

# Cluster de Computación MALTA

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GRUPO MALTA-CONSOLIDER  
UNIVERSIDAD DE OVIEDO

## Cronología

- Junio 2008: Adquisición del equipo
- Septiembre – Octubre de 2008: recepción de los componentes
- Octubre/noviembre/diciembre de 2008: instalación eléctrica
- Diciembre 2008: instalación sistema operativo
- Febrero 2009: LoadLeveler IBM
- Junio 2009: Adecuación de la sala (aire acondicionado)
- Julio 2009: puesta en marcha “oficial” del cluster de computación MALTA
- Junio 2010: Instalación nuevos componentes (>50% calculo + 3Xespacio disco)
- Enero 2011:
  - tiempo de disponibilidad **100%**
  - número de trabajos ejecutados ~**150.000**



## Ampliación 2010

10 servidores IBM Blade HS22, cada uno con:

- 2 procesadores Intel HexaCore 5650 a 2.66 Ghz / 12 MB caché / 1.333 MHz FSB / 95W
- 48 GB de RAM (12x4 GB)
- 2 discos SAS Hot Swap de 300 GB / 10Krpm
- 2 interfaces Gigabit y capacidad Concurrent KVM integrada



1 servidores IBM Blade HS22, con:

- 1 procesador Intel Quad Core 5504 a 2.00 Ghz / 4 MB caché / 800 MHz FSB / 80W
- 12 GB de RAM (6 x 2 GB)
- 2 discos SATA Hot Swap de 500 GB / 7200 rpm
- 2 interfaces Gigabit y capacidad Concurrent KVM integrada



• 1 nuevo chasis **BLADE Center** (mismo modelo que los 3 actualmente disponibles)



28+1 nodos

224 núcleos



40+1 nodos

360 núcleos

## Especificaciones Técnicas

1.2TB RAM, 11TB (688GB RAM, 7TB)

### Chasis 1 – control + calculo

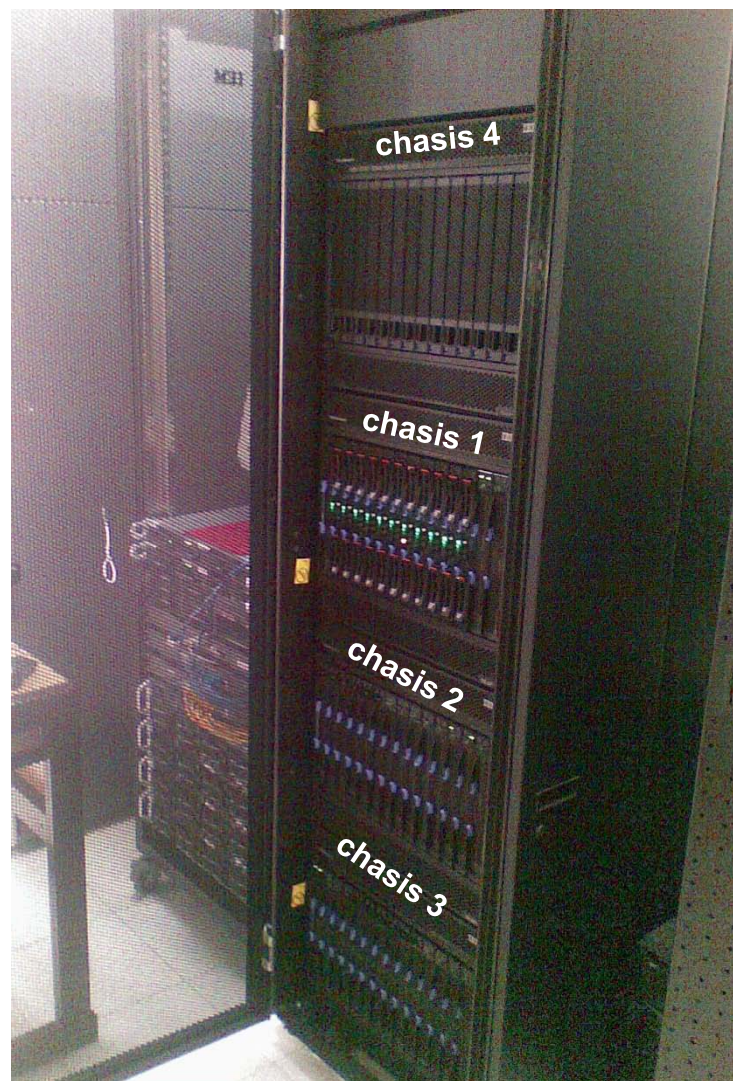
- 10 Intel Hexa Core 5650 a 2.66 Ghz
- 1 Intel Quad Core 5504 a 3 Ghz + 2x5450
- 12/16/32/48 GB de RAM
- discos SAS de 146/300/500 GB

### Chasis 2 – I/O Bounded (14 nodos)

- 2 procesador Intel QuadCore 5450 a 3 Ghz
- 16 (4x4) GB de RAM
- 2 discos SAS de 146 GB
- 2 interfaces Gigabit

### Chasis 3 – CPU Bounded (14 nodos)

- 2 procesador Intel Quad Core 5450 a 3 Ghz
- 32 (8x4) GB de RAM
- 1 discos SAS de 146 GB
- 2 interfaces Gigabit



## Software

### Compiladores

- gcc 4.1.2 (gfortran, g77, gcc, g++)
- Intel (ifort, icc) v11.081
- Python-2.4.3-24

### Librerías matemáticas

- BLACS
- CBLAS (BLAS Library for C)
- FBLAS (BLAS Library for FORTRAN)
- FFTW
- Intel mkl
- LAPACK
- ScaLAPACK

[www.malta-consolider.com](http://www.malta-consolider.com)

### Librerías de cálculo paralelo

- MPICH
- MPICH2
- OpenMPI

### Programas

- abinit
- gnuplot
- octave
- siesta
- VASP (4.7, 5.2)
- Wien2K
- Crystal06
- Gamess
- Quantum Espresso
- Programas “domésticos”



## Nivel de ocupación

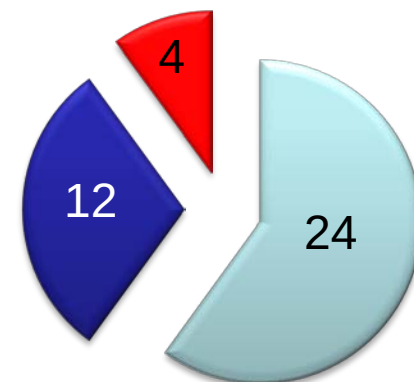
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| nodo16-1  | Avail  | 0   | 0   | Busy   | 8   | 9.02  | 9999 | Intel64 | Linux2 |
| nodo16-10 | Avail  | 0   | 0   | Idle   | 0   | 0.00  | 9999 | Intel64 | Linux2 |
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| nodo16-13 | Avail  | 0   | 0   | Idle   | 0   | 0.00  | 9999 | Intel64 | Linux2 |
| nodo16-14 | Avail  | 0   | 0   | Idle   | 0   | 0.00  | 9999 | Intel64 | Linux2 |
| nodo16-15 | Avail  | 0   | 0   | Idle   | 0   | 0.00  | 9999 | Intel64 | Linux2 |
| nodo16-2  | Avail  | 0   | 0   | Idle   | 0   | 0.00  | 9999 | Intel64 | Linux2 |
| nodo16-3  | Avail  | 0   | 0   | Idle   | 0   | 0.00  | 9999 | Intel64 | Linux2 |
| nodo16-4  | Avail  | 0   | 0   | Idle   | 0   | 0.03  | 9999 | Intel64 | Linux2 |
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| nodo32-7  | Avail  | 0   | 0   | Busy   | 8   | 8.06  | 9999 | Intel64 | Linux2 |
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| nodo48-2  | Avail  | 0   | 0   | Run    | 8   | 8.00  | 9999 | Intel64 | Linux2 |
| nodo48-3  | Avail  | 0   | 0   | Busy   | 12  | 12.00 | 9999 | Intel64 | Linux2 |
| nodo48-4  | Avail  | 0   | 0   | Busy   | 12  | 11.99 | 9999 | Intel64 | Linux2 |
| nodo48-5  | Avail  | 0   | 0   | Busy   | 12  | 12.01 | 9999 | Intel64 | Linux2 |
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Intel64/Linux2 41 machines 28 jobs 176 running tasks  
 Total Machines 41 machines 28 jobs 176 running tasks

The Central Manager is defined on malta

## Dedicación de los nodos

paralelo serie mixto

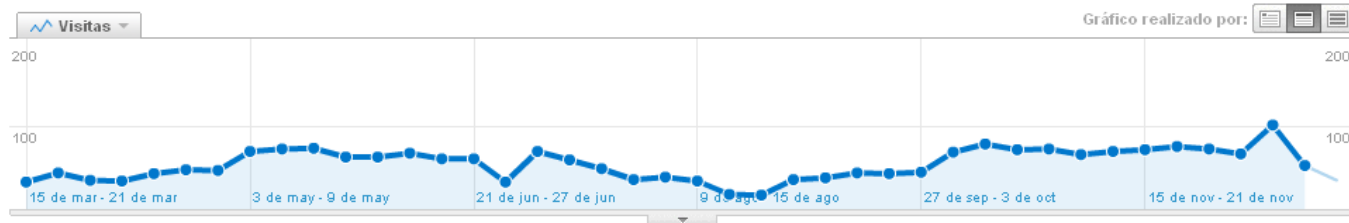


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| sexpress  | 02:00:00                | undefined                | 78            | 80           |
| p12small  | 1+00:00:00              | undefined                | 4             | 120          |
| ssmall    | 1+00:00:00              | undefined                | 78            | 80           |
| psmall    | 1+00:00:00              | undefined                | 119           | 176          |
| p12medium | 7+00:00:00              | undefined                | 4             | 120          |
| smedium   | 7+00:00:00              | undefined                | 78            | 80           |
| pmedium   | 7+00:00:00              | undefined                | 119           | 176          |
| p12large  | 19+18:00:00             | undefined                | 4             | 120          |
| slarge    | 19+18:00:00             | undefined                | 76            | 80           |
| plarge    | 19+18:00:00             | undefined                | 119           | 176          |

## Perspectivas de futuro:

- Instalación de nuevo software
  - sugerencias usuarios
  
- Mantenimiento cluster
  - renovación de licencias
  - actualización sistema operativo + gestor de colas
  - actualización software científico
  
- Nuevo chasis de paralelización masiva
  - cuarto chasis dedicado únicamente a cálculo paralelo
  - red de interconexion infiniband
  
- Directrices última anualidad
  - unidad de almacenamiento
  - acondicionamiento
  - reponer, actualizar, reparar componentes, etc.

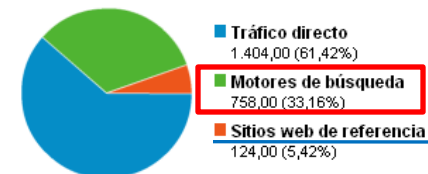
## Año 2009



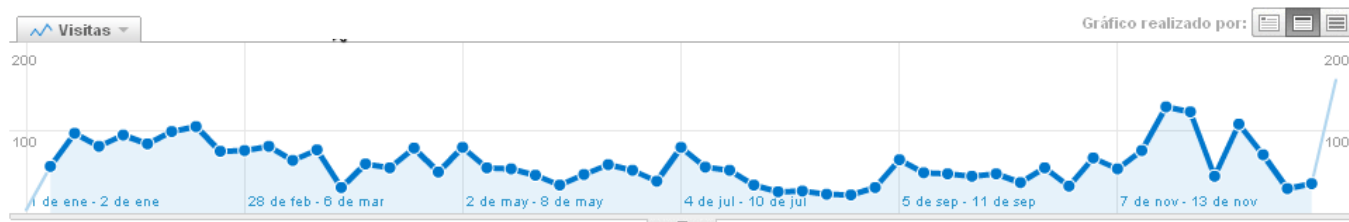
### Uso del sitio

2.286 Visitas

77,87% Porcentaje de rebote



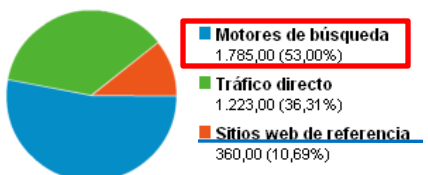
## Año 2010



### Uso del sitio

3.368 Visitas

77,49% Porcentaje de rebote



## Mejoras

- Actualizaciones periódicas
- Ofertas de empleo, Conferencias, charlas, seminarios, convocatorias, noticias, links





Vacío, Criogenia, mecanizado y soldadura de precisión  
Equipos a Presión nº: FAP-147 - EIP-2-75 - ERP-2-3

# REUNIÓN MALTA-CONSOLIDER 2011

Miraflores de la Sierra - 23/27 Enero de 2011

## Infraestructura VII - Centro de Mecanizado -

Samuel Gilliland  
04/02/10 11:40



# Introducción

## Cryovac S.L.

- Se fundió en 1996
- Especializa en criogenia, mecanizado y soldadura de precisión.
- Es fabricante, instalador y reparador de aparatos a presión (FAP-147, EIP-2-75 y ERP-2-3)
- Tenía orígenes industriales pero está entrando cada vez más en el mundo de investigación... sobre todo a altas presiones.
- Es el taller oficial del grupo MALTA
- Apoya al grupo MALTA suministrando principalmente (pero no exclusivamente) celdas y equipos a presión.
- Tiene un centro de mecanizado subvencionado por MALTA.
- Ofrece precios especiales a investigadores MALTA.
- Tiene empleados muy guapos.





# Equipos



Centro de mecanizado



Laboratorio de vacío



Laboratorio de control de calidad



Taller



Vacío, Criogenia, mecanizado y soldadura de precisión  
Equipos a Presión n°: FAP-147 - EIP-2-75 - ERP-2-3



# Nuestros productos

## Anevac CRYO

- Líneas de transferencia criogénica
- Dispositivos criogénicos aislados por vacío (válvulas, separadores de fase etc.)
- Dewars a medida
- Criostatos
- Instalaciones criogénicas (recuperación de He, resonancia magnética, conservación criogénica etc.)



## Anevac VACUUM

- Cámaras de vacío
- Componentes de vacío (bridas, reducciones, flexibles, codos, colectores, pasamuros etc.)
- Equipos de vacío (bombas, válvulas, instrumentación etc.)
- Sistemas portátiles de vacío

## Anevac SPUTTERING

- Sistemas de deposición de capas delgadas
- Fuentes magnetrón
- Equipos auxiliares (precámaras, portasustratos etc.)

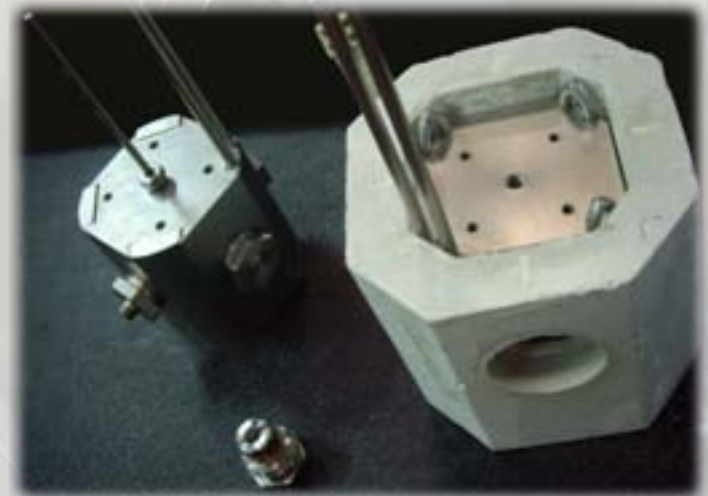
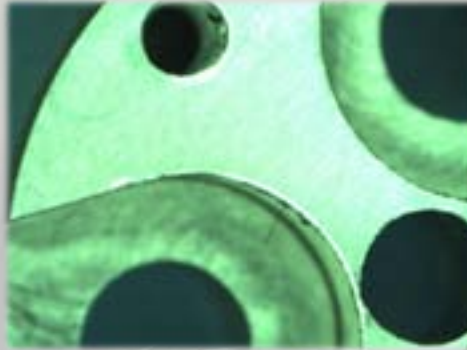
# Nuestros productos

## Anevac PRESSURE

- Instalaciones industriales a alta presión (oxicombustión, envasado de gases, PSA, mezcladores etc.)
- Cámaras de atmósfera controlada
- Recipientes y reactores a presión
- Presurizadores de alimentos



## Anevinco



Celdas de gran volumen



Celdas DAC



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# Nuestros productos

## Anevac CONTROL

- Control automático de vacío
- Control de presión de gas
- Equipos para calibración de presión
- Control automático de temperatura
- Control de líquidos criogénicos
- Controladores programables (PLC) e interfaces

### Control de presión en celdas



Sistema automatizada



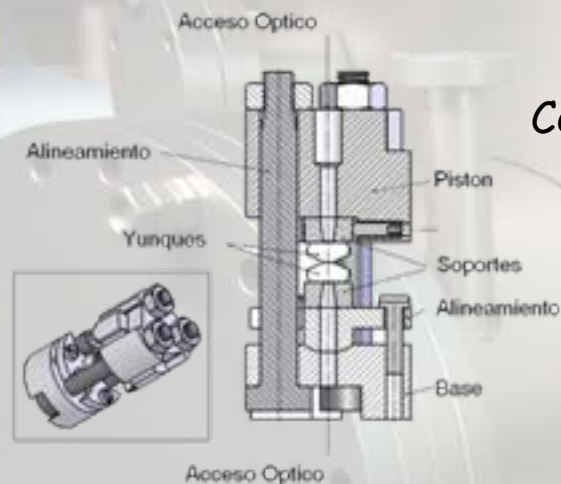
Controlador para celda  
de membrana



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# Nuevos proyectos

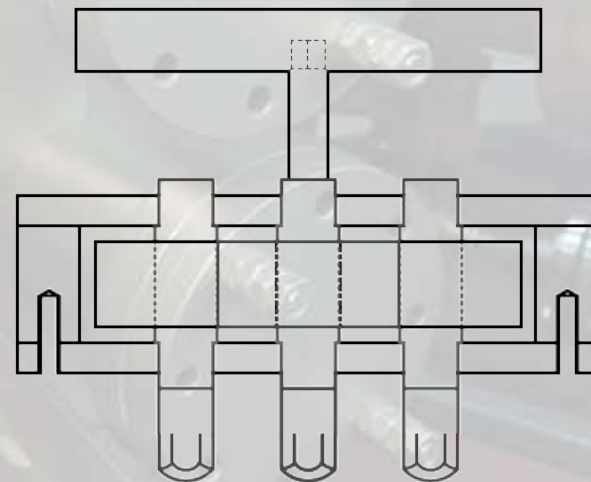
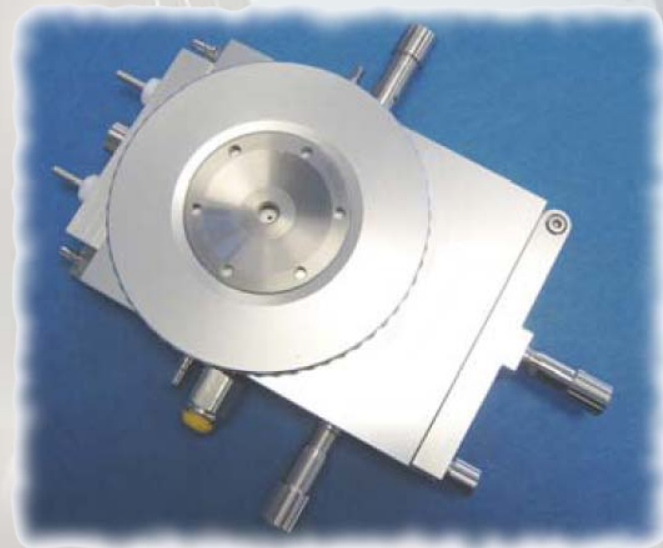


Celda para difracción  
← de neutrones ←

Control de temperatura  
→ para celdas DAC →

Modificación de celdas  
DAC para uso a bajas  
← temperaturas ←

Herramienta para  
→ apretar tornillos →



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# Haznos una propuesta

Simplemente rellena nuestra solicitud de pedido disponible en...

<http://www.malta-consolider.com>

o envía un email a...

[tecnica@cryovac.es](mailto:tecnica@cryovac.es)



## ¿Preguntas?

## ¡Gracias por escuchar!

SOLICITUD DE PEDIDO para



P.I. el Palomo  
C. Arquimedes nº40  
28946 Fuenlabrada  
Tel: 91 606 54 63  
Fax: 91 697 70 97  
e-mail: [info@cryovac.es](mailto:info@cryovac.es)

|                      |               |                  |
|----------------------|---------------|------------------|
| Cliente:             | Departamento: |                  |
| Dirección:           |               |                  |
| Población:           |               | Provincia:       |
| Persona de contacto: | Tlf:          | Movil:           |
| E-mail:              | Fax:          | Fecha Solicitud: |

|                                                                                                                                           |              |
|-------------------------------------------------------------------------------------------------------------------------------------------|--------------|
| Tema:                                                                                                                                     | Nº proyecto: |
| Banda tarifaria:                                                                                                                          |              |
| Relación con MALTA: <input type="radio"/> Proyecto MALTA <input type="radio"/> Investigador MALTA <input type="radio"/> Contacto de MALTA |              |
| Referencia / pedido / orden de trabajo / procedimiento negociado / expediente:                                                            |              |

Croquis / planos / fotos / escáner / (rellenar manualmente, adjuntar páginas adicionales si hacen falta):



|                     |      |                           |        |
|---------------------|------|---------------------------|--------|
| Material / calidad: | Uds: | Plazo entrega solicitado: | Página |
| Observaciones:      |      |                           | 1/     |

DOC-G-020



Vacio, Criogenia, mecanizado y soldadura de precisión  
Equipos a Presión n°: FAP-147 - EIP-2-75 - ERP-2-3

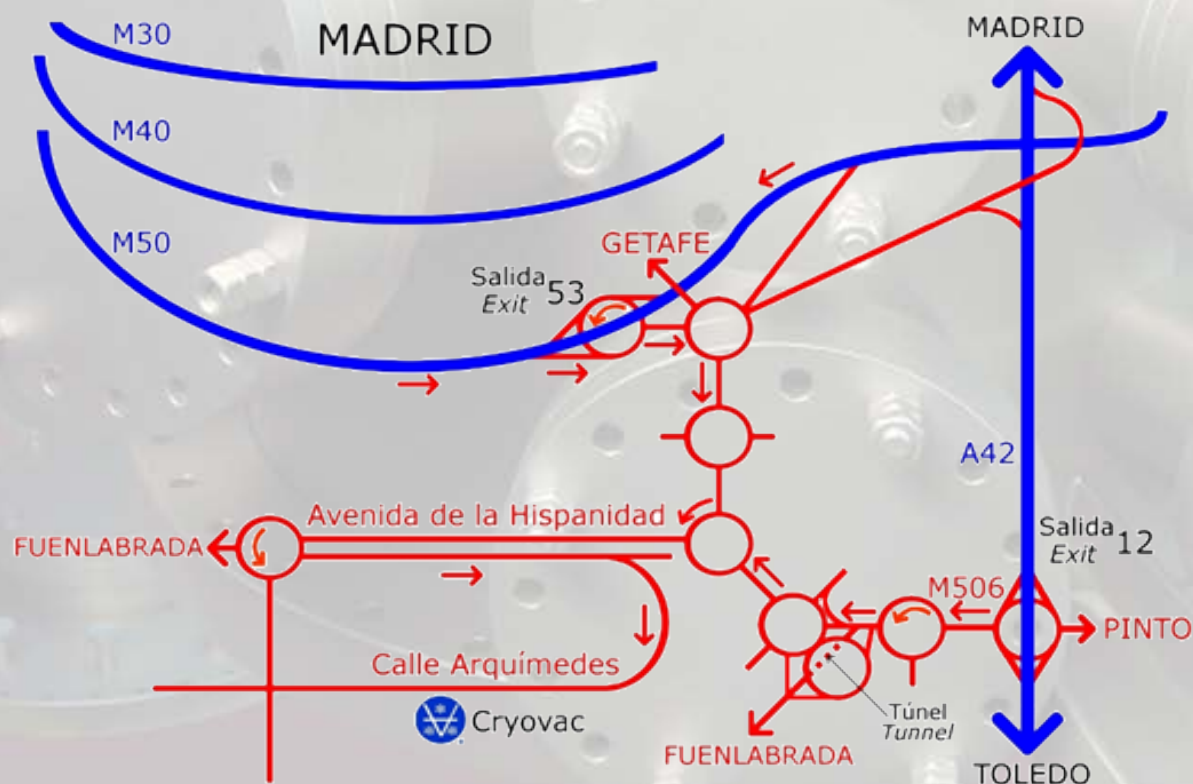
# Detalles de contacto

Cryovac S.L.  
Calle Arquímedes, 40  
28946 Fuenlabrada  
Madrid (Spain)

Telf. (+34) 91 606 54 63  
Fax. (+34) 91 697 70 97

e-mail: [info@cryovac.es](mailto:info@cryovac.es)  
<http://www.cryovac.es/>

Horario de atención: L-V 8:30-17:30



Vacío, Criogenia, mecanizado y soldadura de precisión  
Equipos a Presión n°: FAP-147 - EIP-2-75 - ERP-2-3



Vacio, Criogenia, mecanizado y soldadura de precisión  
Equipos a Presión nº: FAP-147 - EIP-2-75 - ERP-2-3







# MATTER AT HIGH PRESSURE (MALTA)



www.malta-consolider.com



## Scientific goal (II): Molecular systems. Physical properties and chemical reactivity

### b. Pressure-induced reactivity on N, O, C, H materials

Theoretical groups have been involved in the development of computational tools for the analysis of chemical bonding under pressure, derivation of thermal EOS, and systematization of phase transition mechanisms. Their achievements demonstrate the essential character of the variable pressure in chemical reactivity and phase transformations, where the access to specific geometrical configurations (modifying bond distances and angles) plays a fundamental role. This is exemplified in the pressure induced reactivity of unsaturated systems (DMA and CO<sub>2</sub>). A relevant objective of MALTA is to deep in the understanding of the factors affecting high pressure reactivity rather than to provide new reactive processes.



## Methodological Advances

### Chemical Bonding

#### Topology of the Electron Localization Function [1]

Partition of real space into chemically meaningful regions (see figure) allows to identify which are the sensitive electronic entities when pressure is applied. Core of C (green) and O (red), C=O double bond (yellow ring) and C lone pairs (yellow lobes) of molecular CO<sub>2</sub>. Charges (red) and volume (green).

#### Descriptor of non-covalent interactions [2]

The reduced density gradient allows to locate reactive zones liable to experience weak interactions. Formally, it is the derivative of the electron density with respect to the nuclear coordinates. It is used to describe the interactions in molecular crystals and in NCOO biological systems.

### Thermal Effects in EOS and Phase Transition Paths

#### Keeping simplicity beyond the Debye model [3]

The reduced density gradient allows to locate reactive zones liable to experience weak interactions. Formally, it is the derivative of the electron density with respect to the nuclear coordinates. It is used to describe the interactions in molecular crystals and in NCOO biological systems.

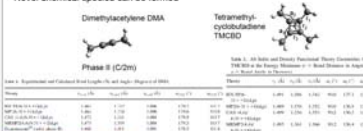
#### Atomic displacements and bond reorganization [4]

The reduced density gradient allows to locate reactive zones liable to experience weak interactions. Formally, it is the derivative of the electron density with respect to the nuclear coordinates. It is used to describe the interactions in molecular crystals and in NCOO biological systems.

## Modeling High Pressure Reactivity in Unsaturated Systems: Application to Dimethylacetylene

### Theoretical Challenge

- Application of moderate pressures leads to a reduction of intermolecular distances
- Novel chemical species can be formed



**Computational strategy**

- Monodeterministic calculations, DFT/B3LYP and MP2 methods were performed with GAUSSIAN.
- GAMESS suite was employed in MCSCF calculations
- Complete active space (CAS) and multireference MP2 (MRMP2) calculations, including four  $\sigma$  electrons placed in two  $\sigma$  and two  $\sigma^*$  orbitals, were used to study the effects of C=C double bond.

Free energy effects were simulated by approaching only two molecules that run parallel along the crystallographic axes of the crystal. Distance R (see figure) controls the degree of freedom in the simulation.

The diagram illustrates the simulation setup for the free energy effects. It shows three molecular structures labeled (a), (b), and (c). Molecules (a) and (b) are positioned parallel to each other, with a vertical double-headed arrow between them labeled 'R'. Molecule (c) is positioned to the right of (a) and (b), with vertical double-headed arrows between it and each of the other two molecules, also labeled 'R'. The molecules are represented as skeletal structures with carbon (grey), oxygen (red), and hydrogen (white) atoms.

**Computational strategy**

- Monodeterminantal calculations, DFT/B3LYP and MP2 methods were performed with GAUSSIAN03
- GAMESS suite was employed in MCSCF calculations
- Complete active space (CAS) and multireference MP2 (MRMP2) methods, including four  $\pi$  electrons placed in two  $\pi$  and two  $\pi^*$  orbitals, were used
- Pressure effects on DMA were simulated by approaching only two molecules that run parallel along the crystallographic c-axes of the crystal. Distance R (see figure) controls the degrees of freedom in the simulation

### Results

- A typical double minima reaction profile is observed
- C-C single bond character appears when R < 2.1 Å
- Transformation predicted follows a path resulting from combination of modes a and c
- When R is large, the most stable situation is described by mode a
- When R decreases, the molecule tend to acquire a reactive geometry similar to that resulting from mode c

### Conclusions

- The transformation path would involve a mixture of the three approaching paths considered in monodeterminantal calculations, the path c having special significance at the onset of the reaction
- It seems feasible that DMA reacts concertedly, due to restrictions in orientation in the solid state to form TMCB and preclude polymerization

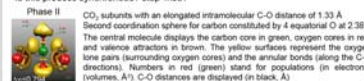
[1] J. Contreras-García, A. Martín-Pendás, B. Silvi and J. M. Recio, J. Chem. Phys. B **113**, 1068 (2009)

[2] E. R. Johnson, S. Keinan, P. Mori-González, J. Contreras-García, A.J. Cohen, and W. Yang, J. Am. Chem. Soc. **132**, 6498 (2010)

## Bases for Understanding Polymerization under Pressure: The Practical Case of CO<sub>2</sub>

### Theoretical Challenge

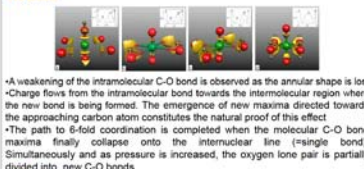
- Simple molecular solids of light elements as CO<sub>2</sub> experiment critical bonding changes toward covalent nets under moderate or high pressure
- Along the progression toward polymeric structures, some intermolecular interactions must strengthen as the intramolecular C=O double bond weakens
- Is this process synchronous? step-wise?



**Computational strategy**

- The ELF topological study has been performed on the all-electron crystalline wave functions computed with the CRYSTAL package
- All-electron calculations have been performed under the DFT framework using C and O basis sets adapted for molecular crystals
- Dirac-Slater exchange and Perdew-Zunger correlation functionals have been used

### Results



### Conclusions

- The electron localization function is able to characterize the pseudopolymeric nature of the bonding intermediates as well as to identify the degree of bond formation
- The rupture of intramolecular bonds and the creation of new intermolecular bonds is carried out in a synchronous manner
- Understanding the transformation of lone pair basins into bonding pairs is of capital interest to predict the coordination of the high-pressure polymorphs

[3] D. Abessi Pérez, Master Thesis, unpublished

[4] M. Marques, M. Florjanczyk, J. Contreras-García, and J. M. Recio, Phys. Rev. B **79**, 104101 (2009)

[5] C. Medavilla, J. Tortajada, V.G. Baonza, J. Comput. Chem. **30**, 415 (2008)

## Acknowledgements

Spanish Ministry of Science & Innovation for project MALTA-Consolider Ingenio 2010 (CSD2007-00045)





RESEAU HAUTES PRESSIONS

# 7<sup>e</sup> FORUM

DE TECHNOLOGIE  
DES HAUTES PRESSIONS

**LA PRESSION POURQUOI ?  
LES NOUVEAUX ENJEUX.**

**Résumés**

[www.rescauhp.org](http://www.rescauhp.org)

**PIERRICK ZERBINO**  
[pierrick.zerbino@synchrotron-soleil.fr](mailto:pierrick.zerbino@synchrotron-soleil.fr)

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**ANDREAS ZERR**  
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LPMTM  
CNRS  
Université Paris Nord  
99, av. J. B. Clément  
93430 Villetaneuse

Un grand merci également aux 6 « participants » de MALTA :

Bérengère Guignon: Instituto del Frío, Consejo Superior de Investigaciones Científicas, Madrid

Idoia Codina Torrella: Planta Tecnologia dels Aliments, Universidad Autónoma de Barcelona

Alfonso Muñoz González: Departamento de Física Fundamental y Experimental, Universidad de La Laguna

Jesús Antonio González Gómez -DCITIMAC, Universidad de Cantabria

José Tortajada Pérez- Departamento de Química Física, Universidad Complutense de Madrid

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### *b. High pressure effects on aqueous solutions of supramolecular aggregates and proteins*

Data on the physical properties of food and food components are still scarce at high pressure. These data are needed to develop and improve food high pressure processes.

Pressure dependence of density, isothermal compressibility, thermal expansivity, specific heat and thermal conductivity are required for numerical simulation of heat and mass transfers and of the coupled microorganism inactivation during high pressure treatment. In this way, treatment uniformity can be determined.

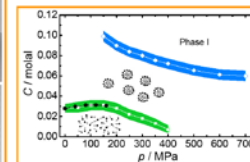
Pressure effects on protein conformation and on aggregation phenomena can be assessed by volumetric and ultrasounds techniques *in situ*. The apparent properties of food components in aqueous solution provide information about their behaviour with pressure in foods. Speed of sound also allows for the determination of critical micellar concentration under pressure.



### High Pressure Measuring Probes

Specific volume and isothermal compressibility were determined for a wide variety of liquid foods and pressure transmitting fluids: pure water, tomato paste, orange juice, whole and skim milk, sodium caseinate aqueous solutions, vegetable oils, silicone oil, castor oil, propylene glycol, ethylene glycol and ethanol.

Ultrasonic measurements were performed in tomato paste and sodium laurate aqueous solutions.



Critical micellar concentration vs. pressure of sodium laurate solutions as determined from speed of sound measurements at 298.15 K.

## References

Otero et al. 2010. Modelling thermophysical properties of food under high pressure. *Crit. Rev. Food Sci.* 50(4): 344-368.

Guignon et al. 2010. Volumetric properties of pressure-transmitting fluids up to 350 MPa: water, ethanol, ethylene glycol, propylene glycol, castor oil, silicone oil and some of their binary mixture. *J. Chem. Eng. Data*, 55, 3017-3023.

Guignen et al.: 2010. Specific volume of liquid water from (253.15 to 323.15) K and pressures up to 350 MPa by volumetric measurements. *J. Chem. Eng. Data* **55**, 3338–3345.

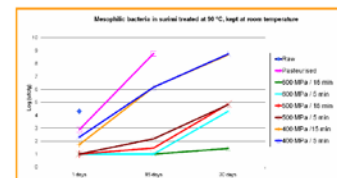
Aparicio et al. 2011. Thermal expansion coefficient and heat capacity from sound velocity measurements in tomato paste from 0.1 MPa up to 350 MPa as a function of temperature. *J. Food Eng.*, submitted.

### c. Microbiology under extreme conditions of pressure and temperature

### Pressure assisted thermal sterilisation (PATS)

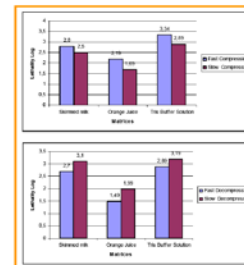
The synergy between pressure and temperature and the effect of adiabatic heating can be used to process solid food and even reach the sterility. An inlet temperature of 90 °C can go up to nearly 120 °C when a pressure of 600 MPa is applied. The benefit of this adiabatic heating is that it is homogeneous and instantaneous.

Important reduction on microbial load of crab sticks can be achieved by applying pressures above 400 MPa, but the combination of 600 MPa and initial temperature of 90 °C produced a sterile product able to withstand at least one month at room temperature.



### Effect of pressure increase and pressure release rates

The effect of non isobaric part of pressure treatment has been studied on *E. coli* inoculated into several matrices. The effectiveness of treatment was matrix dependant, but in all cases, the slowest compression rates caused the lowest lethality, while the fastest decompression was the least effective.



## Acknowledgements

Spanish Ministry of Science & Innovation for project MALTA-Consolider Ingenio 2010 (CSD2007-00045).



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# MATTER AT HIGH PRESSURE (MALTA)

Scientific goal (II): Molecular systems. Physical properties and chemical reactivity

a. Pressure as a probe on unsaturated systems of N, O, C, H

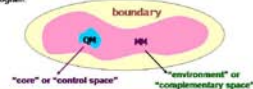
- Modelling Chemical Reactivity at High Pressure
- Raman Characterization of Carbon-based Materials under Pressure



## Modelling Chemical Reactivity at High Pressure

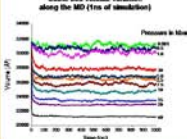
### METHODOLOGY QM/MM + MD

A mixed QM/MM methodology and molecular dynamics (MD) simulations including an external hydrostatic pressure is presented that produces a more experimentally realistic environment featuring hundreds of water molecules. Our objective here is to obtain an appropriate method to incorporate an external hydrostatic pressure in the hybrid QM/MM and MD simulations of the chemical reactivity in complex chemical systems. To this end, a simulation of pure water has been used to probe the quality of MM part. One water molecule in the middle on the cubic box of water molecules has been treated by quantum mechanics, while the rest of the molecules are treated classically by means of the TIP3P potential. All the simulations have been calculated using DYNAMO program.

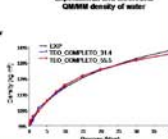


### RESULTS

Cubic box volume variation along the MD (1ns of simulation)



Experimental and theoretical GCMC density of water



## Raman Characterization of Carbon-based Materials under Pressure

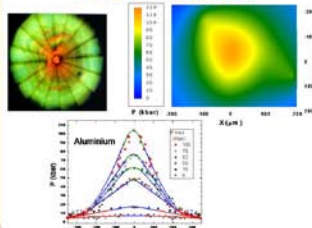
### PRESSURE STATIC VS. UNIAXIAL



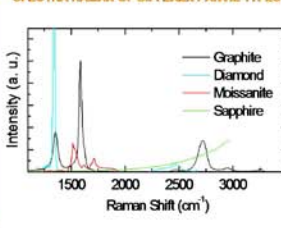
### CELL DETAILS



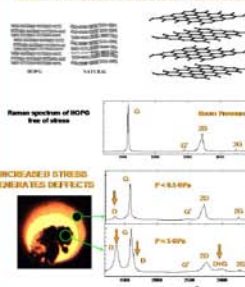
### PRESSURE UNIAXIAL PROFILE



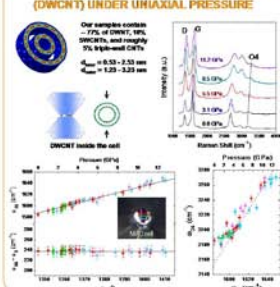
### SPECTRA RAMAN OF DIFFERENT ANVIL TYPES



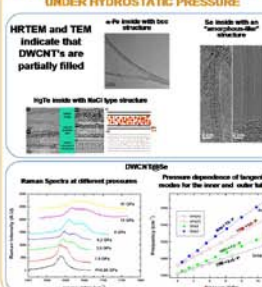
### GRAPHITE UNDER UNIAXIAL PRESSURE



### DOUBLE WALL CARBON NANOTUBES (DWCNT) UNDER UNIAXIAL PRESSURE



### DWCNT FILLED WITH AMORPHOUS ELEMENTS UNDER HYDROSTATIC PRESSURE



## Acknowledgements

Spanish Ministry of Science & Innovation for project MALTA-Consolider Ingenio 2010 (CSD2007-00045)



# MATTER AT HIGH PRESSURE (MALTA)



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- Infrastructures and Facilities acquired
- Technological goals
- Scientific goal (I): Water and life-related systems
  - a. Synthesis, properties and stability of gas clathrates



## Infrastructures and Facilities for All Scientific Goals acquired for MALTA



CLUSTER/NET COMPUTATIONAL FACILITIES



WORKSHOP: HIGH PRESSION MACHINE



RAMAN RESEARCH SYSTEM T64000



X-RAY DIFFRACTION EQUIPMENT, Xcalibur A System



FTIR SPECTROSCOPIC SYSTEM, Bruker Model IFS-120HR

## Technological Goals

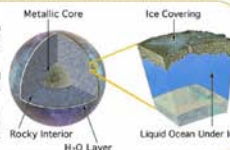
- Development of new high pressure cells: anvil and large volume devices.
- Development of new optically active materials as pressure and temperature sensors.
- Design of a novel laser heating system for diamond anvil cells.
- Implementation of diagnostic devices in high pressure industrial equipments.

## Scientific goal (I): a. Synthesis, spectroscopic properties, PTX diagrams of Gas Clathrates

Clathrate crystallization from saline cryomagmas in planetary conditions

### INTRODUCTION

- EUROPA SATELLITE: Assuming a salty ocean (hydrated salts detected and evidence of magnetic field induced).
- It is interesting the formation and stability of gas clathrates under Europa aqueous ocean conditions.
- Dissociation of clathrates can be an explanation of the presence of the detected gases in the surface and some geological features.



### EXPERIMENTAL APPARATUS

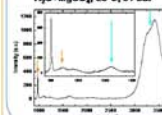
- Stainless steel high pressure chamber coupled to a Raman spectrometer to make the analysis "in situ".
- Clathrate formation has been performed from aqueous solution of  $MgSO_4$  (5%, 10%, 17% in weight) at six different pressures: 18, 20, 25, 30, 50 and 65 bar.
- Changes in the pressure imply the formation (decrease) or dissociation (increase) of the clathrate.



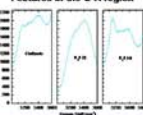
### RESULTS

#### COMPARISON

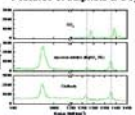
$H_2O+MgSO_4$ ; 25°C, 31 bar



#### Features in the O-H region

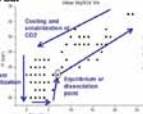
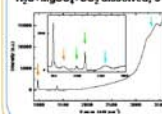


#### Features of sulphate & CO2

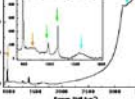


#### EXPERIMENTS

$H_2O+MgSO_4+CO_2$  dissolved; 5°C, 30 bar

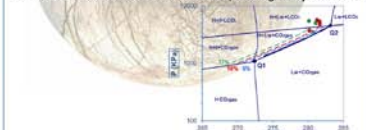


Clathrate  $CO_2$ ; 5°C, 28 bar



### CONCLUSIONS

1<sup>st</sup>  $CO_2$  Clathrates in presence of aqueous solution of  $MgSO_4$  at different concentrations have been formed and dissociated, obtaining the equilibrium lines.



2<sup>nd</sup> Our results suggest that this crystallization of clathrates may explain the geology of Europa satellite, proposing the following model to the magmatic differentiation process:



3<sup>rd</sup> Similar experiments, with other salts detected by Galileo's Near Infrared Mapping Spectrometer, will be carried out since characterization of the clathrate properties will help to detect them in planetary conditions, and understand their role in the activity of the icy moons.

## Acknowledgements



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# MATTER AT HIGH PRESSURE (MALTA)

Scientific goal (III): Structure, stability and reactivity of minerals

**METASTABILITY OF WURTZITE AND ROCK-SALT PHASES IN  $Zn_{1-x}Co_xO$  NANOPARTICLES INVESTIGATED BY OPTICAL ABSORPTION AND RAMAN SPECTROSCOPY UNDER PRESSURE**



## INTRODUCTION

> Colloidal nanocrystals of wurtzite  $Zn_{1-x}Co_xO$  have been prepared using hydrolysis and condensation of acetates solution in DMSO [1].



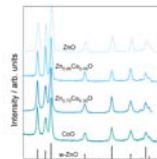
> XRD patterns indicate a good crystallization in the wurtzite structure.

> TEM images show spherical nanoparticles with an average diameter of ca. 4 nm.

> We report an investigation of their optical and lattice-dynamical properties under high pressure by means of optical absorption and Raman spectroscopy.

## SYNTHESIS AND CHARACTERIZATION

### X RAY DIAGRAMS



XRD pattern of the  $Zn_{1-x}Co_xO$  nanocrystalline samples. Wurtzite hexagonal pattern is also shown.

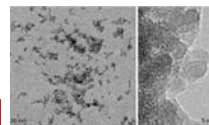
Spherical nanoparticles 4 nm in size

### CRYSTAL STRUCTURES

Hexagonal **Wurtzite (W)** Cubic **Rock-salt (RS)**



### TEM IMAGES



## OPTICAL PROPERTIES

### $Zn_{1-x}Co_xO$ ABSORPTION SPECTRA

As in thin films, absorption spectra of  $Zn_{1-x}Co_xO$  nanoparticles show three main differences with respect to pure W-ZnO [2]:

- ⇒ The energy of the fundamental band-to-band absorption edge is shifted.
- ⇒ A broad band related to charge-transfer transition appears at energies just below (and overlapping) the band-to-band edge.
- ⇒ Well defined absorption bands related to d-d transitions of tetrahedral  $Co^{2+}$  are observed.

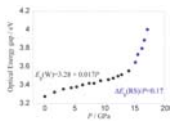
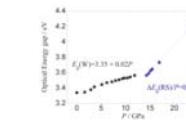
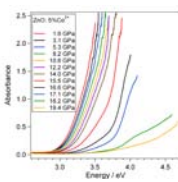
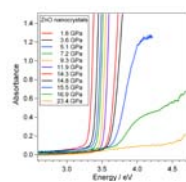
### NON-REVERSIBLE W – RS PHASE TRANSITION UNDER HIGH PRESSURE

The non-reversible transition to the RS phase is observed in the three absorption features [3].

- ⇒ The fundamental edge shifts to much higher photon energies in both ZnO and  $Zn_{0.9}Co_{0.1}O$  and changes from direct to indirect gap at  $P \approx 14$  GPa.
- ⇒ The charge-transfer band virtually disappears (or overlaps the direct transition of the RS phase).

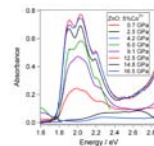
### $Zn_{1-x}Co_xO$ RAMAN SPECTROSCOPY UNDER HIGH PRESSURE

- ⇒ The pressure dependence of the zone-center phonons ( $E_{2g}$ ,  $A_1$ , and  $E_1$ ) was measured for the w-ZnO: 5% $Co^{2+}$  up to the hexagonal-cubic transition near 14 GPa.
- ⇒ Above the transition pressure no active Raman modes were observed.
- ⇒ In the pressure down-stroke, Raman measurements also indicate that the studied nanoparticles remain in the RS phase at ambient pressure.

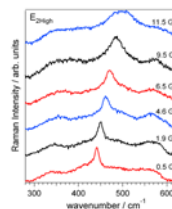


### Metastability

|                 | Bulk    |         | Thin-film |         | Nanoparticles |         |
|-----------------|---------|---------|-----------|---------|---------------|---------|
| P/GPa           | W-to-RS | RS-to-W | W-to-RS   | RS-to-W | W-to-RS       | RS-to-W |
| ZnO             | 9       | 3       | 9         | 3       | 15-16         | -       |
| $Zn_{1-x}Co_xO$ |         |         | 9-11x     | 3-11x   | 15-16         | -       |



⇒ The d-d  $Co^{2+}$  absorption band around 2 eV decreases its intensity by a factor of 10 and shifts to higher energies (2.5 eV) as a consequence of the modification from a tetrahedral to an octahedral coordination symmetry.



### References

- [1] D.A. Schwartz, N.S. Norberg, Q.P. Nguyen, J.M. Parker, D. R. Gamelin, J. Am. Chem. Soc. 125, 13205 (2003).
- [2] J. A. Sans, A. Segura, J. F. Sánchez-Royo, Ch. Ferrer-Roca, and E. Guitiérrez, Phys. Status Solid (b) 244, 407 (2007).
- [3] F. Decrempey, J. Pellicer-Porres, F. Datchi, J. P. He, A. Polian, F. Baudouin, J. Z. Jiang, Appl. Phys. Lett. 81, 4620 (2002).

## Acknowledgements

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# MATTER AT HIGH PRESSURE (MALTA)

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Scientific goal (III): Structure, stability and reactivity of minerals

Pressure-Temperature-Composition (PTx) diagrams and physical properties of:  $ABO_3$  ( $AO-BO_2$ ),  $ABO_4$  ( $AO_2-BO_2$ ) and  $AB_2O_4$  ( $AO-B_2O_3$ ) oxides



## INTRODUCTION

Pressure-induced transition of  $ABO_4$  compounds have been recently reviewed on the light of the most recent experimental and theoretical advantages.

Here we present some recent studies of different compounds where we combine experimental results, from the different MALTA facilities and other experimental facilities, with ab initio methods. Combining the experimental studies with the theoretical first principles simulations we can clarify the complex behaviour of some system under extreme conditions and also it provide some new predictions of novel high pressure phases coming from the ab initio simulations.

### Experimental techniques:

X-ray diffraction experiments: MALTA Xcalibur diffractometer, ESRF, DIAMOND and APS sources.

Raman experiments: University of Cantabria MALTA, and University of Valencia (MALTA)

DAC experiments, with different transmission media (see references).

### Theoretical details.

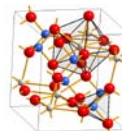
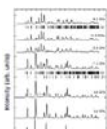
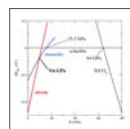
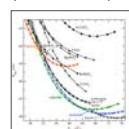
Ab initio total-energy and lattice-dynamics calculations in the framework of the density functional theory, DFT, and the pseudopotential method. MALTA Univ. Oviedo Cluster, MALTA Univ. La Laguna cluster and RES supercomputers.

Projector-augmented wave pseudopotentials, PAW, different exchange correlations functionals, LDA, GGA

Highly converged calculations, structures fully relaxed and forces well converged in order to have the phonon spectra at Gamma point using the direct method to obtain the dynamical matrix. Full phonon dispersion and phonon DOS can be obtained.

### Some recent examples:

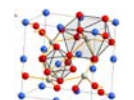
$ABX_4$  compounds have many technological applications. Knowledge of the HP structural behavior of orthophosphates is relevant for mineral physics and chemistry. (The  $APX_4$  compounds depending on the ionic radio crystallize on zircon or monazite structures). In recent years, there have been many studies dealing with their pressure-driven phase transition.



Monazite structure

In ambient conditions,  $TbPO_4$  crystallizes in the tetragonal zircon structure ( $I_4/amd$ ,  $Z=4$ )

In this structure, the Tb and P cations are coordinated by 8 and 4 O anions, respectively.



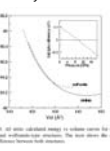
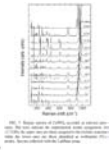
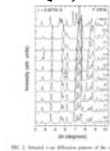
The  $SrUO_4$ -type structure

| Compound | Structure | Tb coord. | Average Tb-O dist. (Å) | P coord. | Average P-O dist. (Å) |
|----------|-----------|-----------|------------------------|----------|-----------------------|
| 0        | zircon    | 8         | 2.37                   | 4        | 1.55                  |
| 10.3     | monazite  | 8+1       | 2.42                   | 4        | 1.54                  |
| 20.5     | scheelite | 8         | 2.31                   | 4        | 1.54                  |
| 77.1     | $SrUO_4$  | 9         | 2.24                   | 6        | 1.65                  |

| P (GPa) | Structure | Tb coord. | Average Tb-O dist. (Å) | P coord. | Average P-O dist. (Å) |
|---------|-----------|-----------|------------------------|----------|-----------------------|
| 0       | zircon    | 8         | 2.37                   | 4        | 1.55                  |
| 10.3    | monazite  | 8+1       | 2.42                   | 4        | 1.54                  |
| 20.5    | scheelite | 8         | 2.31                   | 4        | 1.54                  |
| 77.1    | $SrUO_4$  | 9         | 2.24                   | 6        | 1.65                  |

Good agreement between experiments and theory. Theoretical calculations help to identify the first pressure transition from Zircon to Monazite. X-ray diffractions confirms the monazite structure and Raman results are in good agreement. A second phase transition predicted from theory to a new  $SrUO_4$  phase, where P has six-fold coordination.

$CuWO_4$  crystallizes in a triclinic structure, with both cations octahedrally coordinated by O atoms.



Good agreement, the theoretical results confirm the proposed high pressure phase.

| Compound | Structure | Tb coord. | Average Tb-O dist. (Å) | P coord. | Average P-O dist. (Å) |
|----------|-----------|-----------|------------------------|----------|-----------------------|
| 0        | zircon    | 8         | 2.37                   | 4        | 1.55                  |
| 10.3     | monazite  | 8+1       | 2.42                   | 4        | 1.54                  |
| 20.5     | scheelite | 8         | 2.31                   | 4        | 1.54                  |
| 77.1     | $SrUO_4$  | 9         | 2.24                   | 6        | 1.65                  |

### References:

J. López-Solano et al. Phys. Rev. B **81**, 144126 (2010)  
J. Ruiz-Fuertes et al. Phys. Rev. B **81**, 224115 (2010)

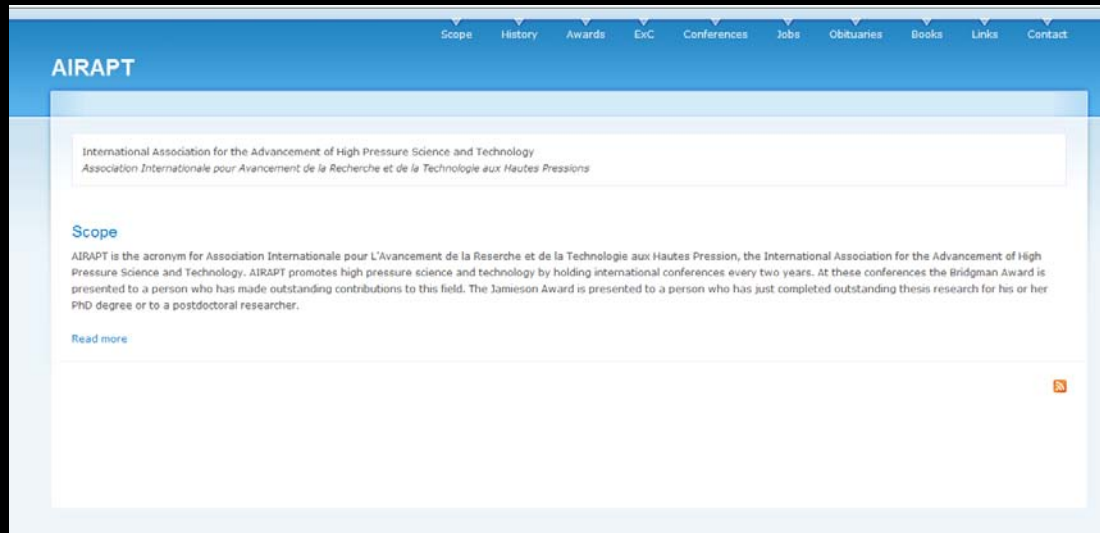
## Acknowledgements



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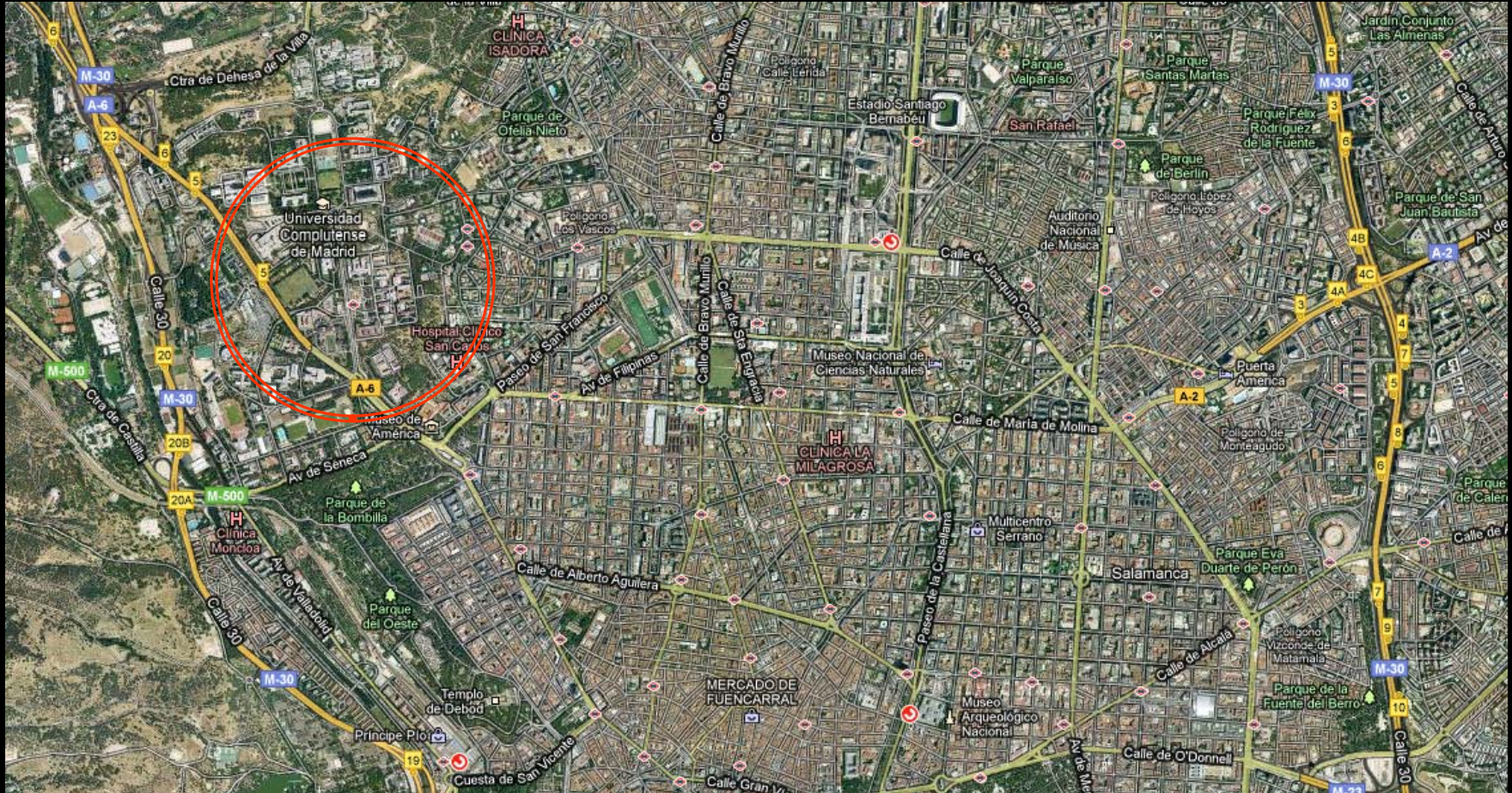








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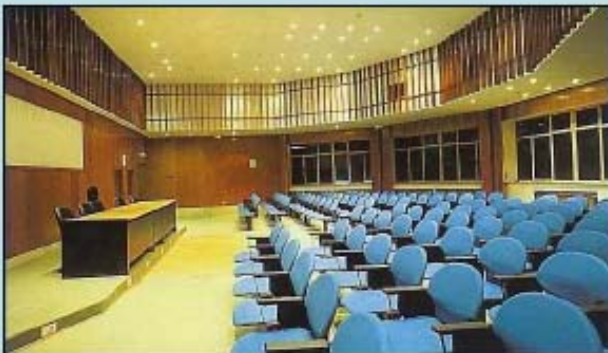


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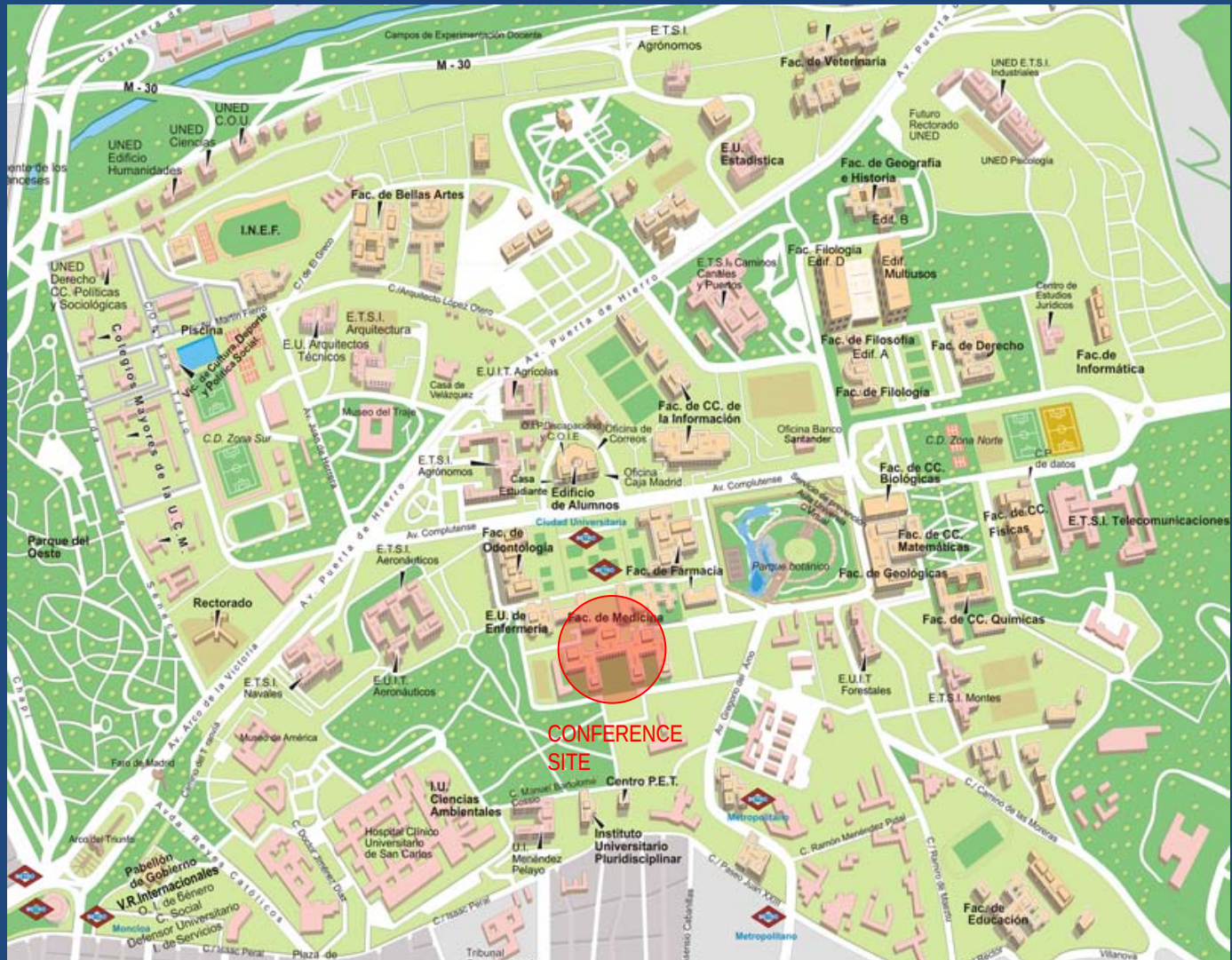
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[www.esmadrid.com/en/portal.do](http://www.esmadrid.com/en/portal.do) (traveling, hotels, culture, ...)

[www.malta-consolider.com](http://www.malta-consolider.com) (updates & general info)

Contact: [vgbaonza@quim.ucm.es](mailto:vgbaonza@quim.ucm.es)

# AIRAPT

[Home](#)

## Conferences

AIRAPT sponsors a conference periodically, usually every two years, at a site chosen by the Executive Committee. Proposals to organize a conference are submitted by people involved with high-pressure research and have attended several AIRAPT conferences. Presentation of a proposal is made at the meeting of the Executive Committee of the AIRAPT conference. In addition to the fact that people submitting a proposal should be active members of the high-pressure community, other important considerations in the process are the expected cost of registration, travel, local hotels and the availability of lodging for students. Based primarily on these presentations, the Executive Committee chooses the future site or sites. Attendance at AIRAPT conferences generally varies from 300 to 500.

- [\[20110124\] Dynamic Phenomena under Extremes, Austin, TX \(USA\), January 24-28, 2011](#)
- [\[20110306\] 26th International Conference on Interaction of Intense Energy Fluxes with Matter, Kabardino, Russia, March 1-6, 2011](#)
- [\[20110502\] 5th CCM international conference on pressure metrology \(Berlin, Germany, May 2-5, 2011\)](#)
- [\[20110520\] 3rd International Conference on High Energy Density Physics 2011. Lisbon, Portugal, May 2011](#)
- [\[20110828\] EHPRG-49, Budapest, Hungary, 28.08-02-09.2011](#)
- [\[20110925\] AIRAPT-23, Mumbai, India, 2011](#)
- [\[20120000\] 50th EHPRG Meeting, Thessaloniki, Greece, 2012](#)
- [\[20130000\] 51st EHPRG Meeting, London, UK, 2013](#)
- [\[20130000\] AIRAPT-24 & APS-SCCM, Seattle, USA, 2013](#)
- [\[20150000\] Joint AIRAPT-25 and EHPRG-53, Madrid, Spain, 2015](#) 
- ▷ [Past Conferences](#)

[\[20110124\] Dynamic Phenomena under Extremes, Au](#)



## Conferences

AIRAPT sponsors a conference periodically, usually every two years, at a site chosen by the Executive Committee. Proposals to or involved with high-pressure research and have attended several AIRAPT conferences. Presentation of a proposal is made at the AIRAPT conference. In addition to the fact that people submitting a proposal should be active members of the high-pressure community, the expected cost of registration, travel, local hotels and the availability of lodging for students. Based primarily on these factors, the future site or sites. Attendance at AIRAPT conferences generally varies from 300 to 500.

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- [\[20150000\] Joint AIRAPT-25 and EHPRG-53, Madrid, Spain, 2015](#) 
- ▷ [Past Conferences](#)

[\[20110124\] Dyna](#)

## Executive Committee

AIRAPT Executive Committee (since 2009)

### Officers

- [R. Boehler](#), USA, *President*
- [P. Loubeyre](#), France, *Vice President*
- [I. Silvera](#), USA, *Vice President*
- [J. S. Schilling](#), USA, *Treasurer*
- [G. G. N. Angilella](#), Italy, *Secretary*

### Members

- [K. Aoki](#), Japan
- [R. Ahuja](#), Sweden
- [V. Brazhkin](#), Russia
- [T. Cui](#), China
- [F. Datchi](#), France
- [I. Grzegory](#), Poland
- [T. Irifune](#), Japan
- [J. Kamarad](#), Czech Republic
- [G. I. Kanel](#), Russia
- [J. Loveday](#), UK
- [F. Rodriguez](#), Spain
- [B. Winkler](#), Germany
- [C.-S. Yoo](#), USA

### Ex-officio members

- [W. Nellis](#), USA, *Past President*
- [S. Klotz](#), France, *Editor of High Pressure Research*
- [D. Klug](#), Canada, *Chairman of Gordon Conference*



# *The European High Pressure Research Group*

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*Meetings*

*Committee*

*Award*

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*Lists*

*Links*

## **High Pressure links**

- The [EHPRG website](#) and the [High Pressure Directory at EHPRG](#) are maintained by [Giuseppe Angilella](#)

Until 2002, the EHPRG website used to be hosted by the [University of Leuven](#), Belgium, and maintained by [Gert Van Tendeloo](#)

## **Scholarly Societies**

- [International Association for the Advancement of High Pressure Science and Technology \(AIRAPT\)](#)
- [European Physical Society](#)
- [COMPRES](#), COnsortium for Materials Properties Research in Earth Sciences
- [International Organization for Crystal Growth](#)
- [International Union of Crystallography \(IUCr\)](#)

## **High Pressure Research Laboratories**

In alphabetical order per country. Please, contact the [webmaster](#) if your laboratory is not in the list.

- [High pressure equipment](#) at [Institut Laue-Langevin](#), Grenoble, France
- [The Bayerisches Geoinstitut](#), Bayreuth, Germany
- [High Pressure Research Center](#), Warsaw, Poland
- [Institute for High Pressure Physics](#), [Russian Academy of Science](#), Troitsk (Moscow Region), Russia
- [Centre for Science at Extreme Conditions](#), Edinburgh, UK
- [Liverpool High Pressure Laboratory](#), Liverpool, UK



# The European High Pressure Research Group

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## The European High Pressure Research Group

### EHPRG Committee Members

Since the Joint AIRAPT and EHPRG International Conference in Uppsala, 2010:

- **G. G. N. Angilella**, University of Catania, Italy, *Chairman*
- **S. Klotz**, Université Pierre et Marie Curie, Paris, France *Secretary*
- **K. V. Kamenev**, The University of Edinburgh, UK, *Treasurer*
- **O. Andersson**, Umeå University, Umeå, Sweden
- **R. Ahuja**, Uppsala University, Uppsala, Sweden
- **E. Boldyreva**, Novosibirsk State University, Russia
- **D. Christofilos**, Aristotle University of Thessaloniki, Greece
- **F. Decremps**, Université Pierre et Marie Curie, Paris, France
- **L. S. Dubrovinsky**, Bayerisches Geoinstitut, Bayreuth, Germany
- **E. M. Dumay**, Université Montpellier 2, Montpellier, France
- **V. García-Baonza**, Universidad Complutense de Madrid, Madrid, Spain
- **M. Hanfland**, ESRF, Grenoble, France
- **P. Lazor**, Uppsala University, Uppsala, Sweden
- **I. Loa**, CSEC, University of Edinburgh, Edinburgh, UK
- **A. Mathys**, Nestlé Research Center, Lausanne, Switzerland
- **A. Muñoz**, Universidad La Laguna, Tenerife, Spain
- **P. Postorino**, Università di Roma La Sapienza, Roma, Italy
- **R. Pucci**, Università di Catania, Catania, Italy
- **A. San Miguel**, Université Lyon 1, Lyon, France
- **Ex-officio members:**
  - **R. Böhler**, Max-Planck Institut für Chemie, Mainz, Germany, *President of the AIRAPT*
  - **E. P. O'Reilly**, Tyndall National Institute (NMRC), Cork, Ireland, *Chairman of the Condensed Matter*

[Giuseppe Angilella](#)

[Belgium, and mainta](#)

[ology \(AIRAPT\)](#)

[in the list.](#)

[v Region\), Russia](#)



## International Conference on High Pressure Science and Technology

### AIRAPT-23



25<sup>th</sup> September (Sunday)  
to  
30<sup>th</sup> September (Friday) 2011

#### Venue:

Bhabha Atomic Research Centre  
Mumbai, India

#### Supported by:

- International Association for the Advancement of High Pressure Science and Technology (AIRAPT)
- Department of Atomic Energy, India
- Defence Research & Development Organization
- Materials Research Society of India

#### Scope of the Conference

This will be an interdisciplinary conference on high pressure science and technology. The program will include invited plenary and topical lectures, parallel sessions, poster sessions, and exhibits of equipment and products.

#### Topics of the conference:

- Theory, modeling and simulations
- Structure, equation of state, phase transitions and kinetics
- Optical, acoustic, electrical and magnetic properties
- Thermophysical properties
- New developments using neutron and synchrotron sources
- Molecular and organic solids
- Liquids and amorphous materials
- Water and ices
- Supercritical fluids
- Geophysical, marine and planetary science
- Chemical Reaction and kinetics
- Synthesis of new materials
- Superhard materials
- Superconducting and novel materials
- Material response under Shock waves
- Mechanical properties
- Bioscience and food technology
- Hot and cold isostatic pressing process
- High pressure instrumentation
- Spectroscopy at static and dynamic pressure
- Hyper velocity impact phenomena
- Melting at static and dynamic high pressures
- High pressure chemistry
- Effect of static and dynamic pressures on bacteria

#### Important Dates

|                    |                   |
|--------------------|-------------------|
| Abstract Due       | 30 April 2011     |
| Early Registration | 30 May 2011       |
| Conference Start   | 25 September 2011 |
| Manuscript Due     | 30 September 2011 |

#### Registration fee

|                                          |                   |
|------------------------------------------|-------------------|
| Delegates from Foreign Countries         | Euro 450          |
| Student delegates from foreign countries | Euro 225          |
| Indian delegates                         | Rs. 4000          |
| Indian Student delegates                 | Rs. 2000          |
| Accompanying person                      | Euro 100/Rs. 2000 |

(All registered delegates will receive conference kits. Delegates and accompanying persons are welcome to visit the exhibitions and cultural programme, and invited to refreshments, lunches and dinners on the conference days.)

