X AUSE Conference &
V ALBA User's Meeting

Catalin Popescu

Future of High Pressure @ ALBA

08.09.2022

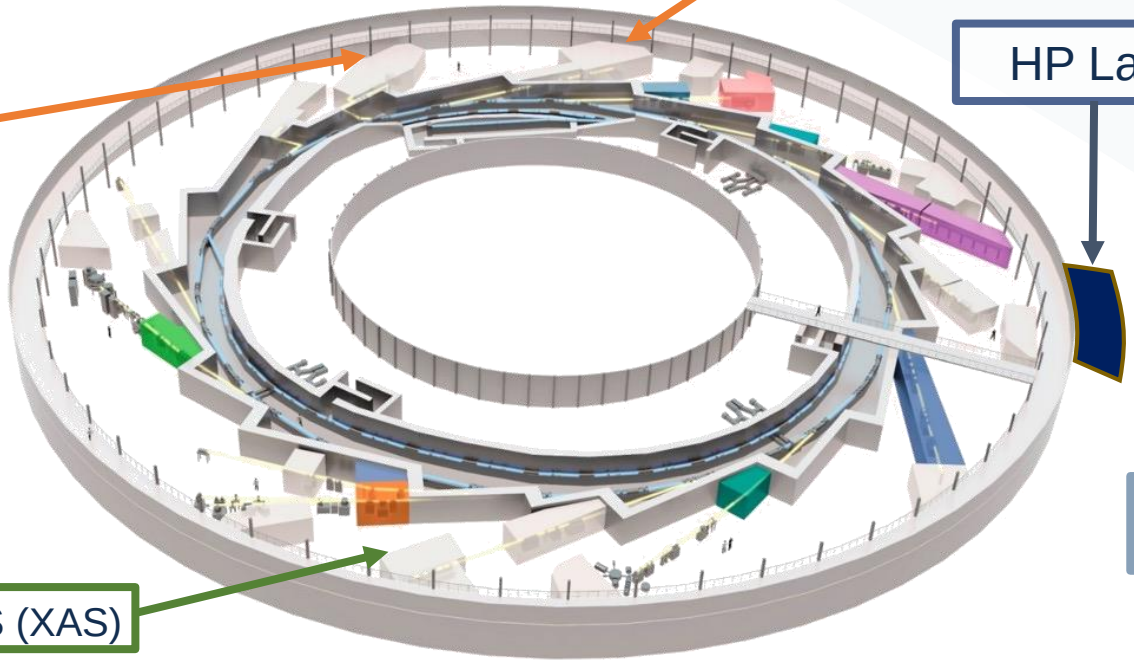
Outline

- status of HP techniques: statistics, current capabilities, support services
- swot analysis
- gap analysis
- overview

HP techniques at ALBA

HP facilities :

- MSPD (HP station) + MIRAS
- HP Lab
- MATEX (proposed in 1st call 2021)



