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

Y. Le Godec, E. Boulard, G. Bromiley, N. Guignot, G. Hamel, J.P. Itié, A. King, M. Mezouar, G. Morard, J.P. Perrillat, J. Phillipe
etc…..

IMPMC
Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie
(Sorbonne University, Paris, France)

Y. Le Godec 30'

Intro Paris-Edinburgh-press

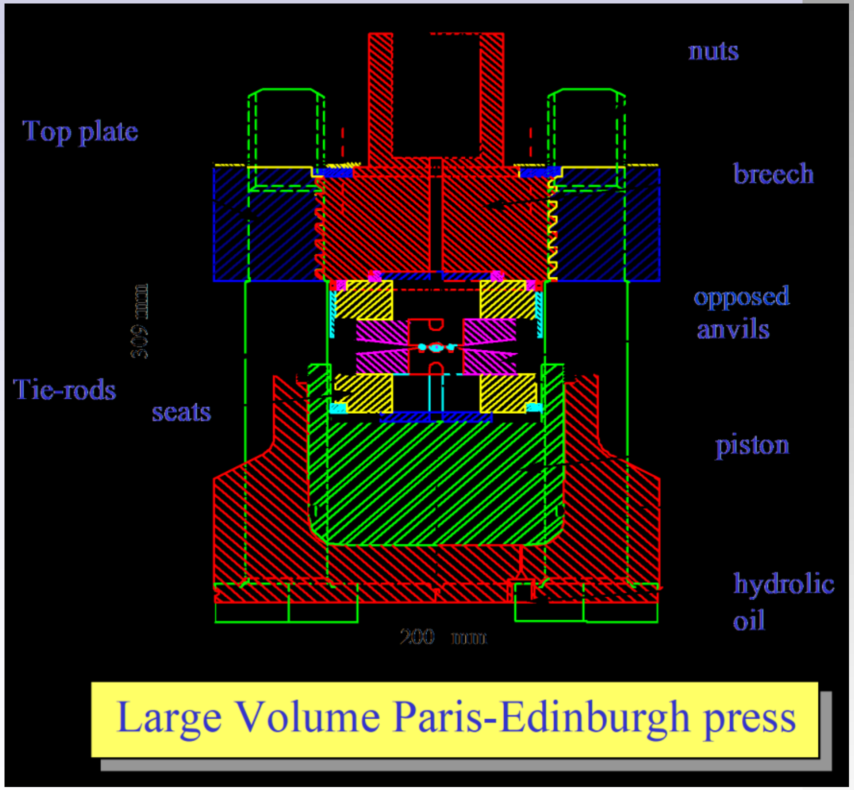
- state-of-the-art
- Recent developments for tomography experiments (Rotopec)

Brief introduction of the Paris-Edinburgh press

Paris-Edinburgh press

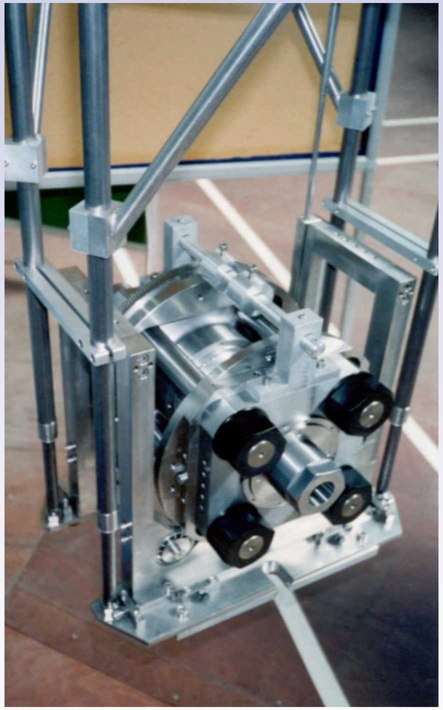
Designed by J.M. Besson, G. Hamel and G. Weill (1991) in France

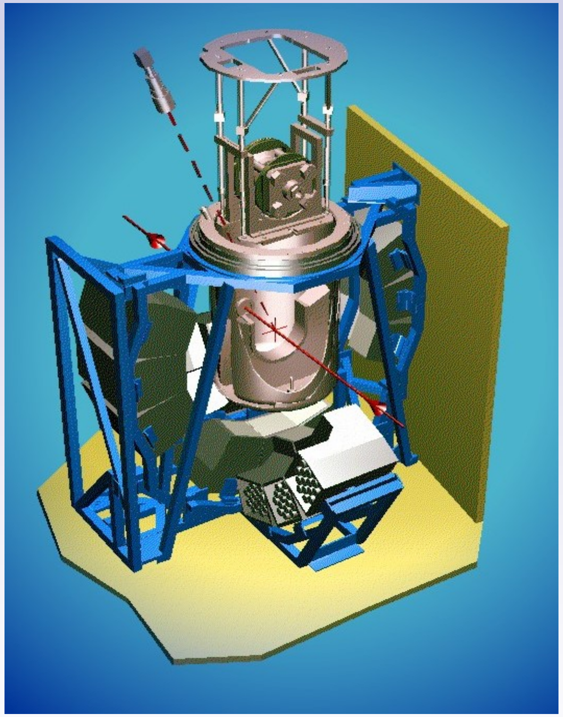
an alternative, in some cases, to the usual very big large volume presses developed in the other countries

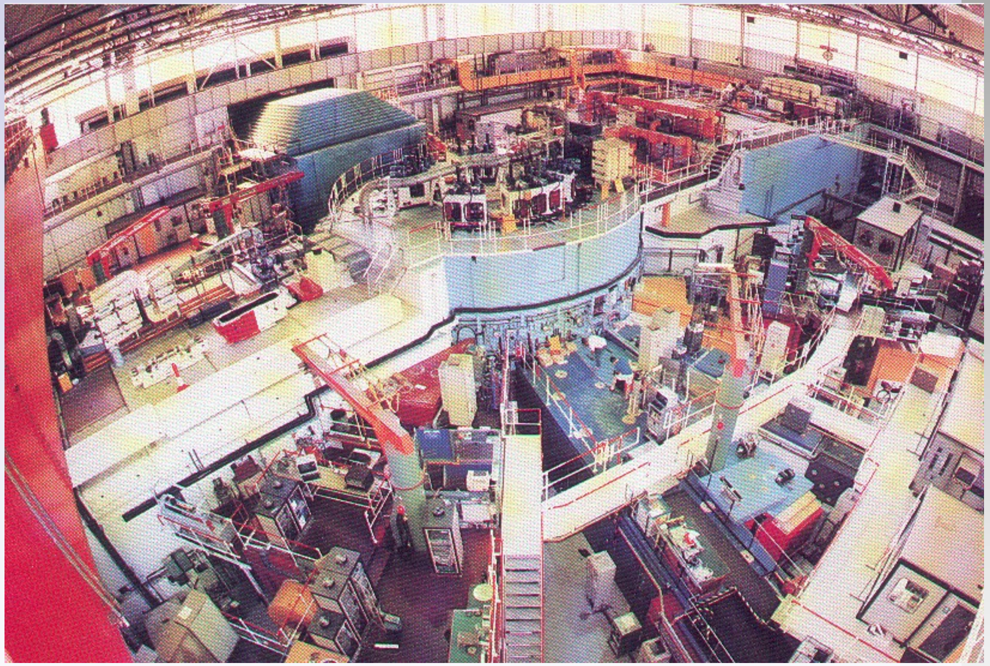


Its originality comes from its hydraulic ram, which is a key breakthrough. It has been very carefully designed (by finite element calculations) to minimize both size and weight. The result is a ram with a 250 tonnes capacity which weighs only 50 kilogrammes and fits into a 30 cm cube

Originally developed for time-of-flight neutron scattering RT experiments at ISIS

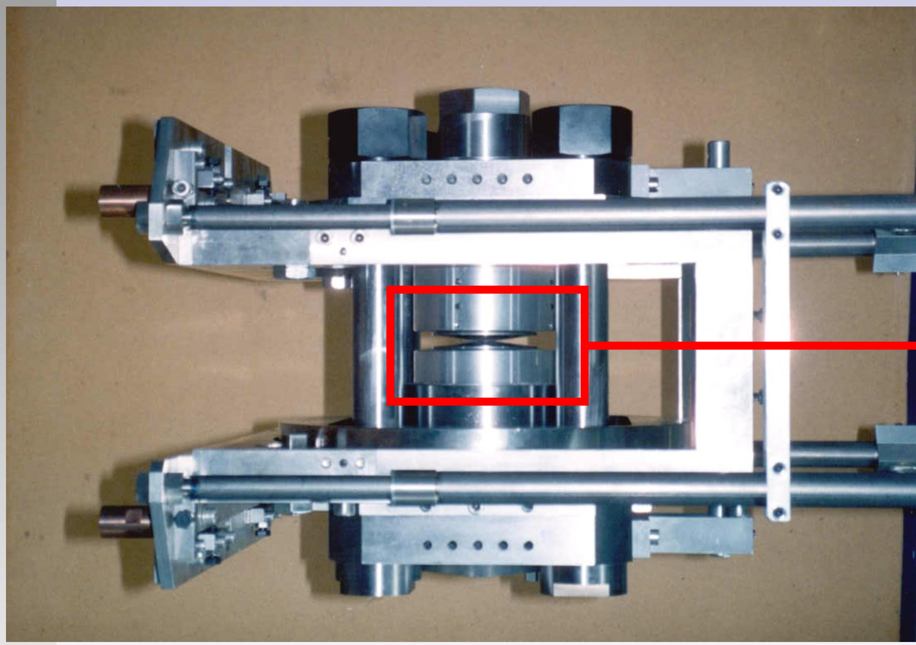




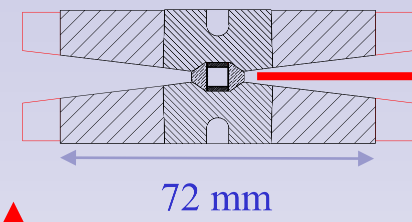


J.M. Besson, R.J. Nelmes, G. Hamel, J.S. Loveday, G. Weill and S. Hull, *Physica (Amsterdam)* **180&181B**, 907 (1992)

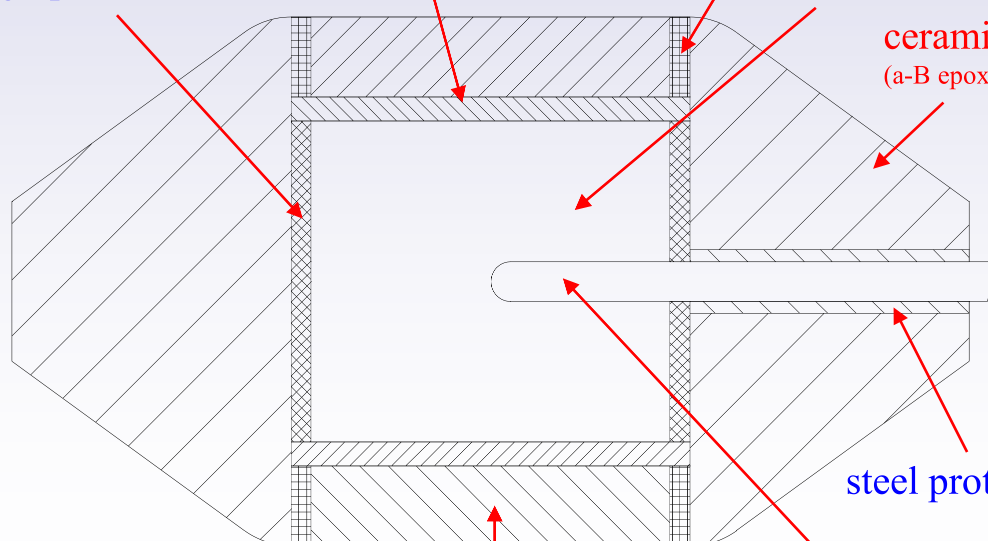
This press has been adapted later for high (P,T) measurements

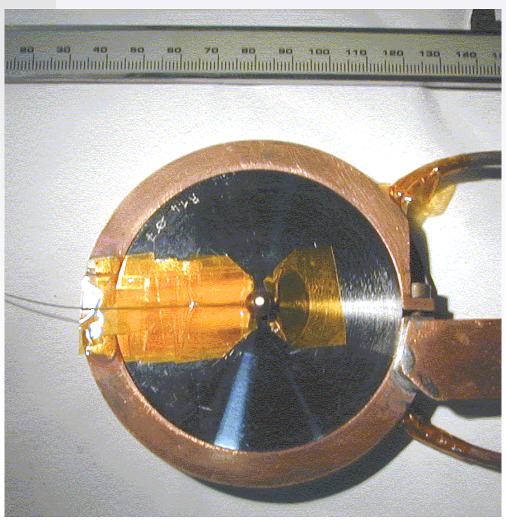


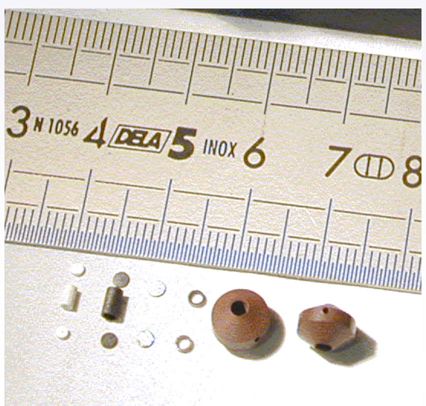
typical set-up



Pseudo-conoidal profile
for anvils
ceramic gasket





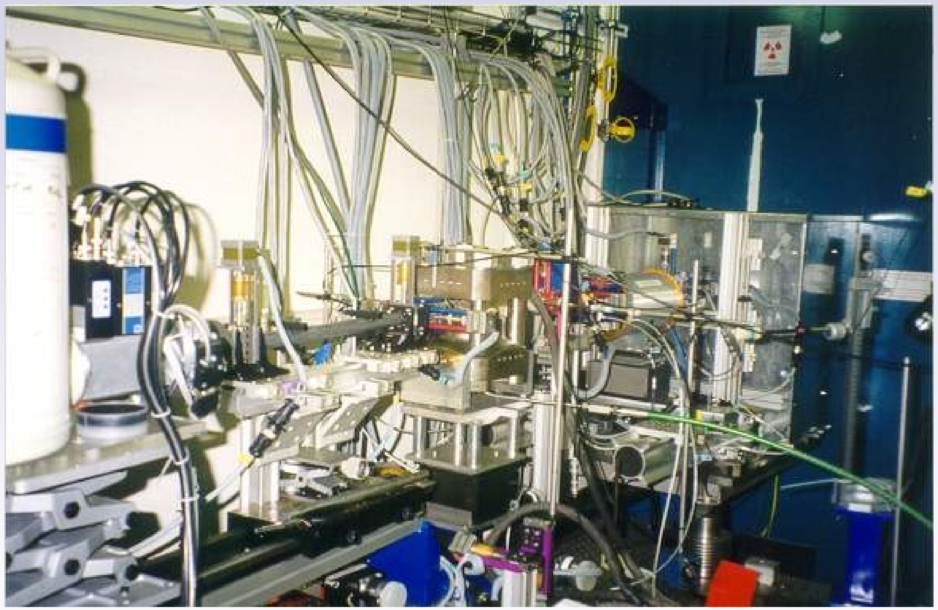


Various parts of the set-up

M. Mezouar, PhD thesis

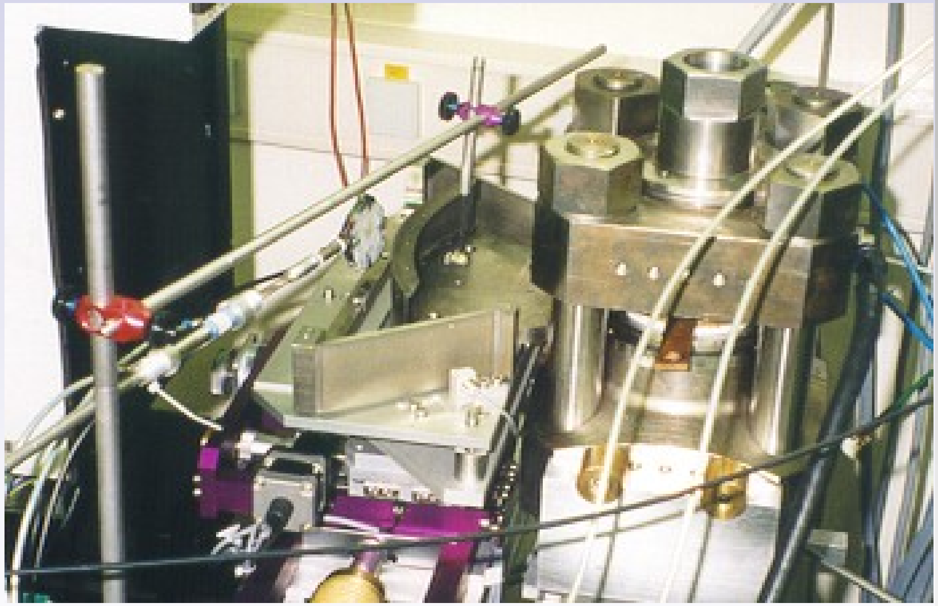
This basic design of the PE press with internal heating has been adapted for a wide range of *in situ* high (P,T) measurements :

X-ray diffraction (LURE and ID30, ID27, ESRF)



Energy-dispersive mode

P. Grima, A. Polian, M. Gauthier, J.P. Itié, M. Mezouar, G. Weill and J.M. Besson *J. Chem. Solids*. **56**, 525 (1995)

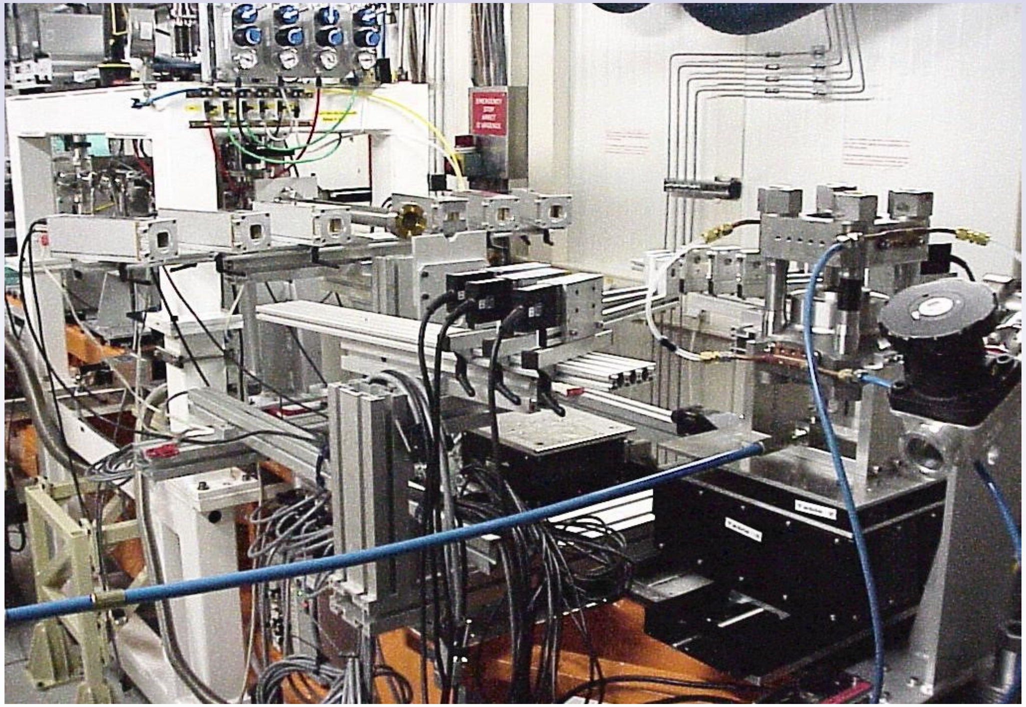


Angle-dispersive mode

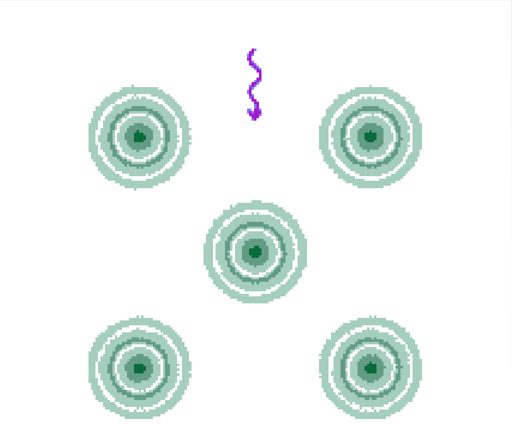
M. Mezouar, T. Le Bihan, H. Libotte, Y. Le Godec and D. Häusermann, *Journal of Synch. Rad*. **6**, 1115 (1999)

This basic design of the PE press with internal heating has been adapted for a wide range of *in situ* high (P,T) measurements :

EXAFS (BM29, ESRF)

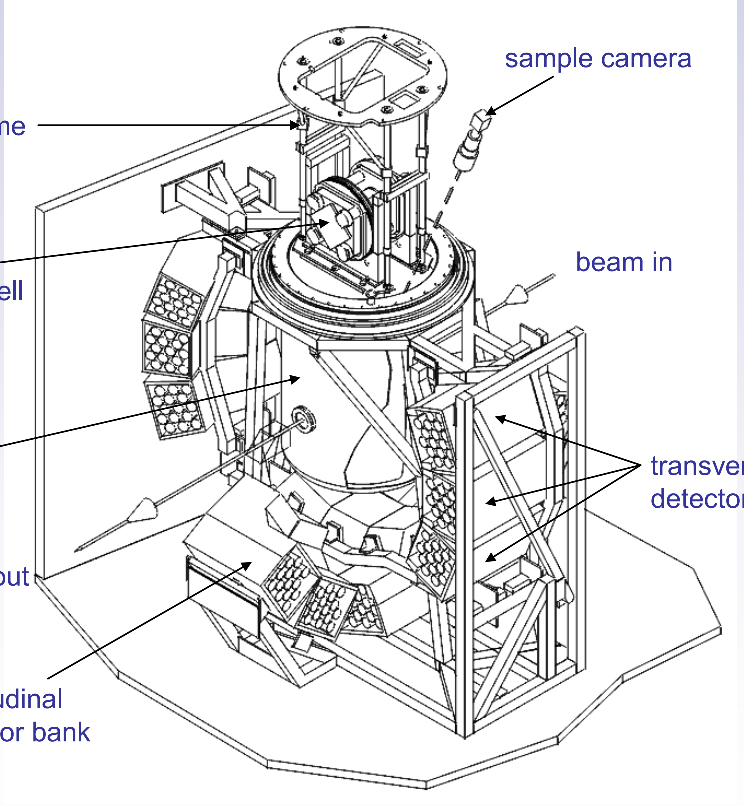


Y. Katayama, M. Mezouar, J.P. Itié, J. M. Besson, G. Syfosse, P. Le Fevre, and A. Di Cicco,
J. Physique IV, Colloq. **7**, C2-1011 (1997)



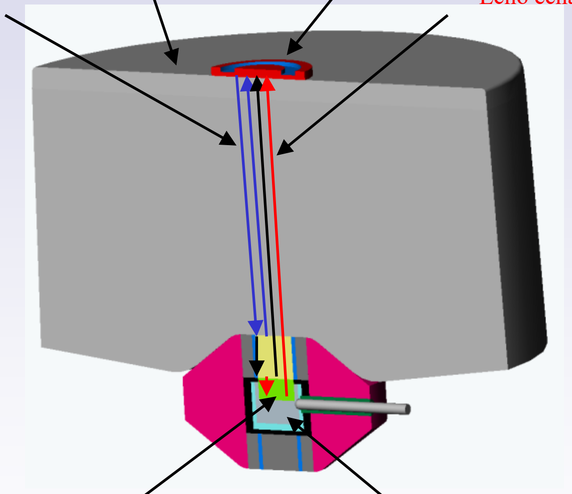
This basic design of the PE press with internal heating has been adapted for a wide range of *in situ* high (P,T) measurements :

Neutron diffraction (Pearl, ISIS)



Y. Le Godec, et al *High Pres. Res.*, **21**, 263. (2001)

Ultrasonic studies (IMPMC)

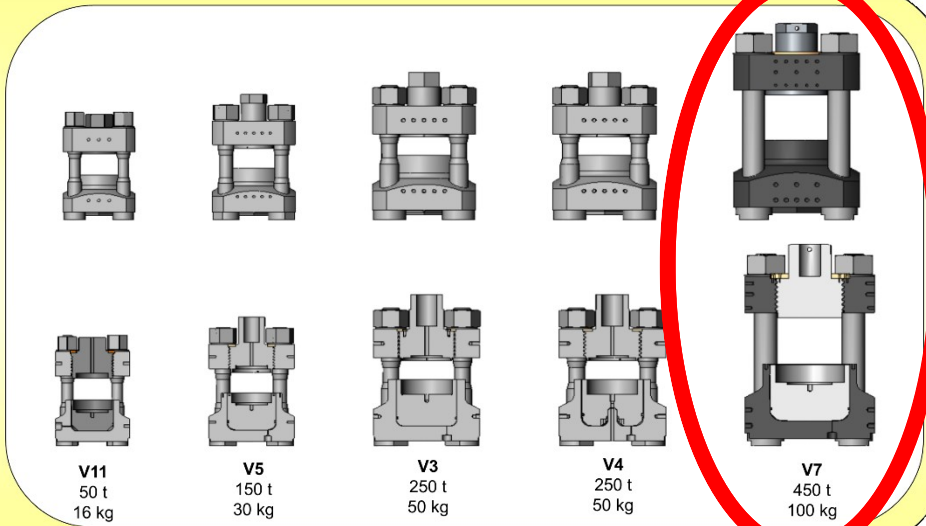


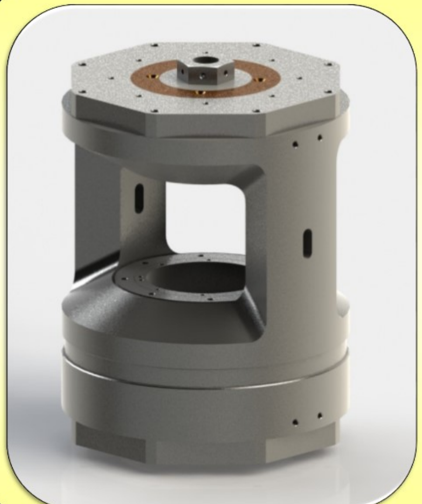
R. Debord, D. Leguillon, G. Syfosse, M. Fisher, *High Pres. Res.*, **23**(4), 451. (2003)

Type V :

4 colomns

J.M. Besson *et al.*, Physica 180&181B, 907 (1992).

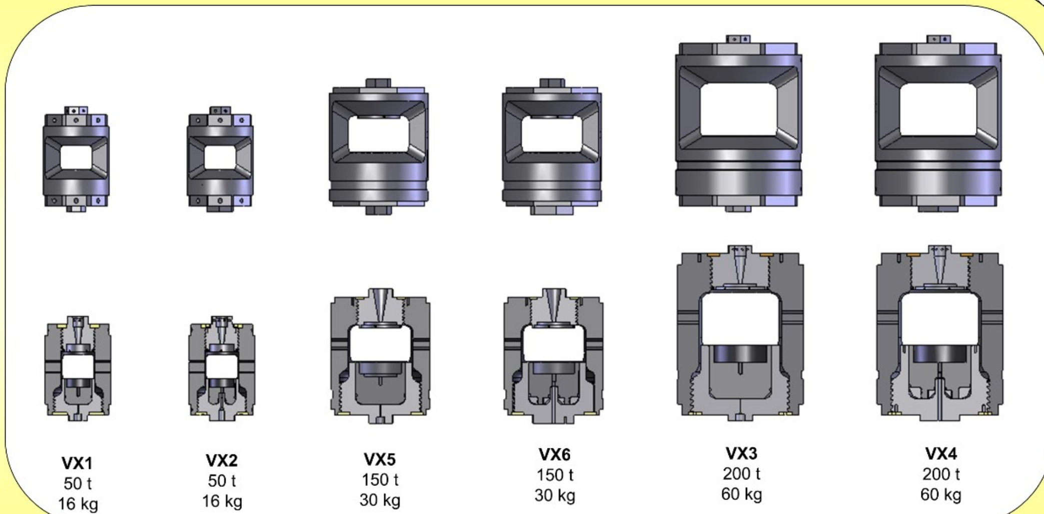




Type VX :

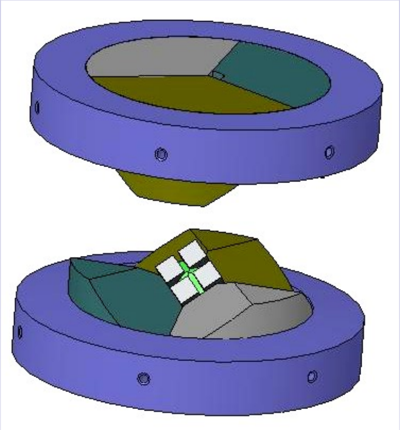
2 colomns

S. Klotz, et al. High Press. Res., 24, 219–223 (2004).

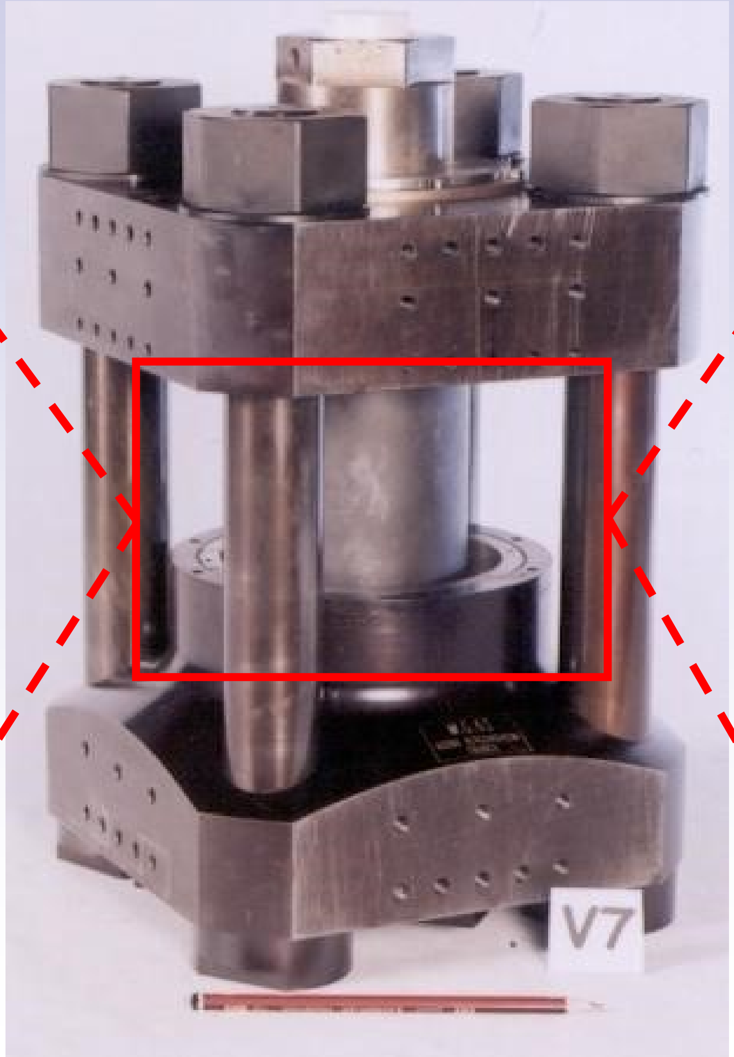


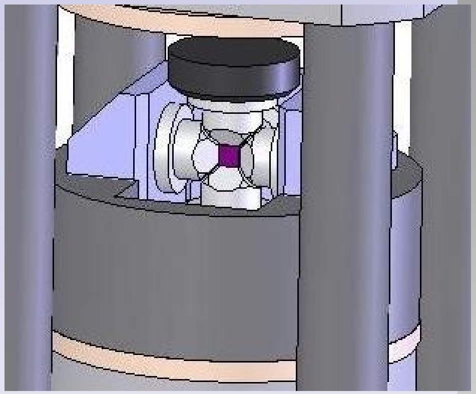
To show the modularity of this press to various techniques, I will continue this talk with the V7, the biggest press of the PE Press family

During the last years we developed various modules that can be easily and quickly adapted into the V7/PE press in order to perform various *in situ* or *ex situ* experiments