#### Exhibitions

Space will be available for exhibitors at the conference site

#### Contact Address

Dr. Satish C. Gupta,  
Chairman, Organizing Committee  
Head, Applied Physics Division,  
Bhabha Atomic Research Centre,  
Trombay, Mumbai 400085, India

Email: [airapt2011@barc.gov.in](mailto:airapt2011@barc.gov.in)  
[airapt23@gmail.com](mailto:airapt23@gmail.com)

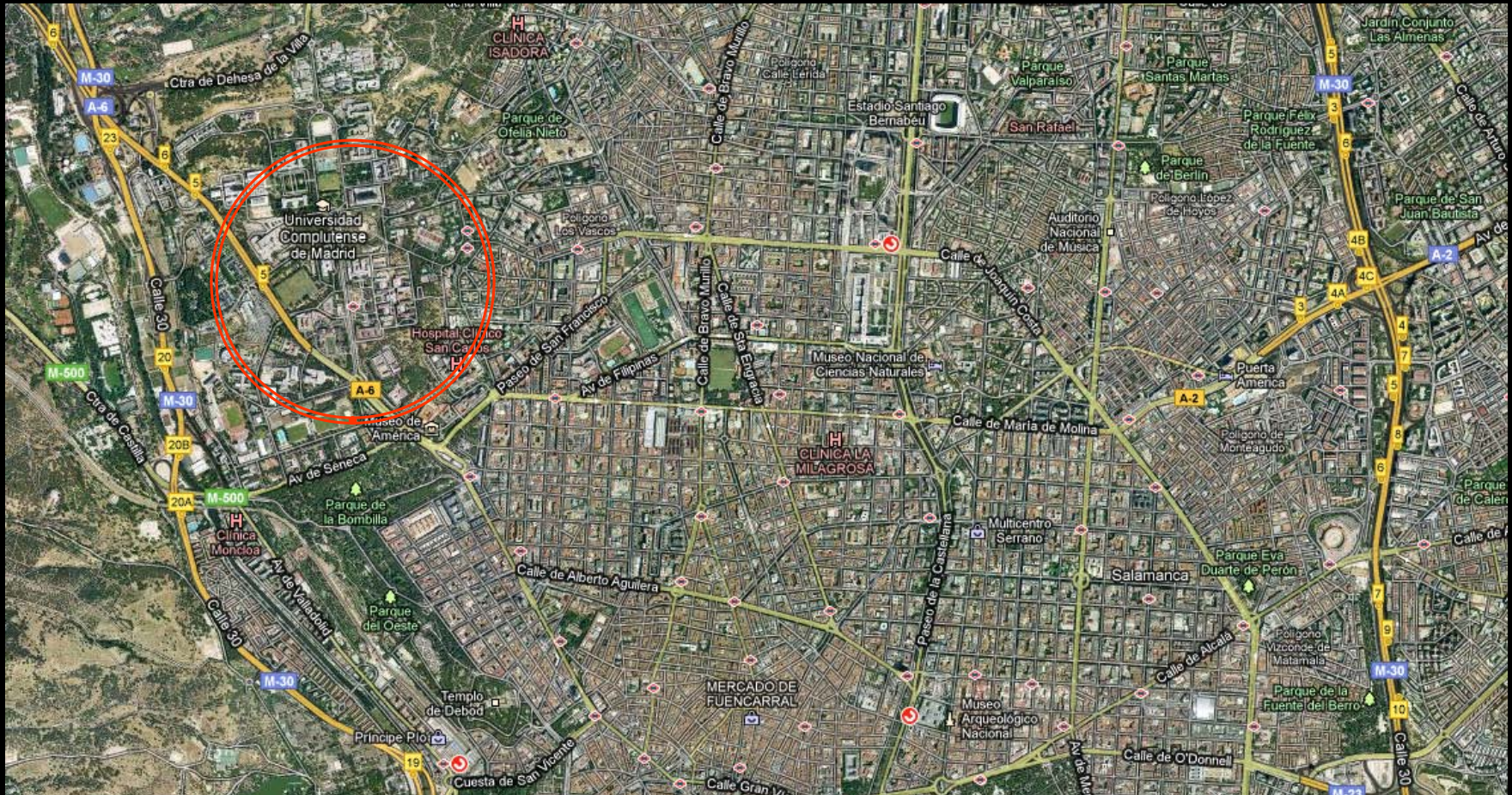
Website:

Phone: +91 22 25595475

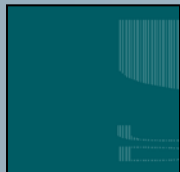
Fax No.: +91 22 25505151 and +91 22 25519613



# MADRID 2015



**Infraestructura Espectrómetro Raman**  
**Informe de Gestión**  
**Jesús González**  
**Fernando Rodríguez**  
**CITIMAC, UC**  
**Reunión Malta Consolider**  
**Miraflores de la Sierra (Madrid) 23-27 de**  
**Enero 2011**



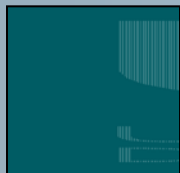
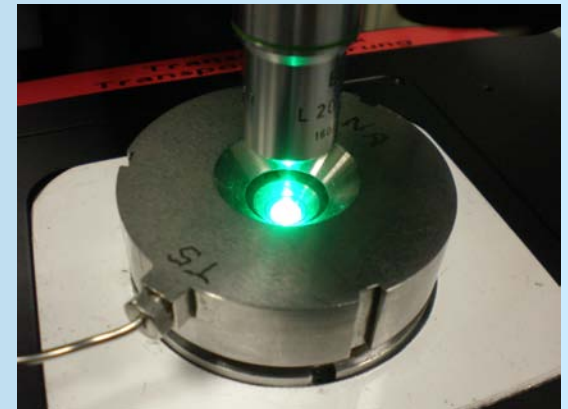
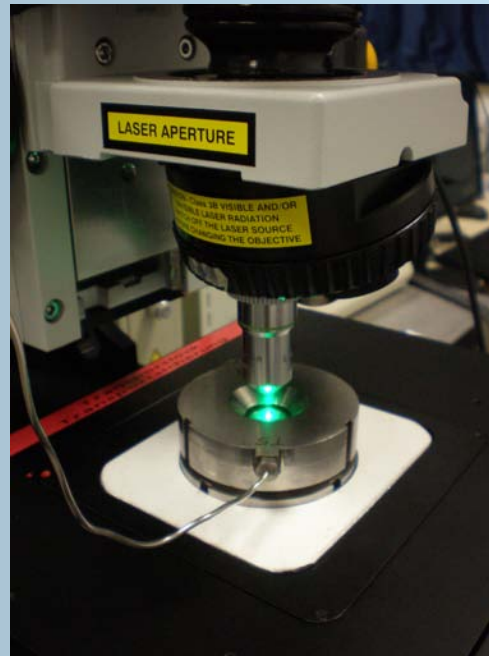
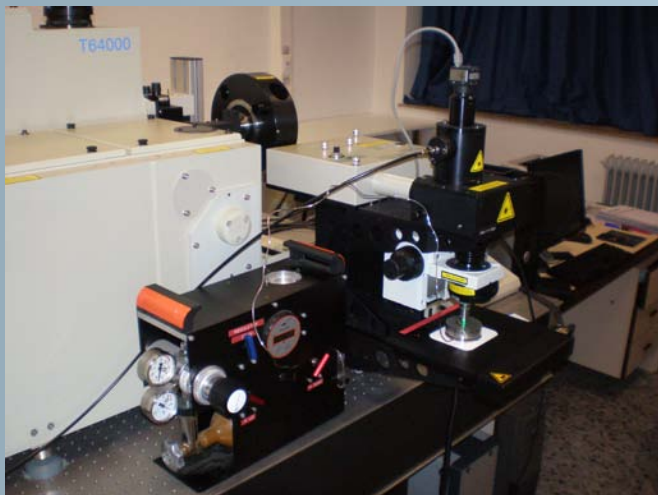
**Grupo de Altas Presiones y**  
**Espectroscopia**  
<http://grupos.unican.es/apye>  
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# Grupo de Altas Presiones y Espectroscopia

## Raman T-64000



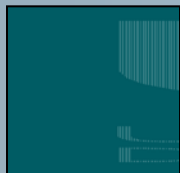
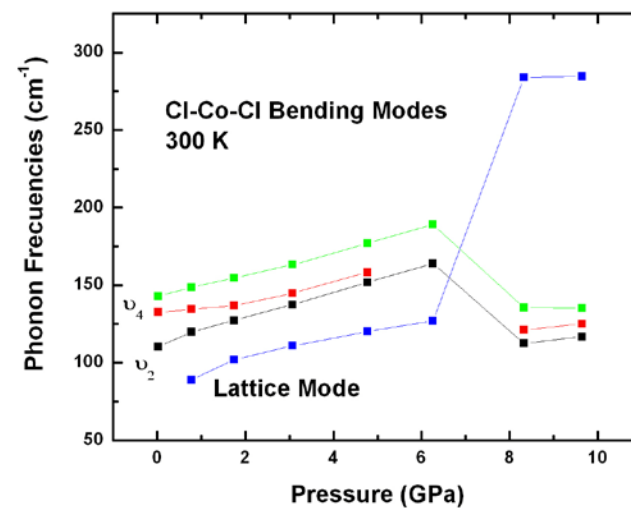
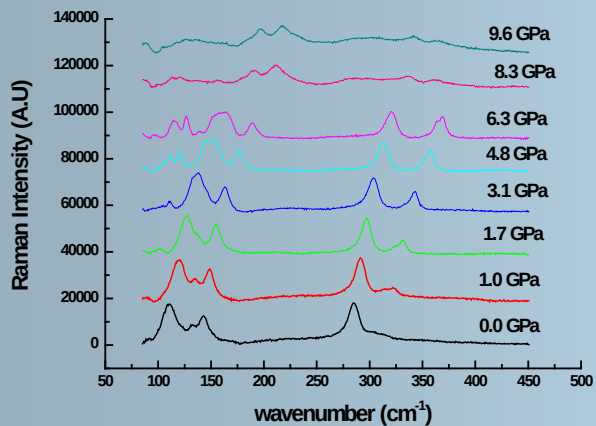
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Espectroscopia**  
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# Grupo de Altas Presiones y Espectroscopia

## Lucie Nataf y Fernando Rodríguez

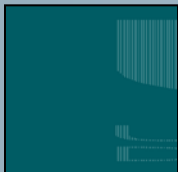
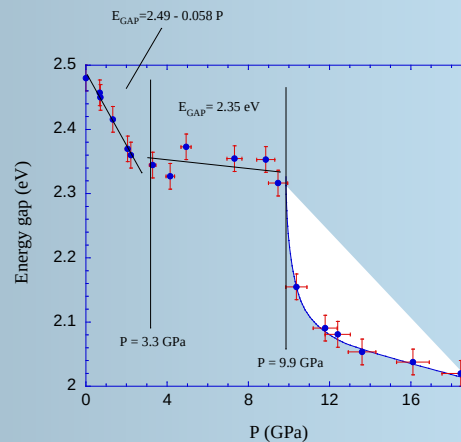
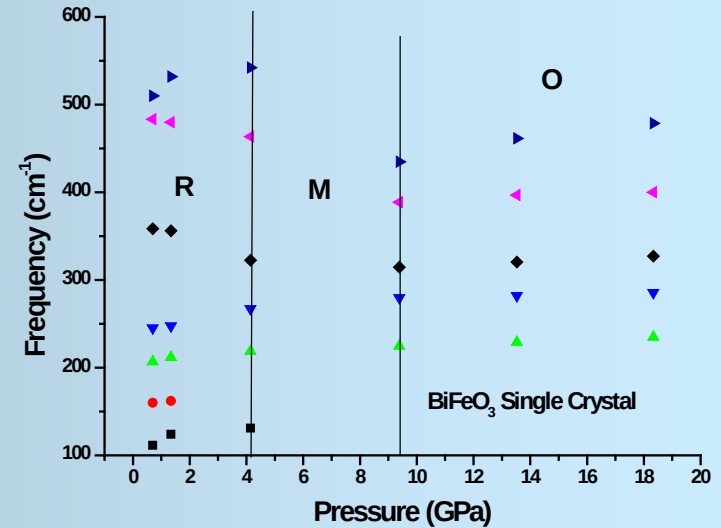
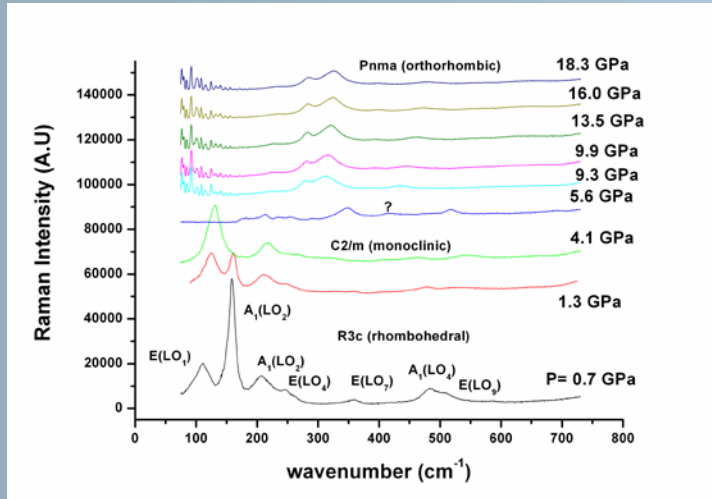
### Cs<sub>2</sub>CoCl<sub>4</sub>



# Grupo de Altas Presiones y Espectroscopia

## Susana Gómez y Fernando Rodríguez

### Multiferroico BiFeO<sub>3</sub>



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<http://grupos.unican.es/apye>  
**FACULTAD DE CIENCIAS**

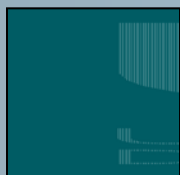
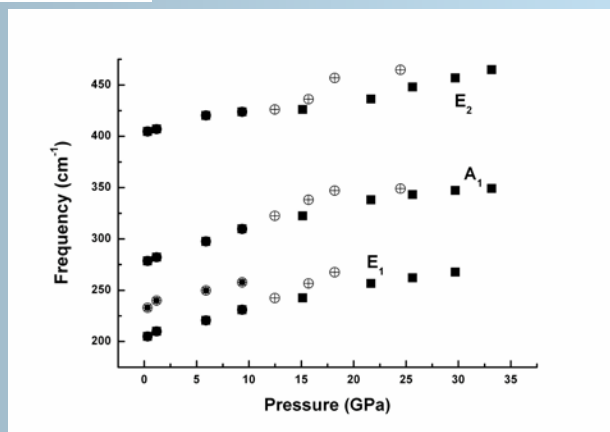
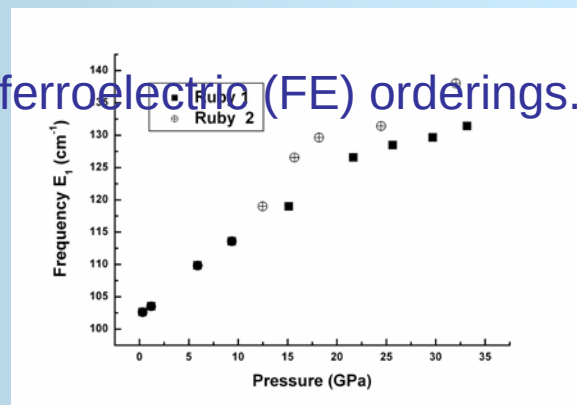
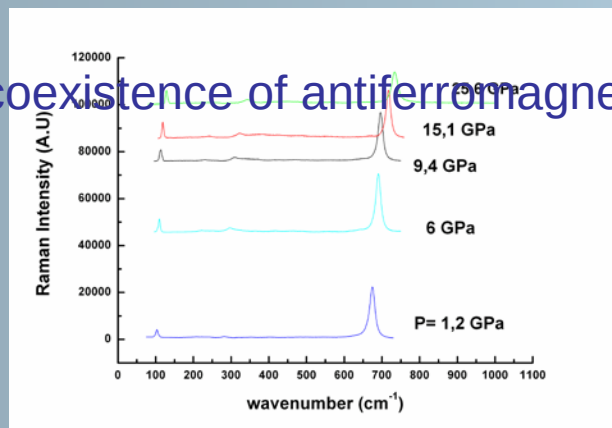




Grupo de Altas Presiones y Espectroscopia  
Fernando Aguado y C. Wang (Academia de Ciencias China, Beijing)

HoMnO<sub>3</sub> (hexagonal perovskite with space group  $P6_3cm$   $Z=6$ )  
multiferroic material characterized by the

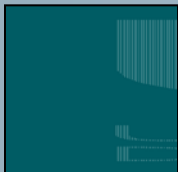
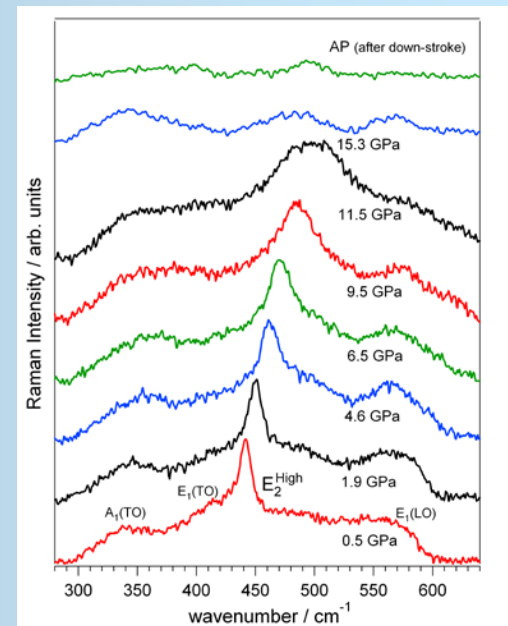
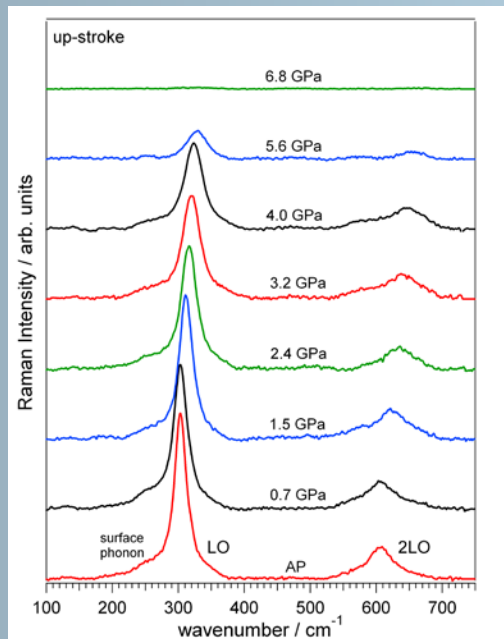
coexistence of antiferromagnetic (AFM) and ferroelectric (FE) orderings.



# Grupo de Altas Presiones y Espectroscopia

Rosa Martin y Rafael Valiente

## Nanopartículas de CdS y ZnO: Co 5%



UNIVERSIDAD DE CANTABRIA



MÁSTER EN CIENCIA DE MATERIALES

“Síntesis y caracterización de materiales compuestos de matriz polimérica con nanotubos de carbono multipared (MWCNT's) reforzada con fibra de vidrio”

FUNDACIÓN CENTRO TECNOLÓGICO DE COMPONENTES (CTC)



Autor: Carmen Manteca Martínez  
Director: Jesús Antonio González Gómez  
Codirector: Ángel Yedra Martínez

Santander, Octubre 2010

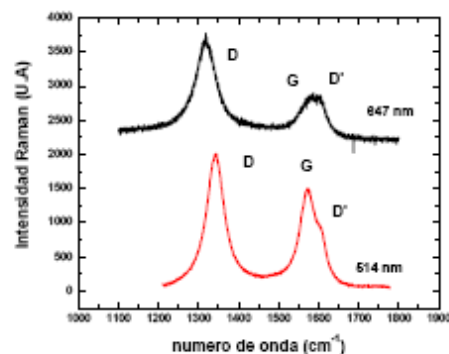
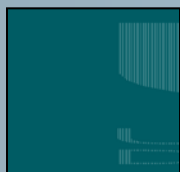


Figura 32. Espectro Raman de una muestra de nanotubos multipared de Nanocyl.

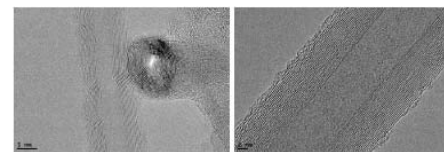


Figura 35. Imagen TEM de una muestra de MWCNT de Nanocyl y de un nanotubo de carbono de una muestra de MWCNT de Nanocyl.

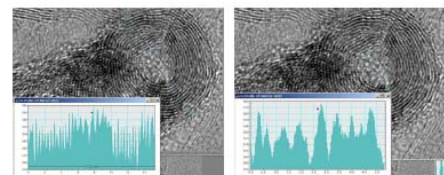


Figura 37. Medios del diámetro externo e interno de un nanotubo de carbono de una muestra de MWCNT de Nanocyl.

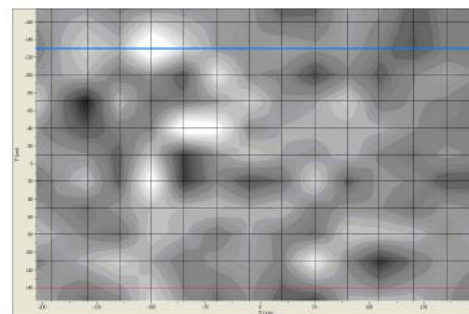


Figura 33. Mapa de distribución de la muestra PIUD\_0\_1\_3C.

Grupo de Altas Presiones y  
Espectroscopia

<http://grupos.unican.es/apye>

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# Caracterización de tensiones residuales y análisis microestructural en fundición mediante espectroscopia Raman

por  
Borja Estandía Coterón  
Master en Ciencia de Materiales

SANTANDER  
Octubre de 2010

Tutores:

D. Jesús A. González Gómez  
D. Jose A. Alvarez Laso

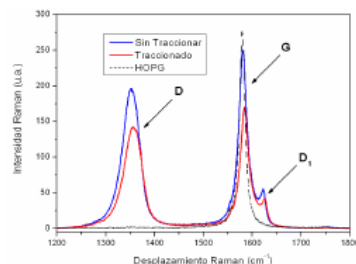
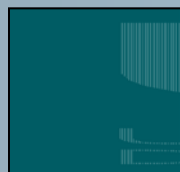


Figura 3.6: Espectros Raman en primer orden de dispersión de las muestras de grafito laminar, traccionado y sin traccionar, y grafito HOPG.

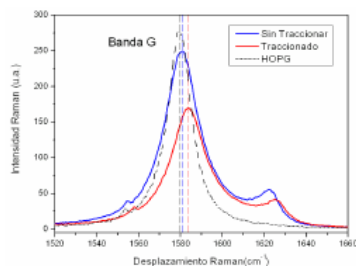


Figura 3.7: Espectros Raman de la banda G de las muestras de grafito laminar, traccionado y sin traccionar, y grafito HOPG.

aquellas que presentan un desplazamiento a frecuencias mayores, como es el caso, y *Tensiones de Tracción* las que propician un desplazamiento a frecuencias menores. Estos desplazamientos se cuantifican como  $5 \text{ cm}^{-1}$  por Gigapascal (GPa) de presión [3][4][5].

$$\frac{d\omega}{dP} = 5 \text{ cm}^{-1} \text{ GPa}^{-1} \Leftrightarrow 1 \text{ GPa}^{-1} = 10 \text{ kbar}$$

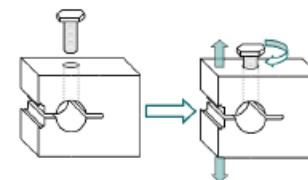
En la Tabla 3.1 se recogen los valores obtenidos de la modelización de las bandas correspondientes con la dispersión Raman de primer orden de los espectro representados en las figuras 3.4 y 3.5.

Tabla 3.1: Principales valores de las diferentes estructuras Raman en Grafito Laminar calculados con Origin 7.0.

| Muestra        | Asignación de banda | Posición $\tilde{\nu}$ ( $\text{cm}^{-1}$ ) | FWHM $\omega$ ( $\text{cm}^{-1}$ ) | Intensidad $I$ (u.a.) | $I_D/I_G$ |
|----------------|---------------------|---------------------------------------------|------------------------------------|-----------------------|-----------|
| Sin Traccionar | D                   | 1351,78                                     | 36,12                              | 196,72                | 0,79      |
|                | G                   | 1580,67                                     | 20,43                              | 250,09                |           |
| Traccionado    | D                   | 1355,83                                     | 38,10                              | 142,63                | 0,84      |
|                | G                   | 1583,96                                     | 21,50                              | 169,85                |           |

## Próximos estudios:

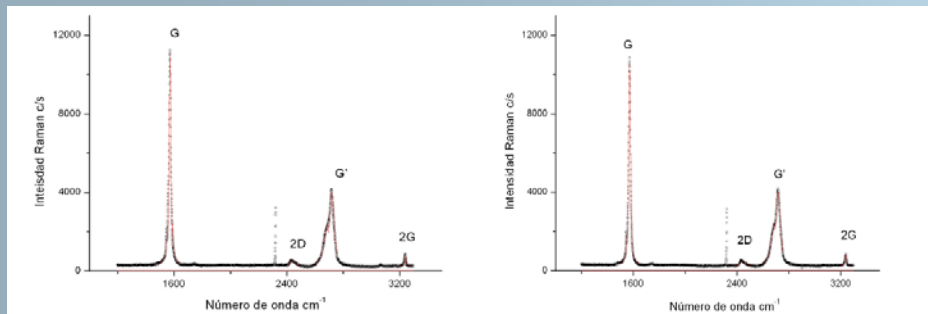
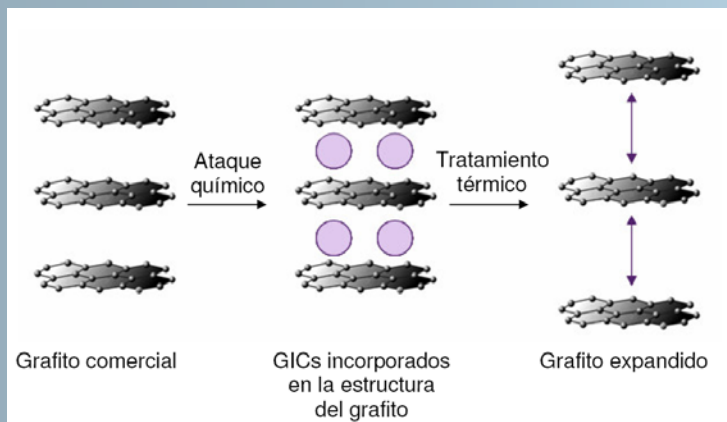
Una vez **confirmado el uso de grafito como sensor de presión** se pretende validar cuantitativamente los resultados obtenidos en este estudio mediante una modelización de elementos finitos sobre **probetas de diseño propio** evaluándolas simultáneamente mediante espectroscopia Raman.



# Colaboración CTC

## Tesis Master en Ciencia de Materiales

### Pablo Mantilla



UNIVERSIDAD DE CANTABRIA

Facultad de Ciencias

DEPARTAMENTO DE CIENCIAS DE LA TIERRA  
Y FÍSICA DE LA MATERIA CONDENSADA



MASTER EN CIENCIA DE MATERIALES

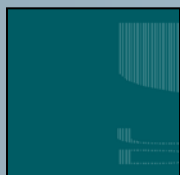
"Desarrollo de materiales compuestos PCM/matriz base carbono"

Autor: Pablo Mantilla Gilart  
Director: Ángel Yedra Martínez  
Empresa: Fundación Centro Tecnológico de Componentes (CTC)



SANTANDER, 2009 - 2010

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# Grupo de Altas Presiones y Espectroscopia

## Antonio Barreda y Fernando Rodríguez

### Matriz Orgánica+MnBr<sub>4</sub>

UNIVERSIDAD DE CANTABRIA  
III MASTER EN CIENCIAS DE MATERIALES



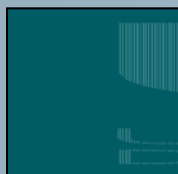
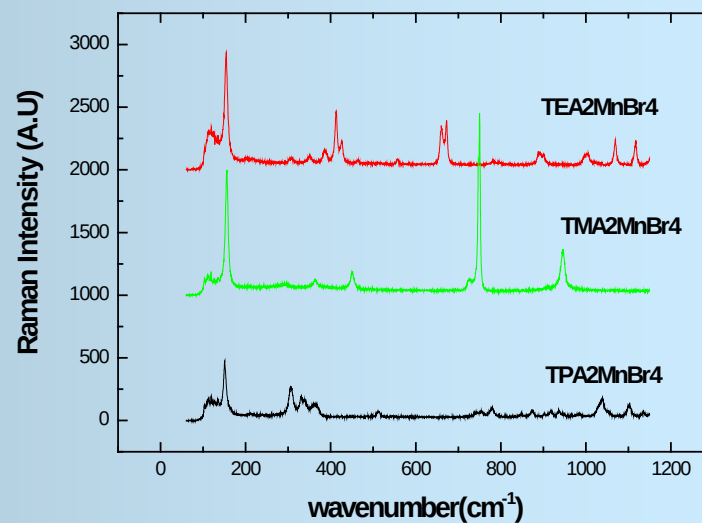
**Desarrollo de técnicas espectroscópicas para estudio de micromuestras sometidas a condiciones extremas**

**Aplicación al estudio de la eficiencia luminiscente en sistema**  
 $[(C_nH_{2n+1})_4N]_2MnX_4$  (X:Cl, Br; n=1-3)

Proyecto Final de Master en Ciencias de Materiales  
Santander, octubre de 2010

Autor: - **José Antonio Barreda**  
Director: - **Fernando Rodríguez**

Dep. Ciencias de la Tierra y Física de la Materia Condensada  
Facultad de Ciencias



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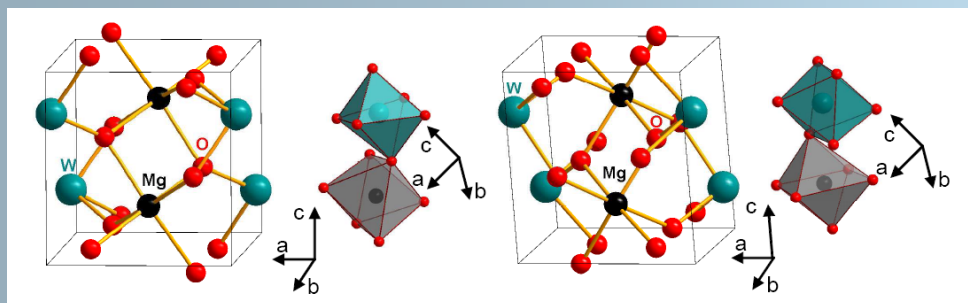


Figure 1 (color online). Schematic view of the wolframite structure with a detail of both octahedra (left) and of the P1 triclinic structure (right).

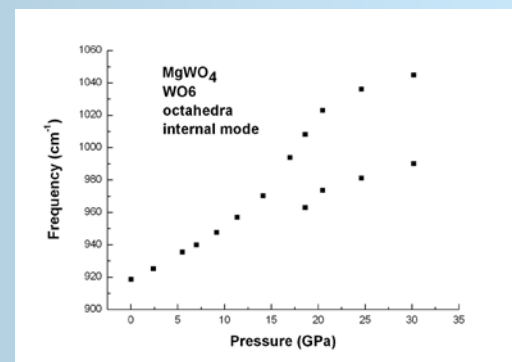
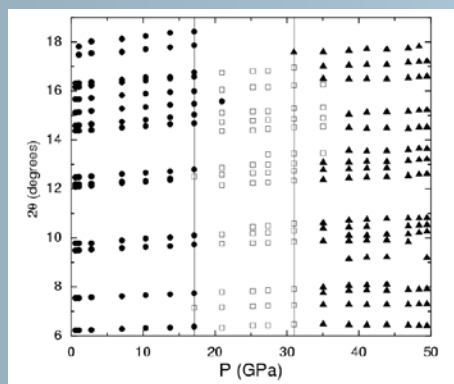
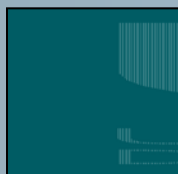


Figure 3.  $\text{MgWO}_4$  peaks evolution with pressure. Circles: wolframite, squares: triclinic phase, triangles: second HP phase. The vertical lines indicate transition pressures.



# Raman characterization of carbon materials under non-hydrostatic conditions

E. del Corro, M. Taravillo, V.G. Baonza

1 MALTA-Consolider Team, Departamento de Química Física I, Facultad de Ciencias Químicas, Universidad Complutense de Madrid, Plaza de Ciencias 3, 28040-Madrid, SPAIN

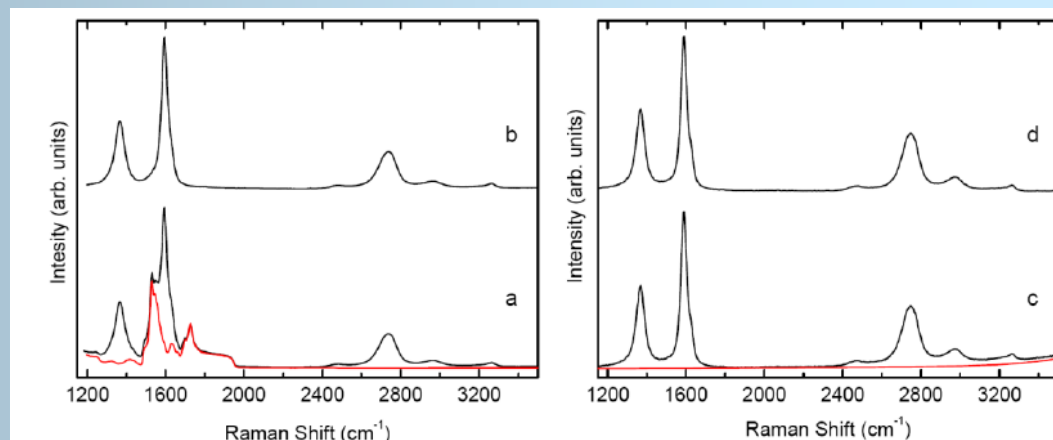
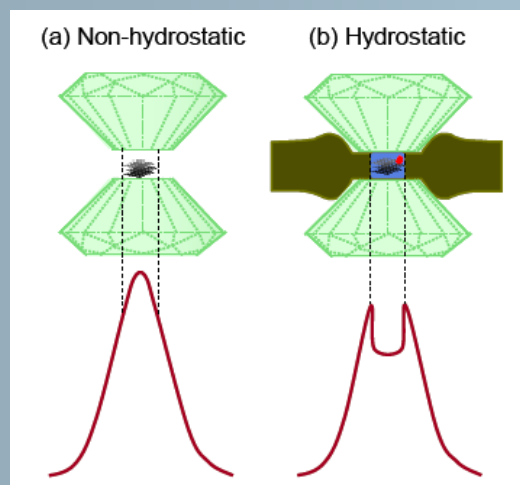
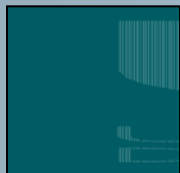


Fig. 4 – (a, c) Measured Raman spectra of two graphite samples (black lines) by using moissanite (left) and sapphire (right) anvils. Both samples were first compressed to effective stresses above 10 GPa and the stress was released down to room pressure. The spectra shown in the figure are those measured around 3 GPa. In both figures the background contribution from the anvils is shown as a red line. (b, d) Background-corrected Raman spectra of the two HOPG samples.



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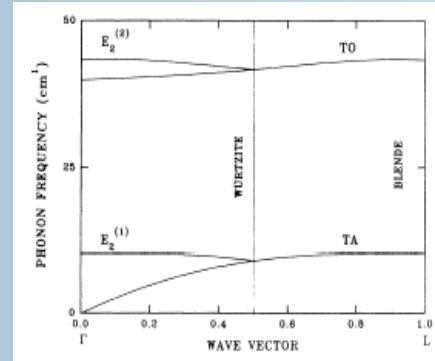
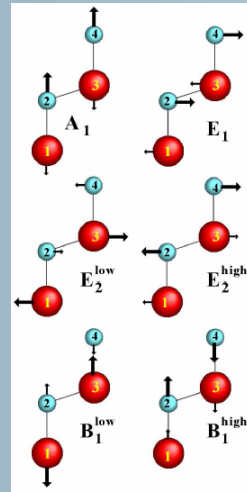
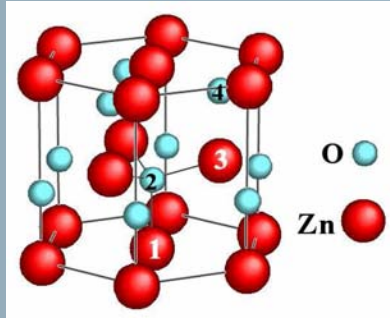
<http://grupos.unican.es/apye>

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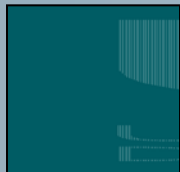
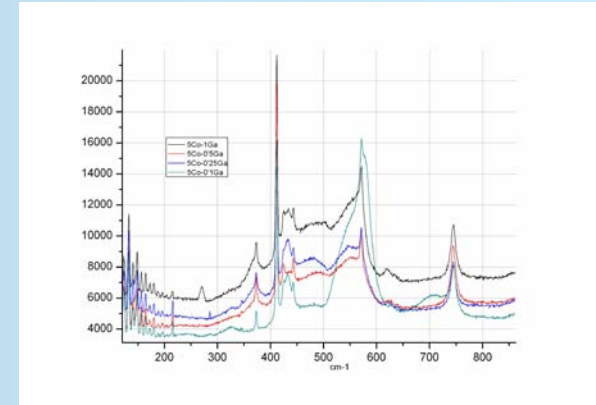
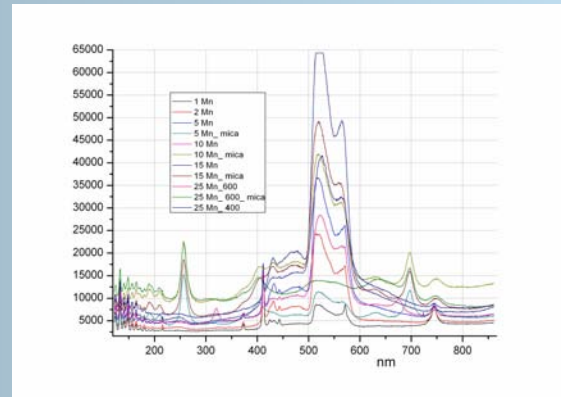
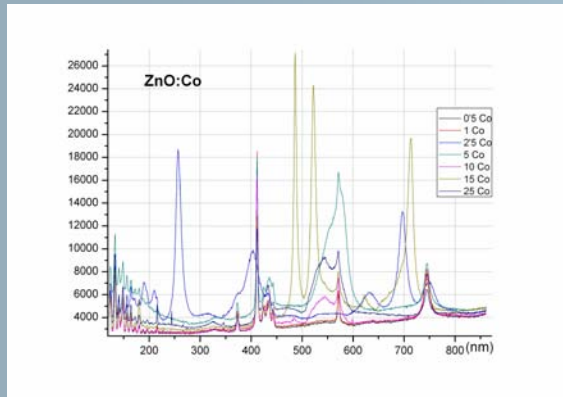
Universidad de Valencia

Profesor Alfredo Segura y Gloria Almonacid (Estudiante de Doctorado)  
Caracterización de Películas delgadas de ZnO, dopadas con Co, Mn y Ga



$$\Gamma(\text{opt}) = 1A_1 + 2B_1 + 1E_1 + 2E_2.$$

Correspondence between the Brillouin zone of ZB and Wurtzite crystal along the [111] direction



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# Phonon softening on the specific heat of nanocrystalline metals

D P Rojas, L Fernández Barquín, J Rodríguez Fernández, L Rodríguez Fernández  
Grupo de Magnetismo del CITIMAC, Universidad de Cantabria

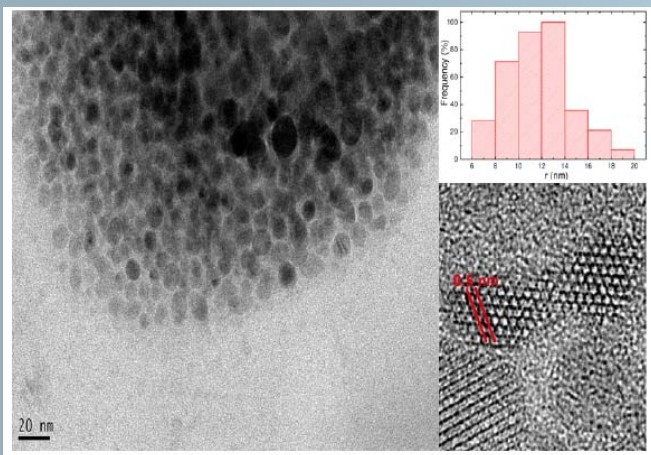


Figure 2. TEM image of nc LaAl<sub>2</sub> showing nanoparticles with a mean size of 12 nm. In the insets, details of the distribution of the particle sizes (aboveright) and high resolution TEM images of a particle (below right) are given. The interatomic distance between lattice planes inside a particle is around 0.5 nm.

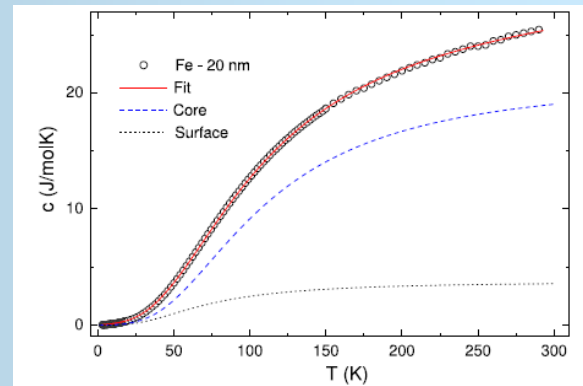


Figure 4. Temperature dependence of the specific heat of nc-Fe with a mean particle size of 20 nm. The solid line is a fitting using equations (1) and (2). Partial phonon contributions from the core and surface of the particles are also presented.

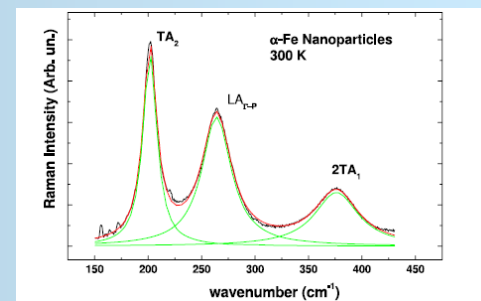
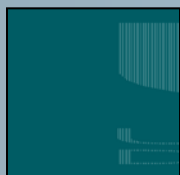


Figure 5. Raman spectrum of nc-Fe (20 nm) at 300 K showing overtones of acoustic phonons (TA<sub>1</sub> and TA<sub>2</sub>) in the -N direction and LA in the -P ones. The solid lines are the result of the fitting of the peaks with Lorentzian functions.



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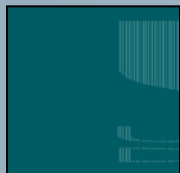
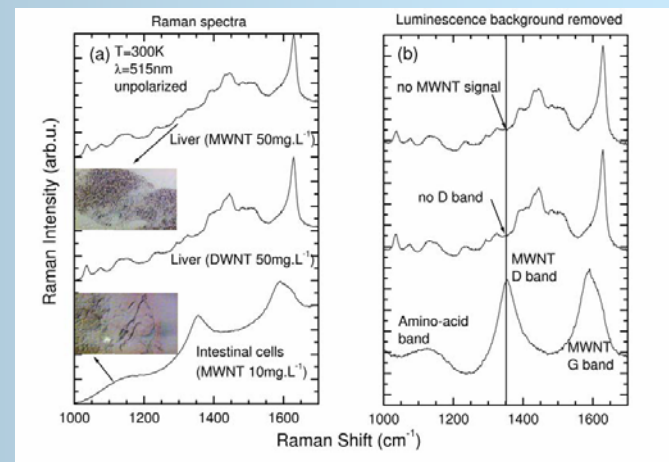
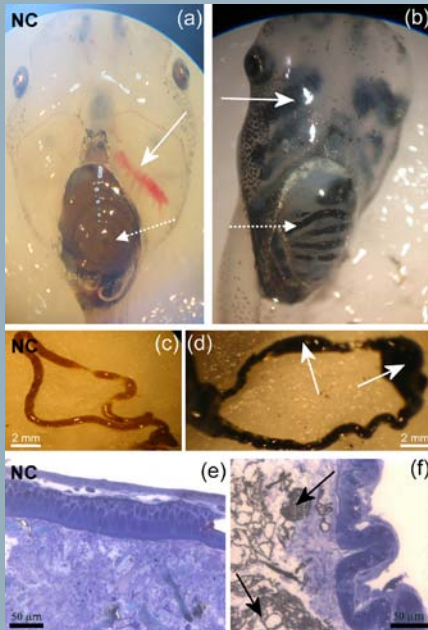


## Characterisation and *in vivo* ecotoxicity evaluation of double-wall carbon nanotubes in larvae of the amphibian *Xenopus laevis*

Light photomicrographs of *Xenopus* larvae exposed 12 days to 10 mg L<sup>-1</sup> of CNTs compared to the control group:

general observation of entire larvae, binocular observation and histology of intestine. NC: negative control. On Fig (a and b), plain white arrows indicate traps and dotted white arrows show the intestine. Plain white arrows on Fig. (d) show the distended aspect of the intestine in larvae exposed to CNTs, as compared to the intestine of non-exposed larvae (Fig. (c)).

Black arrows in Fig. (f) indicate the presence of black fibrous structures in the intestine lumen of CNT-exposed larvae. Sections shown in Fig. (e) and (f) were stained with methylene blue.

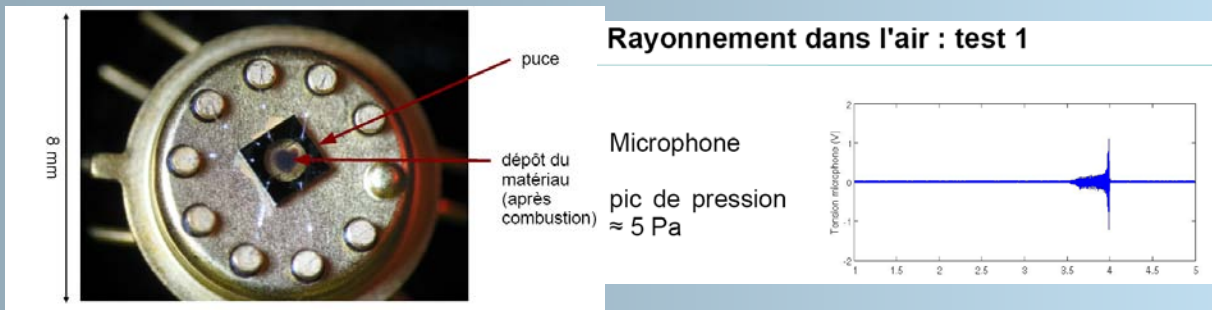


# Measurements of ignition properties at lab scale under oil well conditions: Pressure-Temperature (hexamine cobalt nitrate) Nano-Explo

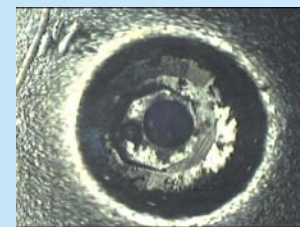
B. Chaudret (LCC, UPS, Toulouse), J.M. Broto (LNCMI, CNRS, Toulouse), E. Flahaut (CIRIMAT, Toulouse)

Rayonnement acoustique de la combustion d'un matériau énergétique: (hexamine cobalt nitrate)

Ignition by Heating at  $P=0$  (hexamine cobalt nitrate)

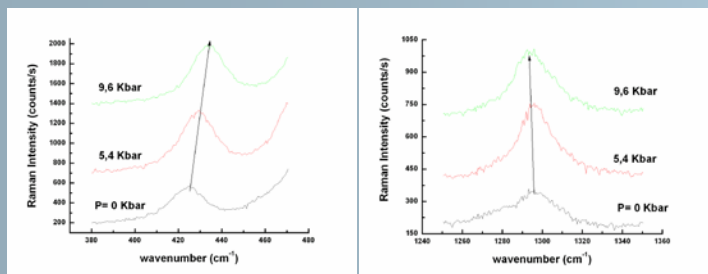


Before Heating



After Heating at 270 ° C

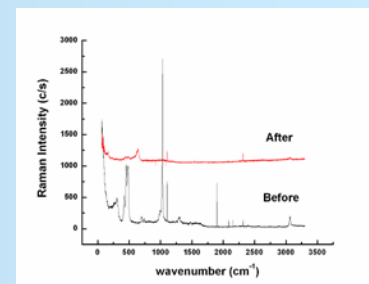
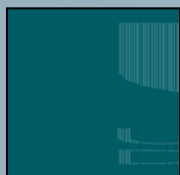
Raman Scattering Under Pressure (hexamine cobalt nitrate)



Preliminary conclusions:

- No phase transitions of (hexamine cobalt nitrate) up to 10 Kbar at room temperature
- The ignition temperature at atmospheric pressure is about 270° C
- The ignition temperature at 10 Kbar is about 240° C
- In-situ (P,T) Raman and optical measurements for ignition sensor

Symmetric bending mode NH<sub>3</sub>



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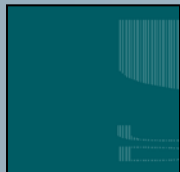
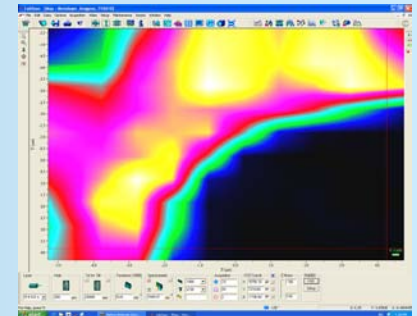
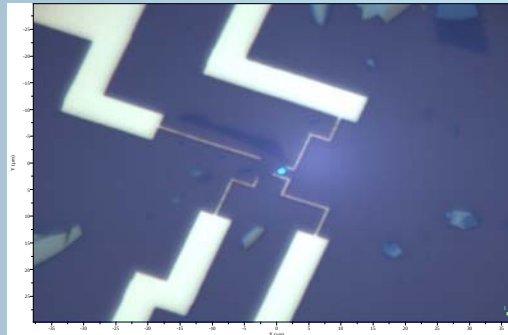
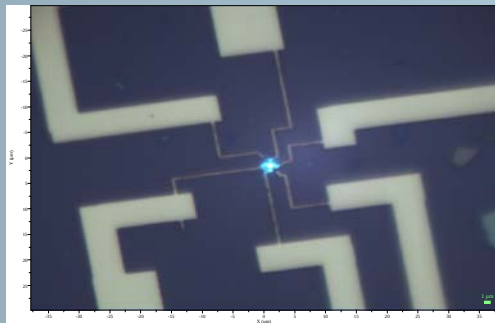
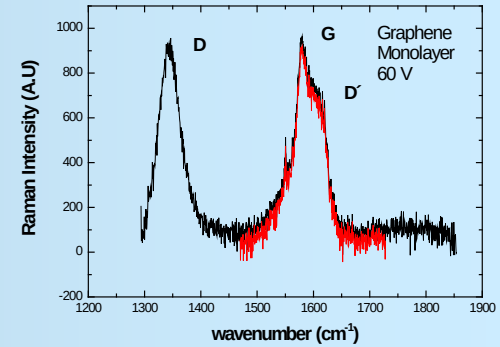
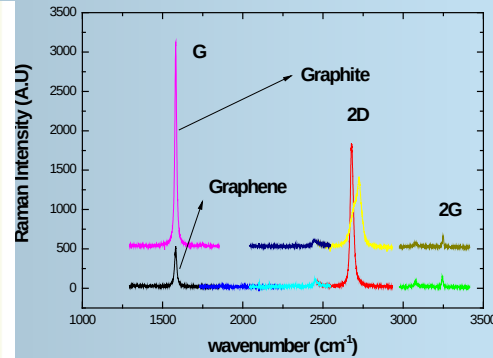
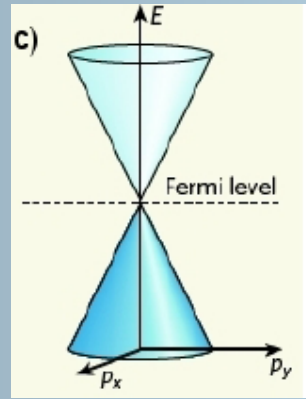
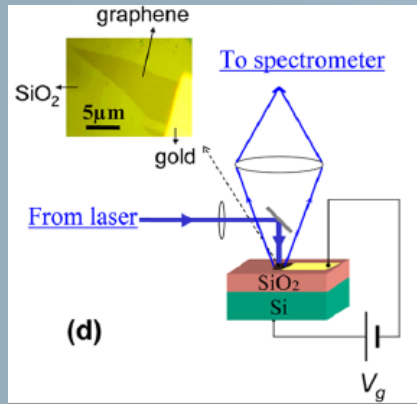
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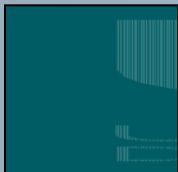
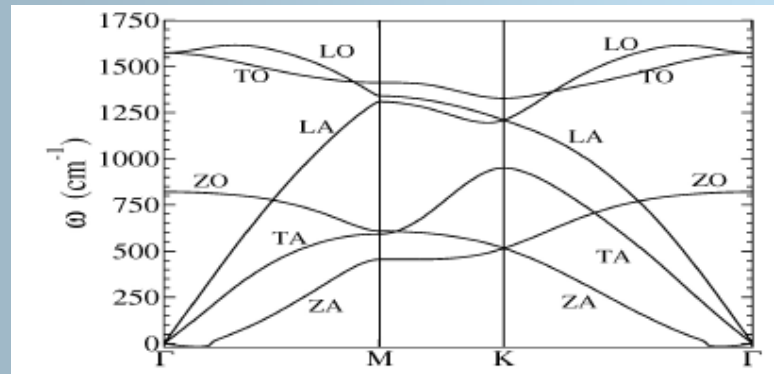
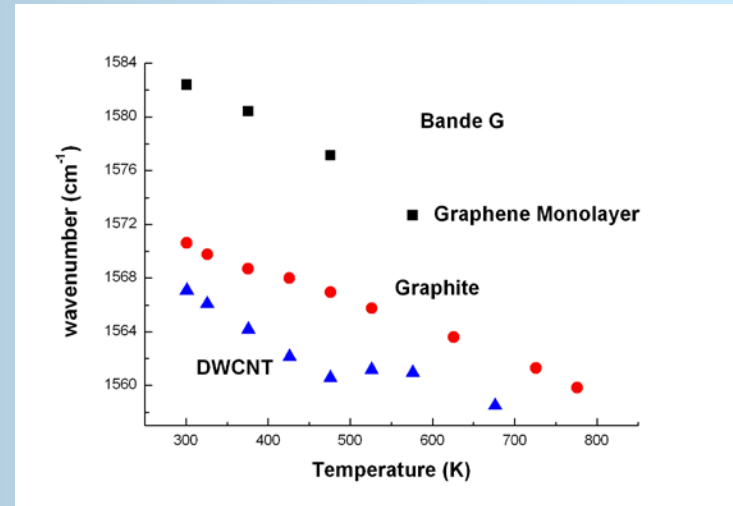
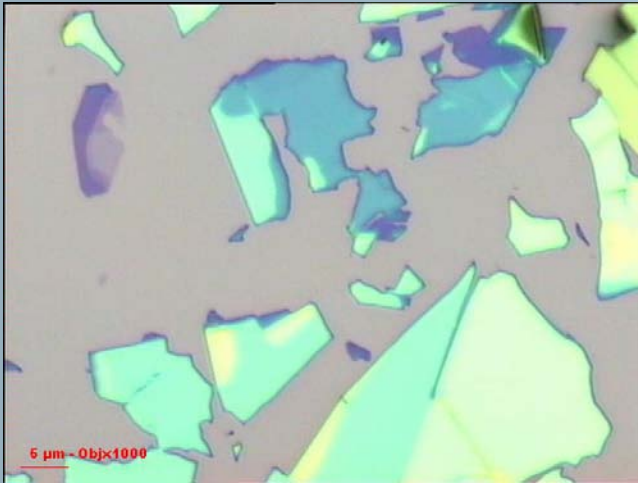
Colaboración Internacional con el LNCMI (CNRS-Toulouse)- Universidad de Toulouse  
 Profesor Jean Marc Broto y Jean Marie Poumirol (Estudiante de Doctorado)  
 Visita de 1 semana al laboratorio. (22/03/2010)  
 Grafeno Monolayer Exfoliado  
 Raman en funcion del Voltaje de Gate



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# Platina Calefactora hasta 1300 K en colaboración con Antonio Barreda

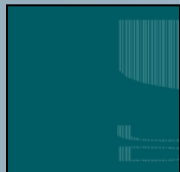


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## **Proyectos:**

- Concepción de celdas para difracción de neutrones en altas presiones con gran volumen de muestra, CDTI- Cryovac (Madrid), Noviembre 2010**
- Determinación de la micro-estructura en diferentes tipos de hierro fundido por medio de la Espectroscopia Raman, Saint Gobain (SODERCAN) Diciembre 2010**  
**En colaboración con SCTI-Servicios Científico-Técnicos de Investigación**  
**Universidad de Cantabria.**
- Optimización del proceso de colada y mejora de la calidad de los componentes mediante la técnica de análisis térmico y espectroscopia Raman, Nissan (SODERCAN) Noviembre 2009, en colaboración con SCTI-Servicios Científico-Técnicos de Investigación Universidad de Cantabria.**
- Caracterización de materiales nanocompuestos, Centro Tecnológico de Componentes (CTC-SODERCAN) Septiembre 2009**



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# Publicaciones 2010

- Kyle W. Galloway, Stephen A. Moggach, Pascal Parois, Alistair R. Lennie, John E. Warren, Euan K. Brechin, Robert D. Peacock, Rafael Valiente, Jesús González,,Fernando Rodríguez, Simon Parsons and Mark Murrie,

## **Pressure-induced switching in a copper(II) citrate dimer**

CrystEngComm, **12**, 2516–2519 (2010)

- Marius Millot, Jean-Marc Broto, Jesus Gonzalez, and Fernando Rodríguez

## **Trigonal field acting at the Cr<sup>3+</sup> 2E states in ruby from magneto-optical measurements under high pressure**

Phys. Rev. **B 81**, 075120, (2010)

- Edgar Beldandia, Marius Millot, Jean-Marc Broto, Emmanuel Flahaut, Fernando Rodriguez , Rafael Valiente , Jesus Gonzalez

## **Pressure dependence of Raman modes in double wall carbon nanotubes filled with 1D Tellurium**

Carbon, **48**, 2566 (2010)

- Marius Millot, Jean-Marc Broto, Sylvie George, Jesús González, and Alfredo Segura

## **Electronic structure of indium selenide probed by magnetoabsorption spectroscopy under high pressure**

Phys. Rev. **B 81**, 205211, (2010)

- Ch. Power, I. Molina, L. Chacon y J. Gonzalez

## **Absorción óptica a altas presiones del TLGaSe<sub>2</sub>**

Revista Mexicana de Física, **56**, 217 (2010)

- Sebastien Nanot, Marius Millot, Bertrand Raquet, Jean-Marc Broto, Arnaud Magrez, Jesús Gonzalez

## **Doping dependence of the G-band Raman spectra of an individual multiwall carbon nanotube**

Physica E, **42**, 2466 (2010)

- J. Ruiz-Fuertes, M. N. Sanz-Ortiz, J. González, F. Rodríguez, A. Segura and D. Errandonea

## **Optical absorption and Raman spectroscopy of CuWO<sub>4</sub>**

Journal of Physics: Conference Series, **215**, 012048 (2010)

-J. Ruiz-Fuertes, S. López-Moreno, D. Errandonea, J. Pellicer-Porres, R. Lacomba-Perales, A. Segura, P. Rodríguez-Hernández, A. Muñoz, A. H. Romero, and J. González

## **High-pressure phase transitions and compressibility of wolframite-type tungstates**

J. Appl. Phys. **107**, 083506 (2010)

-J. Ruiz-Fuertes, D. Errandonea, R. Lacomba-Perales, A. Segura, J. González, F. Rodríguez, F. J. Manjón, S. Ray, P. Rodríguez-Hernández, A. Muñoz, Zh. Zhu, and C. Y. Tu

## **High-pressure structural phase transitions in CuWO<sub>4</sub>**

Phys. Rev. **B 81**, 224115, (2010)

- Marius Millot, Ramon Tena-Zaera, Vicente Munoz-Sanjose, Jean-Marc Broto, and Jesus Gonzalez

## **Anharmonic effects in ZnO optical phonons probed by Raman spectroscopy**

Appl. Phys. Lett. **96**, 152103 (2010)

-D. P. Rojas, L. Fernandez Barquin, J. Rodriguez Fernandez, L. Rodriguez Fernandez and J. Gonzalez

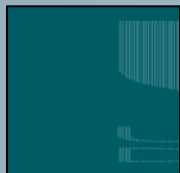
## **Phonon softening on the specific heat of nanocrystalline metals**

Nanotechnology, **21**, 445702 (2010)

-E. del Corro, M. Taravillo, J. Gonzalez, V.G. Baonza

## **Raman characterization of carbon materials under non-hydrostatic conditions**

Carbon, **49**, 973 (2011)



**Grupo de Altas Presiones y  
Espectroscopia**

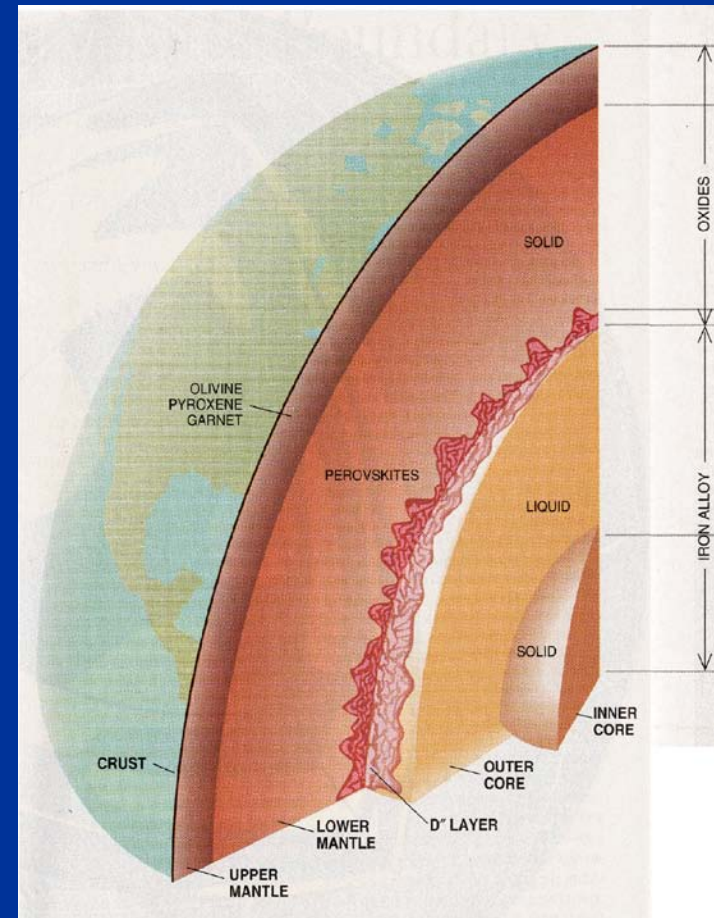
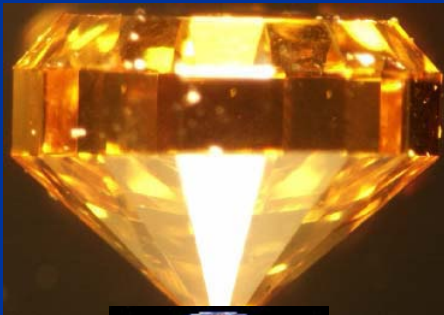
<http://grupos.unican.es/apye>

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# Sistema de Calentamiento Laser

## Universidad de Valencia



# IR - Laser



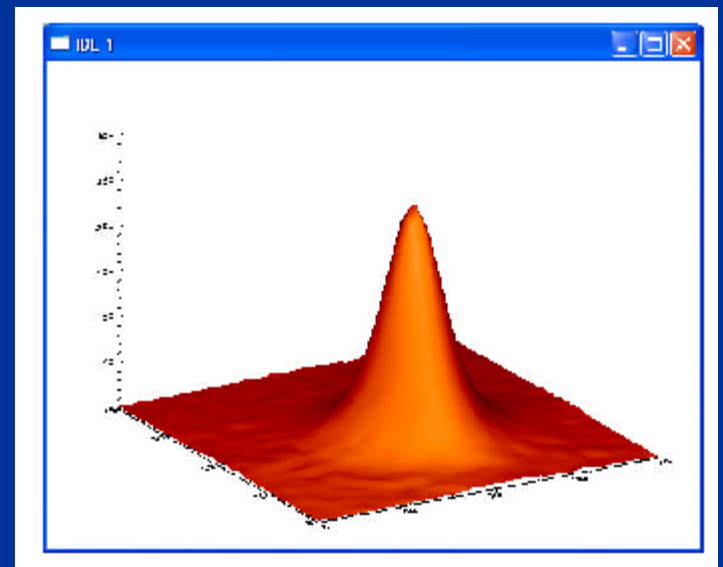
Quantronix - 20 W – Nd:YLF – TEM<sub>00</sub> - 1053 nm

Power Instability: 1%

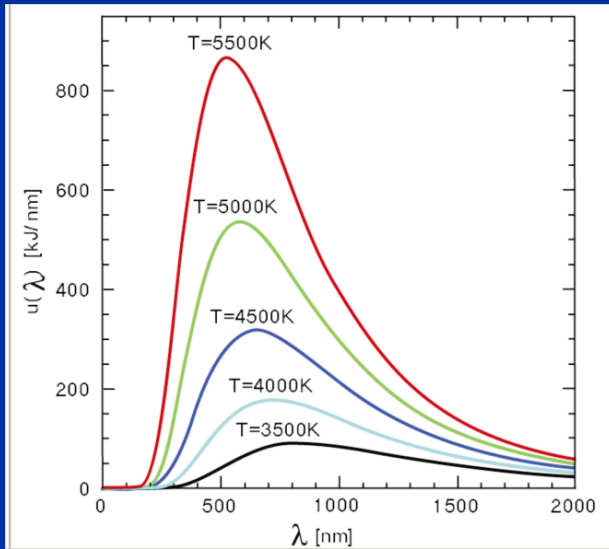
Beam Diameter : 1.5mm at output coupler

Beam Pointing Stability: 50μrad

Beam Divergence: 3.5mrad

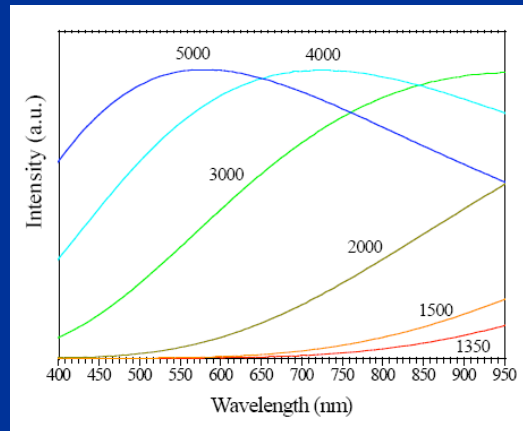
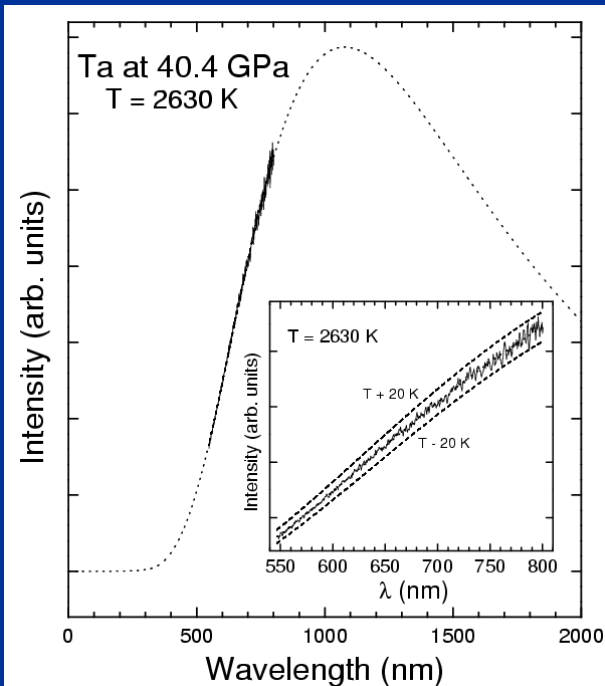
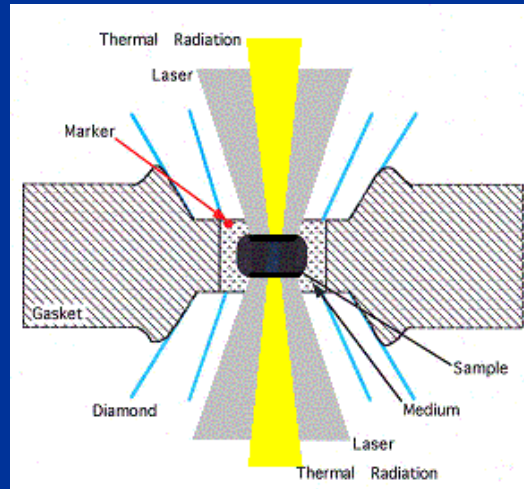
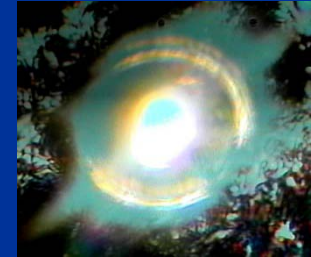






## Planck's law

$$I = \varepsilon \frac{2\pi hc^2}{\lambda^5} \frac{1}{e^{hc/\lambda kT} - 1}$$



## DAC Almax-Boehler P > 1 Mbar

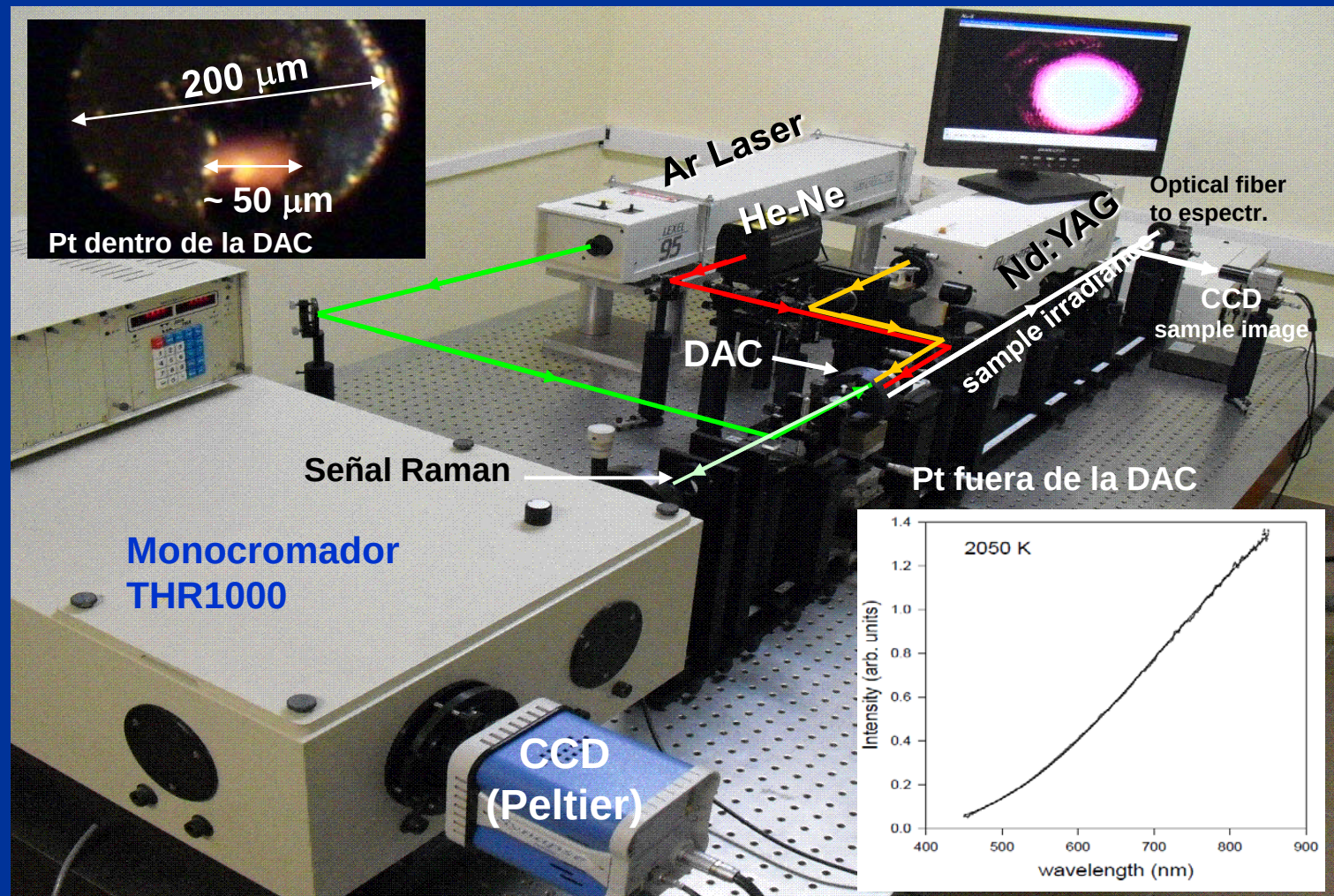


# Equipamiento montado:

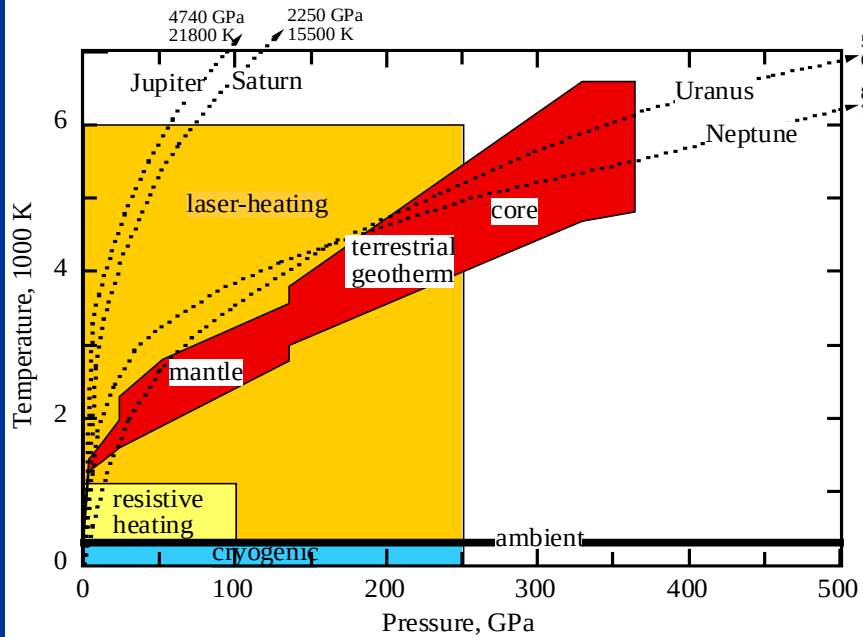
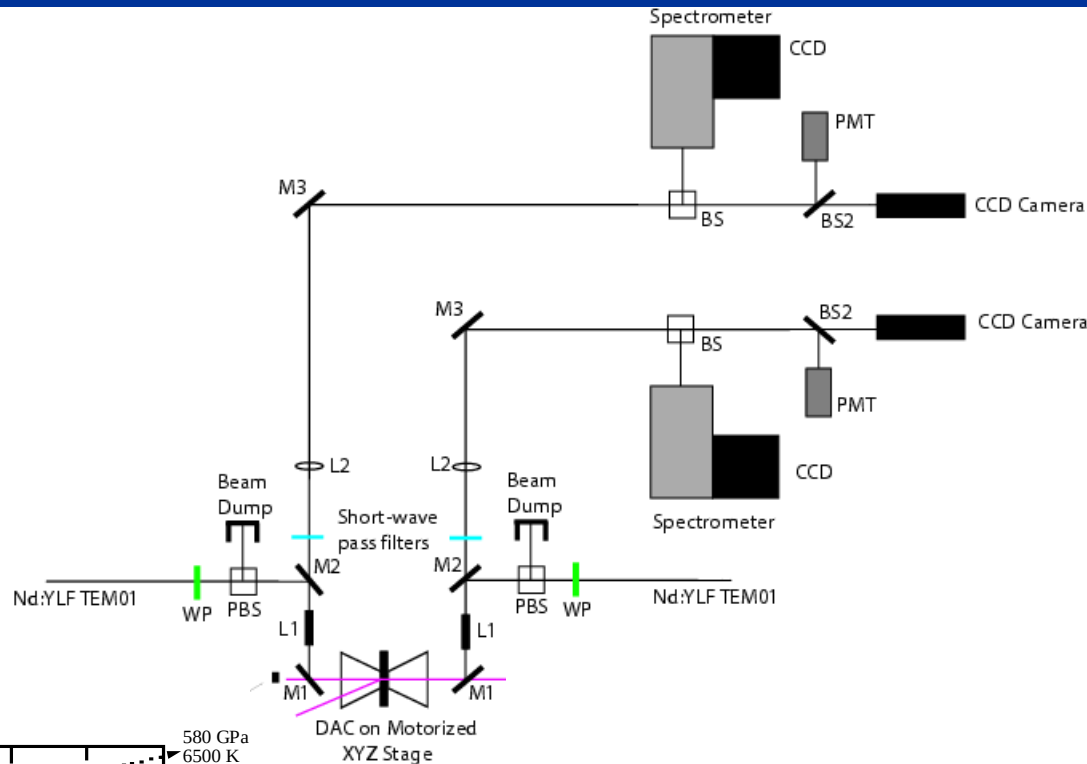
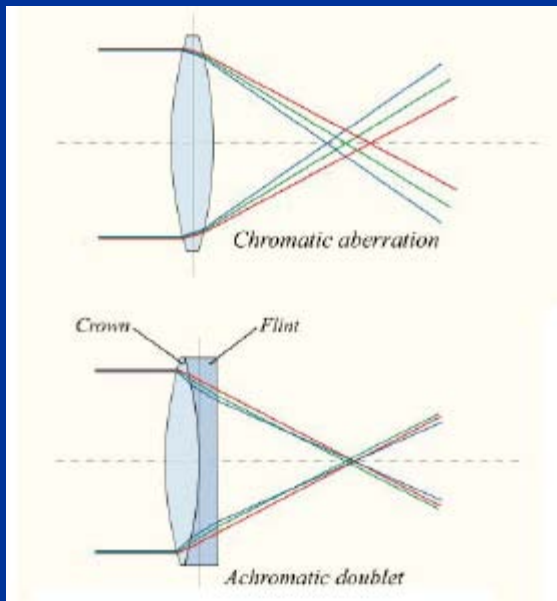
## Calentamiento láser en DAC + Raman *in situ*

Nd:YAG 20 W, 1064 nm

$P_{\max} \approx 1$  Mbar,  $T_{\max} \approx 2500$  K







Optical Fiber Laser

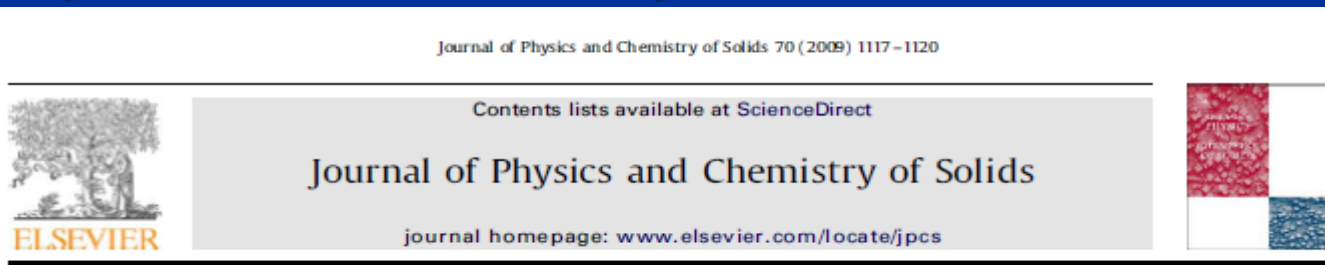
100 W







- Calentamiento dentro y fuera DAC
- Ahora  $P < 60$  GPa,  $T < 2500$  K
- Mediada de presión (RT) y temperatura in situ
- Adquisición temperatura, necesita ser automatizado
- Sistema de calibración y caracterización
- Nuevo espectrómetro (Acton Research) – ahora Ocean Optics
- Nuevo laser
- Double-sided LH (mas ópticas)
- Combinar LH con Raman
- Principal inconveniente: Manpower



Observation of chemical reactions between alkaline-earth oxides and tungsten at high pressure and high temperature

Daniel Errandonea

MALTA Consolider Team, Departamento de Física Aplicada-ICMUV, Fundación General de la Universidad de Valencia, C/Dr. Moliner 50, 46100 Burjassot, Spain



# Prensas de Gran Volumen

1<sup>er</sup> Simposio MALTA-Consolider  
23-27 Enero 2011, Miraflores de la Sierra

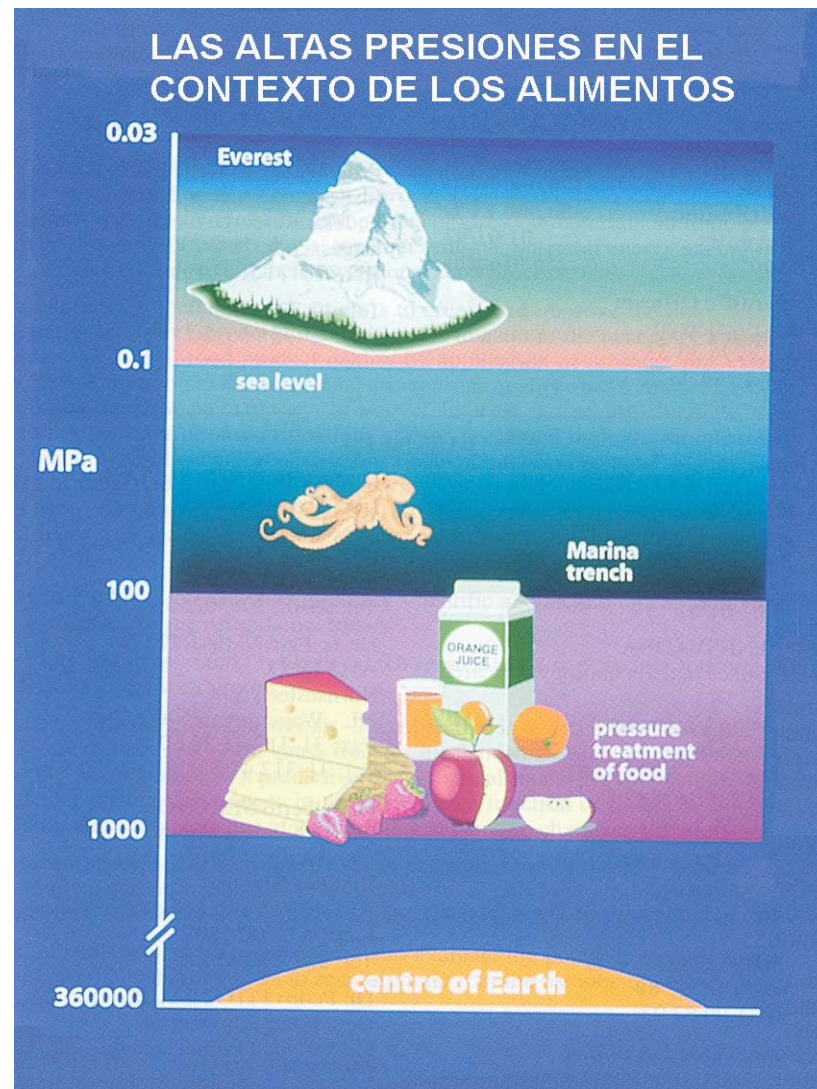
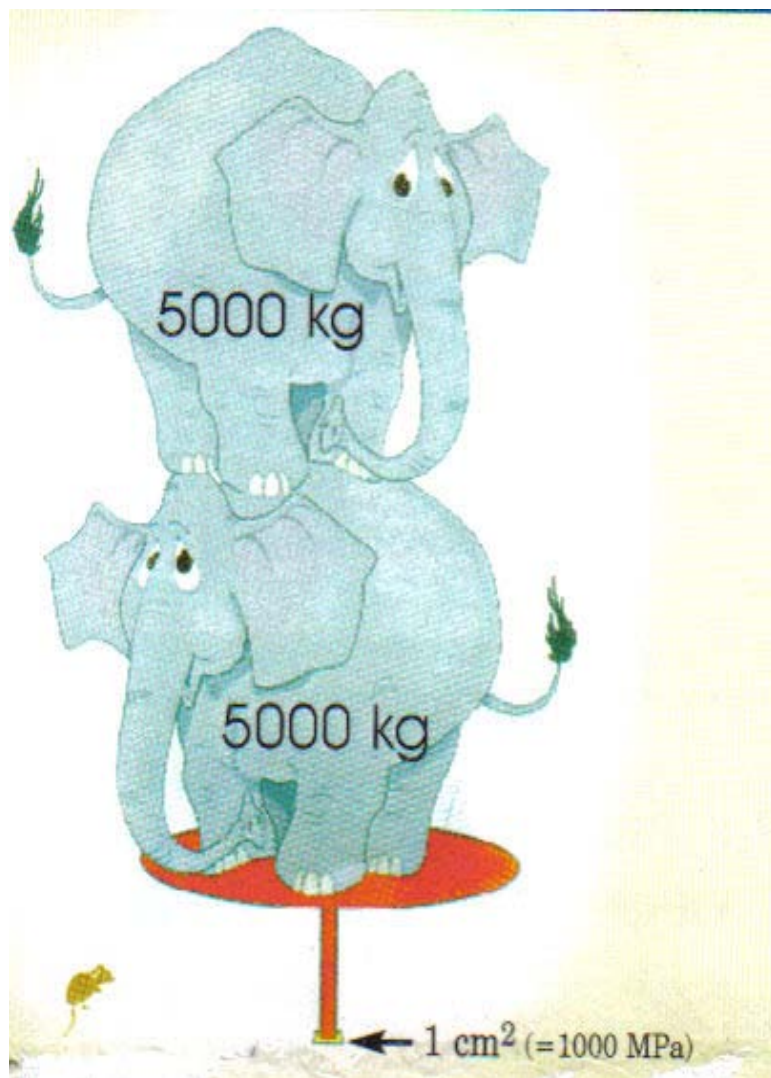
Dr. Ramón Gervilla

CER Planta Tecnología Alimentos  
Universitat Autònoma de Barcelona

[www.cerpta.com](http://www.cerpta.com)

