ESRF high pressure laboratory: present and future

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ESRF, Grenoble, France
Present status
- Staff and services
- Advanced instrumentation available
- Off line facilities

Developments in view of EBS
HIGH PRESSURE AT ESRF USING DAC

ID06, ID15B, ID27, ID10: X-ray Diffraction – Structure, Crystallography, Strain, Deformation, …

ID18: Nuclear Resonance Scattering - Magnetism, Phonons

ID20: Resonant Inelastic X-ray Scattering - Electronic and Magnetic Structure

ID28: Inelastic X-ray Scattering, Diffuse Scattering – Phonons

ID12, BM23, ID24: XAS, XMCD - Local and electronic structure, Magnetism,

ID10: XPCS

ID02, ID26, BM01, BM30, ID09B, ID19, …. 

ID20: 72 Analysers and Panoramic DAC
Cryostat (T > 2.7 K)

Resistive heating (T < 1300 K)

Laser heating (T > 5000 K)

Magnetic field (H < 8 T)

LeToullec type DAC (P > 200 GPa)
HP LAB: STAFF AND SERVICES

STAFF:
- Jeroen Jacobs
- Gaston Garbarino

USER DEDICATED SPACE FOR DAC PREPARATION:
- From diamond purchasing to loaded DAC

HP LAB PURPOSES:
- SERVICE to extreme condition experiments
- LOAN POOL with all equipment for DAC experiments
- DEVELOPMENT of extreme condition equipment requested from beamlines

COMMERCIAL ACTIVITY:
- Around 5 DAC’s sold per year

• High pressure activity is growing every year
• Strong collaboration with beamlines
• Involved in several pioneering and strategic developments

DAC’s loan / year

HP-LAB is located in Sector 21
EQUIPEMENT AVAILABLE FOR USERS:

DAC’s ready to use:

- Standard
- Panoramic
- Large opening

Temperature

- $T > \sim 1000 \text{K}$: laser heating (YAG, CO$_2$)
- $T < 1300 \text{K}$: internal heating
- $T < 700 \text{K}$: external heating
- $T > 3 \text{K}$: CuBe alloy (Non magnetic)

- High T vacuum chamber
- High T inert atmosphere chamber
- Automatic pressure drivers
HP LAB: ADVANCED INSTRUMENTATION AVAILABLE

AVAILABLE SPACE: FOUR DEDICATED ROOMS
- user dedicated space (21.0.12, 21.0.13)
- super-user space (21.0.09)
- gas loading space (21.0.11)

AVAILABLE EQUIPMENT:
- Microscopes
- Spectrometer for ruby fluorescence measurement
- Indentation "stage"
- Laser drilling machine
- Gas loading machine

• Very positive impact on the quality of the produced data
• Increase the number of successful experiments due to the possibility to load / reload onsite
CHEMISTRY AND MICROIMAGING LABORATORY

CHEMISTRY LAB (H. Muller):
- Inert atmosphere sample manipulation, DAC loading
- Possibility to use glove box equipped with microscope ...

MICROIMAGING AND SAMPLE PREPARATION LAB (I. Snigireva)
- Sample preparation, polishing, cutting
OFF-LINE FACILITIES

OFF-LINE LASER ANNEALING (BEL.0.02, 14.0.09)

Present:
- YAG laser (2 x 50 Watts)
- Temperature measurement

Upgrade:
- CO₂ laser
- Raman spectroscopy
- YAG fiber (1 x 100 Watts)
OFF-LINE FACILITIES

OFF-LINE RAMAN SPECTROMETER (BEL.0.02, 14.0.09, 07.0.12)

H₂ vibron up to 180GPa
OFF-LINE CRYOGENIC CAPABILITIES (BEL.0.02, 14.0.09, 07.0.12)

**SES Pool**
- Cryostream (LN$_2$)
- Helijet (He)

**Available at ID15B - ID27**
- Oxford “blue” cryostat
- In-house made cryostats (x3)
- Designed to develop coupled techniques

**EXAMPLE: Test of strain device at low temperature**
- Developed for $\chi$, $\rho$, XRD, IXS, RIXS… Tested and characterized at room T “at home”
- Tested and characterized at low T “at ESRF” in same experimental conditions as on beamline
- Improved thermal contact and performances to be ready for beamtime on ID28-ID32-ID27

(Credits M. Souliou, M Le Tacon group)
Present status
- Staff and services
- Advanced instrumentation available
- Off line facilities

Developments in view of EBS
DEVELOPMENTS IN VIEW OF EBS

NEW AVAILABLE SPACE:
- user dedicated space (21.0.13)

NEW AVAILABLE EQUIPMENT:
- Fume-hood for Be manipulation
- Mouse controlled Micromanipulator
DEVELOPMENTS IN VIEW OF EBS

AVAILABLE SPACE:
- three user dedicated space (21.0.09, 21.0.12, 21.0.13)
- gas loading space (21.0.11)

LOAN POOL
- Improvement external/internal DAC heating system (Rosa - Jarnias)

EQUIPMENTS DEVELOPMENTS
- Improvement Micromanipulator
FEMTOSECOND LASER DRILLING MACHINE

Laser:
- $\lambda = 343, 515, 1030 \text{ nm}$
- Energy per pulse $\sim 80 \mu \text{J} @ 1030 \text{nm}$
- Pulse length $> 240 \text{fsec}$

Advantages:
- femto-sec pulse laser (ablative regime)
- fast, precise and large xy stage
  (100mm x 100mm)
- visualization

Capabilities:
- User friendly
- Sample preparation
- Gasket drilling
- Completely automatized laser machining
- Available for staff / users
- Other possible application consequence of drilling/machining in ablative regime

(Credits O. Hignette, P van der Linden)

Optimization:
- Damaged focusing achromat
- beam expander
- objective (replace lenses)
- visualization
DEVELOPMENTS IN VIEW OF EBS

NANOSECOND LASER DRILLING

FEMTOSECOND LASER DRILLING

50µm STST foil

26A (26A max)

7µm

100µm

100mm

1.2W (5W max)

100µm

100µm

1.2W (5W max)
DEVELOPMENTS IN VIEW OF EBS

NANOSECOND LASER DRILLING

Credits to I. Snigireva

FEMTOSECOND LASER DRILLING
Importance of high level offline and online equipments to perform the most challenging DAC loadings and experiments on-site.

- High pressure laboratory
- Laser drilling
- Sample manipulation
- Inert atmosphere sample manipulation and preparation
- Gas loading
- Off-line characterization: Raman spectroscopy, laser annealing, magnetic and transport properties
CONCLUSIONS - ACKNOWLEDGMENTS

- Importance of high level offline and online facilities to perform the most challenging extreme conditions experiments at ESRF

- HP-lab has a key role and is involved in several pioneering and strategic scientific instrumental developments

- Projects and motivation to keep ESRF high-pressure programme at the forefront of the field within ESRF - EBS.

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