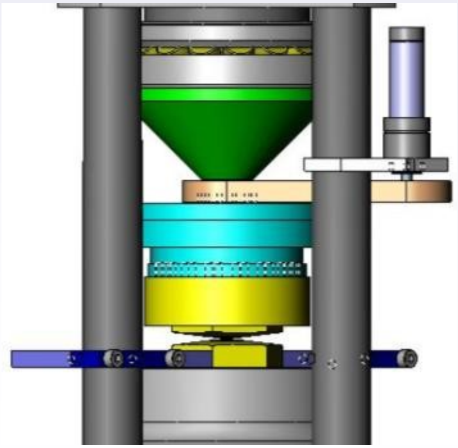


T-cup

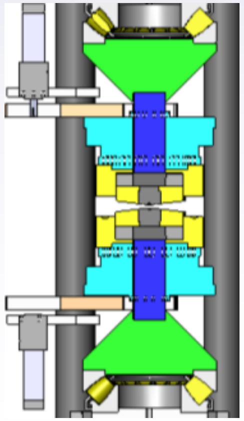




DIA



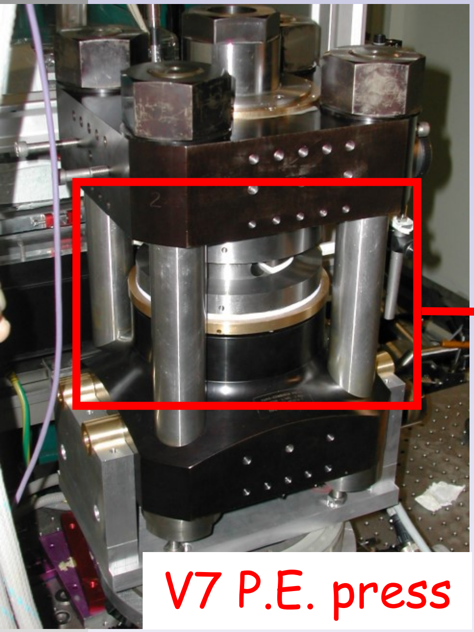
roPEc



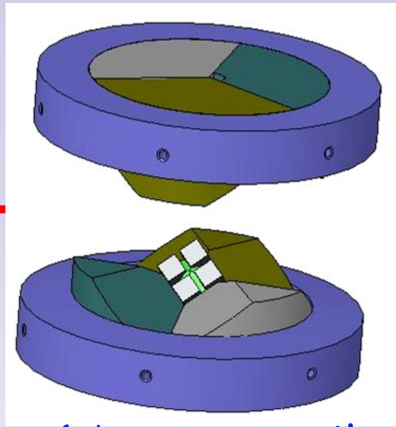
Ro-tomography

LTP HS 2532 (3 years) and LTP 3630 (3 years) at beamline ID27 of ESRF

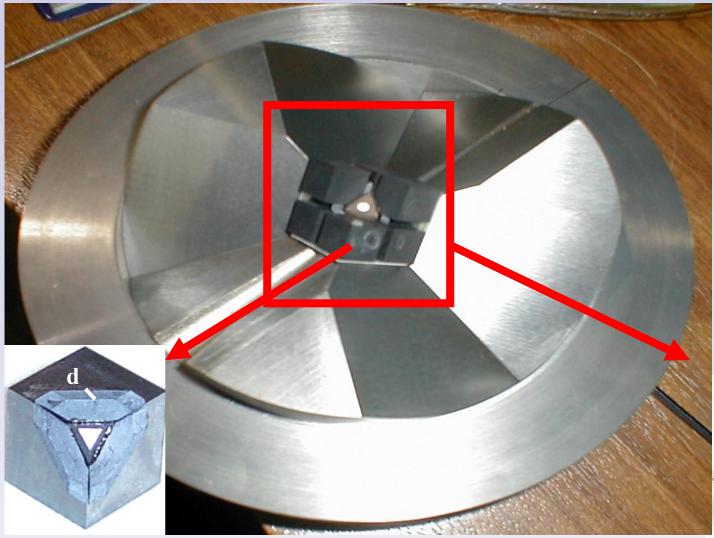
The PE press with T-cup module



V7 P.E. press



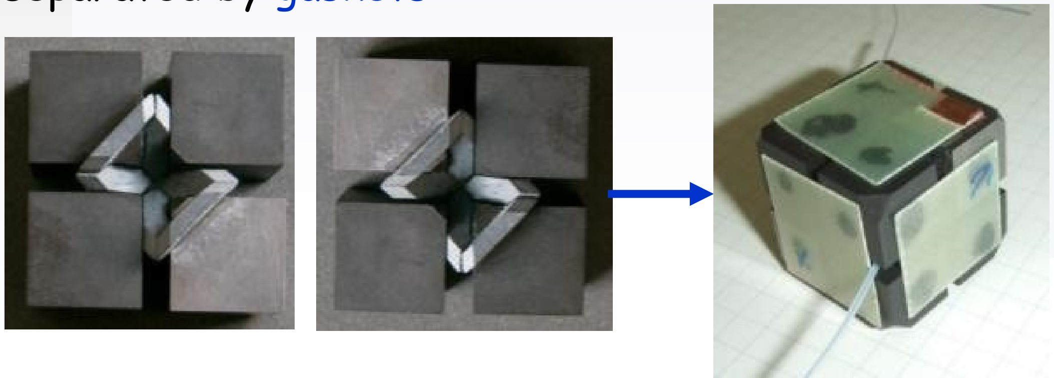
1st stage anvils

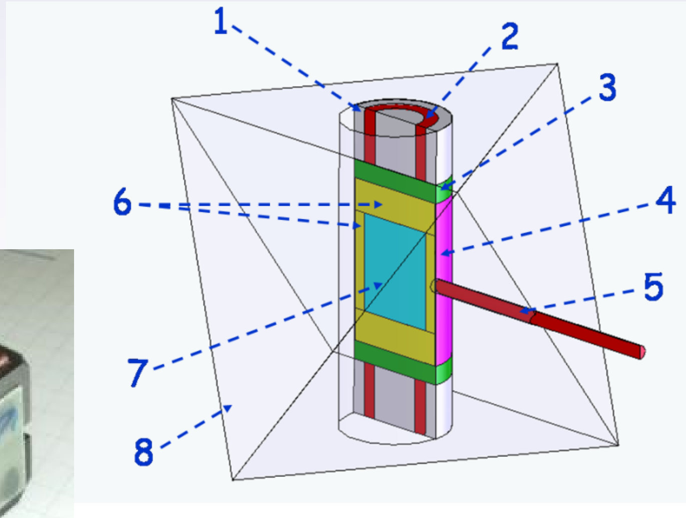


a miniaturized 6-8 KAWAI-type LV device similar to the Stony Brook "T-cup system" which operates routinely to 25 GPa and 2500 K

sample

The second stage consists of **eight c-BN cubes of 10 mm** edge length with a **2 mm truncation** separated by gaskets

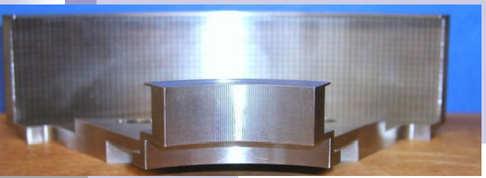




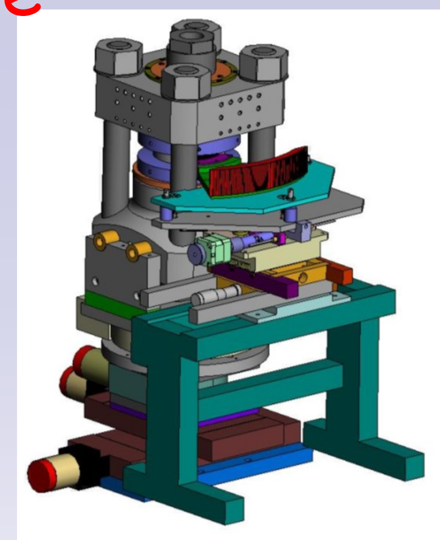
The high pressure set-up is hence a **7 mm amorphous boron epoxy octahedron**

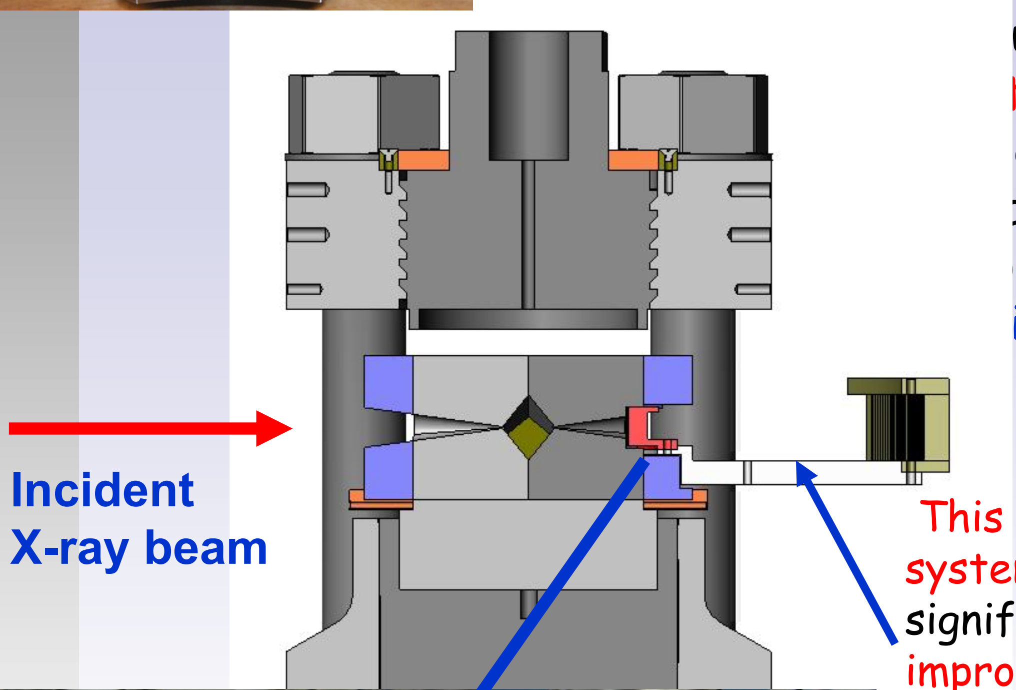
M.T. Vaughan *et al.* *Rev. High Pressure Sci. and Techn.* **7**, 1520 (1998)

The PE press with T-cup module

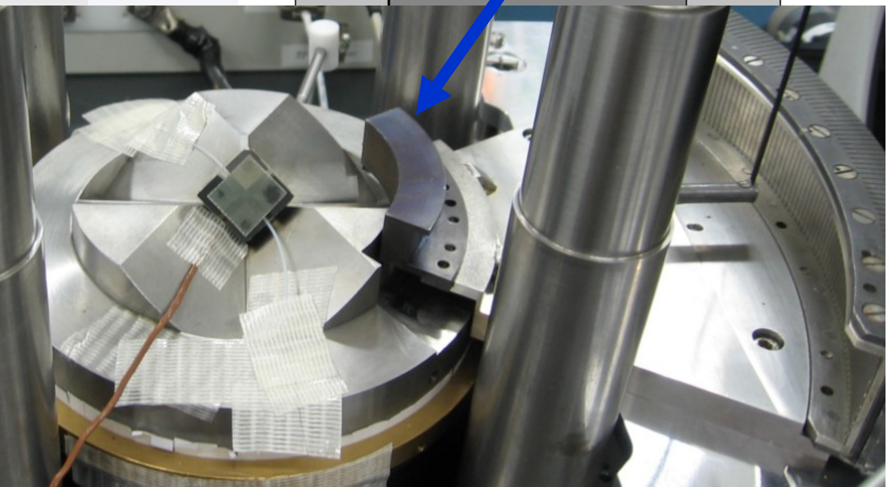


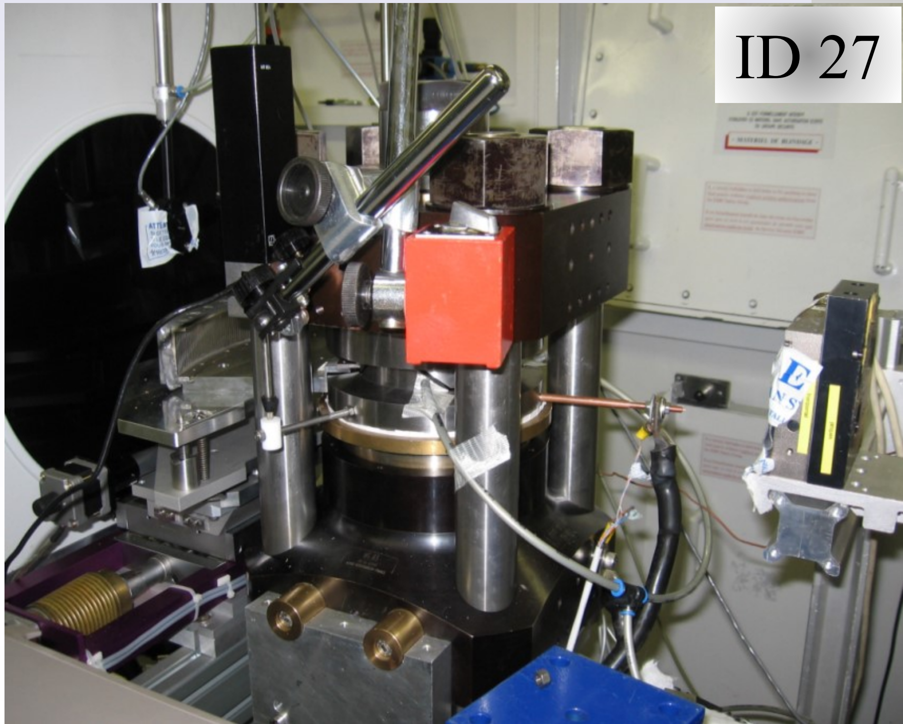
The novel feature of this LV ...us is its extreme ...tness which permits to ...odate optimised Sollers ...and large area CCD ...rs for angle dispersive ...iffraction at ESRF-ID27



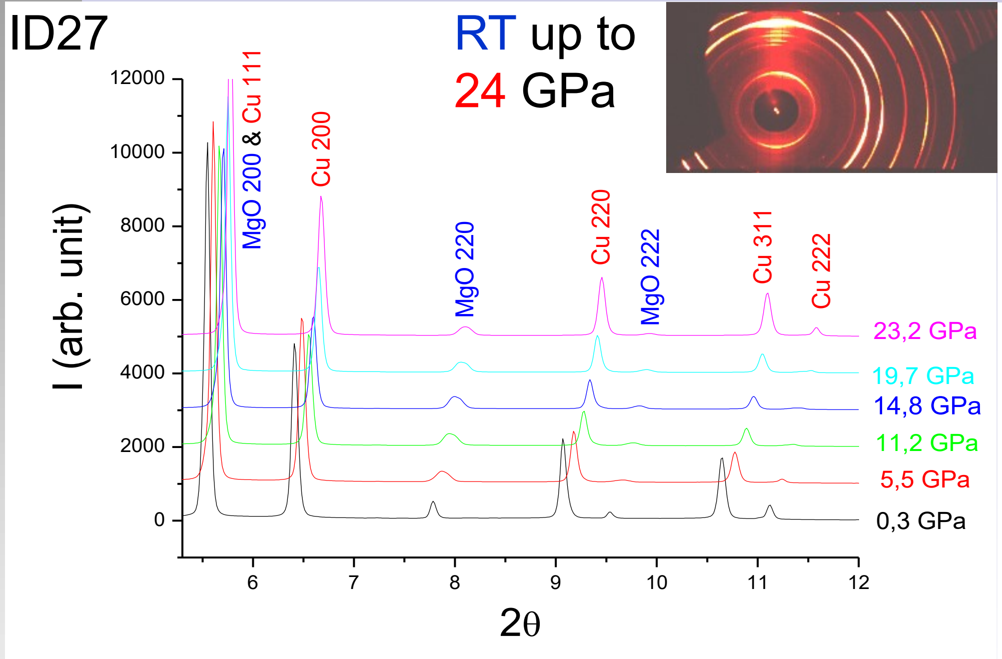


This Soller slit system permits a significant improvement of the signal-to-background ratio and provides clean diffraction patterns of crystalline, amorphous and even liquid samples

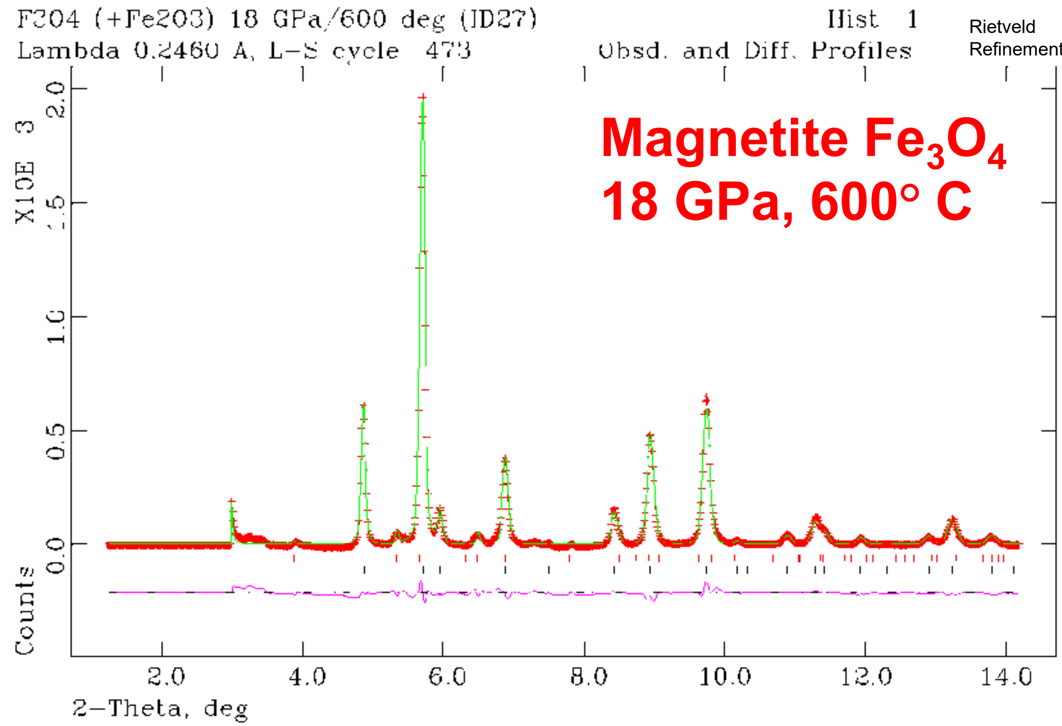




Some typical diffraction patterns



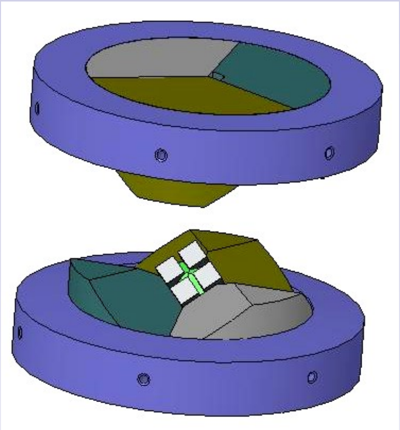
Sample of Copper and MgO



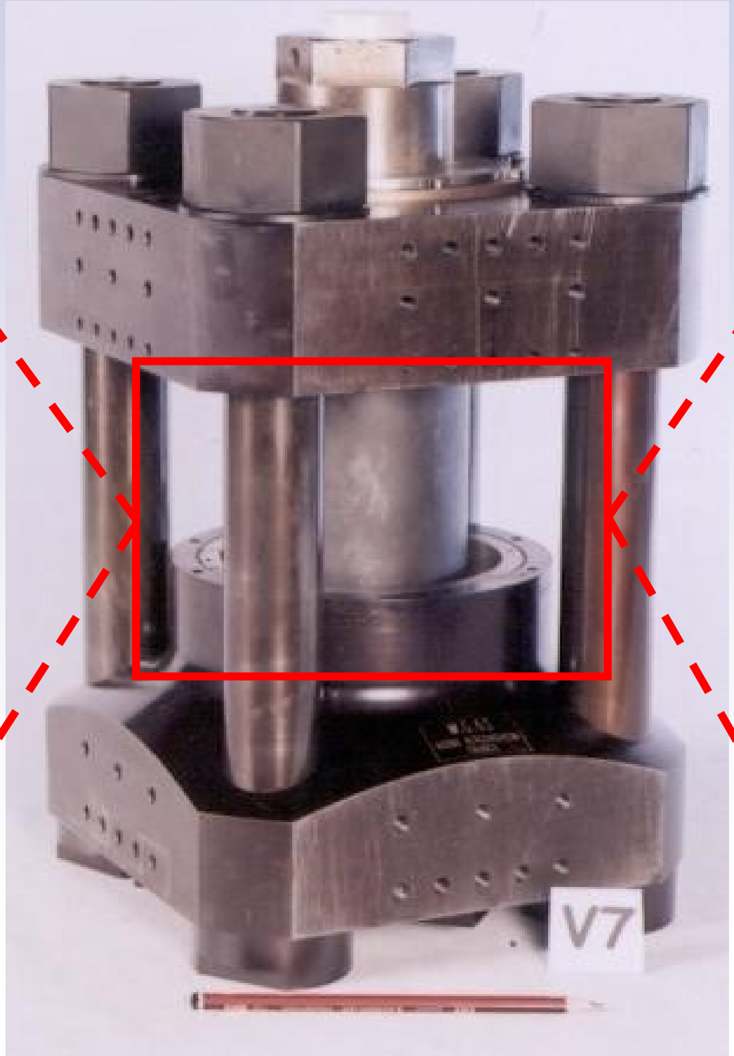
***All** reflections provide from the sample, there is no contaminant signal from the sample environment*

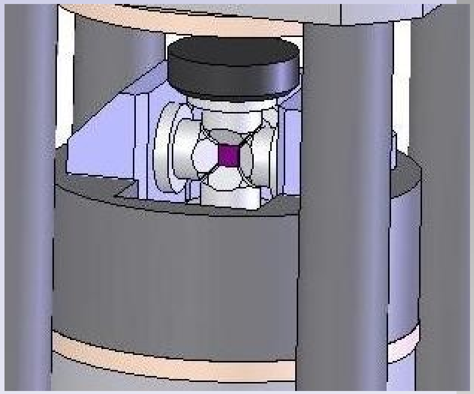
Finally, these experiments showed that the quality of diffraction patterns which can be obtained at HPHT is excellent, and that the data quality is comparable to that obtained with the standard opposed-anvil setup in the Paris-Edinburgh press

The V7/T-cup set-up with this ~ 20% larger overall dimensions allows to accommodate very easily and quickly some other interesting HP modules

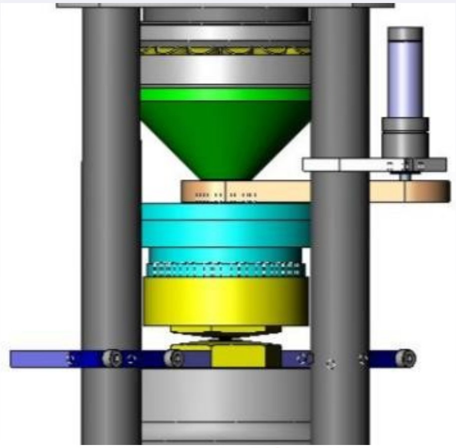


T-cup

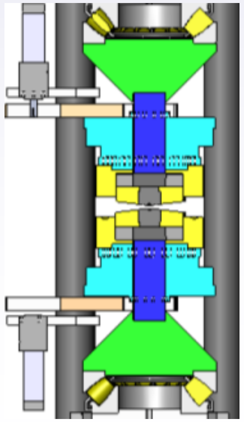




DIA

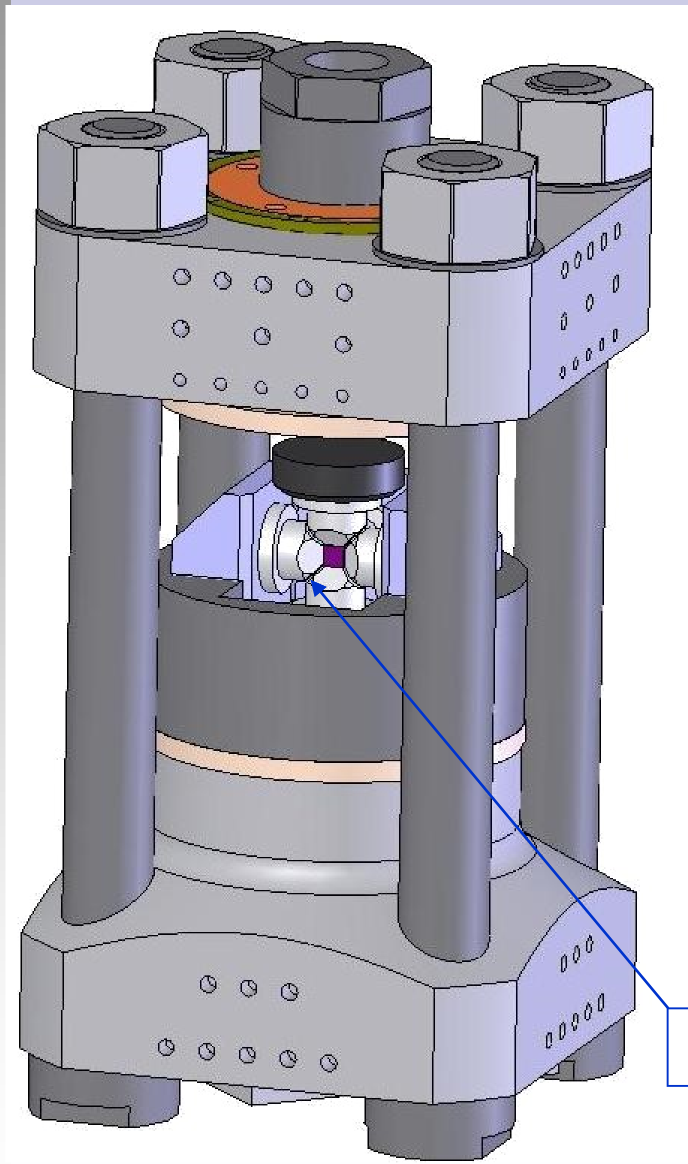


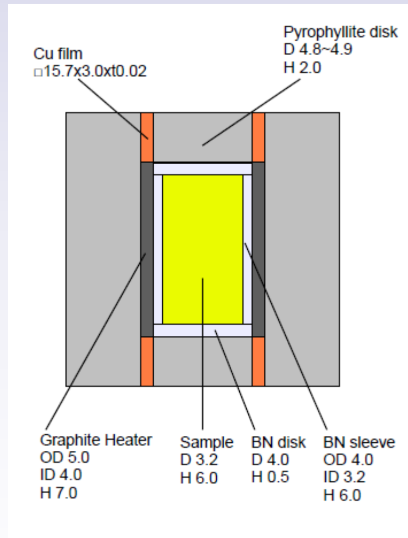
roPEc



Ro-tomography

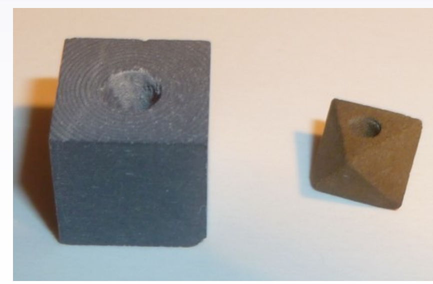
DIA module





a miniaturized DIA module

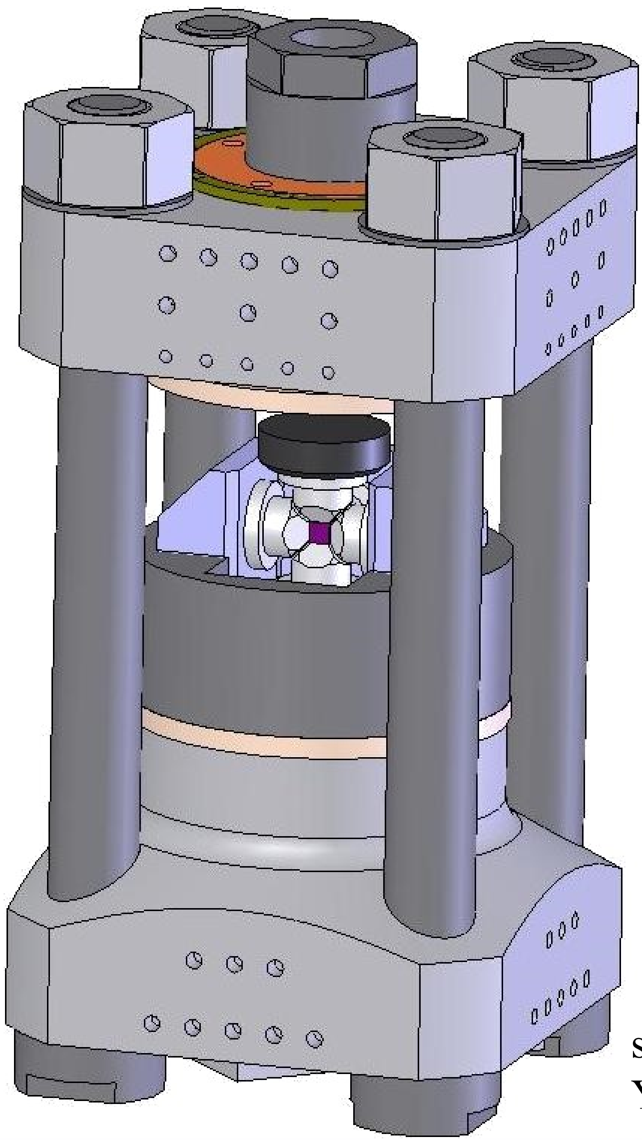
Gasket is cubic



larger sample volume can be achieved

With Prof. A. Gauzzi (IMPMC)

D-DIA press

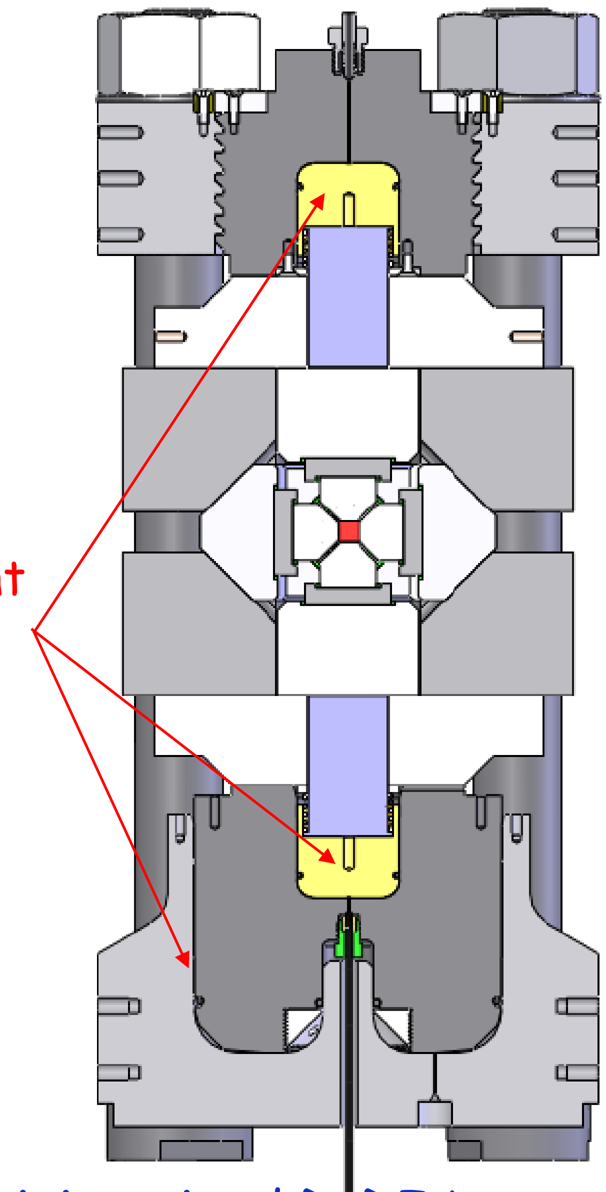


Hence, in the DIA configuration, one pair of opposing anvils can be moved independently from the other anvils

Four miniaturized displacement transducers will be used to measure the displacements of anvils during deformation

similar to the system of
Y. Wang, Rev. Sci. Instrum. 74, 3002 (2003)

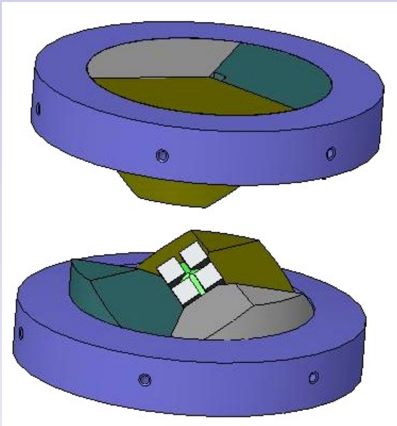
Another possibility



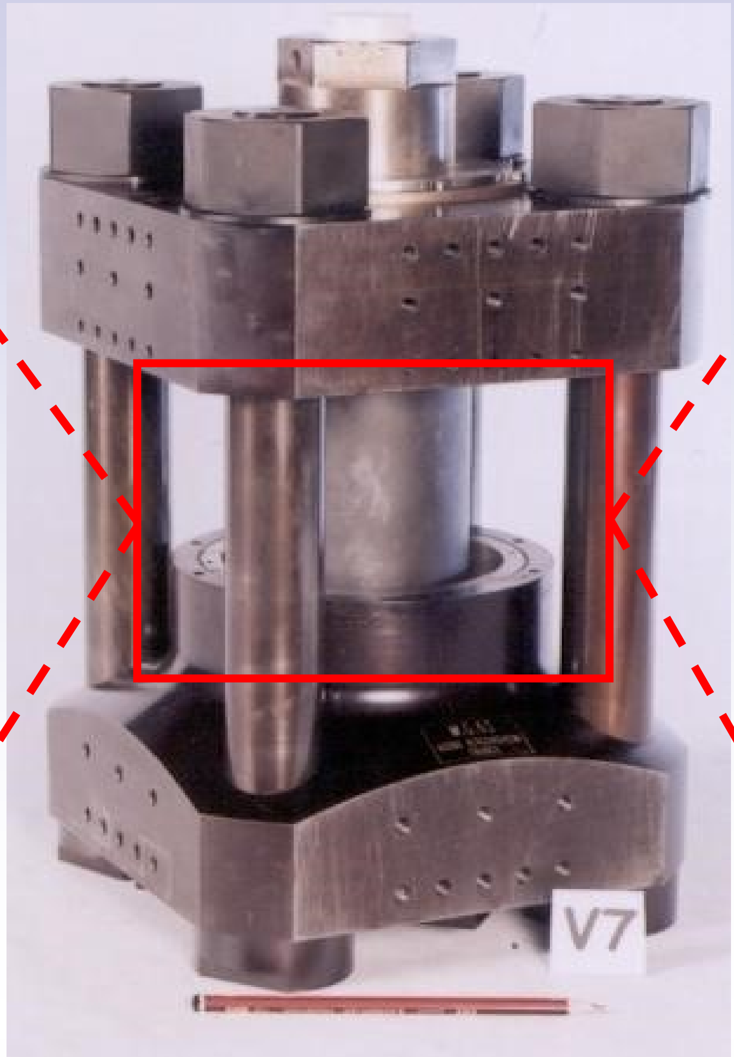
miniaturized D-DIA press

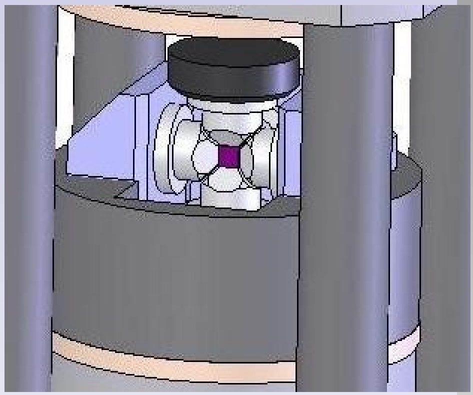
JP. Perrillat, Y. Le Godec *et al.*, Project supported by CNRS

The V7/T-cup set-up with this ~ 20% larger overall dimensions allows to accommodate very easily and quickly some other interesting HP modules

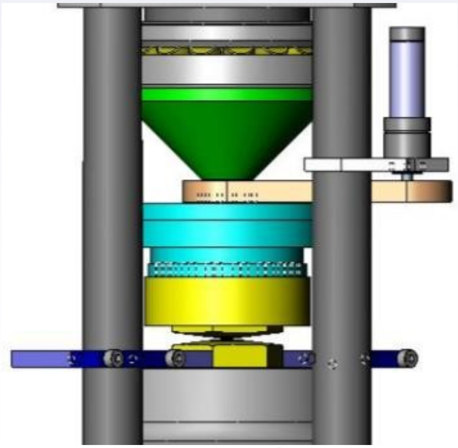


T-cup

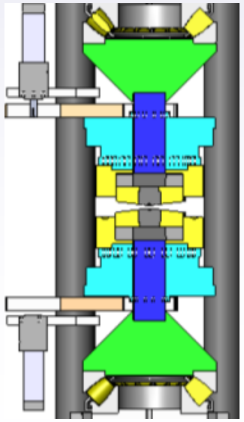




DIA

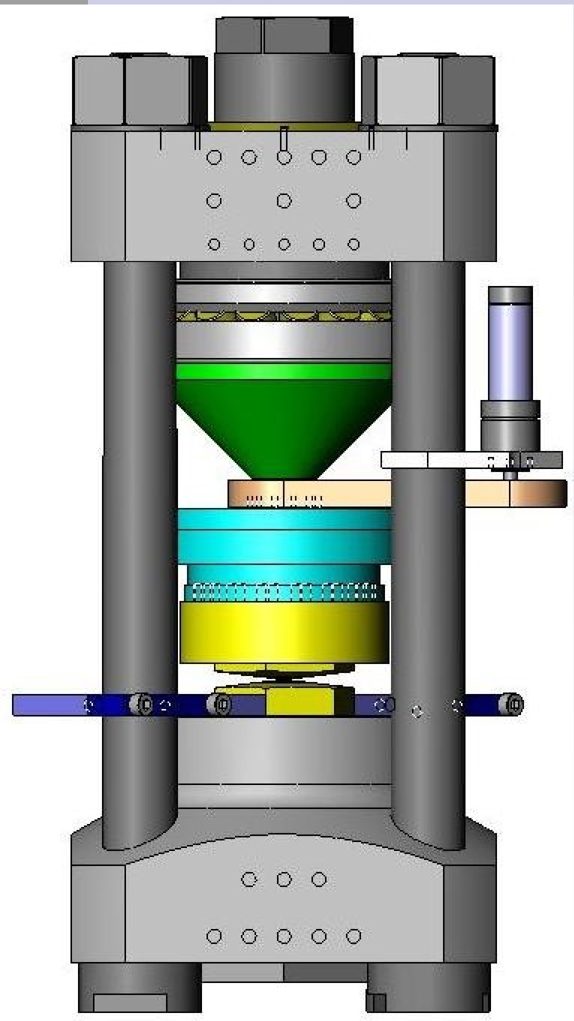


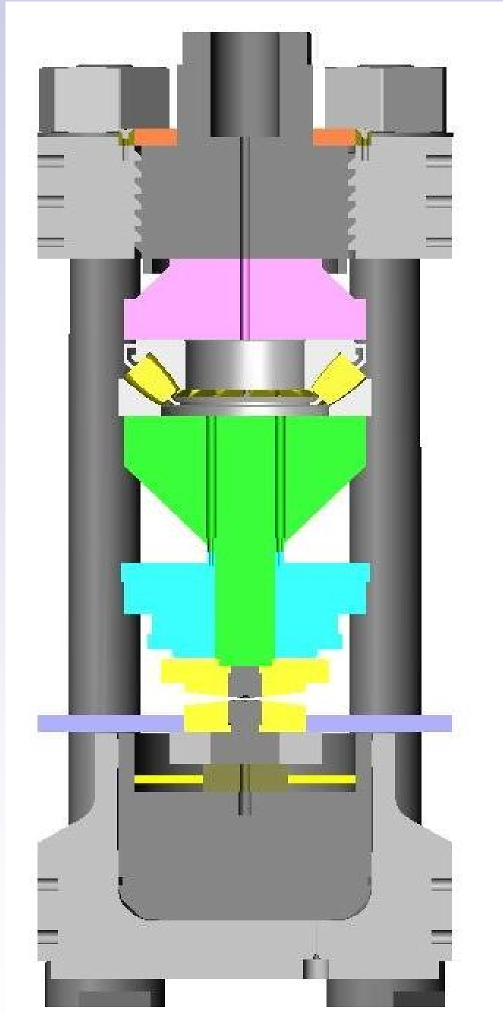
roPEc



Ro-tomography

roPEc module : Torsional deformation under simultaneous high (P,T) conditions (can also synthesize new materials in these unusual conditions !)