# Altas Presiones en Alimentos

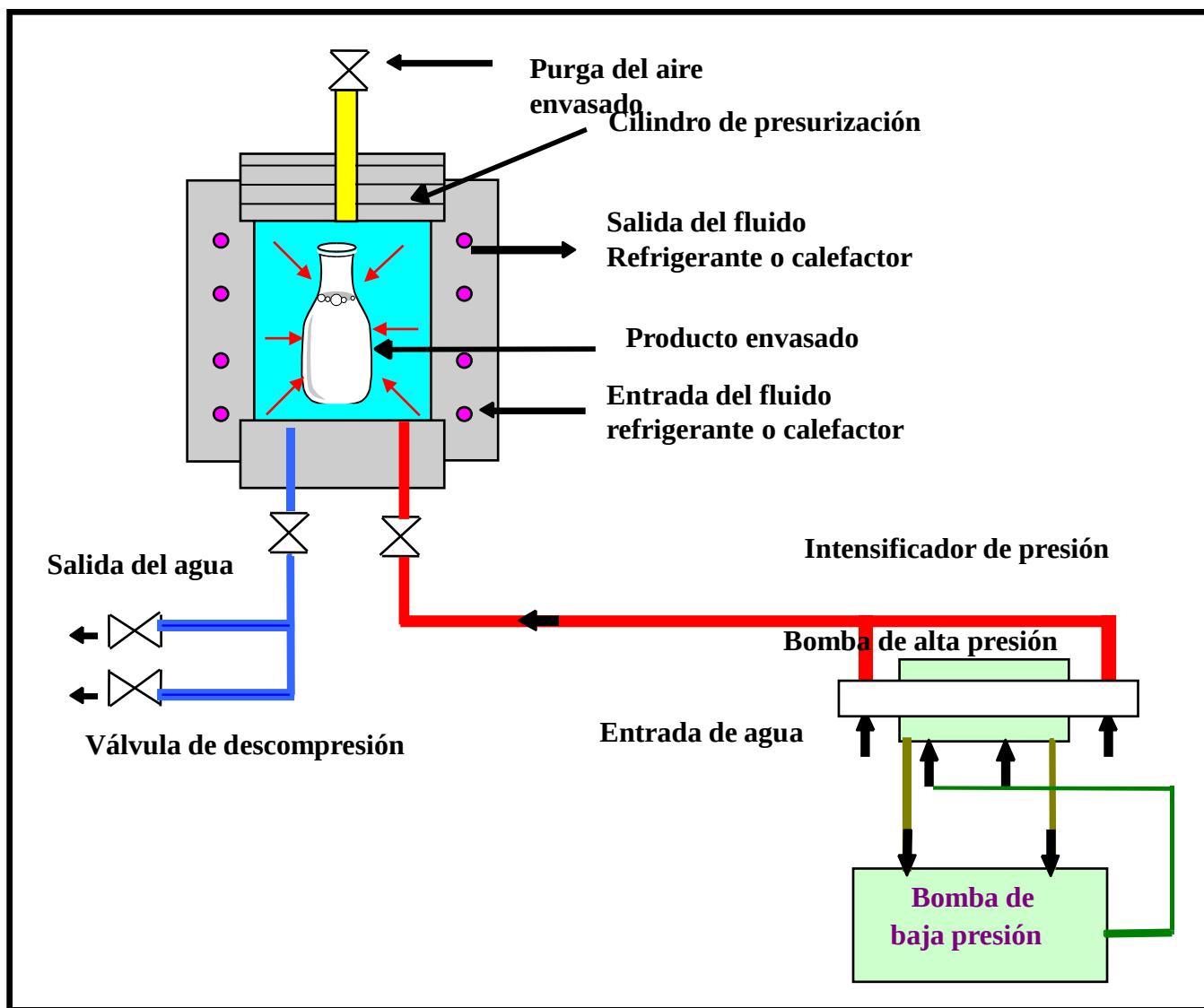
- 2 -  
 Miraflores  
 Enero-2011



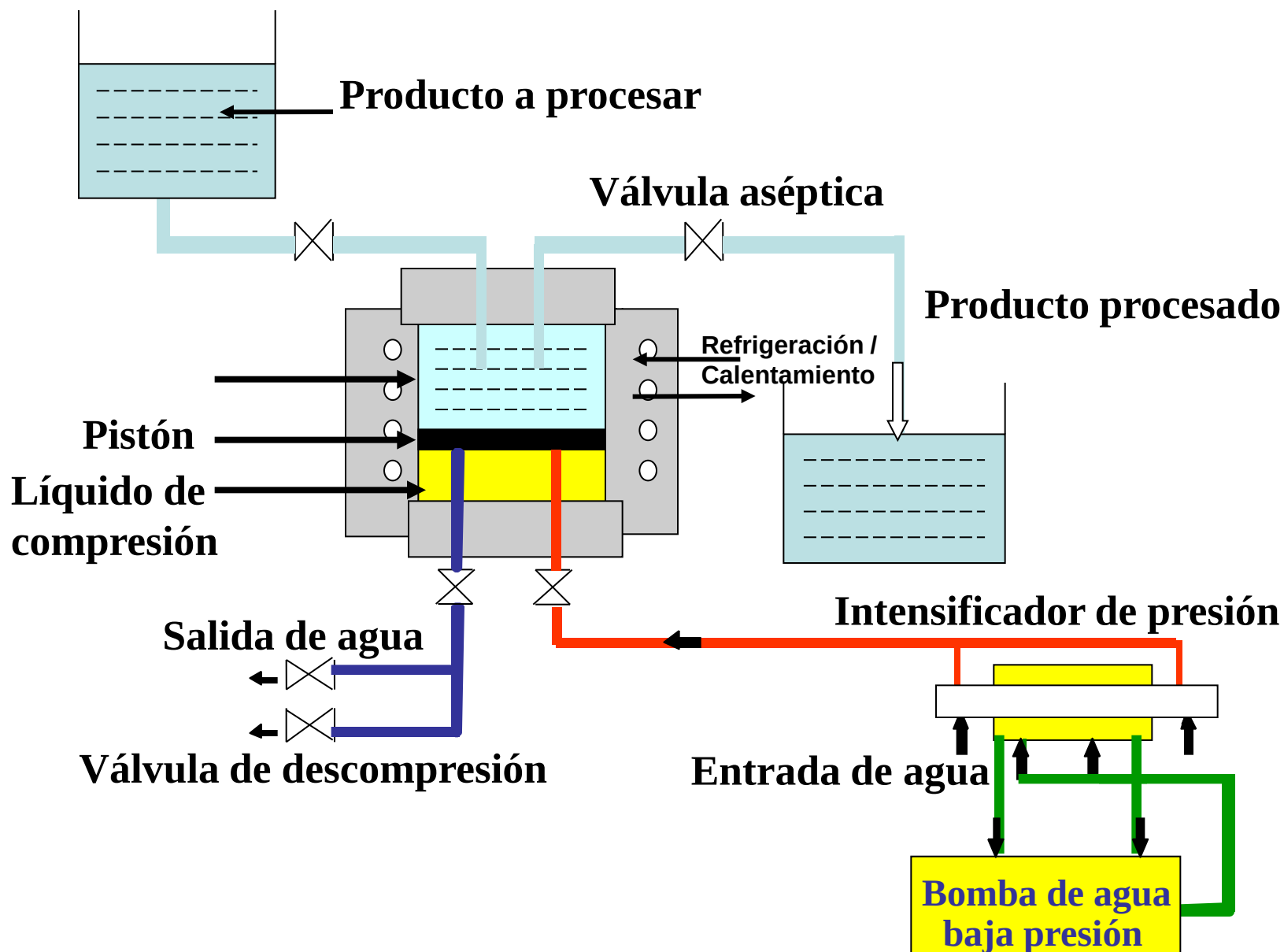
- **Según si fluido transmisor en compartimento del alimento**
  - Directos (líquidos)
  - Indirectos (alimento envasado)
- **Según tratamiento**
  - Tratamientos Discontinuos
  - Tratamientos Semicontínuos
- **Según cierre de la vasija**
  - Cierres de Rosca
  - Cierres de Yunque



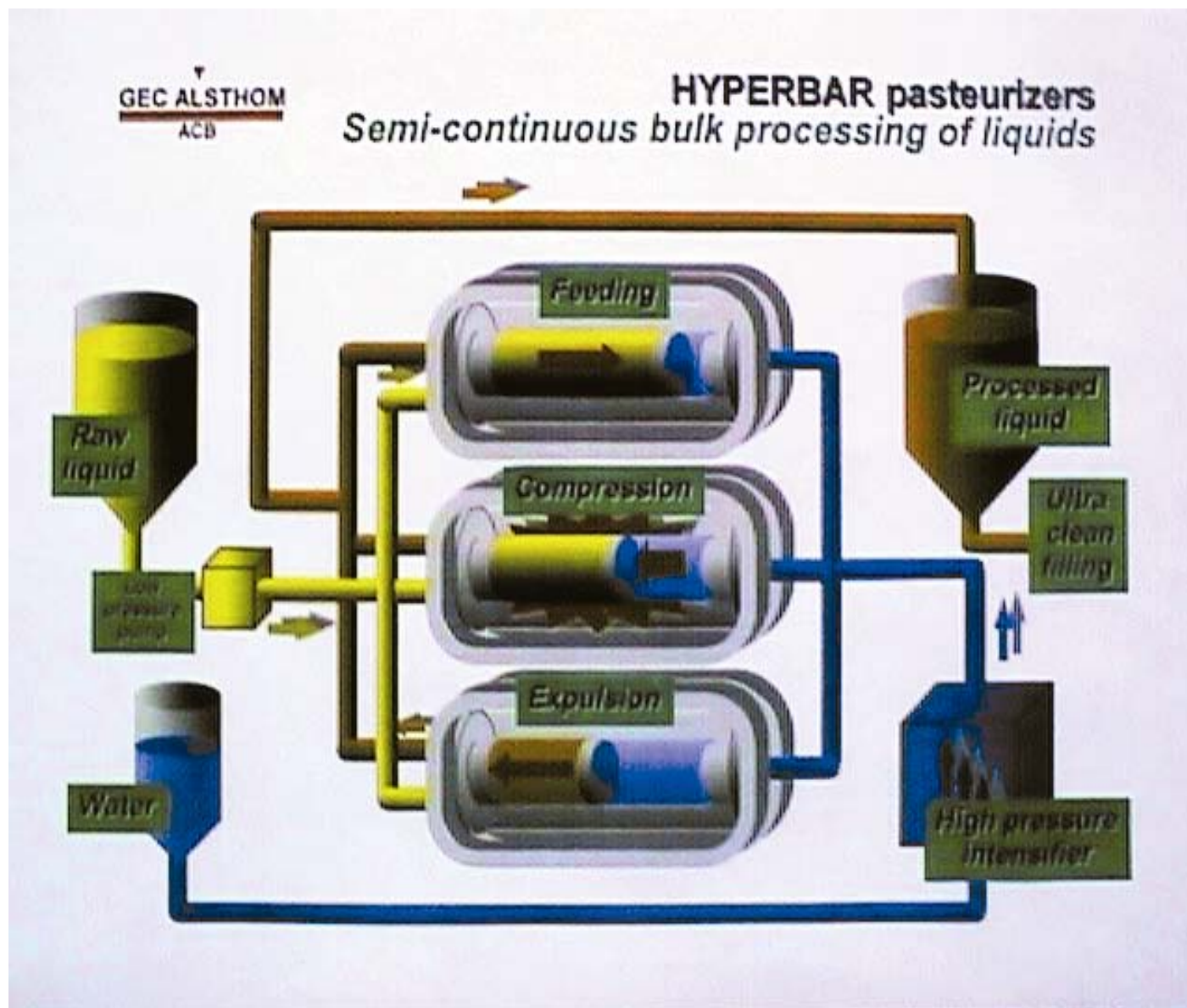
# Equipos Trat. Indirecto (Envasados)



# Equipos Trat. Directo (Líquidos)



# Equipos Semicontínuos





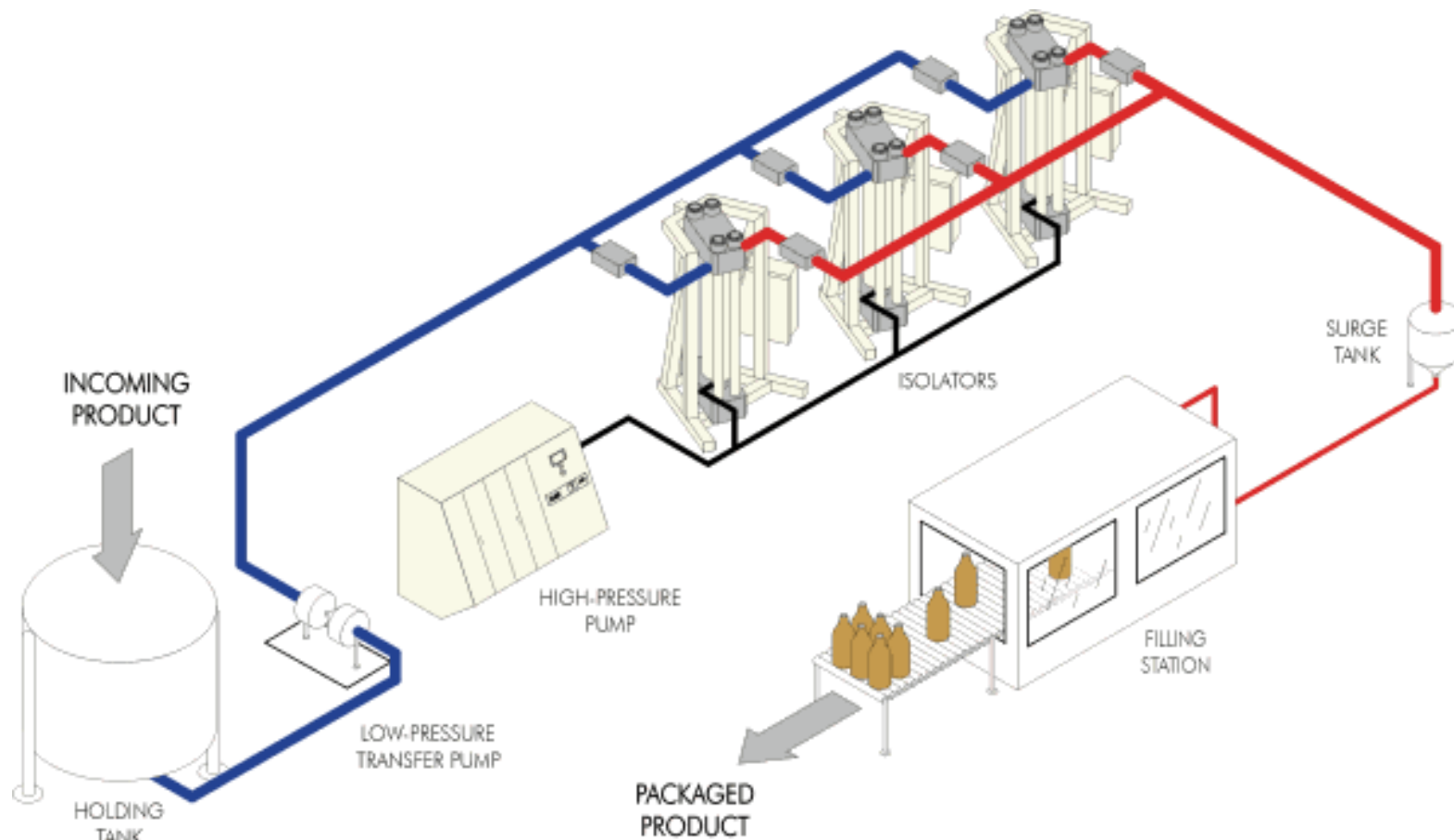
# Equipo Semicontínuo Mitsubishi

- 7 -  
Miraflores  
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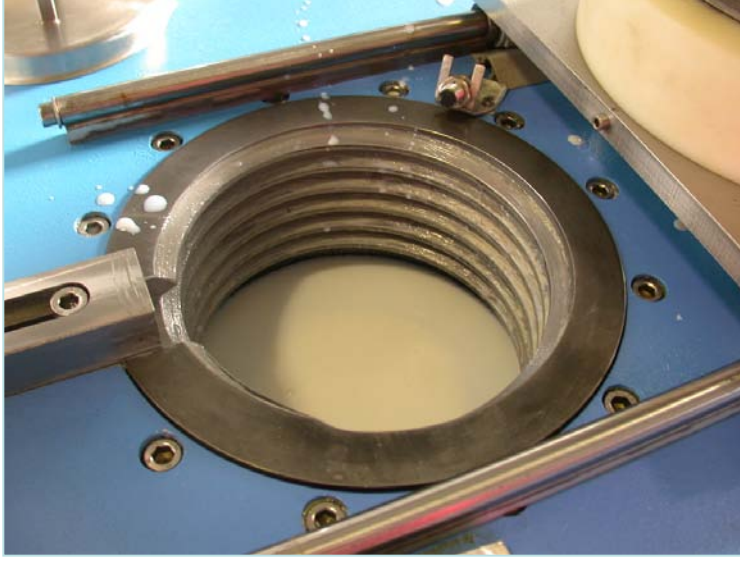
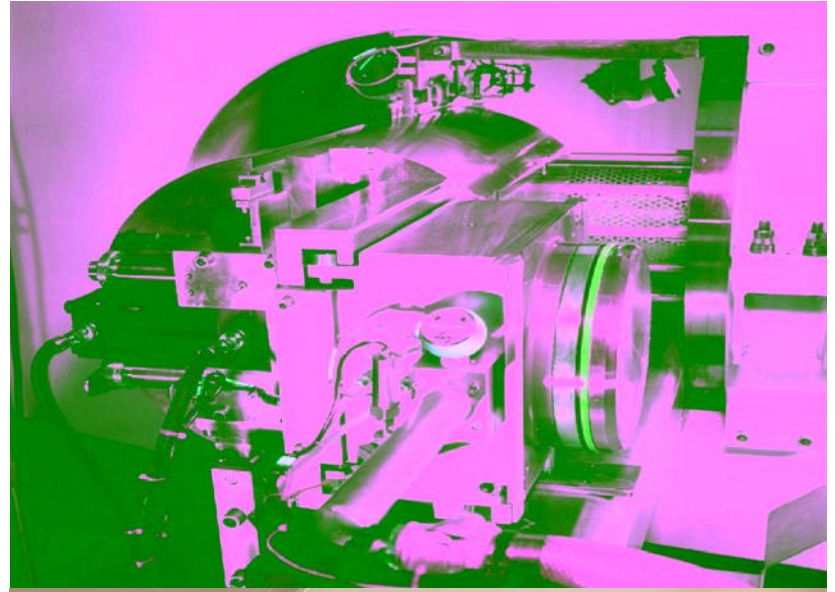


# Equipo Semicontínuo

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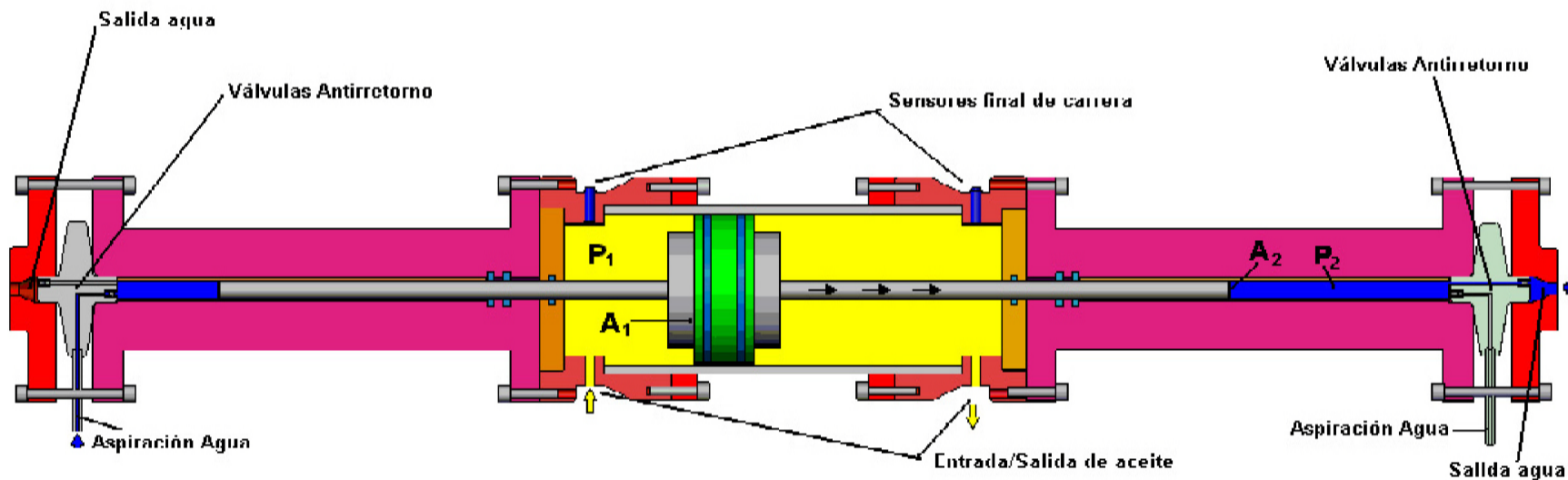
# Cierres en Rosca o Yunque





- Capacidades de la vasija de 50-500 L
- Presiones máximas de trabajo aprox. 600 MPa
- Temperaturas de trabajo de refrigeración (4-10 °C) a moderadas (30-40 °C)
- Ciclos de trabajo entre 3-10 min.
- Intensificadores dobles (altas velocidades de presurización)
- Sistemas de carga y descarga optimizados

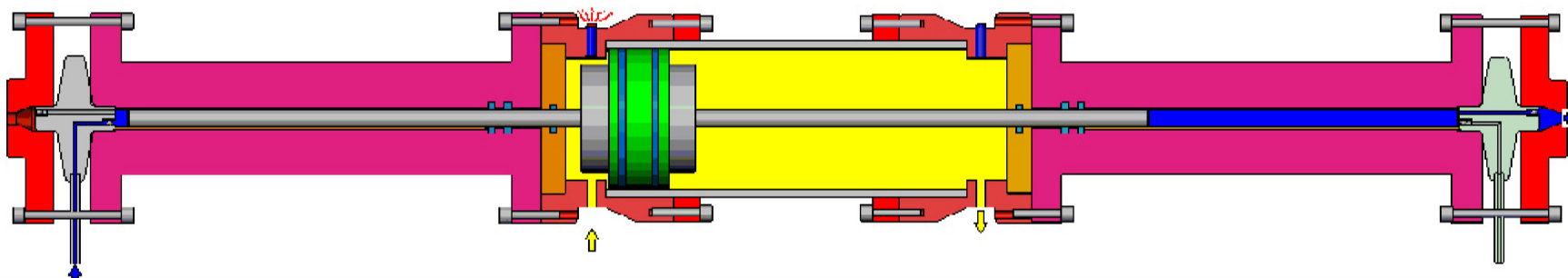
# Intensificadores dobles de altas presiones



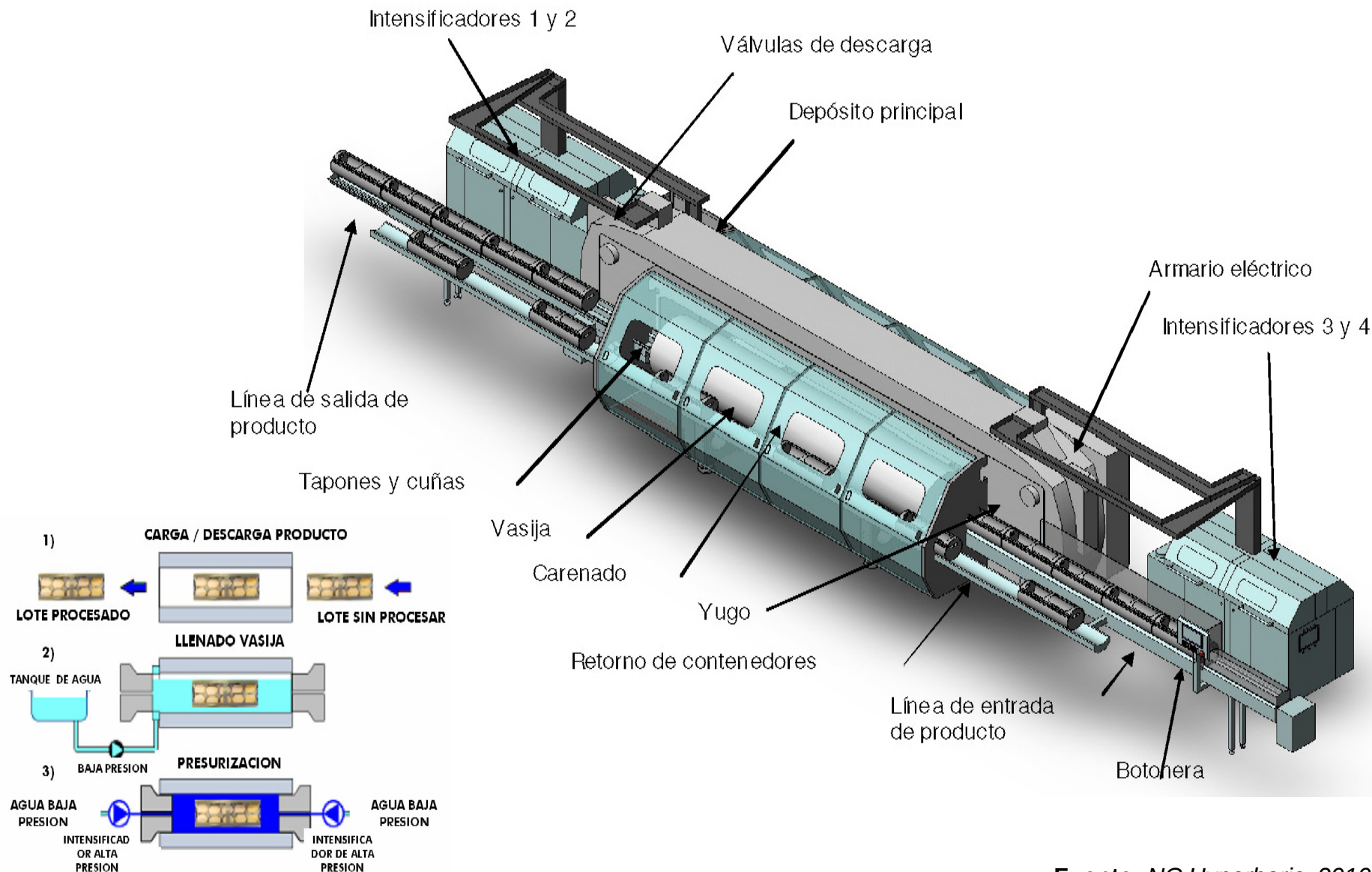
1 2

$$P_1 \cdot A_1 = P_2 \cdot A_2$$

$$A_1 \gg A_2 \quad P_2 \gg P_1$$



# Optimización de la carga y descarga





- **NC-Hyperbaric (E)**
- **Avure Technologies (USA)**
- **Mitshubishi Heavy Industries (J)**
- **Elmhurst Research (USA)**
- **Engineered Pressure Systems Inc. (USA)**
- **Kobeco (J)**
- **Stansted Fluid Power (UK)**
- **Uhde Hochdrucktechnik (D)**
- **Unipress (PL)**

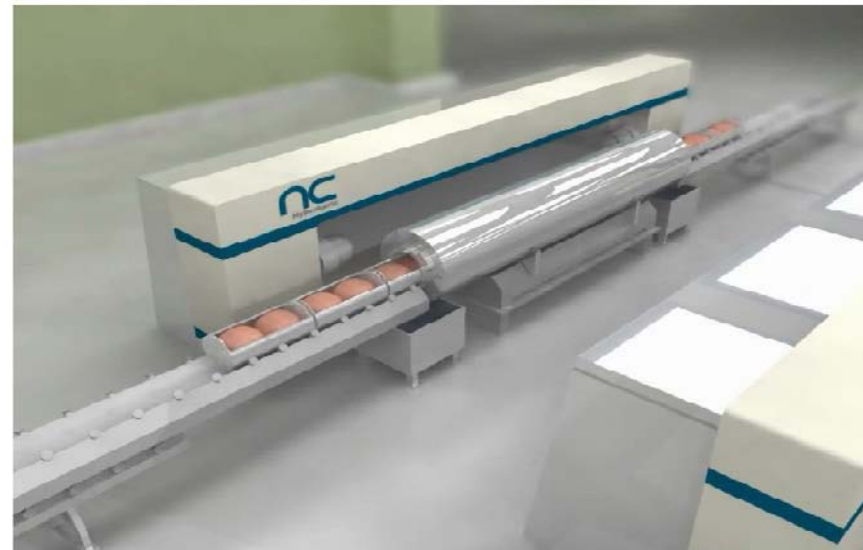
## ACB GEC ALSTOM

- 14 -  
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## NC HYPERBARIC

- 15 -  
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Enero-2011

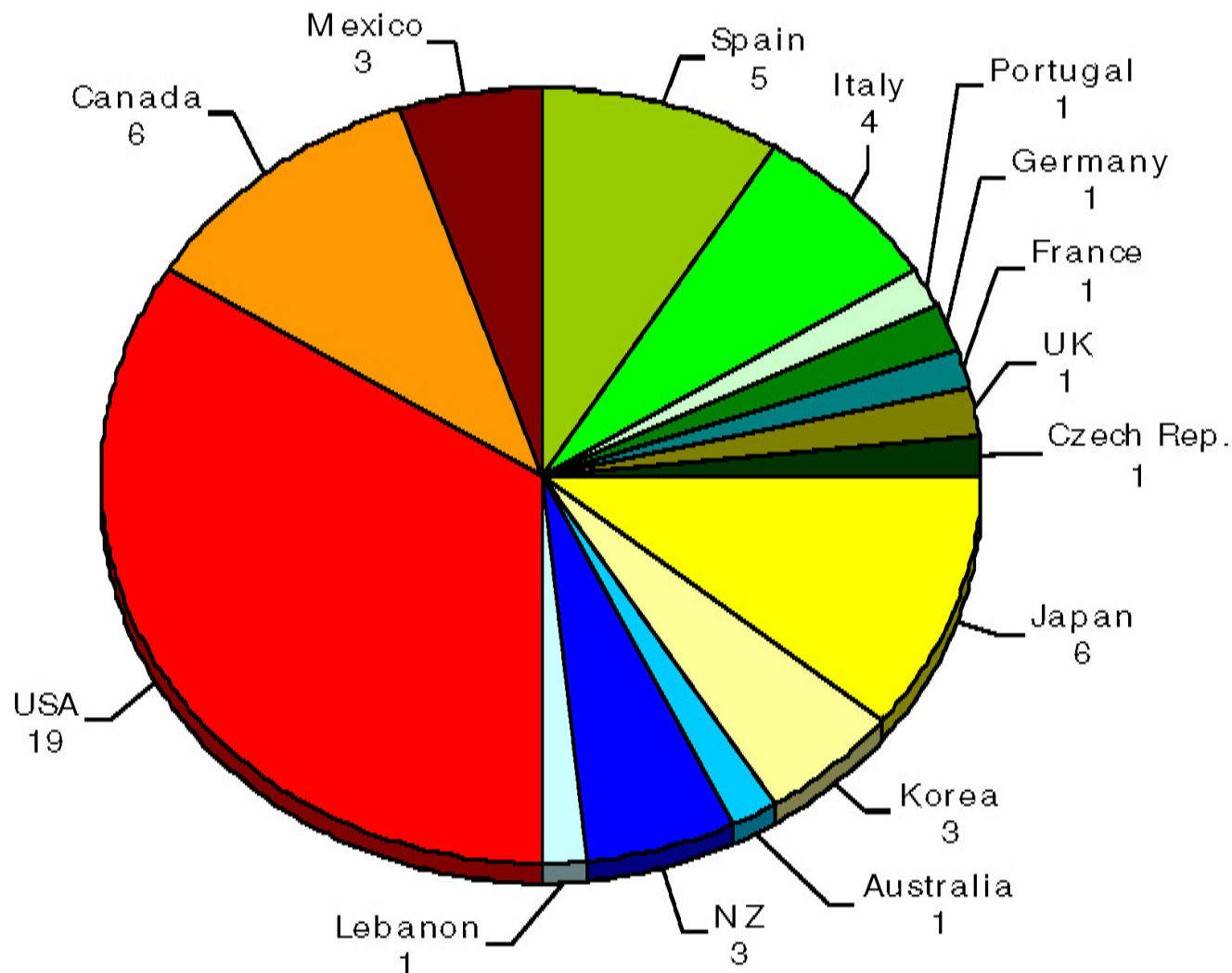






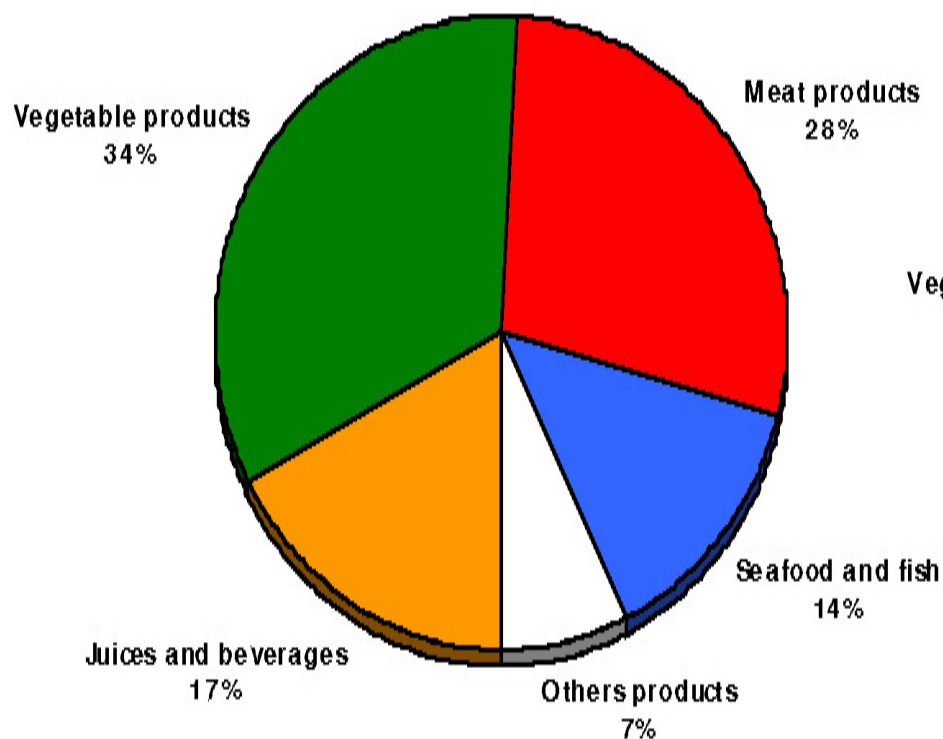
## Empresas usuarias por país

- 17 -  
Miraflores  
Enero-2011

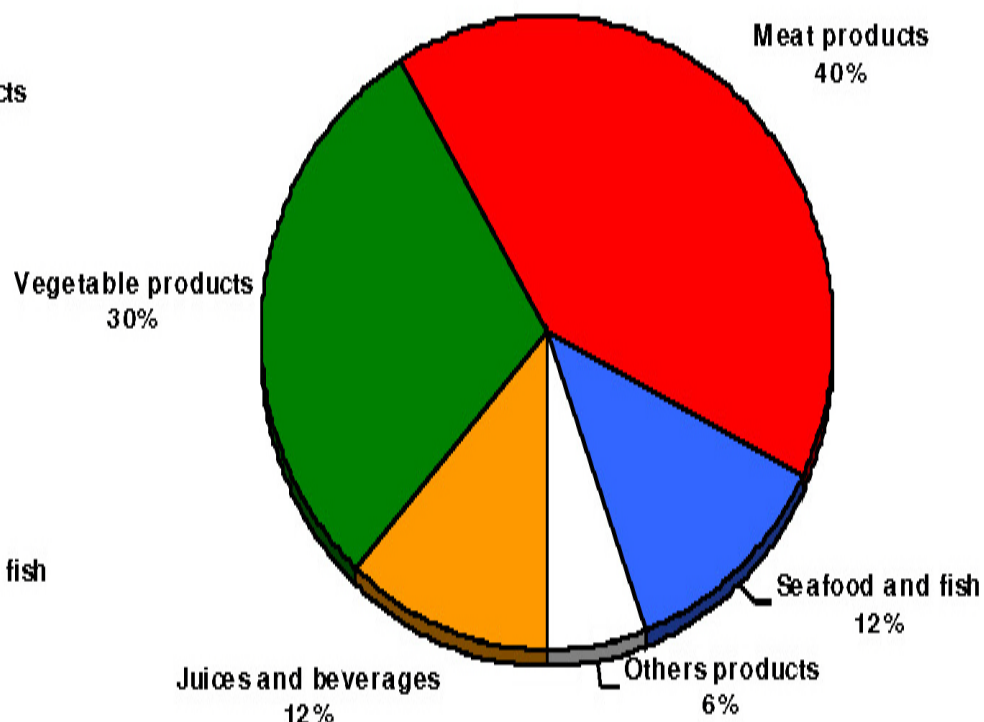


Fuente: NC Hyperbaric, 2010

% del numero total de máquinas



% del volumen total de máquinas



- Alargar la vida comercial, manteniéndose el frescor
- Inactivación enzimática
- Se conservan propiedades funcionales
  - Antioxidantes
  - Antimutagénicas



# Productos vegetales

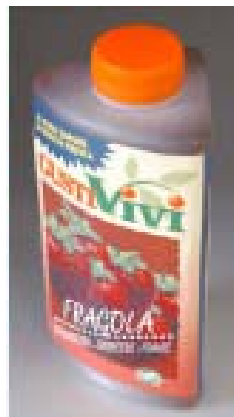




# Zumos de frutas y hortalizas

- 21 -  
 Miraflores  
 Enero-2011

- Alargar la vida comercial, manteniéndose el frescor
- Inactivación enzimática
- Se conservan propiedades funcionales
  - Antioxidantes
  - Antimutagénicas



# Productos cárnicos

- Alargar la vida comercial
- Higienización de loncheados y post-envasados
- Reestructurado y texturizaciones
- Tenderizaciones



# Productos de la pesca

- 23 -  
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- Obertura de ostras y reducción de la carga microbiana
- Recuperación de carne de crustáceos (255 MPa/90 s)
- Bacalao desalado
- Re-estructuración (merluza y salmón)



## **EQUIPAMIENTO ALTAS PRESIONES)**

- Equipo 500 MPa de 2 L (ACB)
- Equipo 900 MPa de 0,3 L (SFP)
- Equipo 800 MPa de 5 L (SFP)

## **EQUIPAMIENTO COMPLEMENTARIO (relevante)**

- Sistemas de Refrigeración y Calentamiento
  - Compresores y baños térmicos
  - Tanque y armario de N<sub>2</sub> líquido
- Sistemas de Envasado
  - Envasado al vacío y en atmósfera
  - Envasado aséptico (Tetra Brick Slim 250 mL)
  - Envasado higiénico (Tetra Recart)



# ACB 500 MPa 2 L

- 25 -  
Miraflores  
Enero-2011



# SFP 900 MPa 0,3 L

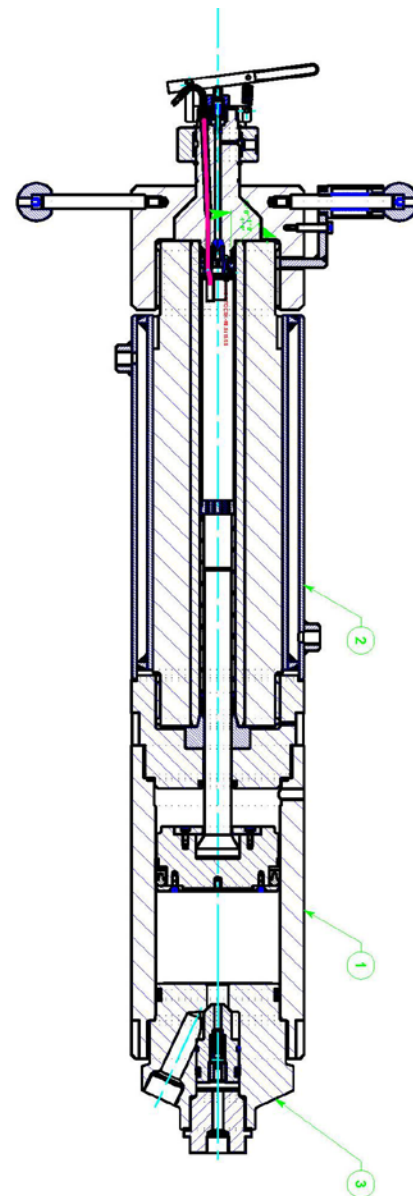
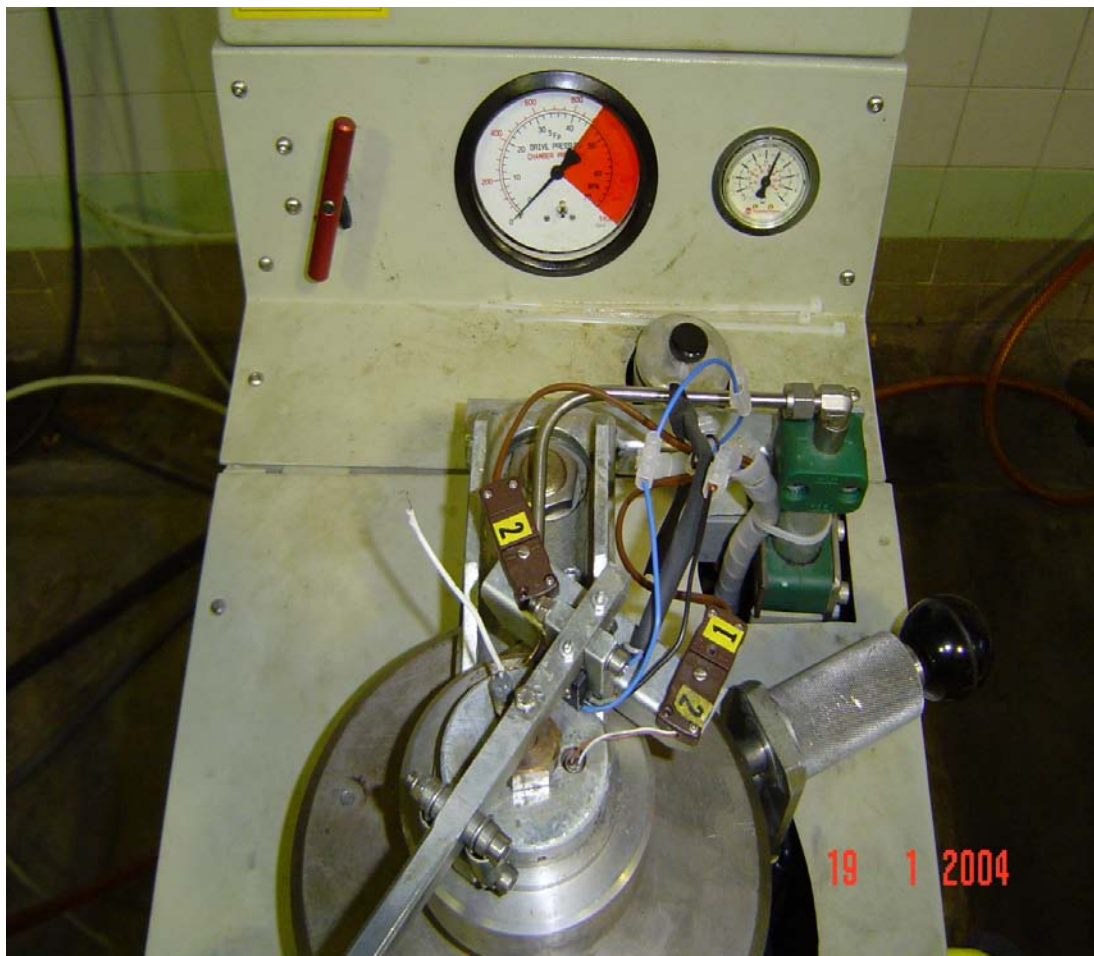
- 26 -  
Miraflores  
Enero-2011



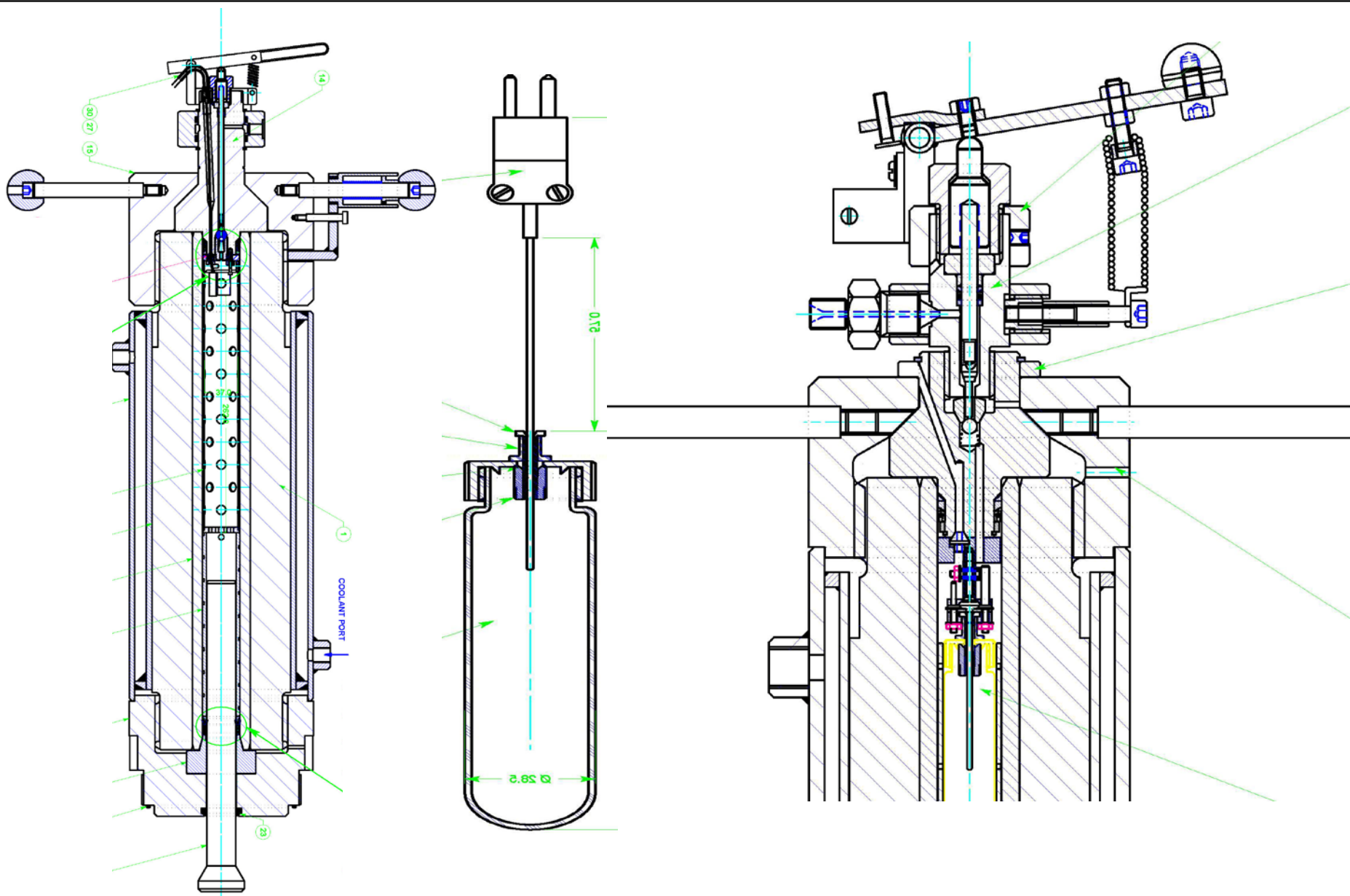


# SFP 900 MPa 0,3 L

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Miraflores  
Enero-2011



# SFP 900 MPa 0,3 L





**SFP 800 MPa 5 L**

**Modelo FPG11500**

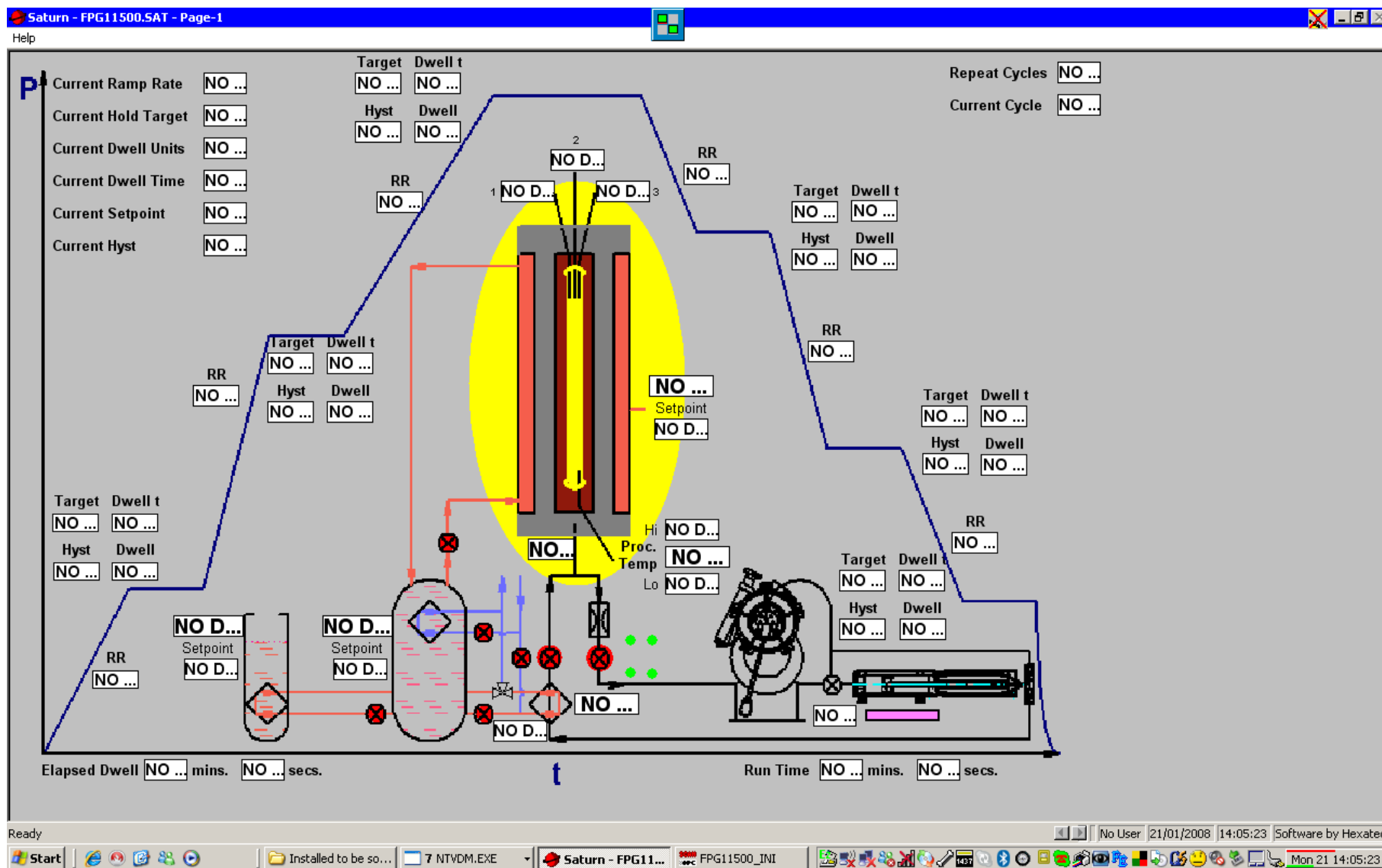
## SFP 800 MPa 5 L

- MEDIDAS CÁMARA INTERIOR = Ø110 mm x 550 mm (aprox. 5 L)
- MÁXIMA PRESIÓN TRABAJO = 800 MPa
- MÁXIMA TEMPERATURA DEL PROCESO 130 °C
- MÁXIMA TEMPERATURA DE PRECALENTAMIENTO 110 °C
- MÍNIMA TEMPERATURA DEL PROCESO -20 °C
- TERMOPARES REMPLAZABLES Y POSICIONABLES 3
- CONTROL Y PROGRAMACIÓN DEL PROCESO AUTOMATIZADO
- RECOGIDA CONTÍNUA DE DATOS DEL PROCESO
- PREACONDICIONAMIENTO MUESTRAS EN BAÑO (110 °C / -20°C)
- APERTURA RÁPIDA Y SENCILLA
- VALVULAS DE DESCOMPRESIÓN POR ETAPAS RÁPIDAS Y MANUAL
- ACONDICIONAMIENTO TÉRMICO INDEPENDIENTE
  - Cámara del Cilindro
  - Pre-llenado y línea de llenado de alta presión
  - Pre-acondicionamiento de las muestras

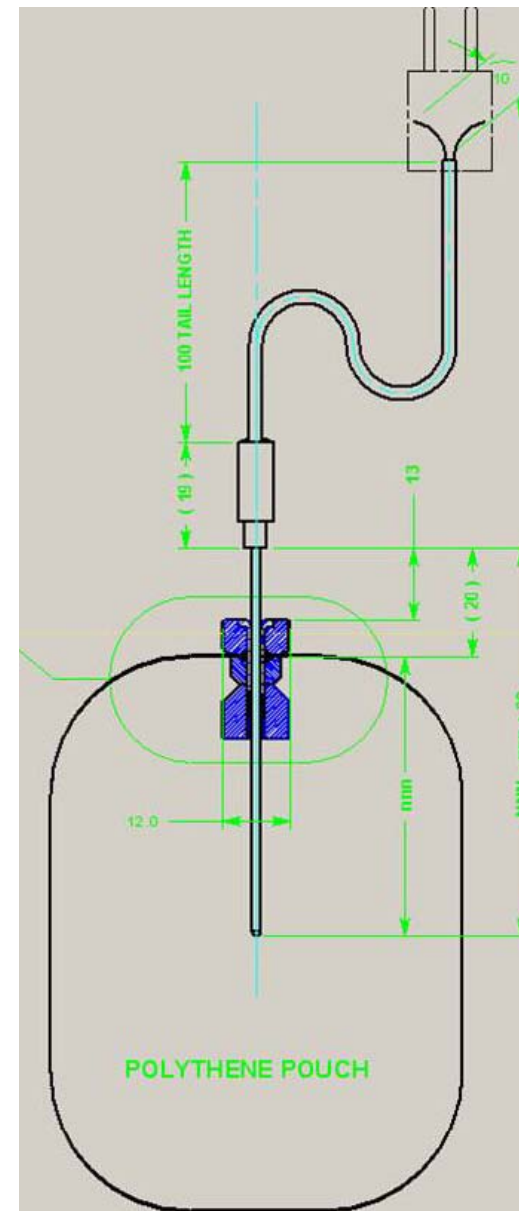
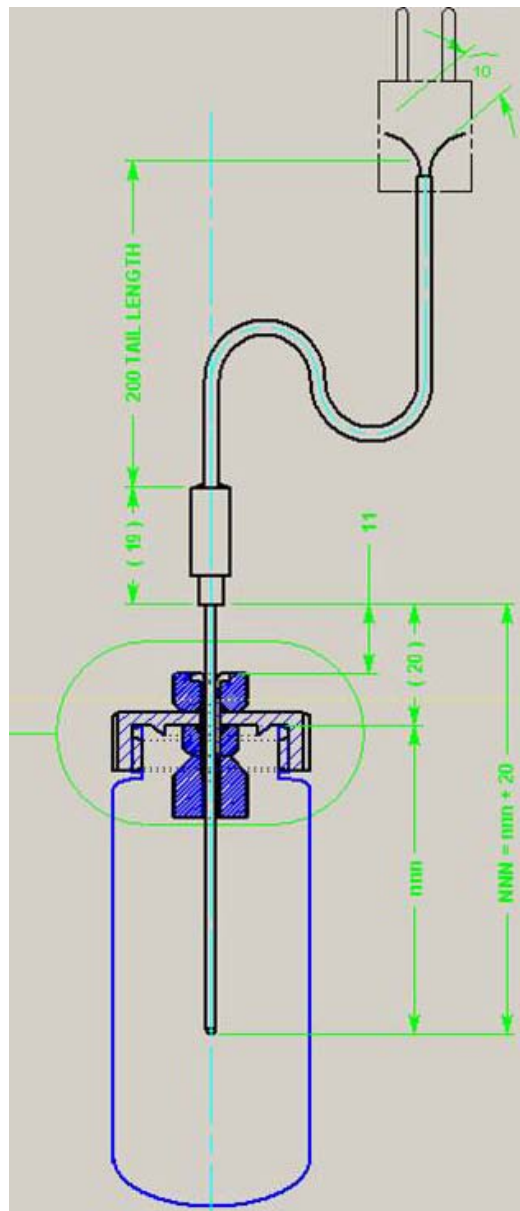
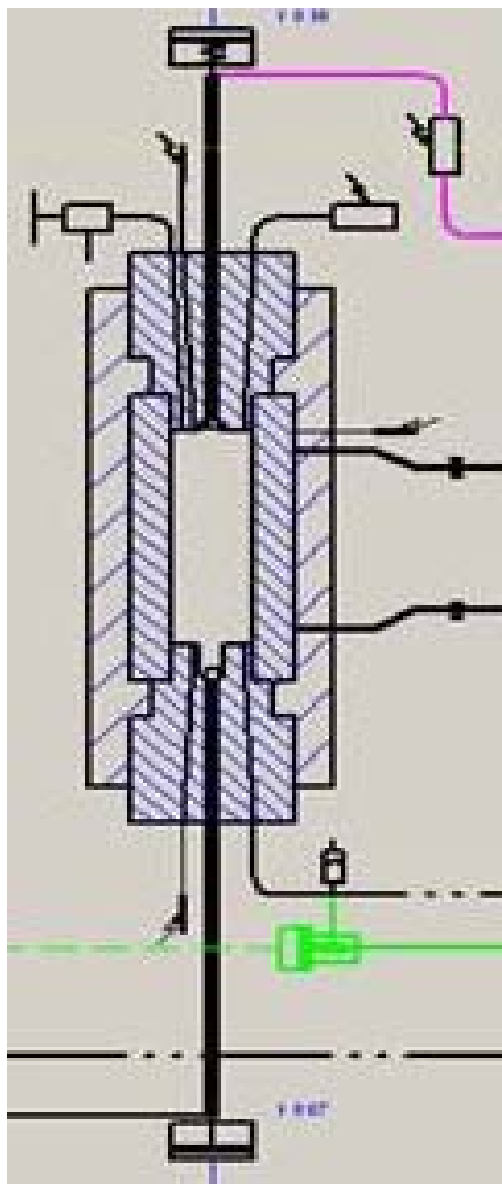
## SFP 800 MPa 5 L

### Panel de Control con Pantalla Táctil

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Miraflores  
*Enero-2011*



# SFP 800 MPa 5 L





# SFP 800 MPa 5 L

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Miraflores  
Enero-2011



# SFP 800 MPa 5 L

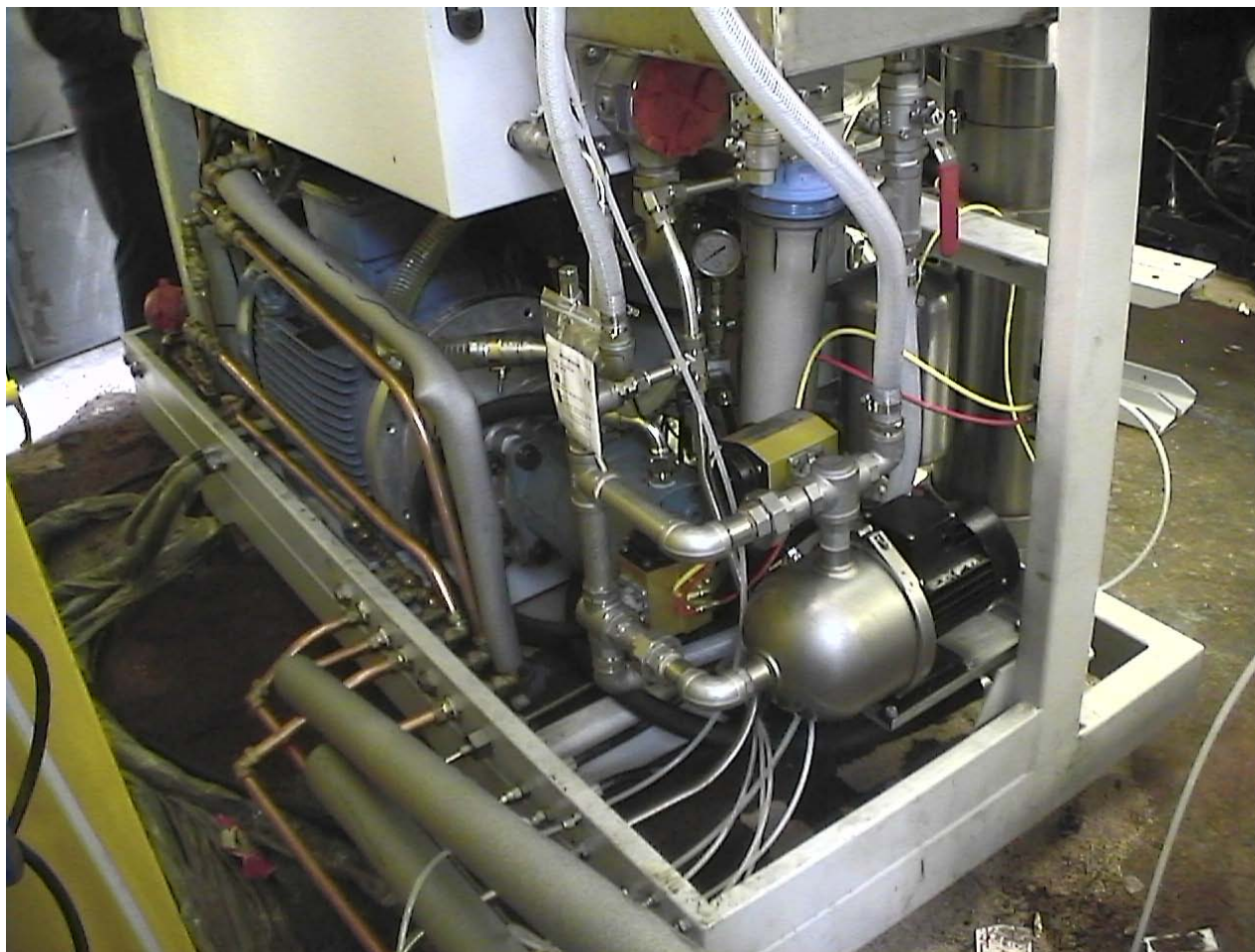
## Intensificador y Válvulas descompresión





# SFP 800 MPa 5 L Bomba de baja presión

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Miraflores  
Enero-2011



# SFP 800 MPa 5 L

## Depósitos de Fluidos Transmisores de Presión





# SFP 800 MPa 5 L

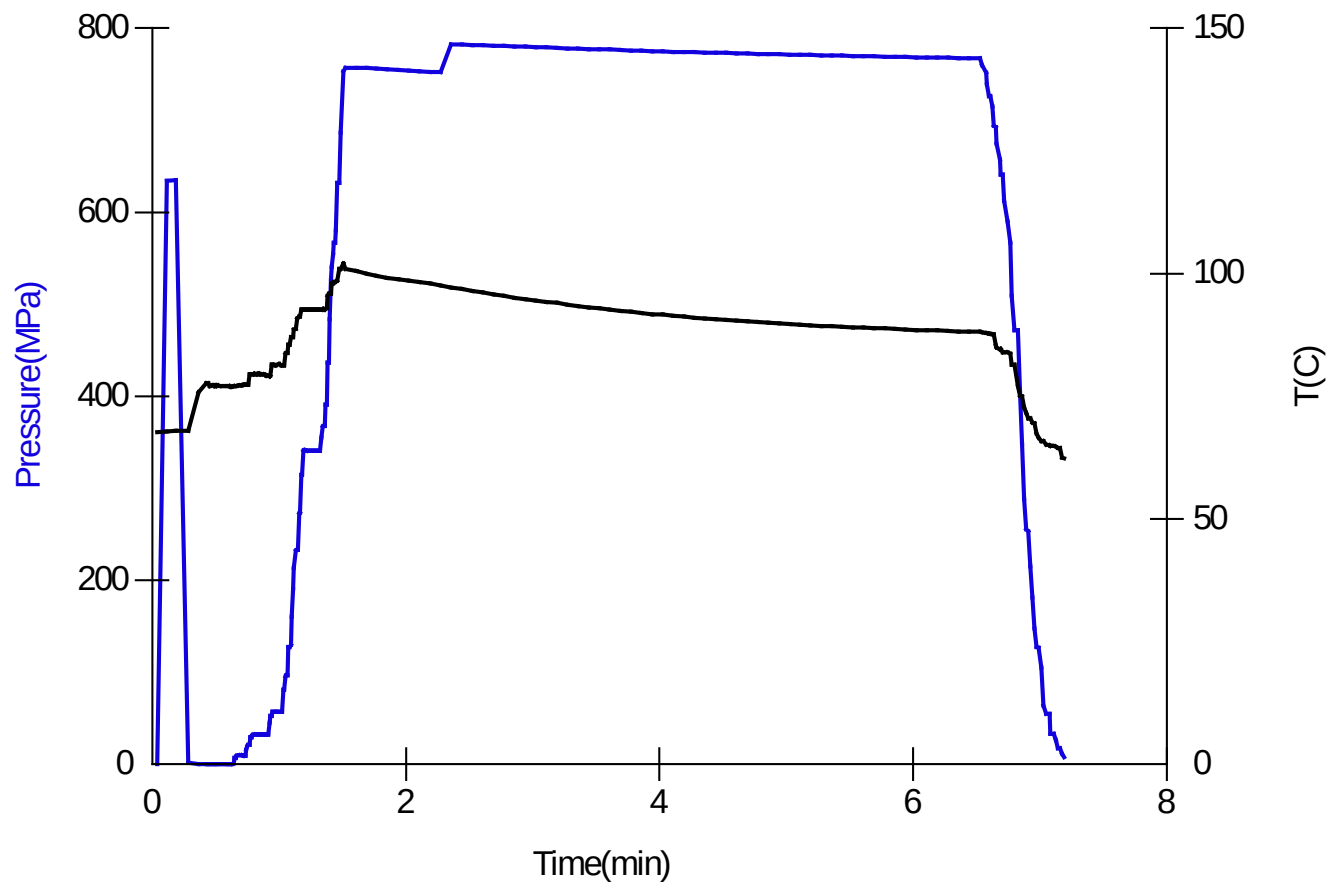
## Depósito Acumulador de Fluidos Térmicos

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Miraflores  
Enero-2011



# SFP 800 MPa 5 L

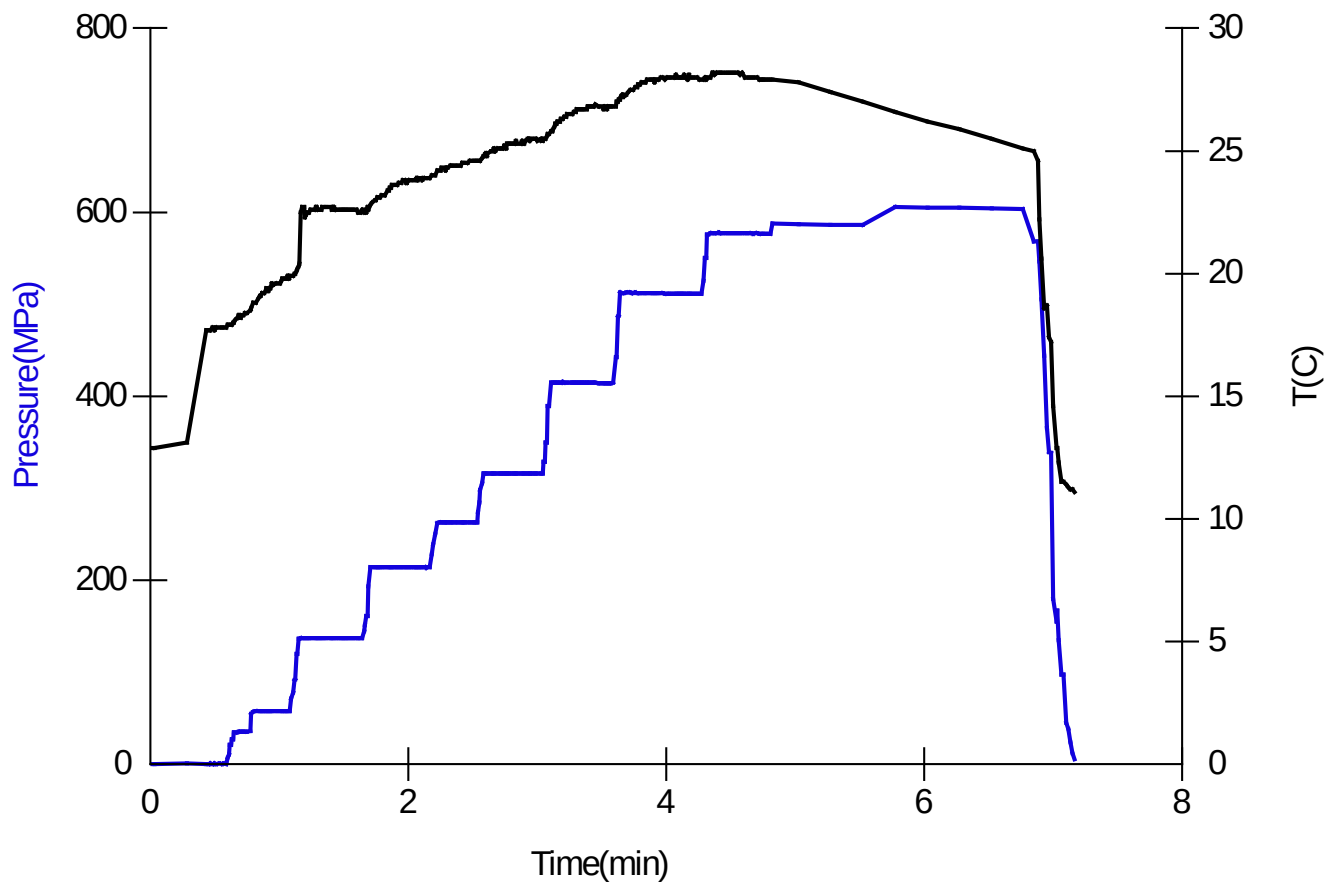
## Rápida SUBIDA y Rápida BAJADA



# SFP 800 MPa 5 L

## Lenta SUBIDA y Rápida BAJADA

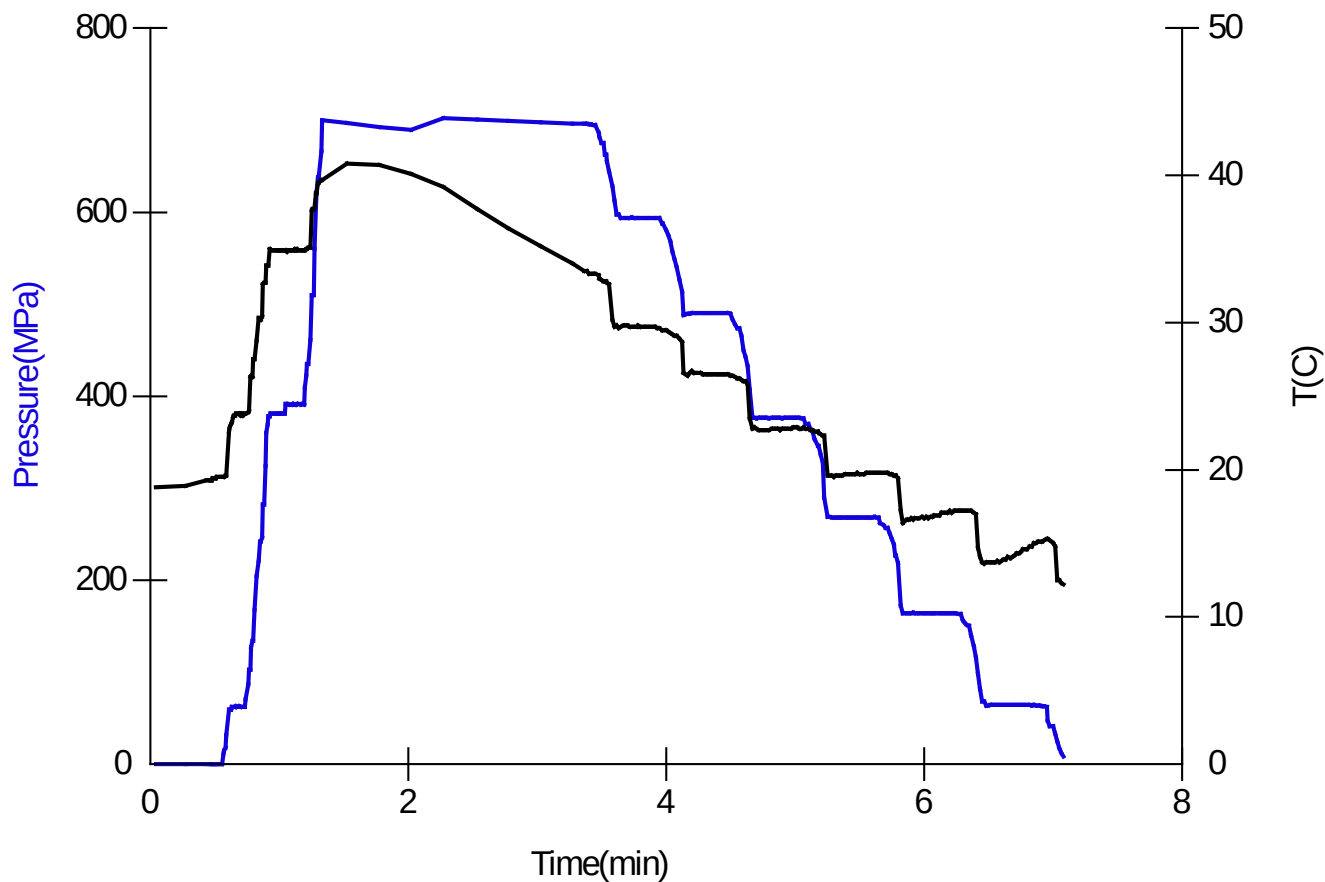
- 39 -  
Miraflores  
Enero-2011



# SFP 800 MPa 5 L

## Rápida SUBIDA y Lenta BAJADA

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Miraflores  
Enero-2011

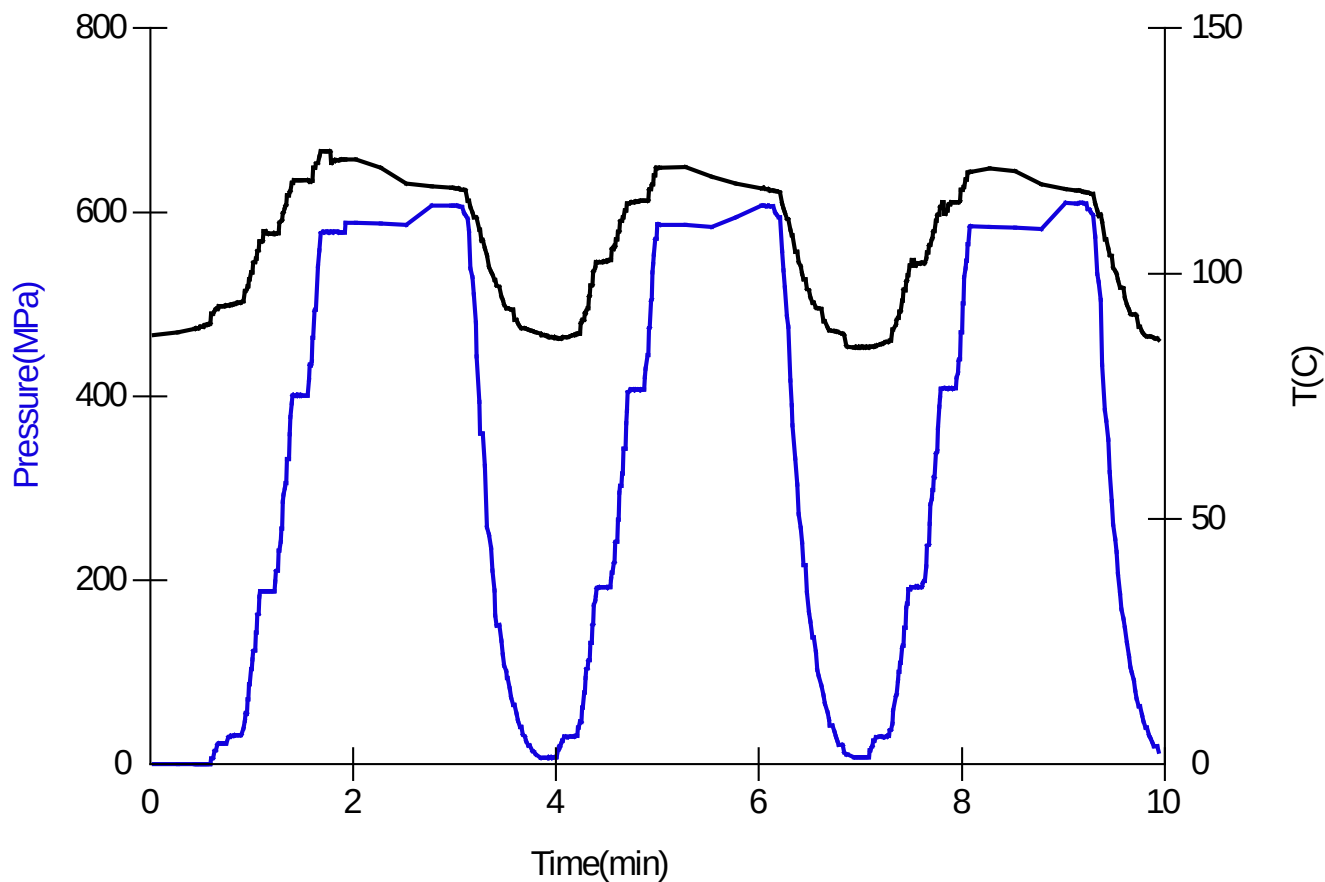




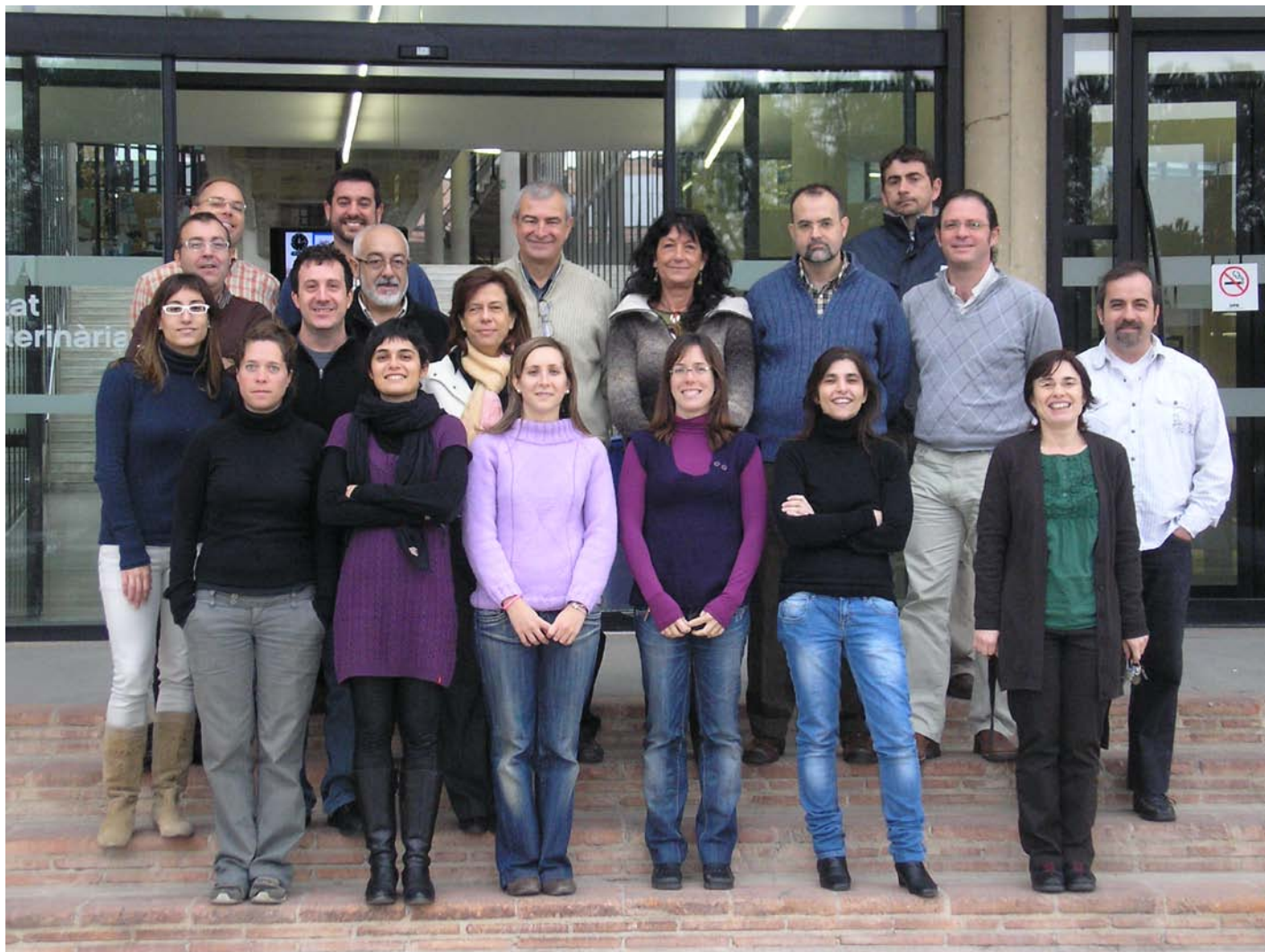
# SFP 800 MPa 5 L

## Multiciclos Simples

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Miraflores  
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# MUCHAS GRACIAS EN NOMBRE DEL CERPTA





MALTA-CONSOLIDER  
ESCUELA DE ALTAS PRESIONES

**MATERIA A ALTA  
PRESIÓN**  
**FUNDAMENTOS Y APLICACIONES**

2011

Eds: R. Valiente, F. Aguado, J.M. Menéndez y J.M. Recio

SERVICIO PUBLICACIONES UNIVERSIDAD DE OVIEDO  
SERVICIO PUBLICACIONES UNIVERSIDAD DE CANTABRIA

# 3 Bloques

- Teoría y Simulaciones
- Técnicas Experimentales
- Aplicaciones

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PRESIÓN  
FUNDAMENTOS Y APLICACIONES

2011

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Eds: R. Valiente, F. Aguado, J.M. Menéndez y J.M. Recio

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# Teoría y Simulaciones

- Termodinámica de las transiciones de fase sólido-sólido J. M. Recio, M. Flórez (UO)
- Mecanismos de las transiciones de fase sólido-sólido M. Flórez, J. M. Recio (UO)
- Simulaciones computacionales (I) J. Junquera, P. Aguado (UC)
- Simulaciones computacionales (II) A. Muñoz, P. Rodríguez (ULL)
- Simulación de reactividad química J. Contreras-García, L. Gracia, A. Beltrán, J. Andrés (UJI)

Eds: R. Vallerote, F. Aguado, J.M. Menéndez y J.M. Recio

Eds: R. Vallerote, F. Aguado, J.M. Menéndez y J.M. Recio

# Técnicas Experimentales

- **Técnicas de alta presión**  
V. García Baonza (UCM)
- **Fundamentos de la dispersión Raman**  
J. González (UC)
- **Espectroscopia óptica**  
F. Rodríguez (UC)
- **Técnicas de radiación sincrotrón**  
A. Segura, J. Pellicer, D. Errandonea (UV)
- **Función dieléctrica y propiedades ópticas**  
A. Segura (UV)
- **Propiedades de transporte y magnetismo**  
D. Errandonea (UV)

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PRESIÓN  
FUNDAMENTOS Y APLICACIONES

2011

Eds: F. Valier

M. Menéndez y J.M. Recio

# Aplicaciones específicas

- **Síntesis de materiales inorgánicos**

M. A. Alario (UCM)

- **Estados localizados en sólidos: defectos e impurezas**

F. Rodríguez (UC)

- **Tecnología de alimentos**

B. Guignon (IF-CSIC)

- **Microorganismos en condiciones extremas**

J. Saldo (UAB)

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- **Nanomateriales**

J. González (UC)

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Eds: R. Valiente, F. Aguado, J.M. Menéndez y J.M. Recio

- **Clatratos y otros materiales de interés planetario**

O. Prieto (IAB-CSIC)

- **Transiciones de fase en el manto terrestre**

F. Aguado (UC)

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# Resumen

- Introducción + 18 capítulos. 440 páginas

- Co-edición entre la U. de Oviedo y la U. de Cantabria

- Servicio de publicaciones: Coste cero. Difusión

2011

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# Trabajo por hacer

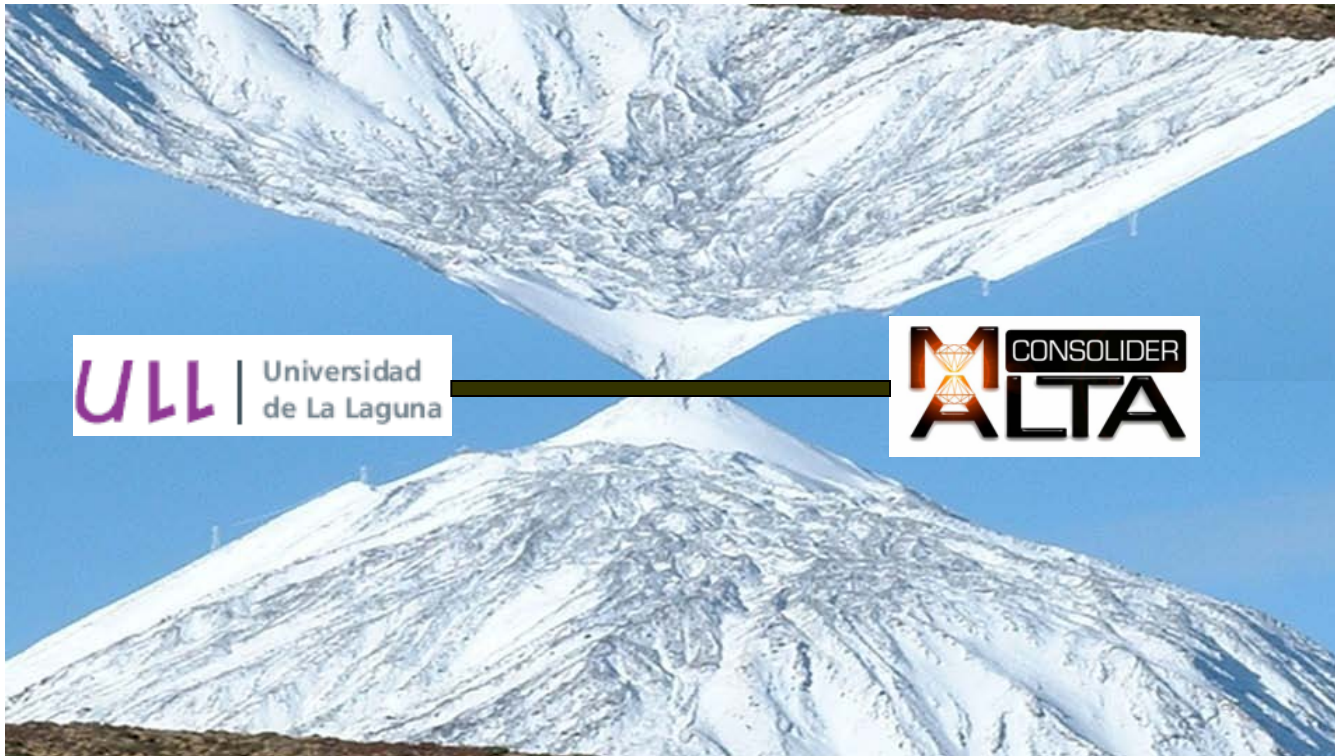
- Los autores deberían preocuparse de pedir los permisos necesarios (Copyright) sobre las figuras sobre las que no tienen derechos de autor. Mejora de figuras
- Revisión del texto
- Concurso de ideas para la portada del libro (abierto a todos los miembros de MALTA)
- Disponibilidad para la V Escuela de Alta Presión. Consejo Editorial (mediados de febrero).