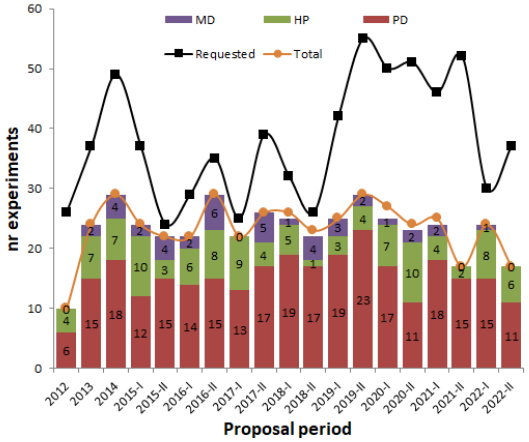
Fully dedicated

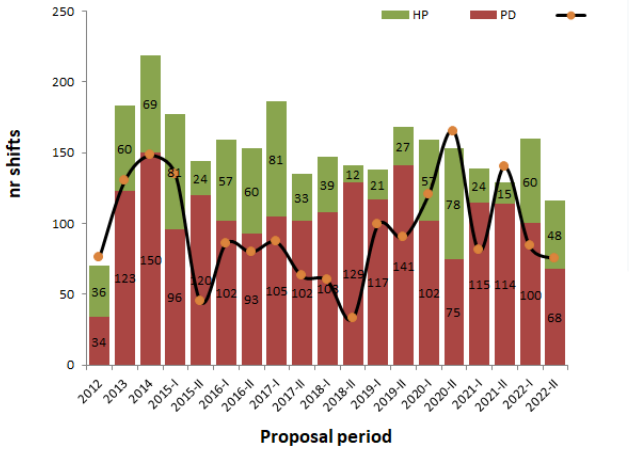
Partially dedicated

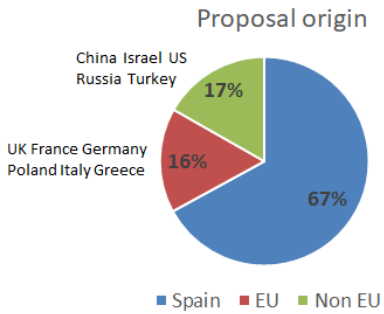
potential

proposed

Statistics on HP activity



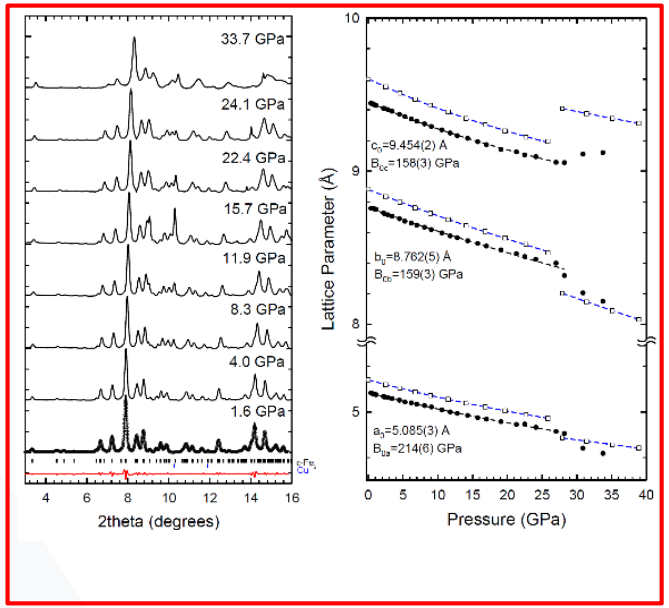




- statistics based on MSPD beamline
- shared BL + 30% quota by staff (natural limit 1 pers)
- high productivity of HP publications (1 /experiment)
- wide community (over 47 groups) & working hard to get beamtime

Current capabilities: facilities

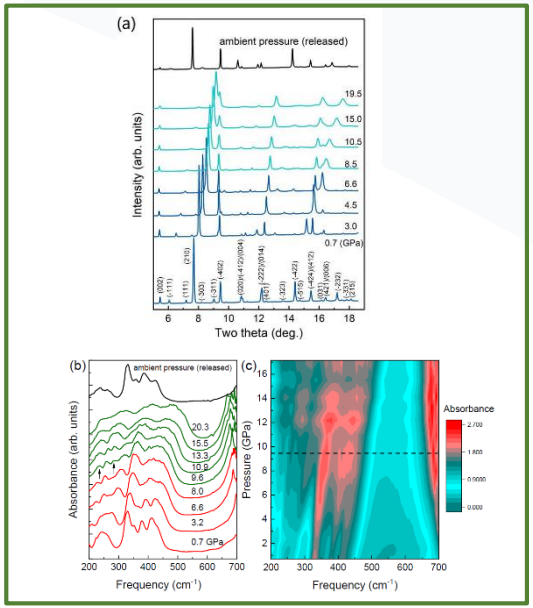
XRD Long range structure



Sans et al. 2018, Nat. Comm.

ID27 ESRF & MSPD @29.2 keV

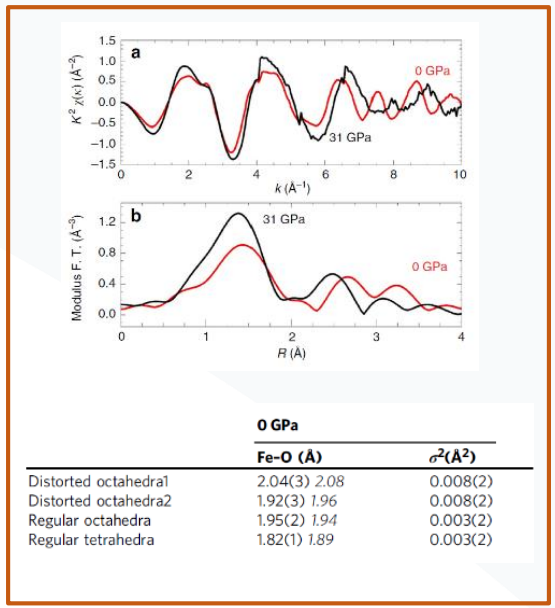
FTIR in situ chemical composition electronic structure