A rotational actuator attached to the upper anvil (while the lower anvil is fixed) allows deformation of the sample in simple shear geometry (torsion) under controlled strain rate

Mechanochemistry studies at HPHT

It is well known that the effect of plastic shear deformation leads to many interesting effects, summarized here :

A significant (by a factor 3 to 10) reduction of pressure transformation for a well known phase transition (PT) and chemical reaction

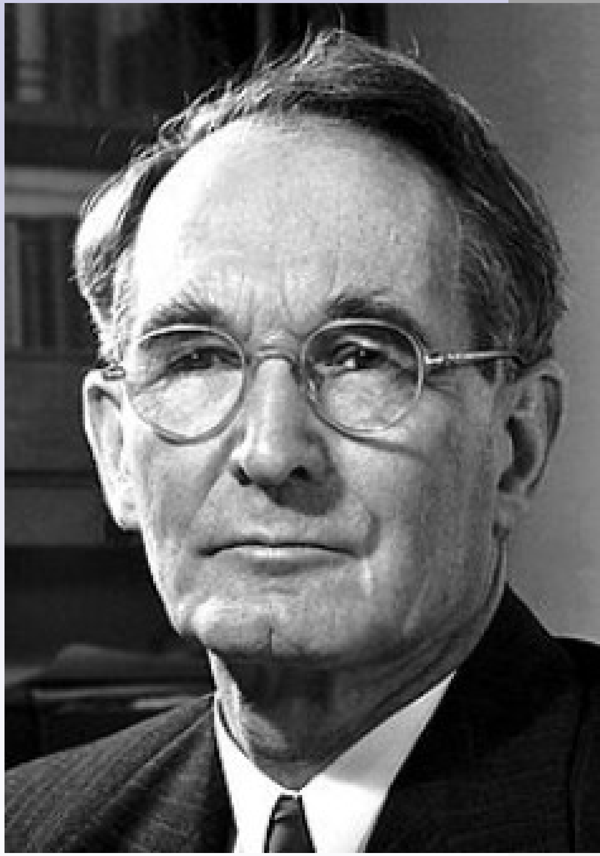
The synthesis of new phases which could not be produced by hydrostatic pressure

New chemical reactions (shear-induced metallization and oxidation, polymerisation)

A substitution of a reversible PT by an irreversible one

A strain-controlled (rather than time-controled) kinetics. It is possible to play with the kinetics of PT

A possiblility to modulate the microstructure and to form bulk nanostructurated materials



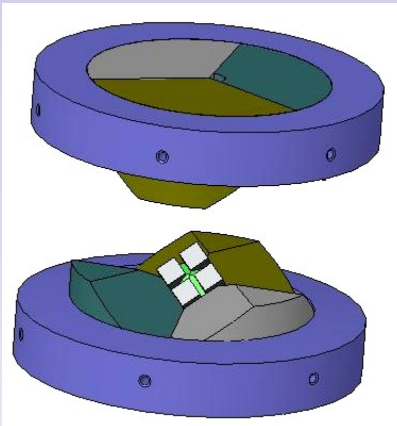
Bridgman (1916)

An old idea but a new unexplored field which could be explored NOW with the roPEc module

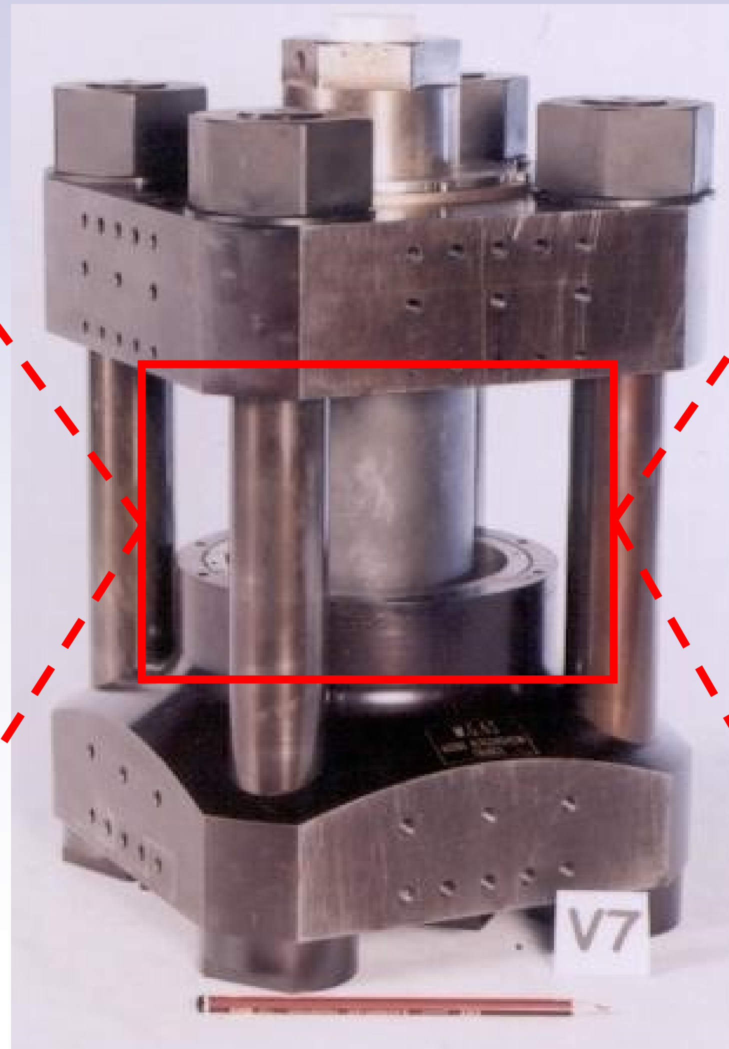
Phys. Rev. 7, 215–223 (1916)

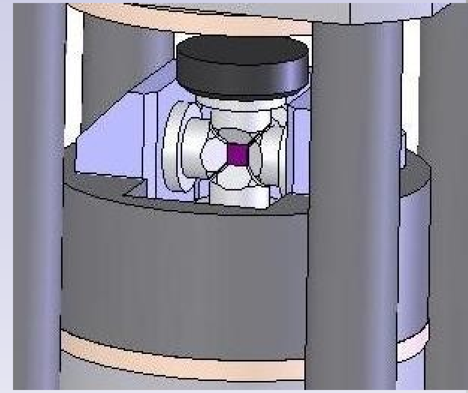
On The Effect of General Mechanical Stress on the Temperature of Transition of Two Phases, with a Discussion of Plasticity

The V7/T-cup set-up with this ~ 20% larger overall dimensions allows to accommodate very easily and quickly some other interesting HP modules

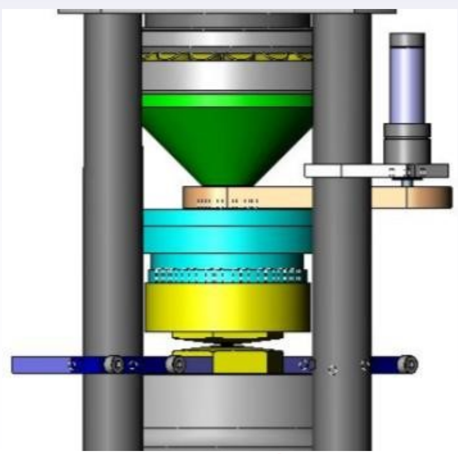


T-cup

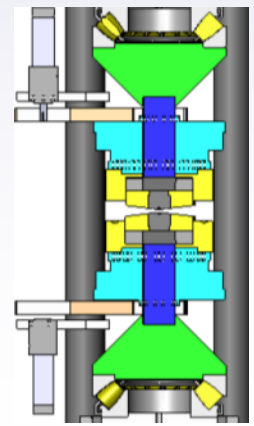




DIA

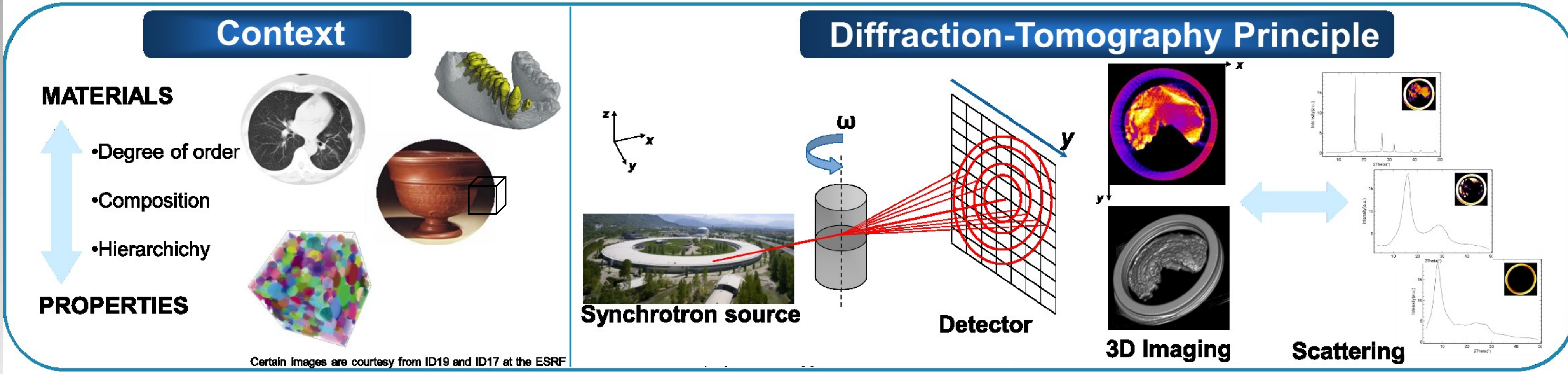


roPEc

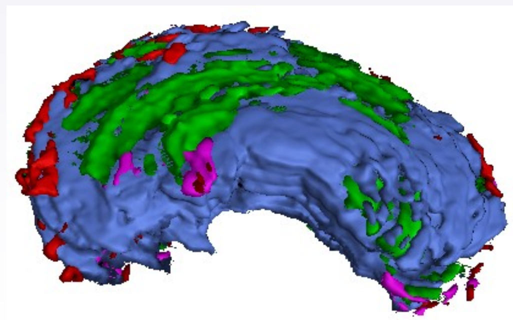


Ro-tomography

SB tomographic imaging is nowadays a powerful technique for non-destructive, high-resolution investigations of a broad kind of materials. High-brilliance and high-coherence third generation synchrotron radiation facilities allow micrometer and sub-micrometer, quantitative, three-dimensional imaging within very short time. Coupling this technique with HP would be very interesting !!!



- Penetrating radiation (X-rays)
- Contrast : absorption, diffraction, fluo
- 2D angular projections
- Reconstruction of 'virtual slices'
- Volume elements (voxels)



Set-up in U.S :
Y. Wang et al., REVIEW OF SCIENTIFIC INSTRUMENTS 76, 073709 (2005)

- 3D probe
- Non destructive
- Phase selective
- High sensitivity
- High spatial resolution

Ex situ tomographic study on recovered samples demonstrated how useful *in situ* high P/T/stress tomographic imaging could be!

- ability to study evolution of textures in multi-phase systems
- time-resolved studies of development of melt networks
- Study of microstructure evolution of composite materials under high pressure, temperature, and during deformation
- Study of strain partitioning in composite materials (strains in the inclusions and the matrix can be determined by mapping the locations and shape change of the inclusions)
- a useful tool in directly measuring volume changes of noncrystalline materials as a function of both pressure and temperature : density of noncrystalline materials at HPHT
- A useful tool for the new “Diffraction-micro-tomography” method to examine *in situ* some minor unidentified phases in high (P,T) synthesised polycrystalline materials. This method could help to precisely determine the local mechanism of high (P,T) synthesis

(Pierre Bleuet et al., *Nature Materials*, 7:468472, June 2008 and M. Alvarez et al., *Applied Crystallography* 44, 163, January 2011 and *Phys. Rev. Lett.* 109, 025502 (2012))

nature materials

Probing the structure of heterogeneous diluted materials by diffraction tomography

PIERRE BLEUET[1], ELÉONORE WELCOMME[2], ERIC DOORYHÉE[3], JEAN SUSINI[1], JEAN-LOUIS HODEAU[3]* AND PHILIPPE WALTER[2]

Physical Review Letters
moving physics forward

PRL 109, 025502 (2012) week ending 13 JULY 2012

“Compressed Graphite” Formed During C_{60} to Diamond Transformation as Revealed by Scattering Computed Tomography

M. Álvarez-Murga,[1,2] P. Bleuet,[3] G. Garbarino,[1] A. Salamat,[1] M. Mezouar,[1] and J. L. Hodeau[2,*]

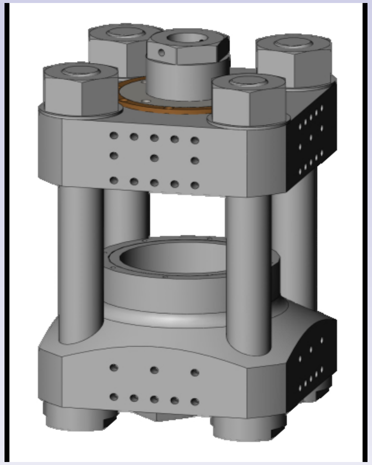
The design of a cell for *in situ* High P/T/stress Tomographies has to satisfy several specific requirements :

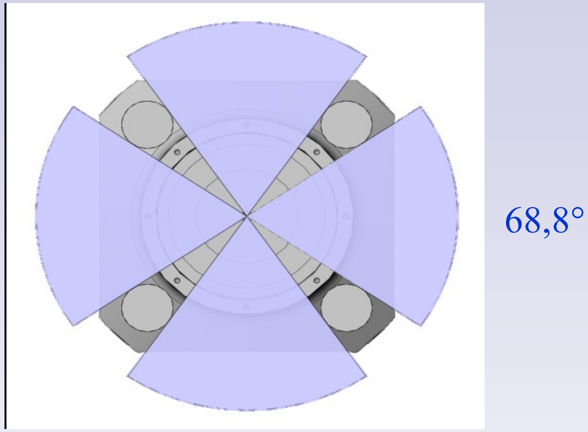
- Large X-rays beams (absorption)
- Monochromatic micro-beams (diffraction)
- Large sample volume (~2 mm^3) to obtain meaningful structural information on microstructure and its evolution
- Angular access of 180° to the sample
- Coaxiality of the anvils, Flatness of sample
- Accurate and simultaneous rotation of the anvils under High Pressure and High Temperature
- Possibility to rotate the anvils in opposite directions (deformation)
- High Energies and high flux
- Transparency of sample environment (absorption)
- Eliminate scattering signal from gasket (diffraction) : compatible with a sollers slits system
- Maintain sample geometry during compression (limit gasket lateral extrusion)
- Easy to handle, portative. The press has to fit on the ID27 Beamline of the ESRF and easily exportable to other beamlines (SOLEIL, DIAMOND)

Paris-Edinburgh presses

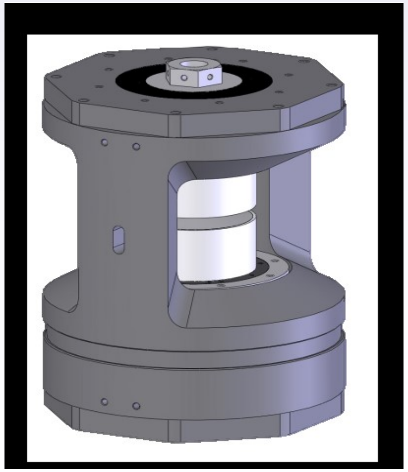
the first simple idea is to rotate the press itself !

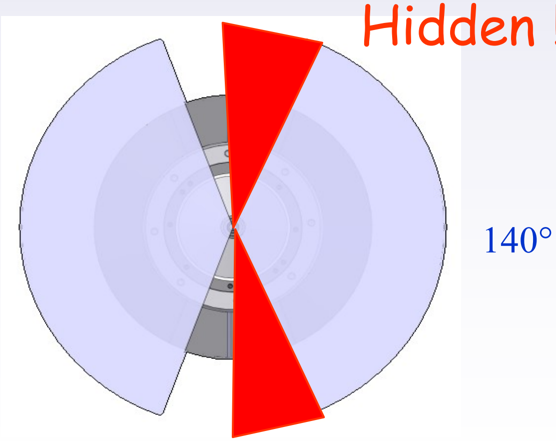
V4 Type
4 columns





VX3 Type
2 columns

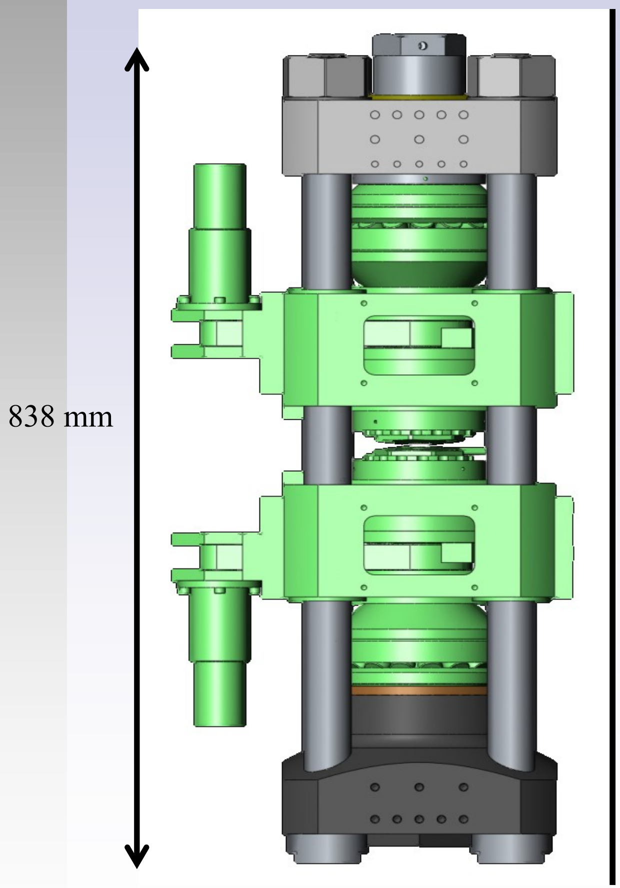


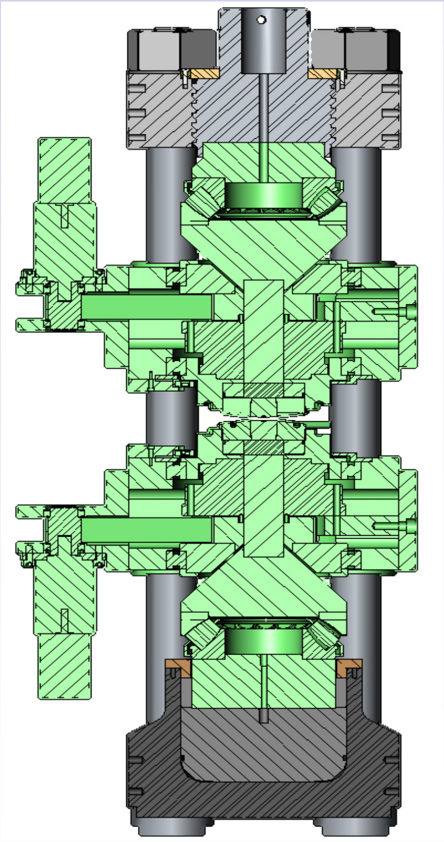


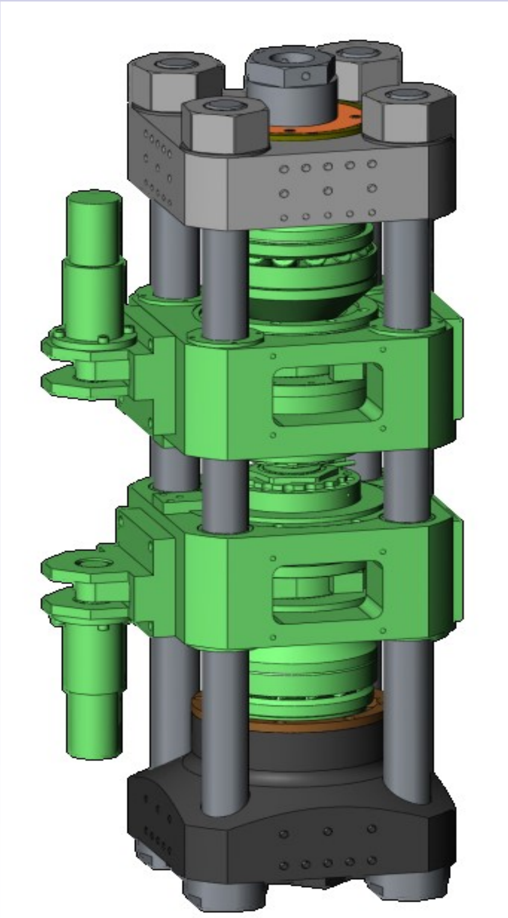
columns are a problem !

it is not possible to scan in excess of 140°. For the tomography scan a minimum rotation of 180° is required. The only solution to this issue is the development of a new system of rotating anvils, which will allow the 180° scanning of the slice

Rotational Tomography
PE module

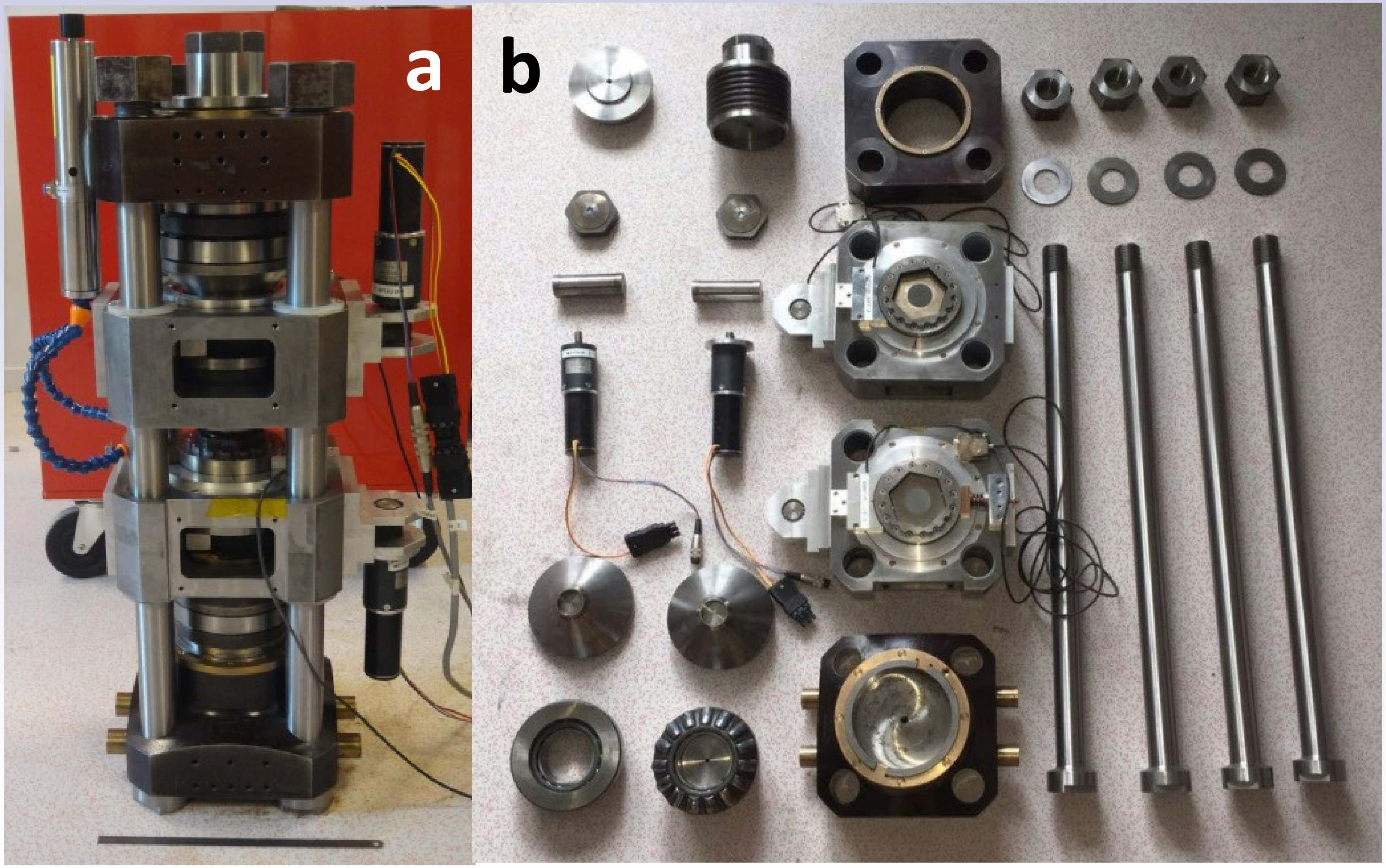






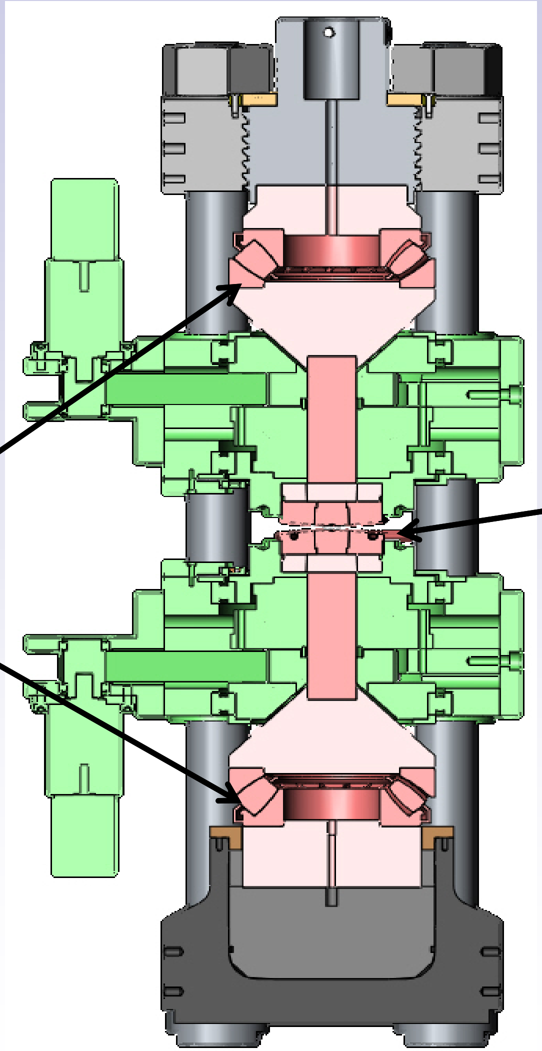
The rotoPEc is easy to install to any beamline (within 20 minutes) and can be removed quickly

Rotational Tomography
PE module



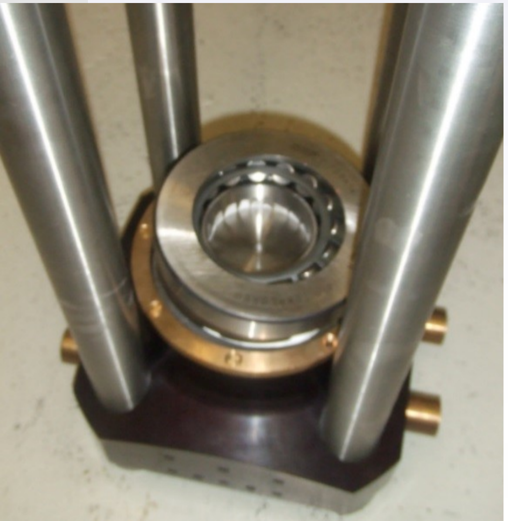
The rotoPEc is easy to install to any beamline (within 20 minutes) and can be removed quickly (10 minutes)