Eds: R. Valiente, F. Aguado, J.M. Menéndez y J.M. Recio

Eds: R. Valiente, F. Aguado, J.M. Menéndez y J.M. Recio

# ***VENCUENTRO DE ALTA PRESIÓN***

***Dedicado al Prof. Dr. Manuel Cardona  
Max Planck Institut Stuttgart.***



***Universidad de La Laguna  
Tenerife, Junio 2011***

## **Objetivos del Encuentro**

***Este será un encuentro interdisciplinar de grupos españoles dedicados a la aplicación de técnicas de Alta Presión en diferentes campos de la Ciencia y la Tecnología. Por tanto, este Encuentro no sólo está dirigido a investigadores, estudiantes de doctorado y licenciados, sino también a profesionales de la empresa privada interesados en conocer las potenciales aplicaciones de las Altas Presiones en procesos industriales. Se impartirán conferencias a cargo de especialistas del campo y habrá una sesión de poster para presentar resultados científicos.***

***En el V Encuentro de Altas Presiones se dará una visión actual sobre esta técnica en diferentes campos de la Geología, la Física o la Química como***

- Propiedades físico-químicas del manto o del núcleo de la Tierra.***
- Síntesis de nuevos materiales.***
- Reacciones químicas bajo condiciones extremas.***
- Nanomateriales y nanotecnología.***
- Estudio de la materia en grandes instalaciones de radiación sincrotrón o de neutrones.***
- Correlación entre estructura y propiedades ópticas, eléctricas o magnéticas en materiales.***
- Simulación computacional de materiales bajo condiciones extremas.***
- Altas presiones en tecnología de alimentos.***
- Astrobiología. Agua y clatratos.***

***Como complemento se llevarán a cabo algunas sesiones prácticas de empleo de las celdas de yunque de diamante en el estudio de propiedades ópticas de sólidos luminiscentes a alta presión y de simulación con ordenador de estructuras sólidas. RECONOCIDO CON 3 CREDITOS DE LIBRE ELECCIÓN POR LA FAC. FÍSICA (ULL)***

### **Comité Científico**

***Dr. Alfredo Segura (U. Valencia)***  
***Dr. Alfonso Muñoz (ULL)***  
***Dr. Valentín García Baonza (UCM)***  
***Dr. Fernando Rodríguez (UCAN)***  
***Dr. José Manuel Recio (U. Oviedo)***  
***Dr. Buenaventura Guamis (UAB)***  
***Dr. Pedro Sanz (CSIC)***

### **Comité Organizador ULL**

***Dr. Alfonso Muñoz González***  
***Dr. Víctor Lavín della Ventura***  
***Dra. Plácida Rodríguez Hernández***  
***Dr. Ulises Rodríguez Mendoza***  
***Dr. Javier López Solano***  
***Lcdo. Sergio Fabián León Luis***



## ASPECTOS A DISCUTIR

- ¿Fechas finales? **27 Junio a 1 de julio**

### -Financiación.

ULL: **4.400 € + ¿1.200 €?**

MICINN: **Acciones Complementarias**

**Modalidad A: organización de jornadas científico-técnico. 14 abril.**

**Prioridad: internacionalidad, >100 asistentes, promoción año de la Química.**

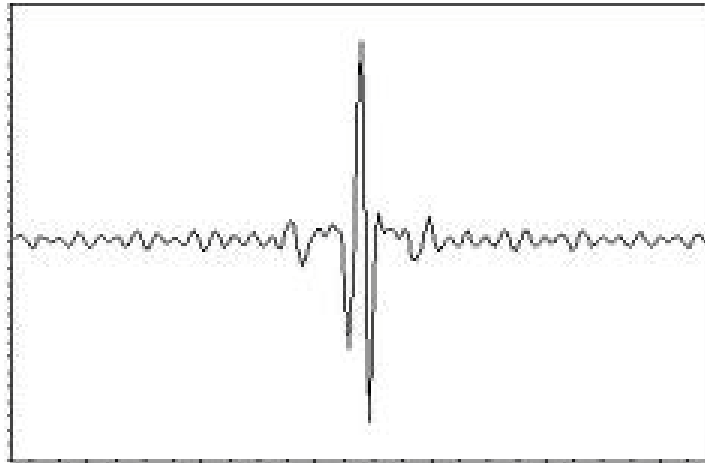
**SPONSORS: Oxford Diffraction, EasyLab, SIE, Cabildo de Tenerife, Hyperbaric...**





| LUNES                         | MARTES                        | MIÉRCOLES                  | JUEVES                                | VIERNES                          |
|-------------------------------|-------------------------------|----------------------------|---------------------------------------|----------------------------------|
| MAÑANA<br>4 charlas<br>45 min | MAÑANA<br>4 charlas<br>45 min | <b>EXCURSIÓN<br/>TEIDE</b> | MAÑANA<br>4 charlas<br>45 min         | <b>MONOGRÁFICO<br/>ALIMENTOS</b> |
| TARDE<br>Prácticas            | TARDE<br>Prácticas            | <b>EXCURSIÓN<br/>TEIDE</b> | TARDE<br>Prácticas<br><br><b>CENA</b> | <b>DESPEDIDA</b>                 |

# High Pressure FTIR spectroscopy setup and future development in new FTIR spectrometer



**Vinod Panchal**  
**Universidad De Valencia**

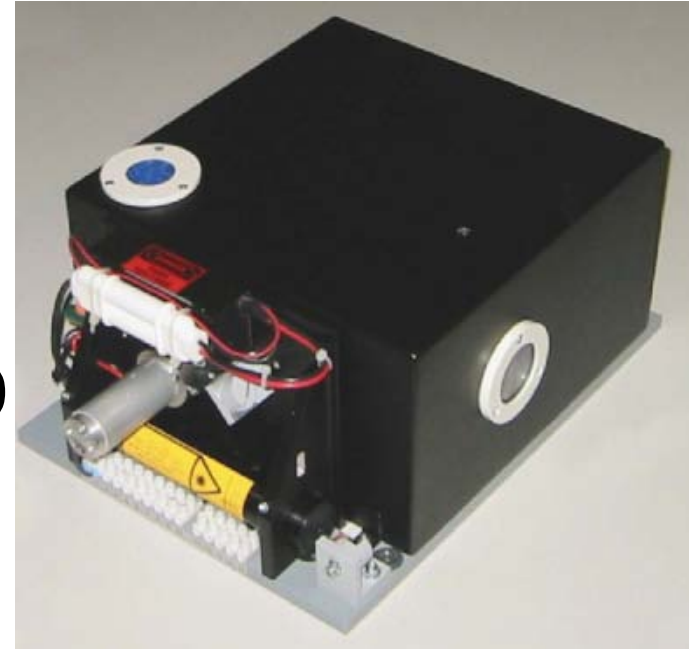
# **plan of Talk**

*Issues to be discussed*

- ❖ High pressure FTIR Setup
- ❖ Preliminary results
- ❖ Over view of new FTIR system (IFS 125HR )  
and plans of future developments

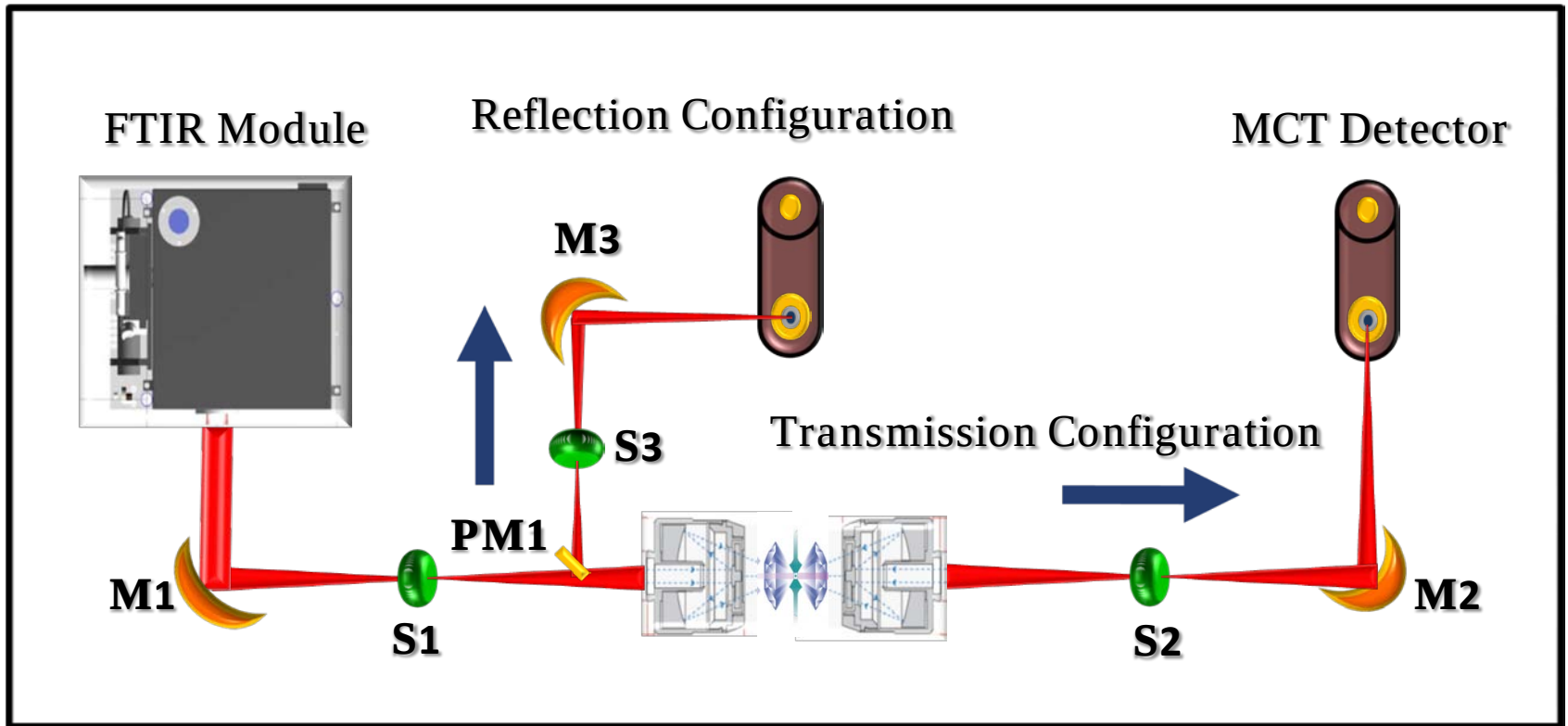
# TEO 400 FTIR MODULE

- ❖ Interferometer : Michelson Type
- ❖ Spectral range : 400-4000  $\text{cm}^{-1}$
- ❖ Resolution : 4 $\text{cm}^{-1}$  (0.5  $\text{cm}^{-1}$  option)
- ❖ Beam Splitter : KBr
- ❖ Reference Laser : He-Ne 633nm
- ❖ Source : Globar (SiC, 1200 °C)



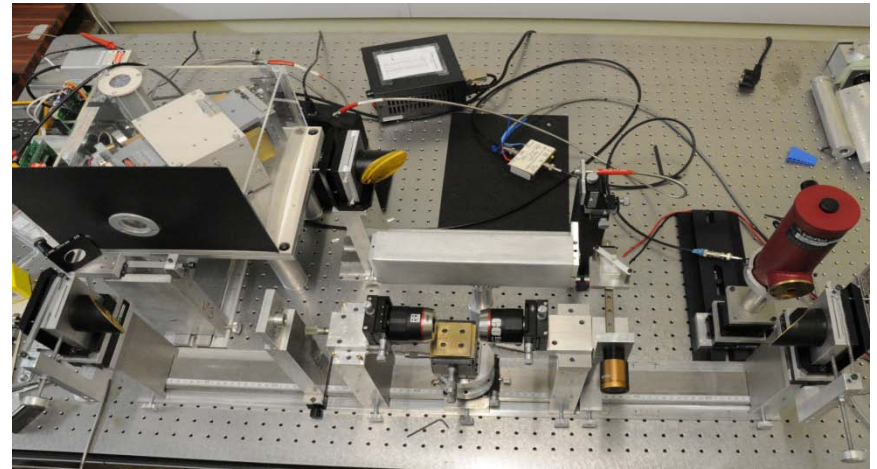
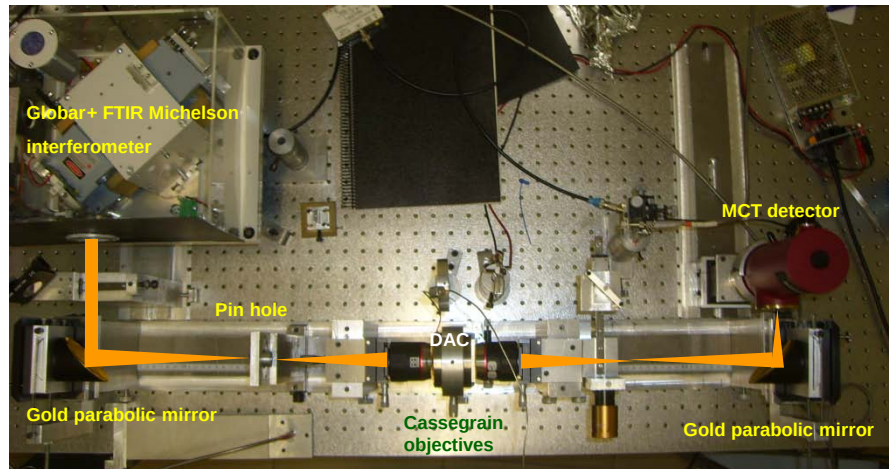


# Optical Layout of HP-FTIR Setup

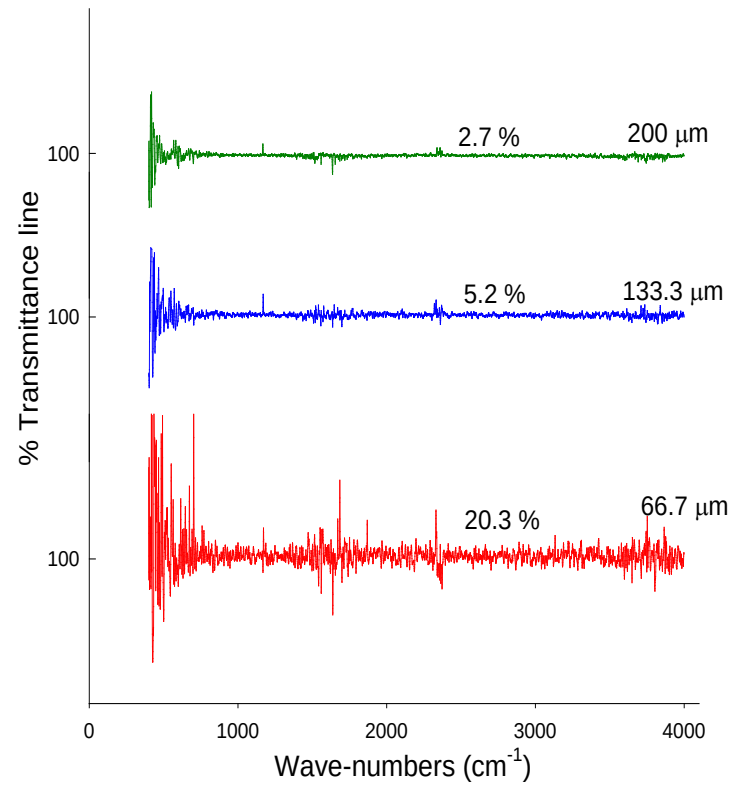


*Optical lay out of high pressure FTIR setup*

# HP-FTIR Setup



*Photograph of actual high pressure FTIR setup*



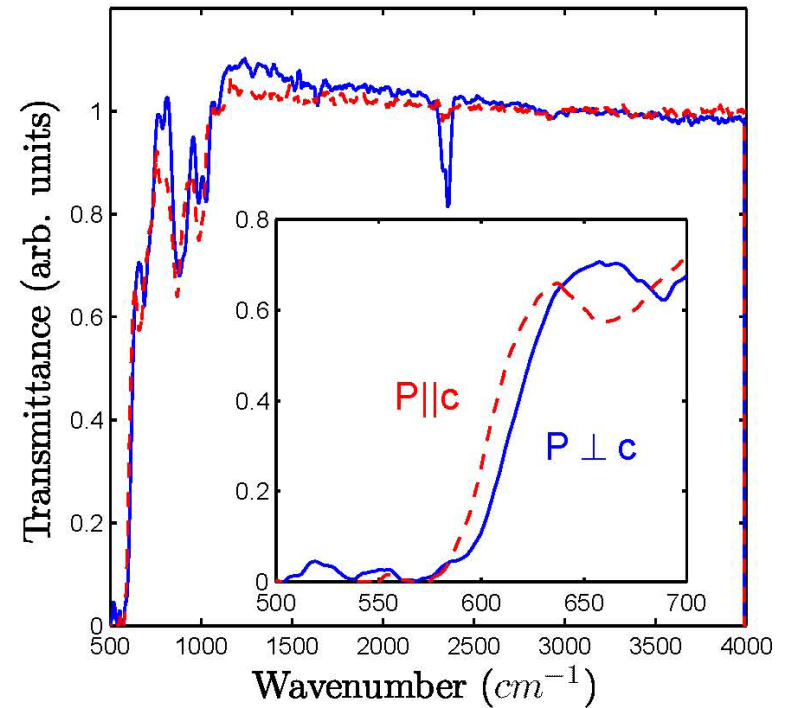
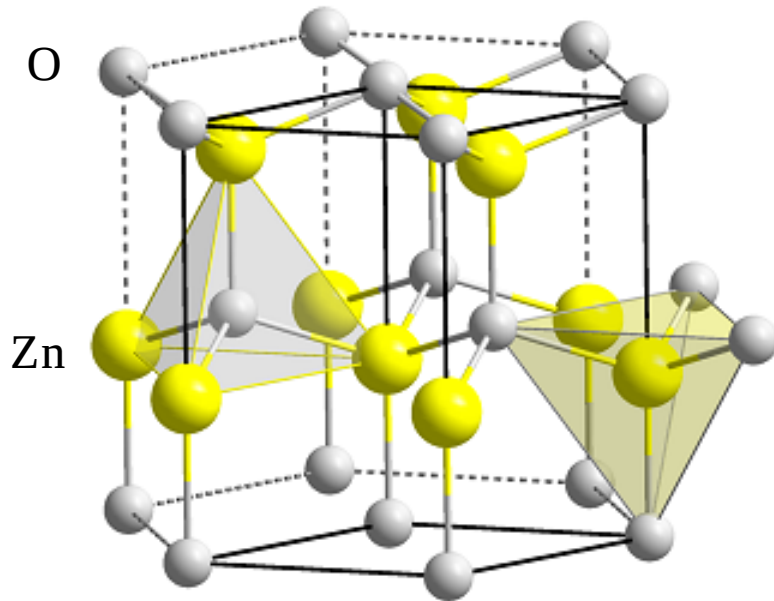
*Peak-to-peak noise for three different apertures*

# Experiments

- ❖ Phonon Absorption
- ❖ Free Carrier Absorption
- ❖ LO-TO Structure
- ❖ Plasma Reflection
- ❖ Refractive index



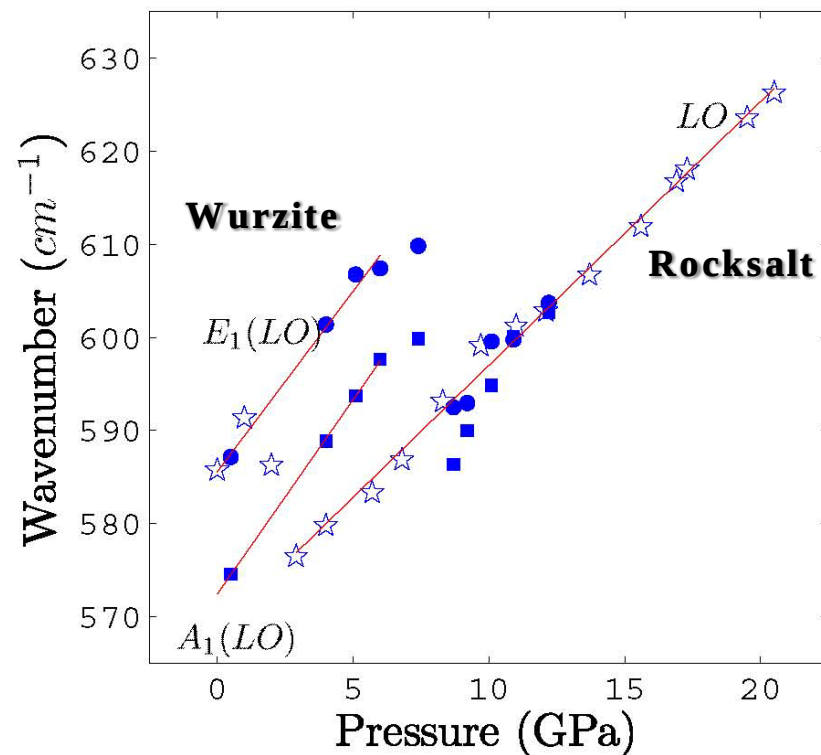
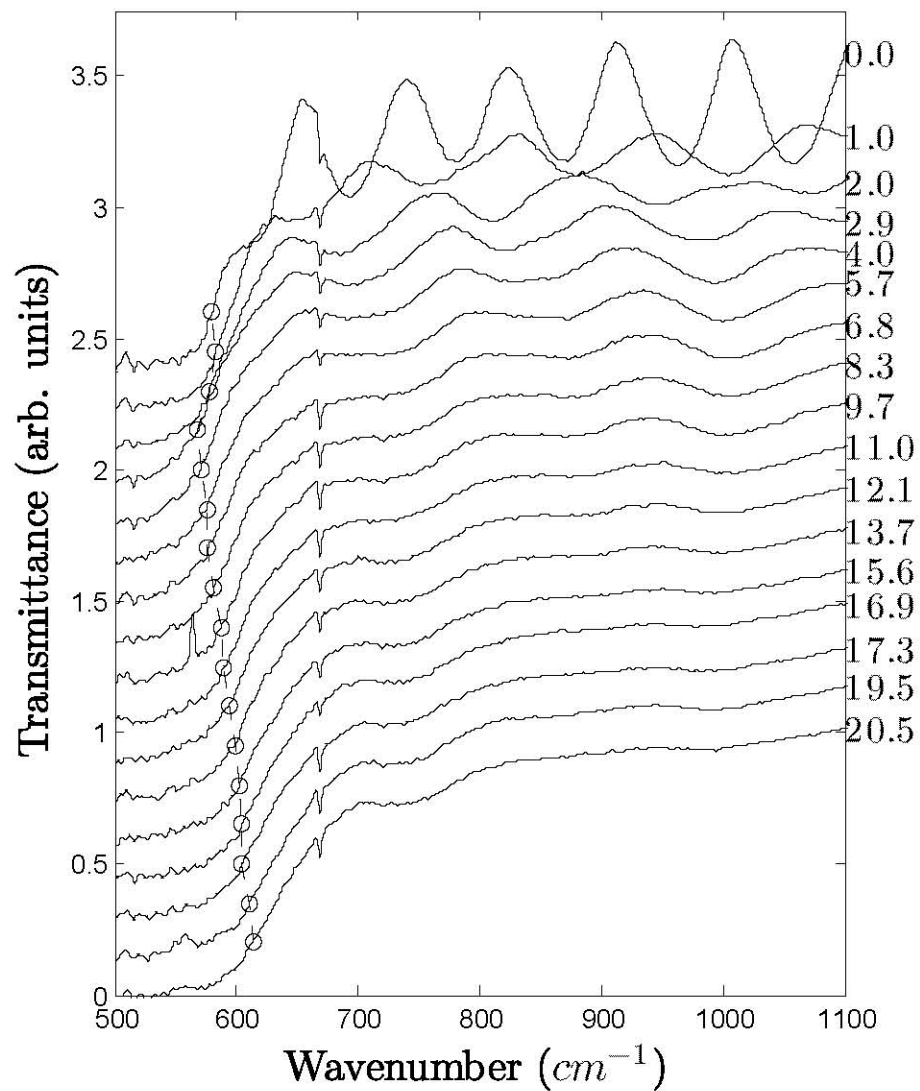
# Wurtzite to Rocksalt transition in ZnO



Wurtzite ZnO crystallizes in  $P6_3mc$  space group ( $C_{6v}^4$  point group)  $Z=2$

$$\Gamma_{\text{opt}} = A_1 + 2B_1 + E_1 + 2E_2$$

*Transmission spectra of Wurtzite ZnO in parallel and perpendicular polarisation. The red line corresponds to  $A_1(\text{LO})$  mode and blue line represents  $E_1(\text{LO})$  mode*

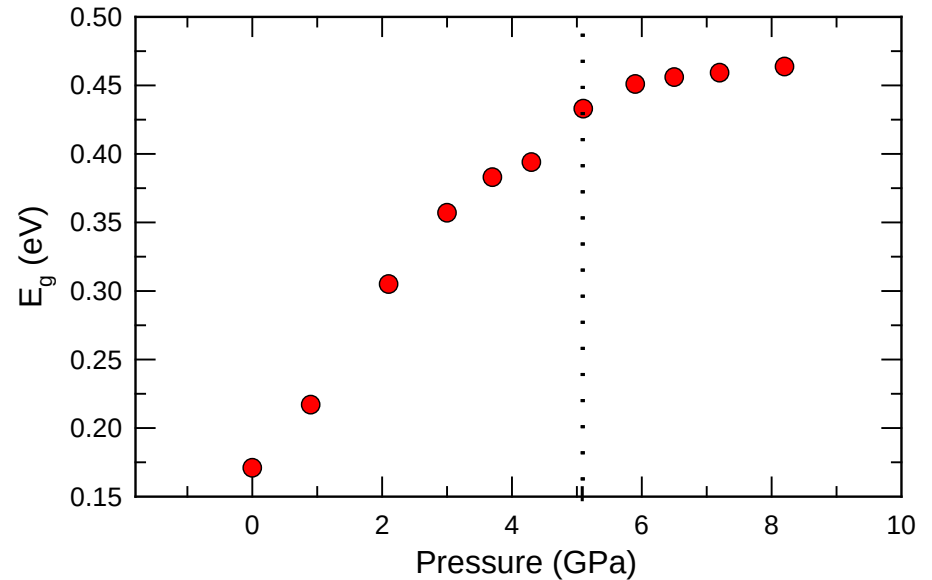
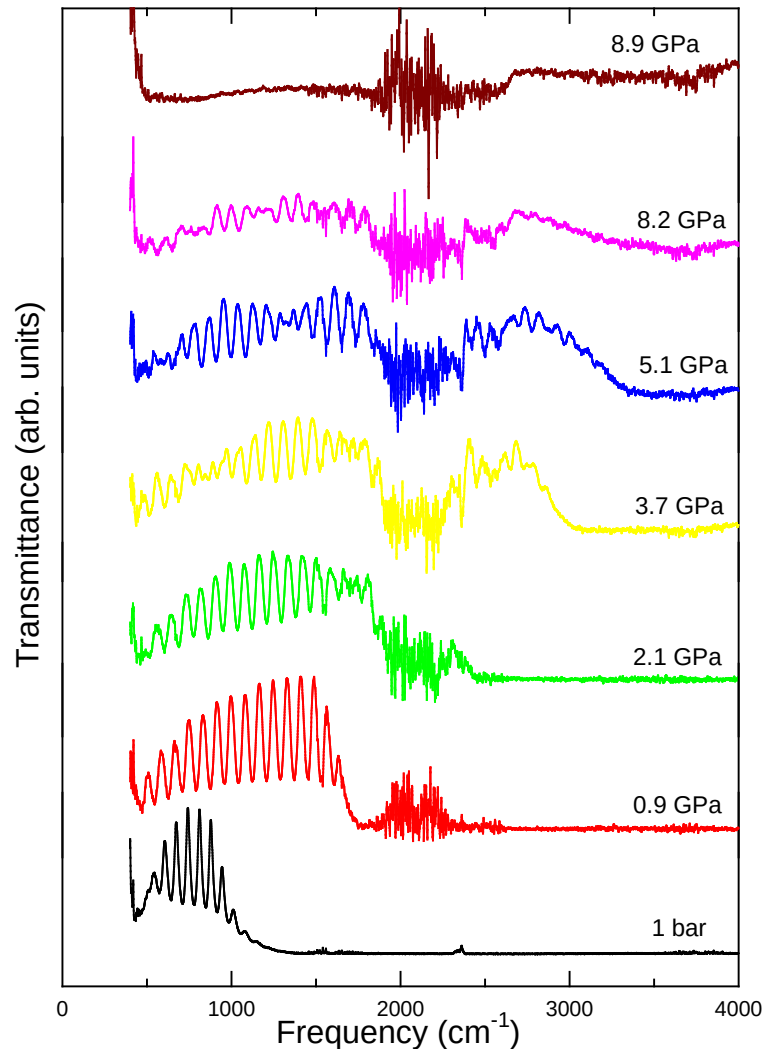


*Pressure dependance of IR modes. Blue filled circles indicates  $E_1(LO)$  mode and square indicates  $A_1(LO)$  modes in single crystal sample. Open star indicates LO mode of powder sample*

# Results

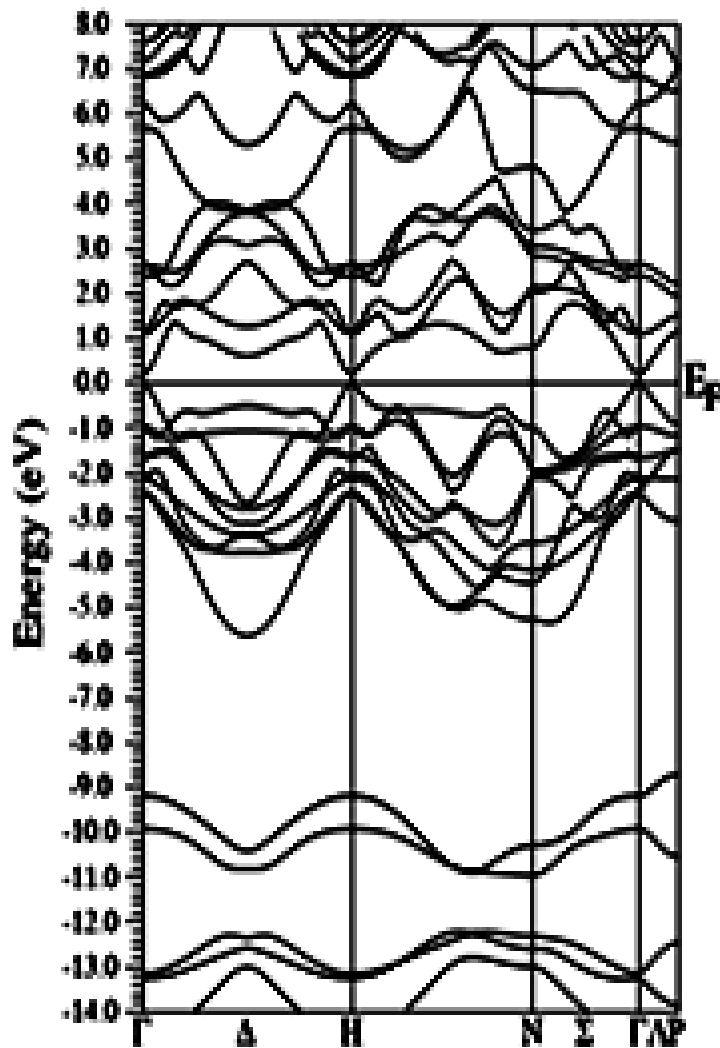
- ❖ The wurtzite to rocksalt transition occurs in ZnO at 7.5 GPa which is consistent with earlier reported values.
- ❖ The phonon wave number for wurtzite and rocksalt phase is given below,  
  
Wurtzite phase :  $A_1(\text{LO}) : 572.4 + 4.17P$      $E_1(\text{LO}) : 585.6 + 3.87P$   
Rocksalt phase :  $\text{LO} : 568.7 + 2.83P$   
(where frequency measured in  $\text{cm}^{-1}$  and pressure in GPa)
- ❖ On release of pressure the wurtzite phase is recovered below 4 GPa

# Topological insulator $\text{Bi}_2\text{Se}_3$ under pressure

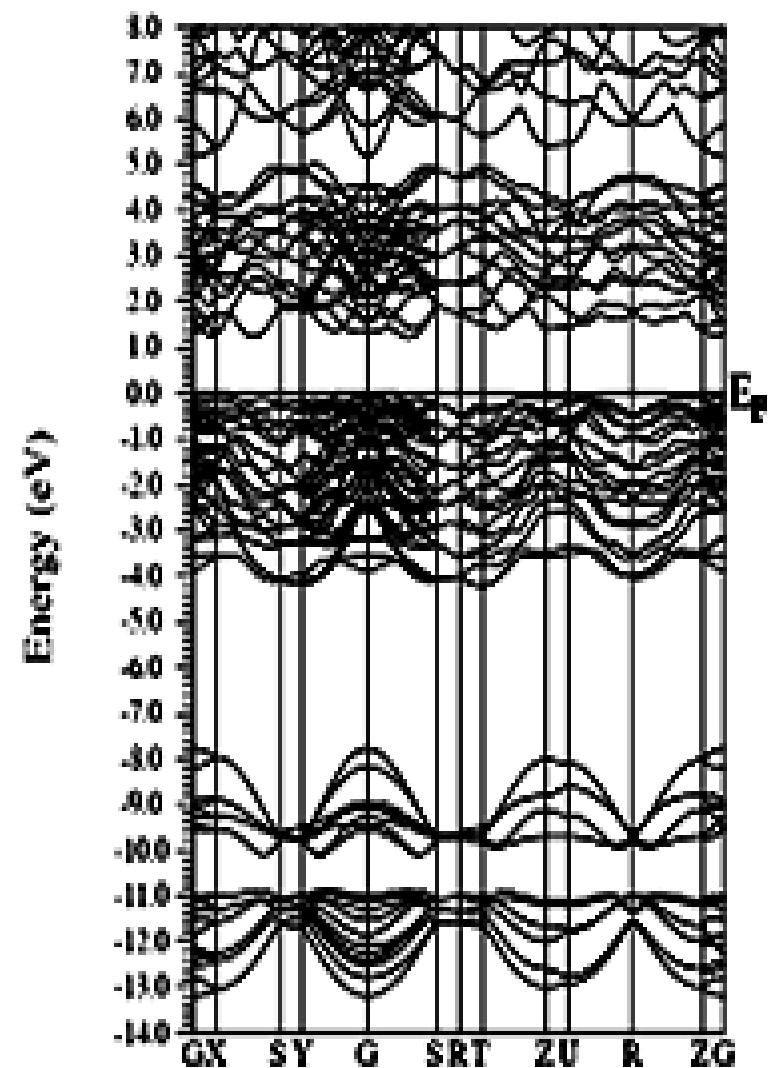


$1380 \text{ cm}^{-1} \rightarrow 0.171 \text{ eV}$





4 GPa  
  
 500 °C



Band Structure of Trigonal  
 $\text{Bi}_2\text{Se}_3$   $E_g = 0.175 \text{ eV}$

Band Structure of Orthorhombic  
 $\text{Bi}_2\text{Se}_3$   $E_g = 1.1 \text{ eV}$

# Results

- ❖ The energy gap in the topological insulator  $\text{Bi}_2\text{Se}_3$  increases linearly till 5.1 GPa with  $dE_g/dP = 0.05 \text{ eV/GPa}$ . Beyond this pressure the energy gap saturates indicating structural changes in  $\text{Bi}_2\text{Se}_3$ .
- ❖ Band structural calculations suggests trigonal to orthorhombic transition at 4 GPa and 500 °C with increase in band gap from 0.175 eV to 1.1 eV.
- ❖ However, at 8.9 GPa  $\text{Bi}_2\text{Se}_3$  becomes fully absorbing. This might be due to generation of defects in new high pressure phase and enhance free carrier absorption.

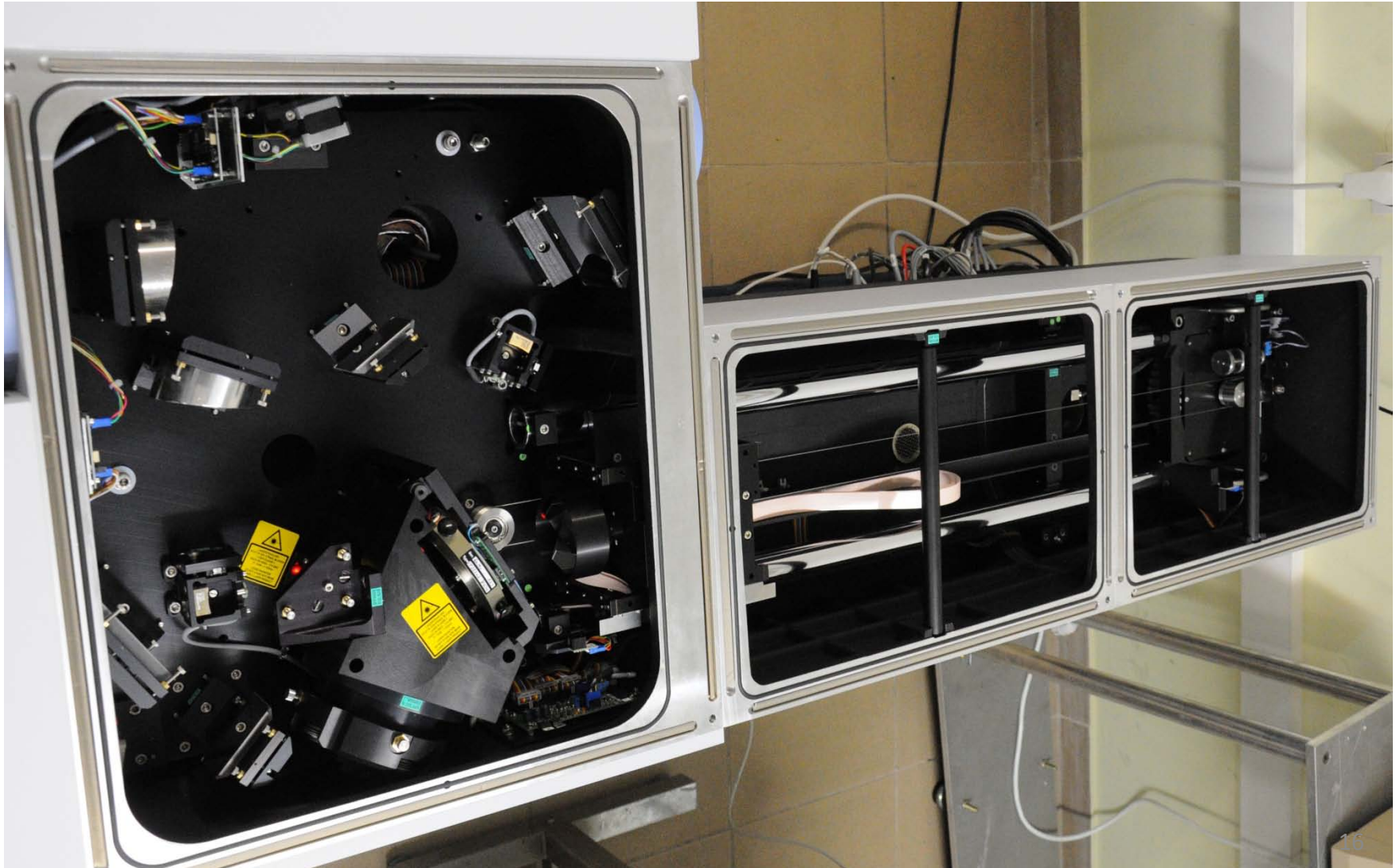
# IFS 125 HR Spectrometer



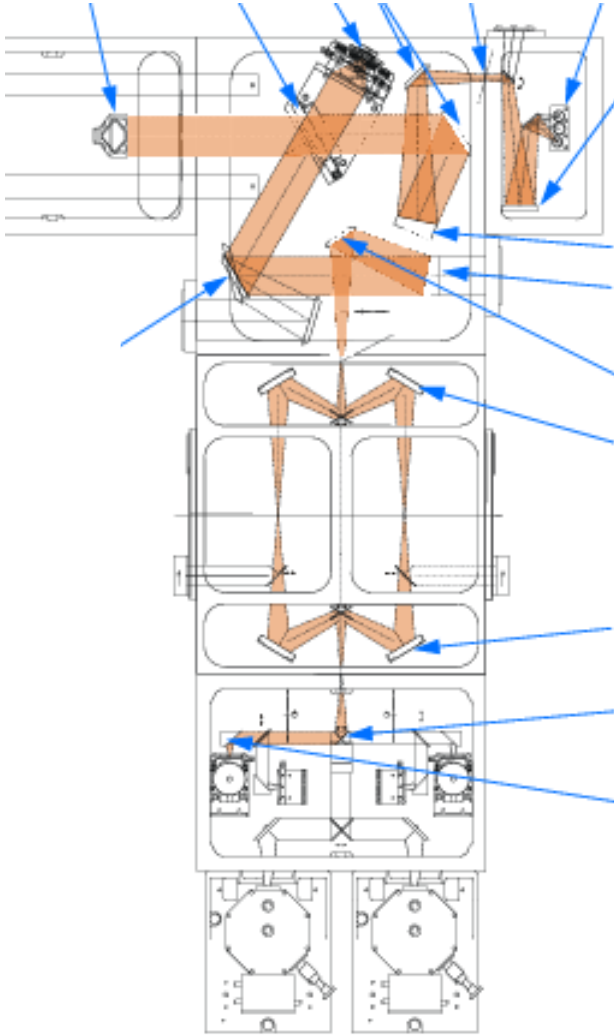
| Spectral Region | Frequency<br>(cm <sup>-1</sup> ) | Source Type  | Detector                     |
|-----------------|----------------------------------|--------------|------------------------------|
| FIR             | 20-1000                          | Mercury lamp | Si-Bolometer<br>or<br>DLATGS |
| MIR             | 600-6000                         | Globar (SiC) | MCT                          |



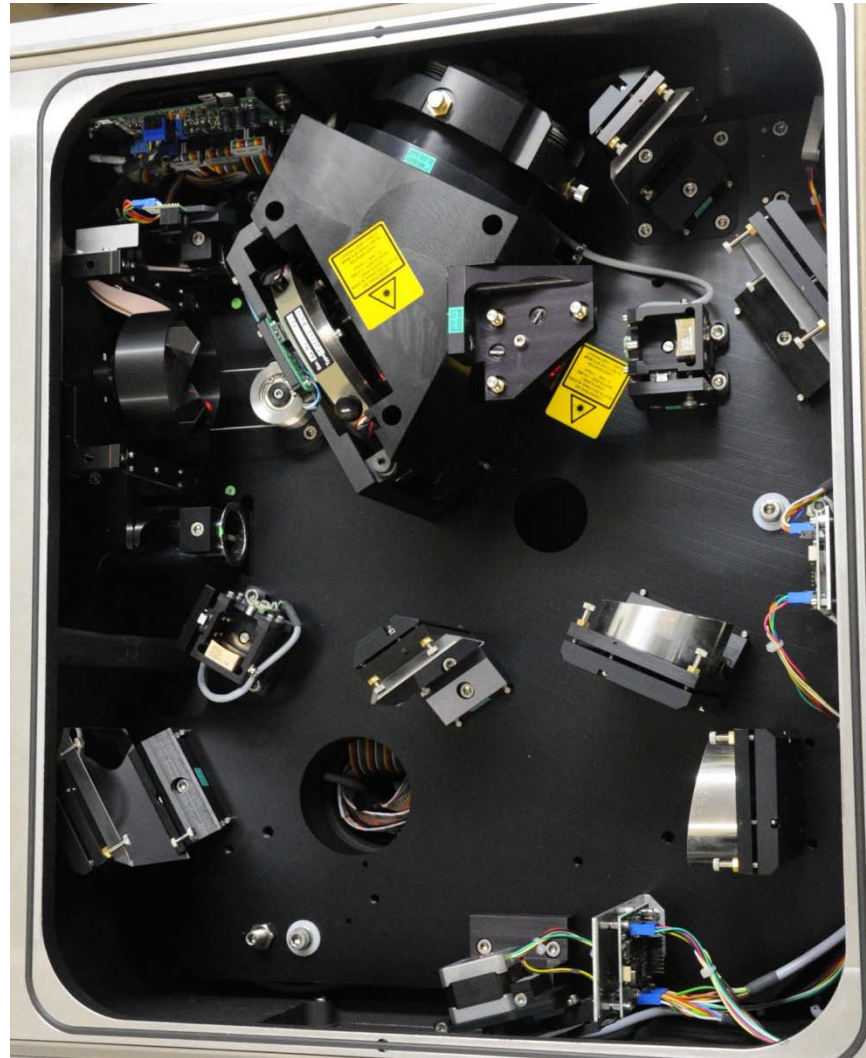
# Interferometer chamber with the extended mirror arm



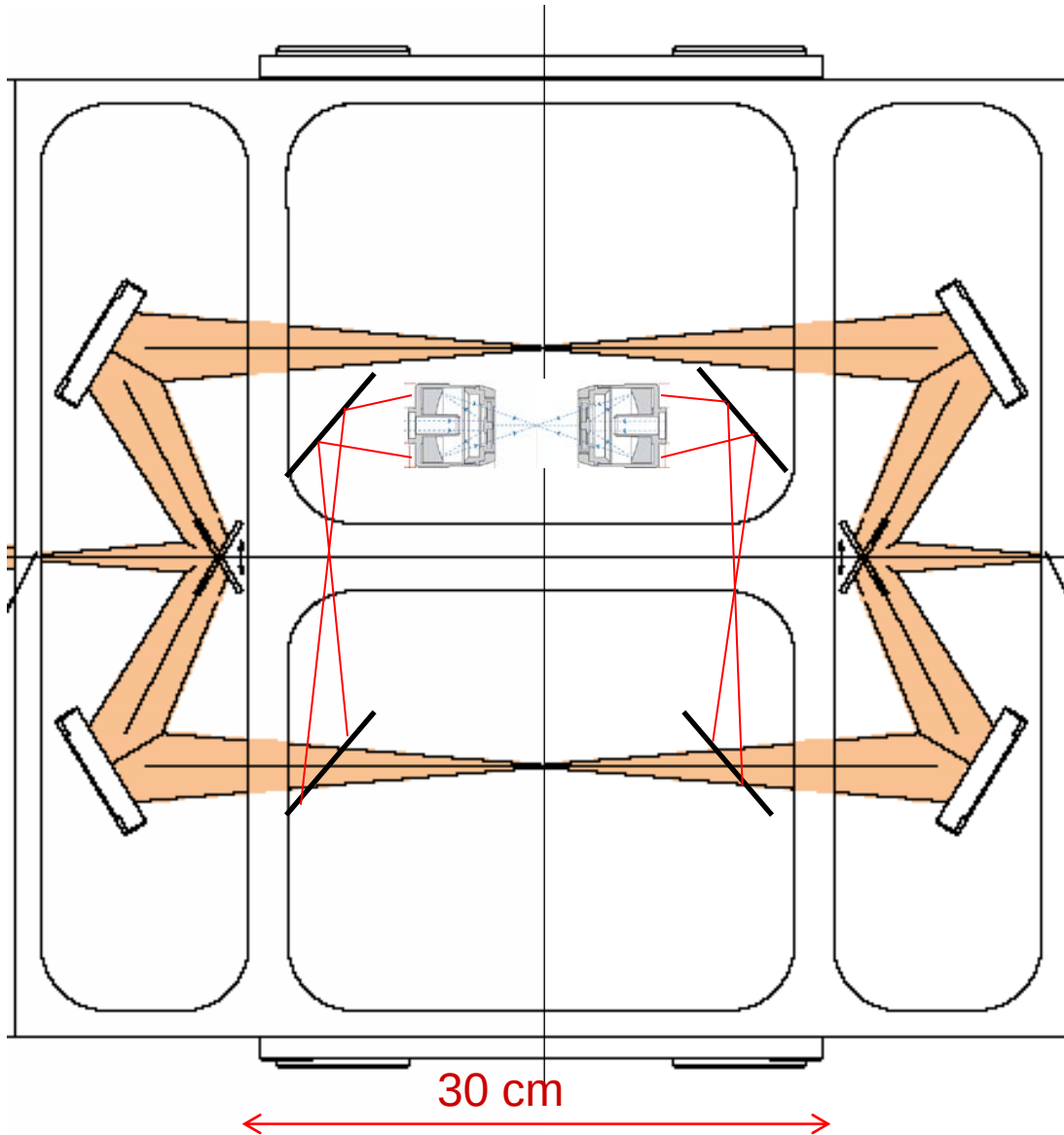
# Interferometer Chamber



*Optical lay out*



# Sample Chamber



**I sincerely thank**

**Prof. Alfredo Segura**

**Dr. Julio Pellicer**

**Technician : David Miguel  
&  
Diego Gomez**



**Thank You**

# X-ray Diffraction in MALTA

CrysAllisPro  
software



Automatic voltage  
regulators

- X-rays generator
- Goniometer

Chiller  
X-rays

At the moment, the diffractometer is mainly devoted to:

- (i) Room pressure single-crystal and powder experiments
- (ii) High-pressure powder experiments

### Usual tools

#### Sample centering:

- visually
- X-rays (macro)

#### Powder calibration vs single-crystal

- Powder standard  
(LaB<sub>6</sub>, CsI, etc...)
- Single-crystal standard (YLID)

#### Mechanical gasket drilling

- stainless steel

### Limitations

#### Instrumental resolution (broad peaks)

Complicated diffraction patterns when:

- Low symmetry structures
- Large lattice parameters
- $P > 20$  GPa

#### Space

- Other type of cells

#### DACs

- Aperture:  
HP single-crystal measurements
- Material

## **Procedure**

- Fill the form and send it to me ([dsantamaria@quim.ucm.es](mailto:dsantamaria@quim.ucm.es))
- If the expertise is available, the DACs should be loaded by the respective groups
- If not, the cells could be loaded in the UCM.
- The form, the manual of the diffractometer (to plan experiments) and the software (to analyse the results) are posted in the intranet of the MALTA webpage.



## Facility rules

- Groups with experimental expertise

Send:

- Application form
- New DACs loaded
- Some extra gaskets preindebted and drilled
- Small amount of sample

- Groups without experimental expertise

Send:

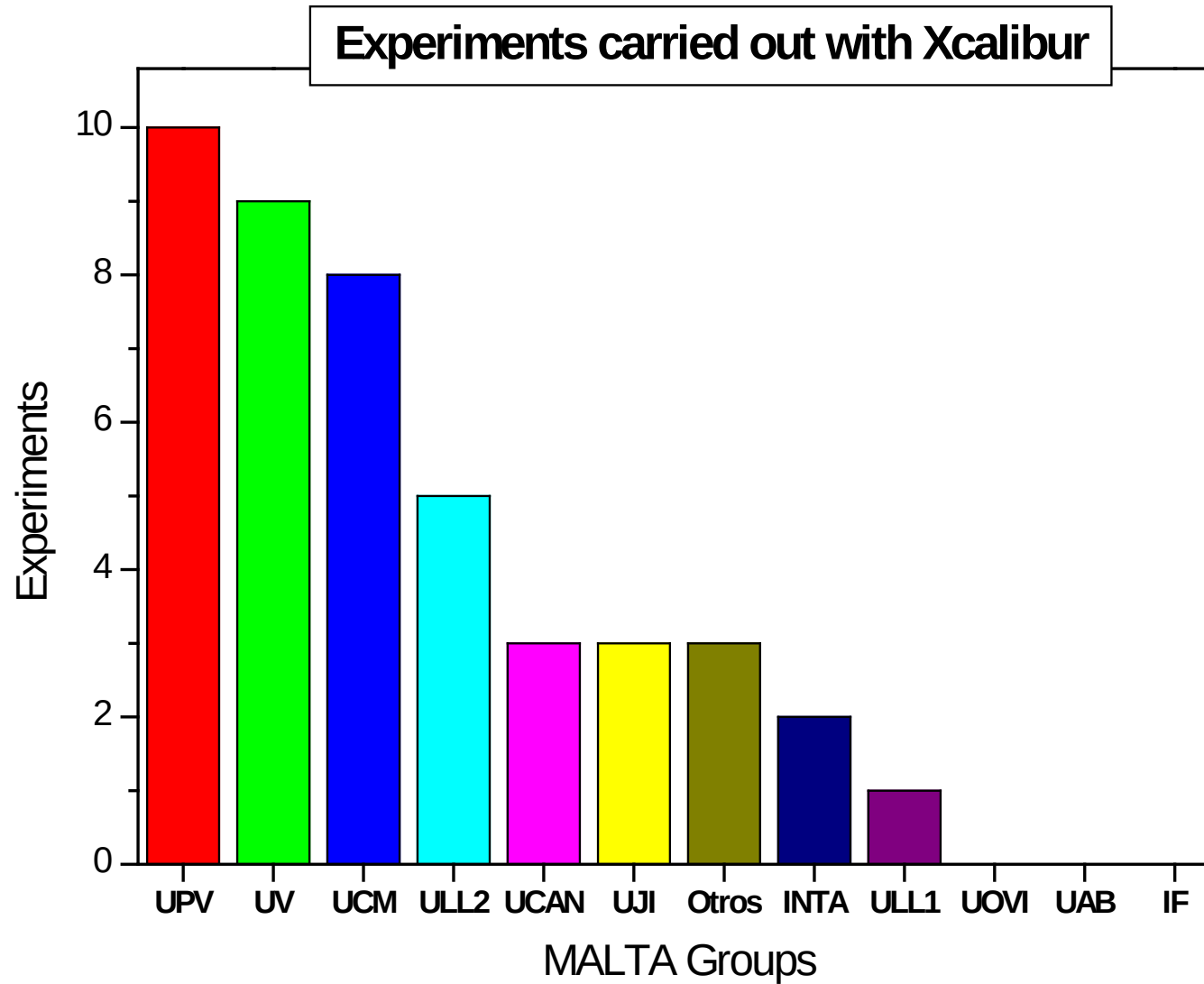
- Application form
- Small amount of sample

The DAC will be loaded in Madrid

- 8 MALTA-groups
- A total of 28 application forms received

- UCM: Graphite,  $\text{Rb}_2\text{S}$ ,  $\text{BaSO}_4$ ,  $\text{SiO}_2$ ,  $\text{Li}_2(\text{Co,Mn})\text{SiO}_4$
- ULL2:  $\text{TbPO}_4$ ,  $\text{HgWO}_4$ ,  $(\text{Mg,Cd,Mn})\text{In}_2\text{S}_4$
- UJI:  $\text{Ti}_{0.5}\text{Zr}_{0.5}\text{PbO}_3$ ,  $\text{BaSO}_4$ ,  $\text{SbPO}_4$
- UCAN: Nanoparticles  $\text{CdS}$  and  $\text{ZnO}$ ,  $\text{HoMnO}_3$
- UV:  $\text{TbPO}_4$ ,  $\text{HgWO}_4$ ,  $(\text{Pb,Cd})\text{MoO}_4$ ,  $(\text{Zr,Hf})\text{TiO}_4$ ,  $(\text{Nd,Ce})\text{VO}_4$ ,  
 $\text{BaSO}_4$
- ULL1:  $\text{Eu}_2(\text{MoO}_4)_3$
- UPV:  $\text{TbPO}_4$ ,  $\text{HgWO}_4$ ,  $(\text{Mg,Cd,Mn})\text{In}_2\text{S}_4$ ,  $(\text{Cd,Hg})(\text{Al,Ga})_2(\text{S,Se})_4$
- INTA:  $\text{FeCO}_3$ ,  $\text{CaCO}_3$
- Otros:  $\text{Pr}_2\text{Ru}_2\text{O}_7$ ,  $\text{CdCr}_2\text{O}_4$ ,  $\text{InVO}_3$

- 8 MALTA-groups



## Theoretical and experimental study of the structural stability of $\text{TbPO}_4$ at high pressures

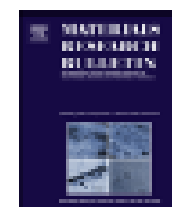
J. López-Solano,<sup>1,\*</sup> P. Rodríguez-Hernández,<sup>1</sup> A. Muñoz,<sup>1</sup> O. Gomis,<sup>2</sup> D. Santamaría-Pérez,<sup>3</sup> D. Errandonea,<sup>4</sup> F. J. Manjón,<sup>5</sup>  
R. S. Kumar,<sup>6</sup> E. Stavrou,<sup>7</sup> and C. Raptis<sup>7</sup>



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Materials Research Bulletin

journal homepage: [www.elsevier.com/locate/materresbu](http://www.elsevier.com/locate/materresbu)



## New high-pressure phase of $\text{HfTiO}_4$ and $\text{ZrTiO}_4$ ceramics

Daniel Errandonea<sup>a,1,\*</sup>, David Santamaría-Pérez<sup>b,1</sup>, Tatiana Bondarenko<sup>c</sup>, Oleg Khyzhun<sup>c</sup>

## High-pressure structural and lattice dynamical study of $\text{HgWO}_4$

F. J. Manjón,<sup>1,\*</sup> J. López-Solano,<sup>2</sup> S. Ray,<sup>1,†</sup> O. Gomis,<sup>3</sup> D. Santamaría-Pérez,<sup>4</sup> M. Mollar,<sup>1</sup> V. Panchal,<sup>5</sup> D. Errandonea,<sup>5</sup>  
P. Rodríguez-Hernández,<sup>2</sup> and A. Muñoz<sup>2</sup>



## High-pressure x-ray diffraction study of bulk and nanocrystalline $\text{PbMoO}_4$

D. Errandonea,<sup>1,a)</sup> D. Santamaria-Perez,<sup>2</sup> V. Grover,<sup>3</sup> S. N. Achary,<sup>3</sup> and A. K. Tyagi<sup>3</sup>

Acta Crystallographica Section B  
**Structural  
Science**

ISSN 0108-7681

Editor: Carolyn P. Brock

## High-pressure experimental study on $\text{Rb}_2\text{S}$ : Antifluorite to $\text{Ni}_2\text{In}$ -type phase transitions.

David Santamaria-Perez\*, Angel Vegas, Claus Muehle and Martin Jansen

## High-pressure X-ray diffraction study of $\text{CdMoO}_4$ and $\text{EuMoO}_4$

D. Errandonea<sup>1,†</sup>, D. Santamaria-Perez<sup>2</sup>, S. N. Achary<sup>3</sup>, A. K. Tyagi<sup>3</sup>, P. Gall<sup>4</sup>, and P. Gougeon<sup>4</sup>

Journal of Applied Physics. Accepted

## Maintenance costs

- X-ray tube replacement: **~ 4000 E**  
(every 1.5 years)
- Total care package  
(Oxford Diffraction) **~ 7000 E/year**

## Final Remarks

- Fully operational equipment (Proposals are welcome).
- Xcalibur as previous step to apply for synchrotron beamtime.
- HP single-crystal measurements to be developed.



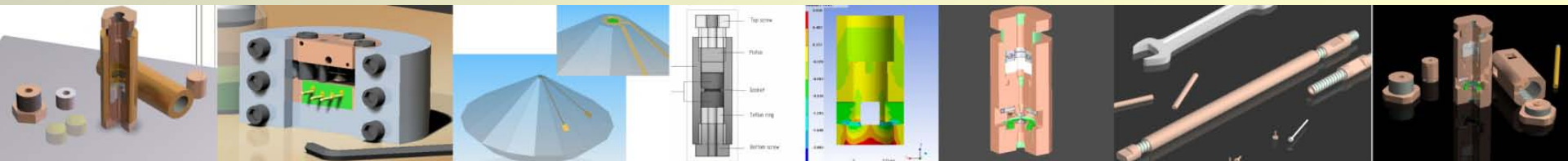
# *“Desarrollo en celdas de alta presión para medidas de propiedades magnéticas”*

*Javier Sánchez-Benítez*

Instituto de Ciencia de Materiales de Madrid (CSIC)

**QUIMAPRES**

Centre for Science at Extreme Conditions  
University of Edinburgh





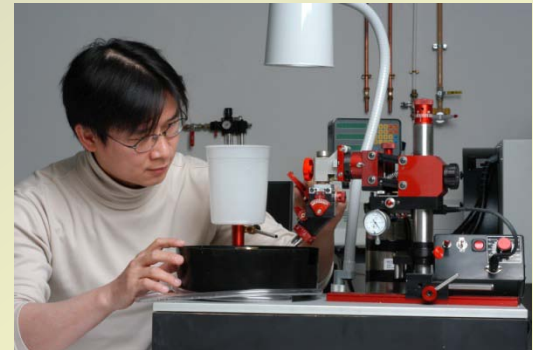
# Esquema

- Instrumentación
- Diseño de celdas y Simulaciones
- Celdas de alta presión para medidas de propiedades magnéticas:
  - long symmetric cell para materiales magnéticamente débiles;
  - electric plug cell para medir presión *in situ*;
  - microcell para medidas de hasta 2.2 GPa;
- Celda de diamantes para medida de transporte a bajas temperaturas

# *Instrumentación en CSEC*

## Maquinaria especializada:

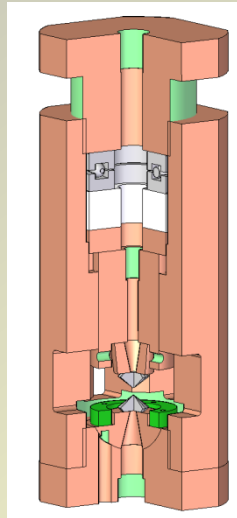
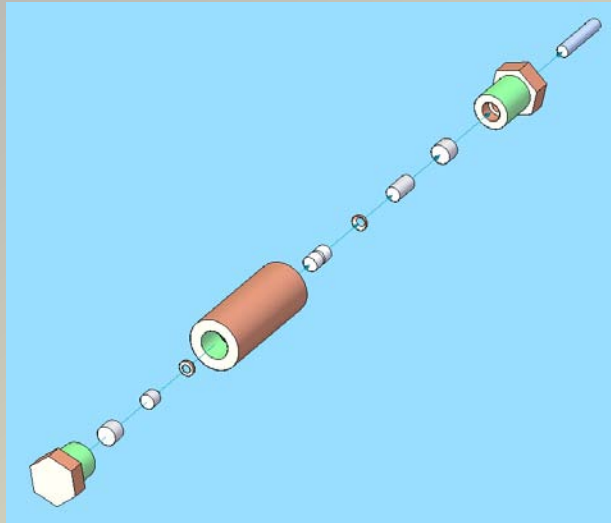
- **Honing machine**
- Precision cut-off and polishing machines
- **Gun drilling machine**
- Laser drilling, cutting and welding centre
- High-speed CNC lathe
- Diamond polishing and faceting machine
- Low current welding system
- **Programmable coil winding machine**
- **Rockwell hardness tester**



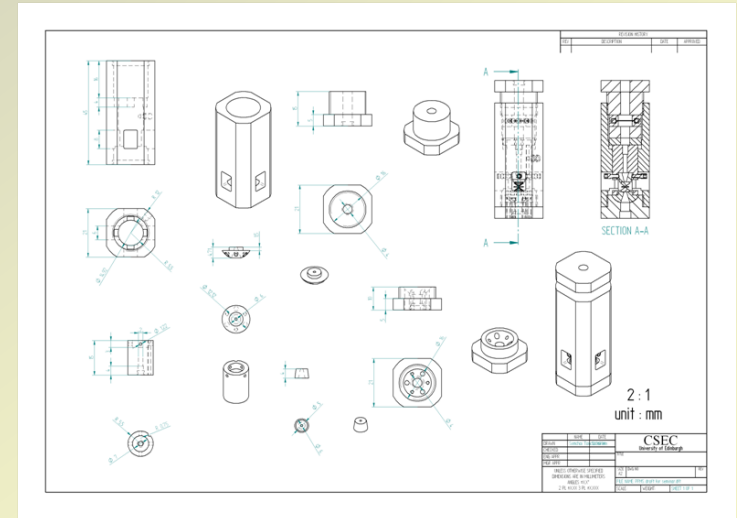
# Herramientas de diseño y simulación

## Software 3D CAD: *Solid Edge*

### Ensamblado 3D



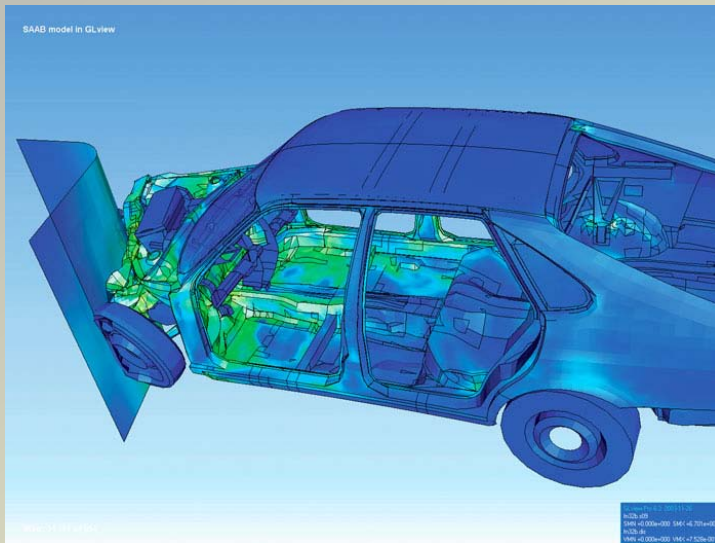
### Dibujos técnicos (2D)



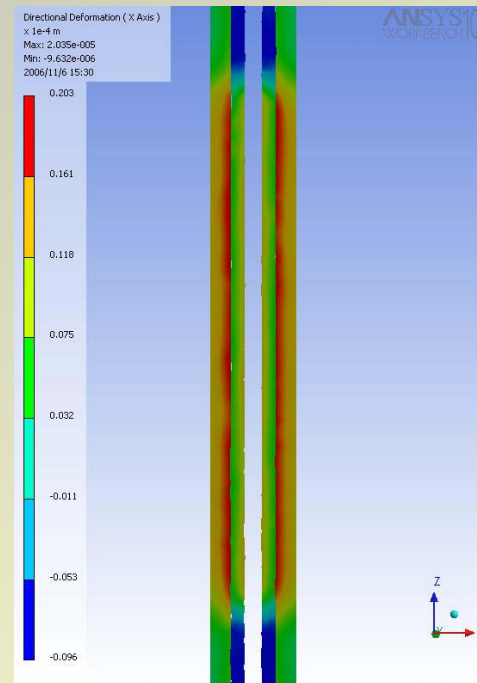
# Herramientas de diseño y simulación

## Finite Element Analysis: ANSYS

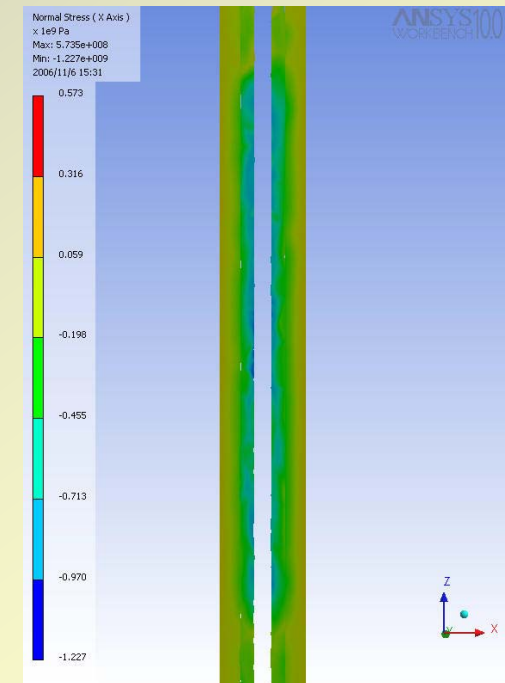
Visualización de cómo un coche se deforma en un choque asimétrico.