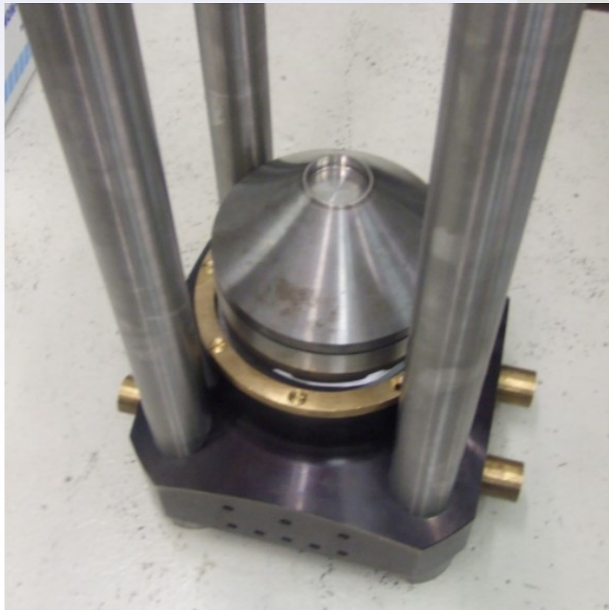
Pressure transmission



2 thrust bearings with a capacity of 125 tonnes

Hexagonal anvils and sample





sufficient to reach 15 GPa in our experiments

Torque transmission

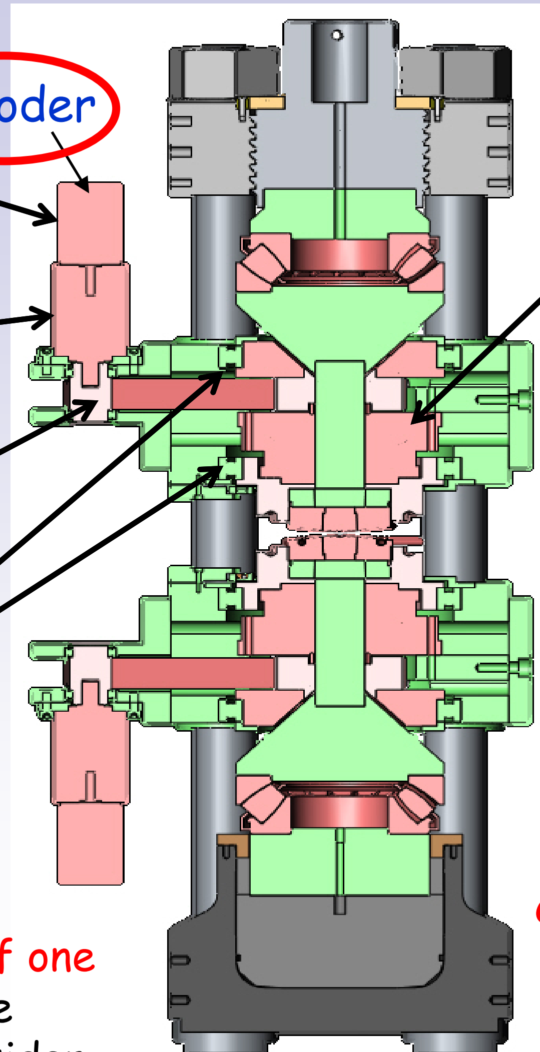
Stepper motors for anvils rotation

encoder

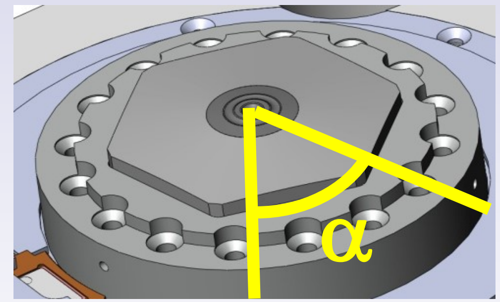
Reduction
100:1

Belt pulley system (with reduction)
3:1

Centering through ball bearings



Gear reducer
Max torque: 892 Nm
160:1



Reduction ratio: 48000:1

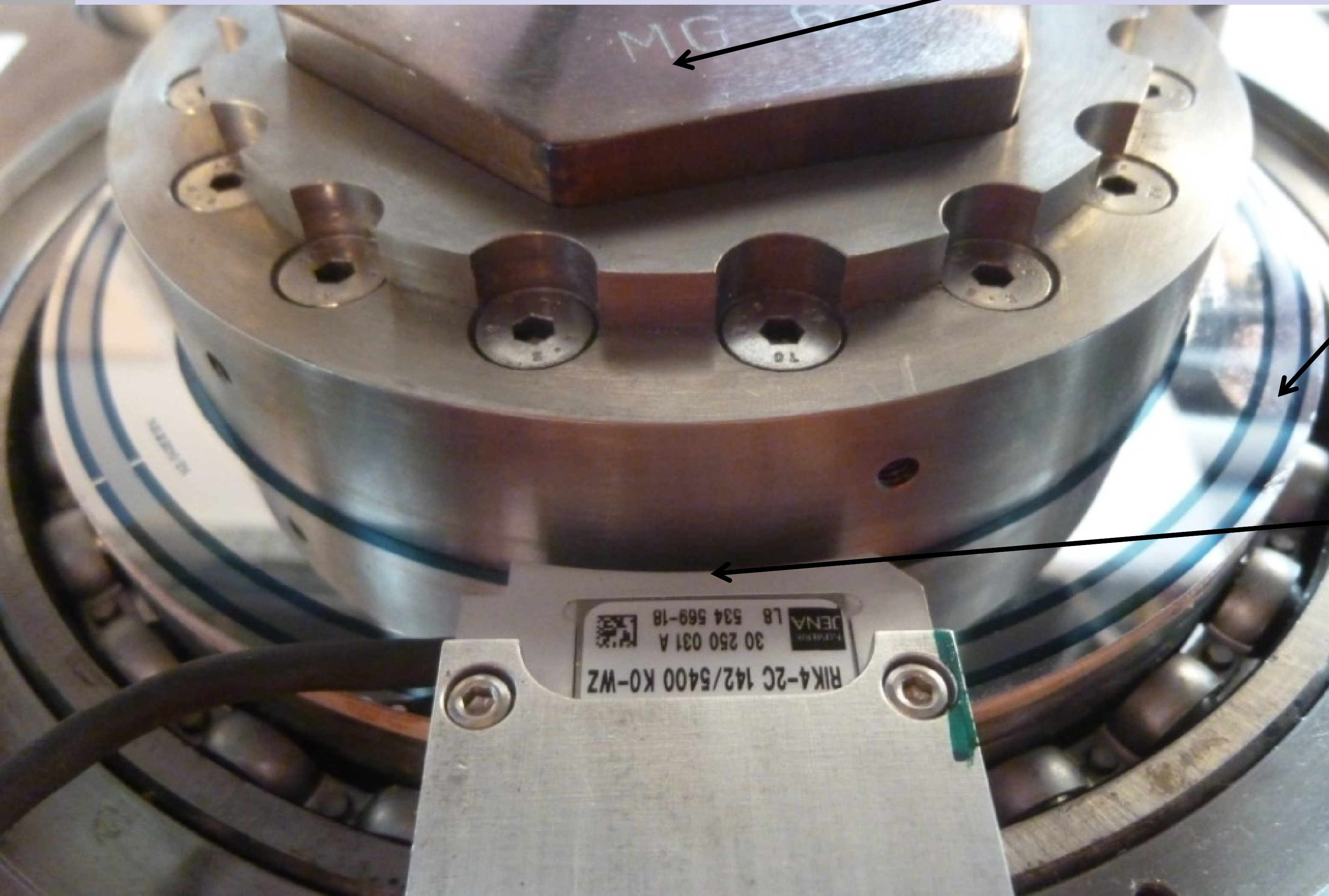
48 000 engine rotations to perform a complete rotation of an anvil (in 9 min with the mean speed of the motor)

To determine the angular position of one anvil, the first idea is to use the encoders of the motors and to consider that the system is perfect (lossless) with the reduction ratio

Accuracy was not sufficient for tomography !
Our idea is to control the motors with two encoders placed directly on the anvils

The idea is to control the motors with two encoders placed directly on the anvils

Hexagonal anvil

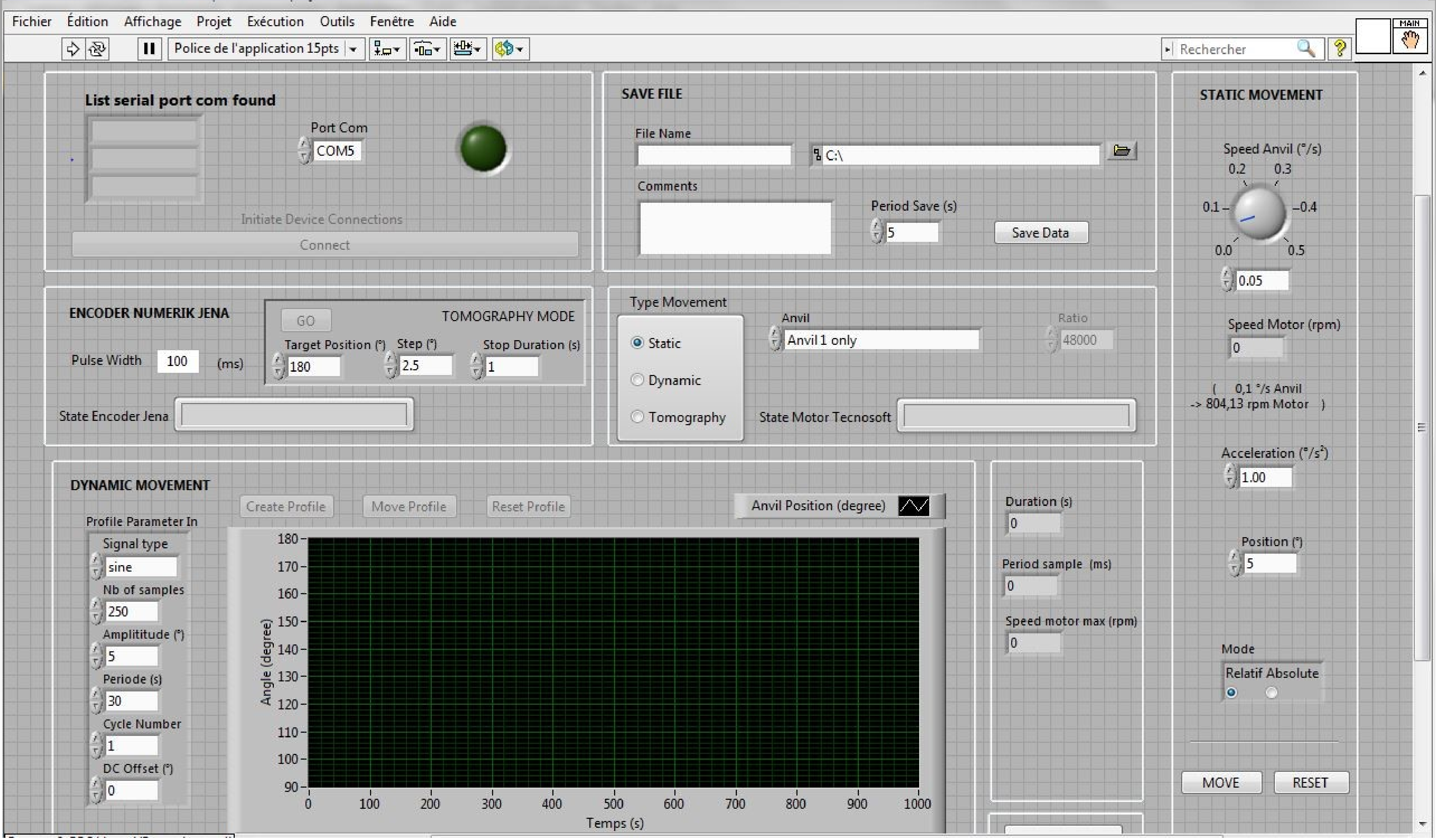


incremental encoder

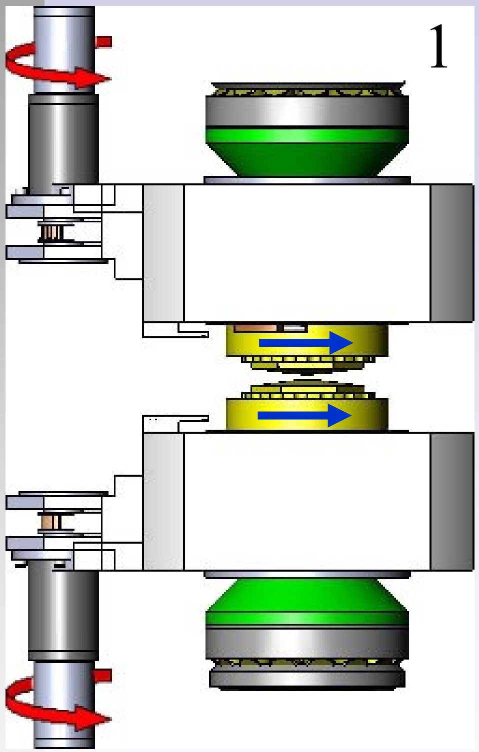
Optical Drive can read the various positions

Resolution 0,005 °

We have developed a software to control the two motors with these incremental encoders in order to monitor the angular position and/or velocity of the anvils

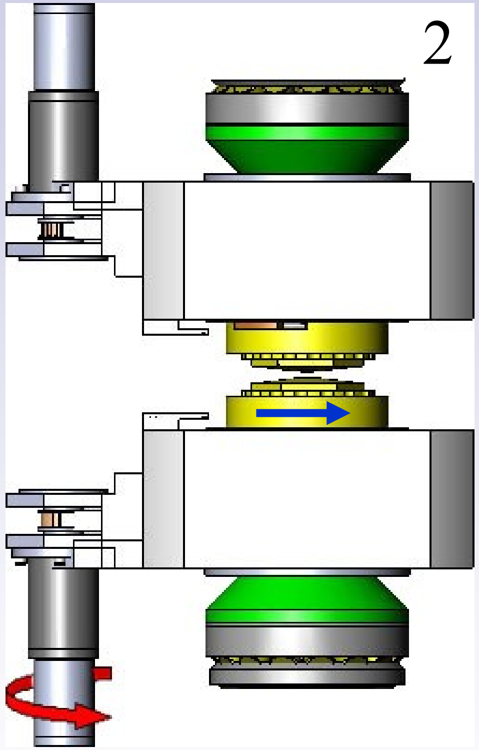


This module can operate under HPHT with 4 modes of operation



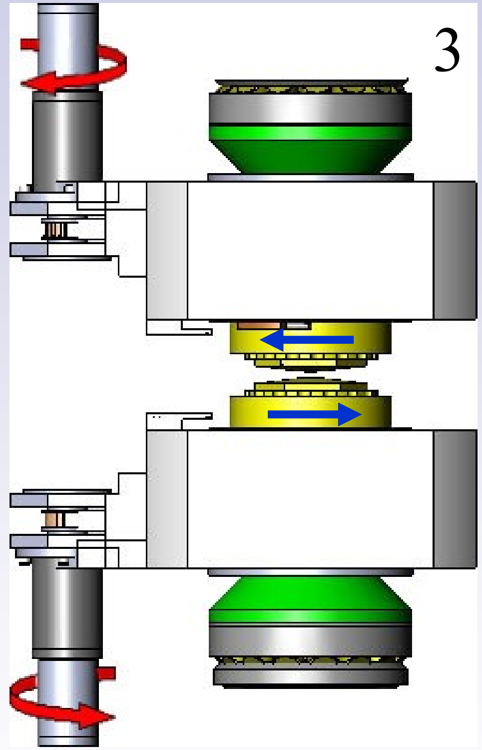
The two anvils rotate in the same direction simultaneously

High P/T tomographies



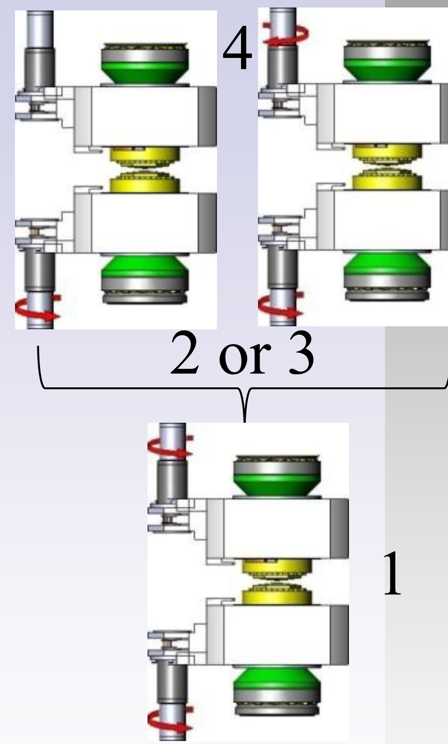
Only one anvil rotates, the other is fixed

High P/T/stress diffraction



The two anvils rotate in opposite directions

High P/T/stress diffraction (fast)

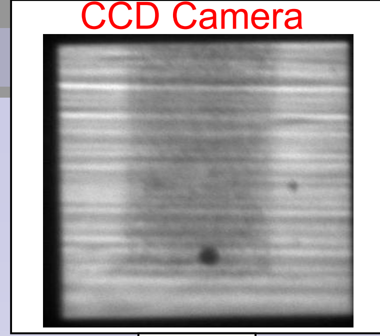


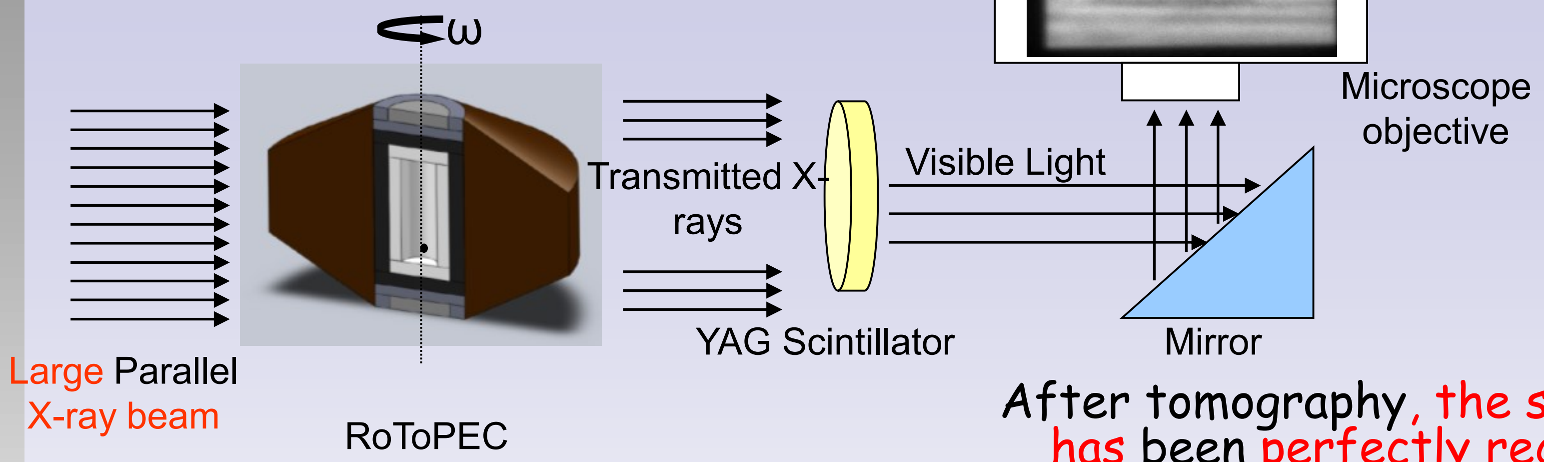
Combination of mode 2 or 3 followed by mode 1

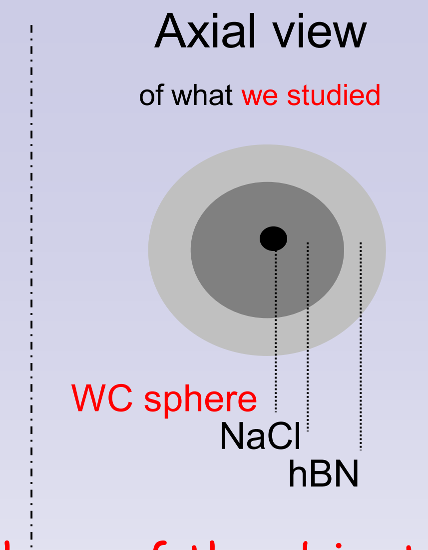
High P/T/stress tomographies

In situ high (P,T) Absorption Tomography

P = 8 GPa and 1000K

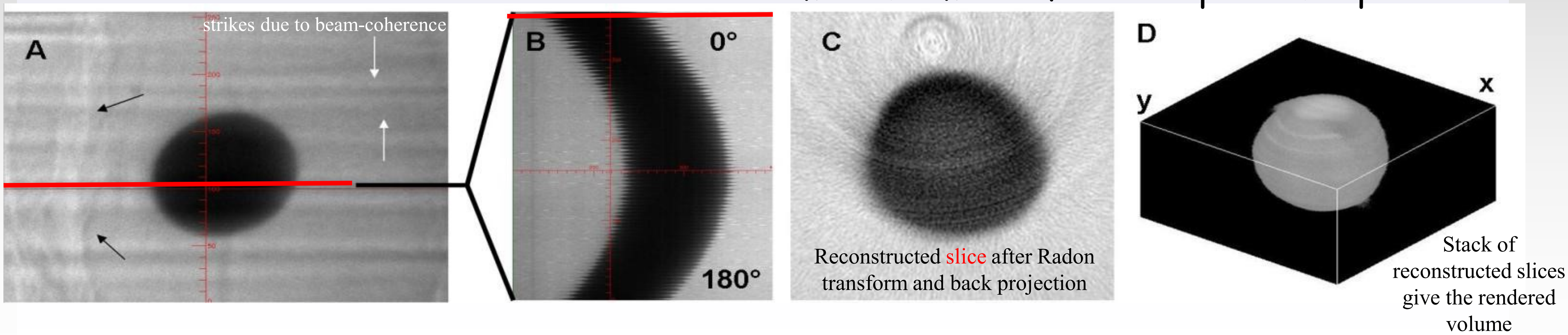




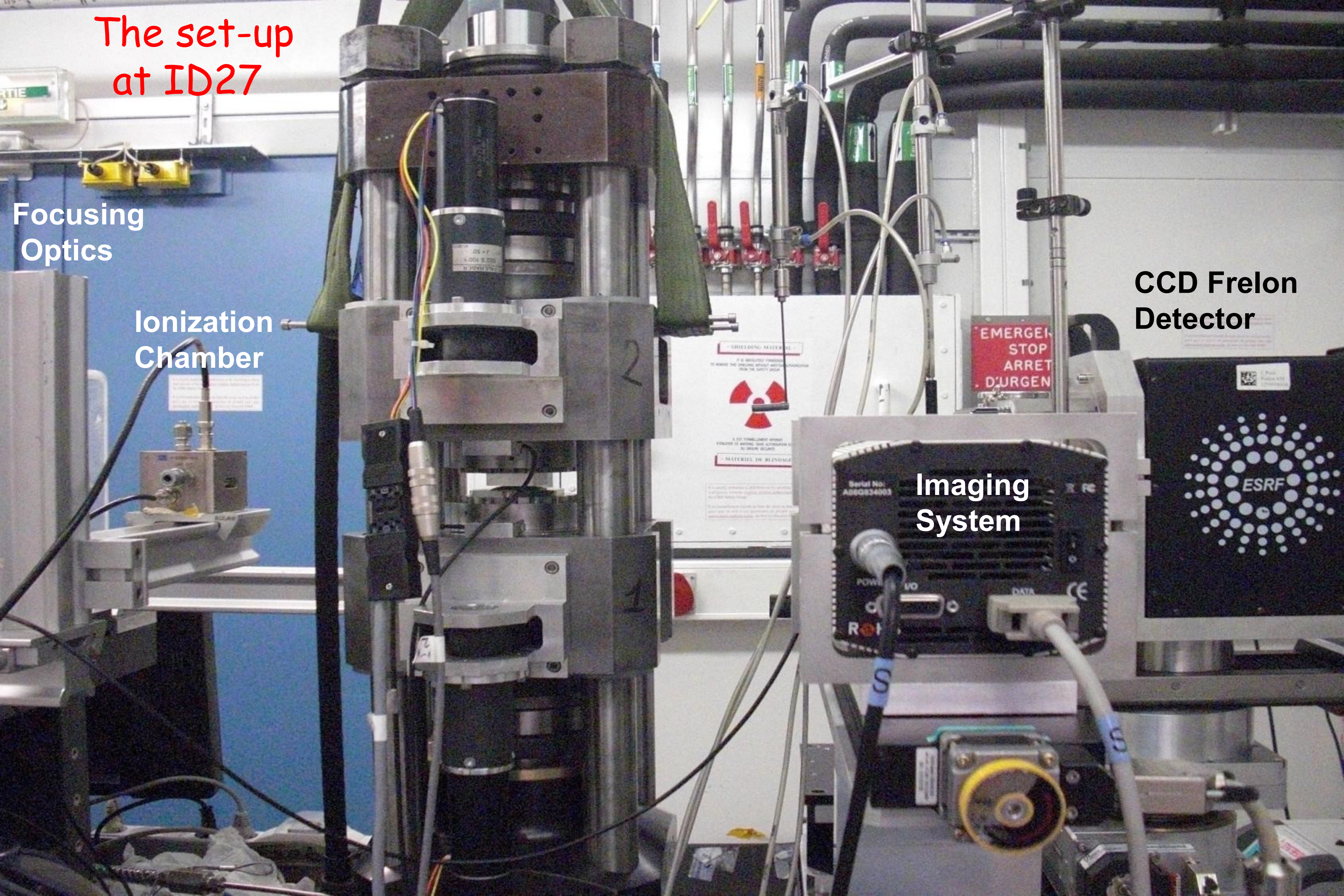


After tomography, the shape of the object has been perfectly reconstructed. The resolution of the new device will be determined from area/volume measurements for each pressure point

360 radiographies, 0.5° angular step



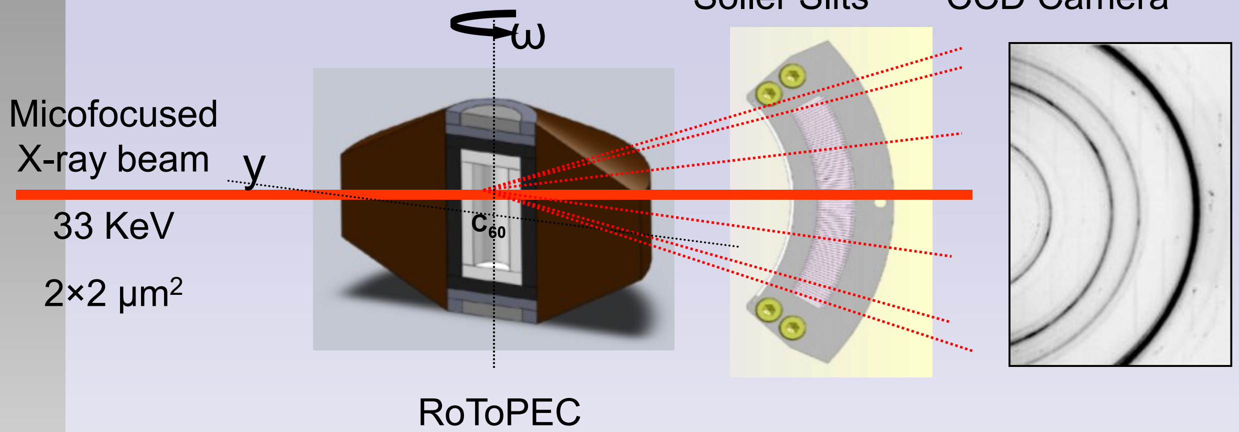
ImageJ

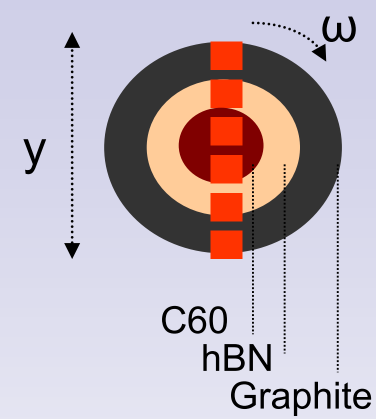

The set-up at ID27
Focusing Optics
Ionization Chamber
CCD Frelon Detector
Imaging System
EMERGE
STOP
ARRET
D'URGEN
ESRF

In situ Diffraction Tomography

P = 13 GPa, 1100 K

Preliminary experiment





Microdiffraction

Ny x Nω

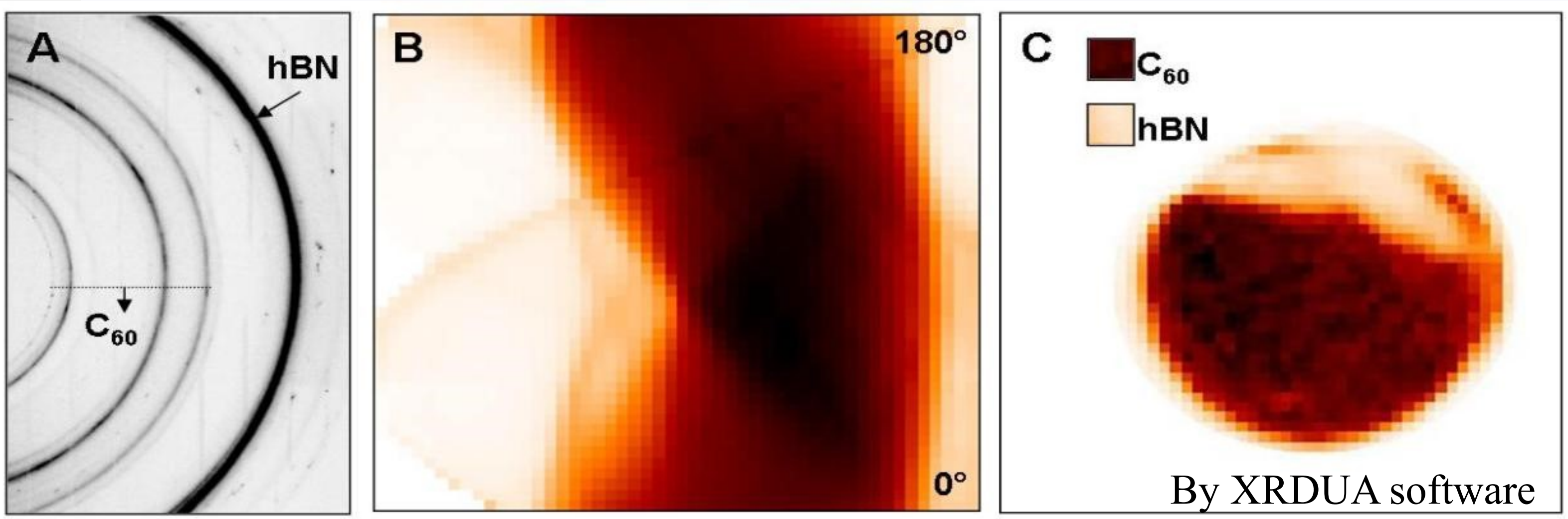
50 x 45 = 2250

2D diffraction images

4° angular step, 26 μm linear step
length 1.3 mm

Scanned

C_{60}

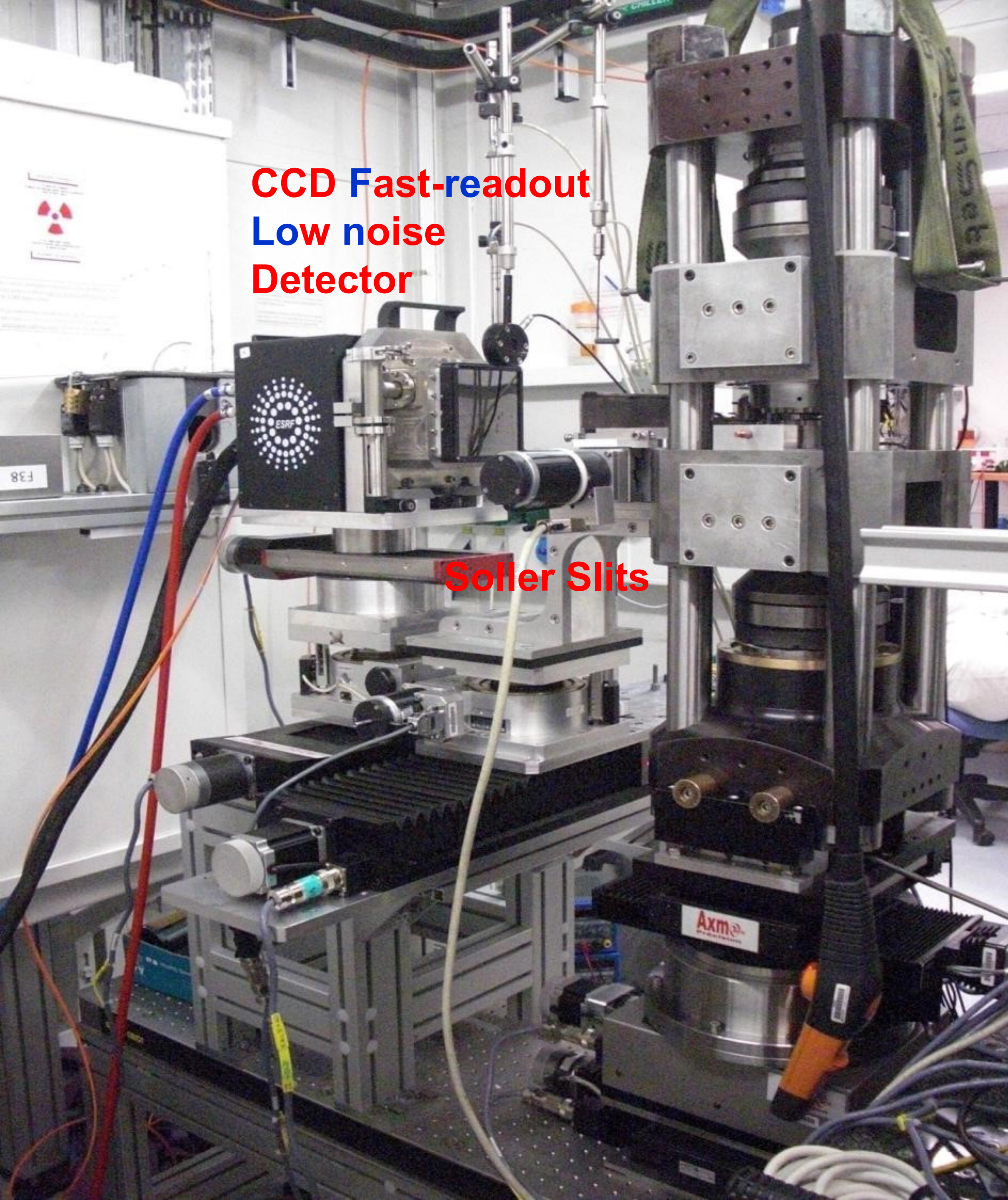


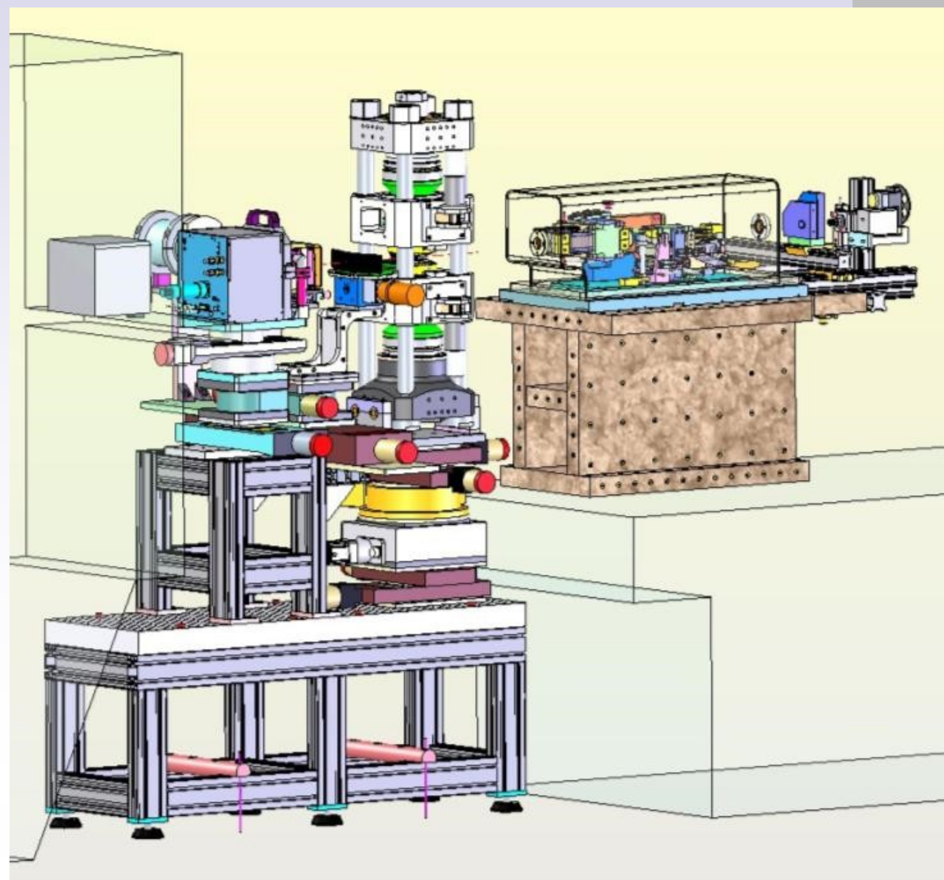
By XRDUA software

To get very interesting information at high P/T/stress conditions :

- Phase distribution - content
- Orientation (texture)
- Crystallite size/microstrain
- Degree of crystallinity

The corresponding set-up at ID27





This new rotational tomography PE press will provide new scientific opportunities for original and unique studies of phase transitions, density, crystallization and deformation in extreme P/T/Stress conditions with these 2 tomographies…