### Deformación



### Stress Axial





# Celdas de alta presión: propiedades magnéticas

## Magnetic Property Measurement System (MPMS) *Quantum Design*, USA



MPMS es un **magnetómetro** que utiliza tecnología Superconducting Quantum Interference Device (SQUID) para medir susceptibilidad magnética

ac y dc: 
$$M = \chi_m H$$

1.8-800 K ; 7 T magnet

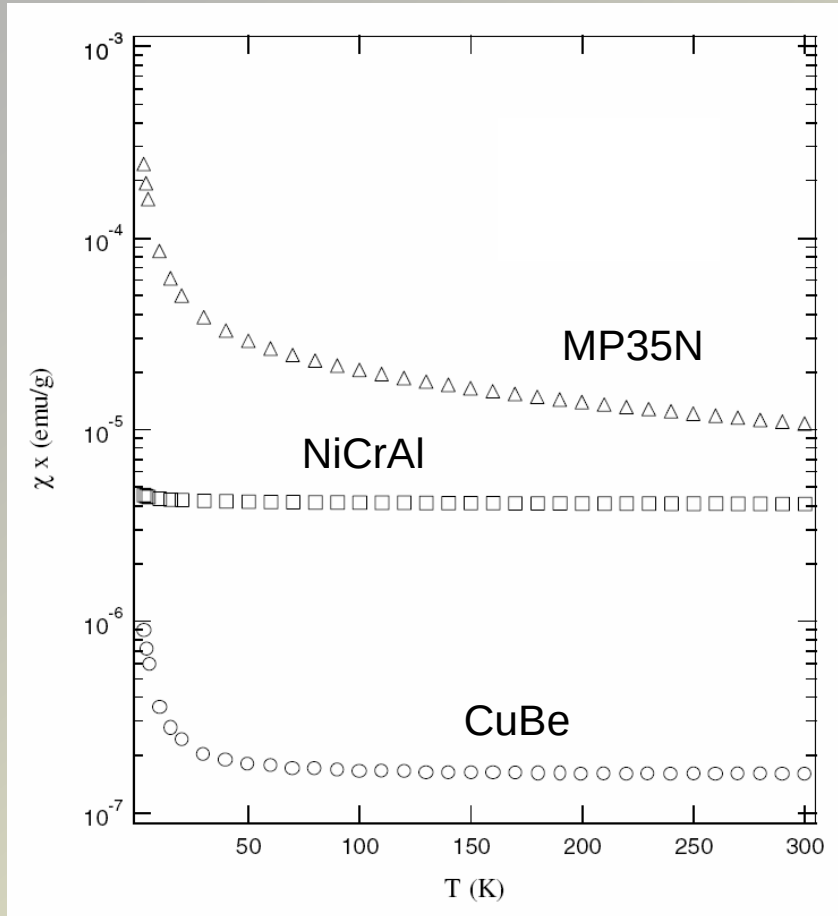
# *Celdas de alta presión: selección de materiales*

- High Strength
- Non magnetic
- Commercial availability
- Machinable



*Heat treatment increases the strength of the materials*

# Celdas de alta presión: selección de materiales



Yield Strength

MP35N 2.3 GPa

NiCrAl 2 GPa

CuBe 1.5 GPa

CuBe: muestras magnéticamente débiles

CuBe+NiCrAl: más alta presión

# *Celdas de alta presión: propiedades magnéticas*

Celdas de Piston-cilindro para MPMS ( $D_{\max} = 9 \text{ mm}$ )

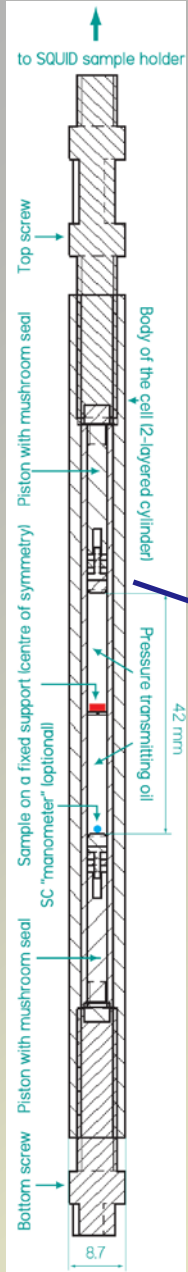




# Celdas de alta presión: propiedades magnéticas

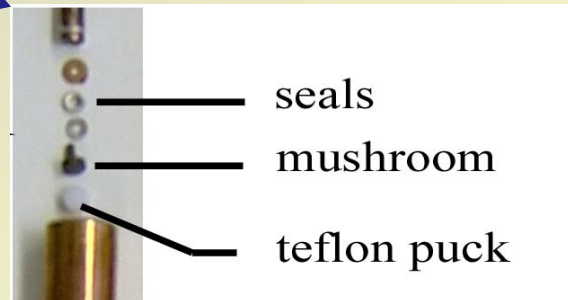
Long symmetric pressure cell (1 GPa)

Adecuado para materiales magnéticamente débiles



$P \approx 0$

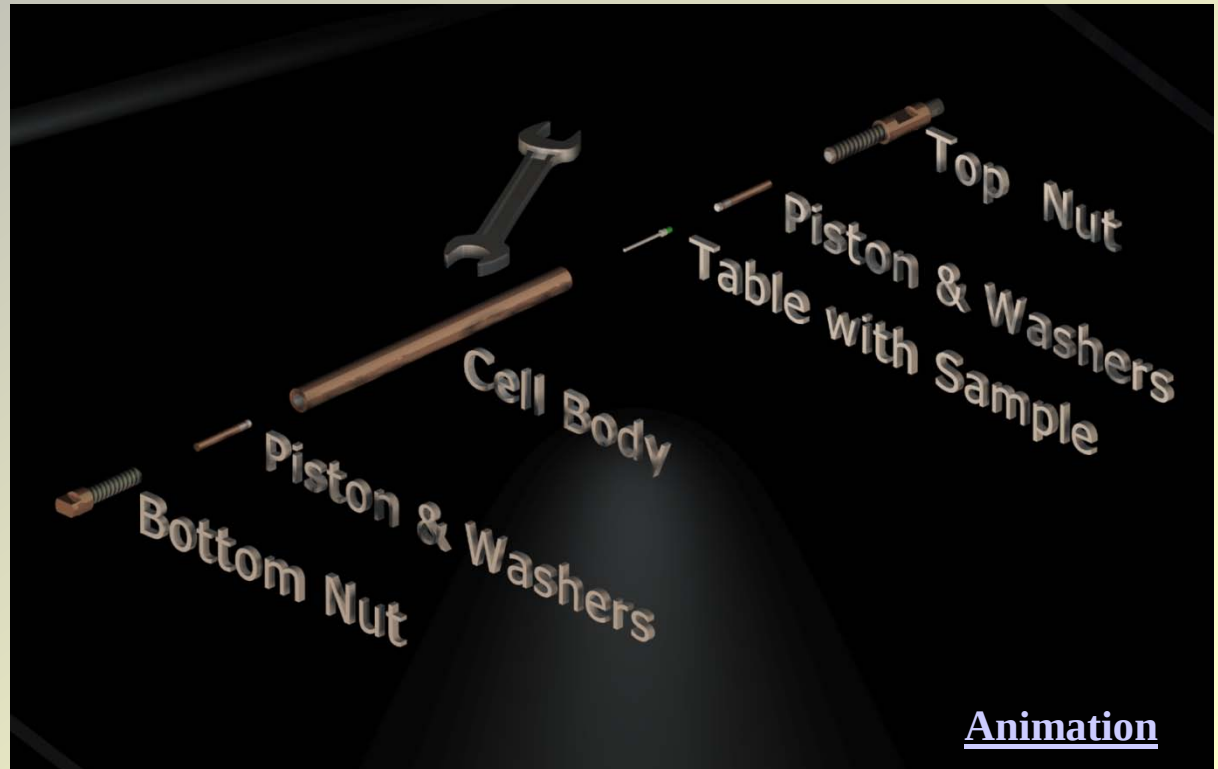
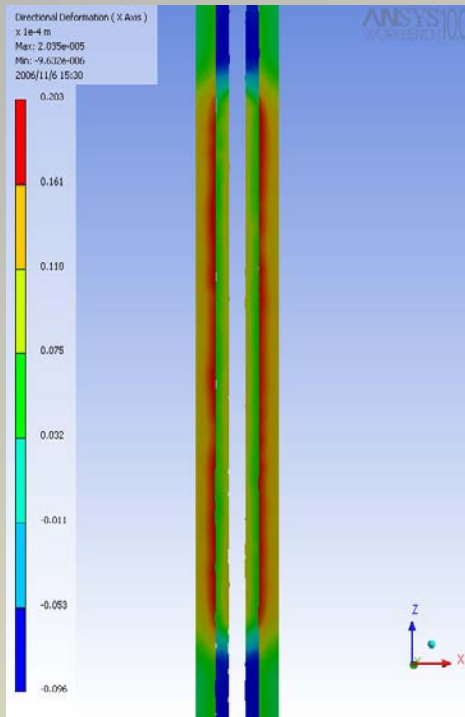
$P > 0$



# Celdas de alta presión: propiedades magnéticas

Long symmetric pressure cell

Cell deformation



# Celdas de alta presión: propiedades magnéticas

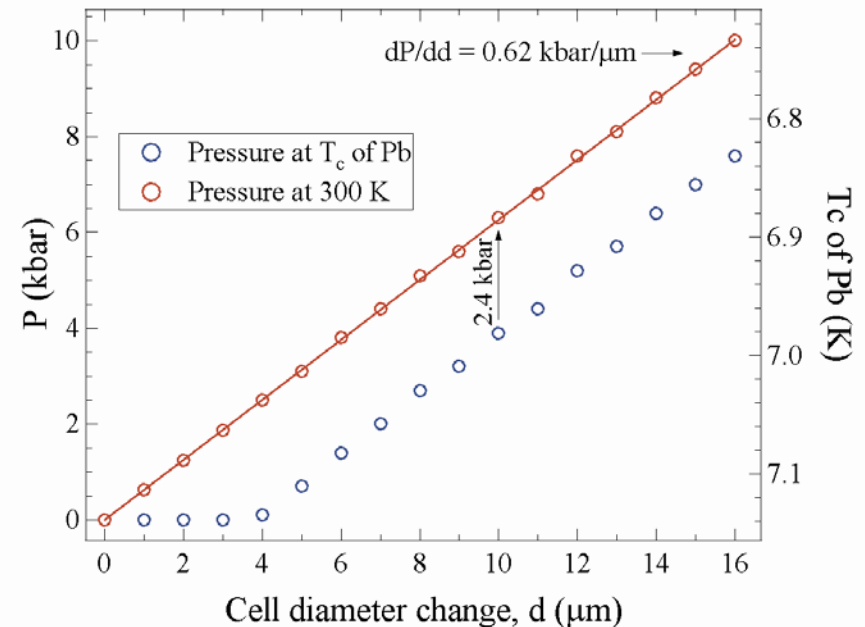
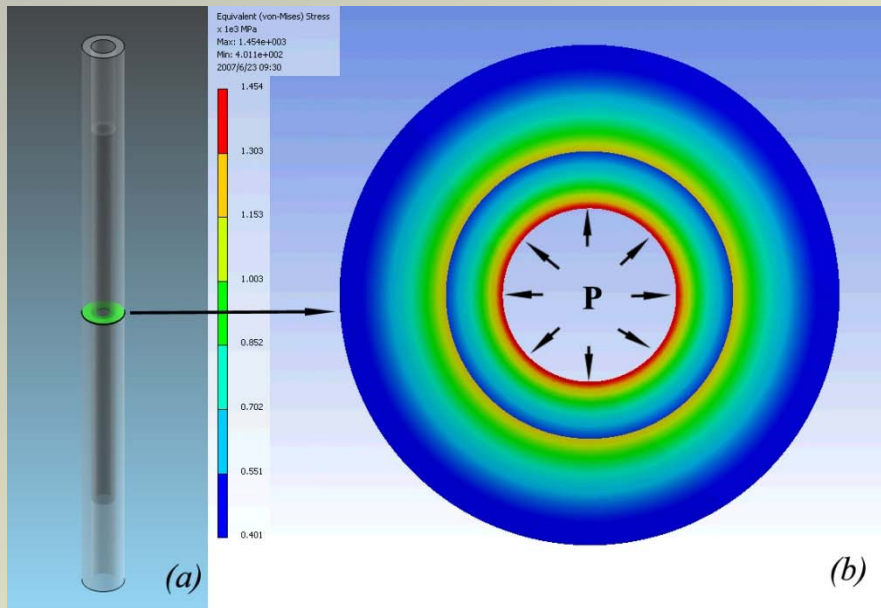
Long symmetric pressure cell:  
medida de la presión – *diámetro*

De la ecuación de Lamé  
se puede derivar que:

$$\frac{dP}{dd} \equiv \frac{E(b^2/a^2 - 1)}{2b(2 - m)}$$

CuBe  $\rightarrow dP/dd = \mathbf{0.62 \text{ kbar}/\mu\text{m}}$

Perfecto acuerdo entre la simulación  
FEA y los datos experimentales.

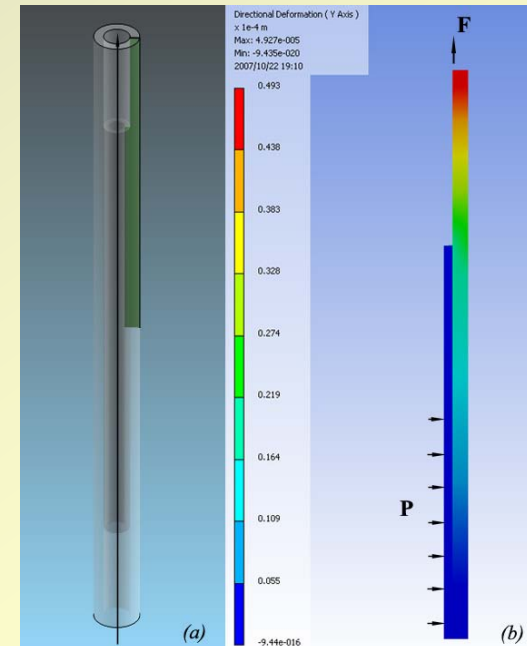
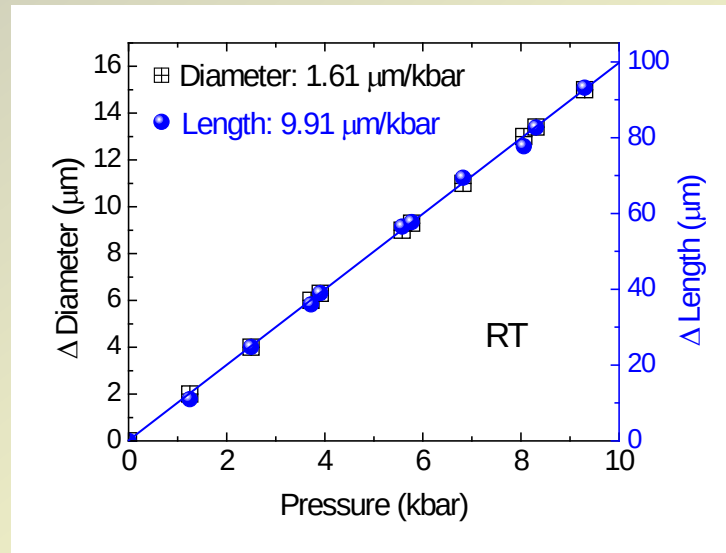
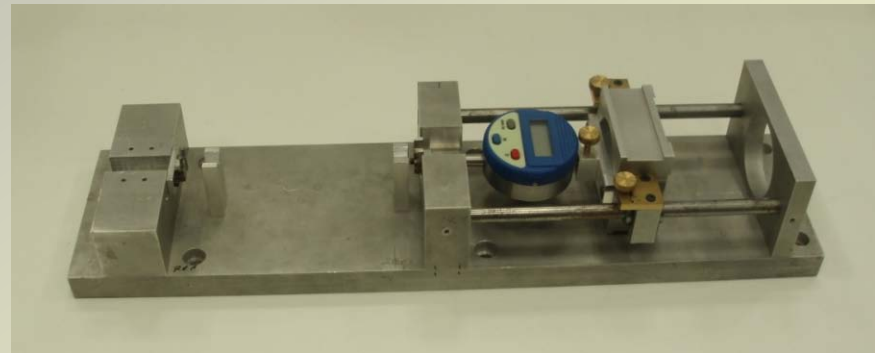
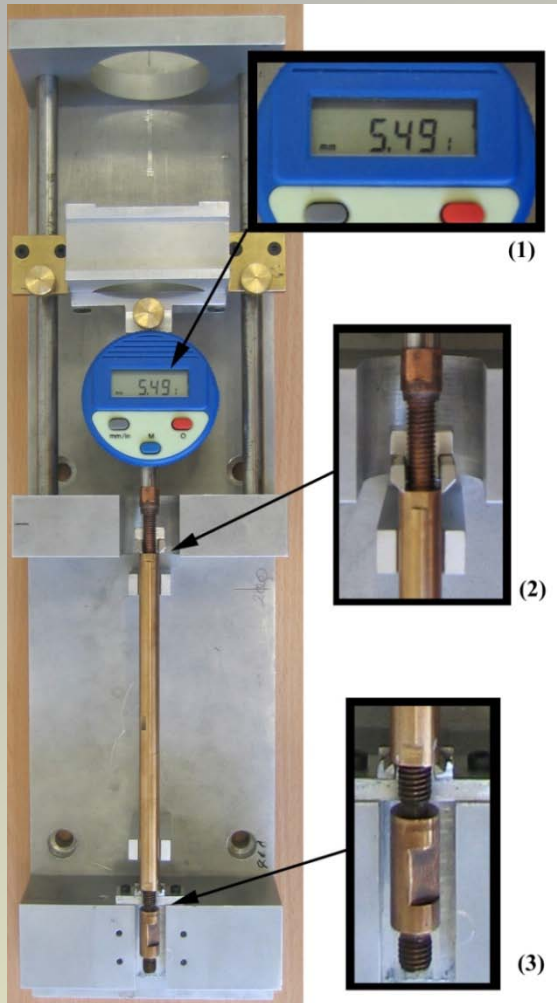


# Celdas de alta presión: propiedades magnéticas

Long symmetric pressure cell: medida de la presión - *longitud*

Accuracy: 1  $\mu\text{m}$

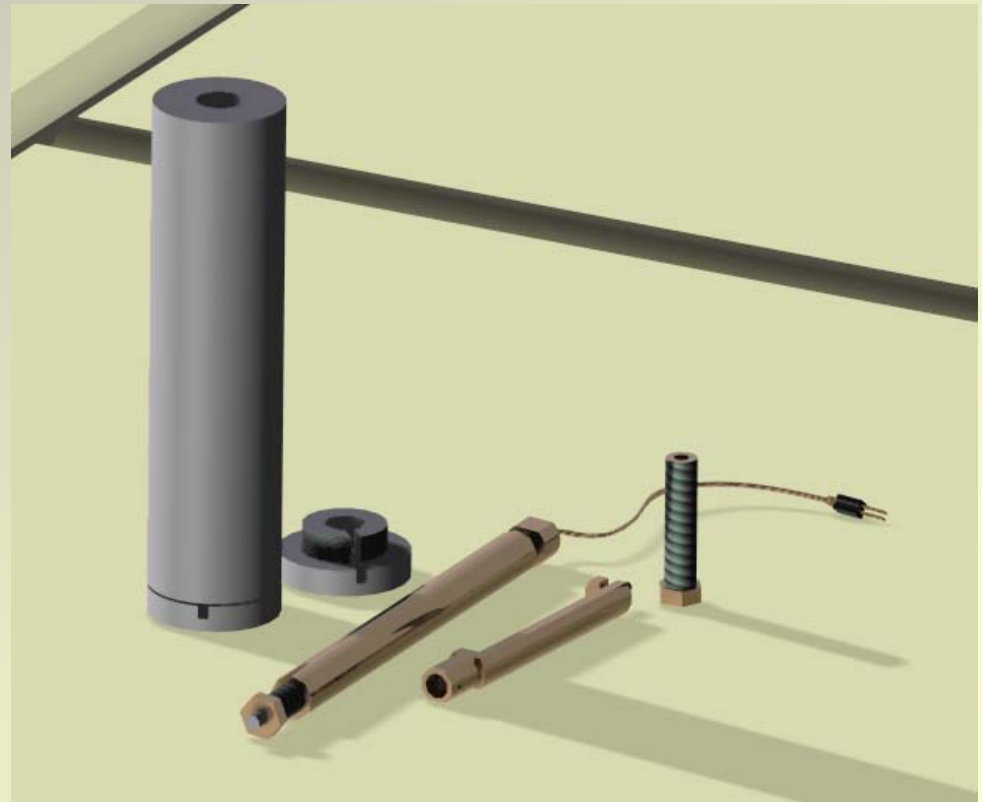
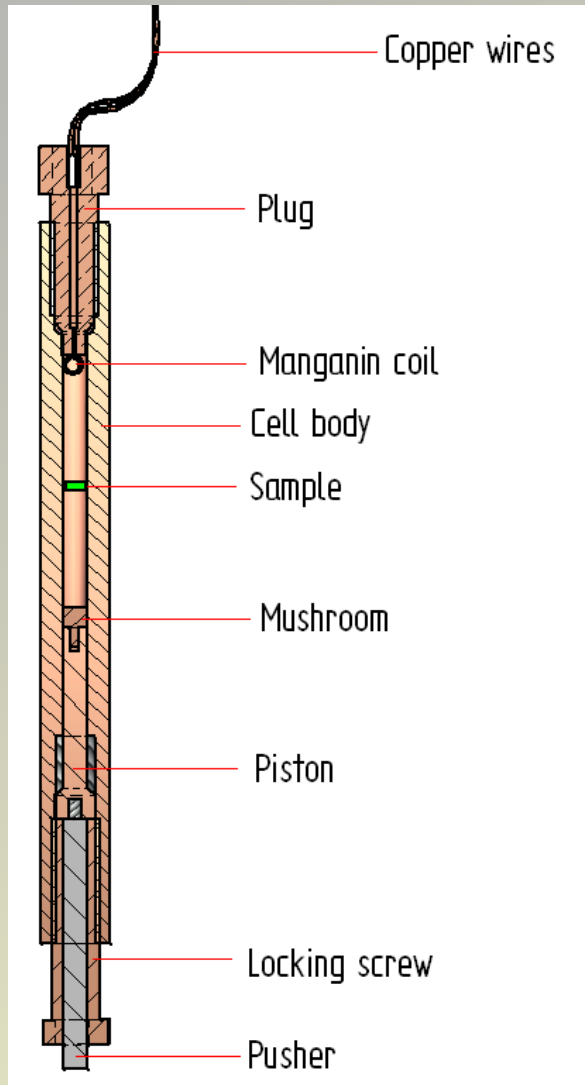
Length-difference meter





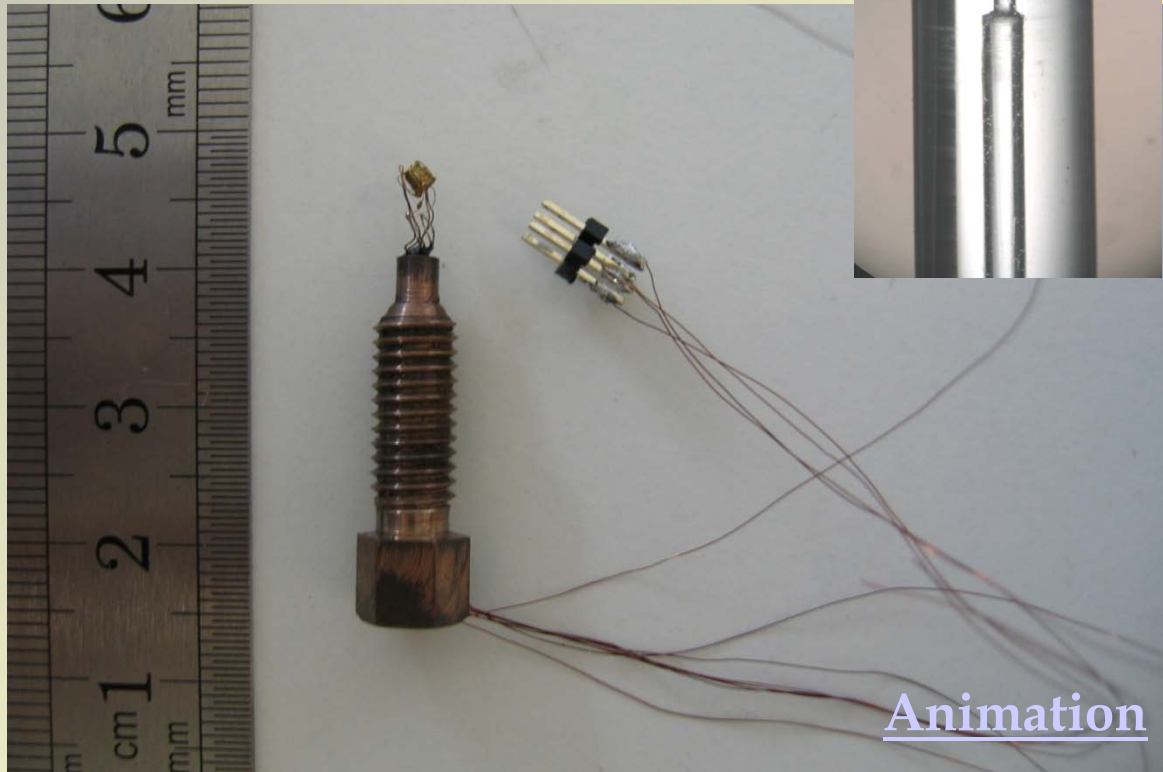
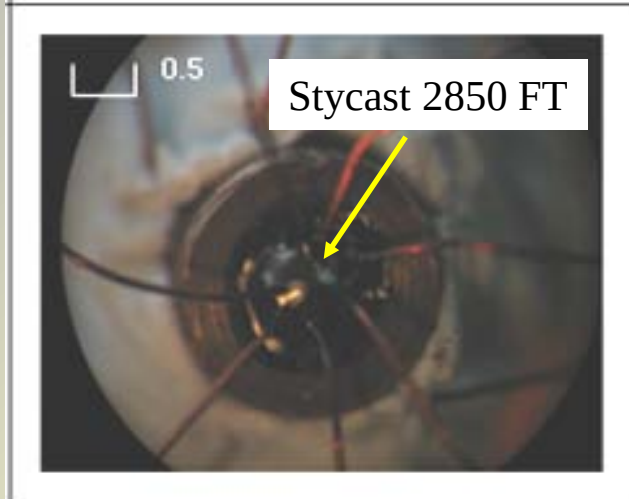
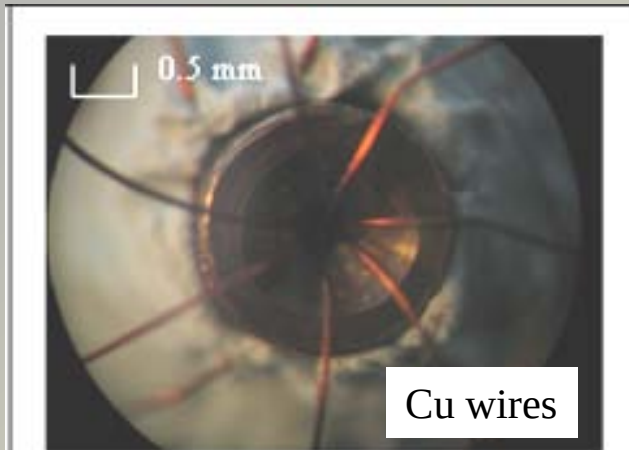
# *Celda de alta presión para monitorizar presión in-situ*

## Plug pressure cell (1 GPa)



# *Celda de alta presión para monitorizar presión in-situ*

Sellado de hilos de Cu – Stycast 2850 FT



Plug + manganin coil + connector

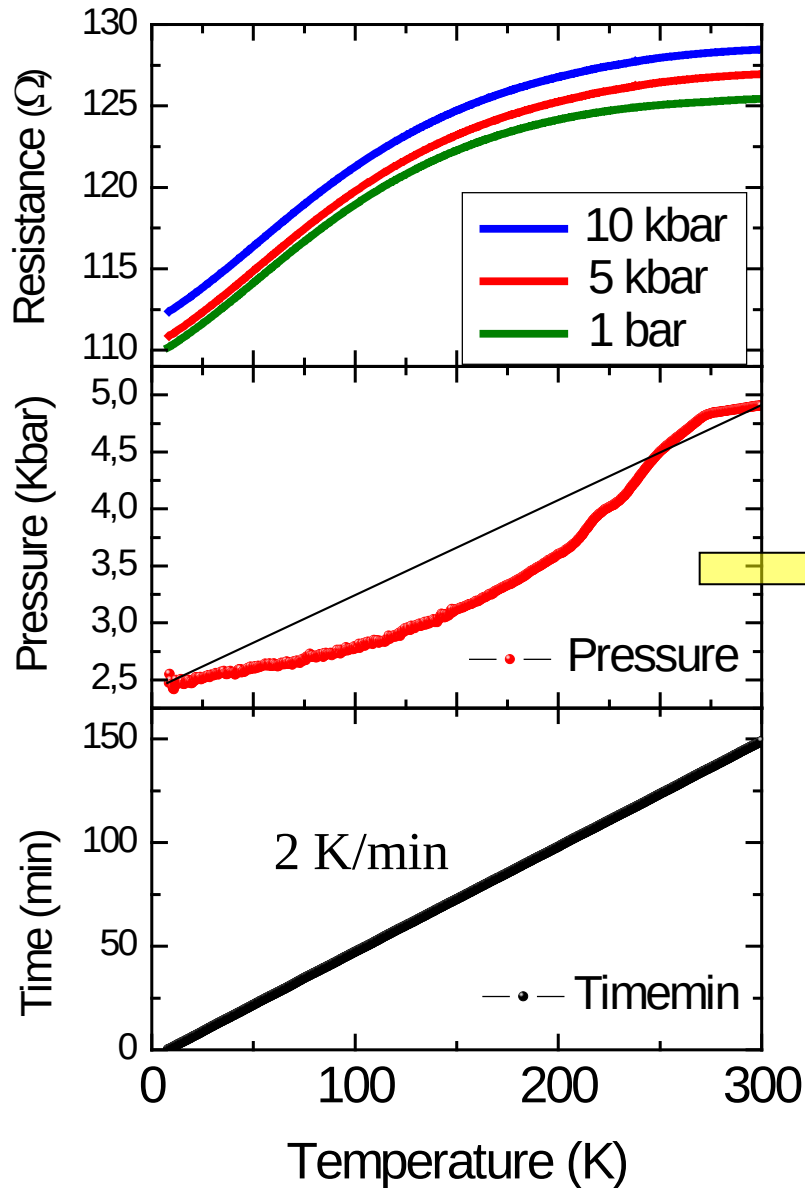
# *Celda de alta presión para monitorizar presión in-situ*

Prensa para aplicar presión

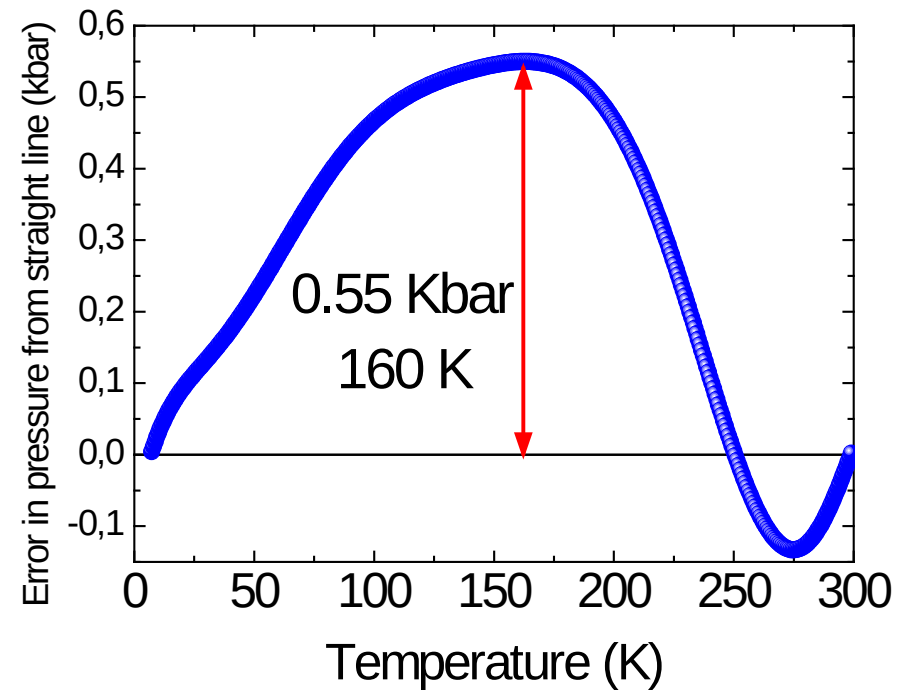


Presión *in situ* a RT

# Celda de alta presión para monitorizar presión in-situ



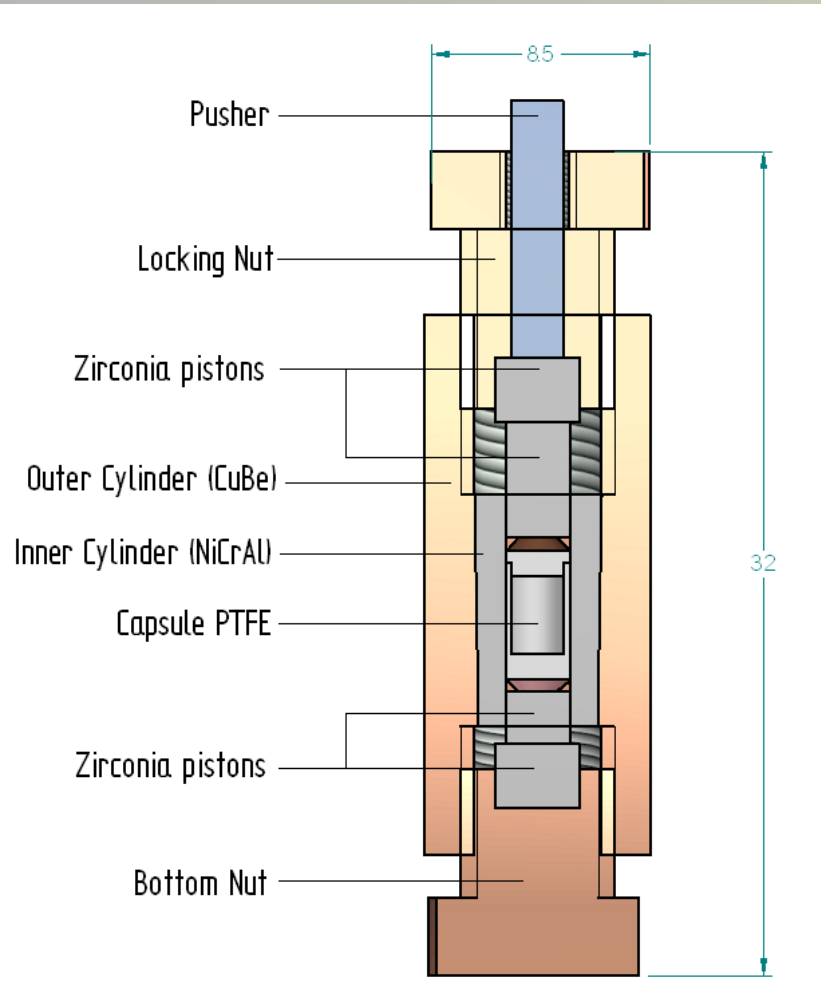
$$P = \frac{1}{\alpha} \left( \frac{R(P)}{R(0)} - 1 \right)$$





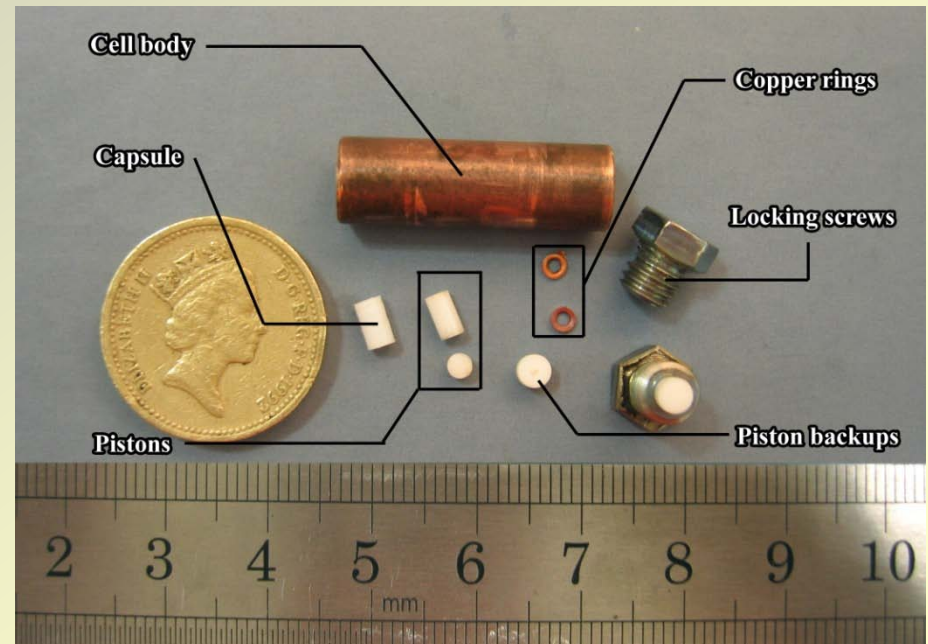
# Celdas de alta presión: propiedades magnéticas

## Microcelda para MPMS

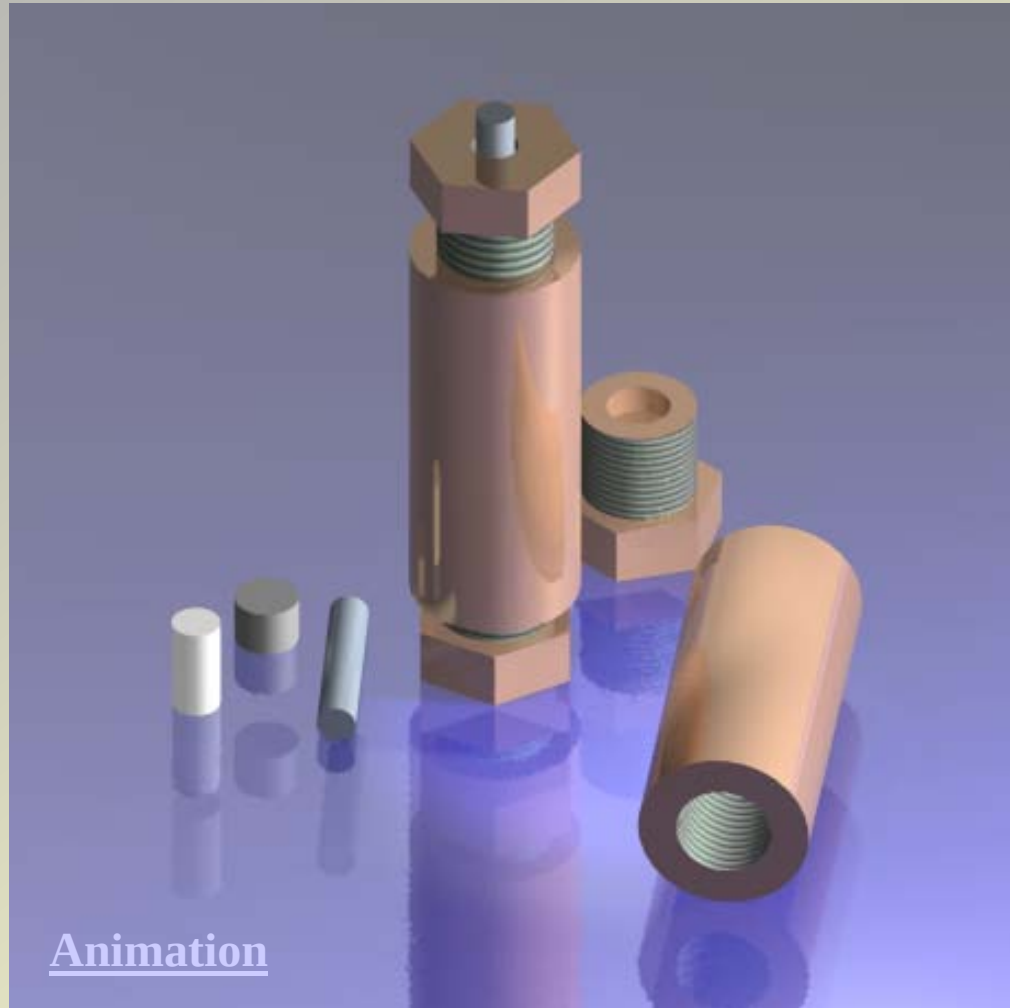


**2.2 GPa**

Susceptibilidad magnética DC y AC



# *Celdas de alta presión: propiedades magnéticas*



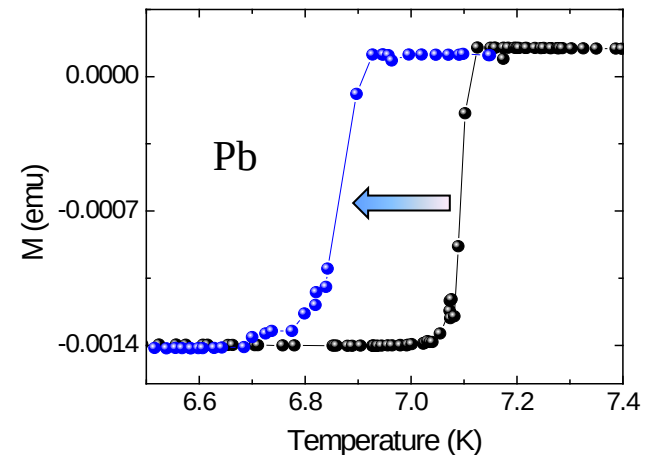
Animation

# Celdas de alta presión: propiedades magnéticas

## Microcelda para MPMS: medida de presión



Cápsula de Teflon: - Muestra  
- Aceite (Daphne)  
- Medidor de presión (Pb, Sn)



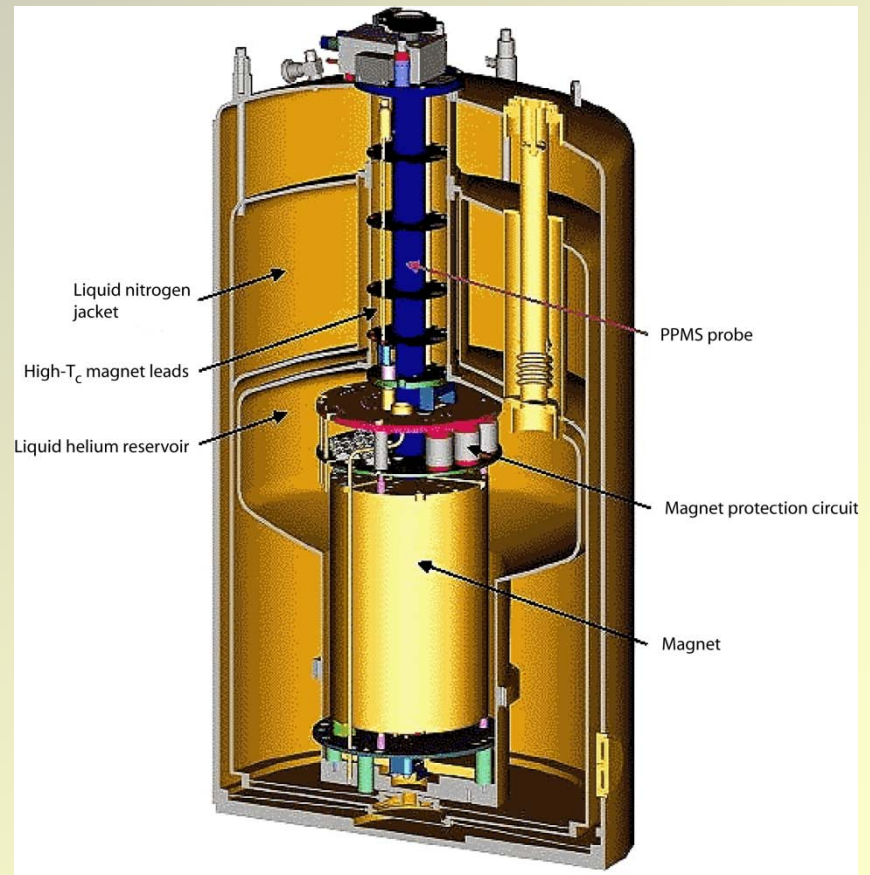
$$\Delta T / \Delta P = 0.365 \text{ K/GPa}$$

# *Celdas de alta presión: transporte*

Physical Property Measurement System (PPMS) *Quantum Design, USA*

PPMS es un instrumento de fácil manejo y versátil basado en un imán superconductor.

9T, 1.8-400 K

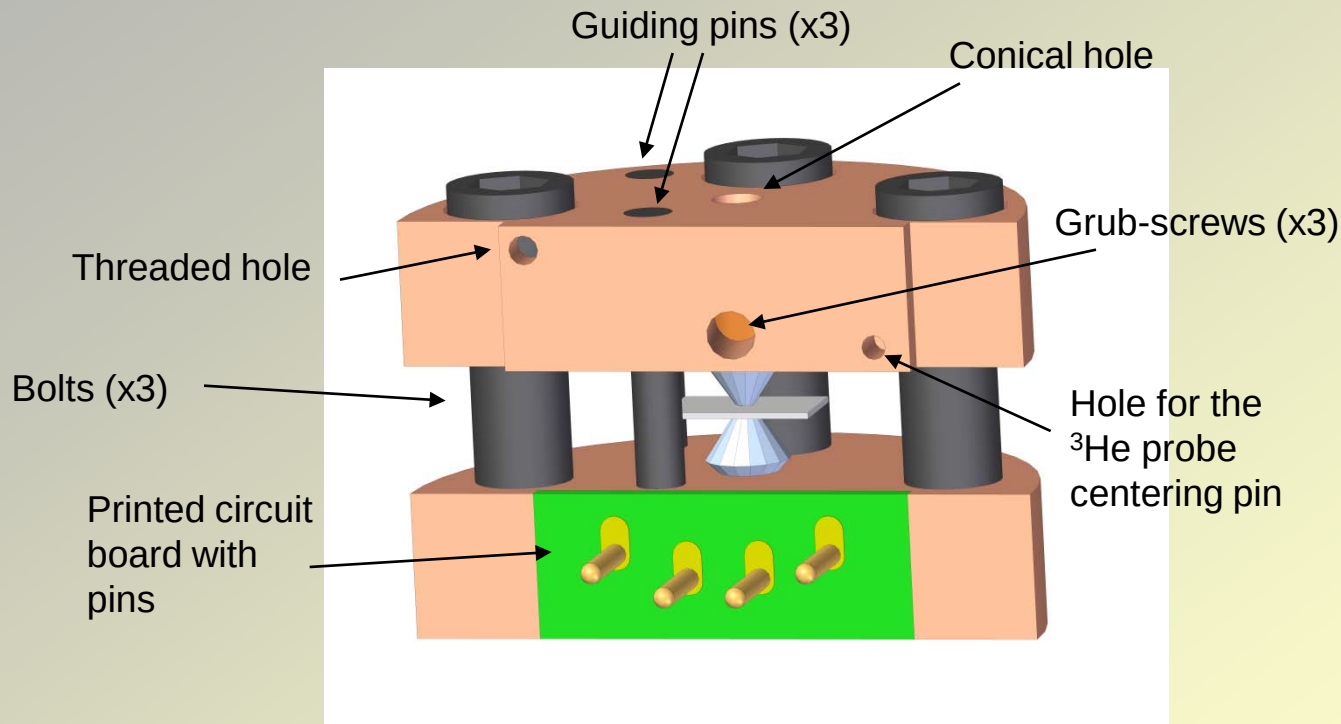




# *Celdas de alta presión: transporte*

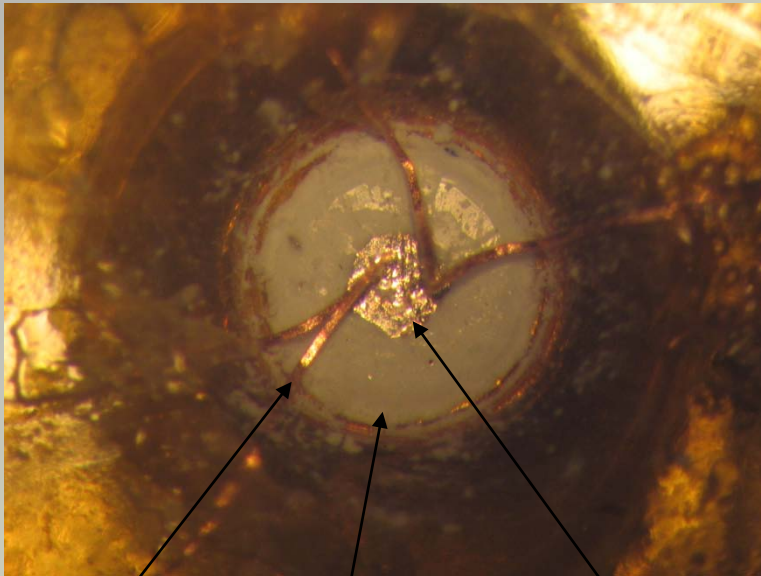
## 3He Celda de alta presión para PPMS

Resistividad con presión hasta 0.5 K



# *Celdas de alta presión: transporte*

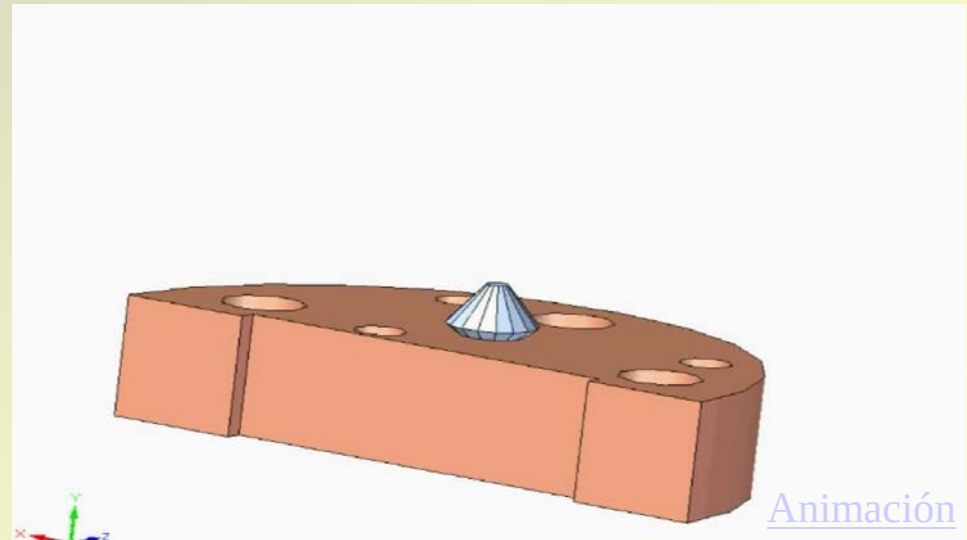
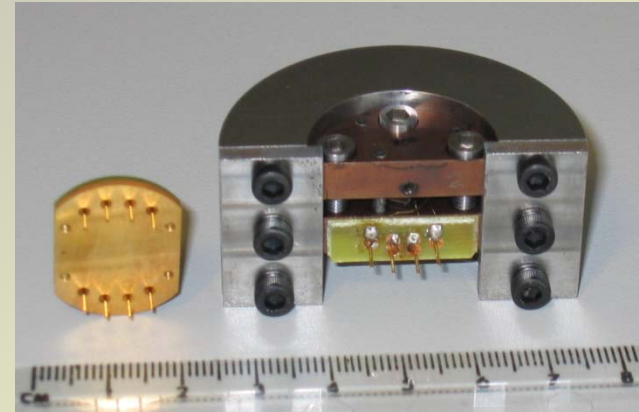
## 3He Celda de alta presión para PPMS



Hilos de Cu

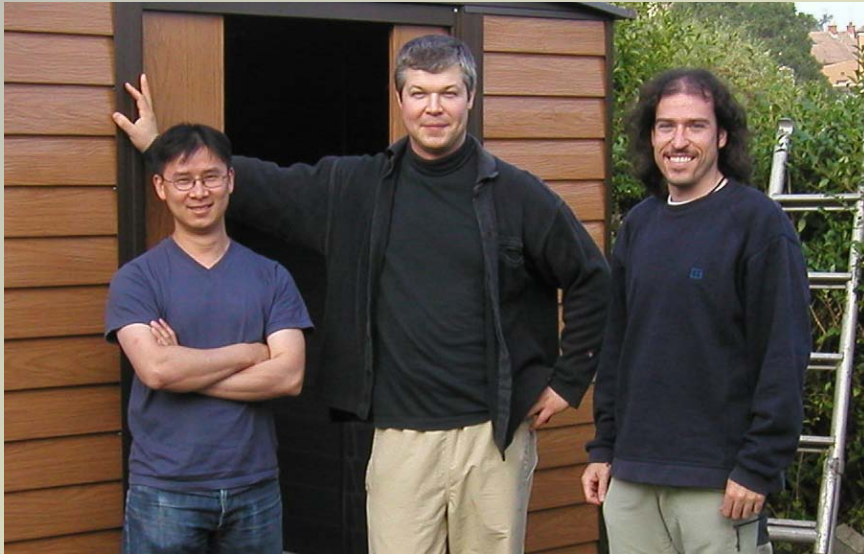
MgO

Muestra



# Agradecimientos

- Dr. Konstantin Kamenev
- Dr. Somchai Tancharakorn
- Andrew Downie and Ronnie Proc (School of Physics Workshop, UoE)
- People in CSEC
- Royal Society and EPSRC







*¡¡Gracias!!*





# Estación experimental de altas presiones en el sincrotrón Alba

Inma Peral

ALBA

<http://www.cells.es>



<http://www.cells.es>

## History:

- 1990: 1<sup>st</sup> attempts (bottom up) to get an accelerator and SL source in Spain
- 1994: Generalitat (Commissió Promotora in 1992) appoints staff for the preparation of a conceptual design report for a SL source
- 1996: LLS (as an IFAE subgroup and funded by Generalitat and OCYT) started to detail the Conceptual Design Report and Scientific Case.
- 1998: Conceptual design report handed to relevant authorities
- 2000: Creation of the LLS Consortium between DURSI and UAB to promote SR Lab.
- 2002: Approval of the project by the Spanish and the Catalan Government.
- 2003: Creation of a Consortium for the Construction, Equipping and Exploitation of the SL Laboratory as well as the governing Commissions: "Rectora" and "Ejecutiva".
- 2003: Appointment of the Chairman of the Executive Commission and the Director
- 2003: Announcement of the positions for the Heads of the 5 Divisions

October 2003: Meeting in Mahon to present the project

February 2004: Meeting in Malaga to start defining scientific cases  
for future beamlines

February 2005: beamlines presentation in the "Scientific Advisory Committee" (SAC)

# PARÁMETROS RELEVANTES DE ALBA:



$E = 3.0 \text{ GeV}$

Circunferencia = 268.8 m

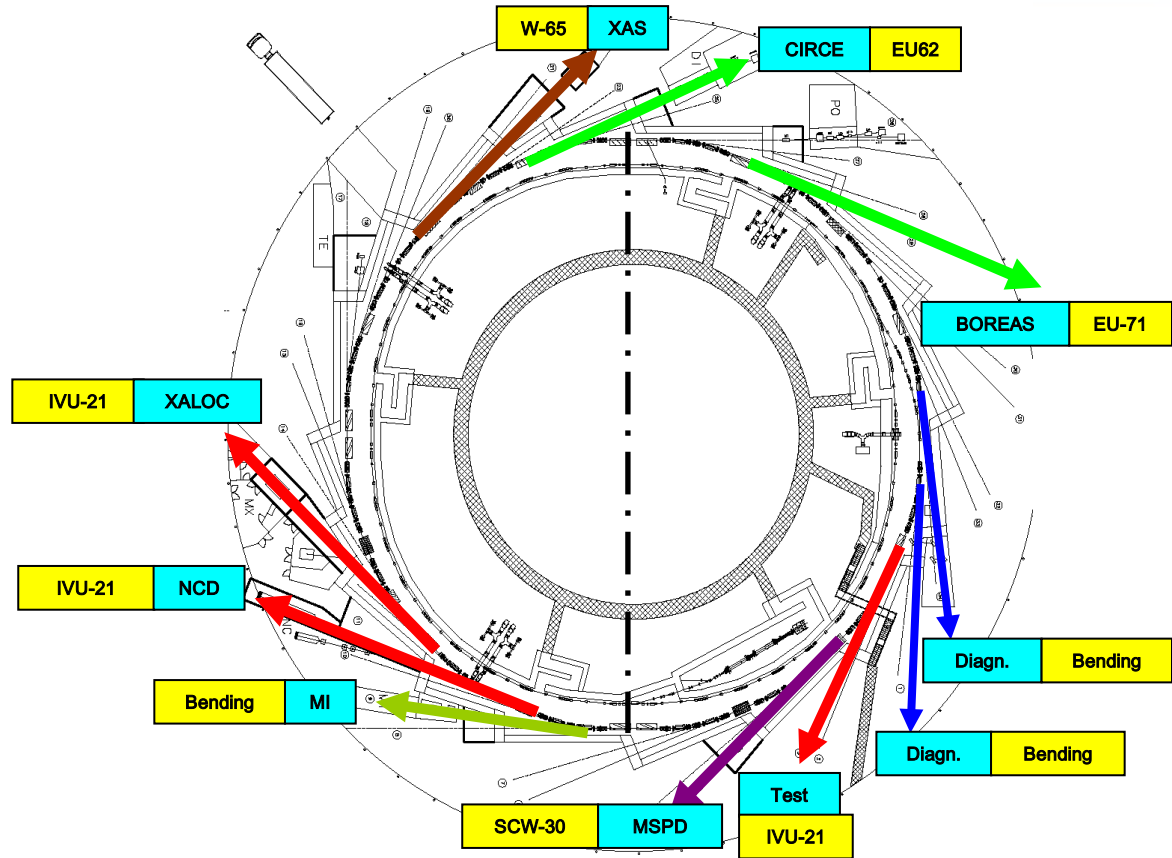
4 fold DBA lattice

$\epsilon = 3.76 \text{ nm.rad}$   
(emitancia)

3 different straight sections:

- 1) 4 x 8 m:
- 2) 12 x 4.3 m:
- 3) 8 x 2.6 m:

32 Líneas



3 son útiles para líneas  
12 son útiles para líneas  
2 son útiles para líneas



## Líneas de la fase I

- 1.- Non Crystalline Diffraction (NCD)
- 2.- Macromolecular crystallography (XALOC)
- 3.- Photoemission Spectroscopy and Microscopy (CIRCE)
- 4.- High Resolution Powder diffraction and high pressure
- 5.- X-ray Absorption Spectroscopy
- 6.- Circular Dichroism and Resonant Scattering
- 7.- X- ray microscopy (MISTRAL)

## Lineas de la fase II

MIRAS "An infrared microspectroscopy beam line for Alba

LOREA "Low energy ultra-high resolution angular photoemission for complex materials"

Microfocus Beamline for Macromolecular Crystallography

Microfocus beamline in Alba

High resolution and micro-focus X-ray Diffraction beamline

AIBA: Alba imaging and Biomedical applications

<http://www.cells.es/Beamlines>

## Plan (fechas de inicio)

- Edificio preparado: Junio 08
- Infraestructura de las líneas: Junio 08
- **Instalación de los componentes opticos de las líneas: Marzo 2010**
- Puesta a punto del Booster: septiembre 2010
- Puesta a punto del storage ring : marzo 2011
- Instalación de los Insertion Devices : junio de 2011
- **Puesta a punto ... a finales 2011**

21 December 2004

Proposal of  
**High-Resolution Powder Diffraction and Microfocus  
Beamline**  
at ALBA  
for Structural Crystallography and Hard Condensed Matter  
Studies.



|        |                                                                                   |    |
|--------|-----------------------------------------------------------------------------------|----|
| 2.     | Scientific case                                                                   | 6  |
| 2.1.   | Experiments using the high-angular resolution of the powder diffractometer        | 6  |
| 2.1.1. | Determination of crystal structures                                               | 6  |
| 2.1.2. | Structural studies on correlated materials showing phase transitions              | 8  |
| 2.1.3. | Microstructural studies                                                           | 10 |
| 2.2.   | Experiments using the high flux of the powder diffraction station                 | 11 |
| 2.2.1. | In situ diffraction studies of catalysts under operating conditions               | 11 |
| 2.2.2. | Kinetic studies of chemical reactions and dynamic studies of phase transitions    | 13 |
| 2.2.3. | Quantitative diffractometry                                                       | 13 |
| 2.2.4. | Residual stress measurements                                                      | 14 |
| 2.3.   | Experiments using the microfocusing capability                                    | 15 |
| 2.3.1. | <del>Microcrystal diffraction studies at ambient and non-ambient conditions</del> | 15 |
| 2.3.2. | Powder diffraction studies at high pressures                                      | 17 |

## Tipos de experimentos en celdas de yunque de diamante con luz sincrotrón:

- Difracción de rayos-x
  - Espectroscopía
  - Scattering inelástico
  - Radiografía de rayos-x
  - Infrarrojo
- Estructura  
Ecuaciones de estado P-V-T
- Estructura local  
tipo de enlace químico

Información sobre luz sincrotrón  
<http://www.lightsources.org>

## Contribuciones de la difracción de rayos-x a altas presiones

Identificación de la **estructura del  $N_2$  polimérico**

Se han resuelto largas controversias respecto al **diagrama de fases** del S.

Se han investigado el efecto de presión en sistemas que son metales a presión ambiental que se espera que sufran transiciones que rompen la simetría,  
p.e. **Li  $\rightarrow$  Li molecular parecido al  $H_2$**

Ha sido **instrumento para comprender nuevas reacciones y fases sólidas.**

Referencias completas en

Hemley et. al, Journal of Synchrotron radiation, 2005

# MSPD application and scientific case



## Scientific case / Application:

Meet the needs of the Spanish user community in:

- Diffraction on polycrystalline samples
- High Resolution Powder Diffraction
- Powder Diffraction at High pressure

Structure Solution  
Structure Refinement  
Real structure analysis  
In-situ diffraction  
Etc. ..

## Technical requirements:

|                             |                                                                     |
|-----------------------------|---------------------------------------------------------------------|
| Maximum Energy range:       | 7 - 50keV                                                           |
| Main Energy range:          | 20 -30keV                                                           |
| Energy Resolution:          | $2 \cdot 10^{-4}$                                                   |
| Focusing Option             |                                                                     |
| Maximum beam:               | H: $400\mu\text{rad}$ , V: $125\mu\text{rad}$                       |
| Two End stations:           | I: High Pressure PD (Diamond Anvil Cells)<br>II: High resolution PD |
| Flexible layout             |                                                                     |
| Maximum available BL length | 42m                                                                 |

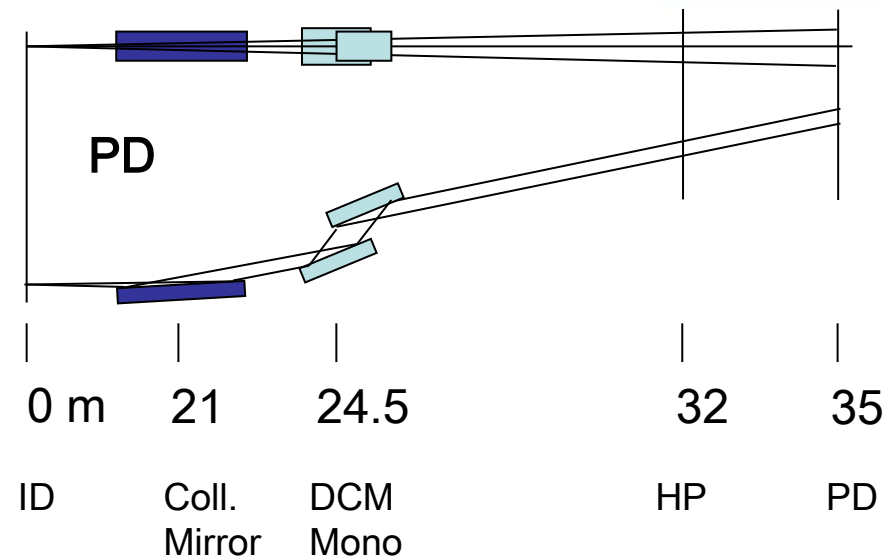
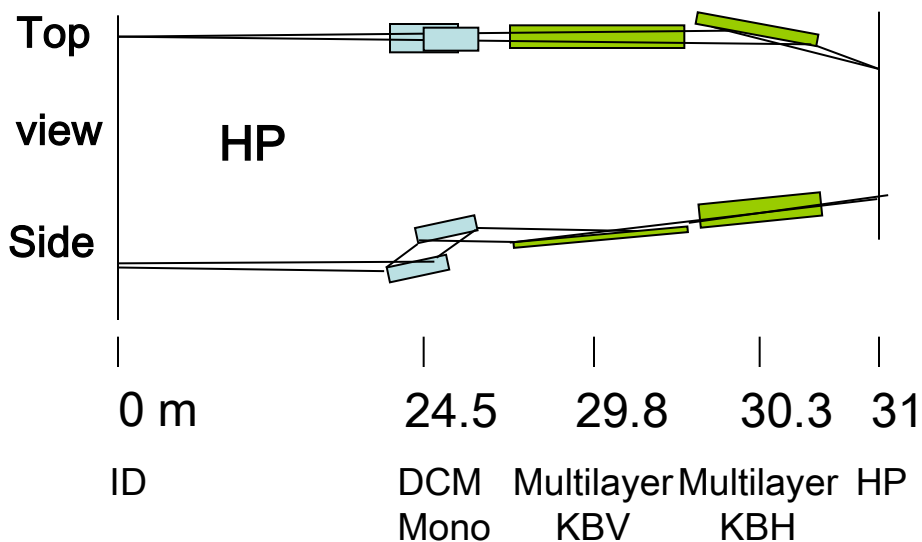
Distance to source:

|                     |            |
|---------------------|------------|
| FE wall:            | 17.5m      |
| Collimating Mirror: | 21m        |
| DCM:                | 25.7m      |
| KB System:          | 29.9/30.3m |
| HP Station:         | 31m        |
| PD station:         | 35m        |

Maximum available: 42m



# Optical layout



## Configuration 1:

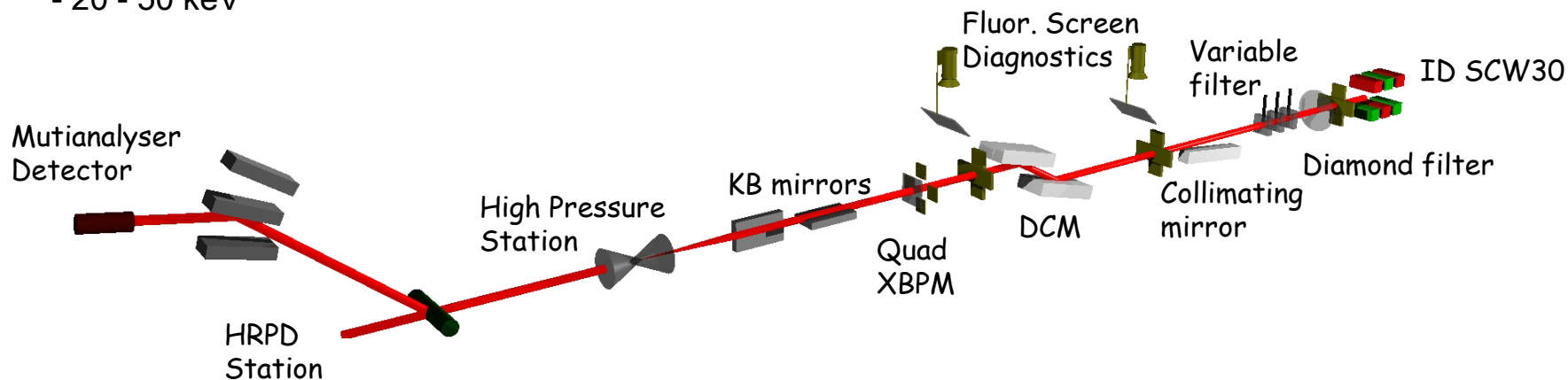
Un-mirrored mode with optional KB mirror

- CVD-diamond heat absorber
- Double Crystal Monochromator DCM (cryo-cooled)
- Multilayer KB mirror (optional)
- 20 - 50 keV

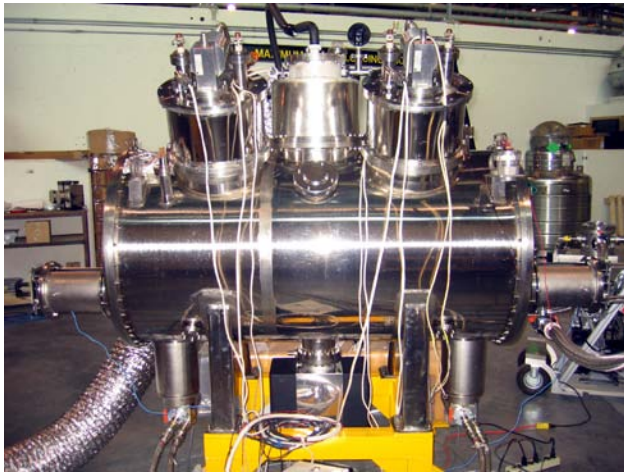
## Configuration 2:

Mirrored mode:

- Collimating Mirror with Pt, Rh, Si coating
- DCM cryo-cooled, Si111
- 7 - 40 keV

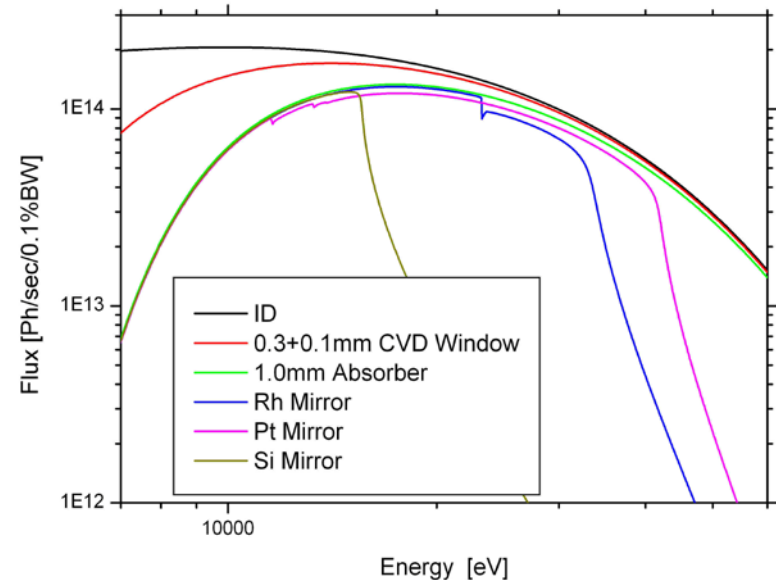


# MSPD Insertion Device



Superconducting Wiggler SCW30 from  
*Budker Institute of Nuclear Physics*

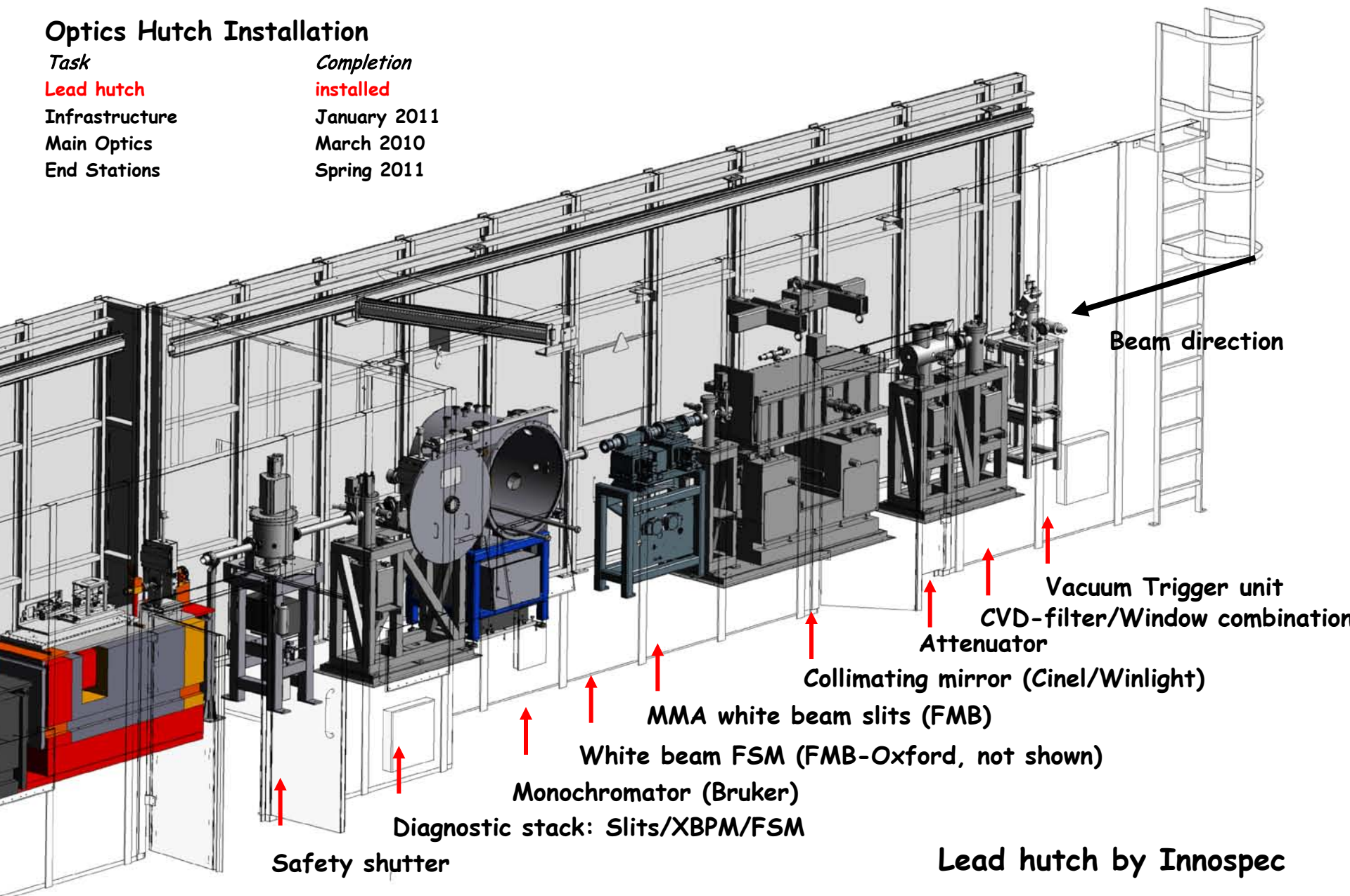
|                        |                          |
|------------------------|--------------------------|
| Variable K, max        | 6.08                     |
| Maximum total Power:   | 18.75 kW                 |
| Power density on axis: | 44.2 kW/rad <sup>2</sup> |
| E <sub>c</sub> max:    | 12.9 keV                 |
| Magnetic Length        | 1.76 m                   |
| Period length          | 30.16 mm                 |
| Number of Periods:     | 58.5                     |
| Max. Field             | 2.16 T                   |



Flux through HxV 200x125  $\mu$ rad  
@ Max K=6.08, 400 mA

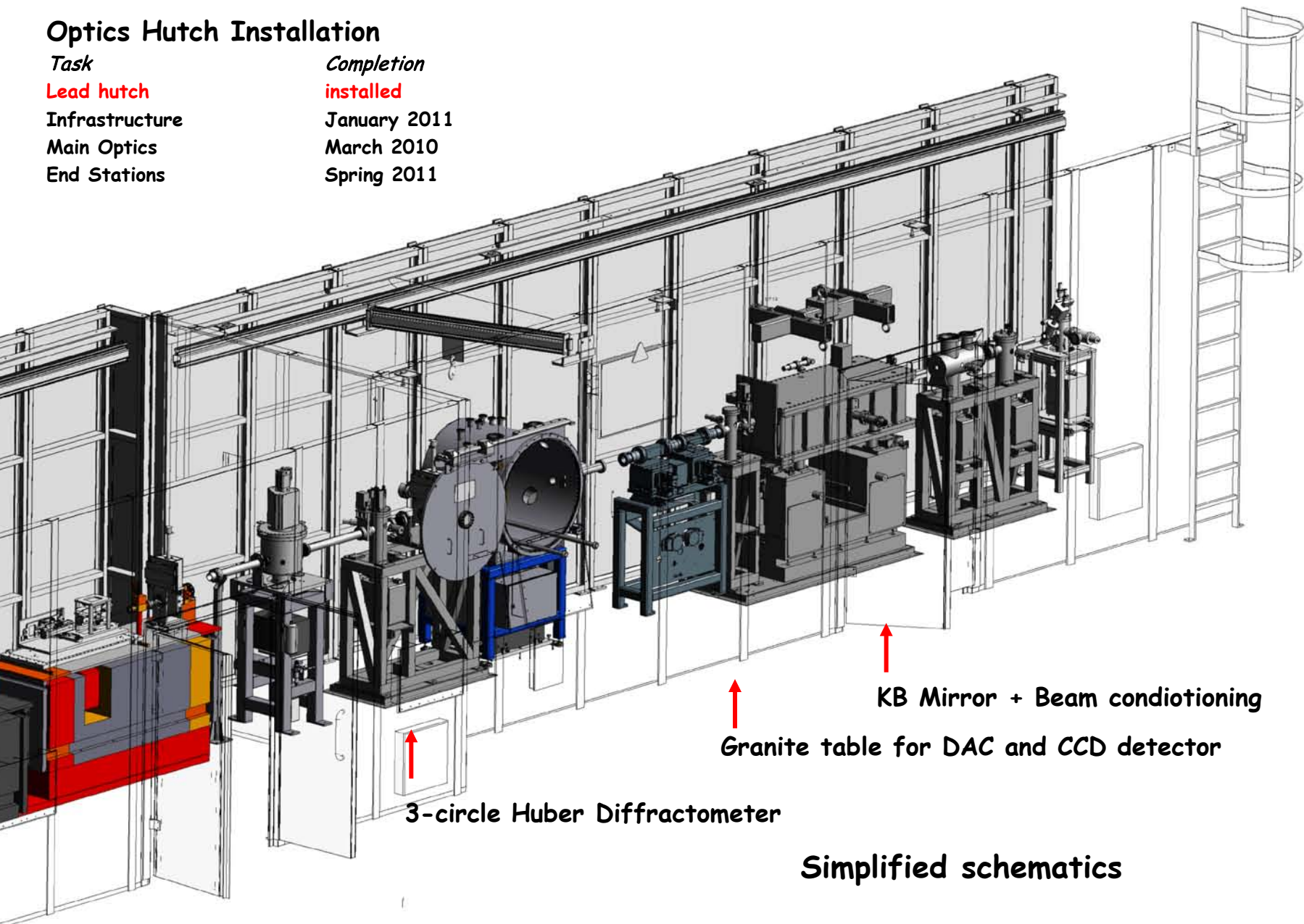
# Optics Hutch Installation

| Task           | Completion   |
|----------------|--------------|
| Lead hutch     | installed    |
| Infrastructure | January 2011 |
| Main Optics    | March 2010   |
| End Stations   | Spring 2011  |



# Optics Hutch Installation

| Task           | Completion   |
|----------------|--------------|
| Lead hutch     | installed    |
| Infrastructure | January 2011 |
| Main Optics    | March 2010   |
| End Stations   | Spring 2011  |



Simplified schematics



# Focusing optics for High Pressure KB Mirror System



## KB- Mirror in Experimental hutch for focusing on HP

ESRF bender design licensed to IRELEC

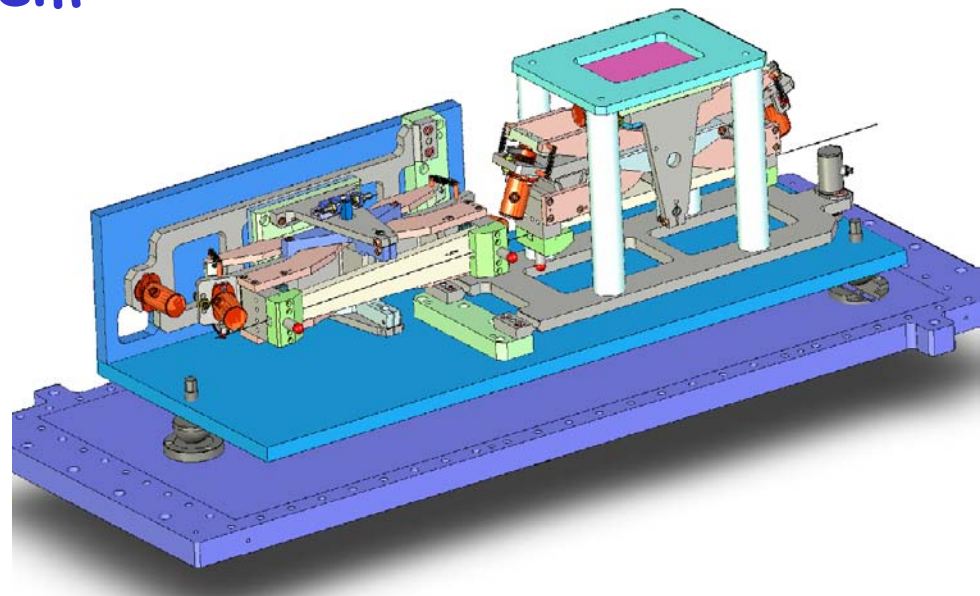
Mirror size 300 x 40 mm

Optical size 240 x 10 mm

Aperture 12 x 12 mm

Energy range 20 – 50 keV

Multilayer mirror (from AXO Dresden)

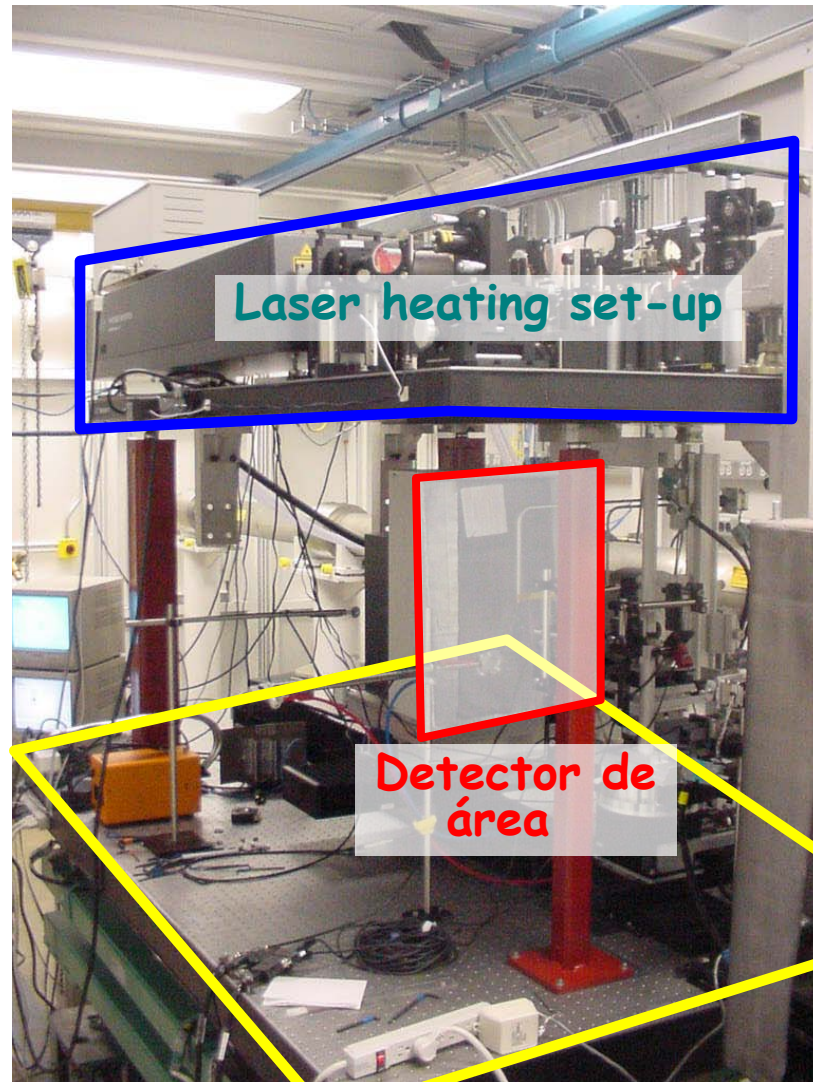


|                      | <b>KBV:</b>    | <b>KBH:</b>    |
|----------------------|----------------|----------------|
| Distance p           | 29.9m          | 30.3m          |
| Distance q           | 1.1m           | 0.7m           |
| M                    | 27             | 43             |
| Substrate            | flat           | flat           |
| Orientation          | [100]          | [100]          |
| S-Roughness RMS      | 0.15nm         | 0.15nm         |
| Slope err. in bender | 1 $\mu$ rad    | 1.5 $\mu$ rad  |
| Coating              | W/Si           | W/Si           |
| Layer pairs          | 110            | 150            |
| Nominal thickness    | 3.0nm          | 2.6nm          |
| Lateral gradient     | 0.41nm / 300mm | 0.57nm / 300mm |
| Interface rough. RMS | 0.3nm          | 0.3nm          |
| Reflectivity @30keV  | 85%            | 83%            |
| Acpt. height @30keV  | 1.7mm          | 2mm            |

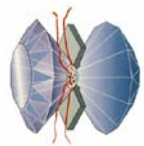
# Cabinas experimentales dedicadas a experimentos de altas presiones



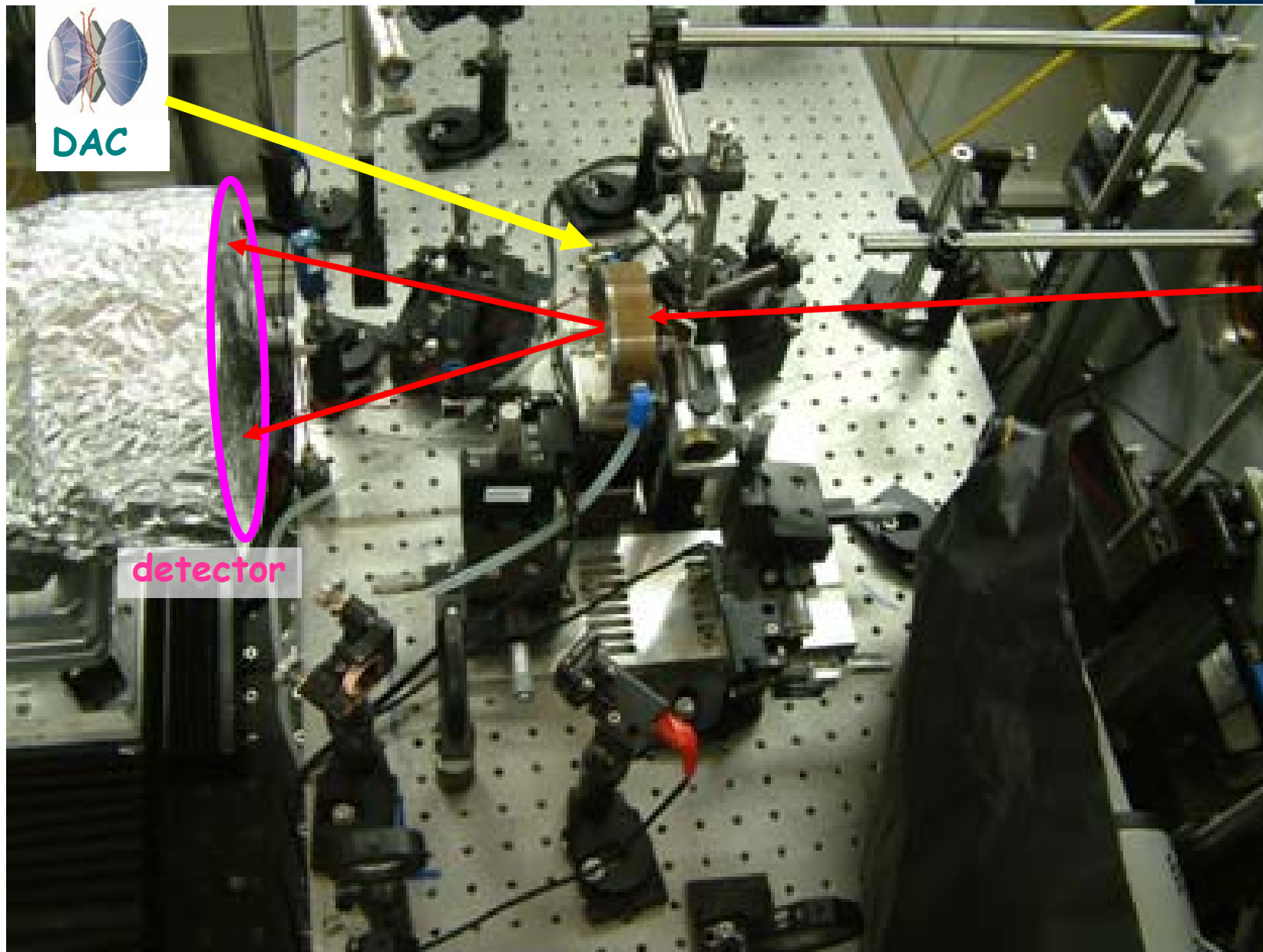
ID27 ESRF  
<http://www.esrf.eu>



ID-B, APS  
<http://www.aps.anl.gov>



DAC



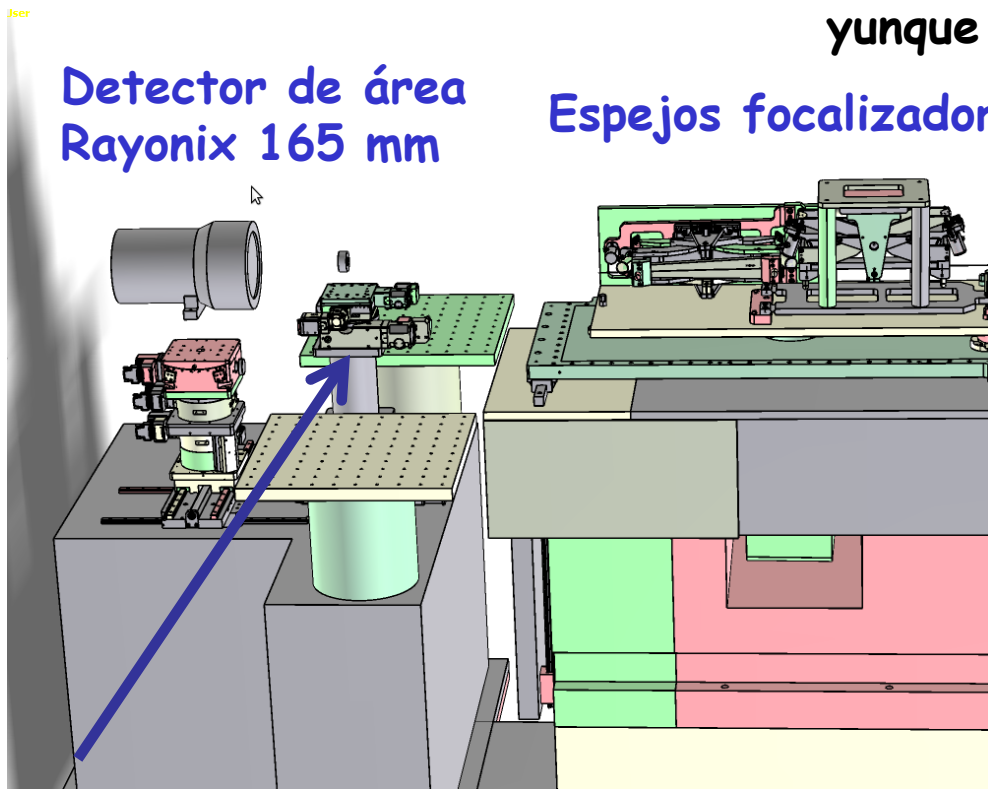
# Diseño detallado de la estación experimental para altas presiones

## Características de la línea

- Tamaño de haz  $30\text{ }\mu\text{m}$  -  $1\text{ mm}$
- Difracción de polvo en celdas de yunque de diamante.

Detector de área  
Rayonix 165 mm

Espejos focalizadores



Difractómetro  
Huber stages



The MSPD beamline will serve the scientific needs of the Spanish user community in the field of powder diffraction at high pressures.

We are preparing the users laboratories and finalizing the design details of the high pressure endstation. The installation and first comissioning will take place during the second half of this year.

... and we are waiting for the resolution of the research grant to equip the endstation with a laser heating set-up [*Proyectos de infraestructuras científico-tecnológica cofinanciadas con el FEDER*]

We are open for discussion, your comments will be welcomed!

# CELDAS PARIS/EDIMBURGO

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Domingo Martínez

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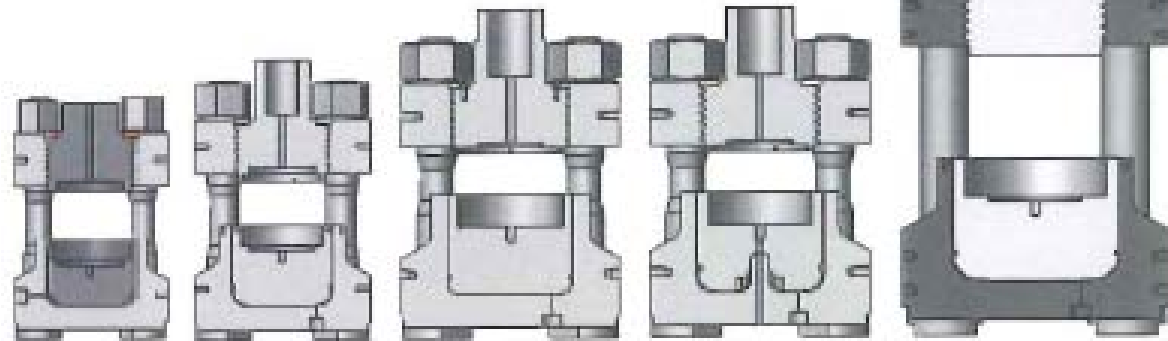
# Índice

---

- ✓ Características de las prensas PE
- ✓ Uso en grandes instalaciones
- ✓ La prensa PE de Valencia

# Características de las prensas PE

## Type V :



**V11**  
Piston Ø80  
50 t  
16 kg  
T → 100K

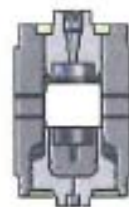
**V5**  
Piston Ø92  
150 t  
30 kg  
T → 100K

**V3**  
Piston Ø114  
250 t  
50 kg  
T → 130K

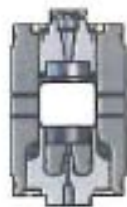
**V4**  
Piston Ø114  
250 t  
50 kg  
T → 130K

**V7**  
Piston Ø150  
450 t  
100 kg  
T → 100K

## Type VX :



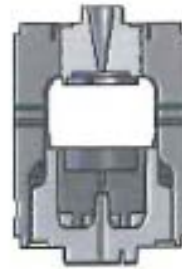
**VX1**  
Piston Ø50  
50 t  
16 kg



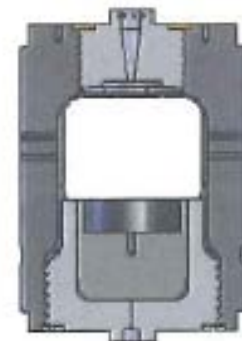
**VX2**  
Piston Ø50  
50 t  
16 kg



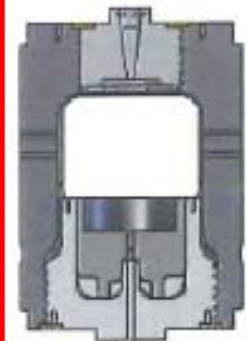
**VX5**  
Piston Ø92  
150 t  
30 kg  
T → 3 K



**VX6**  
Piston Ø92  
150 t  
30 kg



**VX3**  
Piston Ø114  
250 t  
60 kg

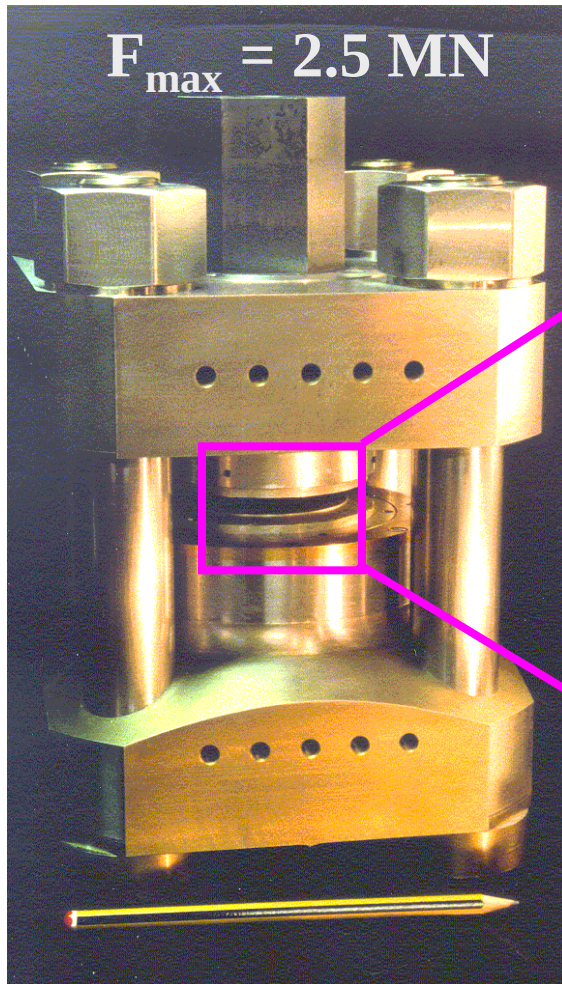


**VX4**  
Piston Ø114  
250 t  
60 kg

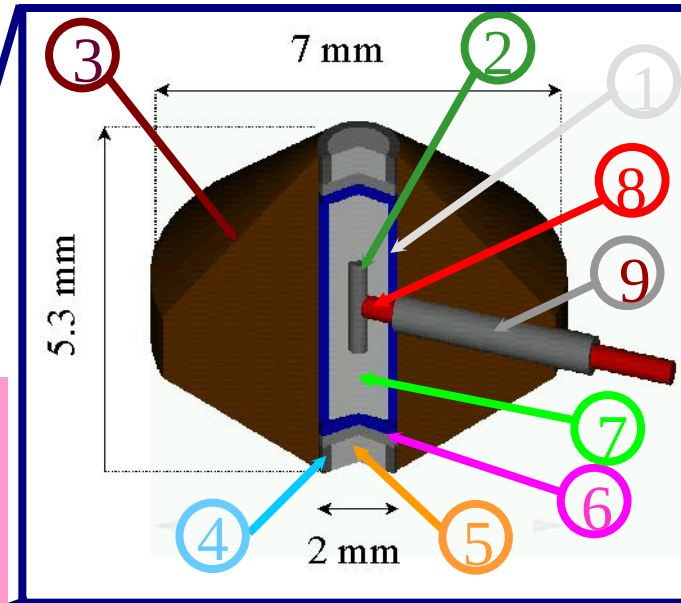
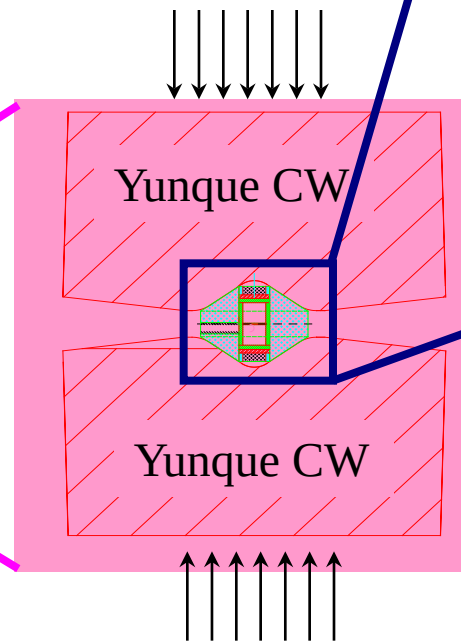


# Características de las prensas PE

## Configuración 2 yunques



V3 (50 kg)



$$P_{\max} \approx 12 \text{ GPa}$$
$$T_{\max} \approx 2000 \text{ K}$$

- 1: horno de grafito
- 2: muestra
- 3: junta de a-B
- 4: electrodo de acero
- 5: cerámica (MgO)
- 6: disco de Mo
- 7: cápsula de BN
- 8: termopar
- 9: tubo de acero

# Características de las prensas PE

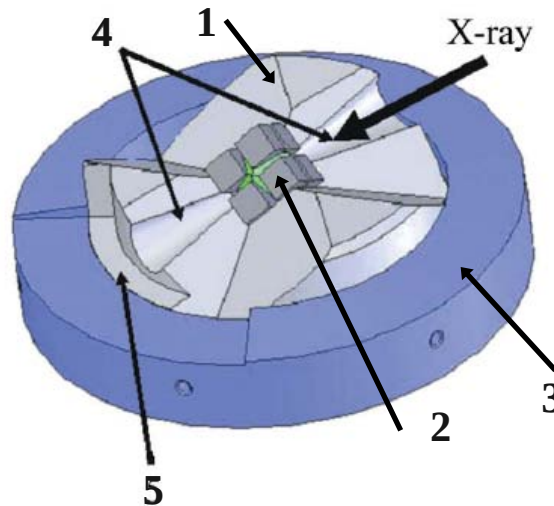
## Configuración multiyunques (T-Cup)

$$F_{\max} = 4.5 \text{ MN}$$

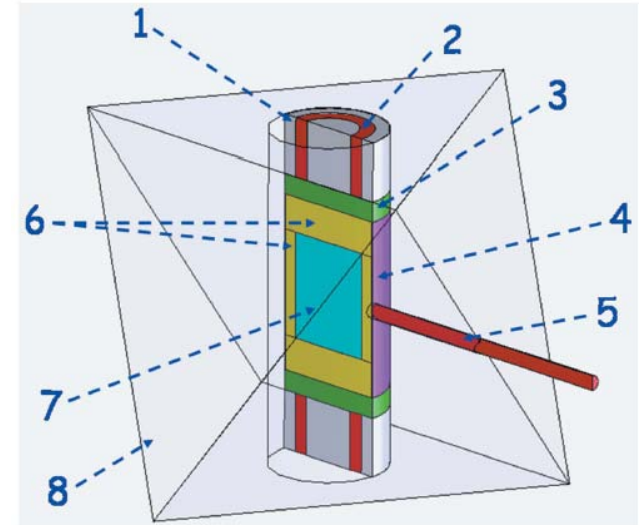


V7 (100 kg)

- 1: yunques primarios
- 2: yunques secundarios
- 3: anillo de sujeción
- 4: canales para X-ray
- 5: rebaje para rendijas



$$P_{\max} \approx 25 \text{ GPa}$$
$$T_{\max} \approx 2000 \text{ K}$$



- 1: cerámica (MgO)
- 2: contacto eléctrico
- 3: disco de Mo
- 4: horno (Re foil)
- 5: termopar
- 6: cápsula de MgO
- 7: muestra
- 8: octaedro a-B

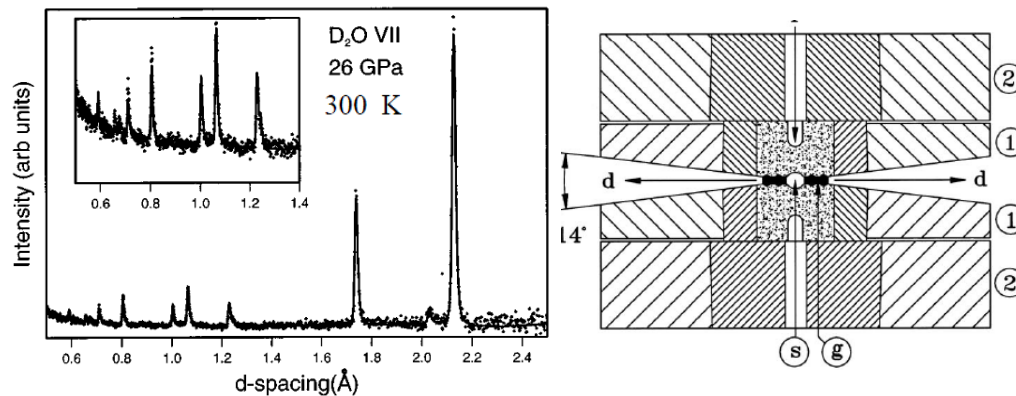
# Uso en grandes instalaciones

## Difracción de neutrones

### Dispersión de energía

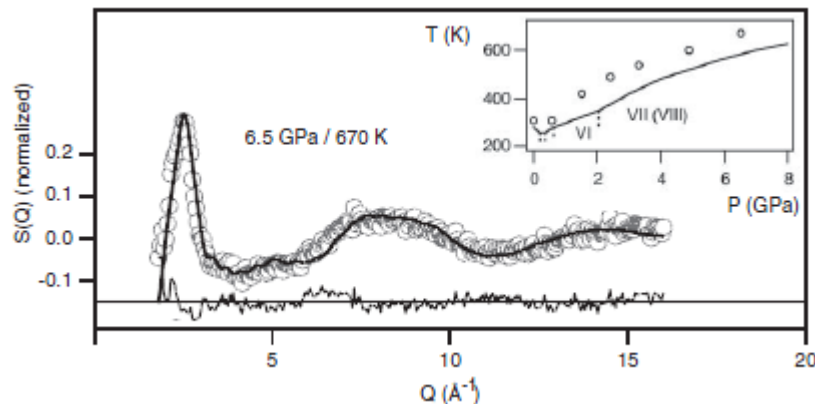
Neutron powder diffraction at pressures beyond 25 GPa

Klotz *et al.* Appl. Phys. Lett. 66 (14), 3 April 1995

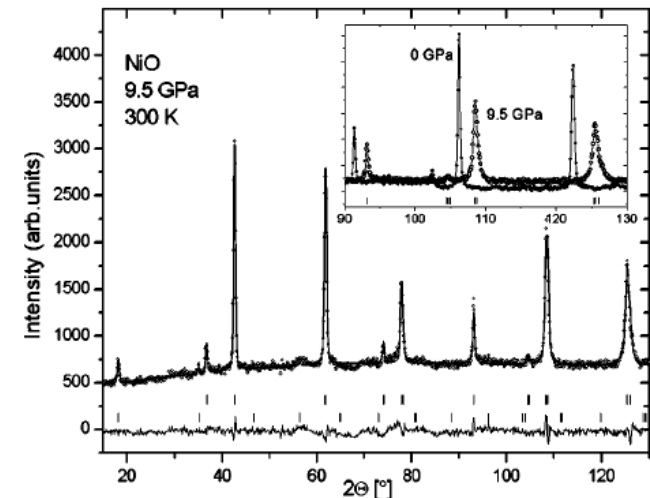
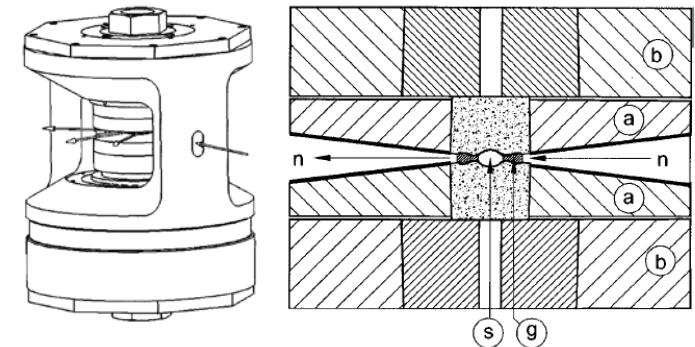


Structure of Dense Liquid Water by Neutron Scattering to 6.5 GPa and 670 K

Strässle *et al.* PRL 96, 067801 (2006)



### Dispersión angular



Angle-dispersive neutron diffraction under high pressure to 10 GPa

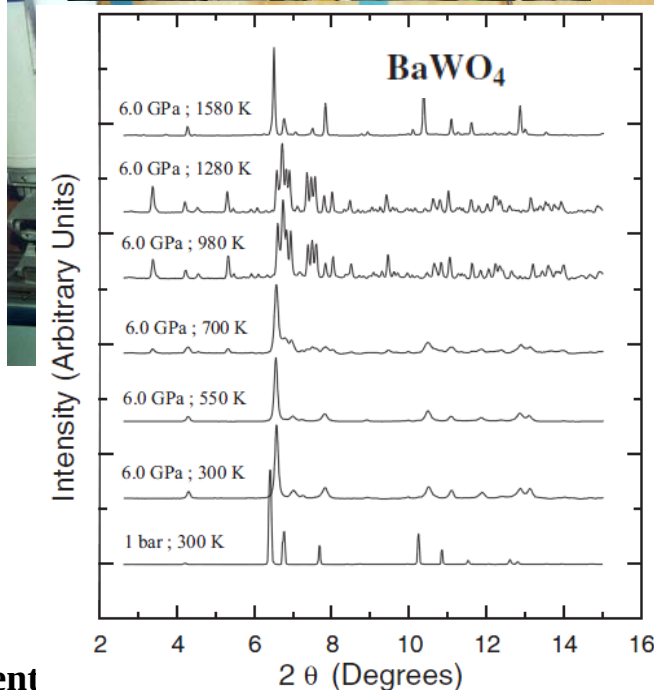
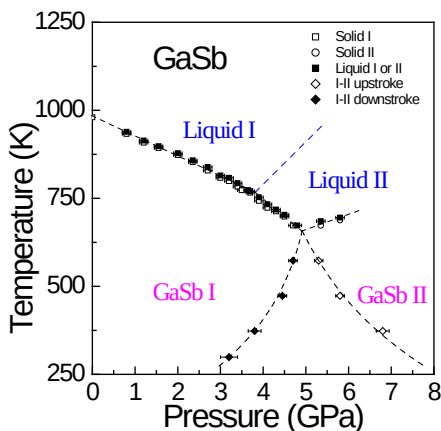
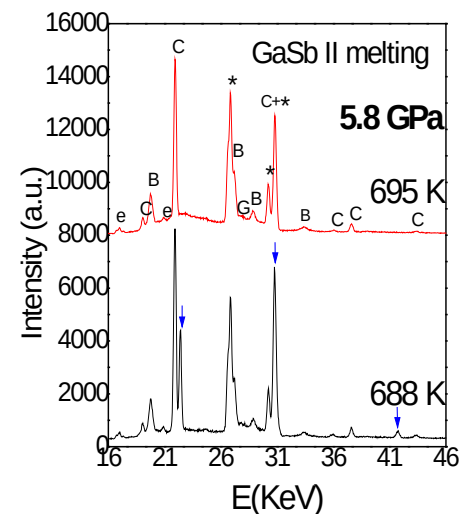
Klotz *et al.* Appl. Phys. Lett. 86, 031917 (2005)



# Uso en grandes instalaciones

## Difracción de rayos X: diagramas p-T

### Dispersión de energía

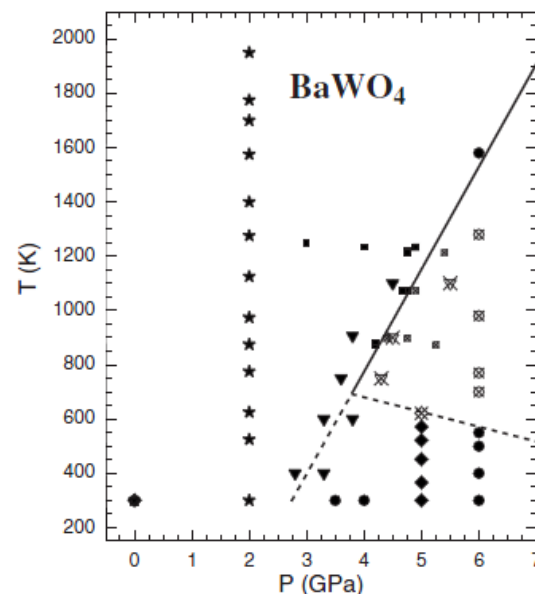


### Dispersión angular

Experimental and theoretical investigation of the stability of the monoclinic BaWO<sub>4</sub>-II phase at high pressure and high temperature

LACOMBA-PERALES *et al.*

PHYSICAL REVIEW B 81, 144117 (2010)



### X-Ray Diffraction Measurement

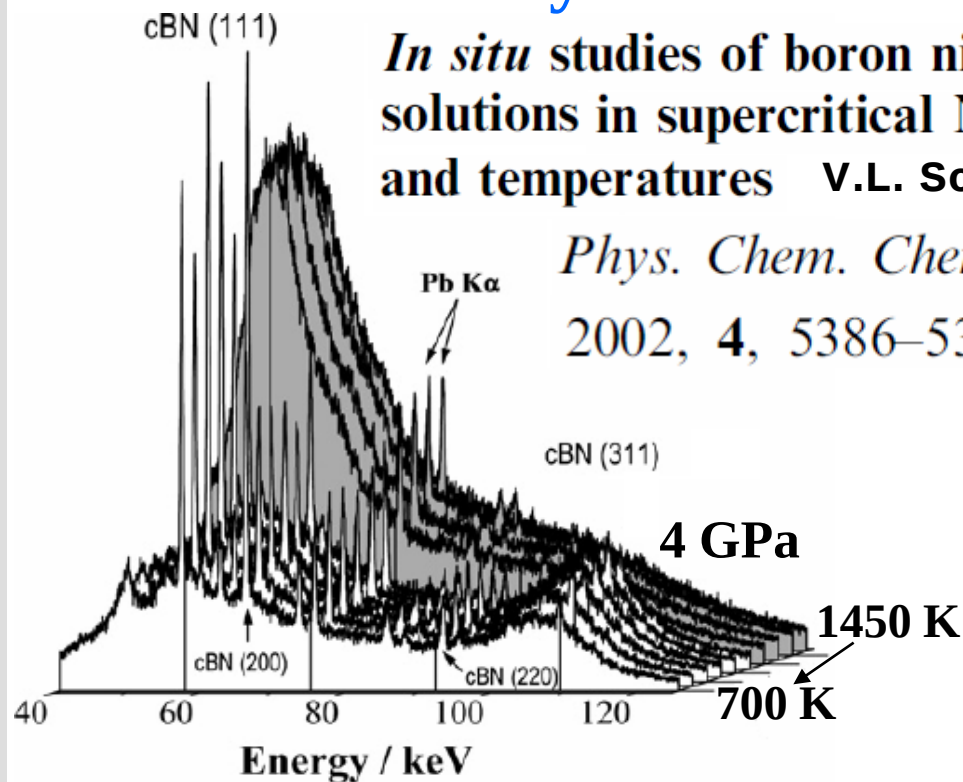
in GaSb under High Pressure and Temperature

Martínez-García et al. *phys. stat. sol. (b)* 211, 475 (1999)



# Uso en grandes instalaciones

## Difracción de rayos X: síntesis



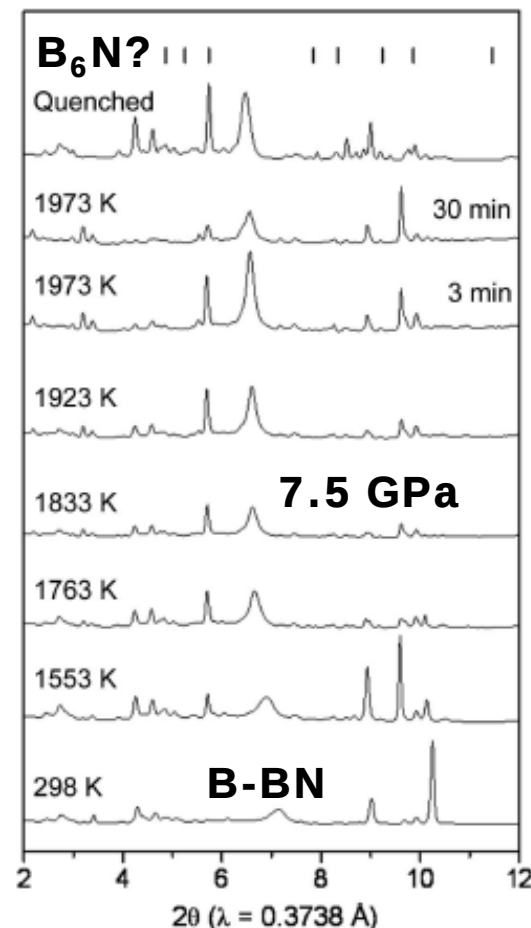
*In situ* studies of boron nitride crystallization from BN solutions in supercritical N–H fluid at high pressures and temperatures V.L. Solozhenko et al.

*Phys. Chem. Chem. Phys.*,  
2002, **4**, 5386–5393

Portable multi-anvil device for *in situ* angle-dispersive synchrotron diffraction measurements at high pressure and temperature

Y. Le Godec, G. Hamel, V.L. Solozhenko, D. Martínez-García, J. Philippe, T. Hammouda, M. Mezouar, W. A. Crichton, G. Morard and S. Klotz

*J. Synchrotron Rad.* (2009). **16**, 513–523

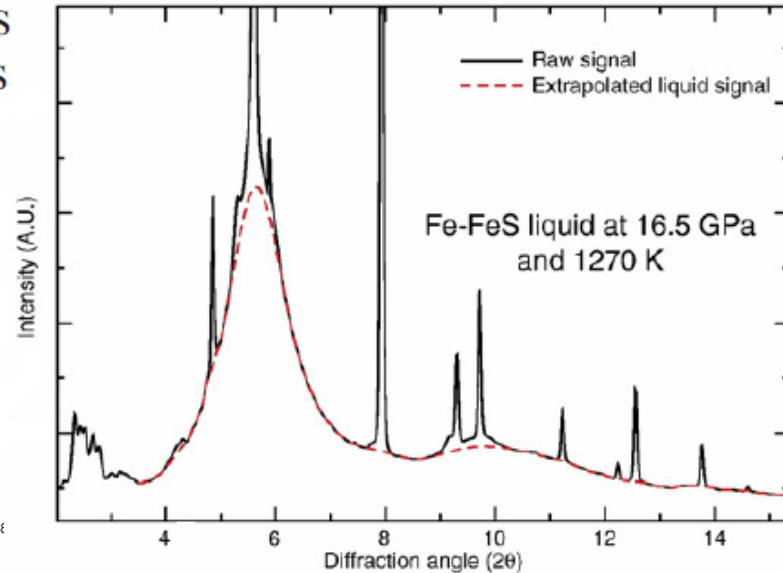
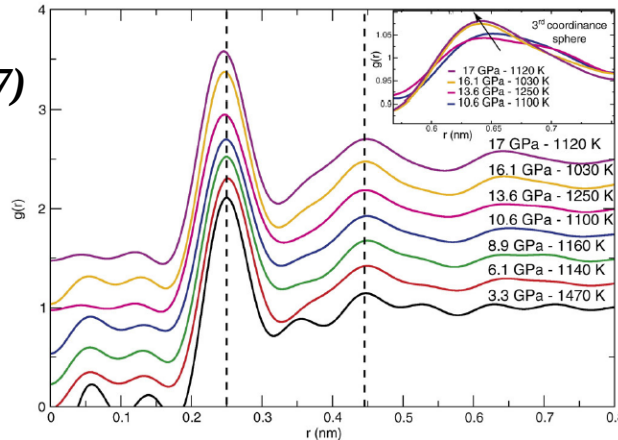


# Uso en grandes instalaciones

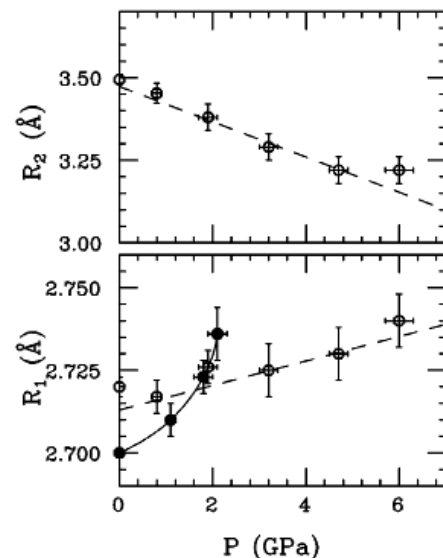
## Difracción y absorción de rayos X: líquidos

Structure of eutectic Fe–FeS melts to pressures up to 17 GPa: Implications for planetary cores

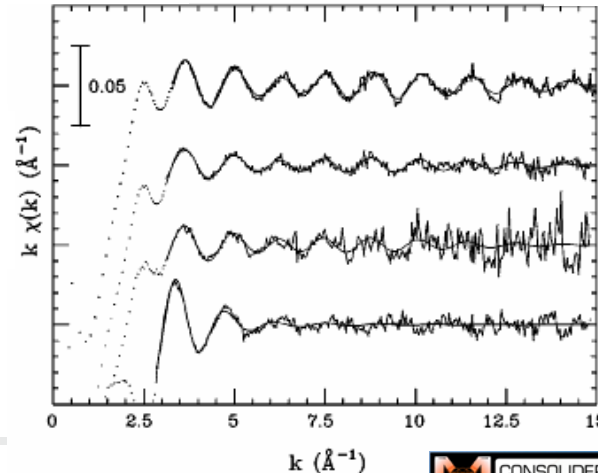
*G. Morard et al.*  
*EPSL 263, 128 (2007)*



## Anomalous Bond Length Expansion in Liquid Iodine at High Pressure



*Buontempo et al PRL 80,1912(1998)*



# Uso en grandes instalaciones

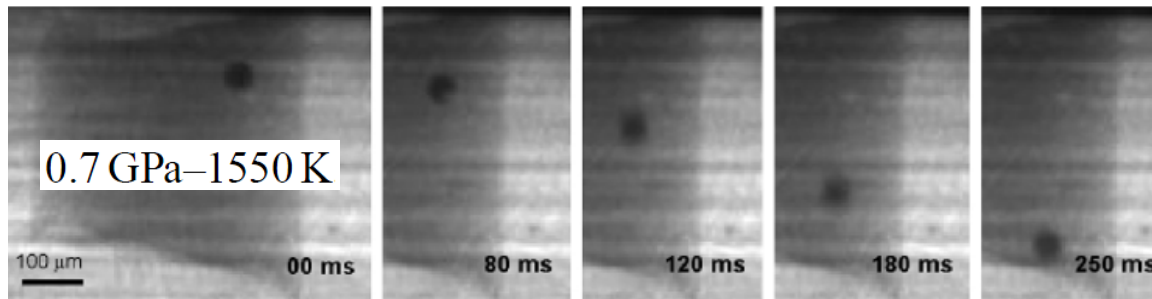
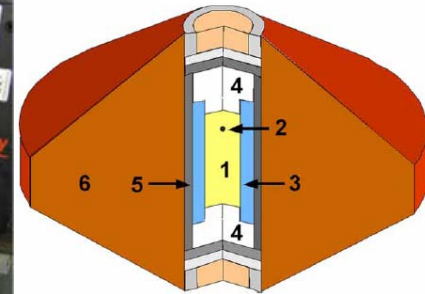
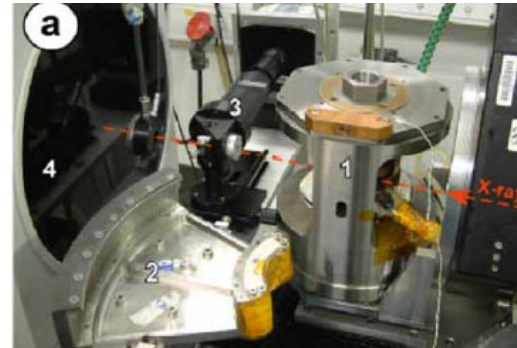
## Otras medidas

### *X-ray tomography*

*In situ* viscometry of high-pressure melts in the Paris–Edinburgh cell: application to liquid FeS

Jean-Philippe Perrillat<sup>a\*</sup>†, Mohamed Mezouar,  
Gaston Garbarino and Stany Bauchau

*High Pressure Research* Vol. 30, 415–423, 2010



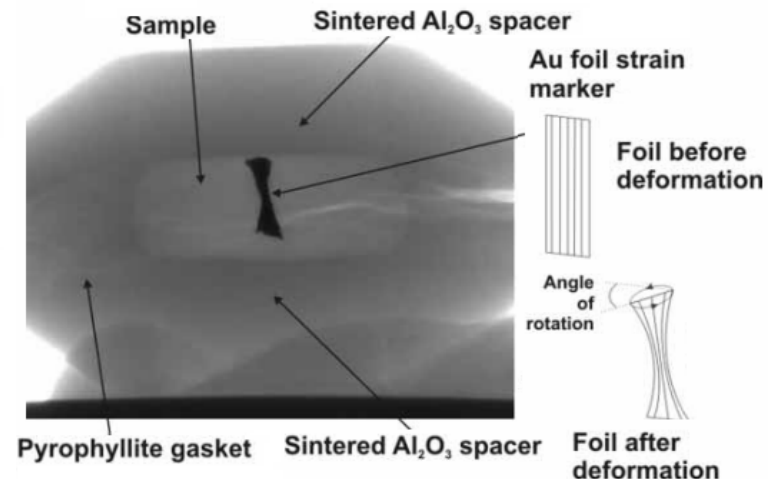
(1) Sample, (2) CW sphere,  
(3) single-crystal diamond capsule,  
(4) boron nitride,  
(5) high-resistivity graphite furnace,  
(6) boron-epoxy gasket.



**A portable high-pressure stress cell  
based on the V7 Paris–Edinburgh  
apparatus**

G. D. Bromiley<sup>a\*</sup>†, S. A.T. Redfern<sup>a</sup>,  
Y. Le Godec<sup>b</sup>, G. Hamel<sup>b</sup> and Stefan Klotz<sup>b</sup>

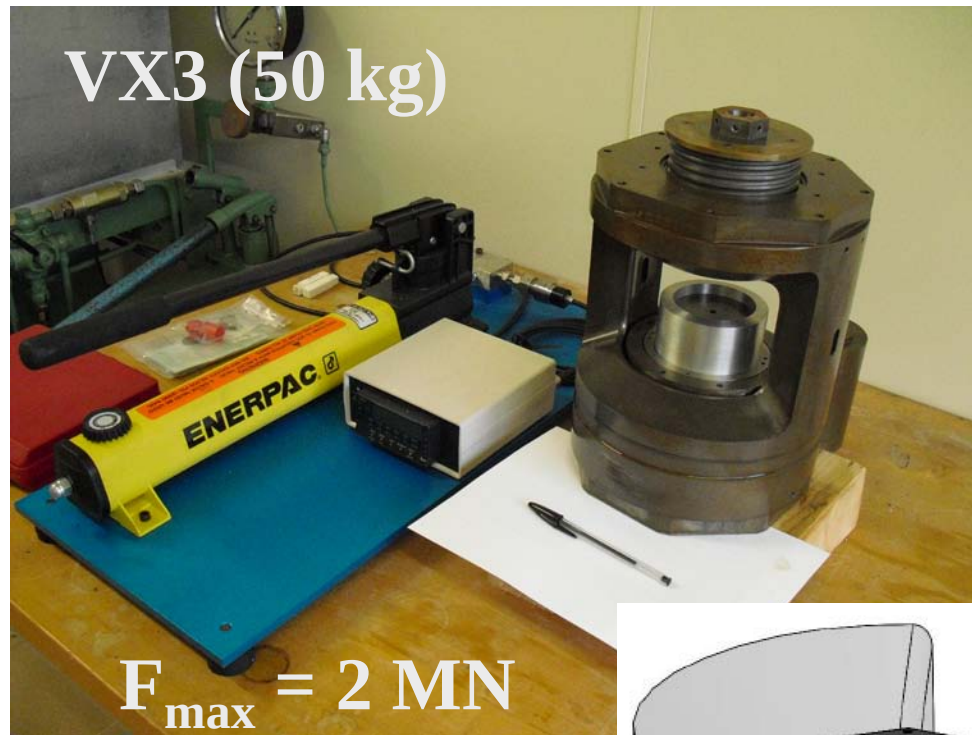
*High Pressure Research*  
Vol. 29, No. 2, June 2009, 306–316





# La prensa PE de Valencia

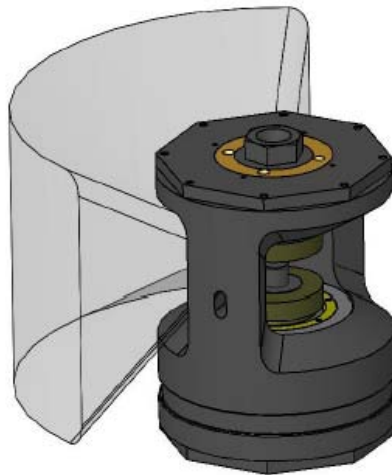
## La prensa



**Aperturas:**

**2 x 140° (horizontal)**

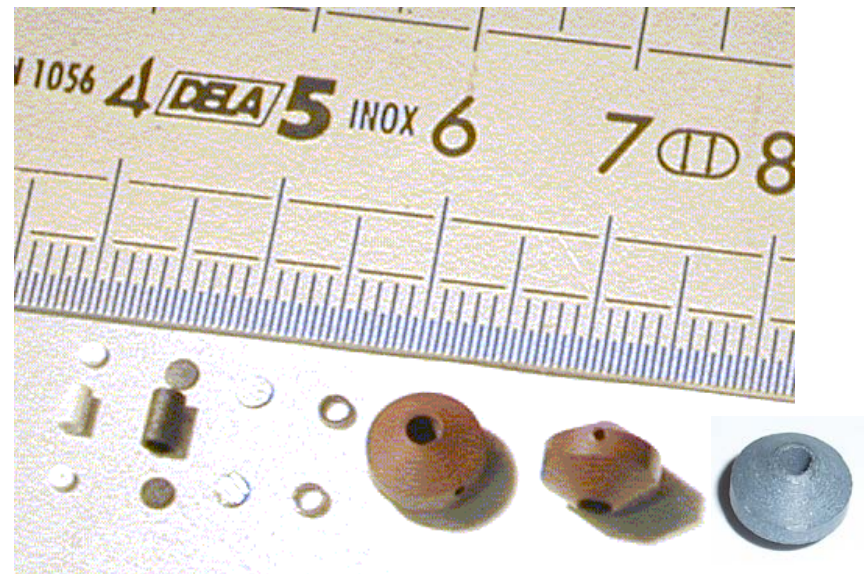
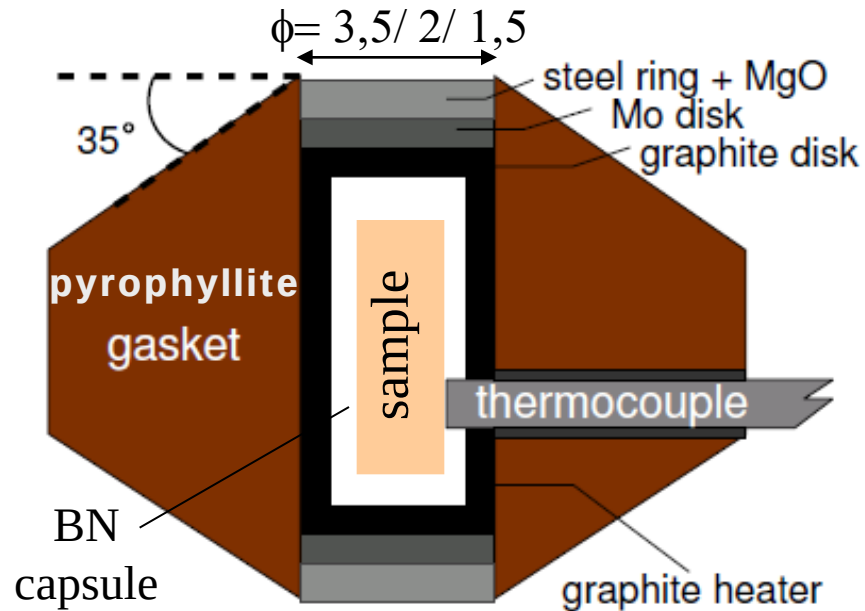
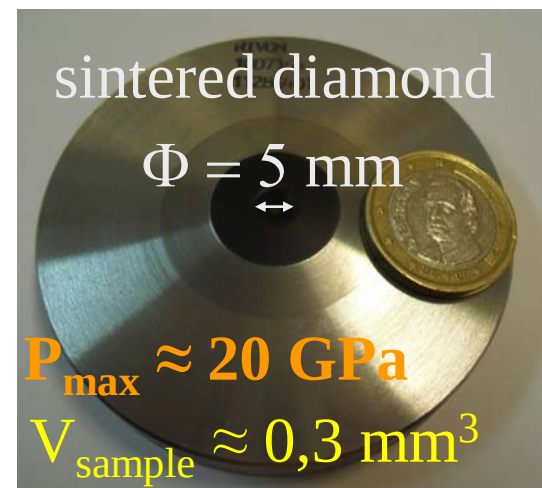
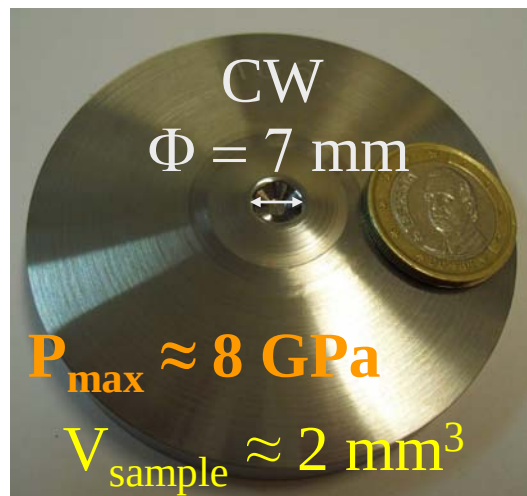
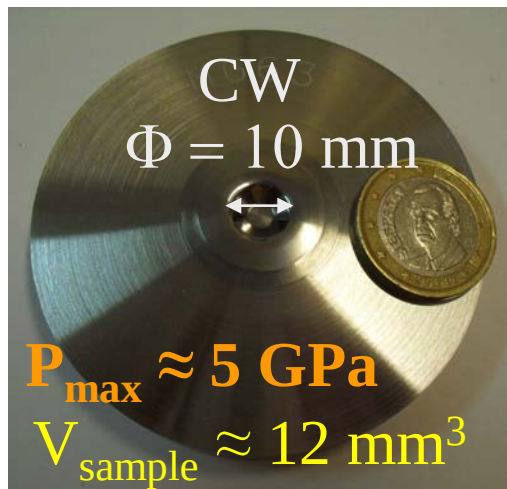
**2 x 60° (vertical)**





# La prensa PE de Valencia

## Los yunques y elementos



$T_{\text{max}} \approx 2000 \text{ K}$

# La prensa PE de Valencia

## Posibilidades

### En laboratorio

- *Resistencia eléctrica ( $p, T$ )*
- *Análisis térmico diferencial (DTA) (?)*
- *Síntesis HP-HT + caracterización ex situ:*
  - Difracción de rayos X*
  - Raman*
  - Microscopía electrónica*
  - Medidas ópticas (?)*

### En grandes instalaciones

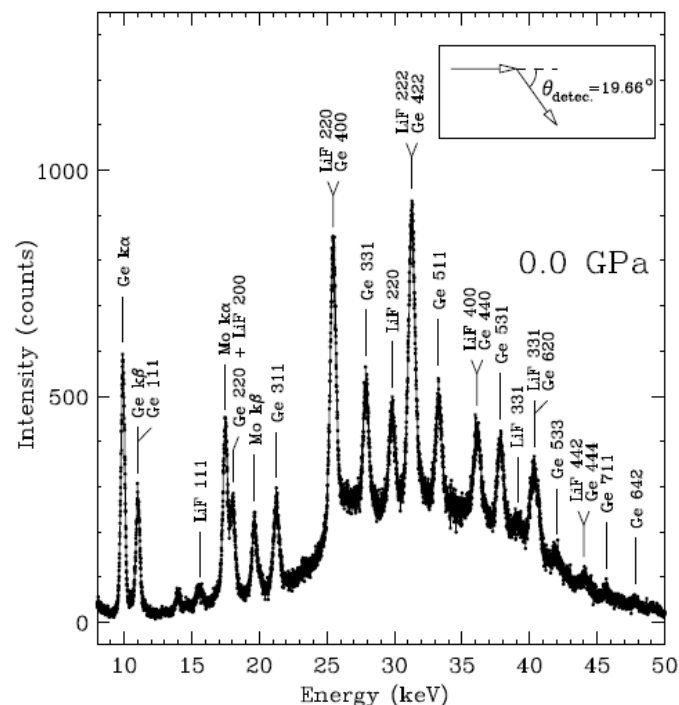
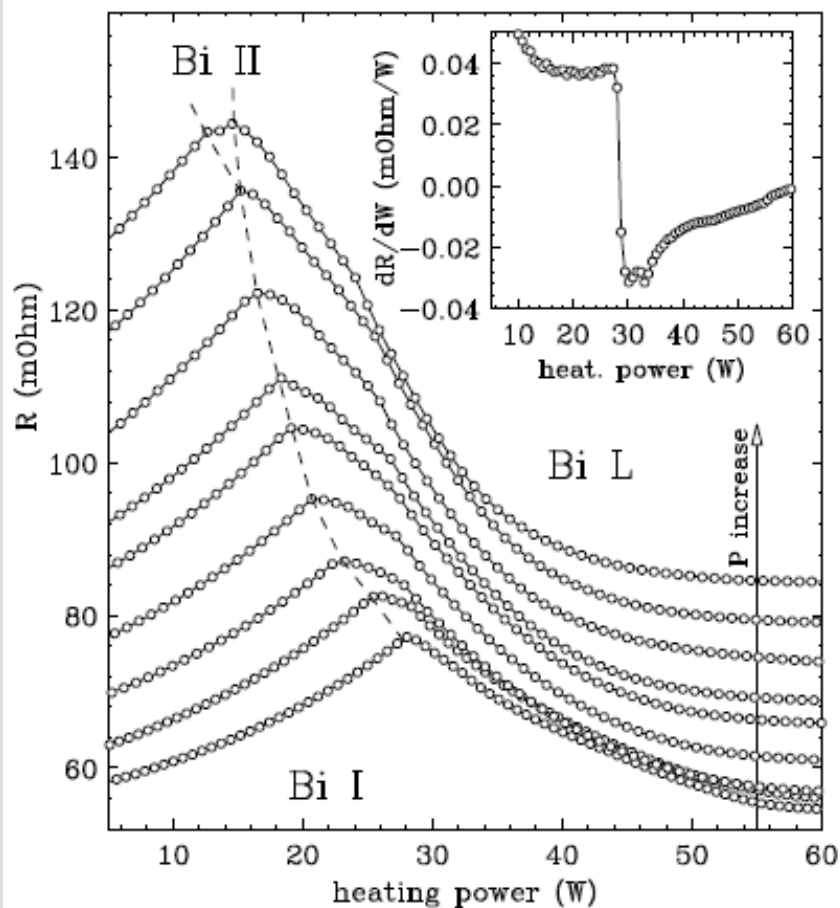
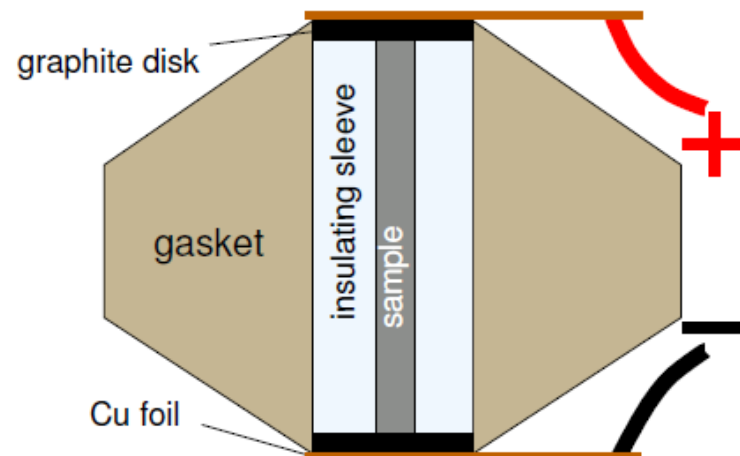
- *Difracción de rayos X (in situ): ESRF, SOLEIL, ALBA*
- *Absorción de rayos X (in situ): ESRF*
- *Difracción de neutrones: ILL, XTREMED*

***¡GRACIAS  
POR VUESTRA  
ATENCIÓN!***

# X-ray diffraction and electroresistance measurement under high pressure and temperature using a large-volume cell

E. Principi, M. Minicucci, A. Di Cicco\*

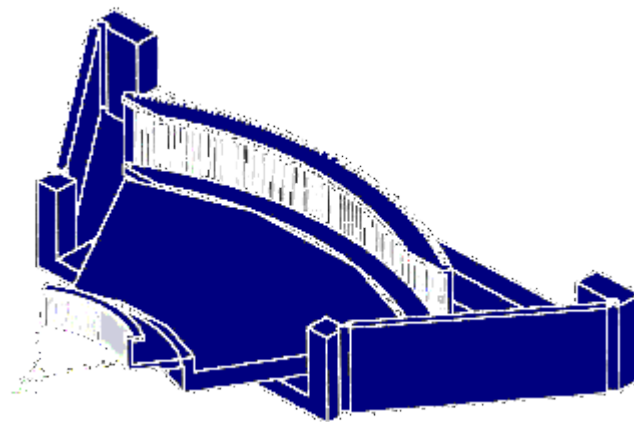
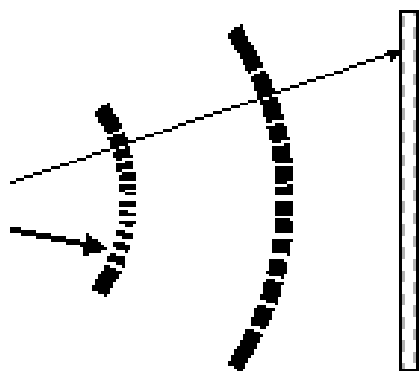
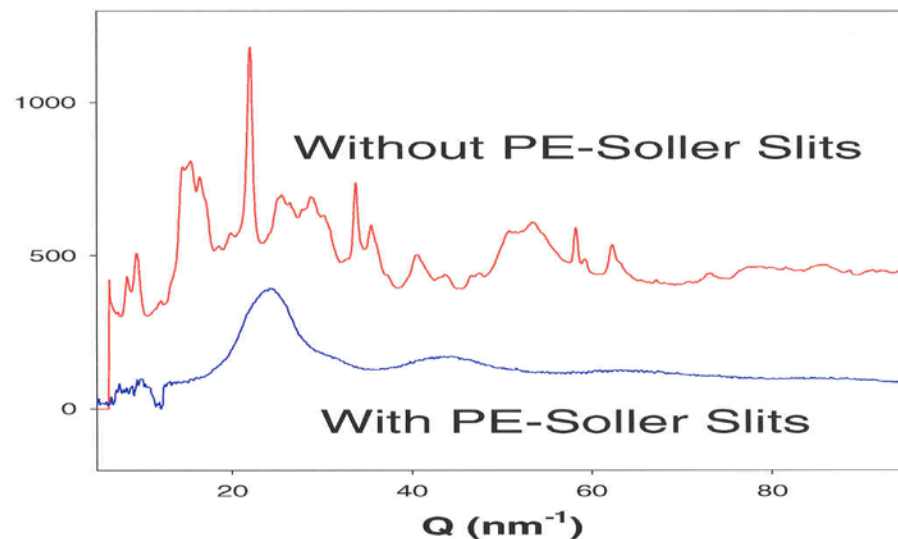
Journal of Physics: Conference Series **121** (2008) 042010



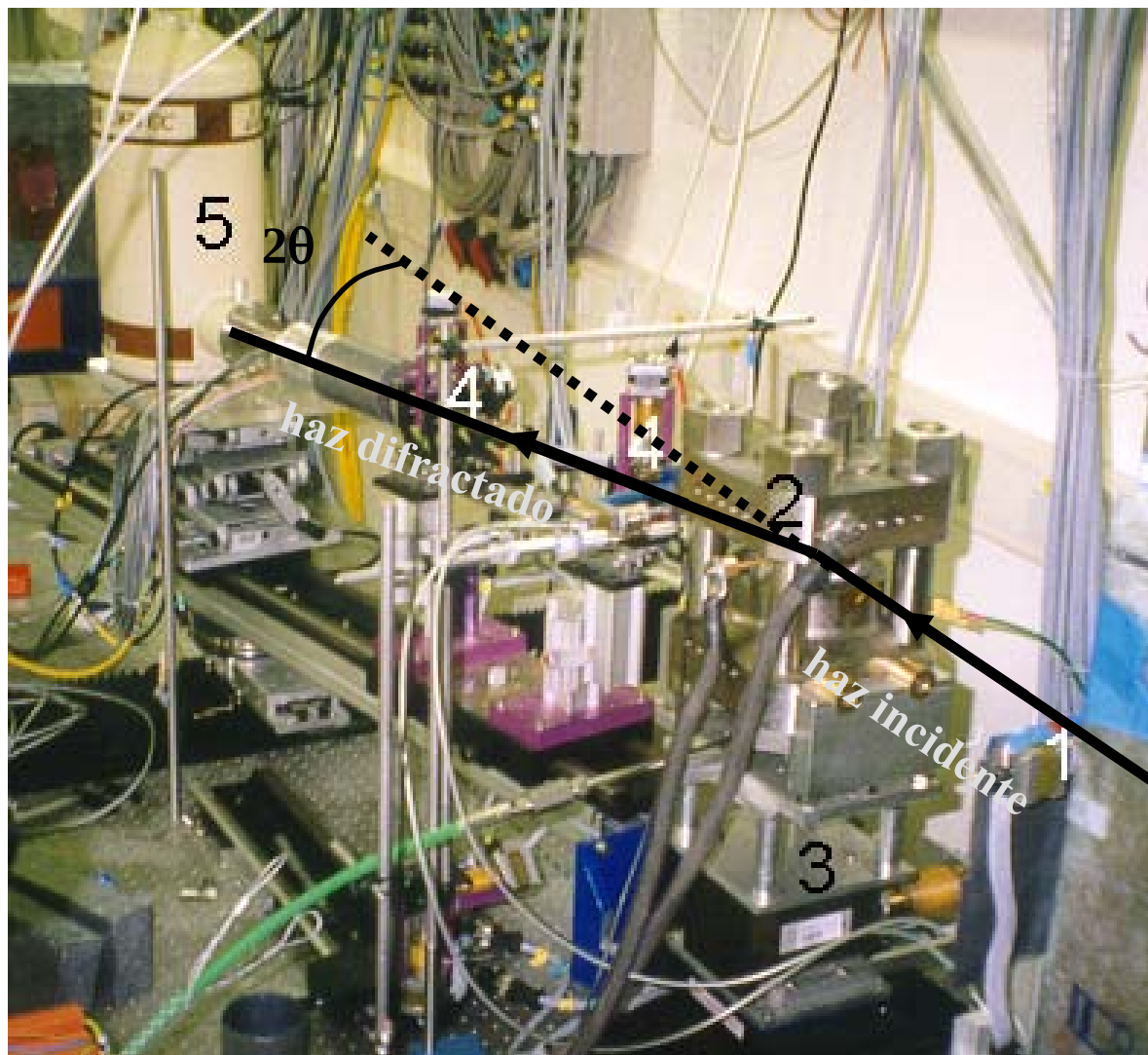


# Multichannel slits system

In monochromatic mode the high background coming from high pressure cells (boron gasket, graphite heater, h-BN) strongly deteriorates the data quality



IP



# Buenas tardes

## Introducción

1º/ Métodos para aplicar la fuerza

2º/ origine y ventajas de la celda à membrana

3º/ Celdas para aplicaciones específicas

4º/ Carga con medios hidrostáticos

Conclusiones



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# HIGH PRESSURE RESEARCH

Volume 29 • Number 2 • June 2009

Editor in Chief: Stefan Klotz  
Regional Editors: Yanbin Wang and Yoshinori Katayama  
Founding Editors: Marvin Ross and Jean-Michel Besson

**50 Years  
Diamond  
Anvil Cell**

**W. Bassett**

**Keynote  
article,  
pp. xx-xx**



ISSN 0895-7959



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## Calibration of the pressure dependence of the $R_1$ ruby fluorescence line to 195 kbar

G. J. Piermarini, S. Block, J. D. Barnett,\* and R. A. Forman

*Institute for Materials Research, National Bureau of Standards, Washington, D. C. 20234*

(Received 29 January 1975)

The pressure dependence of the  $R_1$  ruby fluorescence line has been calibrated at 25 °C against the compression of NaCl. Pressures are determined using the Decker equation of state for NaCl. The dependence is linear to 195 kbar following the equation  $P_{\text{NaCl}} = 2.746(\Delta\lambda)$ , where  $P$  is in kbar and  $\Delta\lambda$  in Å. The uncertainty in the value of the slope,  $dp/d\lambda$ , expressed in terms of a 95% confidence interval is  $2.746 \pm 0.014$  kbar Å<sup>-1</sup>. The coefficient of the quadratic term  $(\Delta\lambda)^2$  is not significantly different from zero; and the quadratic term makes indeed a negligible contribution to the fit. Taking into account the reported uncertainty associated with the Decker equation of state for NaCl, the value of the slope is  $2.740 \pm 0.016$  kbar Å<sup>-1</sup> within a 95% confidence interval.

PACS numbers: 78.60.D, 71.70.E, 62.50., 06.20.H

*High Pressure Research*  
Vol. 28, No. 2, June 2008, 75–126



## Ruby under pressure

K. Syassen\*

*Max-Planck-Institut für Festkörperforschung, Stuttgart, Germany*

*(Received 17 March 2008; final version received 19 May 2008)*

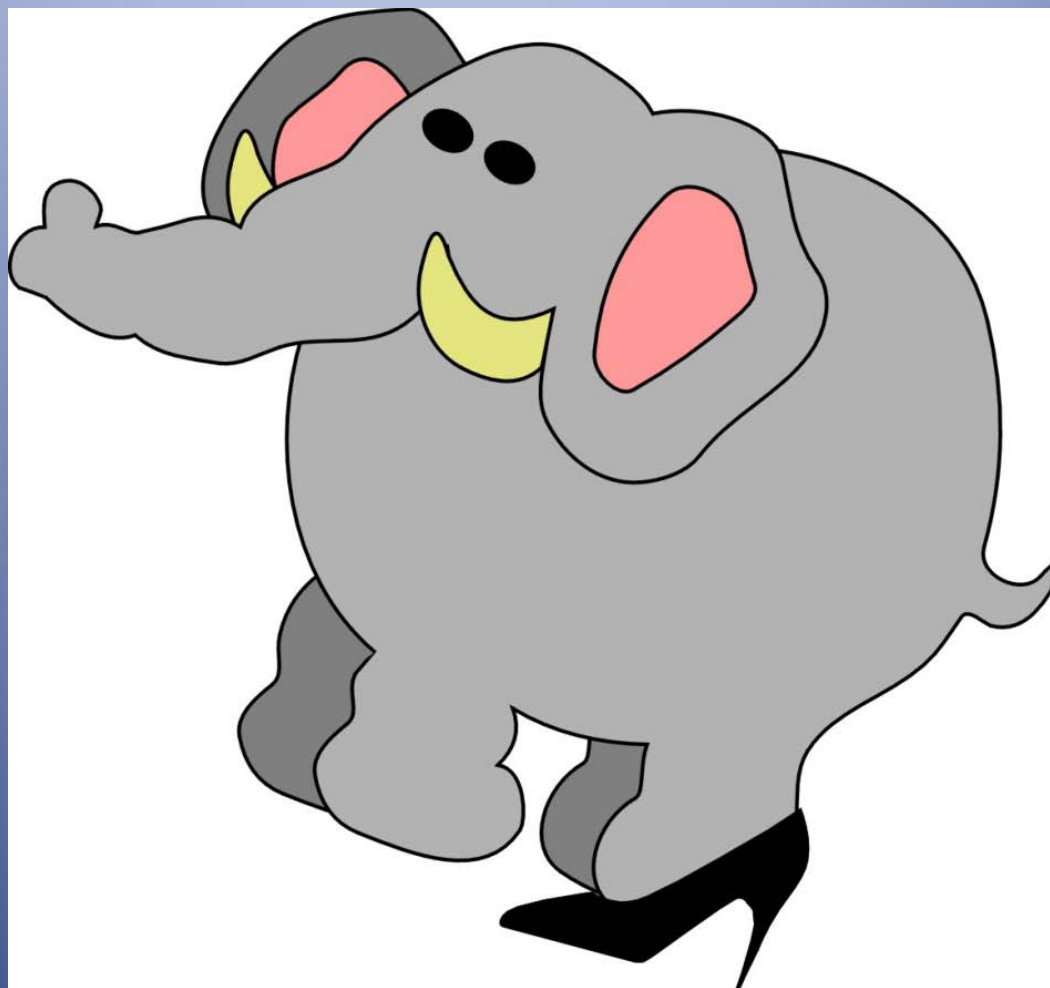
The ruby luminescence method is widely used for pressure measurement in the diamond anvil cell and other optically transparent pressure cells. With this application in mind, we briefly review the ground-state physical properties of corundum ( $\alpha\text{-Al}_2\text{O}_3$ ) with some emphasis on its behavior under high pressure, survey the effects of temperature and stress on the R-line luminescence of ruby (Cr-doped corundum), and address the recent efforts towards an improved calibration of the R-line shift under hydrostatic pressures beyond the 50 GPa mark.

**Keywords:** corundum; ruby; luminescence; high pressure; ruby pressure scale

# 1°/ Métodos para aplicar la fuerza







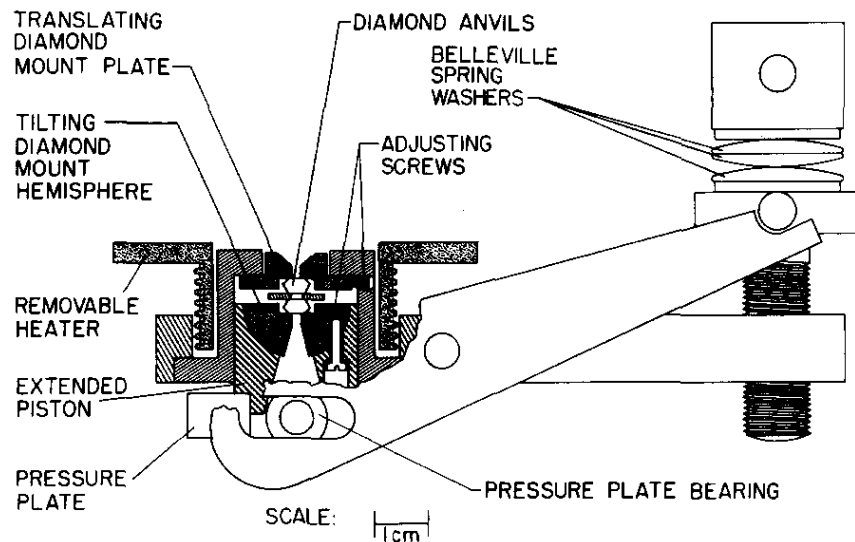
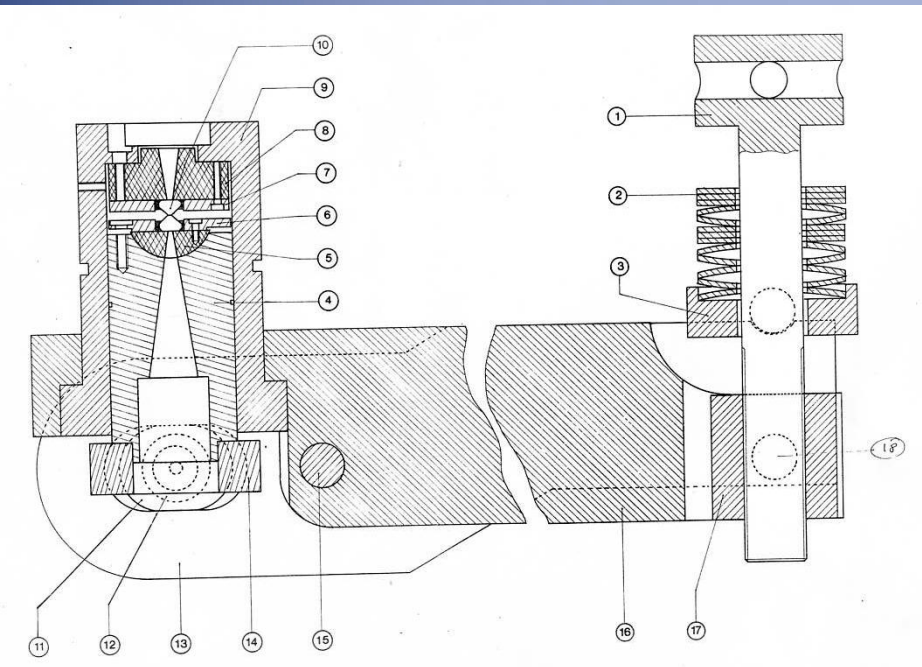
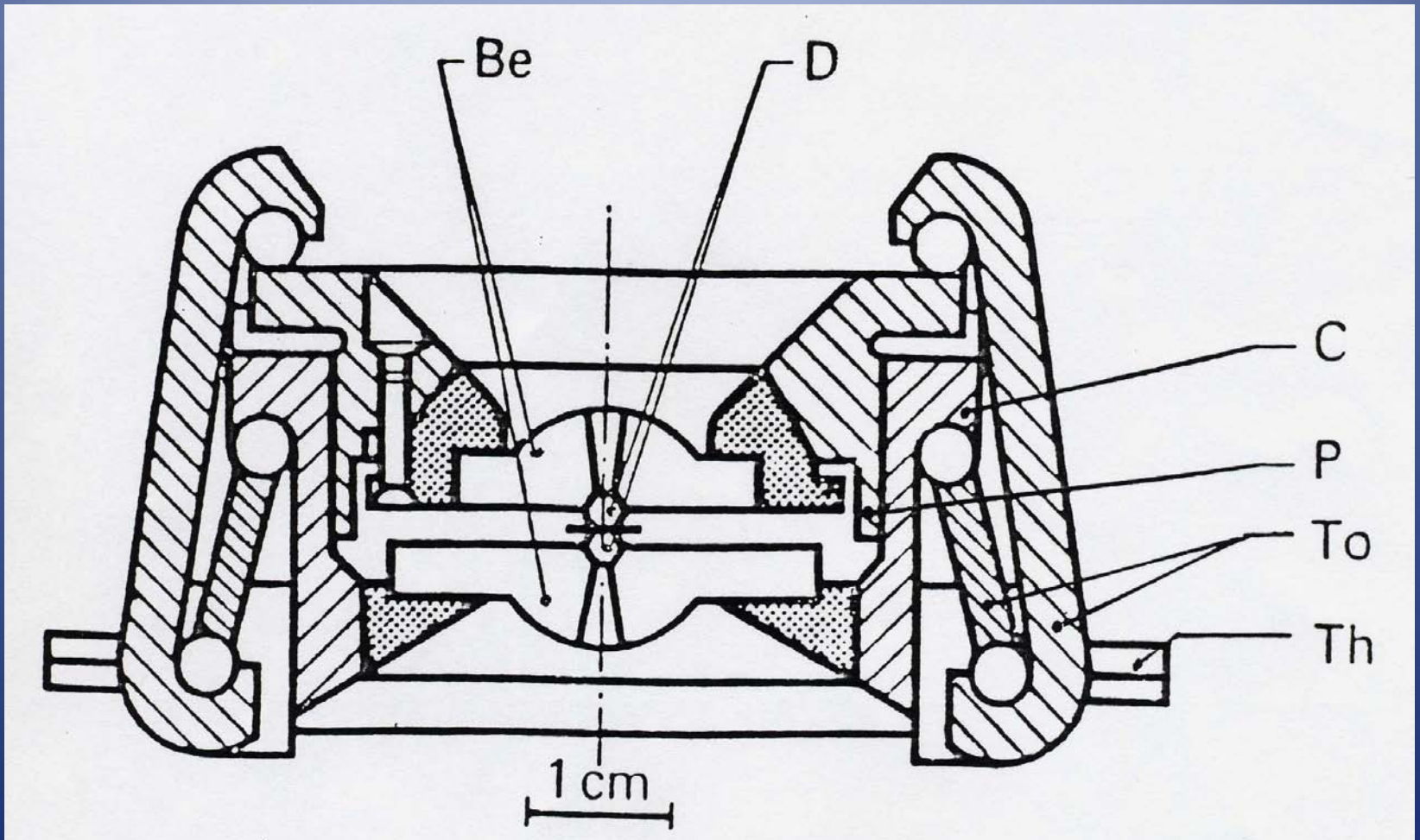


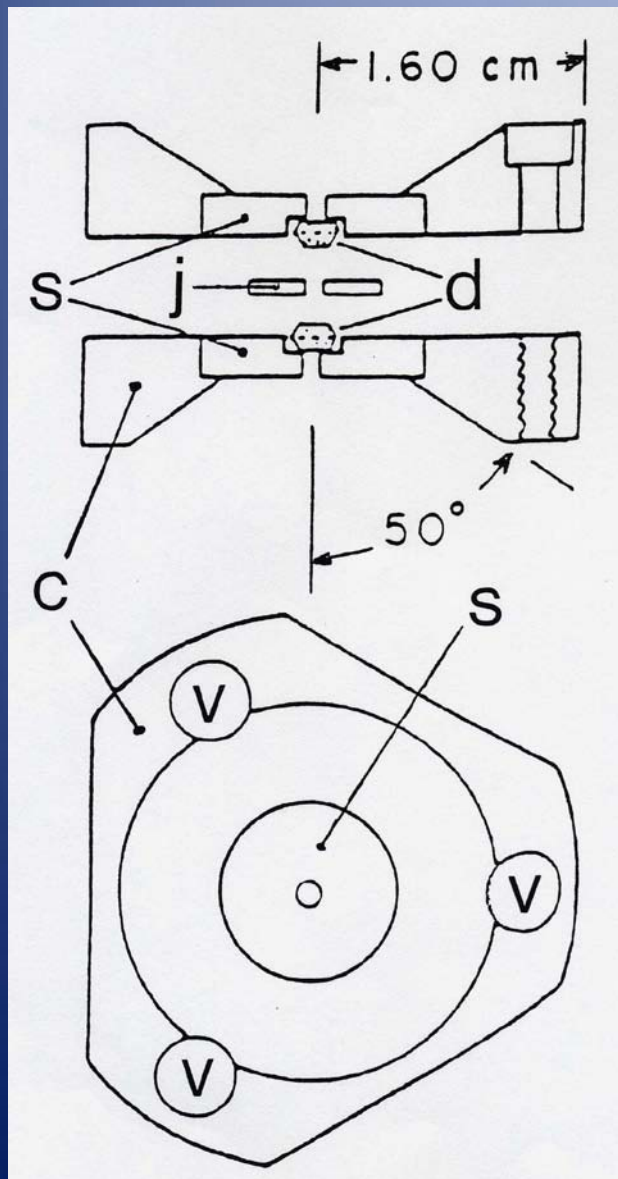
FIG. 3. Cut-away cross section drawing of the Waspaloy pressure cell showing improved alignment design, heater assembly, and spring washer configuration.











## 2º/ origine y ventajas de la celda de membrana

A diamond-anvil high-pressure cell especially designed for a standard precession camera and its operation are described. The applied force is generated by means of a pneumatic device. Single crystals grown by Van Valkenburg's technique are maintained under high pressure (1–30000 bars) at adjustable temperature (20–220°C). A steel goniometer head provides facility of centring and orienting the crystal. Some results are given, including crystal growth, optical observation of the phase transition in cyclohexane and crystal structure of chloroform at ~7000 bars. Conditions for gathering precise intensity data are discussed.