Absorption Tomography +

(large beam, fast)

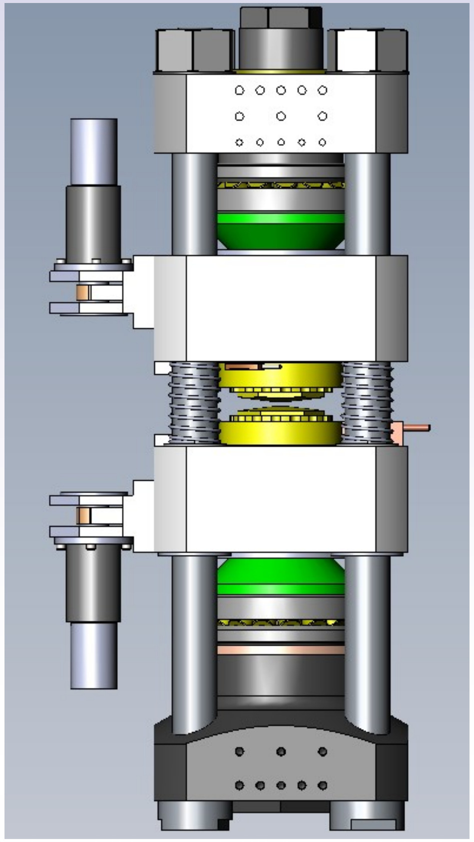
- Density
- Microstructure
- Porosity
- Defects
- Phase content

Diffraction Tomography +

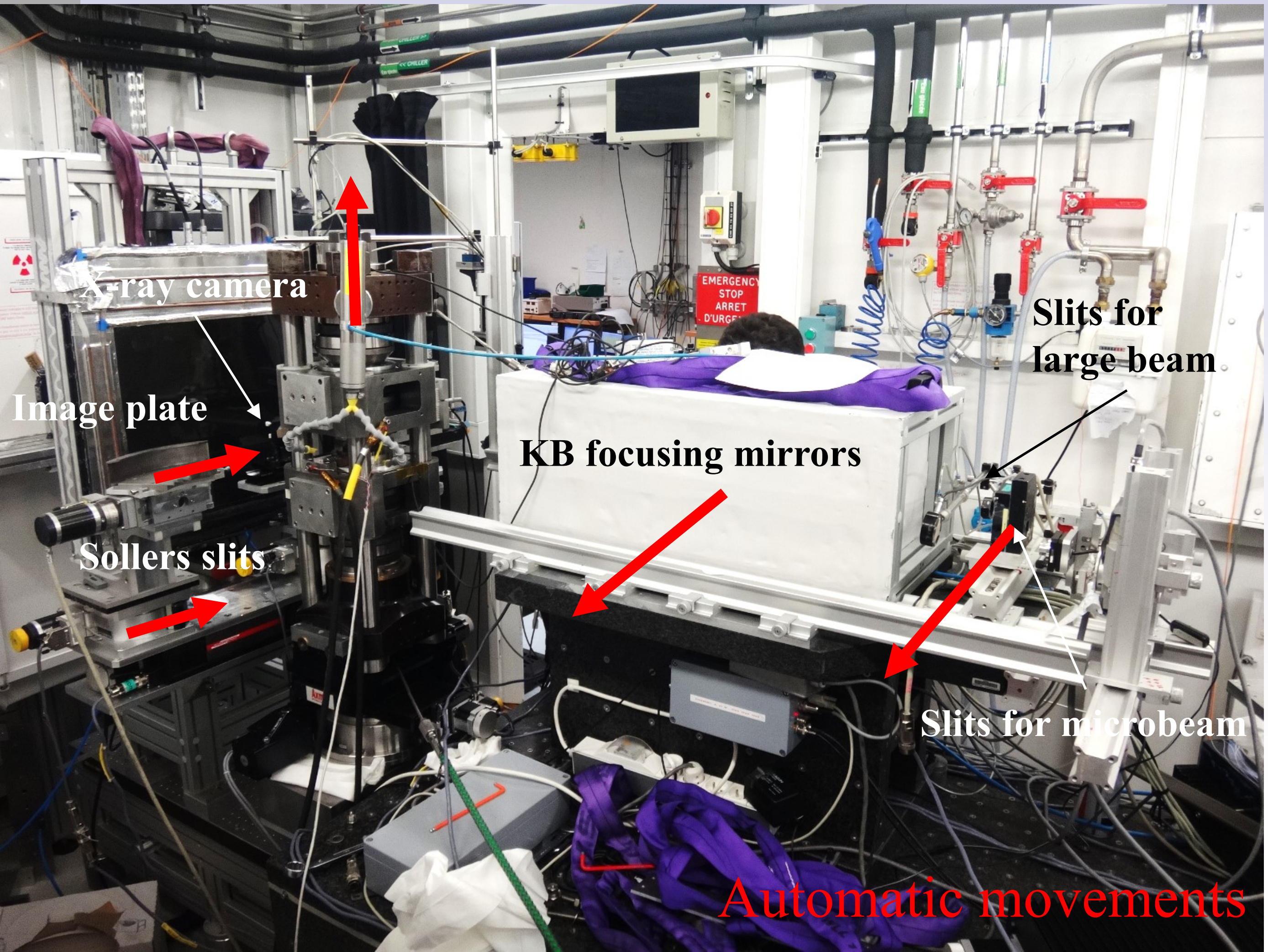
(micro beam, long)

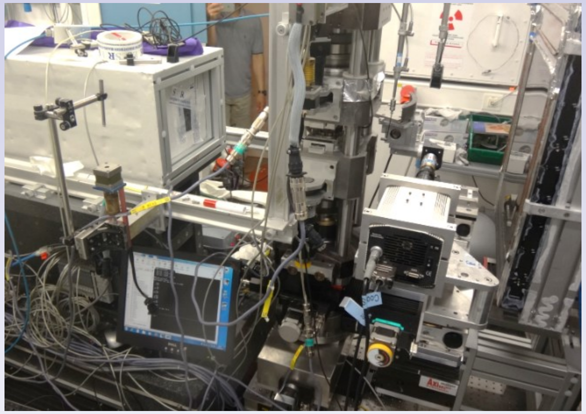
- Phase distribution - content
- Orientation (texture)
- Crystallite size/microstrain
- Degree of crystallinity

High P/T/stress PE cell

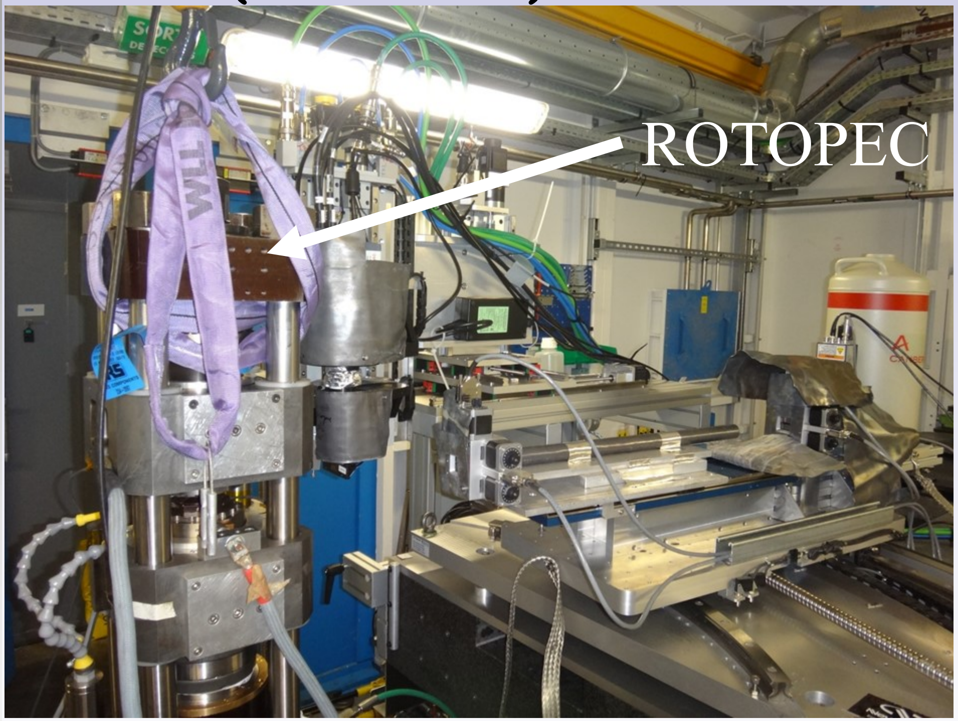


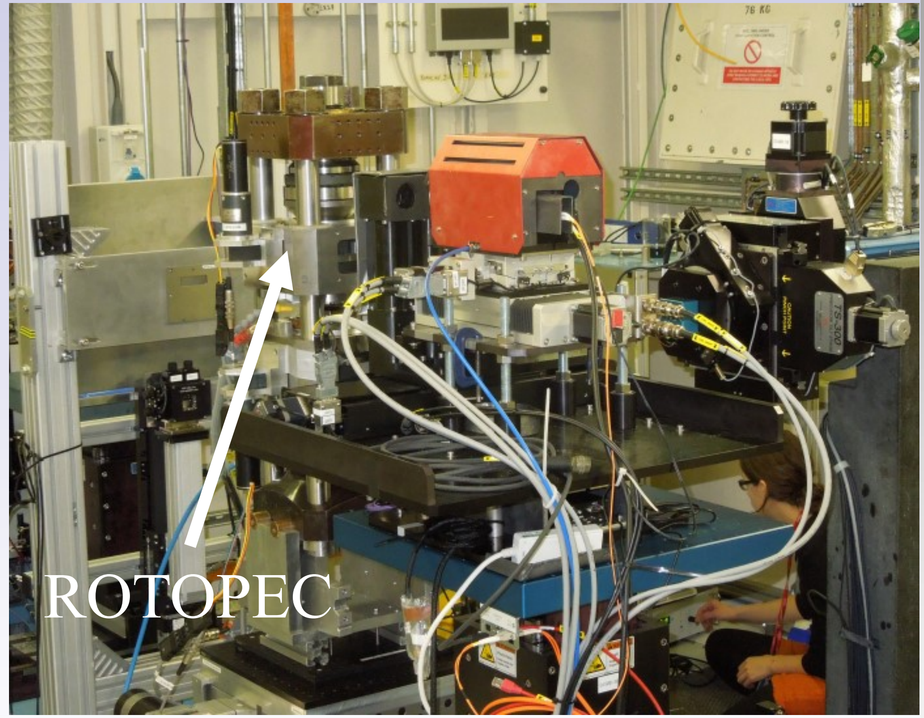
It is now possible to switch during an experiment between the 2 tomographies (absorption and diffraction) by keeping the same sample under HPHT. It is done automatically (without going into the X-ray hutch) via automatic movements and in less than a minute !!





Our portable rotopec press has been easily and successfully adapted to various multi-modal synchrotron experimental set-up, so these experiments are possible not only at beamline ID27 (ESRF), but also at PSICHE (SOLEIL), and I12 (DIAMOND) beamlines





SOLEIL SYNCHROTRON

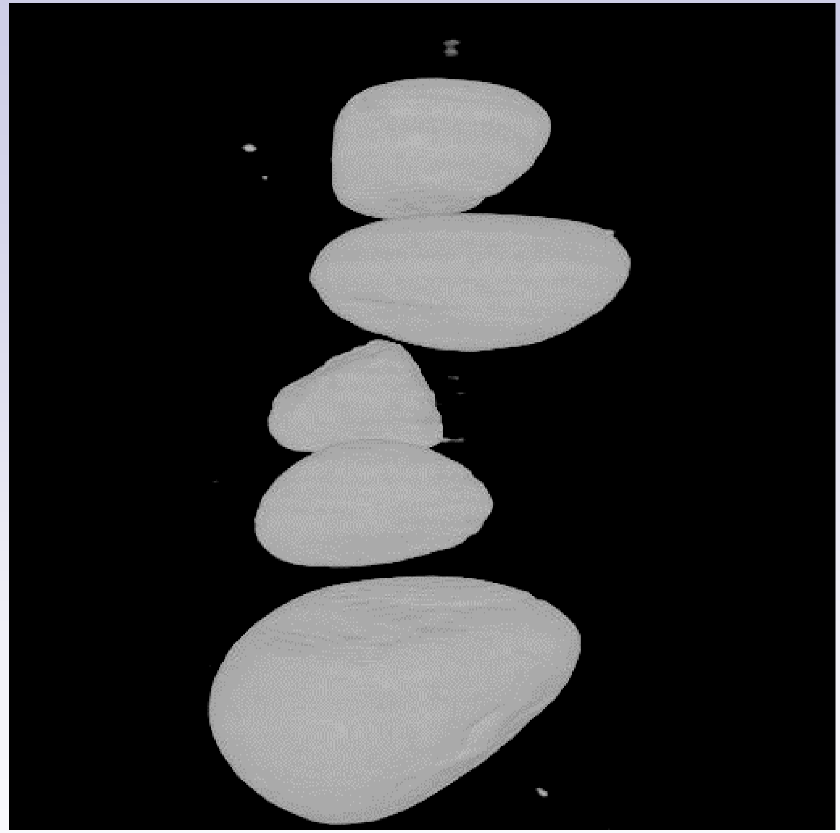
diamond

3 recent studies with this novel rotoPEc portable press

- EOS of non-crystalline materials : feasibility of accurately measuring sample volume as a function of P and T
- Time resolved 3-D imaging of melt migration under extreme conditions
- Measurements of shear modulus and attenuation coefficient Q_G^{-1} under mantle conditions and seismic frequencies

EOS of non-crystalline materials : feasibility of accurately measuring sample volume as a function of P and T

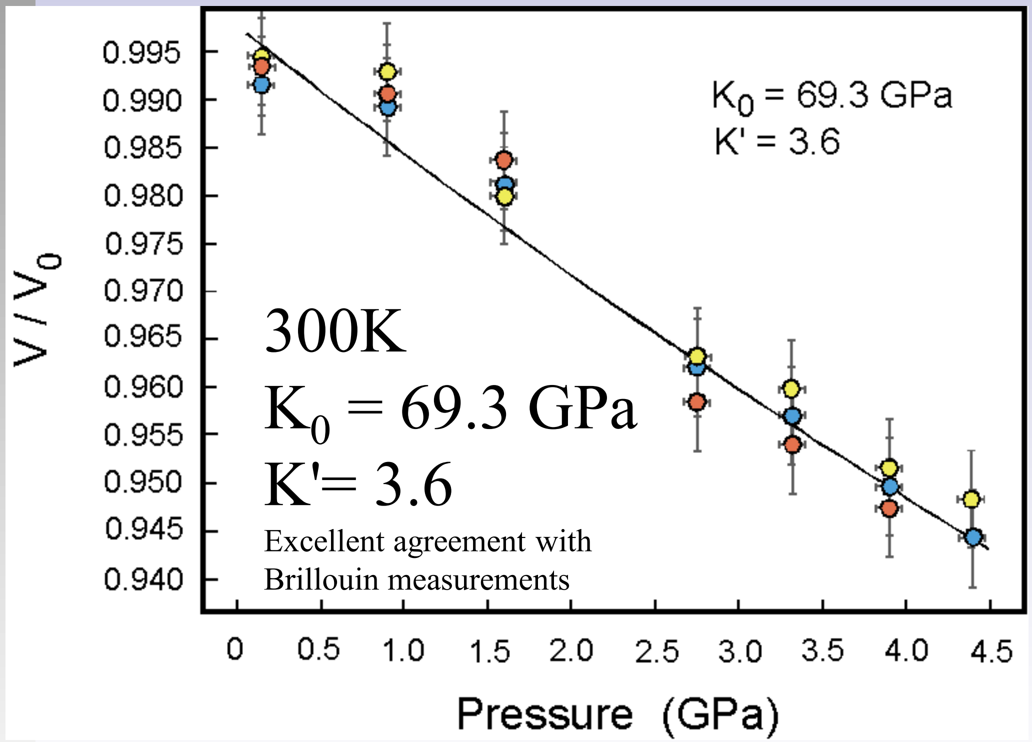
In this experiment at SOLEIL, the sample consisted of vitreous olivine spheres (diameter about 800 μm) embedded in BN powder, hot pressed at various P and T

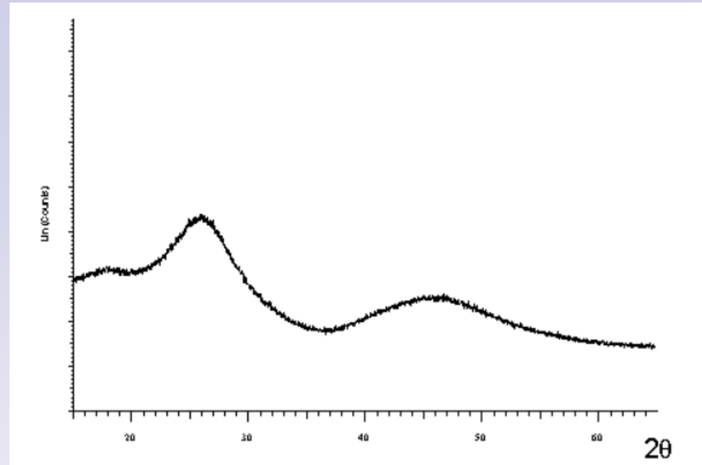


SOLEIL SYNCHROTRON

3D rendering of the five small olivine particles, from which one can extract the volume of the sphere for each P and T

EOS of non-crystalline materials : feasibility of accurately measuring sample volume as a function of P and T





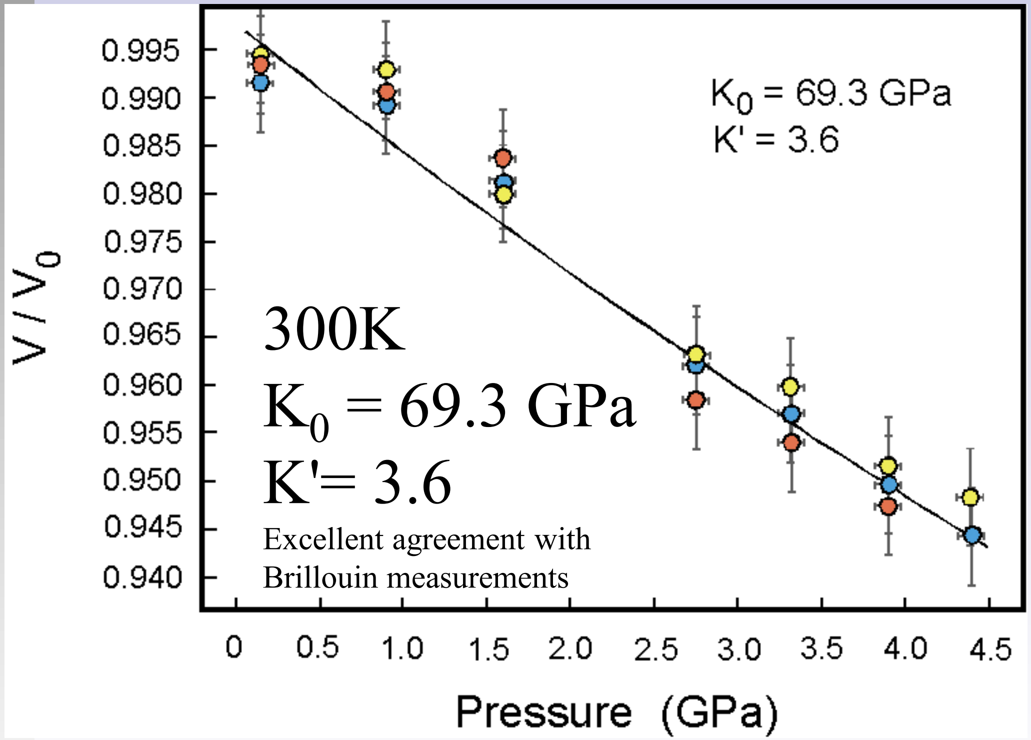
V ?

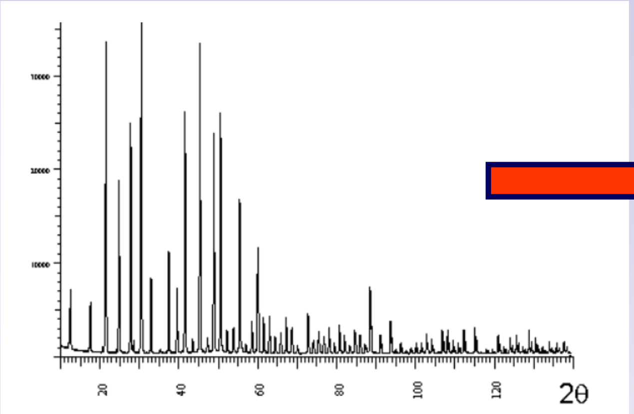
This study justifies our technique as a useful tool in directly measuring volume changes of noncrystalline materials as a function of both P and T

SOLEIL SYNCHROTRON

Talk of J.P. PERRILLAT will detail this study !!

EOS of non-crystalline materials : feasibility of accurately measuring sample volume as a function of P and T



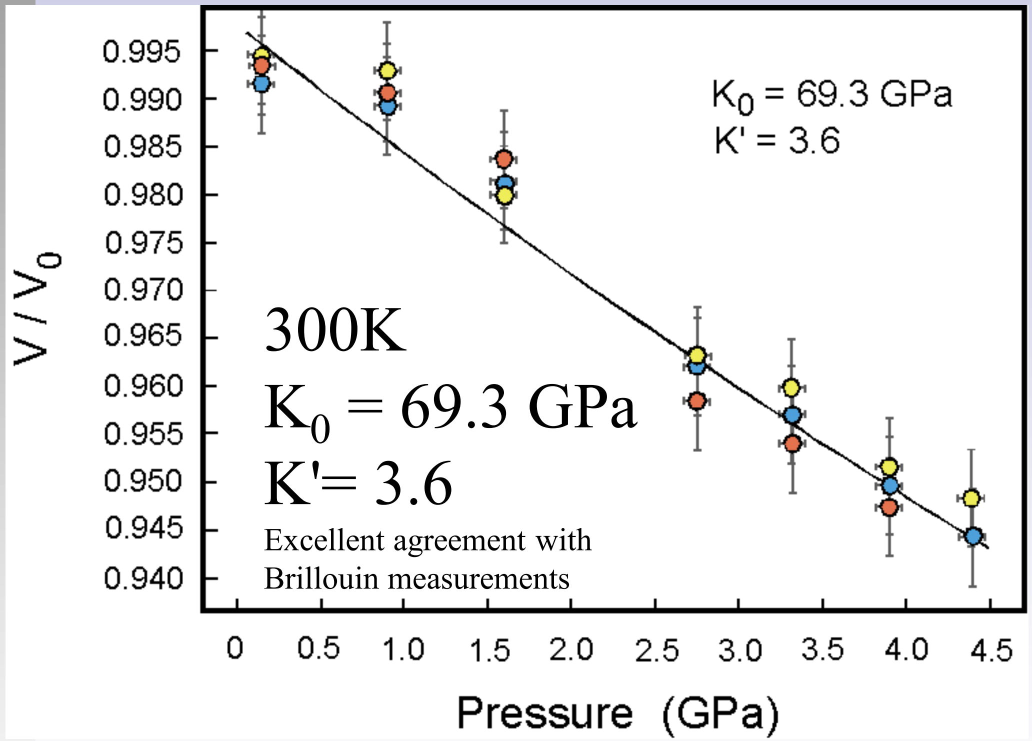


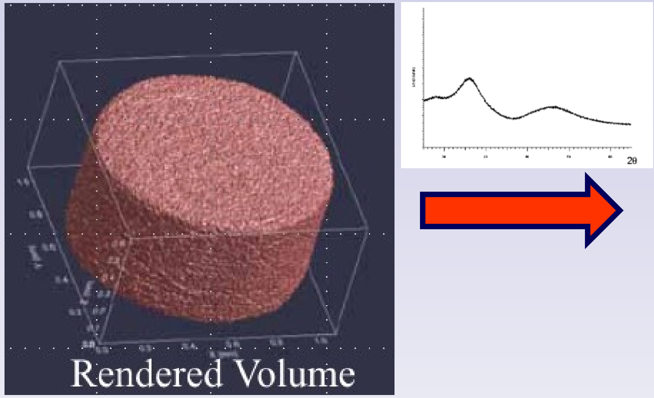
Cell parameter, hence V

Unlike crystalline materials, whose specific density can be evaluated using x-ray diffraction, densities of noncrystalline materials glasses and melts are traditionally more difficult to determine under extreme conditions

SOLEIL SYNCHROTRON

EOS of non-crystalline materials : feasibility of accurately measuring sample volume as a function of P and T





Volume

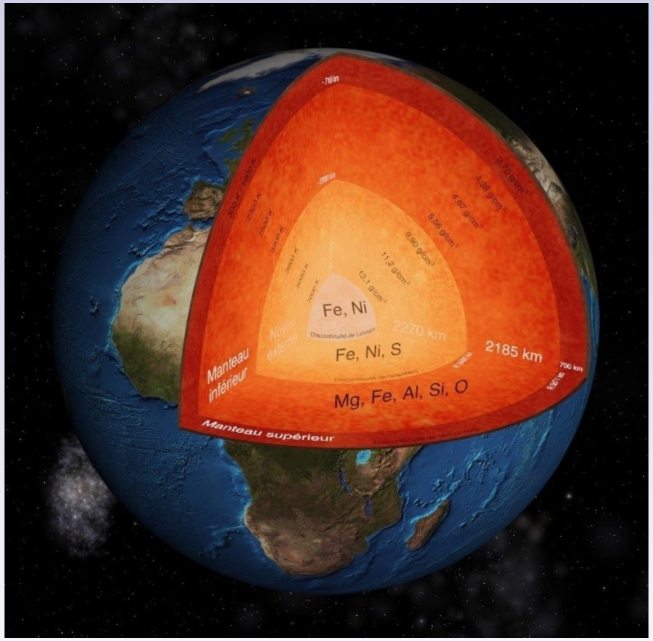
Our rotopec provides now a method to address this long-standing difficulty !

SOLEIL SYNCHROTRON

3 recent studies with this novel rotoPEc portable press

- EOS of non-crystalline materials : feasibility of accurately measuring sample volume as a function of P and T
- Time resolved 3-D imaging of melt migration under extreme conditions
- Measurements of shear modulus and attenuation coefficient Q_G^{-1} under mantle conditions and seismic frequencies

Melt migration in planetary deep interiors



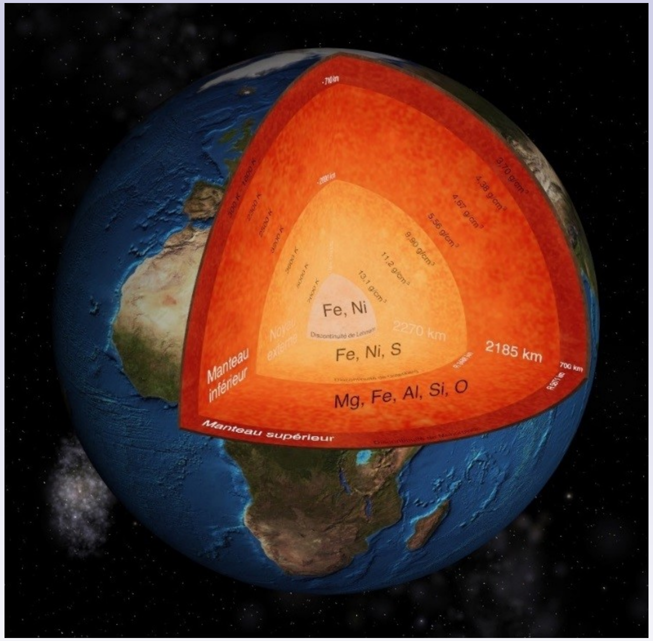
Many geological processes involve extraction of low degree partial melts and several questions arise :

At what point during progressive melting does the melt phase become mobile ?

How does this partial melt move through the remaining solid phase, and through the rest of the Earth ?

Melt release and melt transport processes are important because they influence the geochemistry of the melt phase. In fact, the geochemical signature of melts is typically used to understand where they formed in planetary interiors and how

Melt migration in planetary deep interiors



Many geological processes involve extraction of low degree partial melts and several questions arise :

At what point during progressive melting does the melt phase become mobile ?

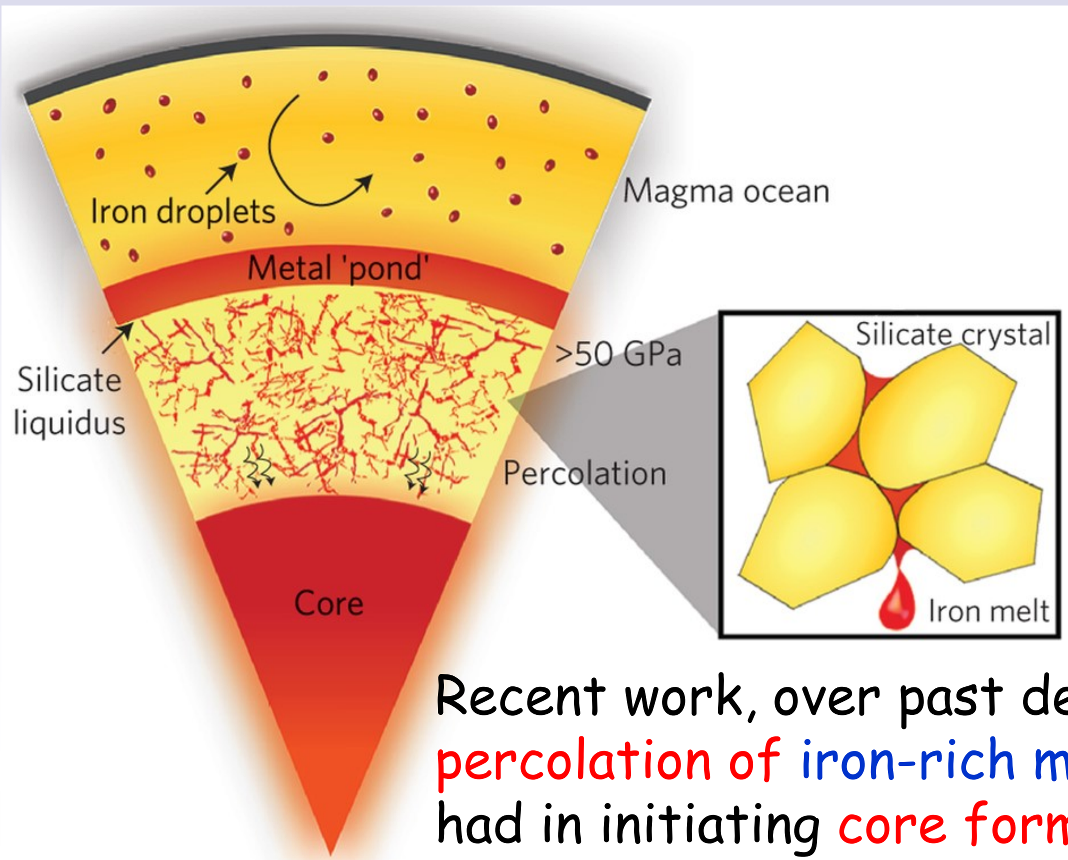
How does this partial melt move through the remaining solid phase, and through the rest of the Earth ?

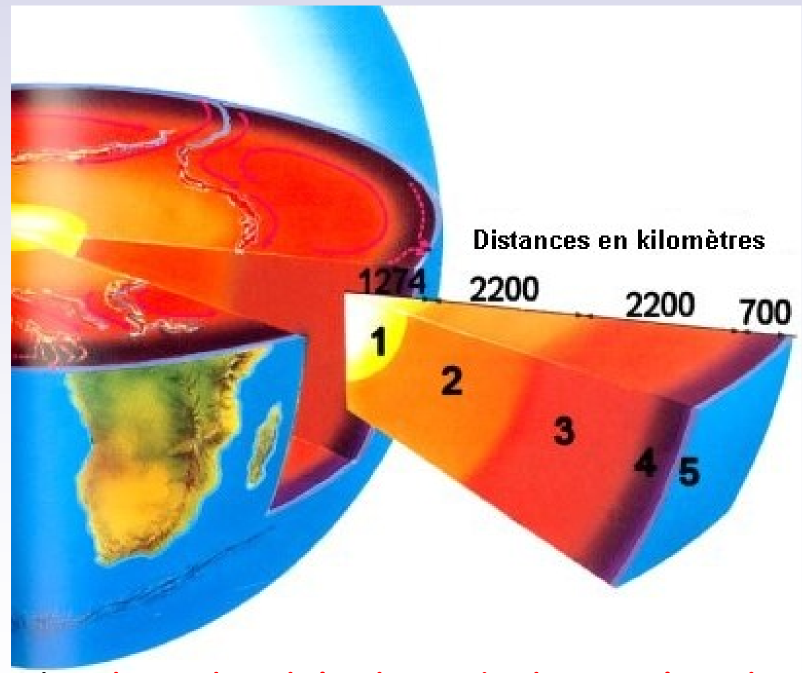
We need to understand melt formation and migration
At present, however, our understanding of these processes under extreme conditions of planetary interiors is limited

Melt migration in planetary deep interiors

Core formation ?