ROGER FOURME

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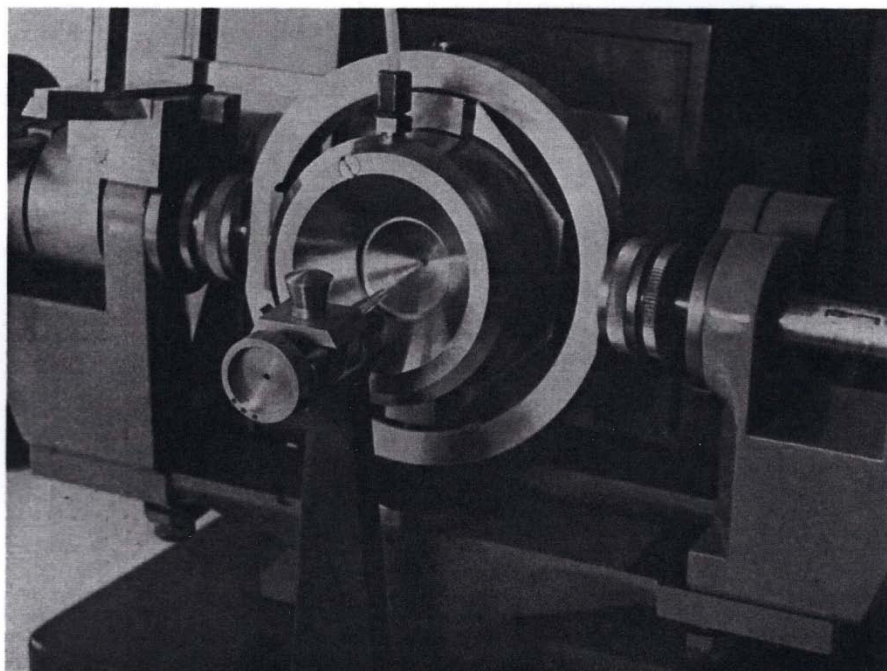
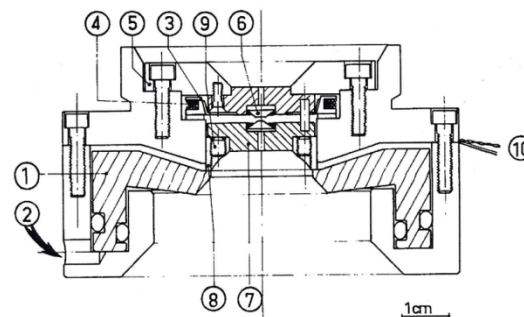


Fig. 5. Chambre à précession Nonius équipée de la cellule haute pression.



3. Schéma de la cellule haute pression. 1 piston annulaire; 2 admission d'azote comprimé; 3 fourreau; 4 dispositif de chauffage; 5 anneau amovible; 6 diamant; 7 porte-diamant (en beryllium, ou, avec une forme différente, en acier); 8 piston; 9 vis réglage; 10 fils d'alimentation électrique et du thermocouple.



## Melting of Helium at Room Temperature and High Pressure

Author(s): J. M. Besson and J. P. Pinceaux

Source: Science, New Series, Vol. 206, No. 4422 (Nov. 30, 1979), pp. 1073-1075

**Abstract.** *Helium has been solidified at room temperature. The melting pressure at 24°C is 115 kilobars, in complete agreement with the Simon equation. An original apparatus was developed for this experiment, which allows loading of the cell at room temperature. Applications to various areas of research are suggested.*

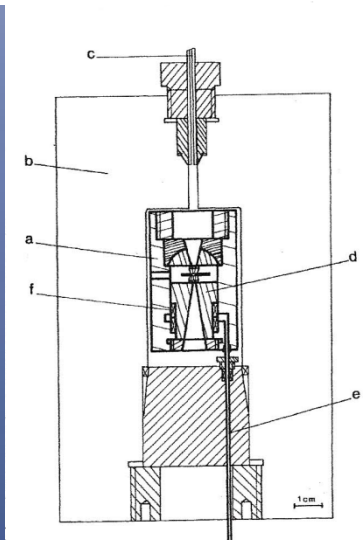


Fig. 1. Sketch of the diamond cell (dark lines) inside the low-pressure vessel (light lines): a, diamond cell (maraging steel); b, low-pressure bomb (5 kbar) (maraging steel, 52RC); c, stainless steel gas-loading capillary; d, moving piston; e, stainless steel capillary feeding the pressure control fluid (helium in this experiment); and f, piston seal.

## Simple double diaphragm press for diamond anvil cells at low temperatures

W. B. Daniels<sup>a)</sup>

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M. G. Ryschkewitsch

*Department of Physics, University of Delaware, Newark, Delaware 19711*

(Received 23 March 1982; accepted for publication 1 September 1982)

A very simple welded double diaphragm press has been constructed and tested. It is suitable for providing the forces and displacements for operation of a diamond anvil cell and requires only a small capillary connecting a helium gas pressure source and the diamond anvil cell.

115 Rev. Sci. Instrum. 54(1), Jan. 1983 0034-6748/83/010115-02\$01.30 © 1983 American Institute of Physics

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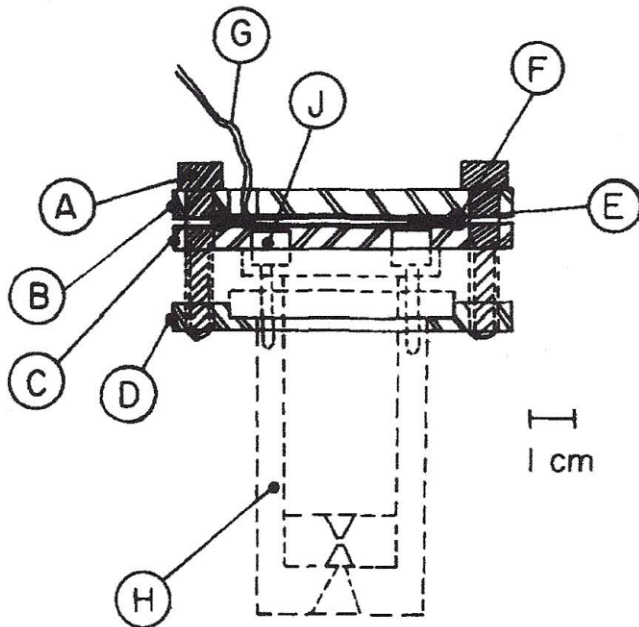


FIG. 1. Cross section of press and diamond anvil cell. (A) Ring of 8, 1/4-28 (6.3 mm) hardened steel socket head screws on 58-mm bolt circle. (B), (C), and (D) Stainless-steel plates of press. (E) Double diaphragm. (F) Relief for weld bead around edge of double diaphragm. (G) 0.8-mm-o.d.  $\times$  0.15-mm-i.d. capillary leading to gas pressure regulator. (H) Body of modified Bassett diamond anvil cell (Ref. 4). (J) Machine screws of gas loading clamp (Ref. 5).

*High Pressure Research*, 1988, Vol. 1, pp. 77-90  
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 Printed in the United Kingdom

# **THE MEMBRANE DIAMOND ANVIL CELL: A NEW DEVICE FOR GENERATING CONTINUOUS PRESSURE AND TEMPERATURE VARIATIONS**

R. LETOULLEC, J. P. PINCEAUX and P. LOUBEYRE

*Laboratoire de Physique des Milieux Condensés\*, T13 E4, Université Paris 6,  
 4 place Jussieu, 75252 Paris, France*

A new design for a diamond anvil cell is described. Its main originality is that the force on the piston is generated by pressurized helium, which pushes an annular membrane. It specially permits fine control and adjustment of the force applied to the anvils, spatial stability of the sample under varying pressure, axial thrust and large optical aperture on both sides of the cell. Selected applications are presented.

**KEY WORDS:** New device, membrane diamond anvil cell, refractive index, optical measurements.

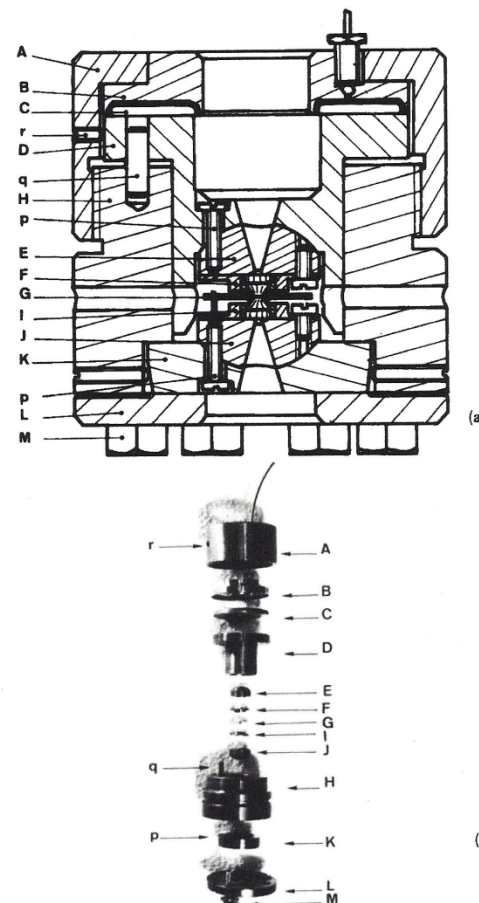
## **1. INTRODUCTION**

The diamond anvil cell (DAC) is a fine quantitative tool, now extensively used for modern high-pressure physics. Its different designs with their specific characteristics have been reviewed by Jayaraman<sup>1,2</sup>.

The main improvement of the diamond anvil cell over other high-pressure apparatuses comes from the fact that diamond is optically transparent over a wide wavelength domain and is still the hardest and strongest known material. This explains why DAC studies are essentially based on optical investigations, from the direct observation of phenomena to light-scattering techniques such as Raman, Brillouin, and infra-red or X-ray measurements; however, these had to be adapted to the study of the microvolumes available in the gasket hole ( $10^{-4}$  mm<sup>3</sup>), which is a common limitation of all DAC.

THE MEMBRANE DIAMOND ANVIL CELL

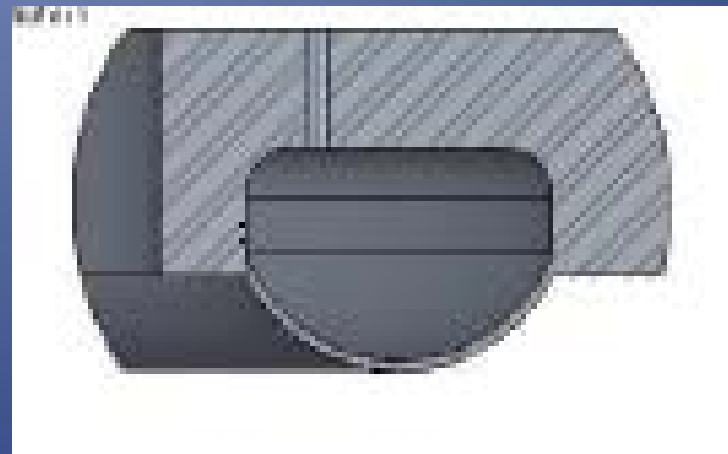
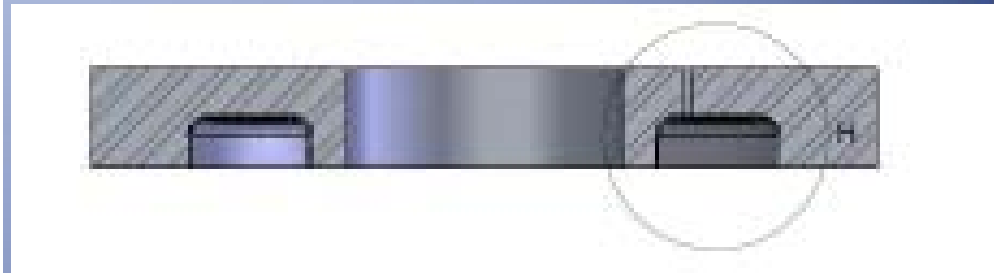
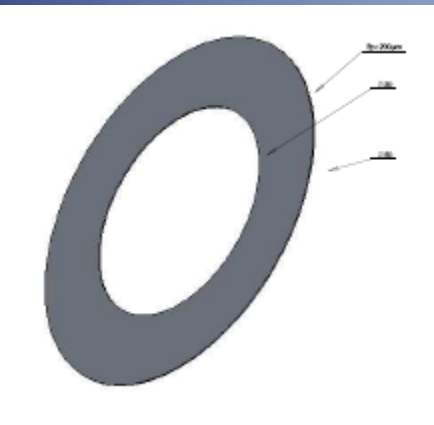
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**Figure 1** The design of the membrane diamond anvil cell is shown in Figure 1(a). Figure 1(b) is a photograph showing its different parts: (A) retaining nut; (B) the support on which the membrane is soldered; (C) push-piece to transmit the force to piston (D); (E and J) half-sphere seats of the diamonds; (G) the gasket; (H) cell body; (I and F) copper pieces in which the diamond anvils are inserted; (K and L) supports of the fixed anvil seat; (M, p and r) screws; (q) a stop.









# 3°/ Celdas para aplicaciones específicas

## Micro-Raman at variable low-temperature and very high pressure

J. C. Chervin, B. Canny, M. Gauthier, and Ph. Pruzan

*Physique des Milieux Condensés (CNRS, URA 0782), Université P. & M. Curie, B 77, T 13, E 4, 4, place Jussieu, F-75252 Paris Cedex 05, France*

(Received 20 May 1992; accepted for publication 14 September 1992)

A setup for micro-Raman investigation at low temperature in a membrane diamond anvil cell is described. This setup allows investigations at variable temperature from room temperature down to 10 K. For a given temperature the system permits a continuous variation in pressure. Performance of the system is illustrated with investigations on benzene and deuterated ice which required pressure cycling (at low temperature) or temperature cycling (at high pressure).





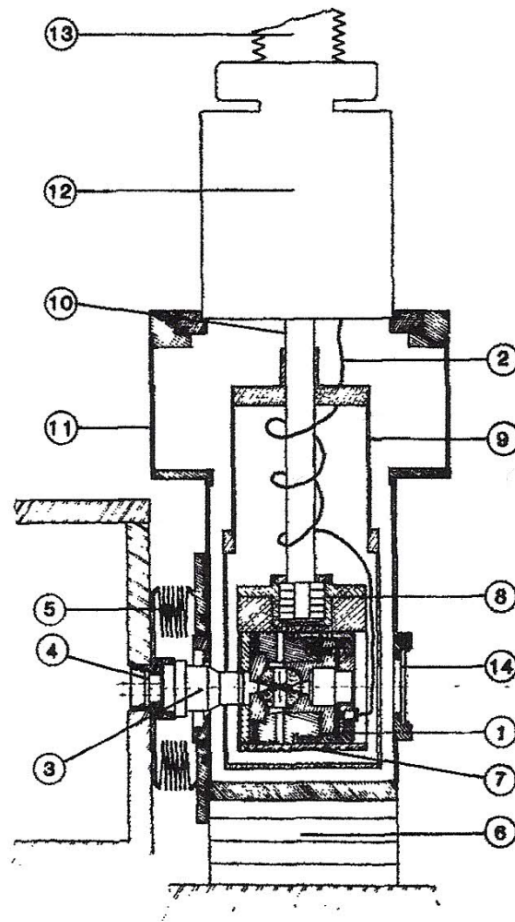
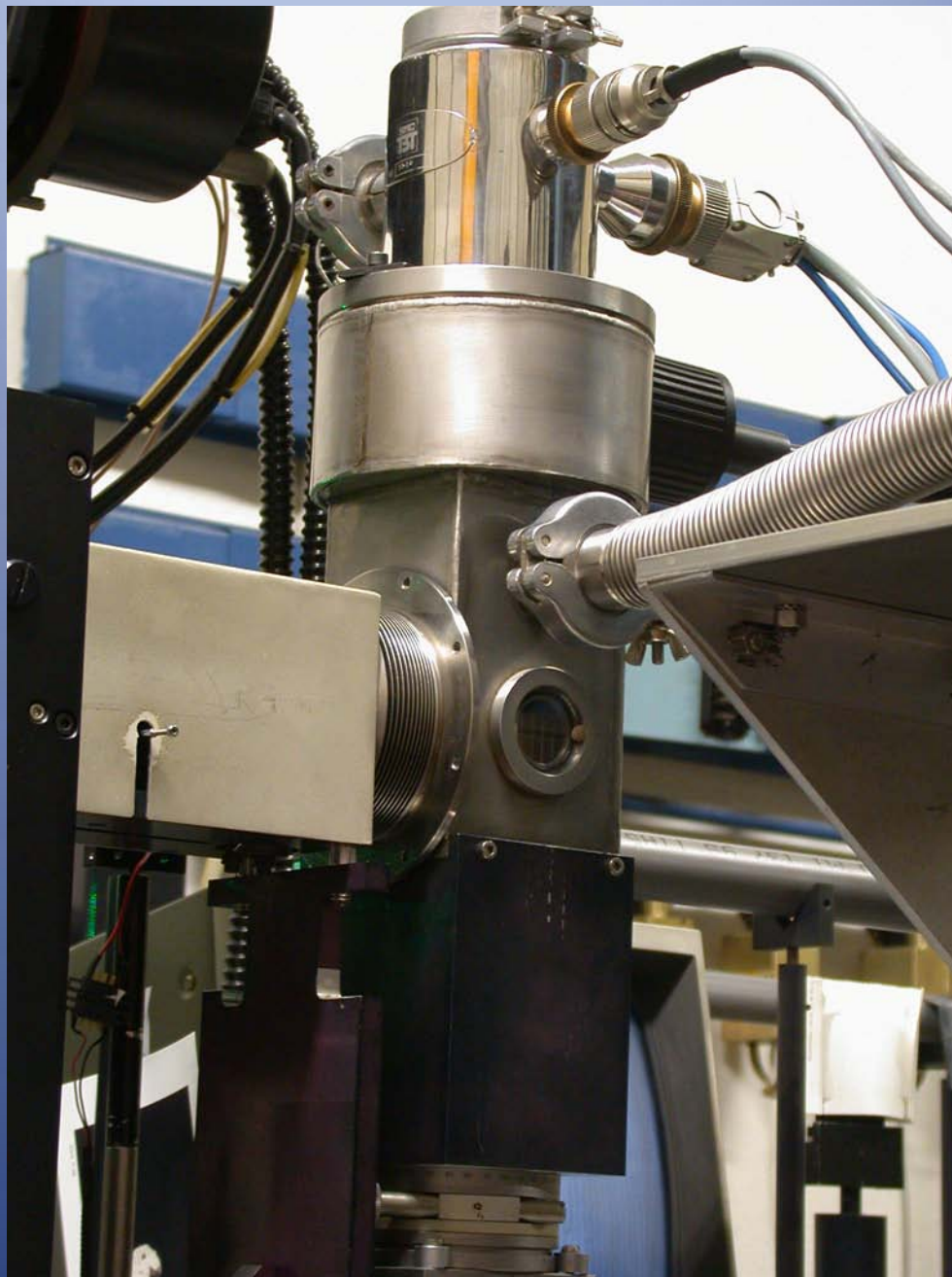
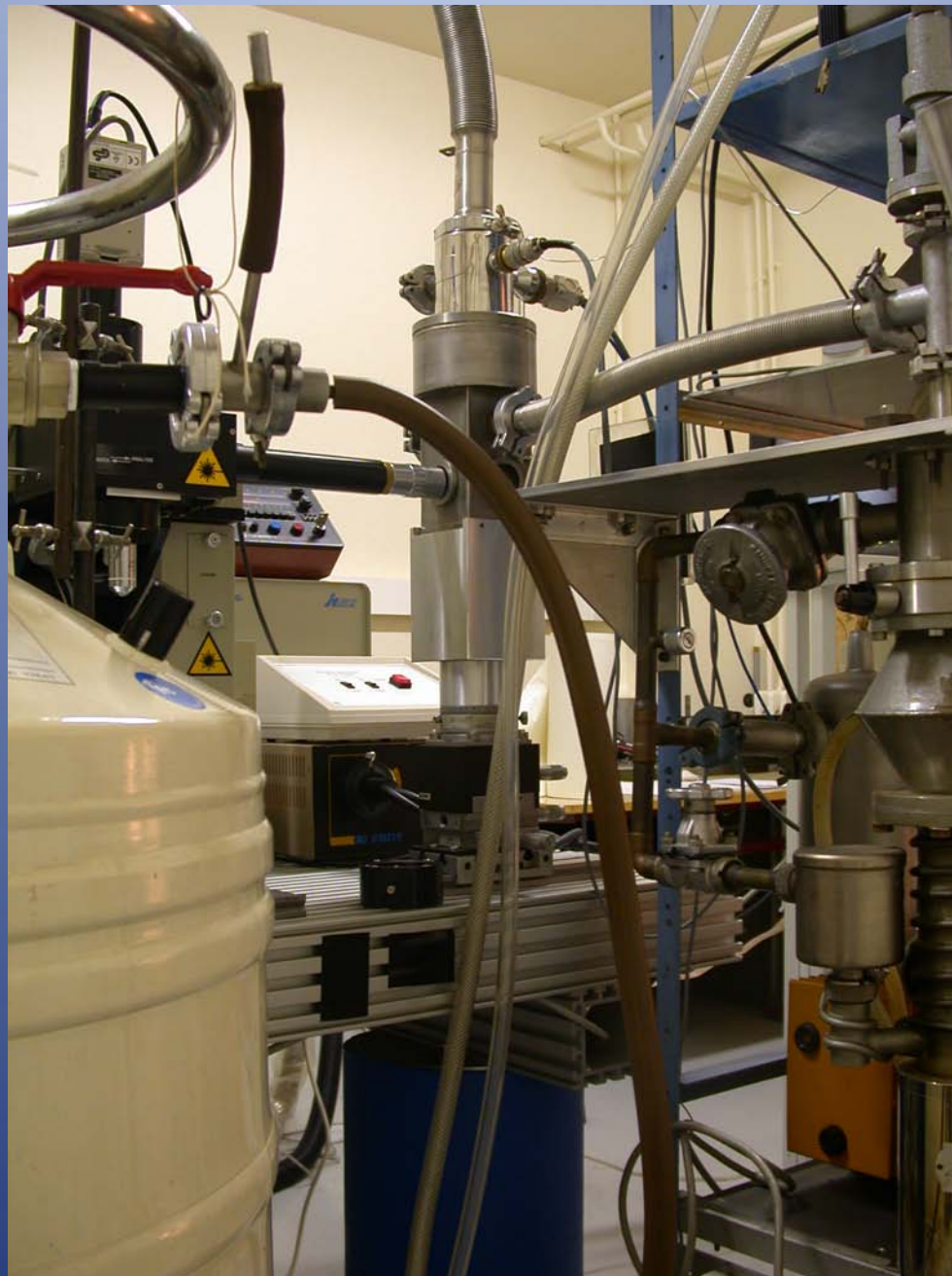


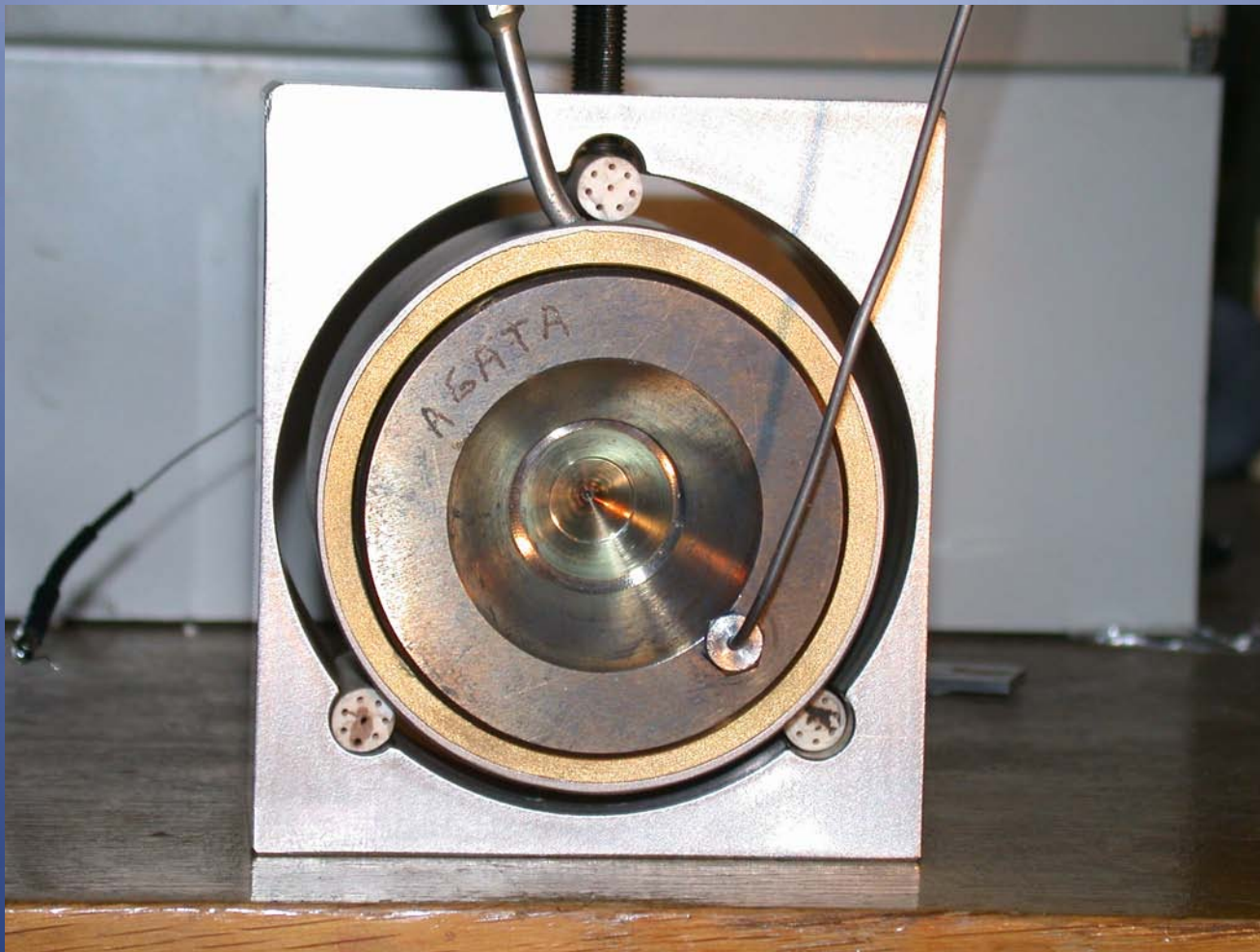
FIG. 1. The helium flow cryostat containing the MDAC and the mechanical coupling between the outer wall and the optical system. (1) MDAC (membrane chamber side); (2) flexible tubing connecting the membrane chamber and the gas source; (3) objective 25 $\times$ ; (4) glass window; (5) bellows; (6) X, Y, Z,  $\theta$  micropositioners; (7) copper sleeve; (8) thermal exchanger; (9) radiation shield; (10) thin-walled stainless-steel tube; (11) outer jacket; (12) cryostat connection; (13) flexible line; (14) glass window.



Miraflores 24 - 27 enero 2011











Miraflores 24 - 27 enero 2011

## A diamond anvil cell with resistive heating for high pressure and high temperature x-ray diffraction and absorption studies

Sebastien Pasternak,<sup>1</sup> Giuliana Aquilanti,<sup>1</sup> Sakura Pascarelli,<sup>1</sup> Roberta Poloni,<sup>1,2</sup> Bernard Canny,<sup>3</sup> Marie-Vanessa Coulet,<sup>4</sup> and Lin Zhang<sup>1</sup>

<sup>1</sup>European Synchrotron Radiation Facility, 6 rue Jules Horowitz, 38043 Grenoble, Cedex, France

<sup>2</sup>Institut de Ciència de Materials de Barcelona (CSIC), Campus de la UAB,

E-08193 Bellaterra, Barcelona Spain

<sup>3</sup>IMPMC-CNRS UMR, 7590 Université Paris VI, 140 rue de Lourmel, 75015 Paris, France

<sup>4</sup>IM2NP-UMR CNRS, 6242 Université Paul Cezanne Campus de St Jérôme, 13397 Marseille Cedex 20, France

(Received 23 April 2008; accepted 17 July 2008; published online 13 August 2008)

In this paper we describe a prototype of a diamond anvil cell (DAC) for high pressure/high temperature studies. This DAC combines the use of a resistive oven of 250 W power in a very small volume, associated with special conical seats for Boehler-type diamond anvils in order to have a large angular acceptance. To protect the diamond anvils from burning and to avoid the oven oxidation, the heated DAC is enclosed in a vacuum chamber. The assemblage was used to study the melting curve of germanium at high pressure (up to 20 GPa) and high temperature (up to 1200 K) using x-ray diffraction and x-ray absorption spectroscopy. © 2008 American Institute of Physics. [DOI: 10.1063/1.2968199]

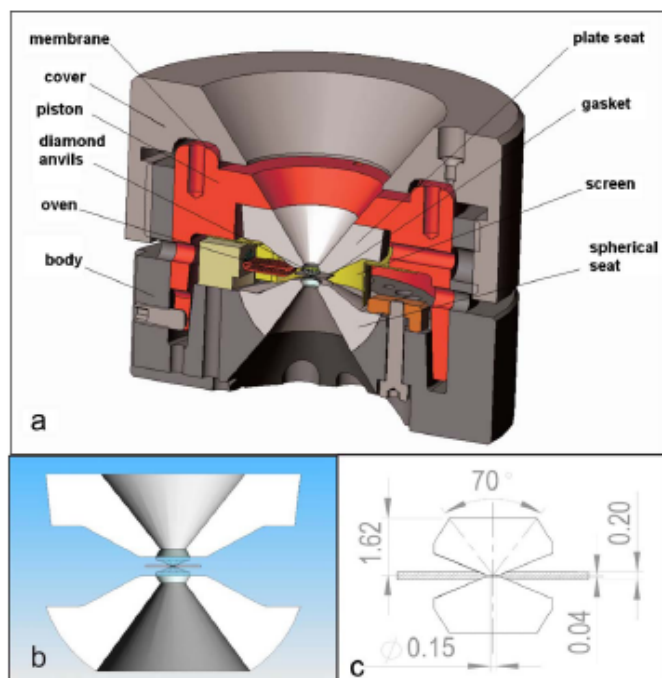


FIG. 1. (Color online) (a) Section view of the high pressure/high temperature DAC. (b) Section view of the anvil seats. (c) Section view of the diamond anvils.

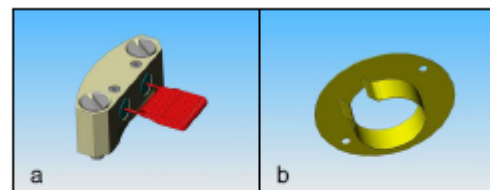


FIG. 2. (Color online) (a) High power resistive oven on its plug. (b) Radiation screen.

## Nueva celda de yunques de diamante por el estudio in situ de fluidos geológicos por espectroscopia

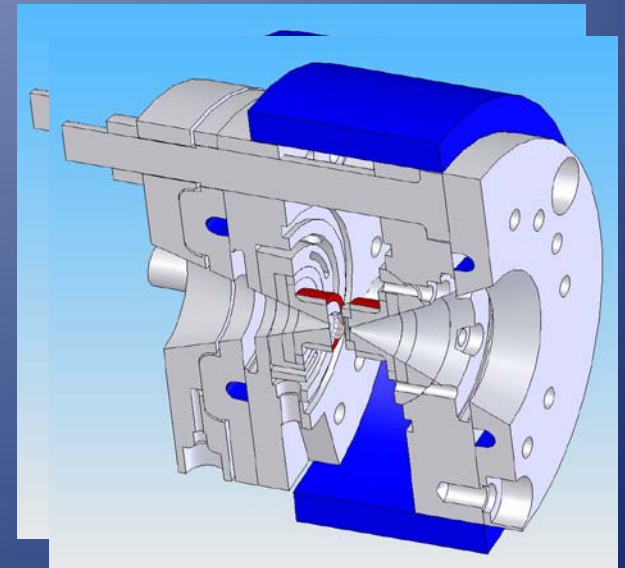
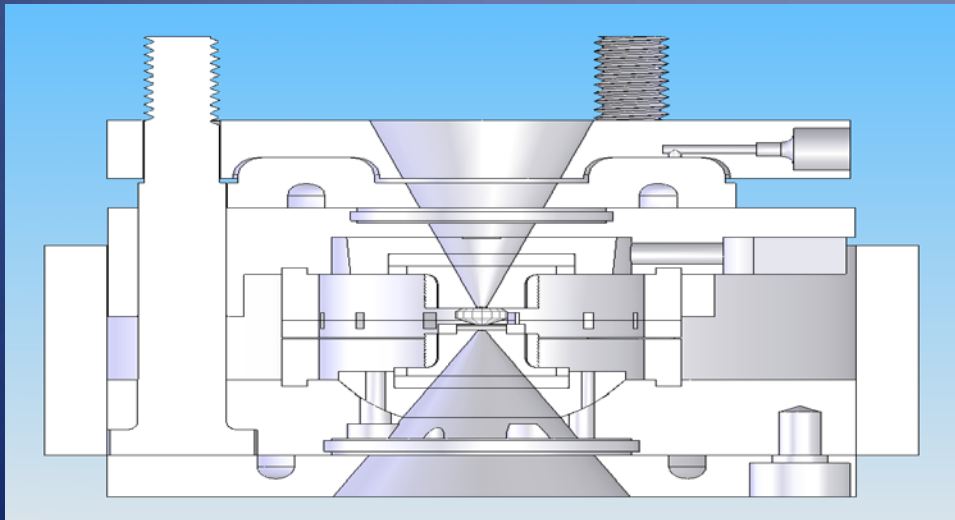
Pascal Munsch<sup>1</sup>, Hélène Bureau<sup>1</sup>, Bernard Couzinet<sup>1</sup>, Andrea Somogyi<sup>2</sup>, Eddy Foy<sup>3</sup>, Guilhem Simon<sup>1</sup>, Stefan Kubsy<sup>2</sup>, Jean-Claude Chervin<sup>1</sup>

1. IMPMC, Campus Boucicaut, 140 rue de Lourmel, 75015 Paris

2. Laboratoire Pierre Süe, UMR9956, CEA Saclay, 91191 Gif sur Yvette

3. Synchrotron SOLEIL, Saint Aubin, BP 48, 91192 Gif sur Yvette

El estudio in situ de los fluidos geológicos de la corteza y el manto terrestre exterior necesitan la utilización de CYD, que permite adaptarse a las condiciones de presión y temperatura de tales fluidos ( desde algunas centenas de MPa hasta 10 GPa y idealmente a la temperatura de 1500 °C). Con el fin de cuantificar las trasferencias químicas des elementos entre los fluidos geológicos ( fluidos acuosos, melts silicatos, fluidos supercríticos) por rayos X, espectroscopia Raman o espectroscopia Infrarroja, nosotros hemos desarrollados una nueva célula de yunques de diamante (CYD).



# Calentamiento laser



## Transformation of benzene to a polymer after static pressurization to 30 GPa

Ph. Pruzan, J. C. Chervin, M. M. Thiéry, J. P. Itié, and J. M. Besson  
*Physique des Milieux Condensés (CNRS-UA 782), T13 E4, Université Pierre et Marie Curie, 75252 Paris Cedex 05, France*

J. P. Forgerit and M. Revault  
*LASIR (CNRS, LP2641) 2, rue Henri Dunant, 94230 Thiais, France*

(Received 24 October 1989; accepted 31 January 1990)

After pressurization to 30 GPa, in a diamond anvil cell, benzene transforms, at room temperature, to a white solid which is stable at ambient pressure. We report here the infrared spectroscopy analysis performed under pressure and at ambient conditions. These preliminary results show that the transformation involves an opening of the benzene rings leading to a highly cross-linked polymer.

6910 J. Chem. Phys. **92** (11), 1 June 1990

0021-9606/90/116910-06\$03.00

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## Chemical and phase transformations of thiophene at high pressures

Ph. Pruzan and J. C. Chervin  
*Physique des Milieux Condensés (CNRS, UA D782), T 13, E 4, B77, Université Pierre et Marie Curie, F-75252 Paris Cedex 05, France*

J. P. Forgerit  
*LASIR (CNRS, LP 2641) 2, rue H. Dunant, F-94230 Thiais, France*

(Received 5 August 1991; accepted 23 September 1991)

Phase transformations of thiophene ( $C_4H_4S$ ) and deuterated thiophene ( $C_4D_4S$ ) were studied by visual observations and Fourier-transform infrared spectroscopy up to 30 GPa. At room temperature, thiophene freezes at 0.4 GPa and undergoes a solid-to-solid transition around 6 GPa and probably another one at 20 GPa. It is shown that an irreversible incomplete transition occurs at 16 GPa. The product of the reaction, a white solid, can be recovered at atmospheric pressure. The infrared analysis of the recovered product gives evidence of a polymer formed by coupling between rings derived from thiophene, but with saturated bonds. Physicochemical arguments suggest the formation of a fibrous compound.

J. Chem. Phys. **96** (1), 1 January 1992

0021-9606/92/010761-07\$06.00

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## A diamond anvil cell for IR microspectroscopy

J. C. Chervin, B. Canny, J. M. Besson, and Ph. Pruzan

*Physique des Milieux Condensés, CNRS-URA 782, Université Pierre et Marie Curie, B 77, 4, place Jussieu, F-75252 PARIS Cedex 05, France*

(Received 27 October 1994; accepted for publication 30 November 1994)

A large optical-aperture membrane diamond anvil cell designed for infrared spectroscopy is described. The cell offers definite advantages compared to existing systems. Other possibilities concerning x-ray diffraction analyses with the cells are mentioned. © 1995 American Institute of Physics.

Rev. Sci. Instrum. 66 (3), March 1995

0034-6748/95/66(3)/2595/4/\$6.00

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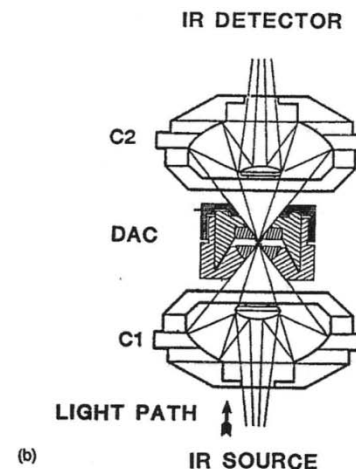
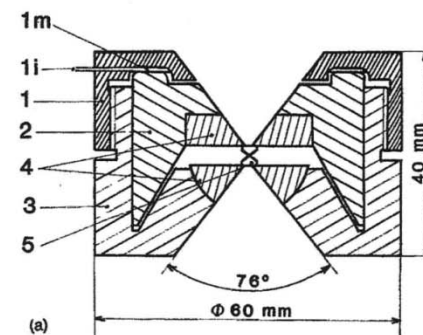


FIG. 1. The membrane diamond anvil cell designed for infrared microspectroscopy. (a) The cell: (1) cap with membrane chamber (1 m) and gas inlet (1i) of the ram, (2) piston, (3) lower body, (4) WC diamond seats, (5) anvils. (b) Schematic diagram of on-axis Cassegrain optics used with the diamond anvil cell for infrared microspectroscopy: C1 and C2 Cassegrain mirrors. The scale is given by the cell's dimensions.





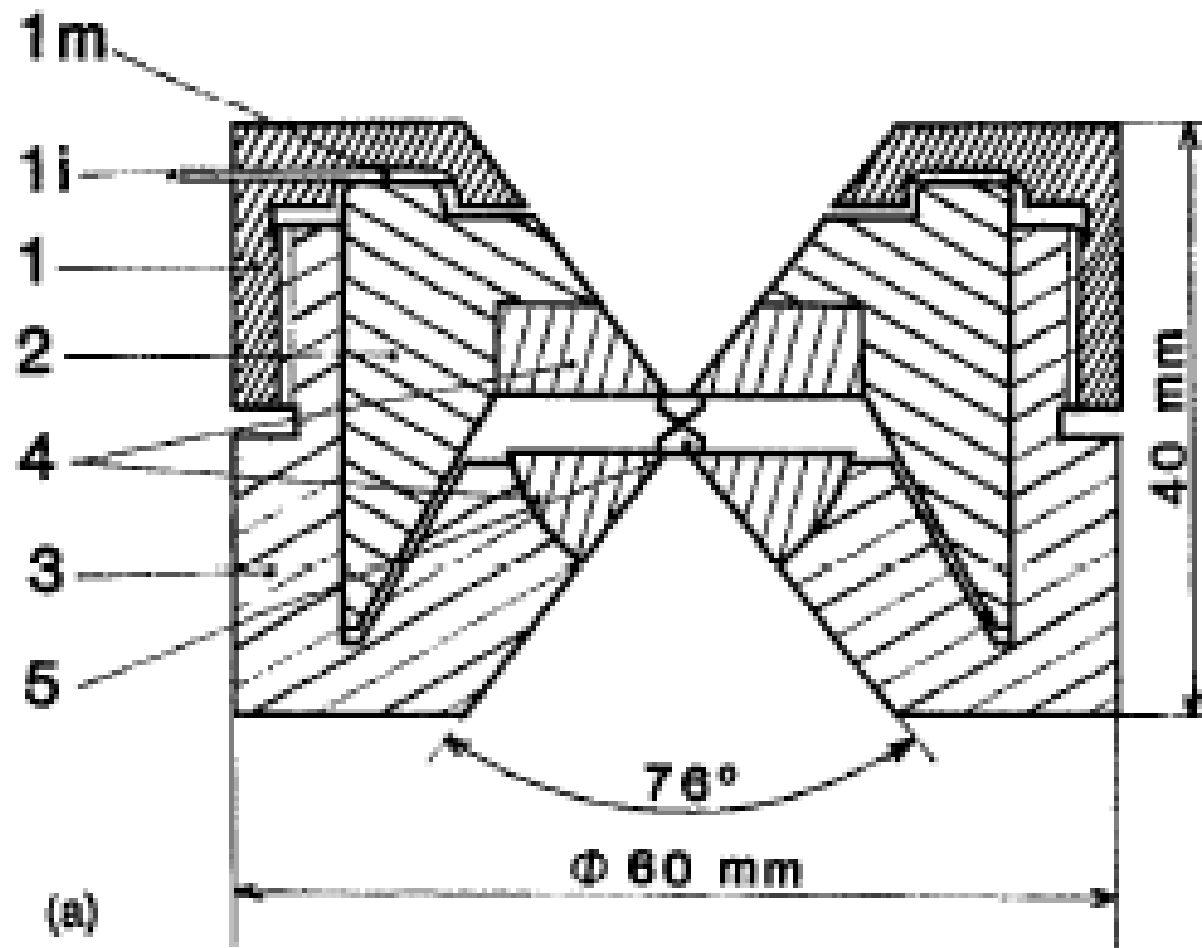
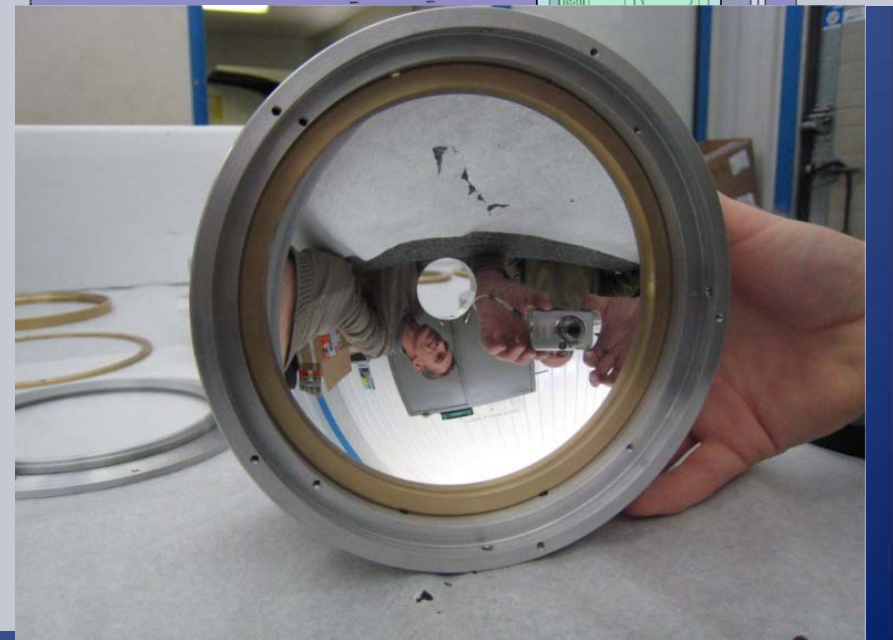
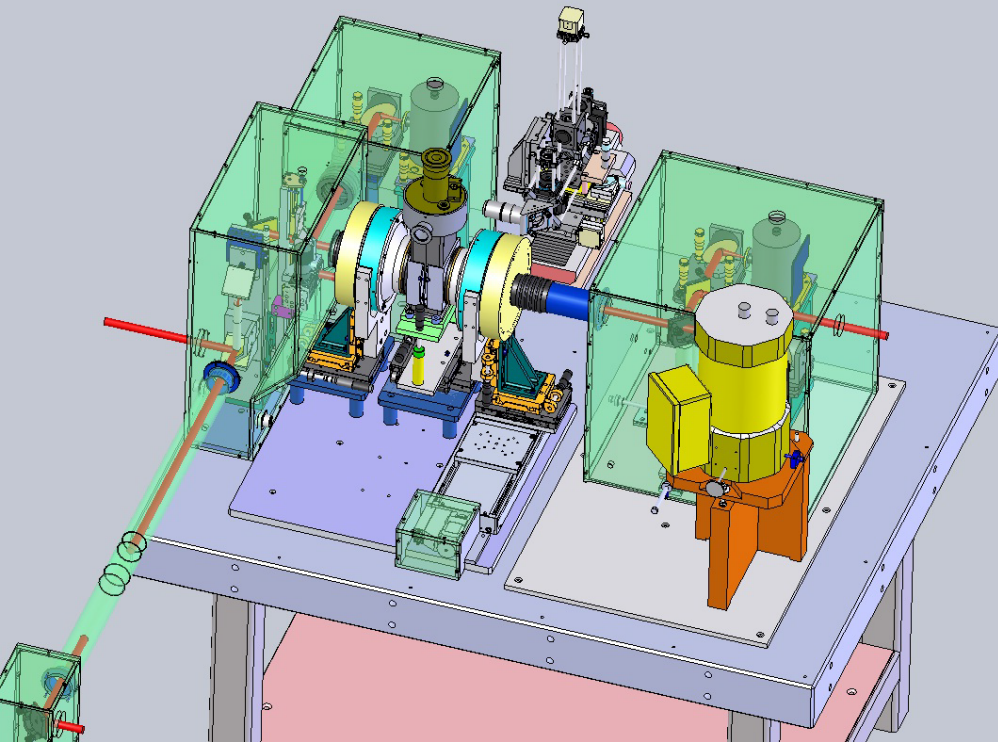
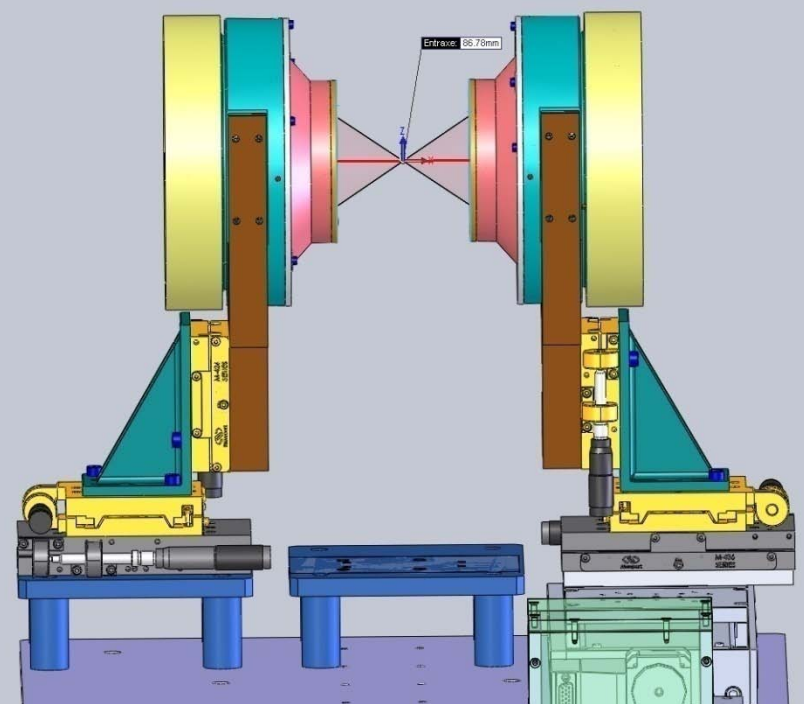
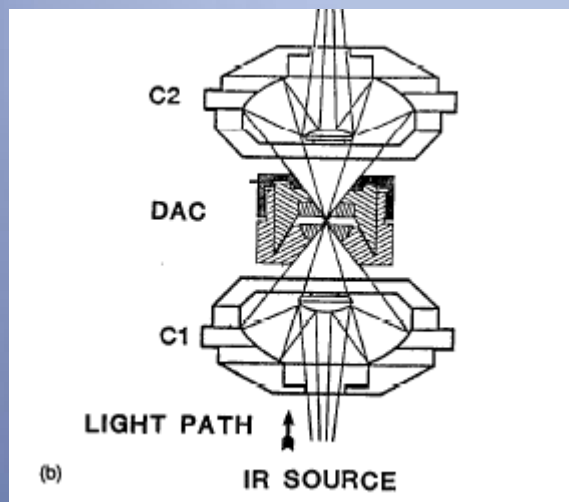


FIG. 1. The membrane diamond anvil cell designed for infrared microspectroscopy. (a) The cell: (1) cap with membrane chamber (1 m) and gas inlet (1i) of the ram, (2) piston, (3) lower body, (4) WC diamond seats, (5) anvils.







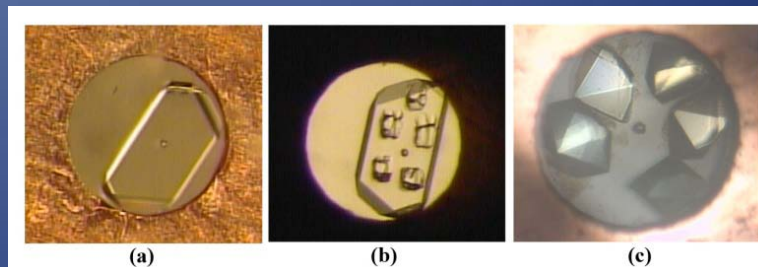
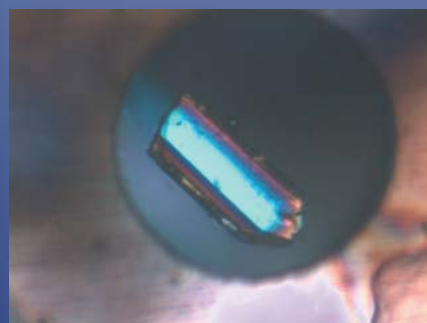
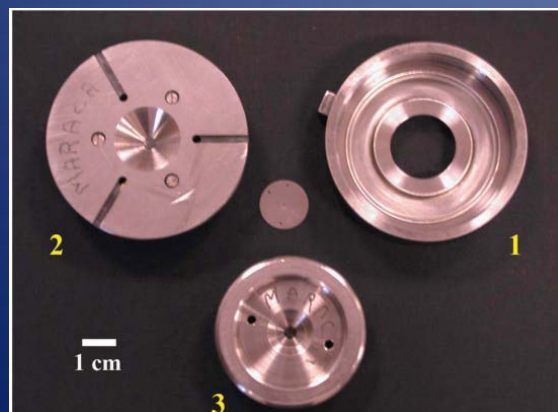
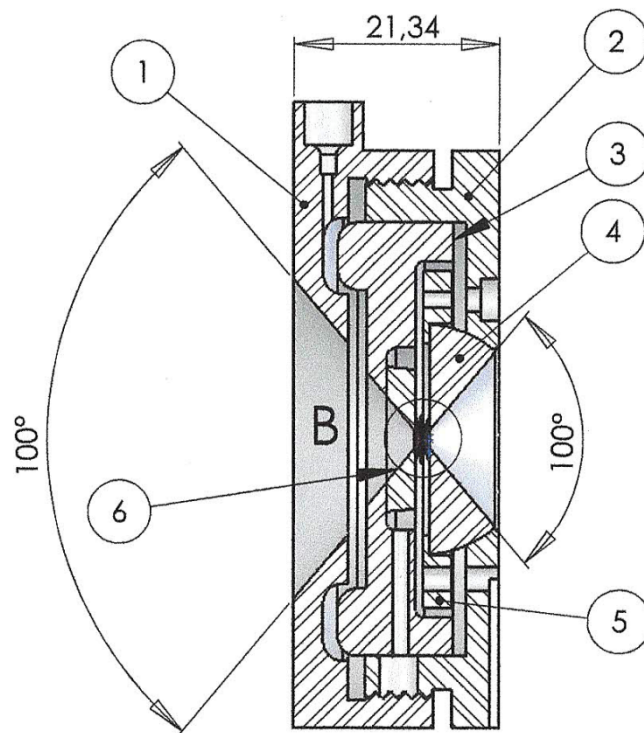


## Toward fully fledged high-pressure macromolecular crystallography

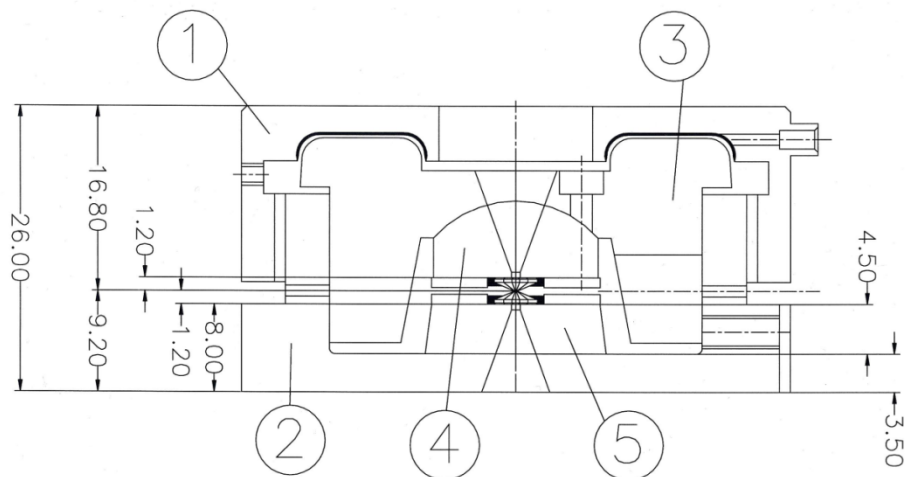
Eric Girard,<sup>a\*</sup> Anne-Claire Dhaussy,<sup>b</sup> Bernard Couzinet,<sup>c</sup> Jean-Claude Chervin,<sup>c</sup>  
 Mohamed Mezouar,<sup>d</sup> Richard Kahn,<sup>e</sup> Isabella Ascone<sup>a</sup> and Roger Fourme<sup>a</sup>

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Pressure allows one to explore the energy landscape and the various conformations of macromolecules. This might be one of the most interesting prospects of combining macromolecular crystallography (MX) with pressure perturbation. This paper presents innovations in high-pressure macromolecular crystallography (HPMX). A new-generation diamond anvil cell extends the technical feasibility of HPMX experiments up to about 2.5 GPa (depending on the sample). Thanks to the large useful aperture (82°) provided by this cell and special loading techniques (use of splinter and/or multiple samples), HPMX can now be more readily applied to crystals with very anisotropic habits and/or low-symmetry space groups, as demonstrated by analysis of diffraction data collected at 140 MPa from orthorhombic urate oxidase crystals. The behaviour of hexagonal crystals of an octanucleotide was investigated up to 2 GPa.







| 5       | PLATEAU                    | 1  | CARBURE314   |     |
|---------|----------------------------|----|--------------|-----|
| 4       | 1/2 SPHERE                 | 1  | CARBURE314   |     |
| 3       | PISTON                     | 1  | cu.be        |     |
| 2       | CORPS                      | 1  | cu.be        |     |
| 1       | capot                      | 1  | cu.be        |     |
| Rep     | Designation                | Nb | Matiere      | Ind |
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| 2.5/1   |                            |    | LE GOFFA     |     |
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*J. Synchrotron Rad.* (1998). **5**, 992–994

## Two recent developments in XMCD

F. Baudalet,<sup>a\*</sup> J. M. Dubuisson,<sup>a</sup> C. Hébert,<sup>a</sup>  
 C. Créoff,<sup>a</sup> L. Pointal,<sup>a</sup> R. Andouard,<sup>a</sup> S. Odin,<sup>a</sup>  
 E. Dartyge,<sup>a</sup> G. Krill,<sup>a</sup> C. Giorgetti,<sup>a</sup> J. C. Chervin,<sup>b</sup>  
 J. P. Itié,<sup>b</sup> A. Polian,<sup>b</sup> A. Fontaine,<sup>c</sup> S. Pizzini<sup>c</sup> and  
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<sup>b</sup>Laboratoire de Physique des Milieux Condensés, Paris VI,  
 4 Place Jussieu BP 77, 75252 Paris CEDEX 05, France,

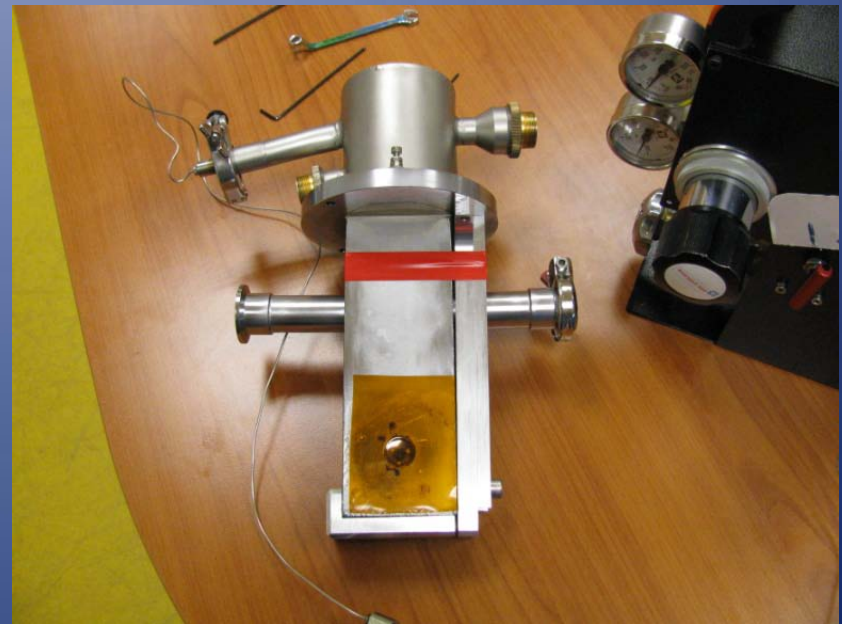
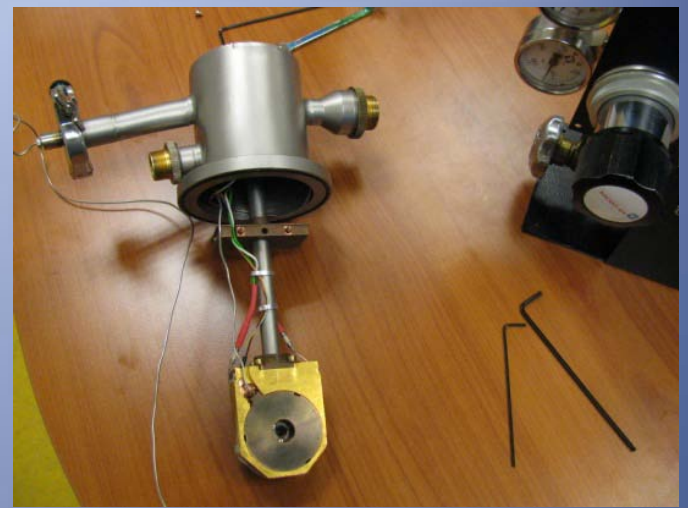
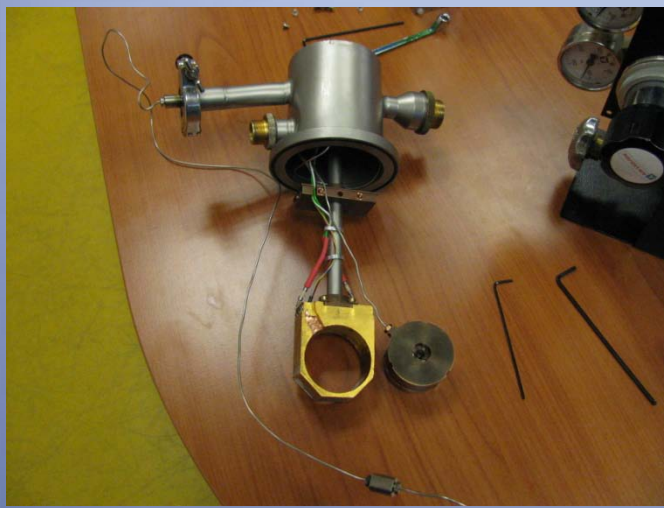
<sup>c</sup>Laboratoire de Magnétisme Louis Néel, Avenue des  
 Martyrs, 38042 Grenoble CEDEX 9, France, and <sup>d</sup>Institut de  
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(Received 4 August 1997; accepted 26 January 1998)

This paper reports on two new technical developments concerning sample environments for X-ray magnetic circular dichroism (XMCD). The first measurements under high pressures of up to 30 GPa are described. The difficulties of combining the techniques of high pressure and XMCD are commented on. The second development involves the use of a fast-switching magnetic field. A new superconducting device is used to perform XMCD measurements on paramagnetic compounds in magnetic fields of up to 6 T. The small amplitude of the XMCD signal imposes, for a given signal-to-noise ratio, a noise less than a few  $10^{-5}$ . The signal-to-noise ratio is improved by the use of a series of acquisitions, switching the magnetic field between each acquisition. A very fast switching mechanism has been built based on mechanical rotation of a superconducting coil, with the sample kept in place inside the coil. The XMCD signals at the  $L_{II,III}$ -edges of paramagnetic rare-earth compounds have been measured at 4.5 K in fields of up to 6 T with a switching time of 11 s.







Bernard Couzinet  
IMPMC



# Magneto-optical Kerr effect under pressure

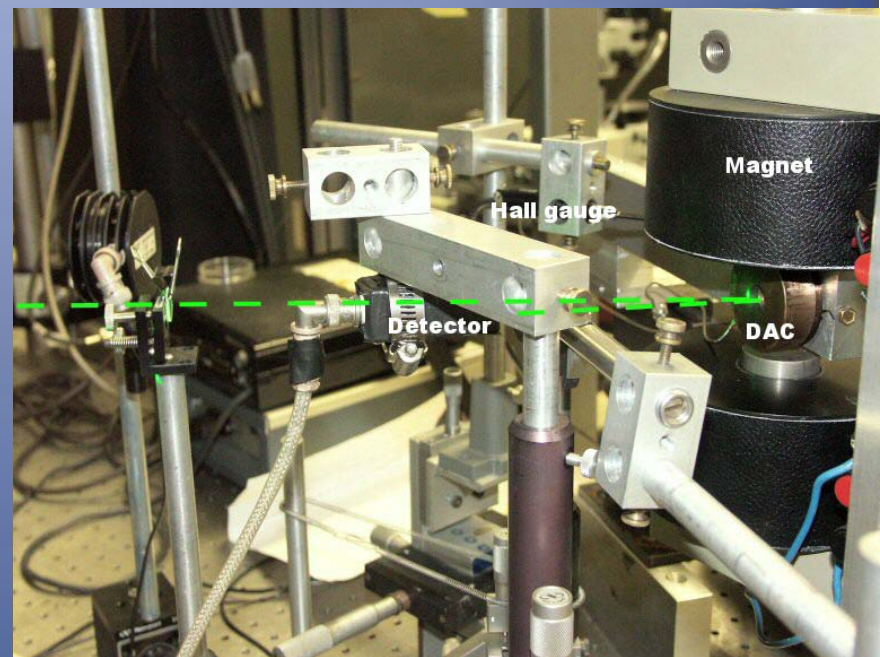
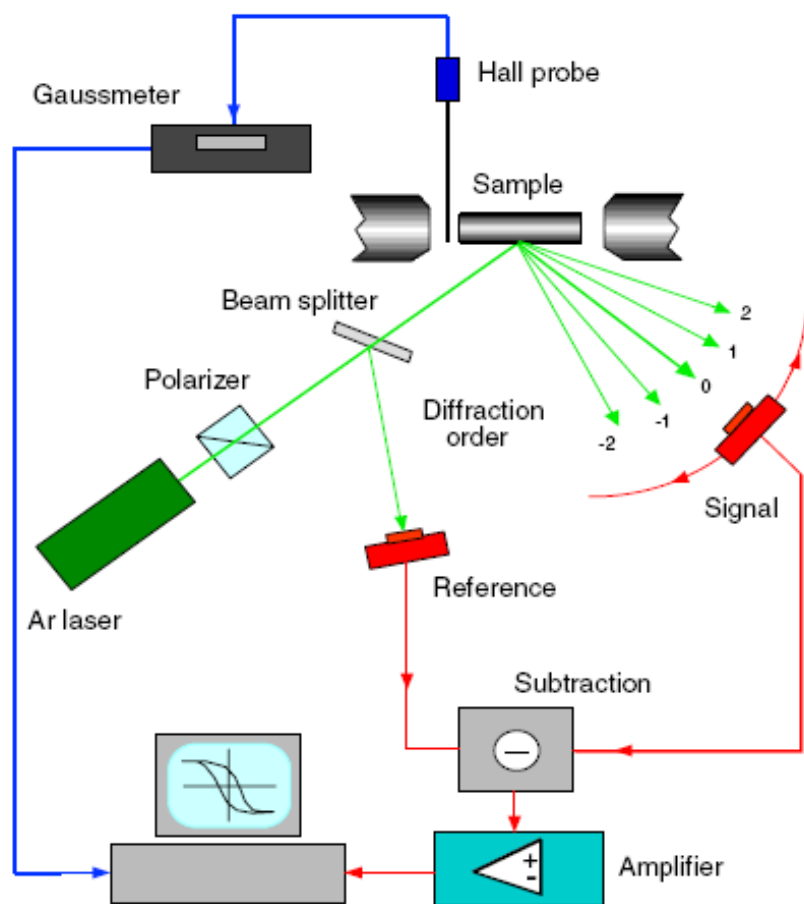
A. Polian<sup>#</sup>, M. Grimsditch\*, P. Vavassori<sup>^</sup>, J. Yi\*, A. Hoffmann\* and V. Metlushko<sup>§</sup>

<sup>#</sup> Physique des Milieux Denses, IMPMC – CNRS – Université Pierre et Marie Curie – Paris - France

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<sup>§</sup> Dept. of Electrical and Computer Engineering, University of Illinois at Chicago, Chicago, IL, USA



## Novel high pressure magnetic measurements with application to magnetite

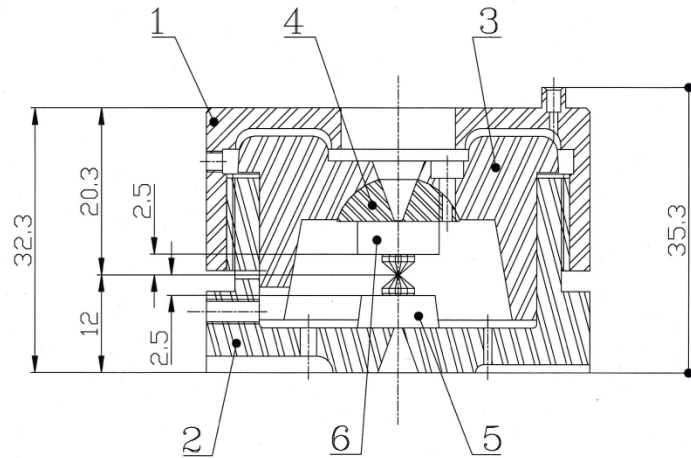
Stuart A. Gilder,<sup>1</sup> Maxime LeGoff,<sup>1</sup> Jean Peyronneau,<sup>2</sup> and Jean-Claude Chervin<sup>3</sup>

Received 24 October 2001; revised 17 January 2002; accepted 18 January 2002; published 22 May 2002.

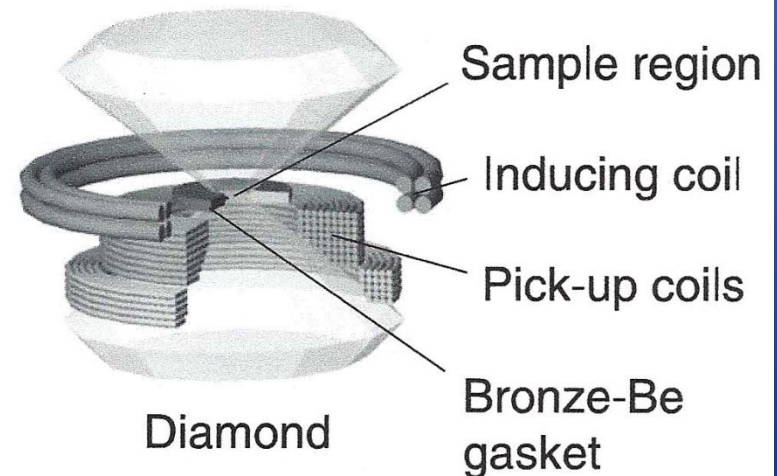
<sup>1</sup>Institut de Physique du Globe, Laboratoire de Paléomagnétisme, Paris, Cedex 05, France.

<sup>2</sup>Institut de Physique du Globe, Laboratoire de Géomateriaux, Paris, Cedex 05, France.

<sup>3</sup>Université Pierre et Marie Curie, Physique des Milieux Condensés, Paris, Cedex 05, France.

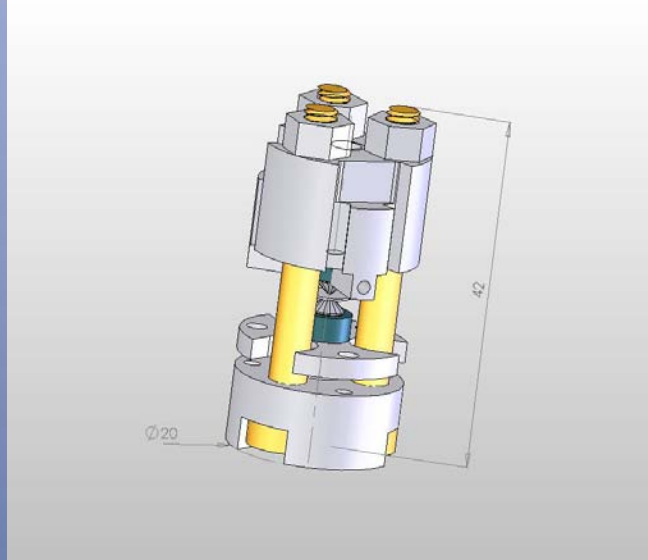


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| 5              | Plateau                        | 1                  | Saphir  |     |
| 4              | 1/2 sphère                     | 1                  | CuBe    |     |
| 3              | Piston                         | 1                  | CuBe    |     |
| 2              | Corps                          | 1                  | CuBe    |     |
| 1              | Capot                          | 1                  | CuBe    |     |
| Rep            | Designation                    | Nb                 | Matiere | Ind |
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**Figure 1.** Simplified illustration of ac susceptibility system.





*High Pressure Research*  
 Vol. 28, No. 4, December 2008, 627–631



## New diamond anvil cell for optical and transport measurements under high magnetic fields up to 60 T

Marius Millot<sup>a</sup>, Sylvie George<sup>a</sup>, Jean-Marc Broto<sup>a\*</sup>, Bernard Couzinet<sup>b</sup>, Jean-Claude Chervin<sup>b</sup>, Alain Polian<sup>b</sup>, Chrystian Power<sup>c</sup> and Jesus González<sup>c,d</sup>

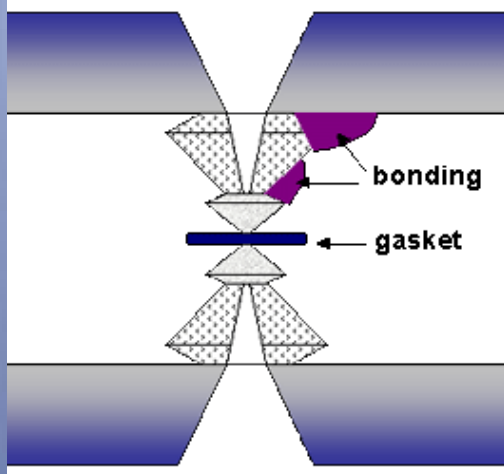
<sup>a</sup>Laboratoire National des Champs Magnétiques Pulsés, Université de Toulouse, Toulouse, France;

<sup>b</sup>Institut de Minéralogie et de Physique des Milieux Condensés, CNRS, Université Pierre et Marie Curie Paris 6, Paris, France; <sup>c</sup>Centro de Estudios de Semiconductores, Facultad de Ciencias, Universidad de los Andes, Mérida, Venezuela; <sup>d</sup>DCITIMAC – Malta Consolider Team, Universidad de Cantabria, Santander, Spain

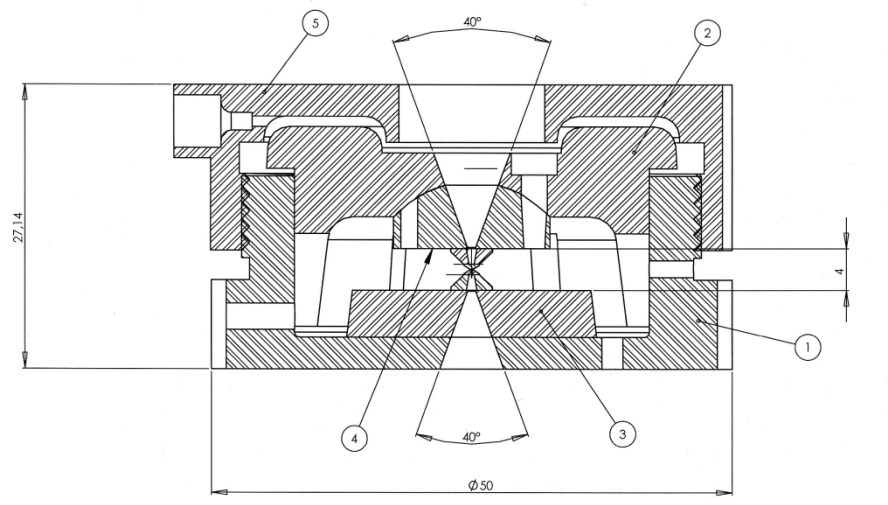
(Received 25 August 2008; final version received 29 September 2008)

We present here a new diamond anvil cell designed to combine high hydrostatic pressure up to 10 GPa, non-destructive, pulsed magnetic fields up to 60 T and low temperature as low as 2 K. Both optical and transport measurements are now achievable. Magneto-photoluminescence set-up under pressure is described in great detail. We report photoluminescence experiments on oriented ruby single crystals exhibiting direct observation of the intrinsic Zeeman splitting of excited states under pressure.

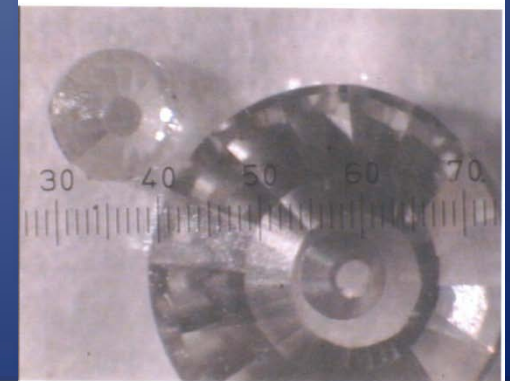
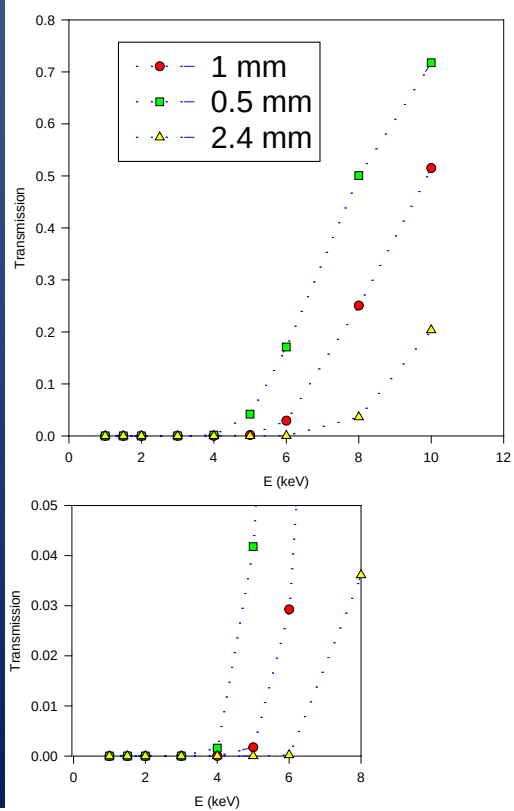




Access to low energy edges



Diamond absorption



Miraflores 24 - 27 enero 2011

## High pressure x-ray absorption spectroscopy at lower energy in the dispersive mode: application to Ce and FePO<sub>4</sub>

J P Itié<sup>1</sup>, F Baudalet<sup>2</sup>, A Congeduti<sup>1</sup>, B Couzinet<sup>1</sup>, F Farges<sup>3</sup> and A Polian<sup>1</sup>

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Received 12 January 2005

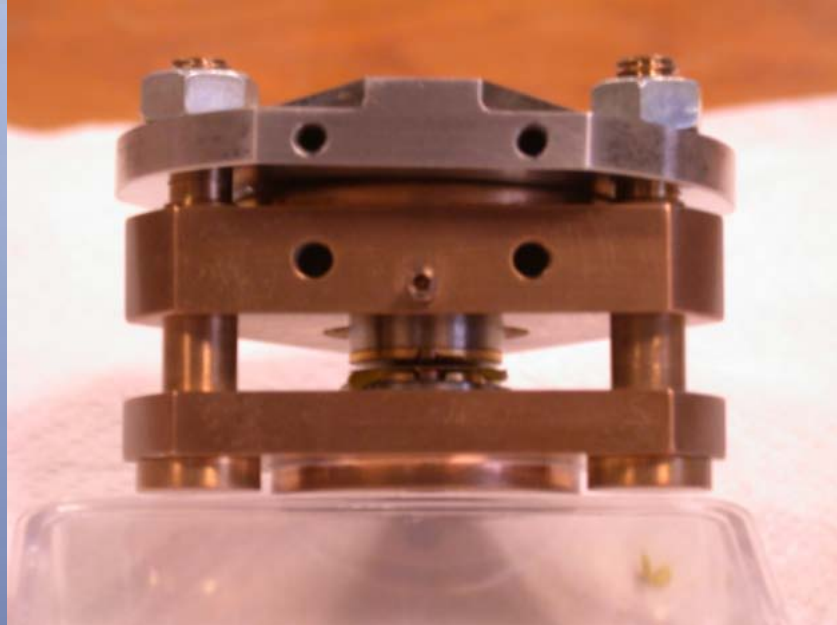
Published 4 March 2005

Online at [stacks.iop.org/JPhysCM/17/S883](http://stacks.iop.org/JPhysCM/17/S883)

### Abstract

X-ray absorption spectroscopy (XAS) using a diamond anvil cell has been extended to lower energies (below 6 keV) thanks to smaller diamond anvils and to a new acquisition set-up on the energy dispersive x-ray absorption beamline of LURE. The valence change of Ce with pressure is followed at the L<sub>III</sub> edge of cerium up to 8 GPa together with the bond length variation during the same experiment. The pressure induced coordination change around the Fe atom in FePO<sub>4</sub> is determined at the Fe K edge through the pre-edge feature behaviour, the XANES modification and the Fe–O bond length increase.