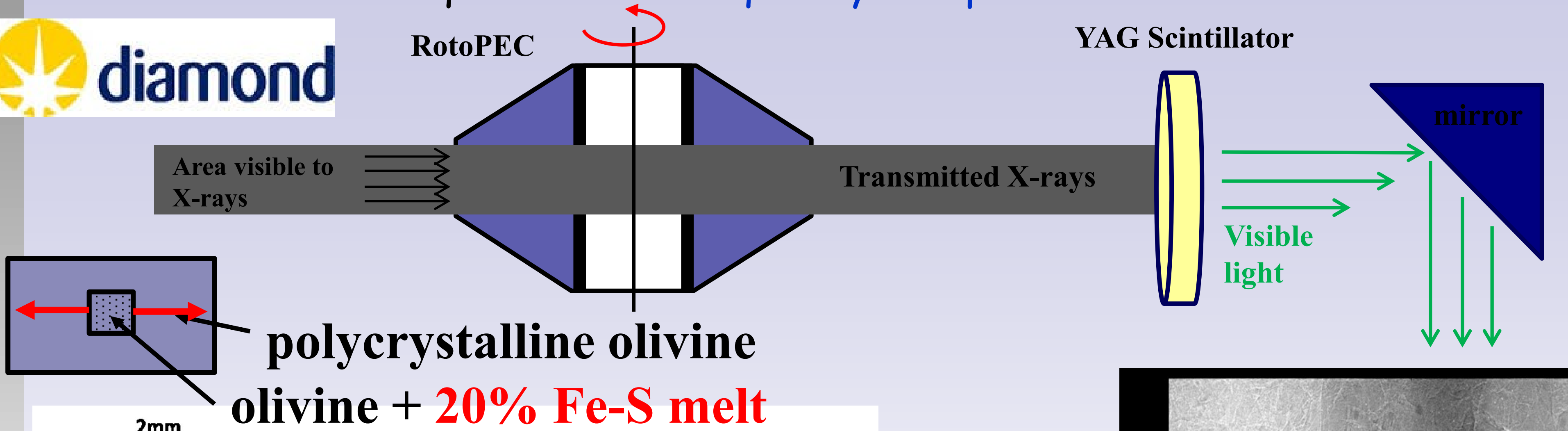
Our understanding of how Earth segregated to form a metallic core surrounded by a thick shell of silicate is constrained by our knowledge of metal-silicate segregation mechanisms under high P,T **and stress**

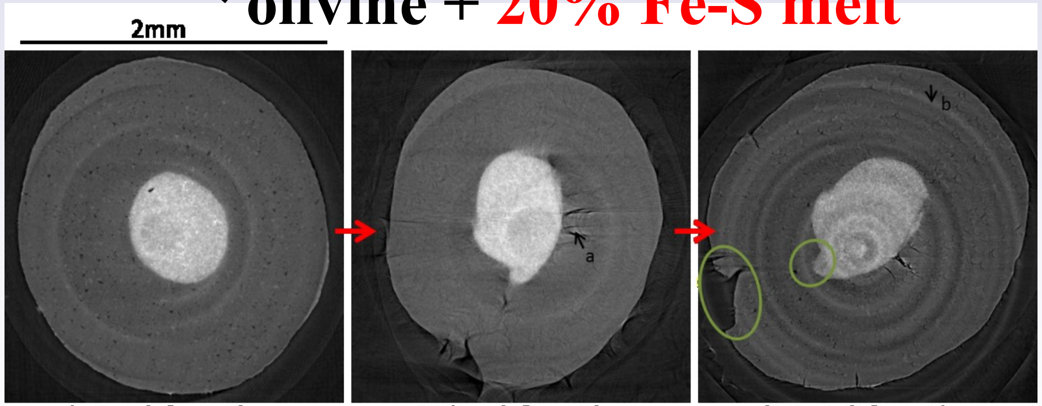




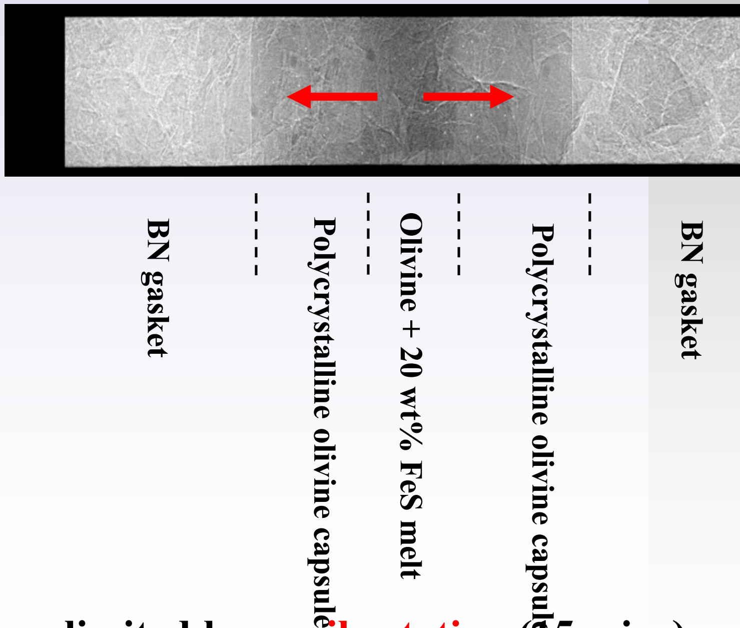
Recent work, over past decade, has highlighted the role that percolation of iron-rich melts through solid silicate could have had in initiating core formation

Visualising melt migration under extreme conditions (HPHT, large deformation) to answer this question : how quickly can planets form metallic cores ?



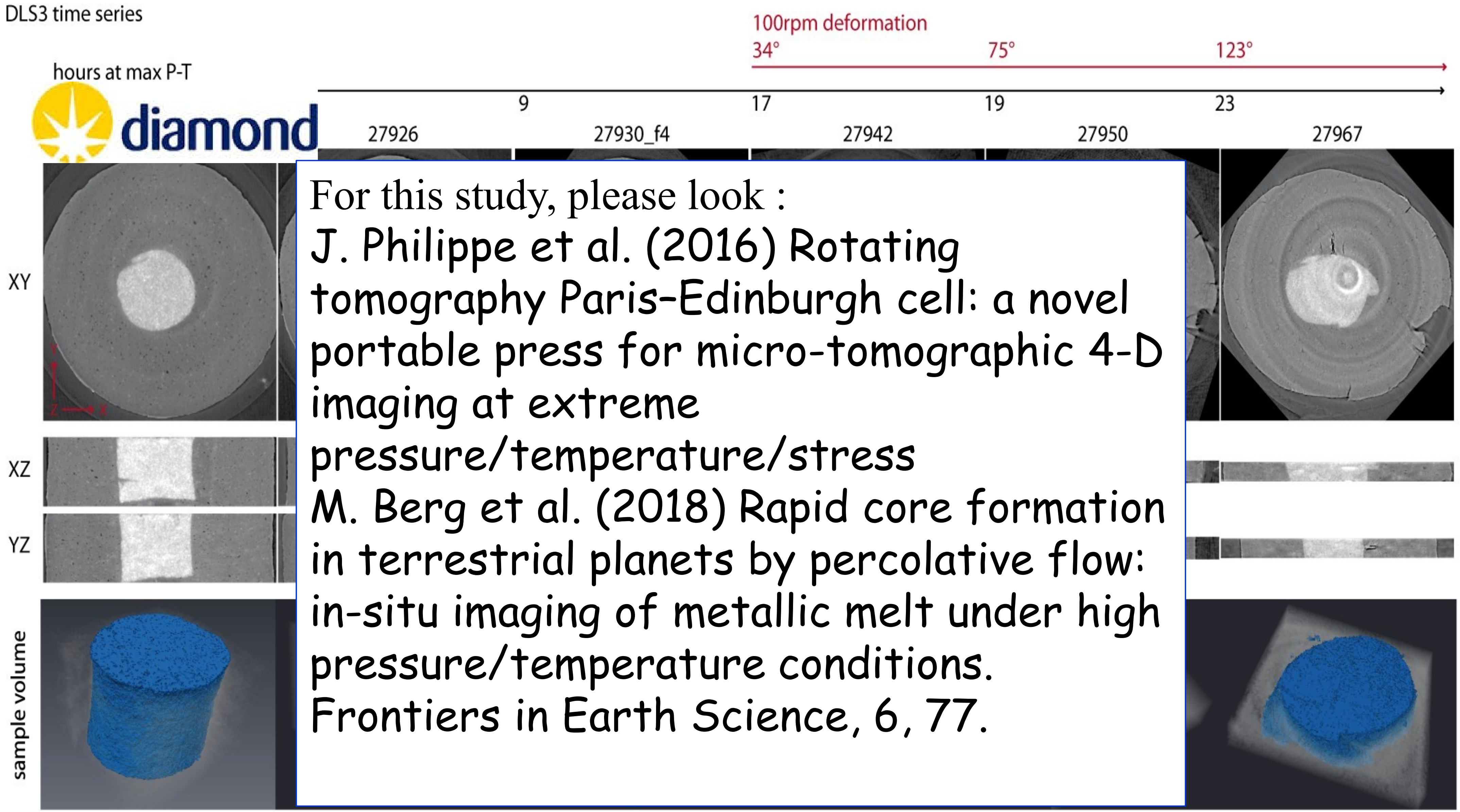


A. Pre-deformation B. Syn-deformation C. Post-deformation



Our experiments performed at DIAMOND aimed at investigating the mobility of Fe-S rich melts: Fe-S melts should migrate under extreme conditions from melt-rich central region of sample assembly into surrounding, melt-free polycrystalline olivine

- Scan time limited by anvil rotation (15 mins)
- But samples stable over this time
- Approx 2µm voxol size achievable;



For this study, please look :

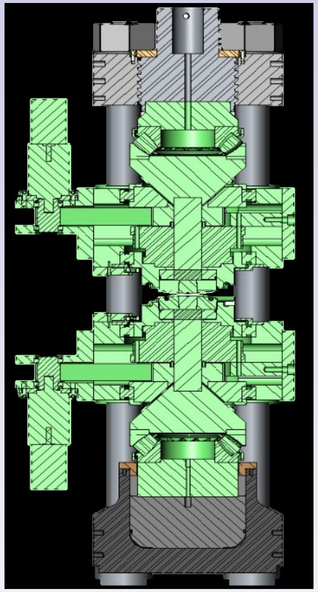
J. Philippe et al. (2016) Rotating tomography Paris-Edinburgh cell: a novel portable press for micro-tomographic 4-D imaging at extreme pressure/temperature/stress

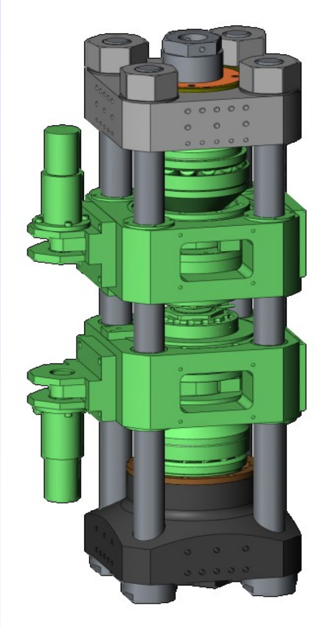
M. Berg et al. (2018) Rapid core formation in terrestrial planets by percolative flow: in-situ imaging of metallic melt under high pressure/temperature conditions. Frontiers in Earth Science, 6, 77.

3-D rendering of the melt-rich central part of the sample during progressive annealing under P and then deformation : deformation enhance melt migration

Melt migration in planetary deep interiors

	distance / µm	time / s	speed / µms-1
M1a short	300	20	15.0
M1a mid	900	20	45.0
M1a long	1150	20	57.5
M1b short	300	20	15.0
M1b long	800	20	40.0
M2 2d	350	540	0.6
M2 short	400	540	0.7
M2 mid	500	540	0.9
M2 long	1500	540	2.8





Clearly this is all very preliminary

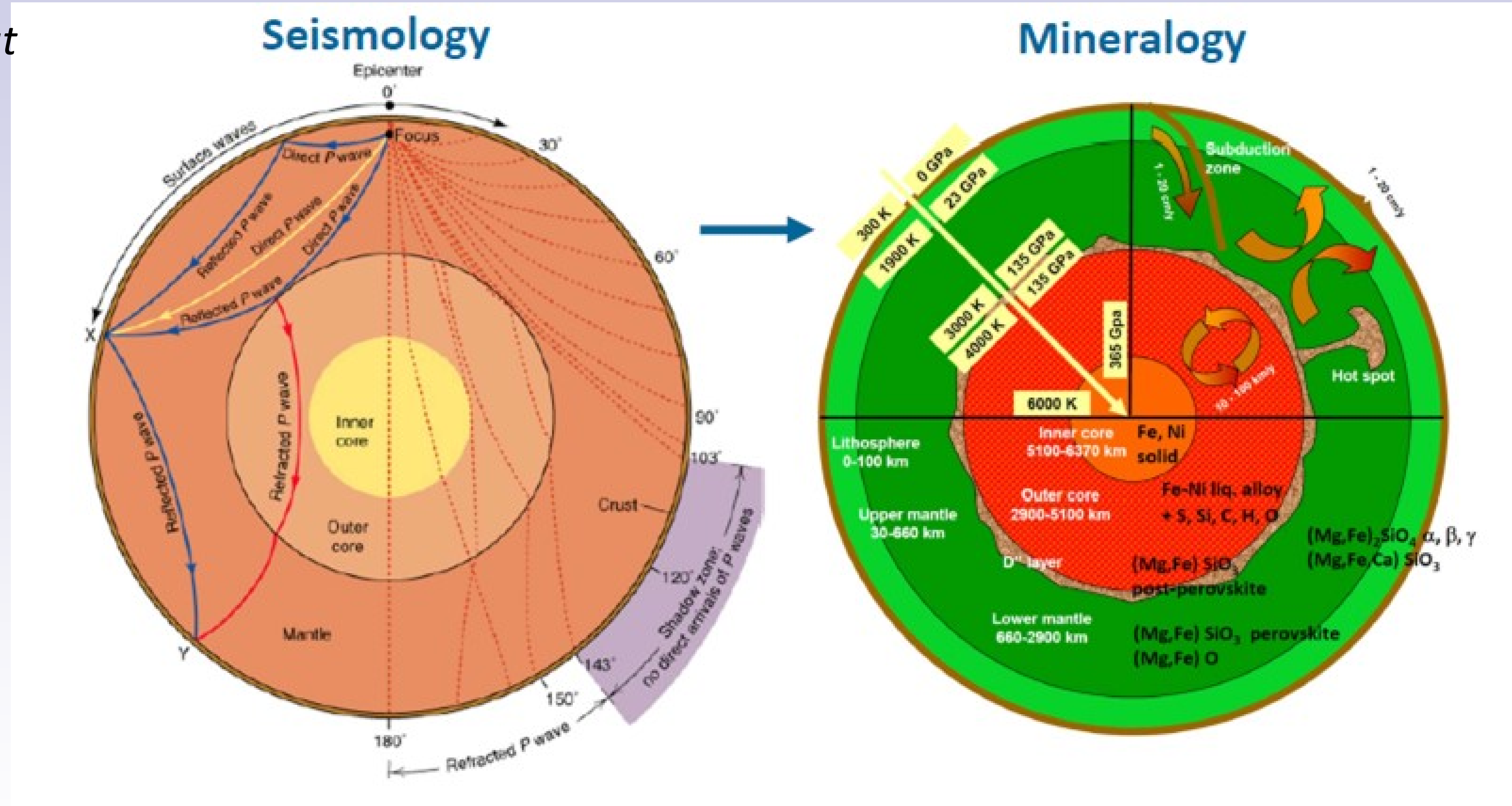
However, we can show that we can extract now with our rotopec press meaningful data on how melts move and melt migration velocities under extreme P/T/Stress conditions

3 recent studies with this novel rotoPEc portable press

- EOS of non-crystalline materials : feasibility of accurately measuring sample volume as a function of P and T
- Time resolved 3-D imaging of melt migration under extreme conditions
- Measurements of shear modulus and attenuation coefficient Q_G^{-1} under mantle conditions and seismic frequencies

Measurements of shear modulus and attenuation coefficient Q_G^{-1} under mantle conditions and seismic frequencies

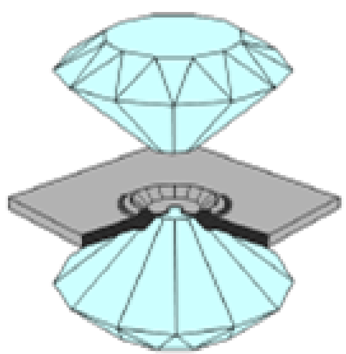
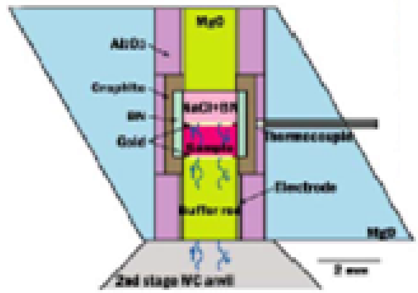
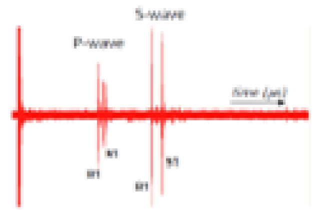
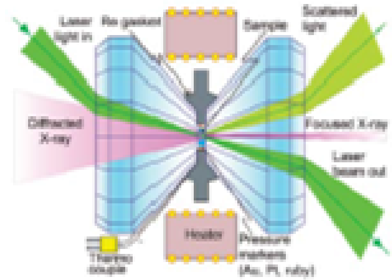
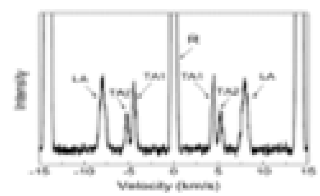
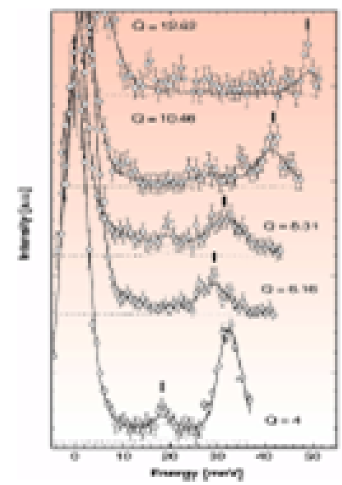
Coll. J.P. Perrillat



Interpretation of seismic data requires the knowledge of compressional and shear wave velocities in minerals

Conventional experimental methods for the determination of sound velocities in geological materials

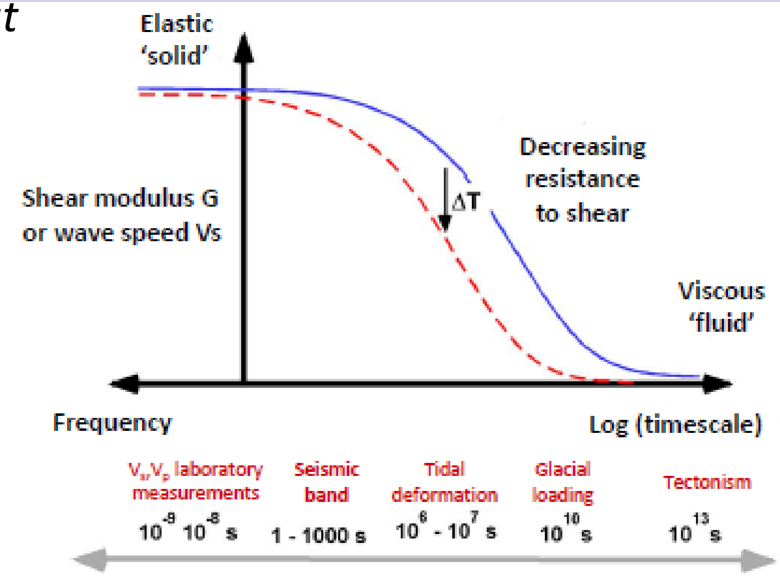
Coll. J.P. Perrillat

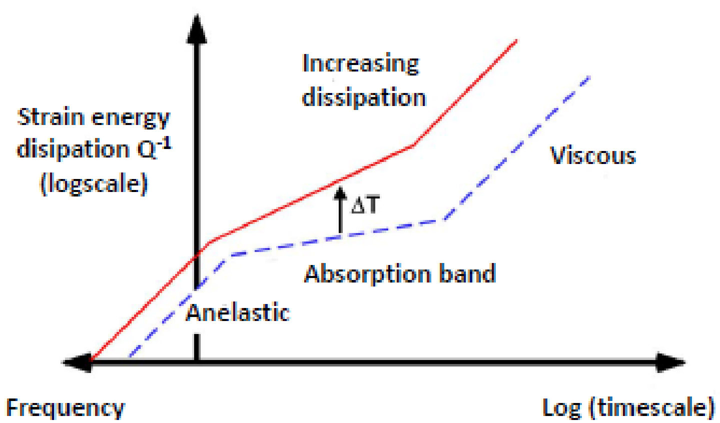
Static compression measurements	*Ultrasonic methods*	*Brillouin Light scattering*	*Inelastic X-ray scattering*
	 	 	
X-Ray & neutron Diffraction			
V (P, T) *Bulk modulus (K)*	*Vp - Vs* *Bulk modulus (K)* *Shear modulus (G)*	*Vp - Vs* *Bulk modulus (K)* *Shear modulus (G)*	*Vp - Vs* *Bulk modulus (K)* *Shear modulus (G)*
frequency	*MHz*	*GHz*	*THz*

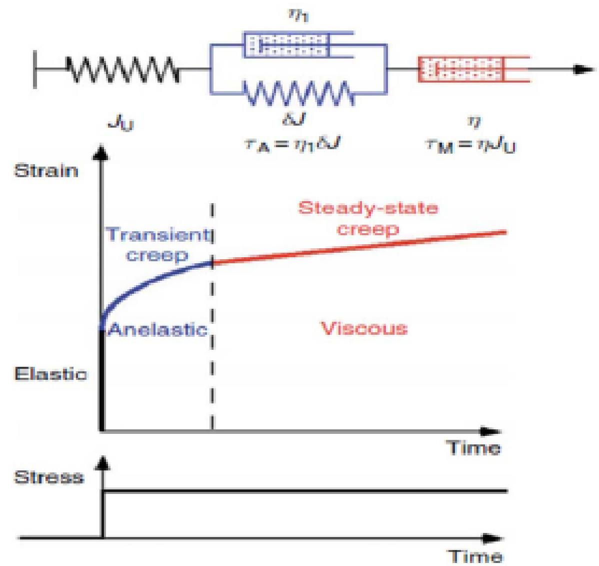
…but in fact teleseismic waves propagate at lower frequencies mHz - Hz

Mantle rocks are visco-elastic solids, with anelasticity

Coll. J.P. Perrillat







Jackson [2005]

Crystallographic mechanisms:
motion of dislocations,
grain boundary sliding,
phase transitions, melting, etc…

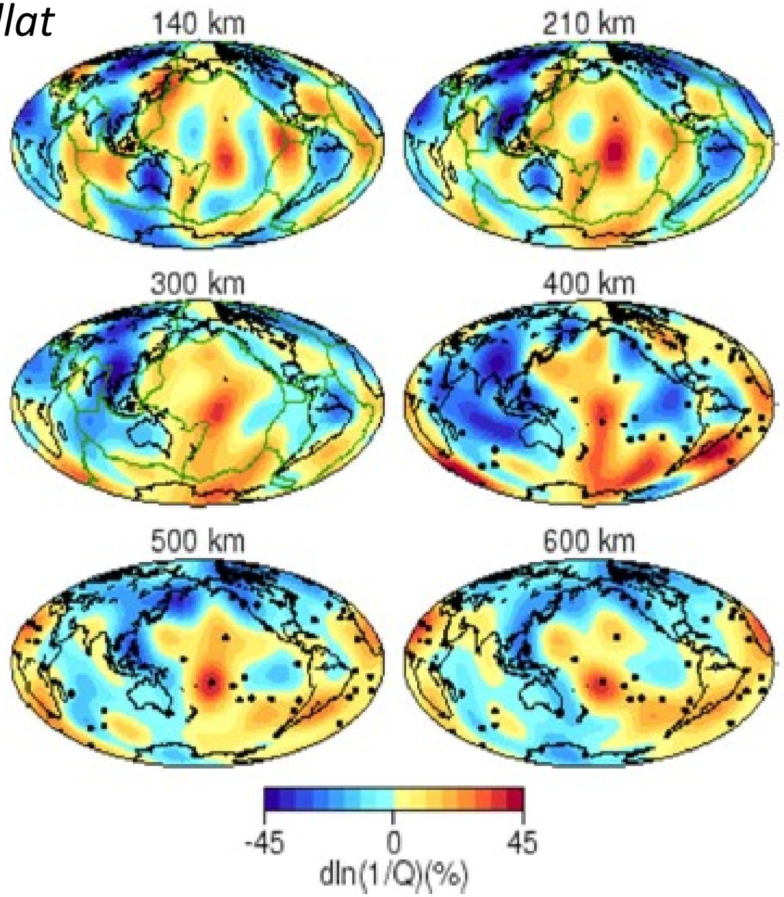
**Attenuation of seismic waves
(« intrinsic attenuation »)**

ESRF

Measurements of shear modulus and attenuation coefficient Q_G^{-1} under mantle conditions and seismic frequencies

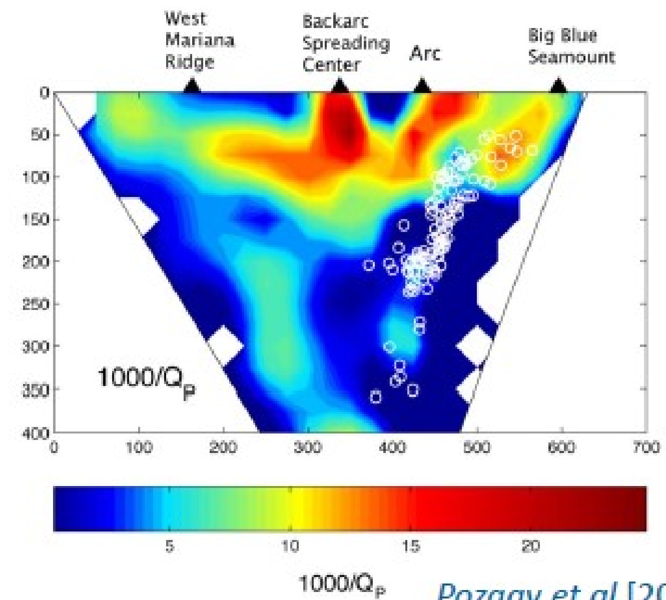
Coll. J.P. Perrillat

Global 3D models



Gung & Romanowicz [2004]

Regional studies



Pozgay et al [2009]

Attenuation furnishes important constraints on temperature, water/melt content, grain sizes… however we lack a mineral database for attenuation coefficient at mantle conditions and seismic frequencies because up to now we lacked an experimental method for these HPHT measurements !

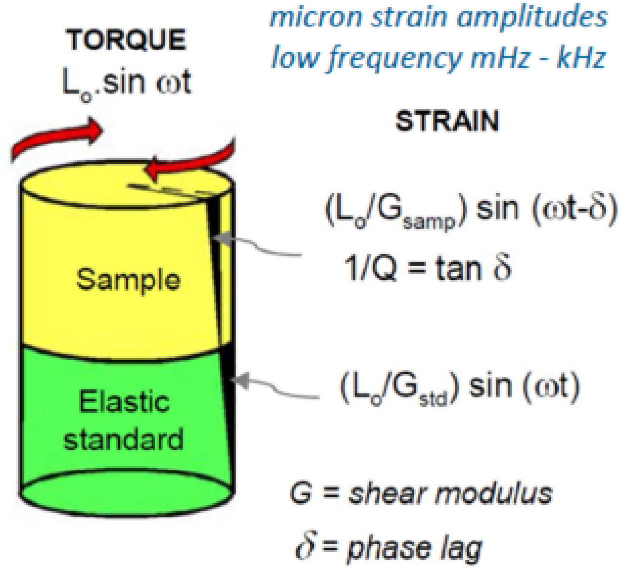
Interpretation of seismic profiles and tomographic models of the Earth's mantle requires HPHT exploring the viscoelastic behaviour of rocks / minerals at low strains and seismic frequencies

The torsional forced-oscillation method, a cyclic loading technique

Coll. J.P. Perrillat

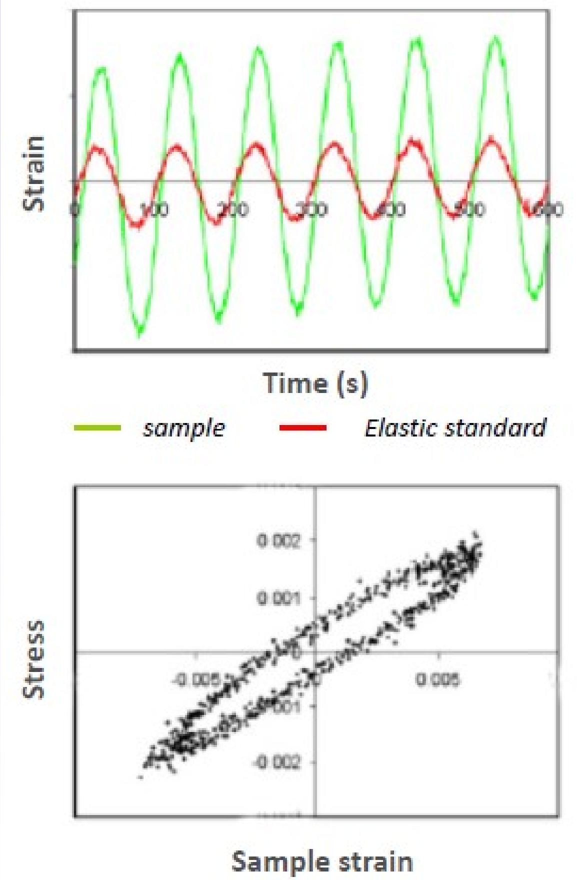
Torsional forced-oscillation method

micron strain amplitudes
low frequency mHz - kHz



Shear modulus G_{samp} = amplitude ratio x G_{std}

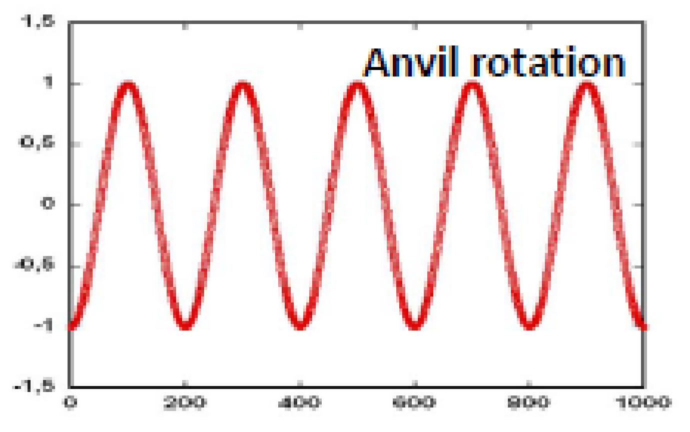
Attenuation Q_G^{-1} = tangent of the phase lag
area of the stress – strain ellipse



The shear modulus is derived from the amplitude ratio and the attenuation of the sample is determined from the phase lag between the deformation of the elastic standard and the sample

Torsional forced-oscillation method : the next step

Coll. J.P. Perrillat



Increasing the P-T range of G and Q_G^{-1} measurements to mantle conditions

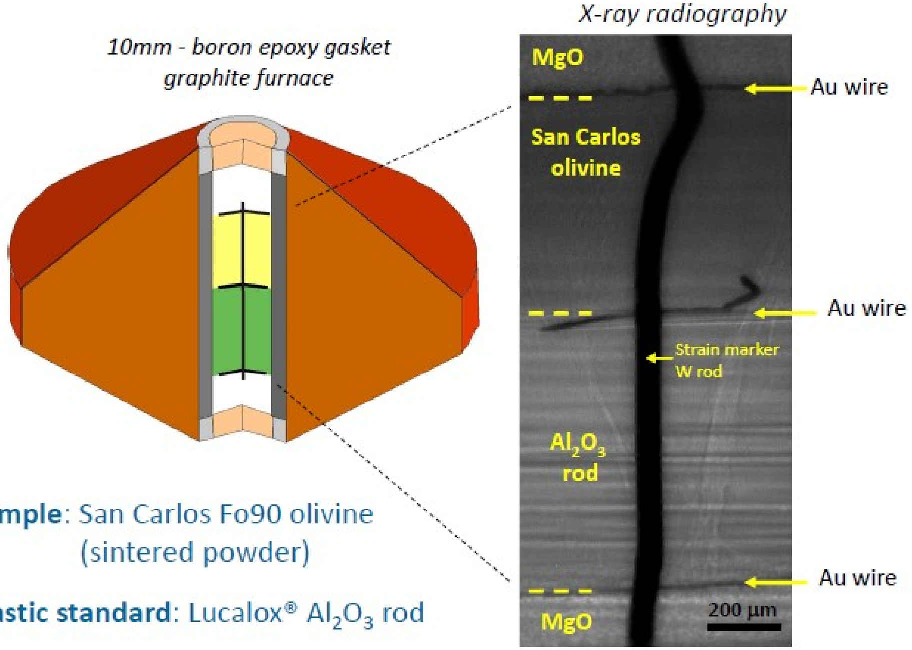
Requirements:

- High pressure device capable of applying a sinusoidal torque on the sample under load

= "RoToPEc" module

- A technique to measure the sample/standard strain with high spatial & time resolution

= X-ray radiography (synchrotron)



Sample: San Carlos Fo90 olivine (sintered powder)

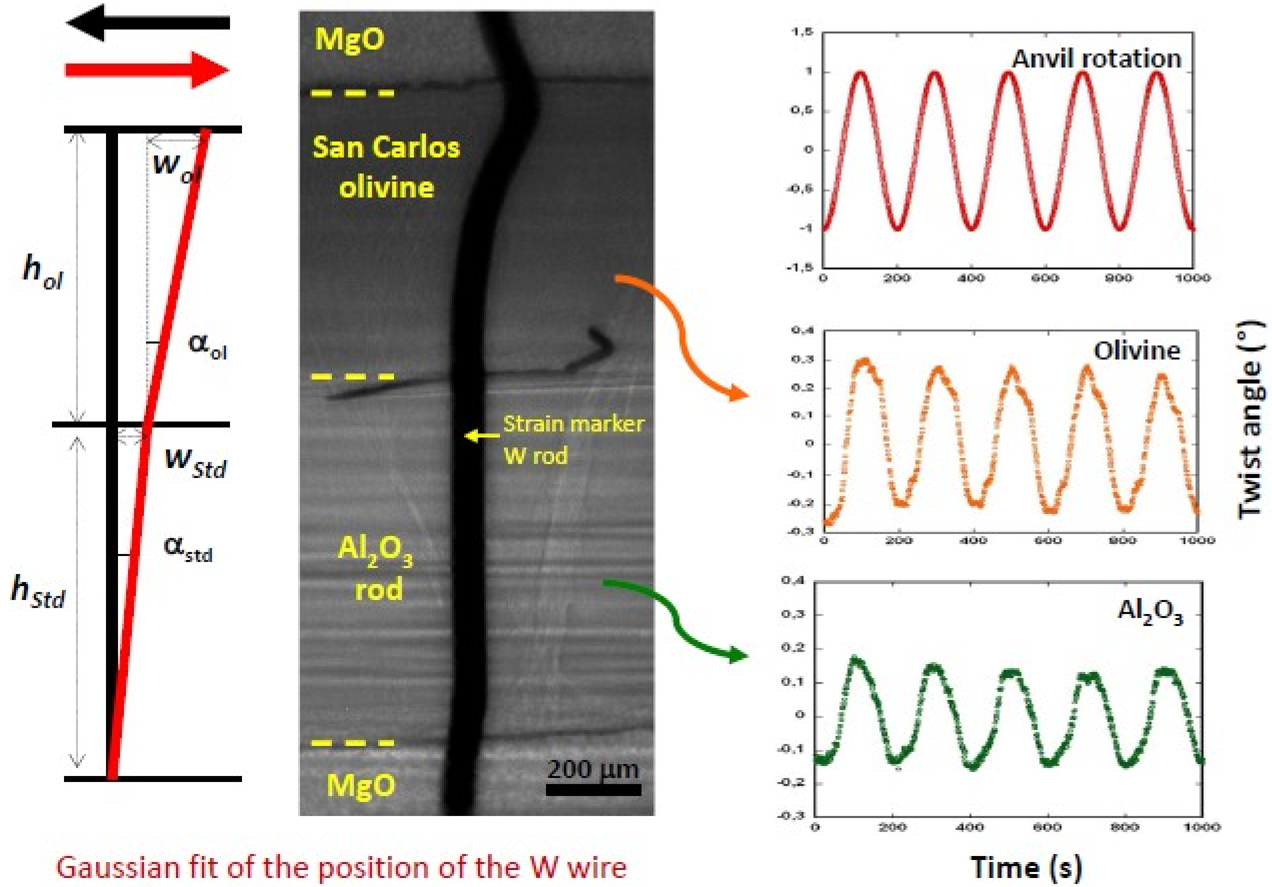
Elastic standard: Lucalox® Al_2O_3 rod

Measurements of shear modulus and attenuation coefficient Q_G^{-1} under mantle conditions and seismic frequencies

Coll. J.P. Perrillat

A sinusoidal shear was set to the sample by the periodic rotation of the bottom anvil of the RoToPEc module by 0.5 – 5° at periods of 100 – 1000 s. While the sample was under cyclic loading, X-ray images were recorded with a time resolution as low as 100ms in order to monitor the deformation of both the sample and elastic standard

Data are still processing but the **feasibility has been clearly demonstrated…**

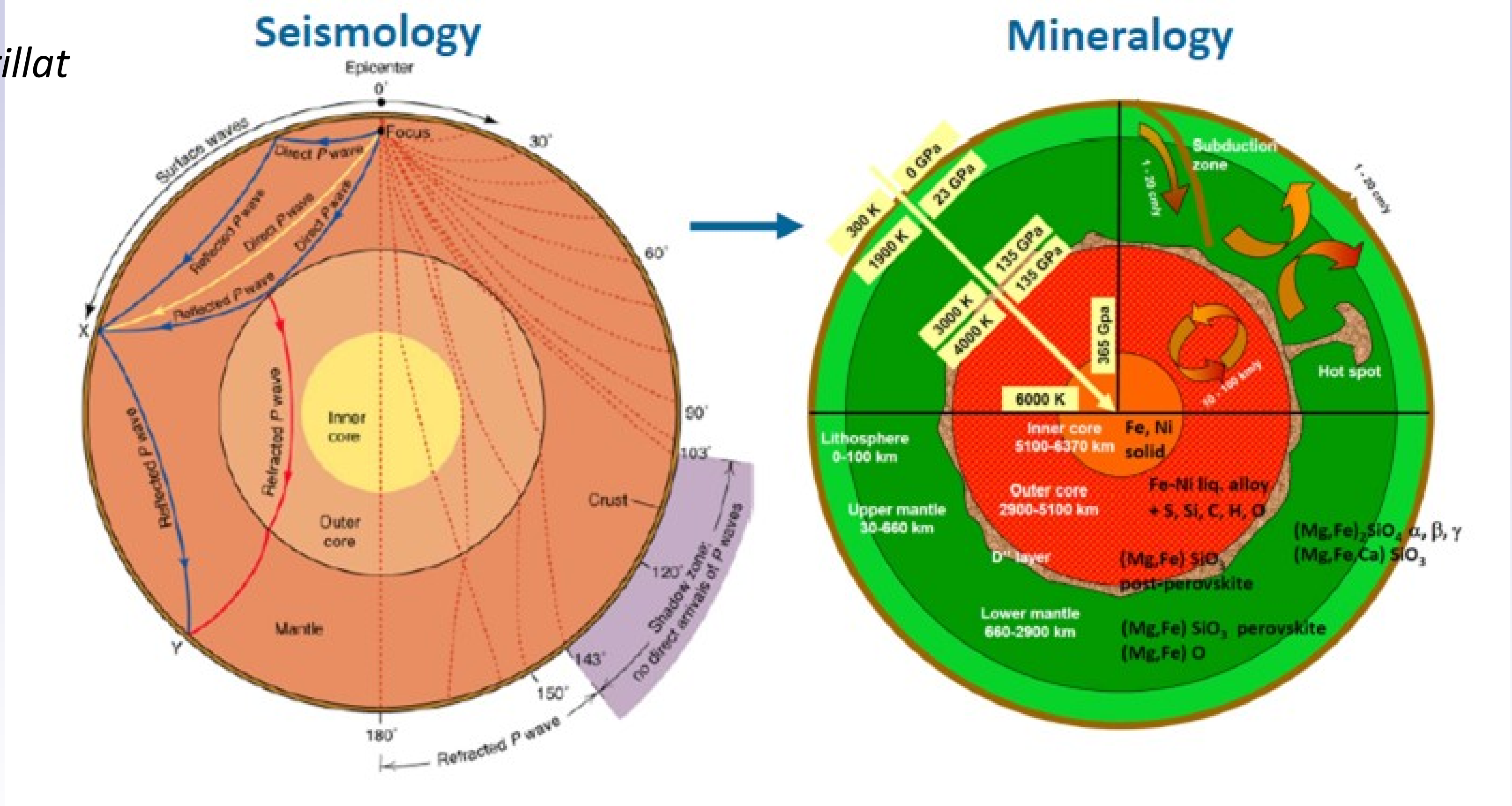


Gaussian fit of the position of the W wire at each interface

ESRF Experimental report n° ES 33

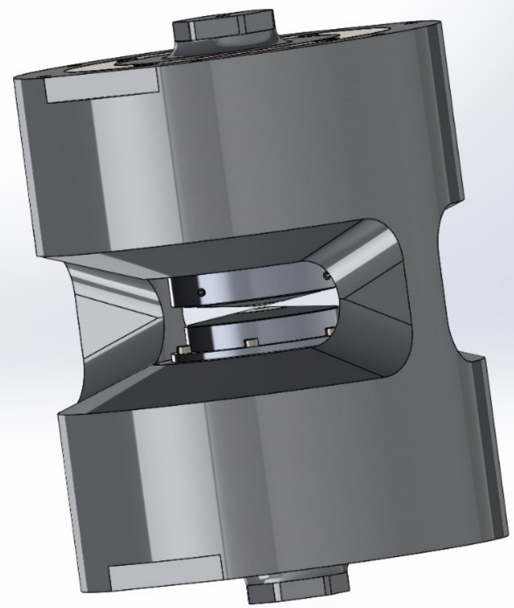
Measurements of shear modulus and attenuation coefficient Q_G^{-1} under mantle conditions and seismic frequencies

Coll. J.P. Perrillat



Attenuation and shear wave dispersion of Earth materials can be investigated at mantle conditions and seismic frequencies

PERSPECTIVES

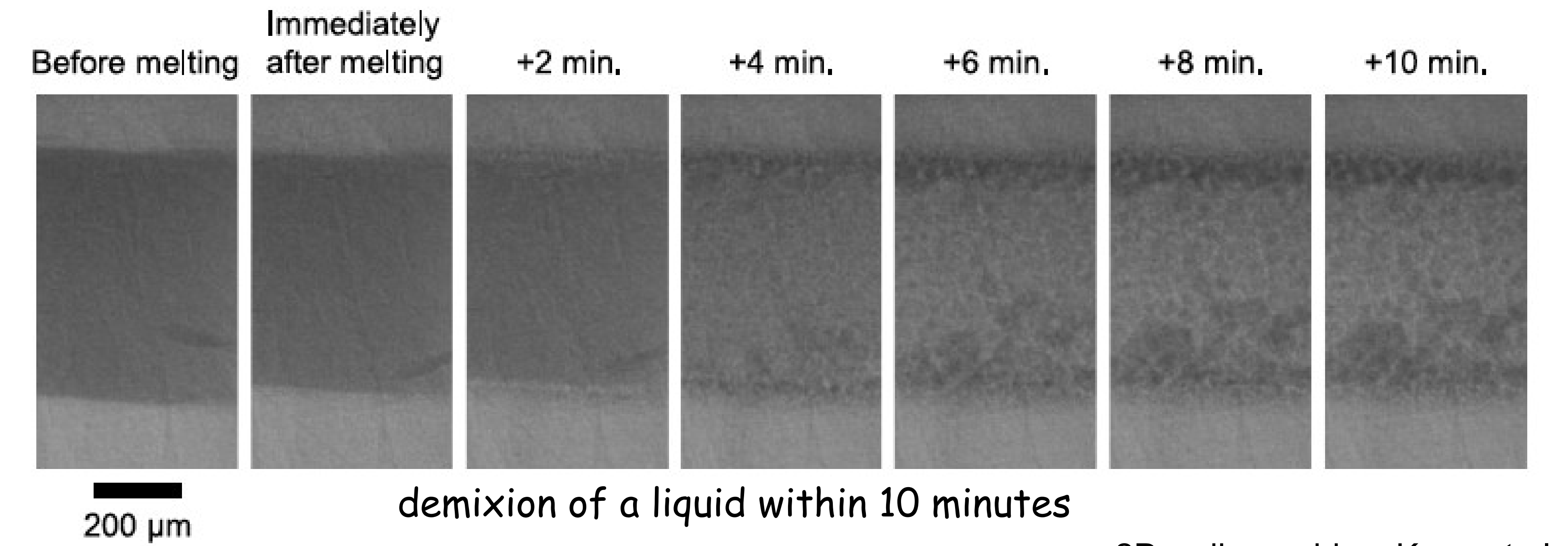


Development of a new Ultrafast Tomography Paris-Edinburgh Cell

UTOPEC

Our current acquisition times of the order of 10 minutes per tomogram represent a significant limitation for dynamic studies of samples that evolve at a rate that is greater than this acquisition time, or for samples which are mechanically unstable under HP and HT conditions

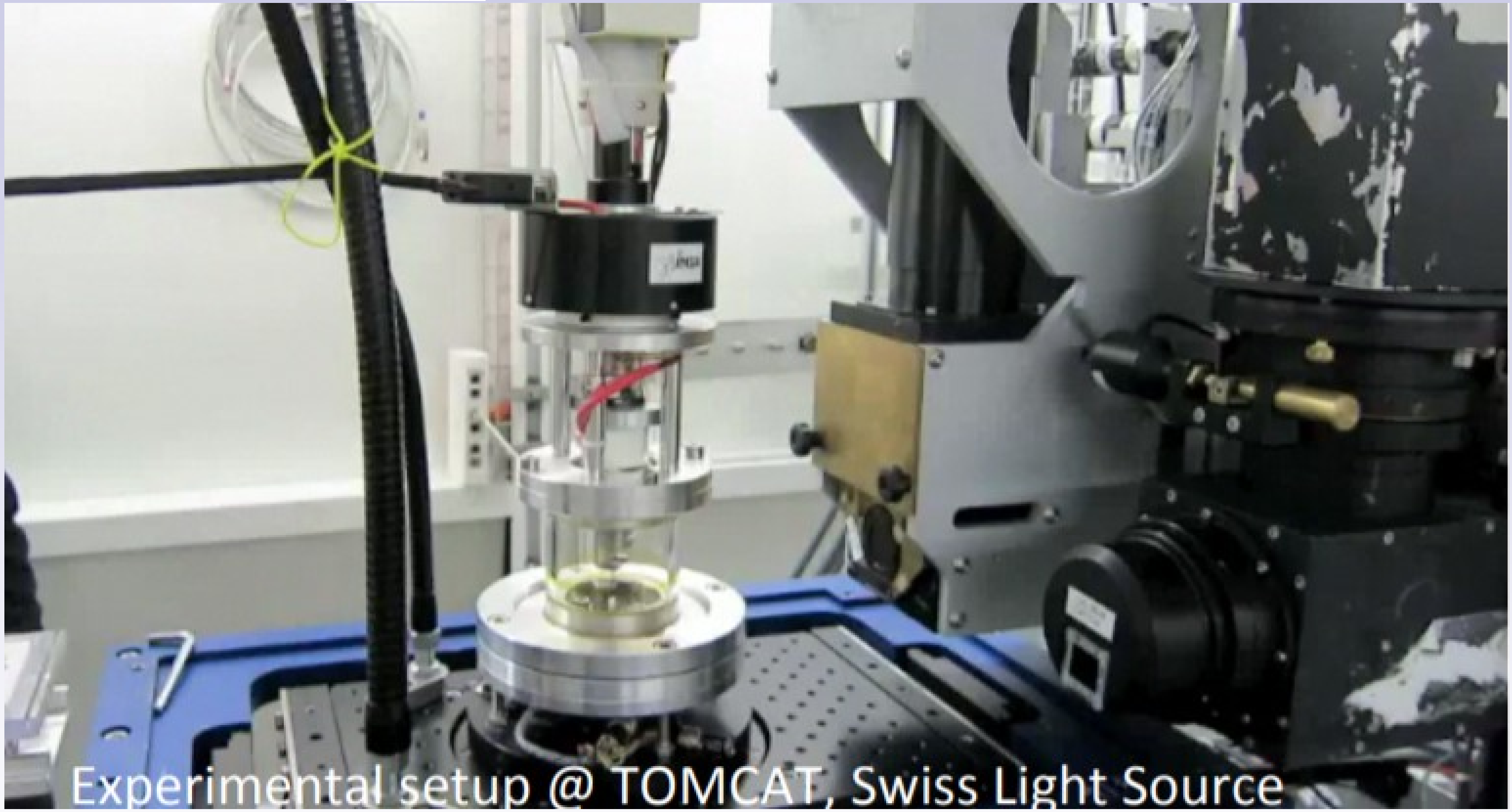
Fast Processes → We need Fast tomography



demixion of a liquid within 10 minutes

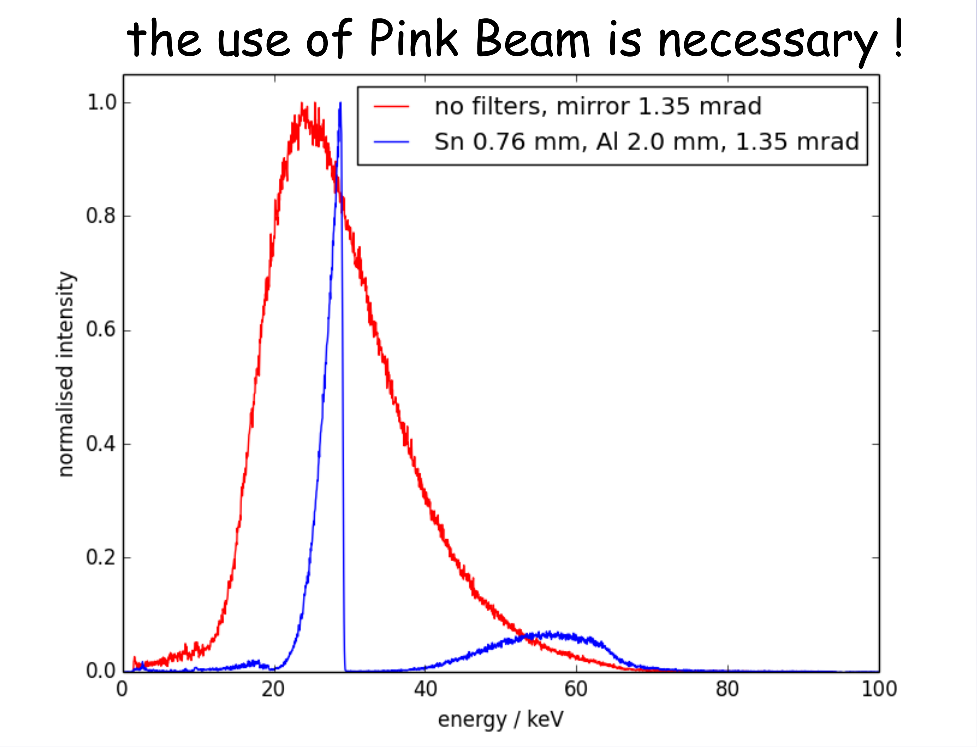
2D radiographies; Kono et al. 2015

- Fast detector 🙂



Recent developments in synchrotron tomography have greatly reduced the acquisition time that can be expected, and second or even sub-second tomography is possible. For that Fast detectors are now capable of kHz frame rate. This enables increasingly fast dynamic processes to be studied in situ such as fracture, solidification or phase transformations

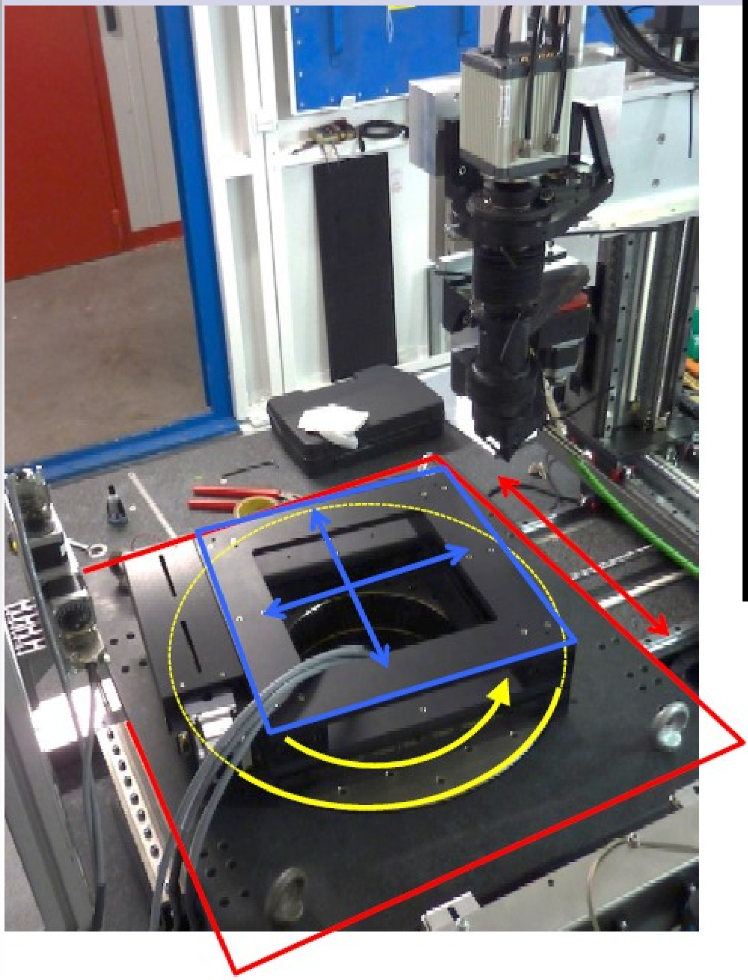
- Fast detector 🙂
- High Brillance of synchrotron source 🙂

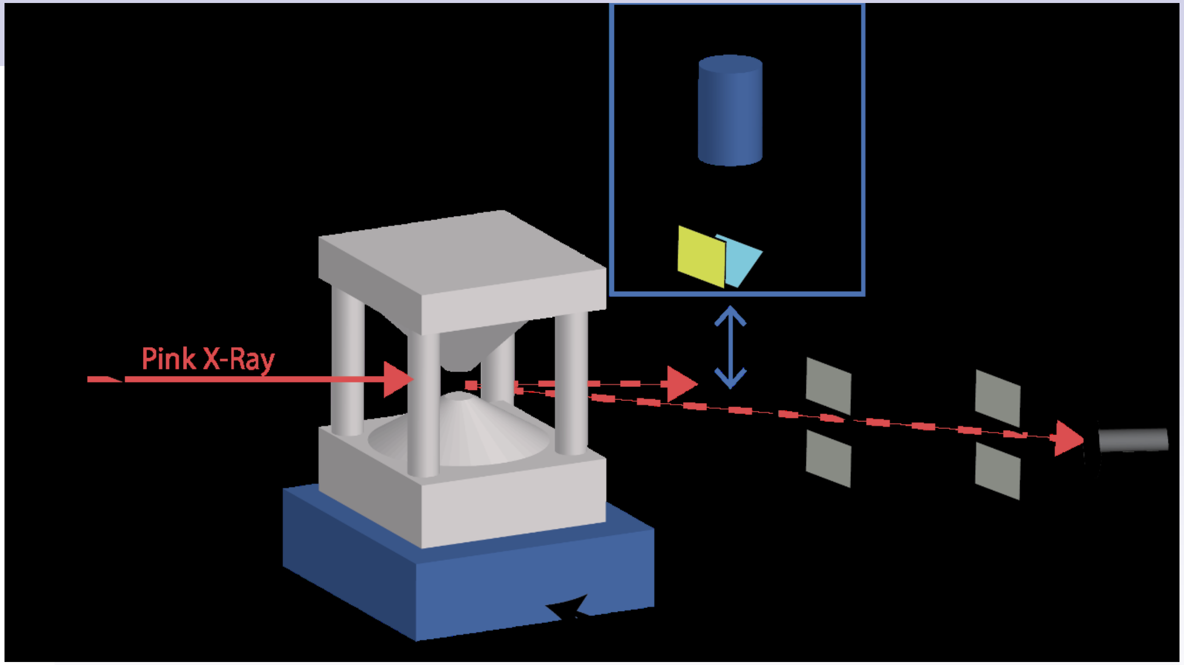


Crystal monochromator optics on a synchrotron beamline typically transmit less than 0.1% of the incident spectrum, and so greatly decrease the flux available. For this reason, monochromatic beams are less suitable for dynamic studies.

By using high-flux wide-bandwidth X-ray illumination (pink beam), the exposure time per radiograph can be reduced accordingly

- Fast detector
- High Brillance of synchrotron source
- Fast (and precise) rotation of the press

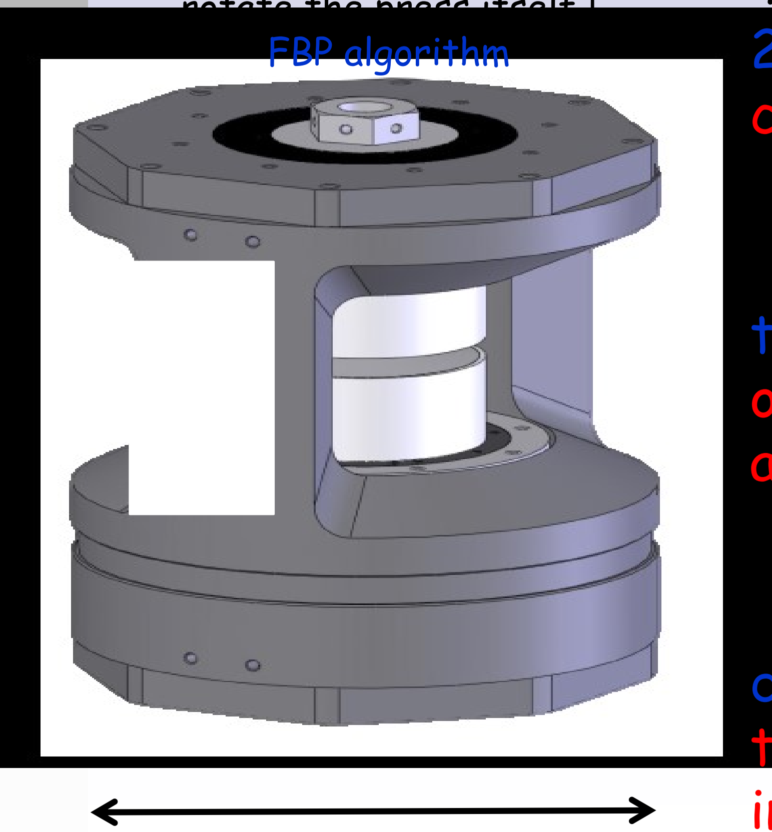




The PSICHE tomograph is based on a high-precision highload-capacity rotation. It has a maximum load capacity of more than 50 kg, and is capable of rotating at 60 rpm with an eccentricity of less than 150 nm

- Fast detector
- High Brillance of synchrotron source
- Fast (and precise) rotation of the press
- A new PE press with severe requirements

Since now we would like to rotate the press itself !



170 mm

The new press should allow working up to 15 GPa and 2000 K. So we need at least a press with 80 tons capacity and 60mm anvils diameter

In order to increase the quality of the reconstructed images, the angular opening will be maximized using a two-column system, like VX, but with a 165° angular opening (!!) in order to reduce the reconstruction artefacts currently observed

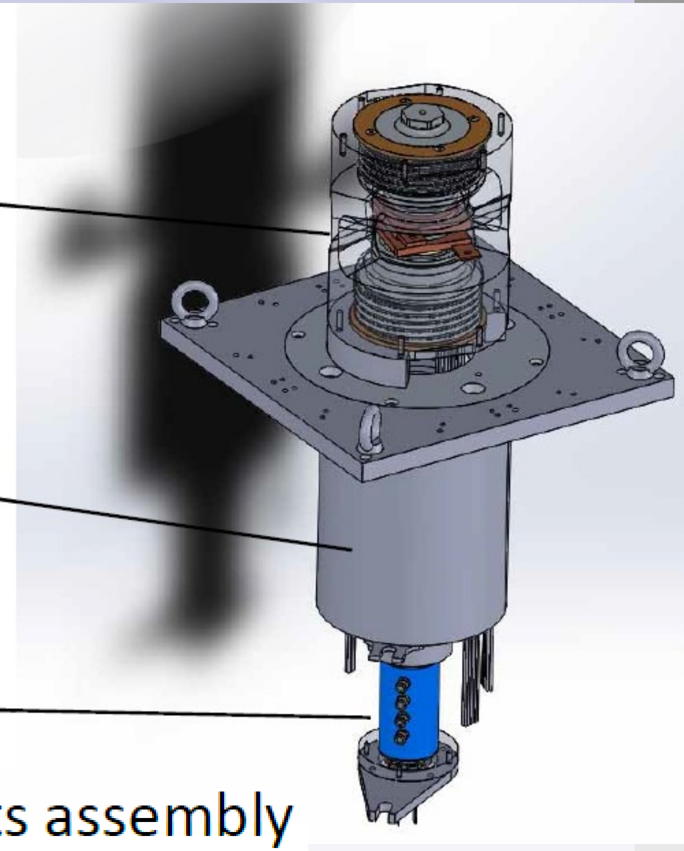
The press should also have a smaller diameter (170 mm) which will allow a smaller sample detector distance to be used. This is particularly important, as the limiting spatial resolution deteriorates with increasing sample-detector distance

The weight of the press should be less than 50 kgs

Rotary Union

Coll. G. Hamel

its adaptation to PSICHE beamline



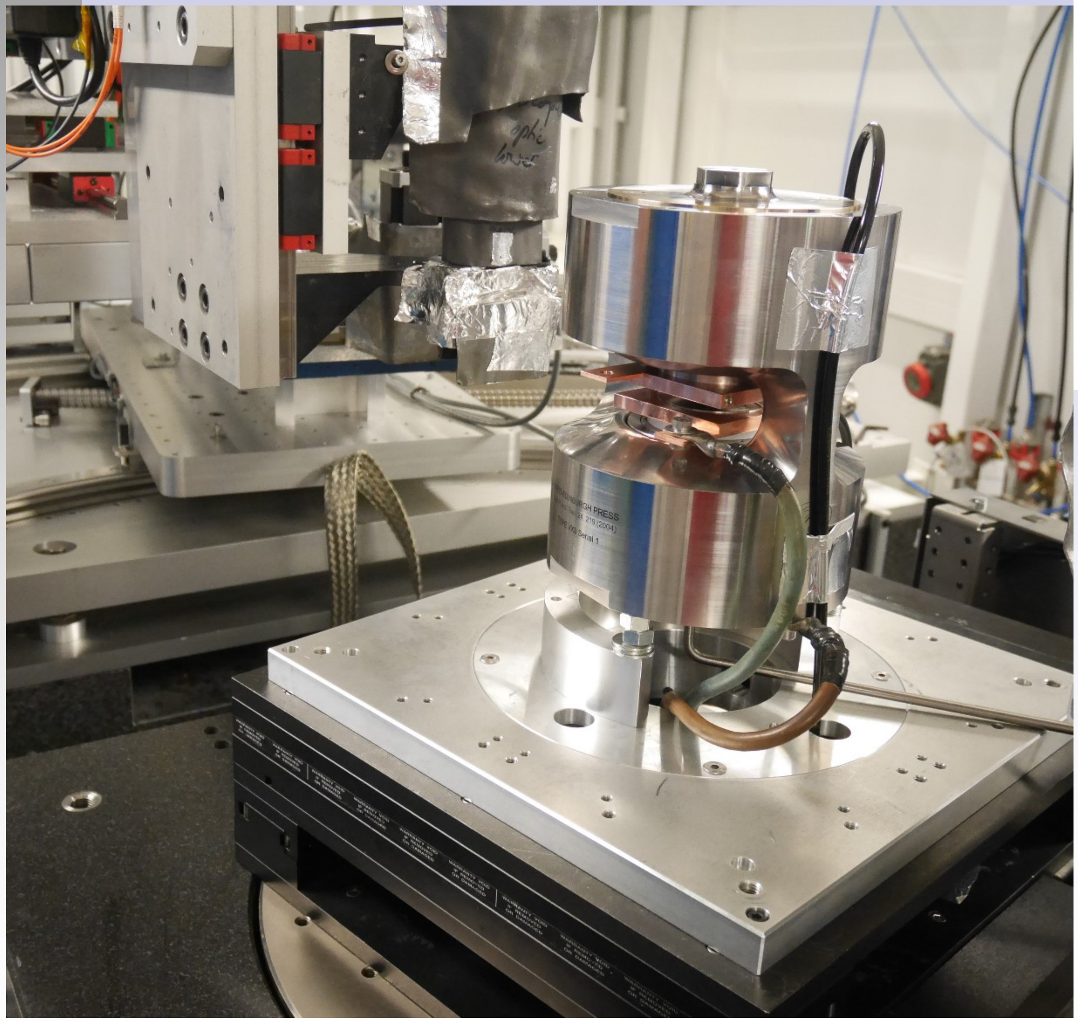
Rotary joints assembly

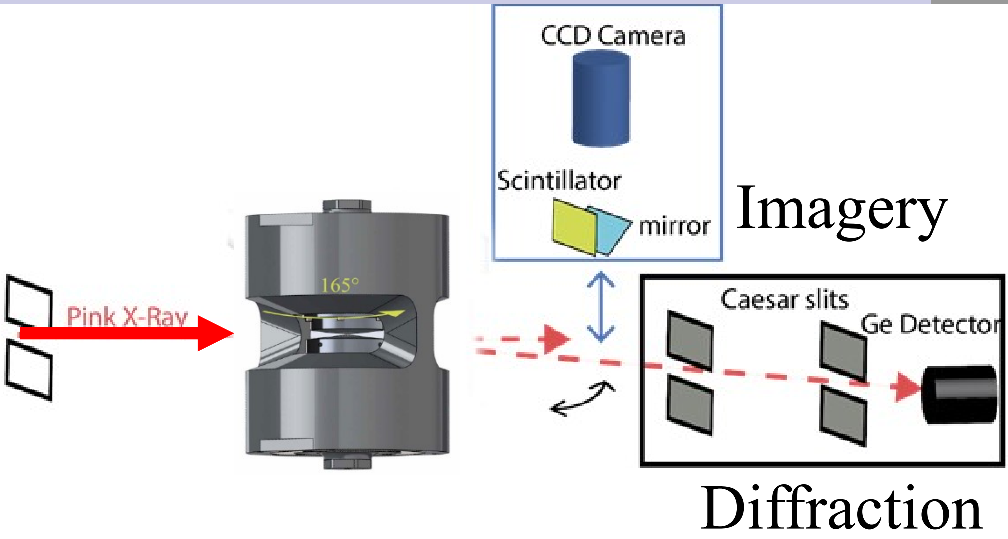
Basically we can rotate the press continuously

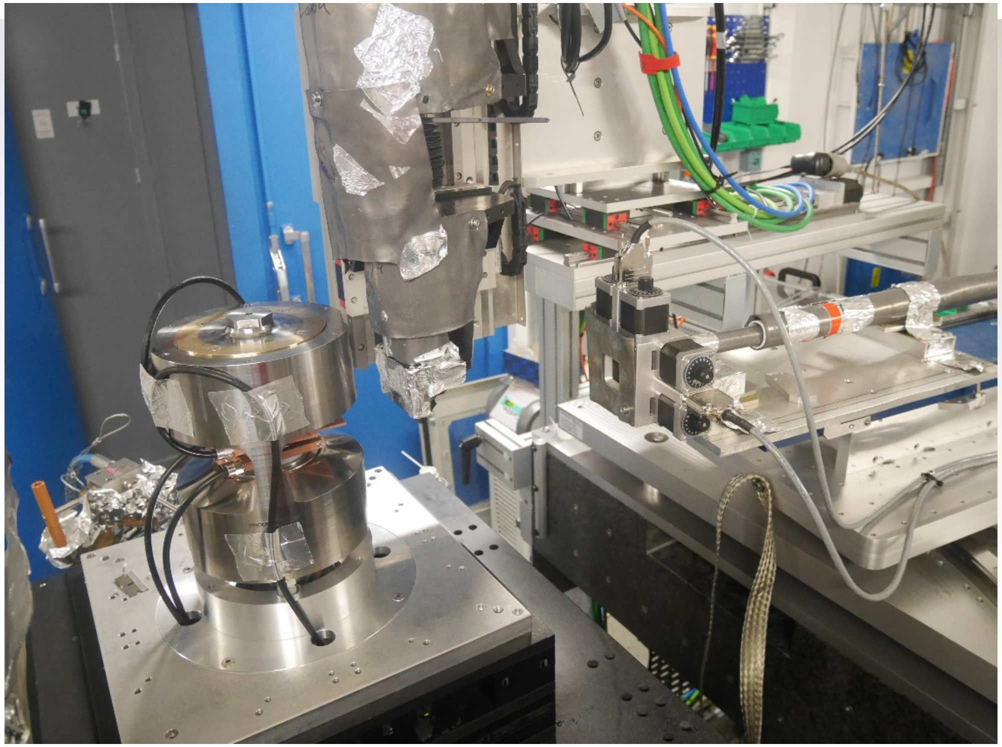
With this improvement, it is possible to acquire in principle a full tomogram in 0.5 s, corresponding to the maximum rotation speed of the stage