(Some figures in this article are in colour only in the electronic version)



APPLIED PHYSICS LETTERS **89**, 231904 (2006)

## Local environment of a diluted element under high pressure: $\text{Zn}_{1-x}\text{Mn}_x\text{O}$ probed by fluorescence x-ray absorption spectroscopy

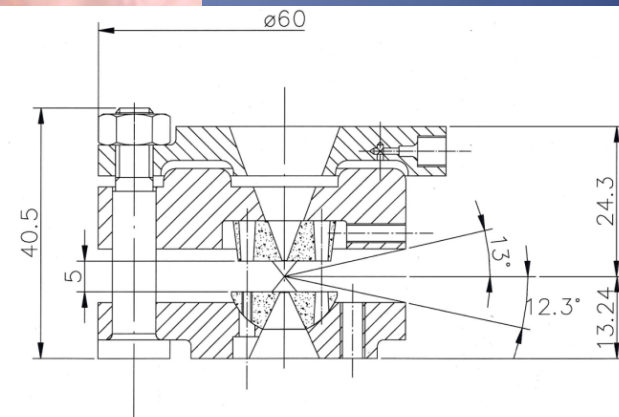
J. Pellicer-Porres,<sup>a)</sup> A. Segura, J. F. Sánchez-Royo, and J. A. Sans  
*ICMUV, Universidad de Valencia, c/Dr. Moliner 50, 46100 Burjassot, Valencia, Spain*

J. P. Itié, A. M. Flank, and P. Lagarde  
*Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin - BP 48, 91192 Gif-sur-Yvette Cedex, France*

A. Polian  
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(Received 27 July 2006; accepted 23 October 2006; published online 4 December 2006)

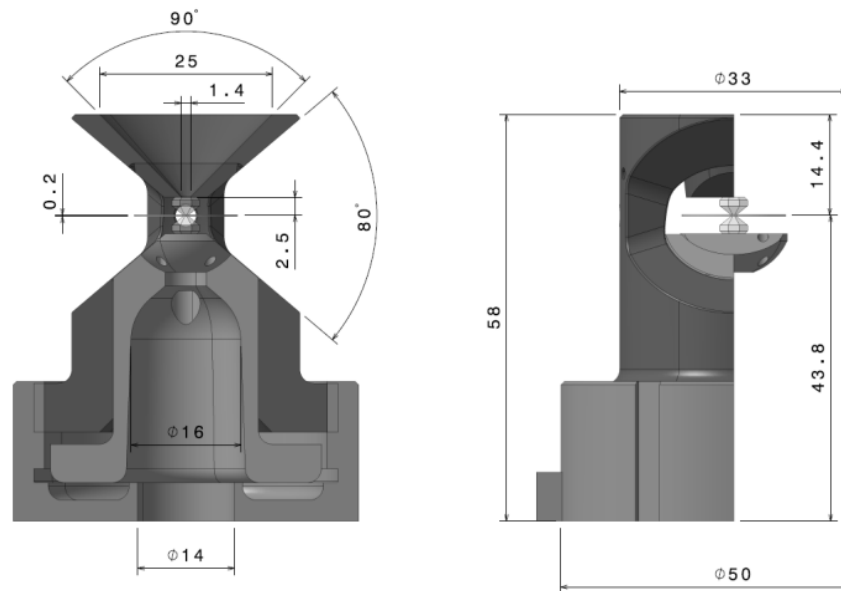
The authors report on x-ray absorption measurements performed on  $\text{Zn}_{1-x}\text{Mn}_x\text{O}$  ( $x=0.05, 0.25$ ) at the Mn *K* edge (6539 eV) under high pressure. Mn is found to substitute for Zn both in the low pressure wurtzite phase and in the high pressure rocksalt phase. The Mn–O bond length is determined to be  $2.02 \pm 0.01$  Å at ambient conditions, with a compressibility similar to that of Zn–O in pure ZnO, being dictated by the ZnO crystalline environment. The phase transition is reversible in  $\text{Zn}_{0.95}\text{Mn}_{0.05}\text{O}$ , but not in  $\text{Zn}_{0.75}\text{Mn}_{0.25}\text{O}$ . The authors demonstrate that fluorescence detected x-ray absorption under high pressure can be used to characterize structural changes around minority elements. © 2006 American Institute of Physics. [DOI: 10.1063/1.2400109]



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|                | Physique des milieux condensés |                    |    |
|                |                                |                    | 00 |



*Prototipo de celda de tipo panorámica  
 (colaboración IMPMC-CNRS-UPMC  
 (JC Chervin) y la Universidad Lyon 1  
 (H. Feret et A. San Miguel)).*



3804

*J. Phys. Chem. B* **2010**, *114*, 3804–3808

## Role of Non-Hydrogen-Bonded Molecules in the Oxygen K-Edge Spectrum of Ice

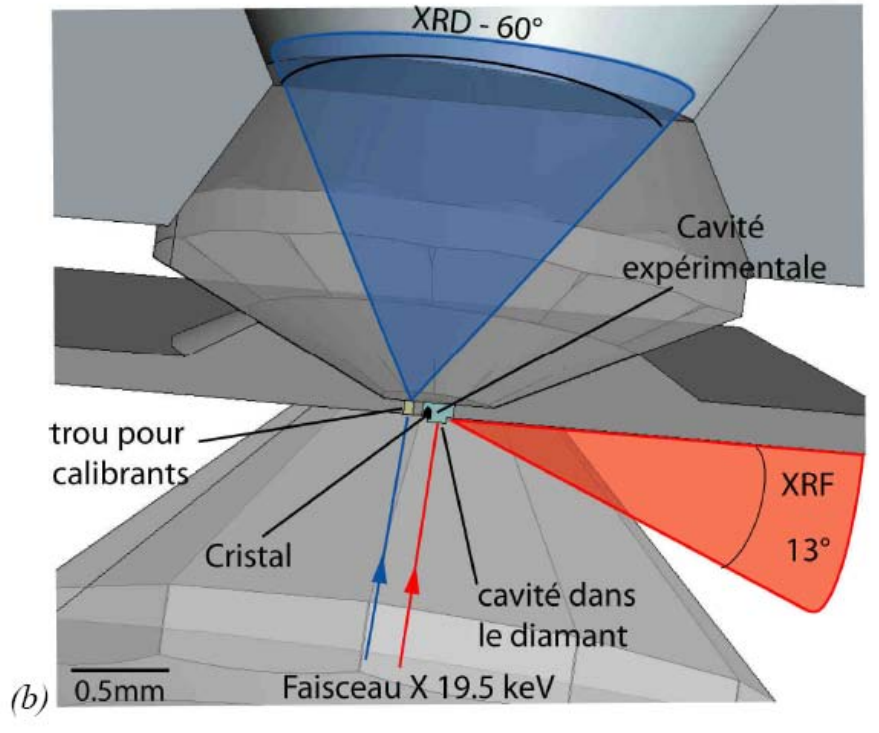
**Tuomas Pylkkänen,<sup>\*,†,‡</sup> Valentina M. Giordano,<sup>‡</sup> Jean-Claude Chervin,<sup>§</sup> Arto Sakko,<sup>†</sup>  
 Mikko Hakala,<sup>†</sup> J. Aleksi Soininen,<sup>†</sup> Keijo Hämäläinen,<sup>†</sup> Giulio Monaco,<sup>‡</sup> and Simo Huotari<sup>‡</sup>**

*Department of Physics, P.O. Box 64, FI-00014, University of Helsinki, Finland, European Synchrotron Radiation Facility, B.P. 220, F-38043 Grenoble, France, and Physique des Milieux Condensés, IMPMC, CNRS-UMR 7590, Université Pierre et Marie Curie, F-75015 Paris, France*

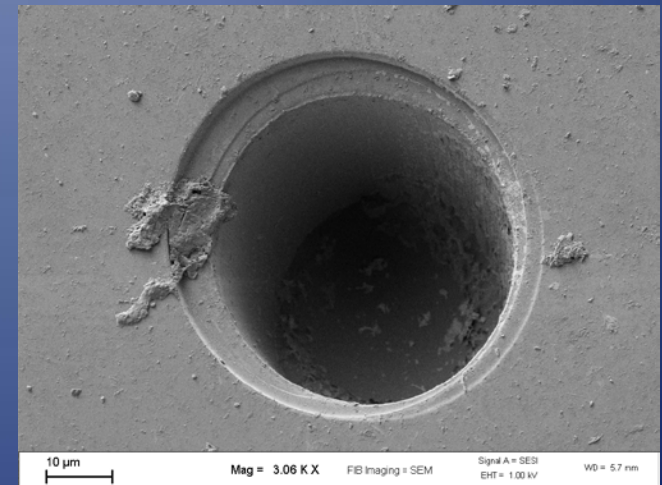
*Received: December 28, 2009; Revised Manuscript Received: February 5, 2010*

We report the oxygen K-edge spectra of ices Ih, VI, VII, and VIII measured with X-ray Raman scattering. The pre-edge and main-edge contributions increase strongly with density, even though the hydrogen bond arrangements are very similar in these phases. While the near-edge spectral features in water and ice have often been linked to hydrogen bonding, we show that the spectral changes in the phases studied here can be quantitatively related to structural changes in the second coordination shell. Density-functional theory calculations reproduce the experimental results and support the conclusion. Our results suggest that non-hydrogen-bonded neighbors can have a significant effect also in the liquid water spectrum. We discuss the implications of the results for the actively debated interpretation of the liquid water spectrum in terms of local structure.





Se ha diseñado una nueva celda llamada FluoX. Esta celda permite medir la señal de fluorescencia X de trazas de elementos en un fluido acuoso a  $90^\circ$  del haz incidente in situ a alta presión y alta temperatura ( leer la tesis de Petitgirard Sylvain, post-doc en la línea ID 27 del ESRF)





# Electrical conductivity of the Earth's lower mantle

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Département des Géomatériaux, Institut de Physique du Globe de Paris,  
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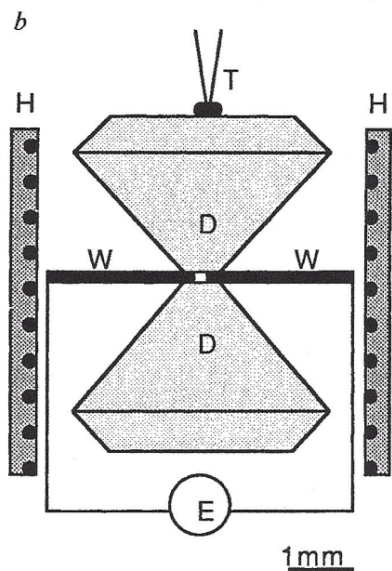
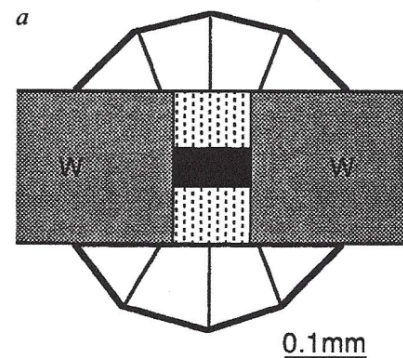


FIG. 1 a Top view of the assemblage: The sample (black) is prepared by transforming the olivine or enstatite (stippled) between the tungsten leads (W). b Schematic view of the apparatus: D, diamonds; W, tungsten leads; T, thermocouple; H, heater; E, electrometer.

**ELECTRICAL** conductivity is an important physical property of the Earth's mantle because it controls the transmission of geomagnetic signals from the core to the surface. The lower mantle, from a depth of 670 km down to the core-mantle boundary (2,990 km), is probably composed of (Mg,Fe)SiO<sub>3</sub> perovskite and magnesio-wüstite, (Mg,Fe)O. Analysis of the transient and secular variations of the geomagnetic field yields values of the lower-mantle conductivity of the order of 1 S m<sup>-1</sup> at a depth of 1,000 km, increasing to ~100 S m<sup>-1</sup> at the core-mantle boundary<sup>1,2</sup>. Information about the physical mechanism of the conductivity and its dependence on temperature and pressure would help to constrain the temperature profile in the Earth. In the only study published so far, Li and Jeanloz<sup>3,4</sup> reported values of the conductivity of the silicate perovskite and of a mixture of perovskite and magnesio-wüstite lower than 10<sup>-3</sup> S m<sup>-1</sup> and concluded that the lower mantle is an insulator, thus casting doubt on the geomagnetic results. We report here the results of measurements of the d.c. electrical

# Electrical transport measurements in a gasketed diamond anvil cell up to 18 GPa

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*Physique des Milieux très Condensés (U.A. 782), Université Pierre et Marie Curie, 75 230 Paris Cédex, France*

G. Weill

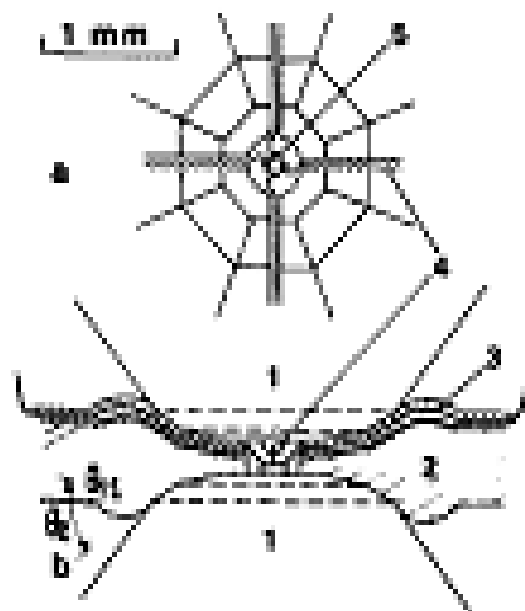
*Département des Hautes Pressions, Université Pierre et Marie Curie, 75 230 Paris Cédex, France*

(Received 1 July 1985; accepted for publication 16 September 1985)

A four-probe technique for electrical and photoelectrical measurements in a gasketed diamond anvil cell is presented. The special anvil profile and the method for the gasket preparation used in these experiments are described. Insulation and continuity of the electrical leads are shown to be satisfactory at least up to 18 GPa. As an example we report photoconductivity measurements in GaAs beyond crossover, under pressure, between direct and indirect band-gap configurations.

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Rev. Sci. Instrum. 57 (1) ; January 1986



**FIG. 1.** (a) Top view of sample and arrangement of the leads. (b) Cutaway view of gasket and anvil profile. Angles  $\theta_1 = 11.5^\circ$ ,  $\theta_2 = 23^\circ$ . (1) Anvil culets, (2) metallic gasket, (3) alumina layer, (4) grooves, (5) sample.



## Yunques implantados

Medidas de presión, de temperatura y de conductividad insertando en la estructura del diamante sensores mediante técnicas de implantación iónica.

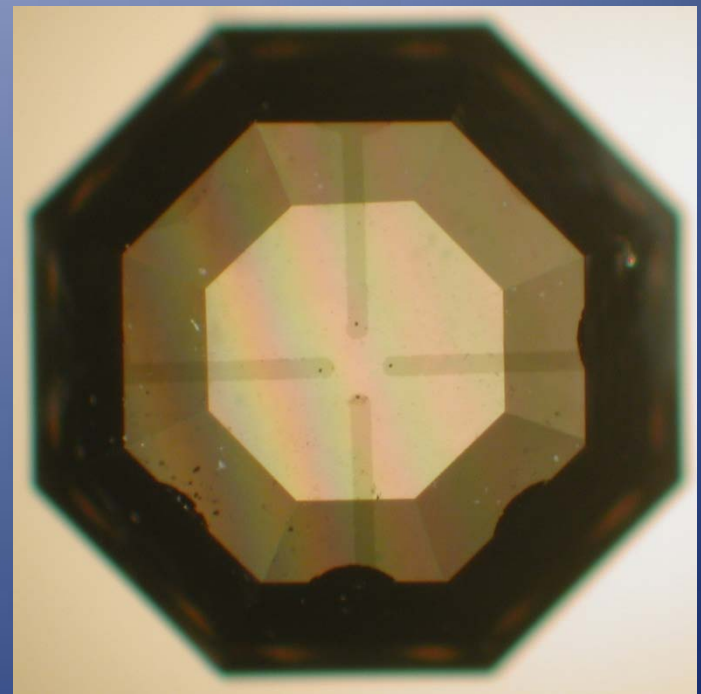
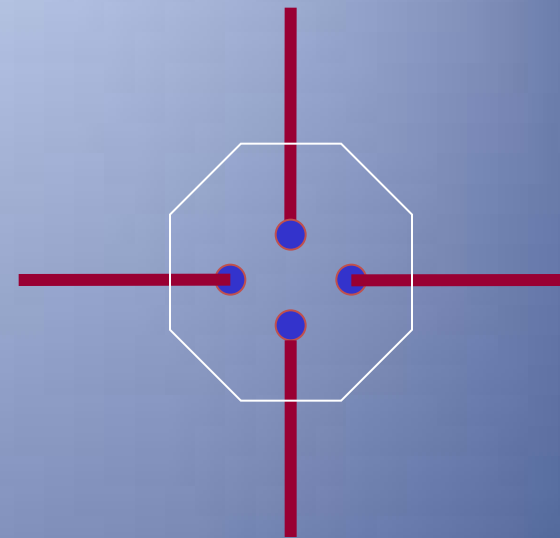
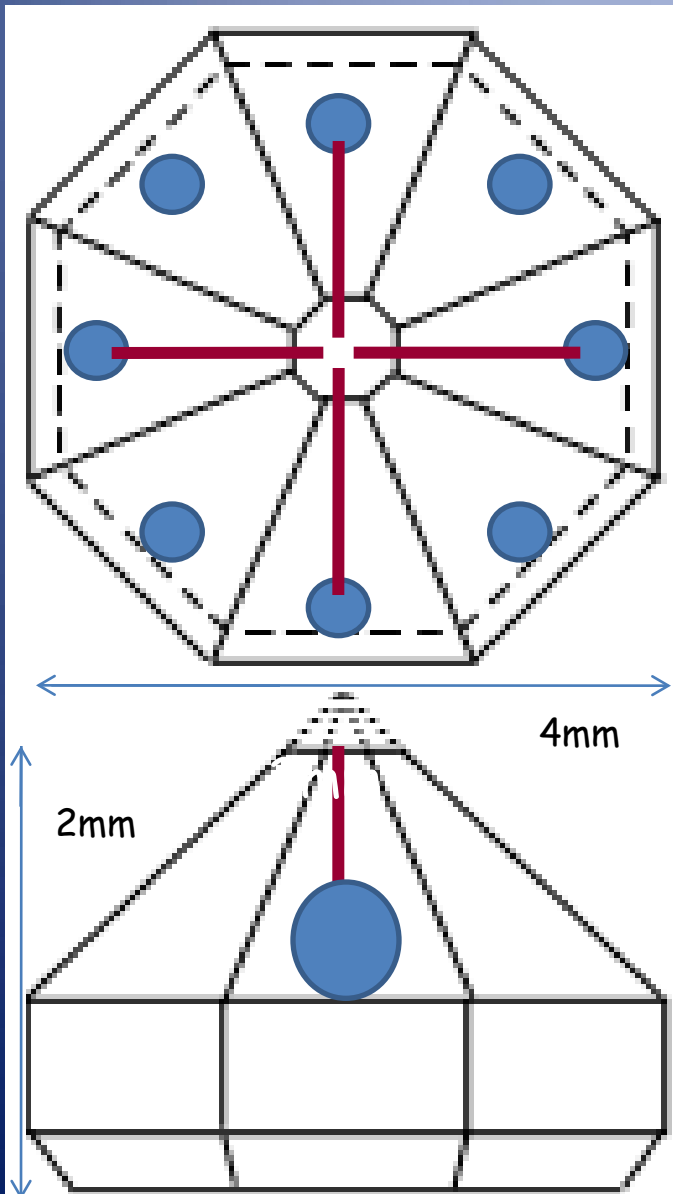
Las estructuras están así lo más cerca posible de la muestra y protegidas por el propio yunque.

## Tres aplicaciones

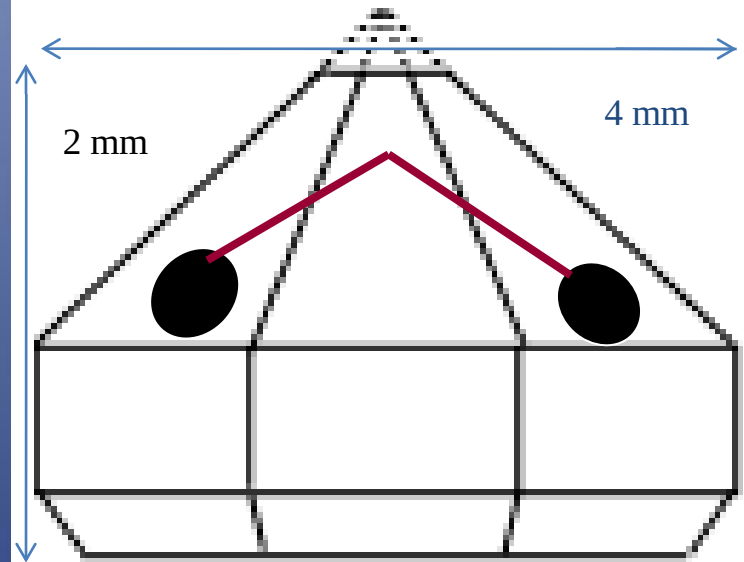
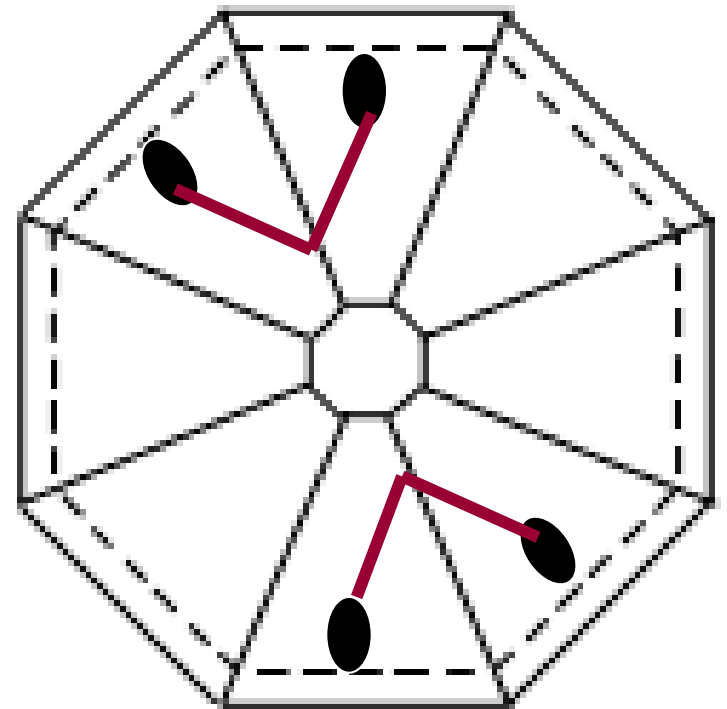
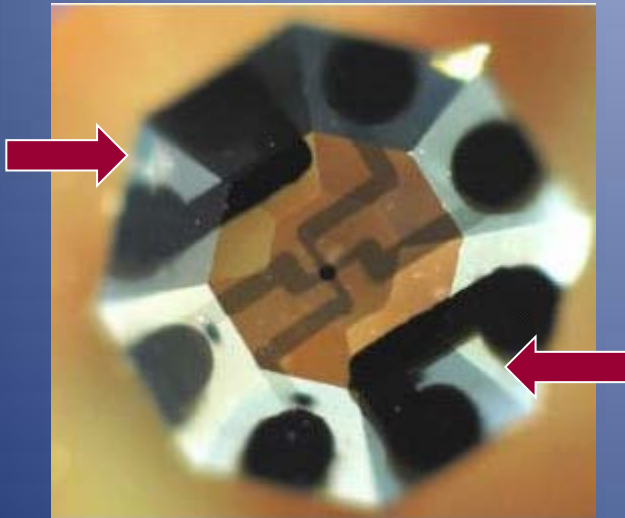
Implantar sensores de **presión** y de **temperatura**

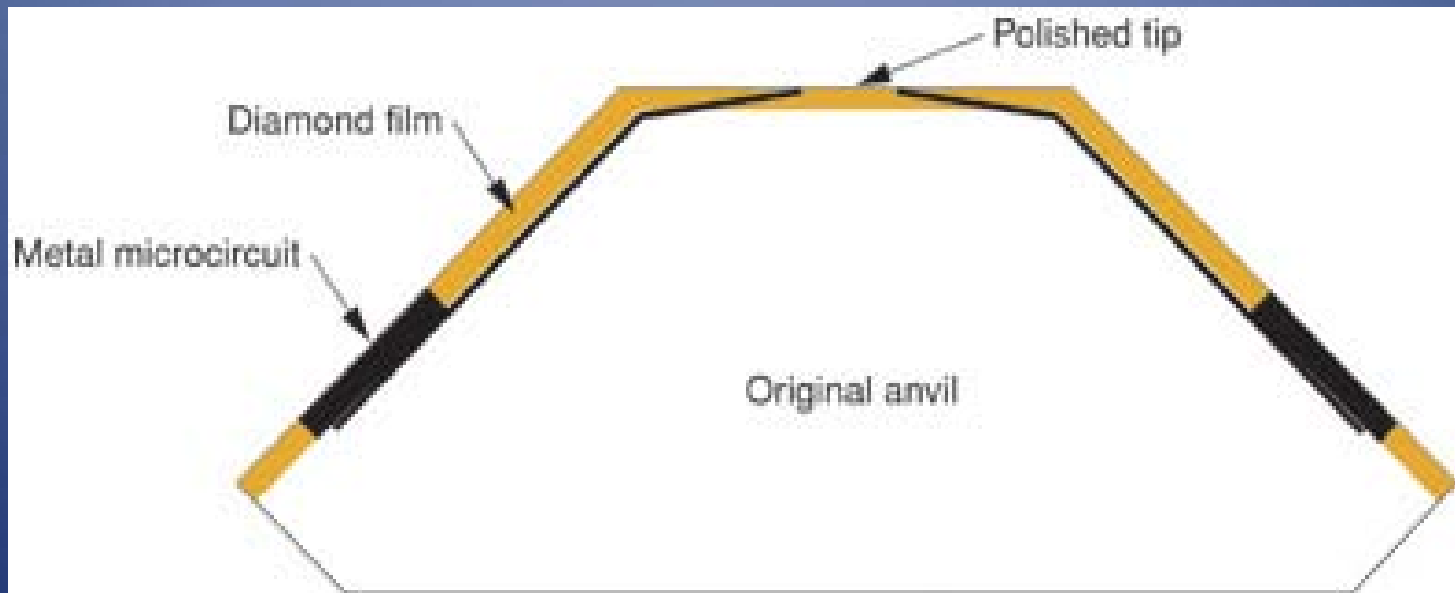
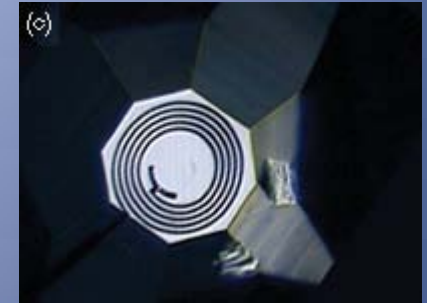
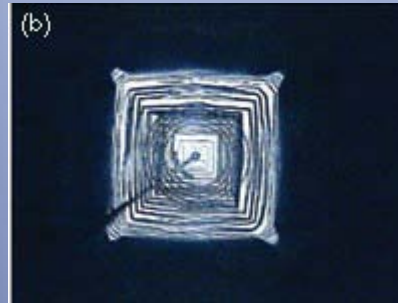
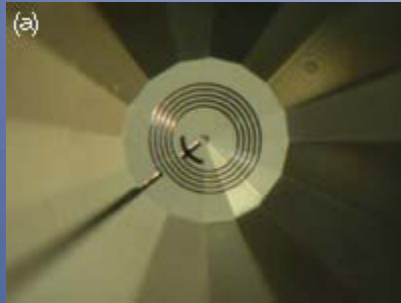
Implantar electrodos para medidas de **conductividad**

Implantar **resistencias** de calentamiento.

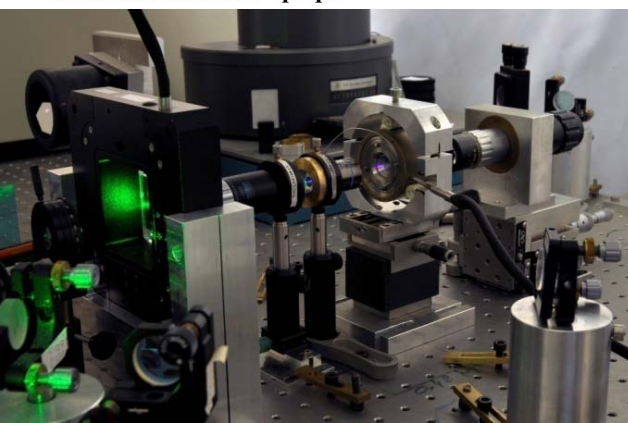
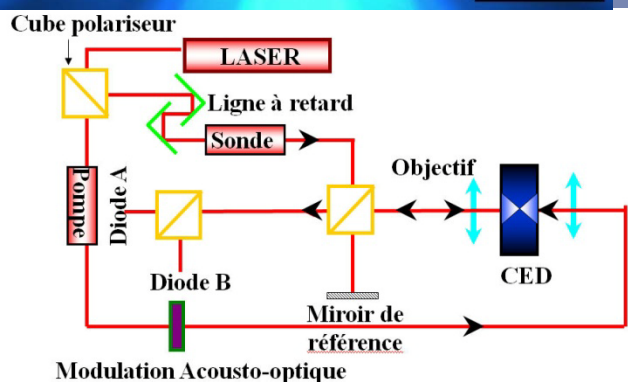
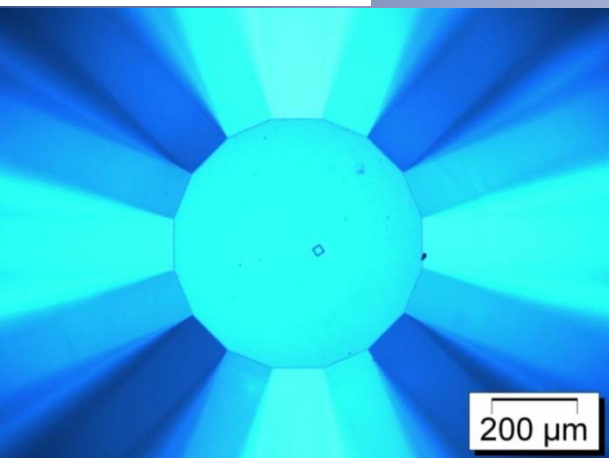


micro-fours









## Liquid mercury sound velocity measurements under high pressure and high temperature by picosecond acoustics in a diamond anvils cell

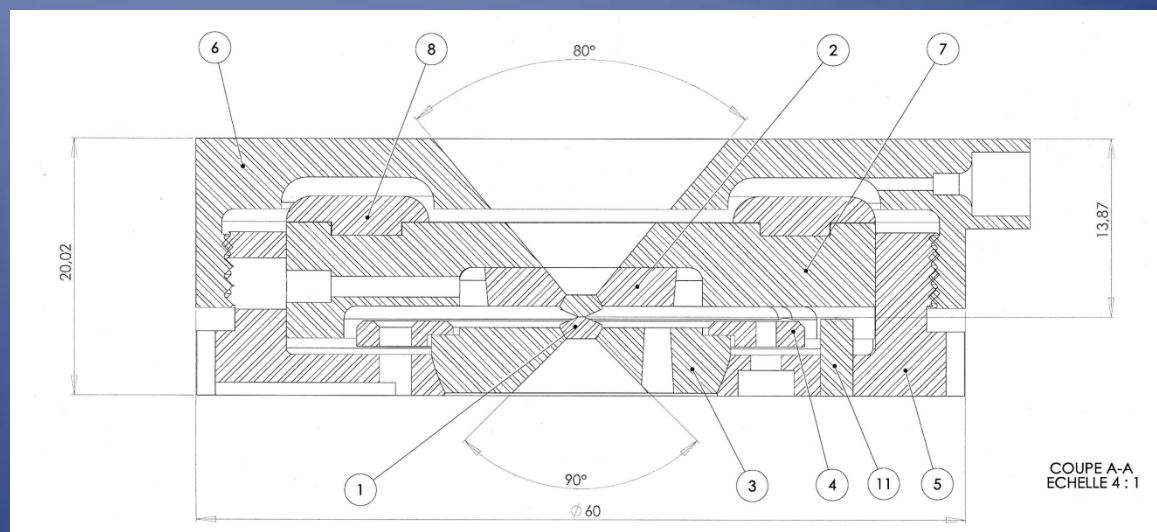
F. Decremps,<sup>1,a)</sup> L. Belliard,<sup>2</sup> B. Couzinet,<sup>1</sup> S. Vincent,<sup>2</sup> P. Munsch,<sup>1</sup> G. Le Marchand,<sup>1</sup> and B. Perrin<sup>2</sup>

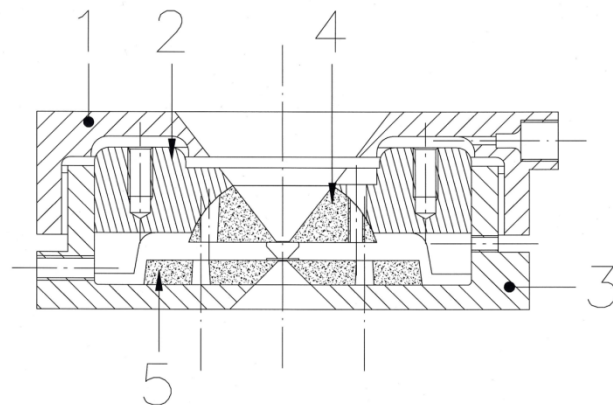
<sup>1</sup>Institut de Minéralogie et Physique des Milieux Condensés, Université Pierre et Marie Curie-Paris 6, CNRS UMR 7590, 140 rue de Lourmel, 75015 Paris, France

<sup>2</sup>Institut des NanoSciences de Paris, Université Pierre et Marie Curie-Paris 6, CNRS UMR 7588, 140 rue de Lourmel, 75015 Paris, France

(Received 28 April 2009; accepted 5 June 2009; published online 8 July 2009)

Recent improvements to measure ultrasonic sound velocities of liquids under extreme conditions are described. Principle and feasibility of picosecond acoustics in liquids embedded in a diamond anvils cell are given. To illustrate the capability of these advances in the sound velocity measurement technique, original high pressure and high temperature results on the sound velocity of liquid mercury up to 5 GPa and 575 K are given. This high pressure technique will certainly be useful in several fundamental and applied problems in physics and many other fields such as geophysics, nonlinear acoustics, underwater sound, petrology or physical acoustics. © 2009 American Institute of Physics. [DOI: 10.1063/1.3160104]





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High Pressure Research  
 Vol. 24, No. 1, March 2004, pp. 25–31



## Development of a low-pressure diamond anvil cell and analytical tools to monitor microbial activities in situ under controlled $P$ and $T$

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Received 31 August 2005; received in revised form 31 October 2005; accepted 14 November 2005  
 Available online 19 December 2005

### Abstract

We have designed a new low-pressure Diamond Anvil Cell (DAC), calibrated two novel pressure calibrants and validated the use of semi-quantitative Raman and X-ray spectroscopies to monitor the fate of microbes, their metabolism or their cellular components under controlled pressures and temperatures in the 0.1–1.4 GPa and 20–300 °C  $P,T$  range. The low-pressure DAC has a 250- to 600- $\mu\text{m}$ -thick observation diamond window to allow for lower detection limits and improved microscopic imaging. This new design allows the determination of cellular growth parameters from automated image analysis, which can be correlated with the spectroscopic data obtained on metabolism, ensuring high quality data collection on microbial activity under pressure. The novel pressure sensors offer the ease of use of the well-known ruby scale, while being more sensitive and reacting to pressure variations instantaneously.

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## COUPLING STATIC AND DYNAMIC COMPRESSIONS: FIRST MEASUREMENTS IN DENSE HYDROGEN

P. LOUBEYRE<sup>a,\*</sup>, P. M. CELLIERS<sup>b</sup>, D. G. HICKS<sup>b</sup>, E. HENRY<sup>f</sup>, A. DEWAELE<sup>a</sup>,  
 J. PASLEY<sup>c</sup>, J. EGGERT<sup>b</sup>, M. KOENIG<sup>c</sup>, F. OCCELLI<sup>a</sup>, K. M. LEE<sup>d</sup>,  
 R. JEANLOZ<sup>d</sup>, D. NEELY<sup>c</sup>, A. BENUZZI-MOUNAIX<sup>c</sup>, D. BRADLEY<sup>c</sup>,  
 M. BASTEA<sup>b</sup>, STEVE MOON<sup>b</sup> and G. W. COLLINS<sup>b</sup>

<sup>a</sup>Département de Physique Théorique et Applications, CEA, Commissariat à l'Energie Atomique, 91680 Bruyères-le-Châtel, France; <sup>b</sup>Lawrence Livermore National Laboratory, P.O. Box 808, Livermore, CA 94551, USA; <sup>c</sup>LULI Ecole Polytechnique, 91128 Palaiseau, France;

<sup>d</sup>Department of Earth and Planetary Science, University of California, Berkeley, CA 94720-4767, USA; <sup>e</sup>Central Laser Facility CLRC Rutherford Appleton Laboratory, Chilton, Didcot OX11 0QX, UK; <sup>f</sup>Dipartimento di Fisica, Università di Milano-Bicocca, 20126 Milan, Italy

(Received 14 August 2003; Revised 27 August 2003; In final form 28 August 2003)

We demonstrate here a laser-driven shock wave in a hydrogen sample, pre-compressed in a diamond anvil cell. The compression factors of the dynamic and static techniques are multiplied. This approach allows access to a family of Hugoniot curves which span the  $P$ - $T$  phase diagram of fluid hydrogen to high density. In this first-of-its-kind experiment, two hydrogen Hugoniot curves have been partially followed starting from pre-compression at pressures of 0.7 GPa and 1.2 GPa. Optical reflectance probing at two wavelengths reveals the onset of the conducting fluid state. The boundary line to conducting fluid hydrogen is suggested.

# 4º/ Cargamento medios hidrostáticos



# Hydrostatic limits of 11 pressure transmitting media

S Klotz, J-C Chervin, P Munsch, and G Le Marchand

IMPMC, Université P&M Curie, 140 Rue Lourmel, 75015 Paris, France

E-mail: [Stefan.Klotz@impmc.jussieu.fr](mailto:Stefan.Klotz@impmc.jussieu.fr)

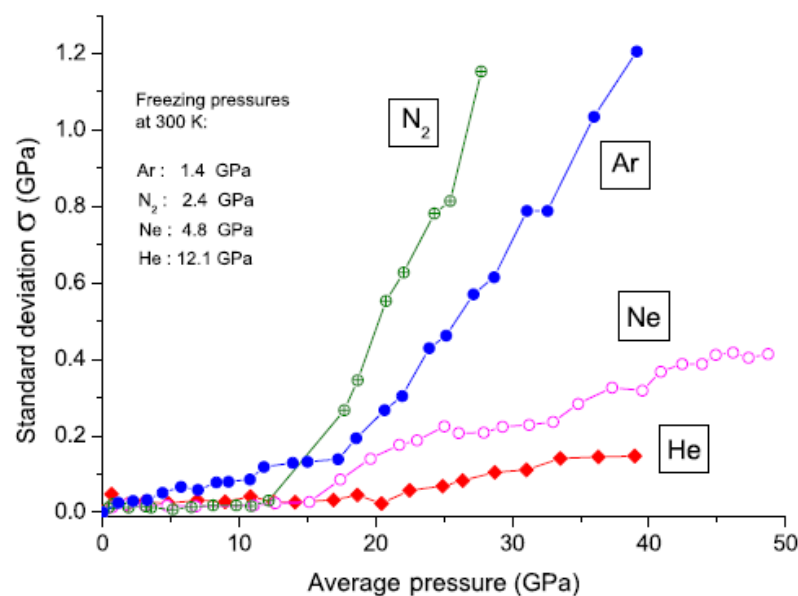
Received 23 December 2008, in final form 10 February 2009

Published 13 March 2009

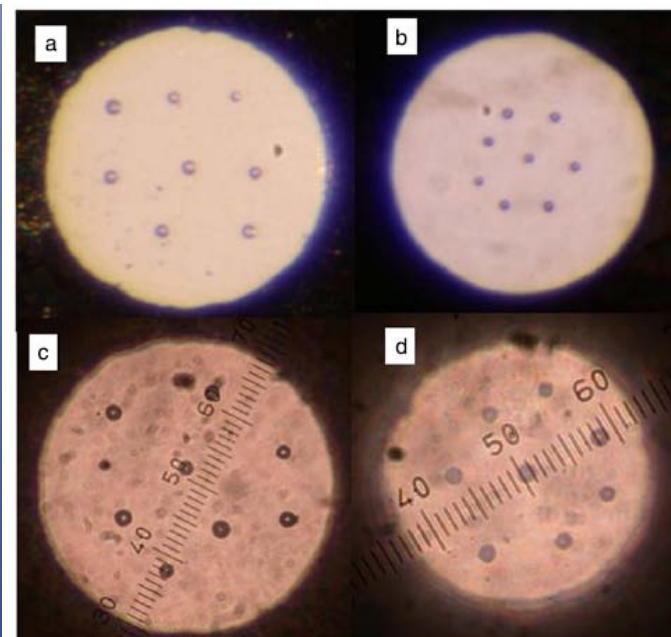
Online at [stacks.iop.org/JPhysD/42/075413](http://stacks.iop.org/JPhysD/42/075413)

## Abstract

We present a systematic and comparative study of the pressure-induced solidification of 11 frequently used pressure transmitting fluids using the ruby fluorescence technique in a diamond anvil cell. These fluids are 1 : 1 and 5 : 1 iso-n pentane, 4 : 1 deuterated methanol-ethanol, 16 : 3 : 1 deuterated methanol-ethanol-water, 1 : 1 FC84-FC87 Fluorinert, Daphne 7474, silicone oil, as well as nitrogen, neon, argon and helium. The data provide practical guidelines for the use of these fluids in high pressure experiments up to 50 GPa.



**Figure 8.** Comparison of pressure dependences of the standard deviation  $\sigma$  of  $N_2$ , Ar, Ne and He. The inset gives freezing pressures at 300 K cited in [26, 27, 28, 29] for Ar,  $N_2$ , Ne and He, respectively.



**Figure 1.** Pictures of DAC loaded with ruby spheres immersed in various pressure transmitting media: (a) 1 : 1 FC84-FC87 Fluorinert, (b) helium, (c) nitrogen, (d) Daphne 7474. Scales in (c) and (d): 1 division = 5  $\mu\text{m}$ .



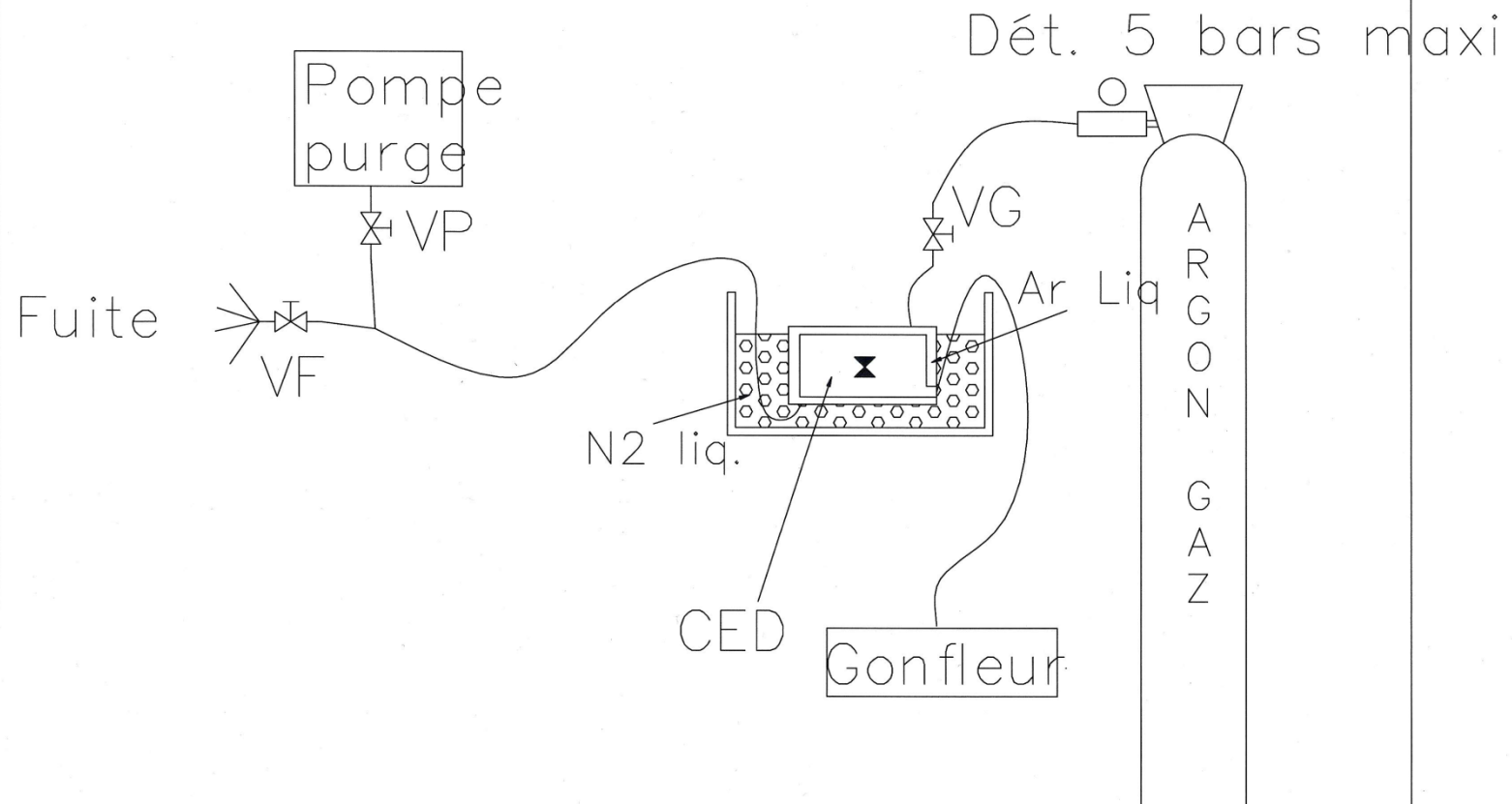
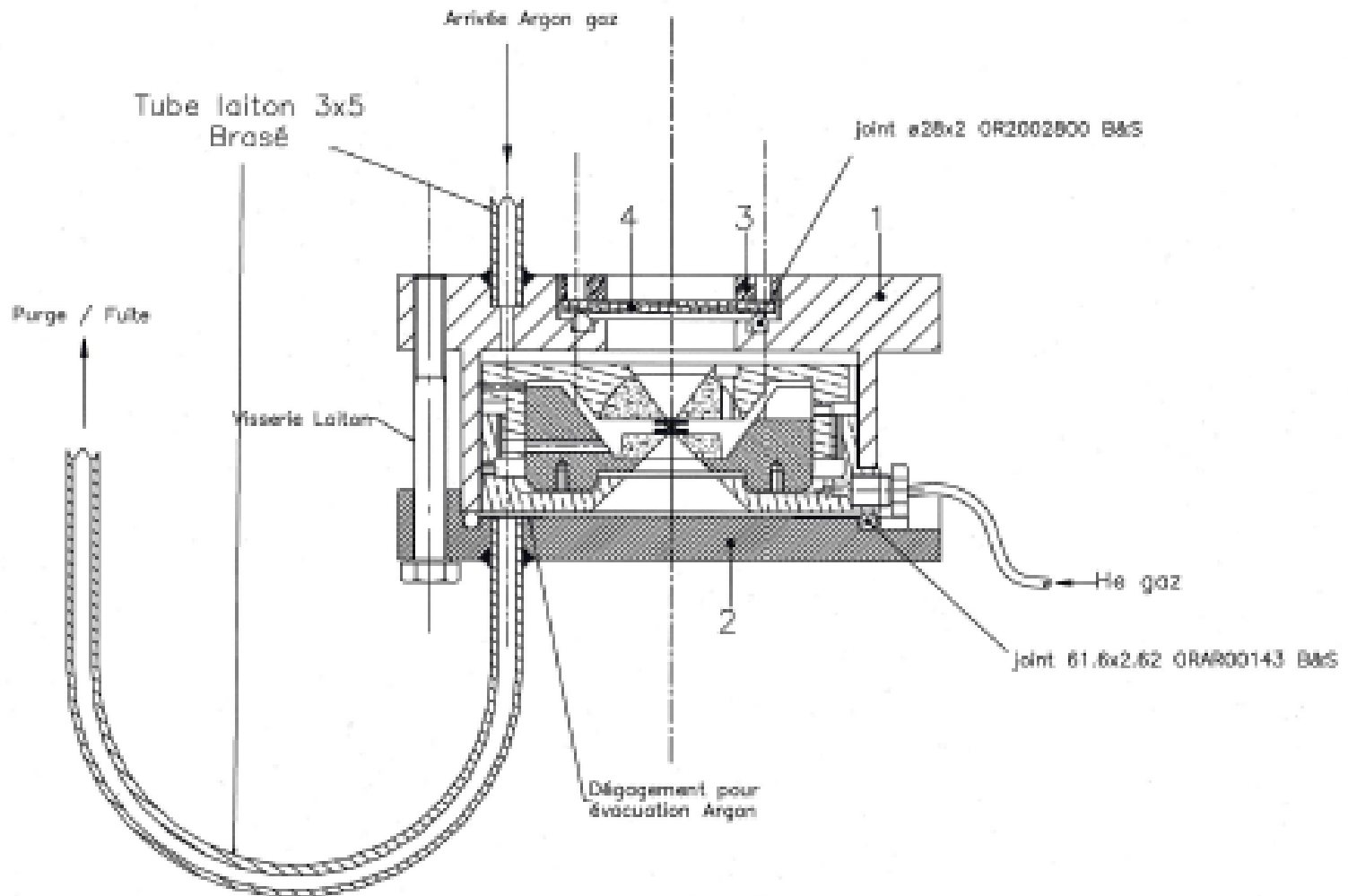


Schéma général chargement argon



High Pressure Research  
 Vol. 23, No. 4, December 2003, pp. 409–415



## OPTICALLY MONITORED HIGH-PRESSURE GAS LOADING APPARATUS FOR DIAMOND ANVIL CELLS

BERNARD COUZINET<sup>a,\*</sup>, NOËL DAHAN<sup>b</sup>, GÉRARD HAMEL<sup>a</sup> and  
 JEAN-CLAUDE CHERVIN<sup>a</sup>

<sup>a</sup>Laboratoire de Physique des Milieux Condensés, CNRS - UMR 7602, Université Pierre et Marie Curie, B 77, 4 Place Jussieu, 75252 Paris CEDEX 05, France; <sup>b</sup>Laboratoire de Mécanique et Technologie, ENS Cachan - CNRS - Université Pierre et Marie Curie, 61 Avenue du Président Wilson - 94235 Cachan CEDEX, France

We describe a high-pressure system built to load rare gases (He, Ar, Ne) in various types of diamond anvil cells, at room temperature. These gases are used as pressure transmitting media to obtain the best hydrostatic compression conditions in high-pressure experiments. Optical windows allow control of the loading process. The loading success rate is close to 100% and the initial pressures in the diamond anvil cell are in between 0.2 and 1 GPa. This system can easily be adapted for loading of various gaseous samples, including gas mixtures, which generally cannot be loaded by cryogenic methods.

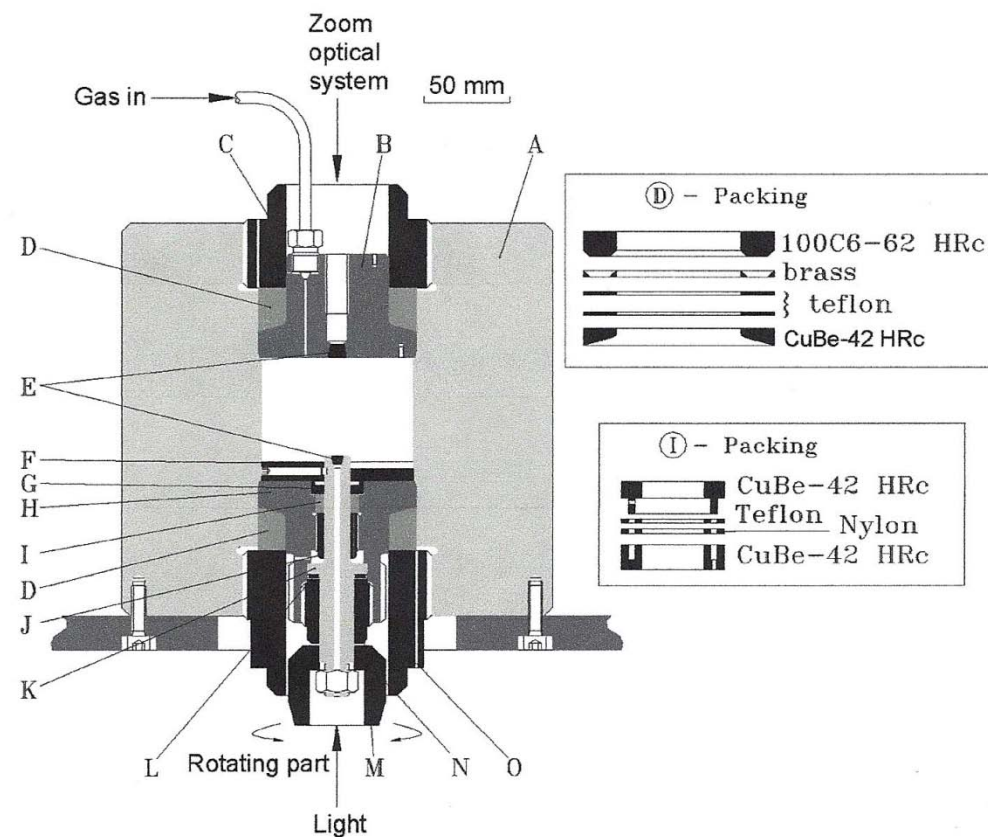


FIGURE 2 Cross-section of the loading apparatus. Insets show the sealing packings.







# Conclusiones

jean-claude.chervin@synchrotron-soleil.fr  
pascal.munsch@impmc.jussieu.fr

# Simulación de procesos a alta presión

**Á.M. Ramos<sup>1</sup>**

Collaborators: **J.M. Bello<sup>1</sup>** **J.A. Infante<sup>1</sup>** **B. Ivorra<sup>1</sup>** **L. Otero<sup>2</sup>** **J.M. Rey<sup>1</sup>** **P. Sanz<sup>2</sup>** **N. Smith<sup>1</sup>**

<sup>1</sup>Departamento de Matemática Aplicada; Universidad Complutense de Madrid; Spain

<sup>2</sup>Instituto de Ciencia y Tecnología de Alimentos y Nutrición (ICTAN); CSIC, Madrid, Spain



**QUIMAPRES**



**Simposio MALTA-Consolider**

Miraflores de la Sierra, 25-1-11

# ¿de qué os quiero hablar (y convencer)?

Uso de la modelización matemática y la simulación numérica

La simulación es la representación de un proceso o fenómeno mediante otro mas simple, que permite analizar sus características.



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# modelos matemáticos: representación abstracta de los procesos

Dichos modelos suelen venir dados por sistemas de ecuaciones (algebraicas, diferenciales ordinarias, en derivadas parciales, etc.) cuyas soluciones nos determinan el comportamiento del proceso que se quiere estudiar

Modelos bien planteados (en el sentido de Hadamard): existencia y unicidad de soluciones y dependencia continua respecto a los datos

La resolución analítica de las ecuaciones es en general imposible, pero gracias a la potencia de cálculo de las computadoras actuales, muchos de estos modelos se pueden resolver de forma aproximada, mediante técnicas y algoritmos numéricos (Elementos Finitos, etc.)

Implementando los algoritmos anteriores en el ordenador se obtienen paquetes informáticos que simulan los procesos, evitando hacer numerosos experimentos → ahorro de tiempo ... y de dinero.

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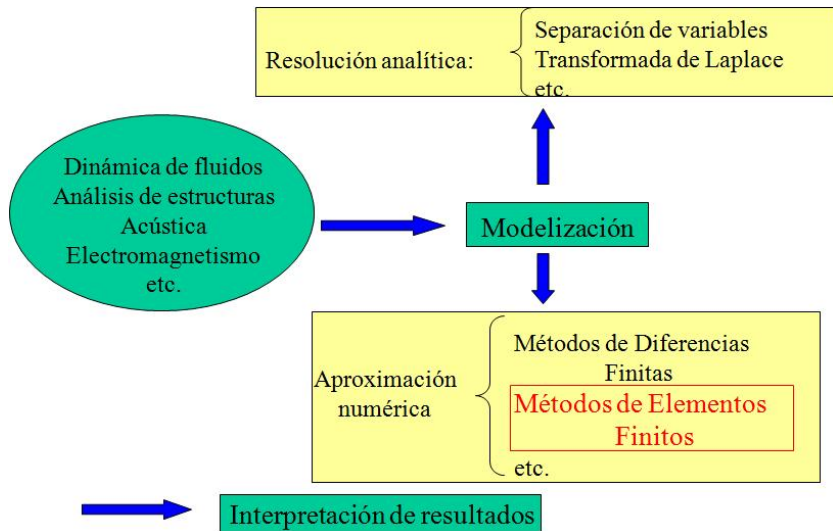
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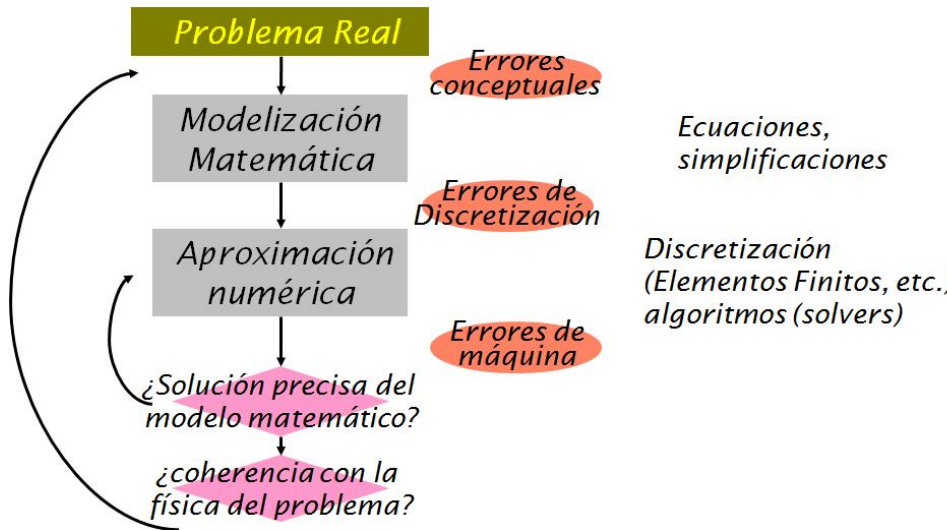
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# ¿cuales son los pasos a seguir?







## Tipos de Procesos a Alta Presión que son objeto de estudio en MALTA:

- 1 Procesos en células de pequeño tamaño (tipo DACs) y altísimas presiones → nuestro grupo no ha trabajado (todavía...) en la modelización de estos procesos.
- 2 Procesos en equipos de altas presiones con vasijas mayores (Ingeniería de alimentos, etc.)

# Esteban España S.A. (Spain), 2005, 600 MPa, NC Hyperbaric



# Modelling HP-T on microorganisms and enzymes

## Model for enzymatic inactivation

### Definition

**Inactivation:** Reduction of undesired biological activity, such as enzymatic catalysis and microbial contamination.

The enzymatic activity of a particular enzyme can usually be described by the first-order kinetic model

$$\begin{cases} \frac{dA(t; T, P)}{dt} = -\kappa(T, P)A(t; T, P) \\ A(0; T, P) = A_0 \end{cases}$$

$A(t; T, P)$ : enzymatic activity at time  $t$ , when the food sample follows a process at temperature  $T$  and pressure  $P$ ;  $A_0$ : initial enzy. act.;  $\kappa$ : inactivation rate

$$\Rightarrow A(t; T, P) = A_0 \exp\left(-\int_0^t \kappa(T(s), P(s))ds\right)$$

Identification of  $\kappa(T, P)$  (Inverse problem). Different  $\kappa$  for different enzymes

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## Model for microbial population

To describe the evolution of a microbial population, we can use the following model:

$$\begin{cases} \frac{dN(t; T, P)}{dt} = -\frac{\ln(10)}{D(T, P)} N(t; T, P) \\ N(0; T, P) = N_0 \end{cases}$$

$D(T, P)$ : *decimal reduction time*, or time required for a 1-log-cycle reduction in the microbial population.

$$\Rightarrow N(t; T, P) = N_0 10^{-\int_0^t \frac{1}{D(T(s), P(s))} ds}$$

Identification of function  $D(T, P)$  (*Inverse problem*). Different  $D$  for different microbes  
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$$N(t; T, P) = N_0 10^{- \int_0^t \frac{1}{D(T(s), P(s))} ds}$$

with  $\kappa(T, P)$  and  $D(T, P)$  suitably chosen.

- You choose curves for  $T$  and  $P$  and you get  $A(t; T, P)$  and  $N(t; T, P)$ .



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Puede que los resultados de este modelo no sean buenos.  
Entonces tenemos que corregir el modelo. **¿qué falta?**

- El modelo de inactividad anterior necesita como input:  $T$  y  $P$ . Pero...
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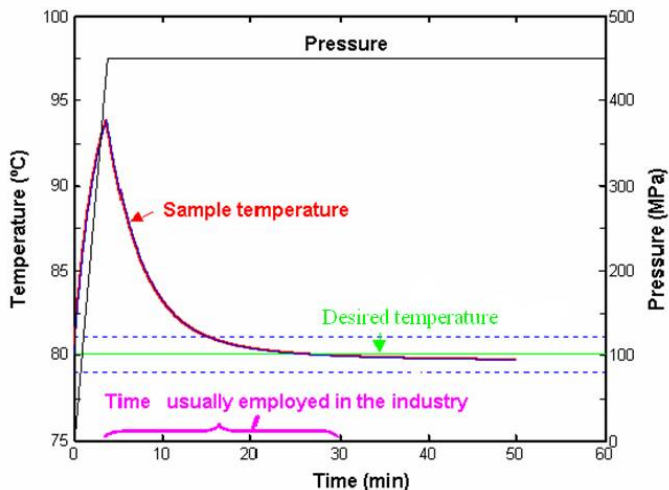
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## Typical pressure/temperature profile



# Models with ODEs or PDEs. Example:

Heat transfer by conduction and convection:

$$\left\{ \begin{array}{l} \rho C_p \frac{\partial T}{\partial t} - \nabla \cdot (k \nabla T) + \rho C_p \mathbf{u} \cdot \nabla T = \beta \frac{dP}{dt} T \text{ en } \Omega^* \times (0, t_f), \\ \rho \frac{\partial \mathbf{u}}{\partial t} - \nabla \cdot \eta (\nabla \mathbf{u} + \nabla \mathbf{u}^t) + \rho (\mathbf{u} \cdot \nabla) \mathbf{u} \\ \quad = -\nabla p - \frac{2}{3} \nabla (\eta \nabla \cdot \mathbf{u}) - \rho \mathbf{g} \text{ en } \Omega_F^* \times (0, t_f), \\ \frac{\partial \rho}{\partial t} + \nabla \cdot (\rho \mathbf{u}) = 0 \text{ en } \Omega_F^* \times (0, t_f). \end{array} \right.$$

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**Problem:** Identification of thermophysical properties and other parameters involved, for arbitrary temperatures and pressures.

### what can we do?

Atmospheric pressure/arbitrary temperature:

parameters known for many food similes and fluids

Arbitrary pressure/temperature:

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### Possible solutions:

- 1 Special formulas in literature for simple food similes: agar, tylose, etc. (see works by P. Sanz and collaborators)
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$$A(t; T, P) = A_0 \exp \left( - \int_0^t \kappa(T(s), P(s)) ds \right),$$

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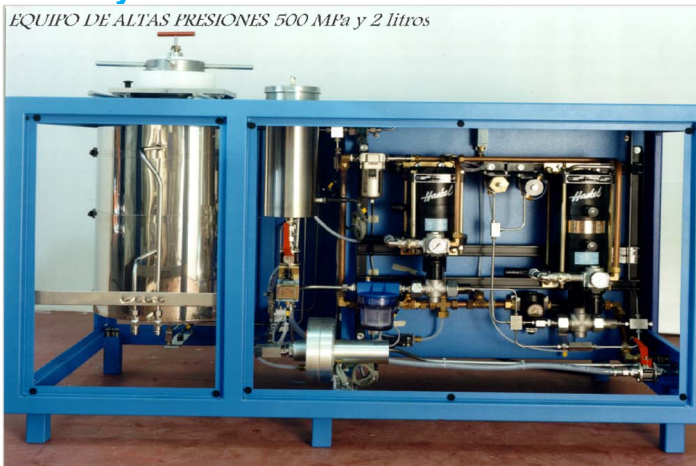


# Simulando procesos reales

Descripción del equipo de altas presiones considerado

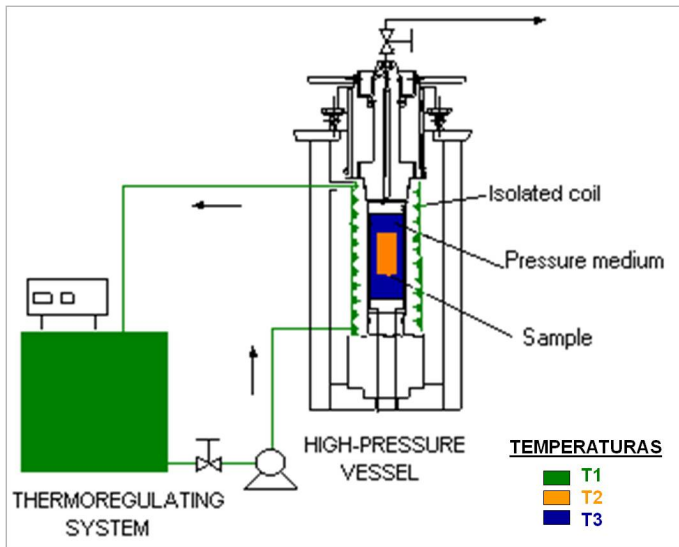
## Equipo en Instituto de Ciencia y Tecnología de Alimentos y Nutrición - CSIC - ACB GEC Alstom

*EQUIPO DE ALTAS PRESIONES 500 MPa y 2 litros*



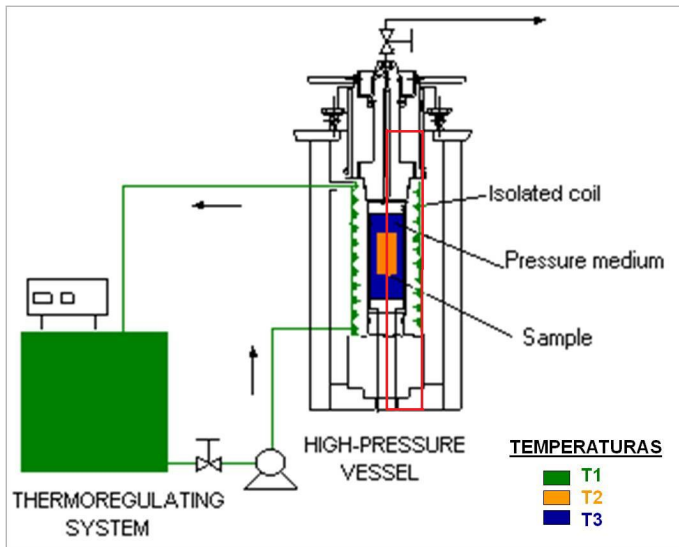
# Simulating real processes

Graphical description of the HP Equipment to be considered



# Simulating real processes

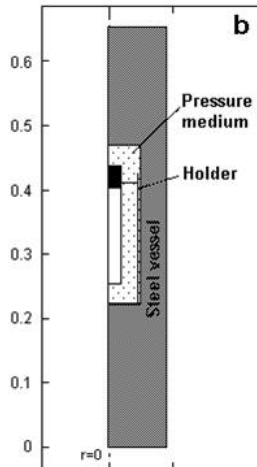
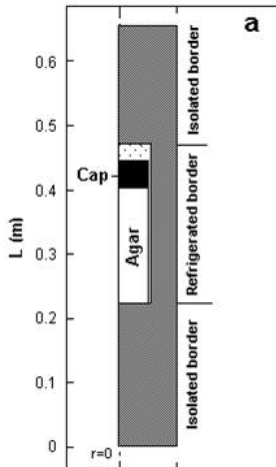
Graphical description of the HP Equipment to be considered



# Simulating real processes

Simplified model (via axial symmetry)

**2D model (r,z) - Computational domain** (big and small food sample)



## Equations governing the process

$$\left\{ \begin{array}{l} \rho C_p \frac{\partial T}{\partial t} - \nabla \cdot (k \nabla T) \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \end{array} \right. = \alpha \frac{dP}{dt} T \text{ en } \Omega^* \times (0, t_f),$$



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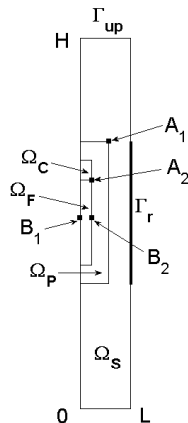
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## Initial and boundary conditions

$$\left\{ \begin{array}{l}
 T = T_r \text{ en } \Gamma_r^* \times (0, t_f), \\
 k \frac{\partial T}{\partial \mathbf{n}} = h(T_{\text{amb}} - T) \text{ en } \Gamma_{\text{up}}^* \times (0, t_f), \\
 k \frac{\partial T}{\partial \mathbf{n}} = 0 \text{ en } \Gamma^* \setminus (\Gamma_r^* \cup \Gamma_{\text{up}}^*) \times (0, t_f), \\
 T = T_0 \text{ en } \Omega^* \times \{0\}, \\
 \mathbf{u}_F = 0 \text{ en } \Gamma_F^* \times (0, t_f), \\
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 p = 10^5 \text{ en } A_1 \times (0, t_f), \\
 p = 10^5 \text{ en } A_2 \times (0, t_f).
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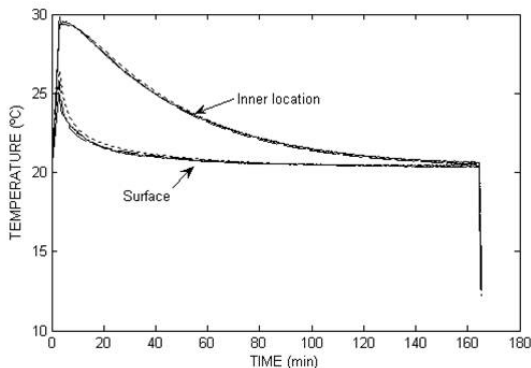


# Simulating real processes

## Example of the choice and validation of the models

L. Otero, A.M. Ramos, C. de Elvira and P. Sanz (2007)

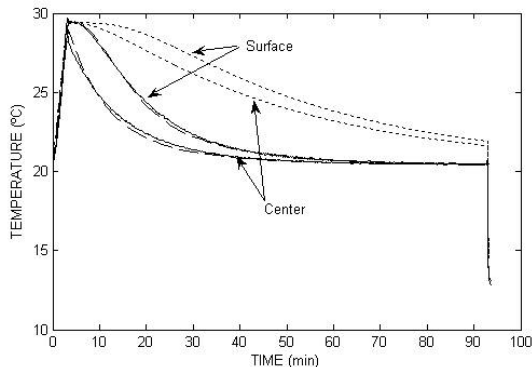
**First model (Heat transfer by conduction).** Big agar sample; 350 MPa;  $T_0 = 20^\circ\text{C}$ . (—) experimental data; (...) predicted values only with conduction; (- - -) predicted values including free convection



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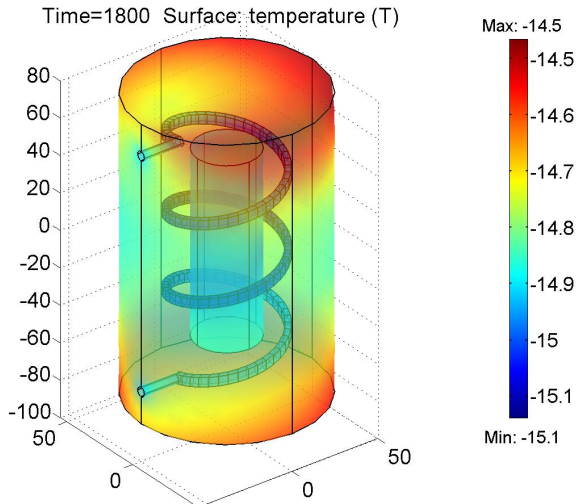
L. Otero, A.M. Ramos, C. de Elvira and P. Sanz (2007)

Second model (Heat transfer by conduction and convection). Small agar sample; 350 MPa;  $T_0 = 20^\circ\text{C}$ . (—) experimental data; (...) predicted values only with conduction; (- - -) predicted values including free convection





También se puede trabajar en 3D:



## Discretization of the enzyme activity

### Solid cases:

$$A(x, t) = A(x, 0) \exp \left( - \int_0^t \kappa(P(s), T(x, s)) ds \right)$$

$$A(x, t_i) \approx A(x, 0) \exp \left( - \frac{\tau}{2} \sum_{j=0}^{i-1} \left( \kappa_j(x) + \kappa_{j+1}(x) \right) \right), \quad i = 1, 2, \dots, n.$$

where

$$\kappa_j(x) = \kappa(P(t_j), T(x, t_j))$$

# Simulating real processes

## Discretization of the enzyme activity

### Liquid cases:

$$A(x, t_i) \approx A(X_0, 0) \exp \left( -\frac{\tau}{2} \sum_{j=0}^{i-1} \left( \kappa_j(X) + \kappa_{j+1}(X) \right) \right), \quad i = 1, 2, \dots, n.$$

$\kappa_j(X) = \kappa(P(t_j), T(X_j, t_j))$ ,  $X_j \approx X(t_j)$  and  $X$  is the trajectory ending at  $x$ .

The values of  $X_j$  are defined by using the implicit algorithm

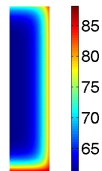
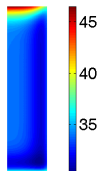
$$\begin{cases} X_n = x \\ X_i = X_{i+1} - \tau \mathbf{u}_F(X_i, t_i), \quad i = n-1, n-2, \dots, 0. \end{cases}$$

We take  $X_i$  as the solution of

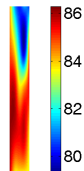
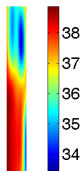
$$\min_{y \in \Omega_F} \|X_{i+1} - y - \tau \mathbf{u}_F(y, t_i)\|_2.$$

## Final Enzyme Activity (LOX)

Solid food



Liquid food

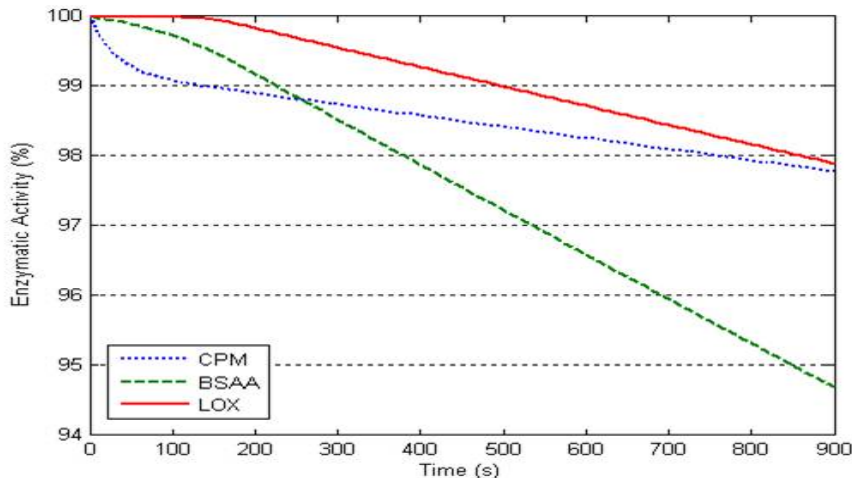


Process P1

Process P2

# Simulating real processes

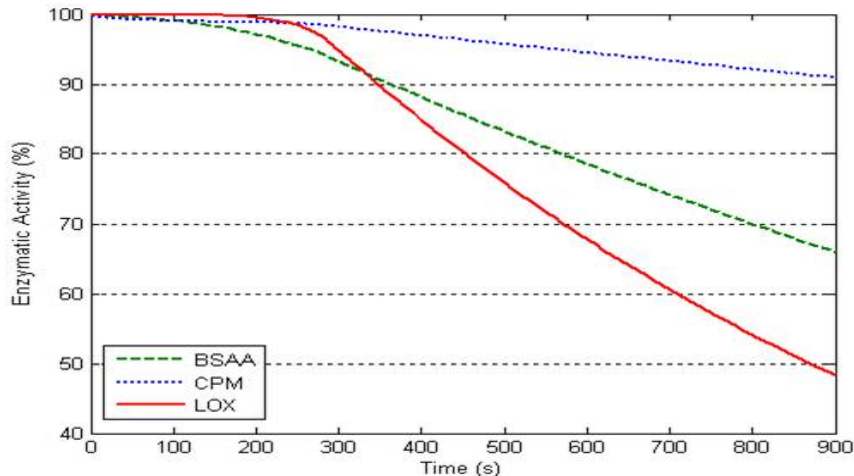
Numerical results for HP Process: 12°C → 20°C / 350 Mpa / Tylose





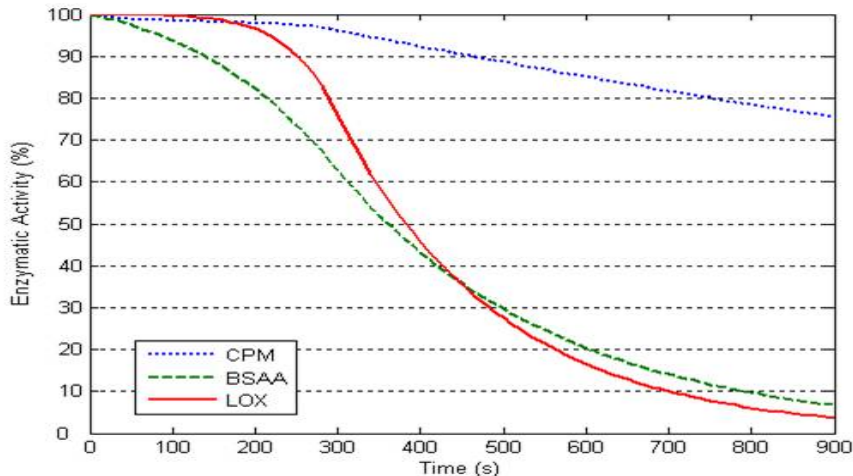
# Simulating real processes

Numerical results for HP Process: 22°C → 44°C / 600 Mpa / Tylose



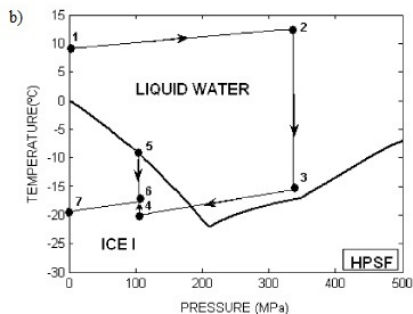
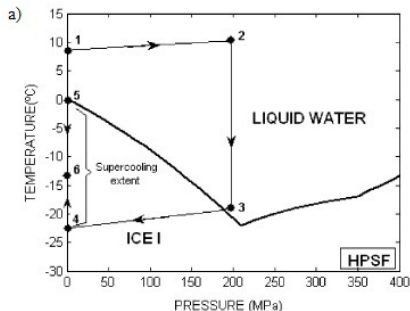
# Simulating real processes

Numerical results for HP Process: 40°C → 56°C / 600 Mpa / Tylose



# Modelling freezing processes with High pressure. HPSF processes

Ponencia de Nadia Smith



We are developing a heat transfer model for HPSF processes with **rapid** (left) or **slow** (right) expansions. For rapid expansion the **phase transition** occurs at **atmospheric pressure** conditions and for slow expansions **under pressure**.

New line of research (with J.M. Bello): Modeling of protein structures (folding and unfolding processes) depending on temperature-pressure conditions.

With the help of A. Rey and his collaborators.

# Conclusiones finales

- La modelización matemática es una herramienta muy útil para el diseño de equipos, la optimización de procesos, etc. que evita la realización de muchos experimentos ... permitiendo ahorrar dinero
- Importancia de la investigación Multi-disciplinar
- En el área de procesos de altas presiones en Ingeniería de Alimentos, hemos desarrollado modelos para predecir distribuciones de temperatura, campos de velocidades, inactivación enzimática y población microbiana.
- Actualmente estamos trabajando en procesos HPSF, comparación de vasijas horizontales y verticales y modelización de estructuras de proteínas.
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