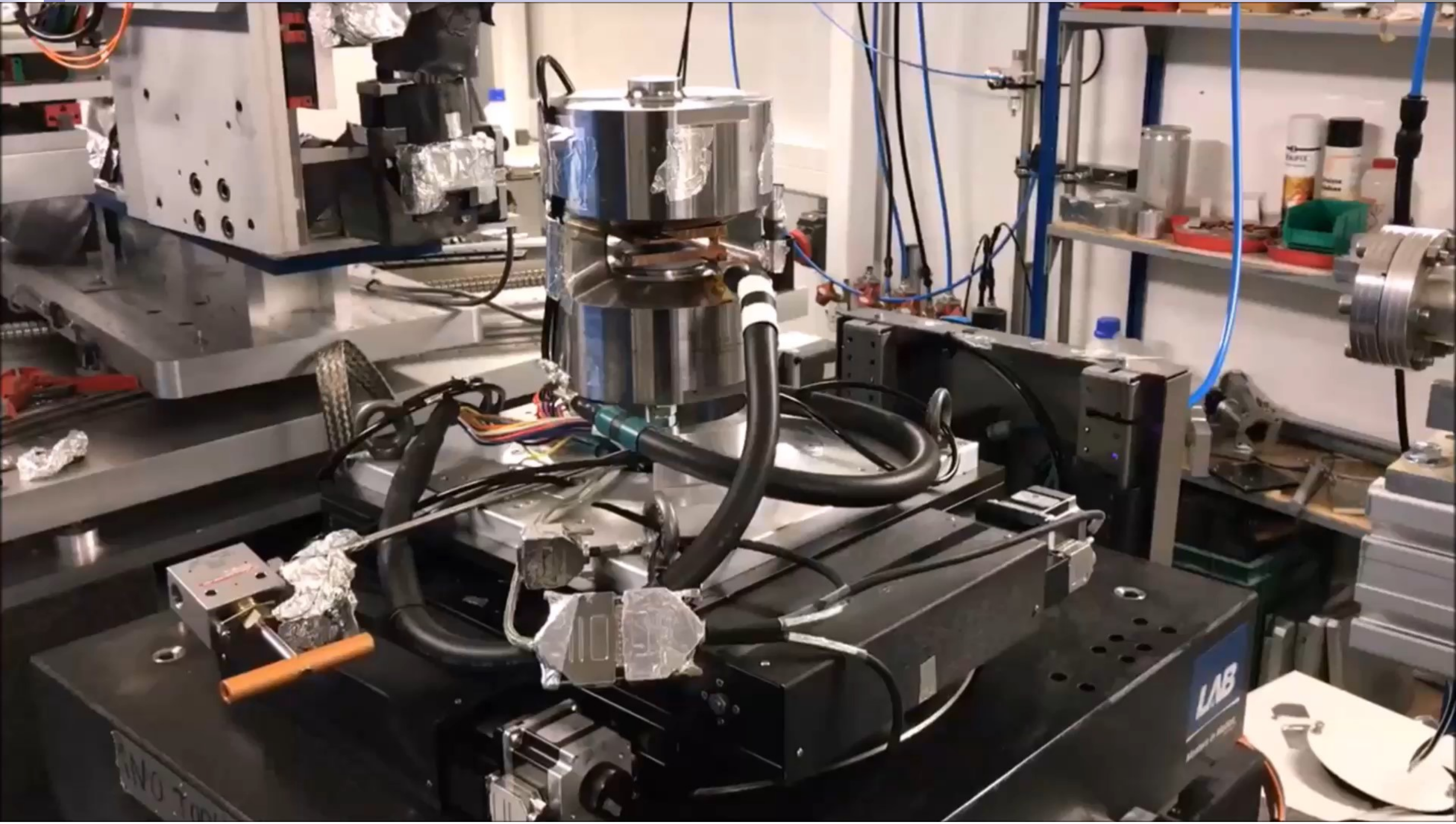
UltraFast acquisitions: < 1 tomo / s

UTOPEC

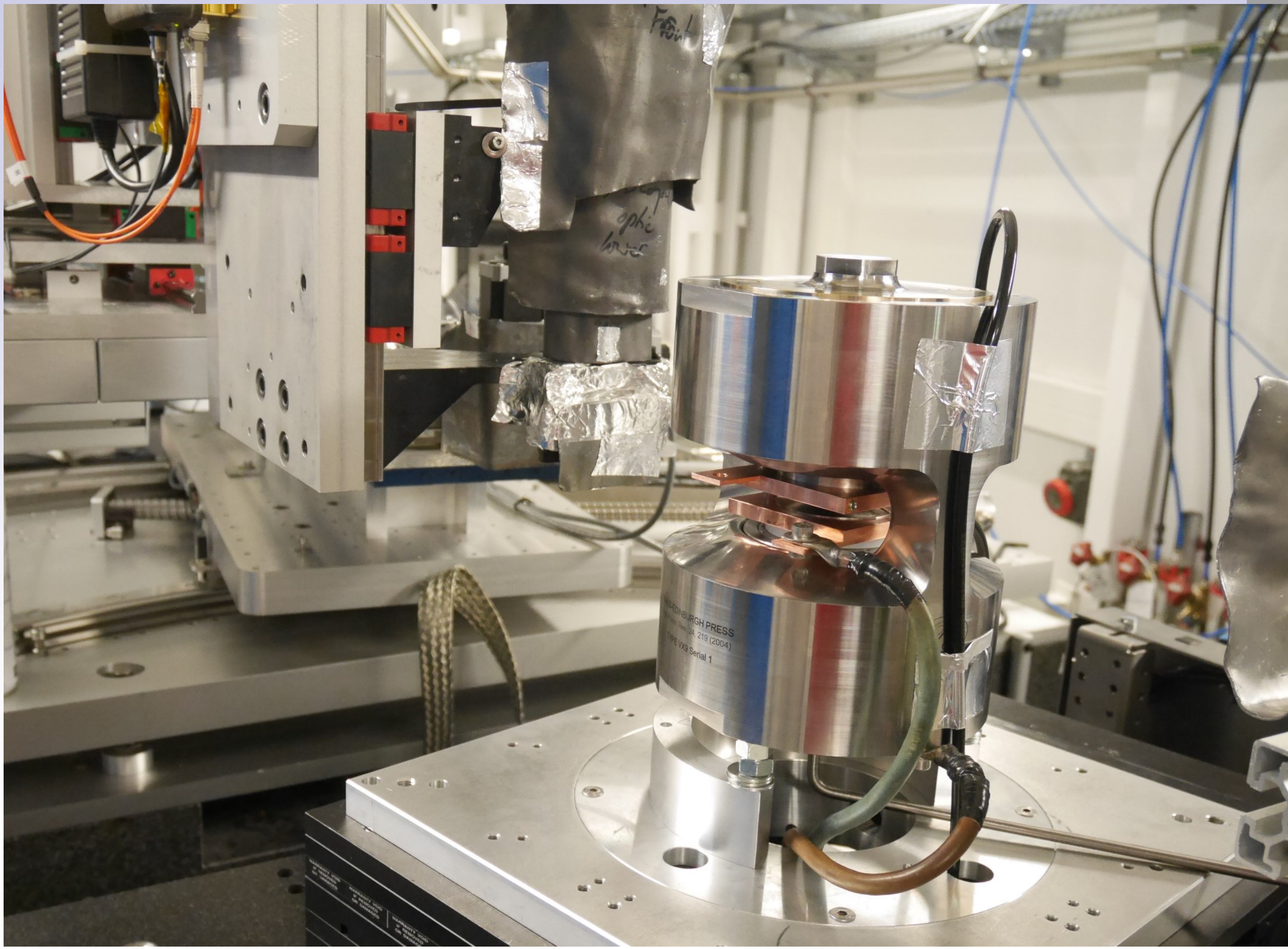



CCD Camera
Scintillator
mirror
Imagery
165°
Pink X-Ray
Caesar slits
Ge Detector
Diffraction





2 recent FAST HPHT Tomography studies



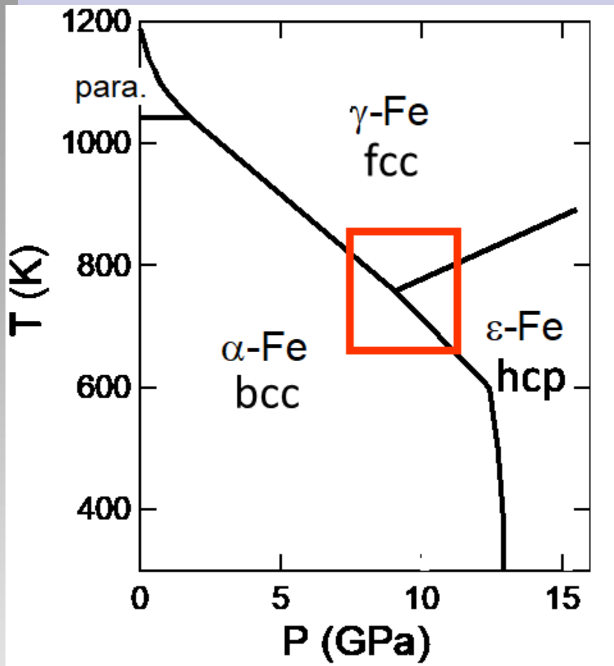
SOLEIL SYNCHROTRON

Psiché

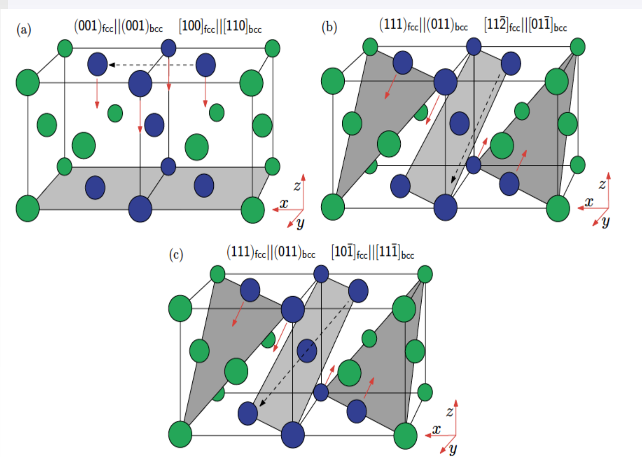
Pression, structure et imagerie par contraste à haute énergie

SOLEIL SYNCHROTRON

Martensitic Transitions of Iron at HP-HT



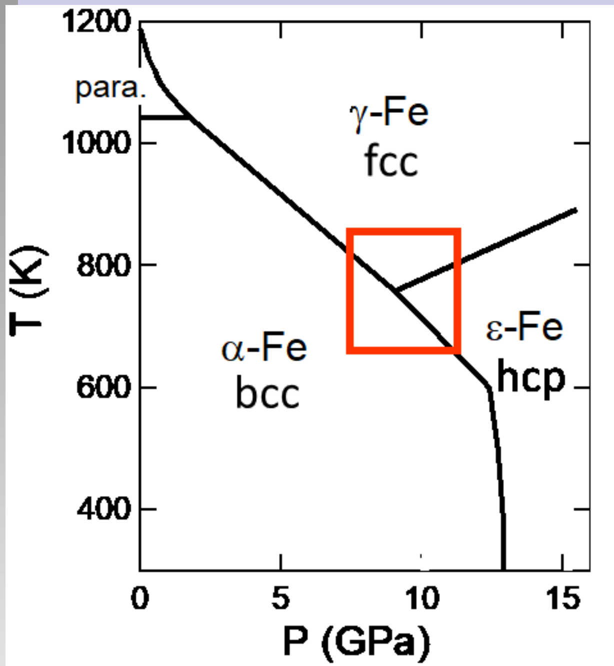
AIM : We studied the mechanisms involved in the HP HT martensitic transformations in iron. These martensitic transitions have profound implications in Fe-based materials technology as well as in planetary science with iron being the main component of terrestrial planetary core. By imaging in situ the orientation of the coexisting phases, we are trying to determine the orientations relations and habit planes for both transitions



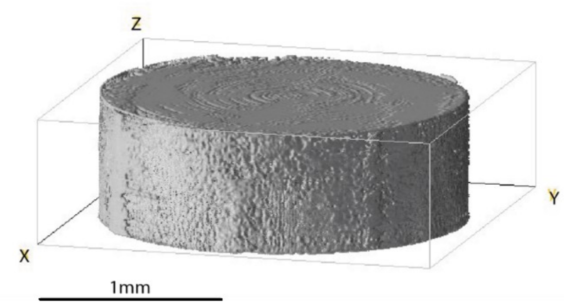
In the literature, three mechanisms of transformation have been proposed involving different orientation relationship between the fcc parent and the bcc phase and our study will be able to discriminate these mechanisms

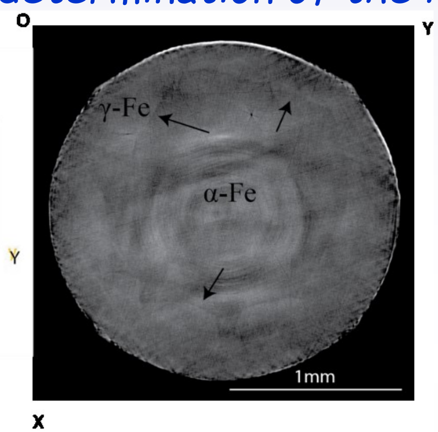
Coll. E. Boulard, A. Deawele et P. Loubeyre (CEA)

Martensitic Transitions of Iron at HP-HT

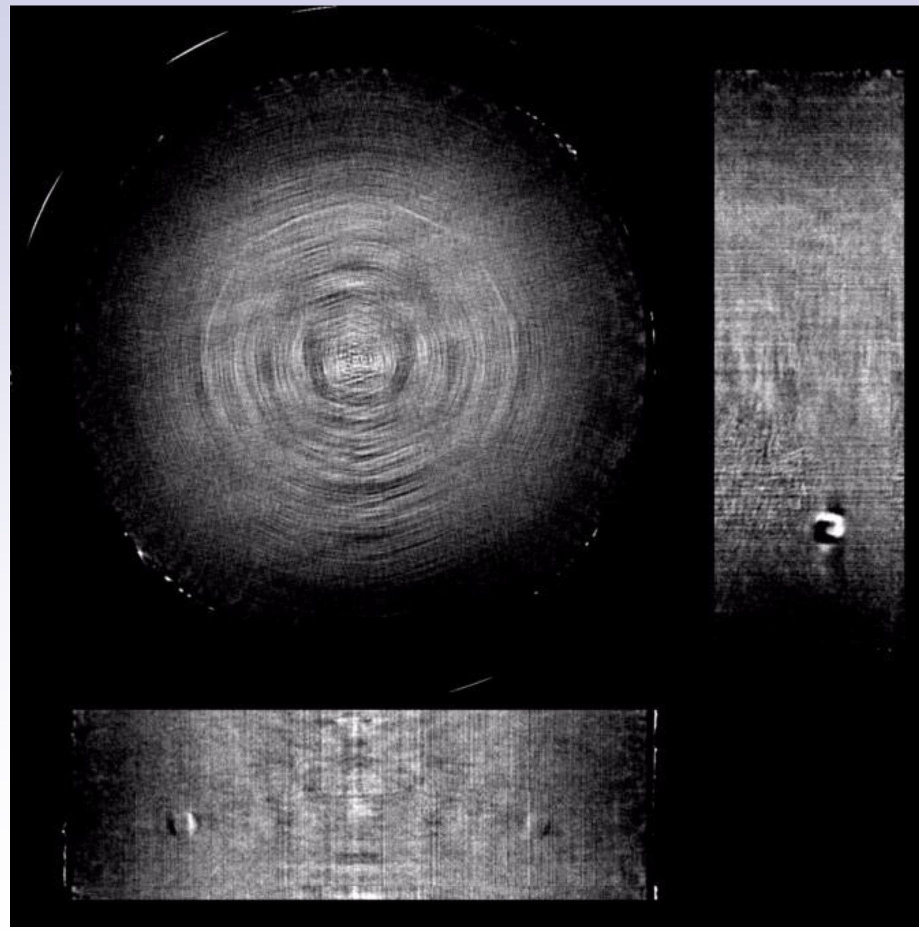


the coexistence of the bcc phase and the fcc phase at the transition and thus the determination of the habit plan



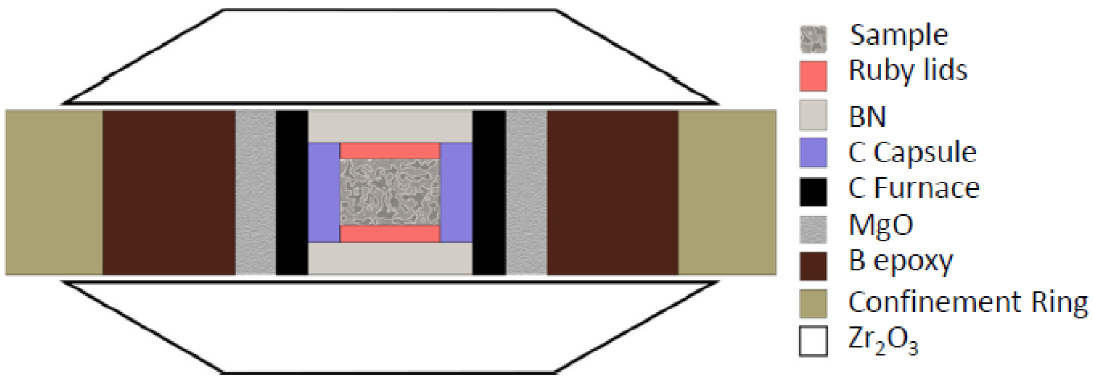


the gamma phase arrives by the edge

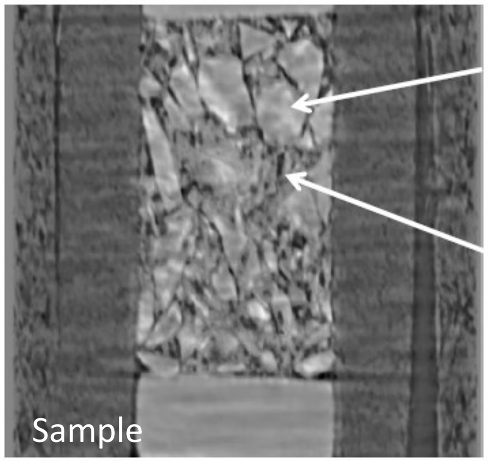


Our experiments provided for the first time information on the orientations relations and habit planes for α-γ and α-ε transitions in iron under HPHT

Transport of magma under mantle conditions

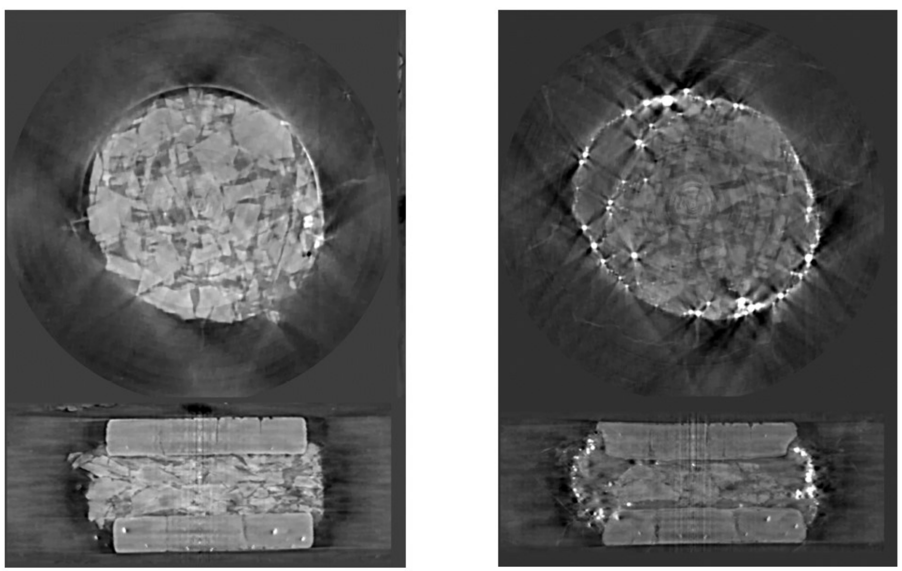


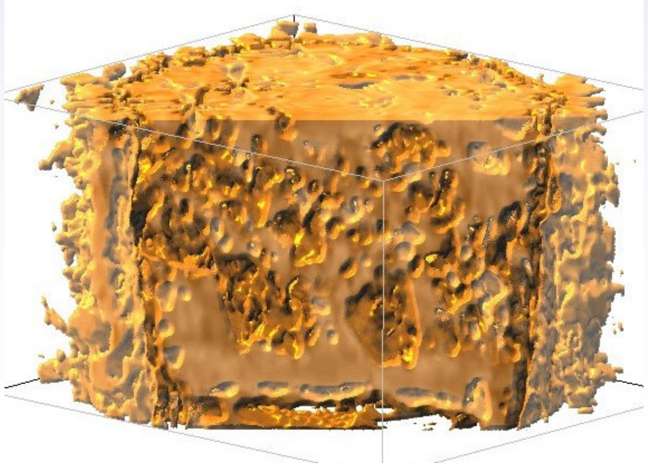
Sample assembly HPHT



3 GPa - 1200 K

3 GPa - 1600 K

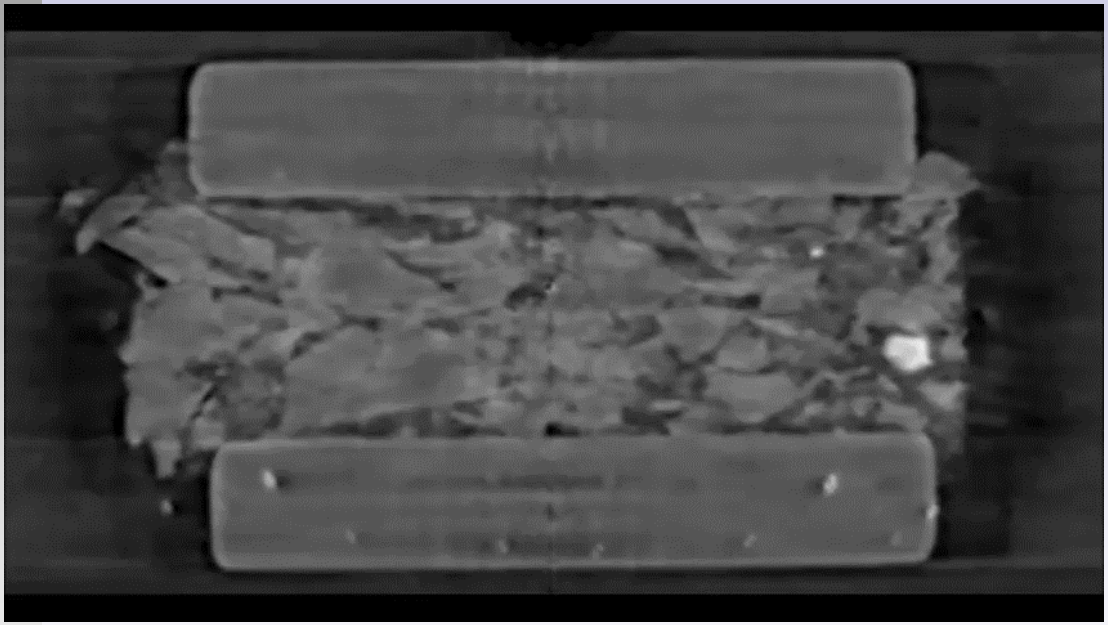


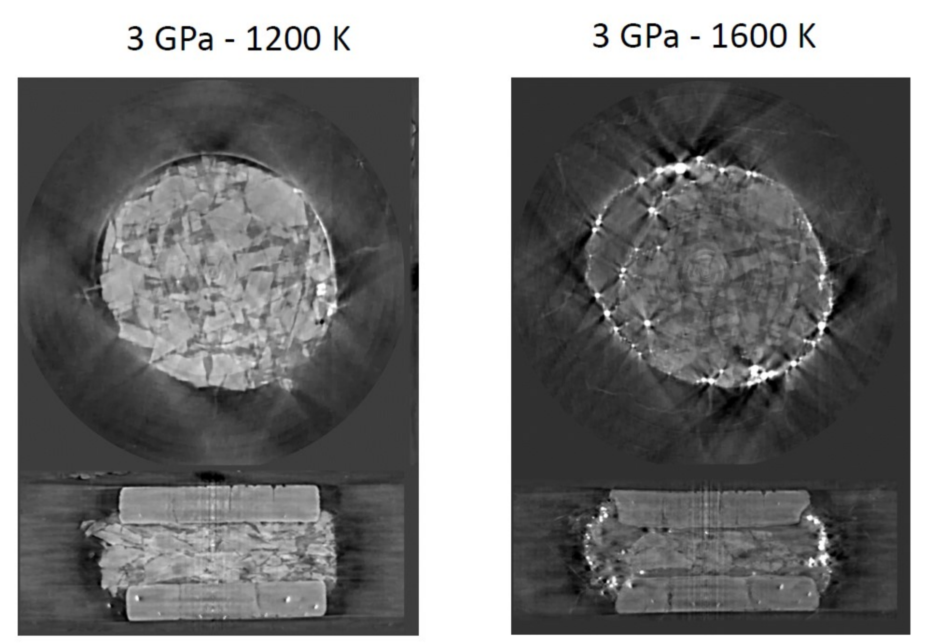


This study made it possible to image high P-T liquids

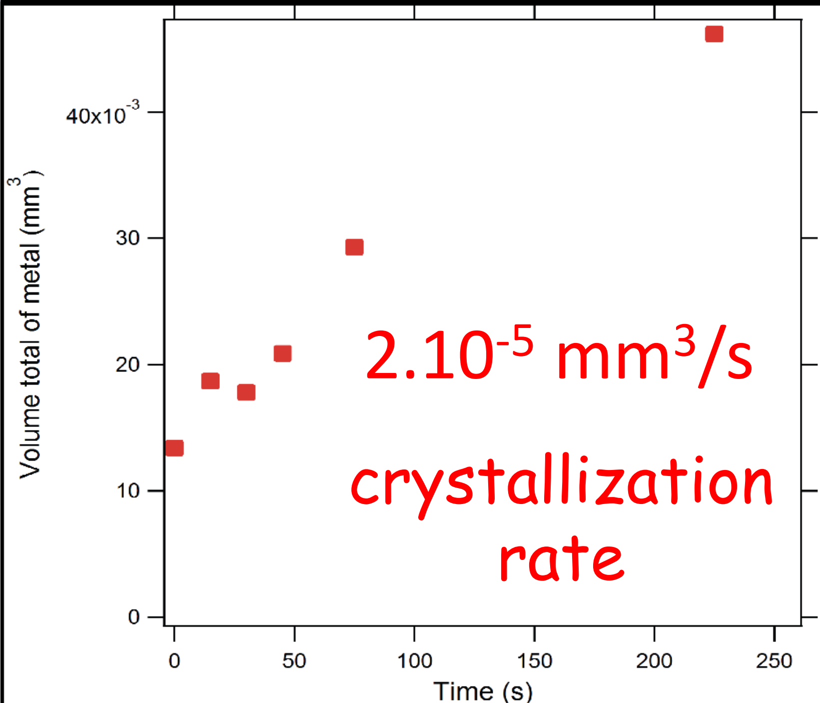
Coll. Eglantine Boulard

Transport of magma under mantle conditions

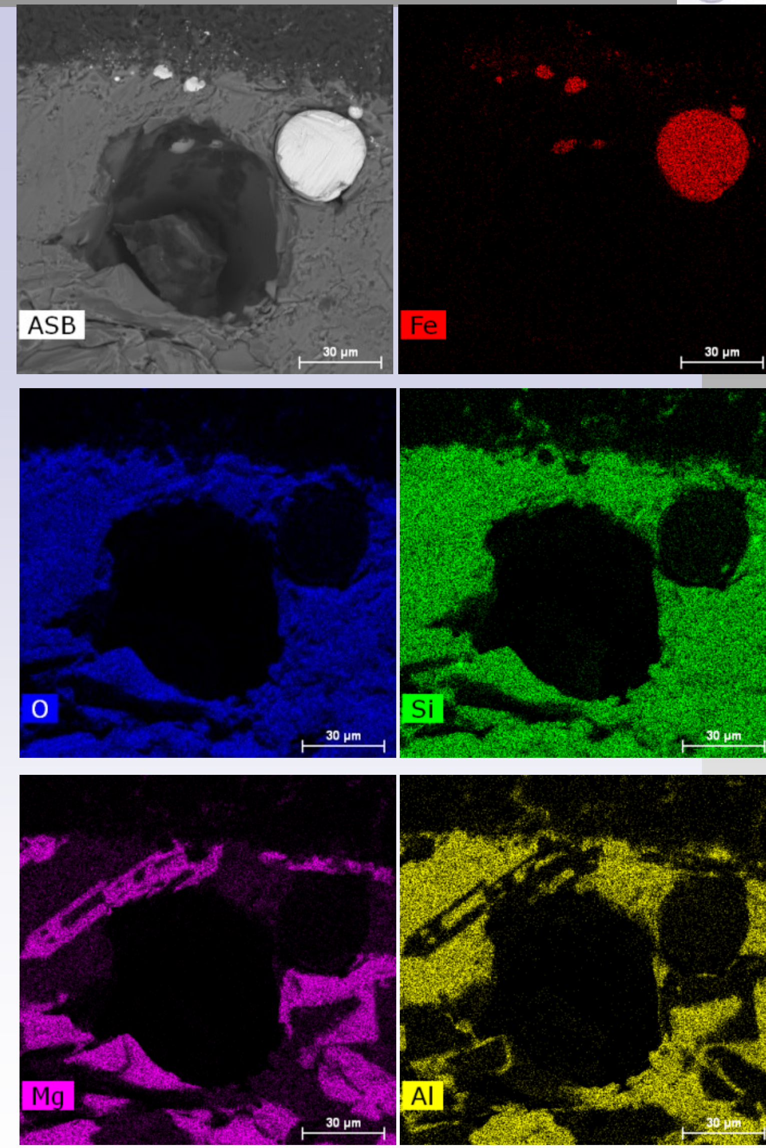




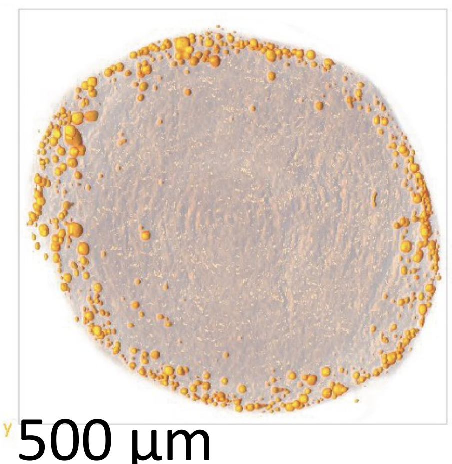
At 3 GPa and above 1100 K, strong deformation of the sample chamber is observed as its volume is reduced drastically due to melting of the rhyolite material. In a few minutes and with increasing temperature the melt reorganized itself toward the border of the capsule. When temperature reached 1600 K, we observed rapid and progressive demixing of liquid iron from the melt that precipitate as iron metallic spherules, probably due to reduction of iron in the melt by the graphite capsule. The fast acquisition time allow us to demonstrate the possibility to measure the iron crystallization rate in situ

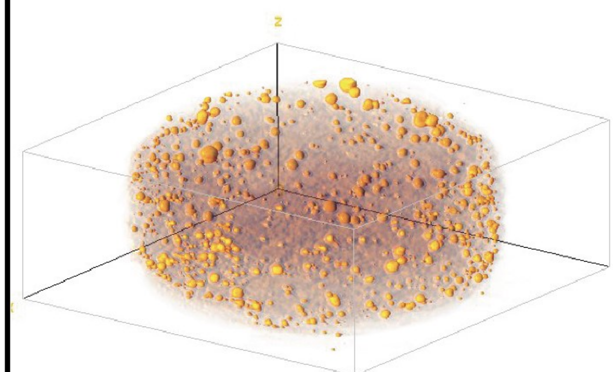


Coll. E. Boulard



SEM confirms that spherules are only Fe





We can obtain quantitative data with our new fast tomography technique at HPHT

New scientific possibilities coming with the ESRF upgrade will allow a better resolution (spatial and temporal) ! It will provide new scientific opportunities for original and unique studies of phase transitions, density, crystallization and deformation in extreme P/T/Stress conditions

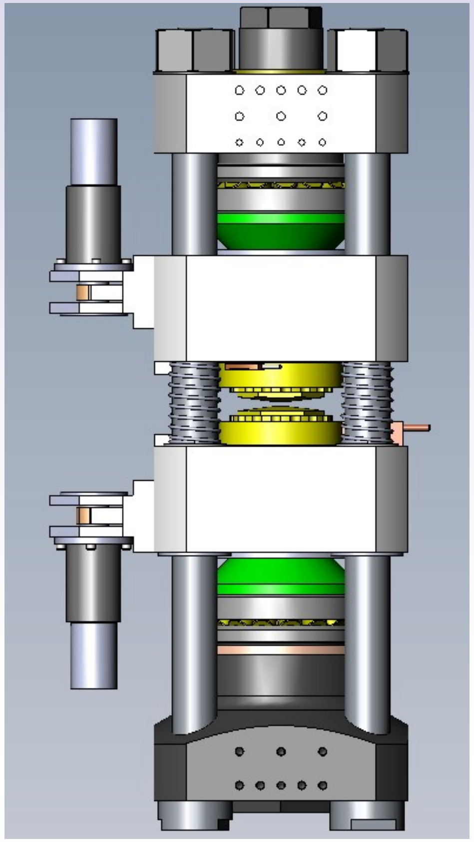
Absorption Tomography **+** **Diffraction** Tomography **+** **High P/T/stress PE cell**

(large beam, fast)

- Density
- Microstructure
- Porosity
- Defects
- Phase content

(micro beam, long)

- Phase distribution - content
- Orientation (texture)
- Crystallite size/microstrain
- Degree of crystallinity



Bibliography

On PE press :

J.M. Besson et al., Physica 180&181B, 907 (1992).
S. Klotz, et al. High Press. Res., 24, 219–223 (2004).
S. Klotz, Techniques in High Pressure Neutron Scattering, CRC press, 2012.

On tomography with PE press :

J. Philippe et al. (2016) Rotating tomography Paris–Edinburgh cell: a novel portable press for micro-tomographic 4-D imaging at extreme pressure/temperature/stress conditions, High Pressure Research, doi:/10.1080/08957959.2016. 1221951.
M. Alvarez-Murga et al. (2017) Development of synchrotron X-ray micro-tomography under extreme conditions of pressure and temperature, Journal of Synchrotron Radiation, 24, 240-247.
M. Berg et al. (2018) Rapid core formation in terrestrial planets by percolative flow: in-situ imaging of metallic melt under high pressure/temperature conditions. Frontiers in Earth Science, 6, 77.
E. Boulard et al. (2018) High-speed tomography under extreme conditions at the PSICHE beamline. J. Synchrotron Rad. 25, 818-825.

Acknowledgments

IMPMC : J. Philippe, E. Boulard, A. Clark, G, Morard, F. Bergame, M. Morand, G. Hamel and S. Klotz

Univ. Lyon : J.P. Perrillat, E. Giovenco

Univ. of Edinburgh : G. Bromiley, M. Berg (and **Cambridge** : S. Redfern)

ESRF : M. Mezouar, M. Alvarez-Murga, S. Bauchau, G. Garbarino

Néel: J.L. Hodeau et A. Prat

DIAMOND : R. Atwood

SOLEIL : N. Guignot, A. King, J.P. Deslandes, J.P. Itié

This work is supported by the French "Réseau Hautes Pressions", DIM OXYMORE and MITI of CNRS