Liang et al. 2022, Phys.Rev.B

MIRAS & MSPD @Alba

XAS local and electronic structure element selective

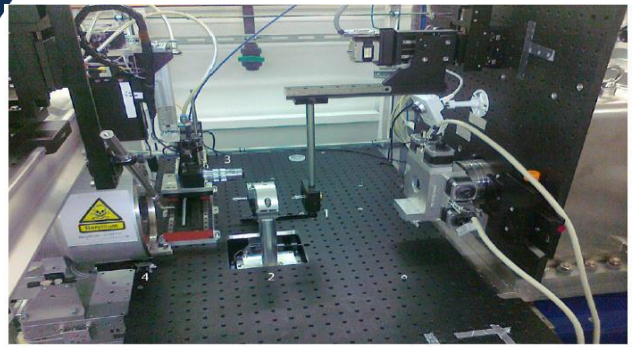


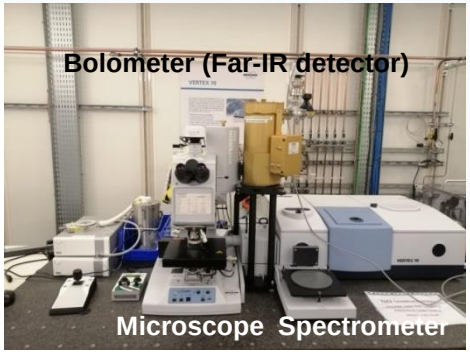
0 GPa		
	Fe-O (Å)	σ^2(Å^2)
Distorted octahedra1	2.04(3) *2.08*	0.008(2)
Distorted octahedra2	1.92(3) *1.96*	0.008(2)
Regular octahedra	1.95(2) *1.94*	0.003(2)
Regular tetrahedra	1.82(1) *1.89*	0.003(2)

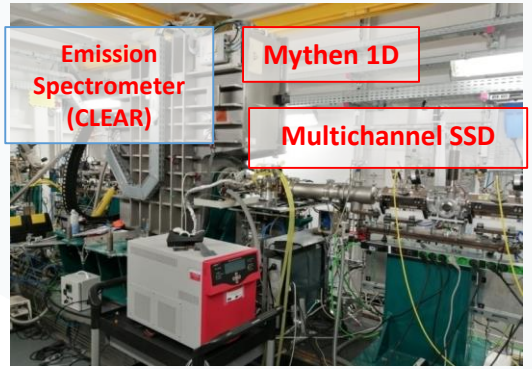
Sans et al. 2018, Nat. Comm.

BM23 ESRF @Fe K edge

Existing HP tools







HP XRD @MSPD

- beam size **25x25 µm²** (FWHM)
- Pmax ~ 100 GPa
- T = 12 K ---> 1300 K
  lHe cryostat, resistive heating

HP IR @MIRAS

- beam size MID-IR: 10 µm
- beam size FAR-IR: 50-70 µm
- photon energy: 1.2 µm to 100µm

HP XAS @CLAESS

- beam size : 61 x 68 µm² (pinhole)
- need for nano polycrystalline
- upgrade matches microfocus program (undulator, KB mirrors)

85%

14%

1%

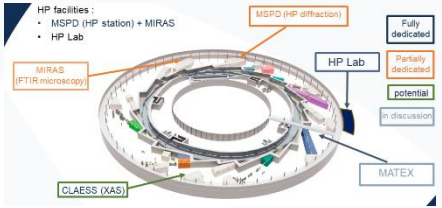
Current capabilities: HP lab

HP Lab purpose:

- service High Pressure BLs
- loan pool and development
- limiting supporting infrastructure (1 person)

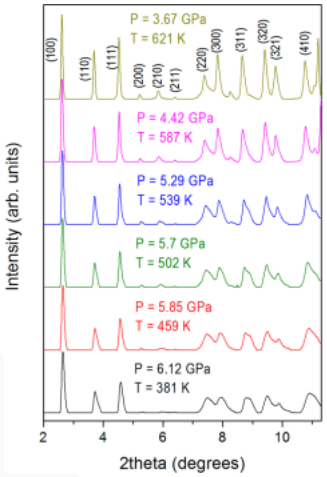
HP Lab equipment:

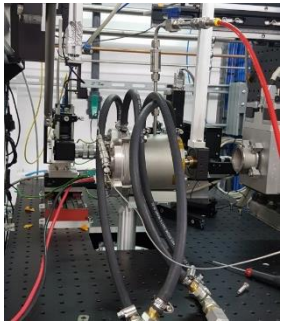
- microscopes for DAC loading
- indentation ‘stage’
- offline PRL for ruby fluorescence
- instrumental development
- laser drilling machine (soon)

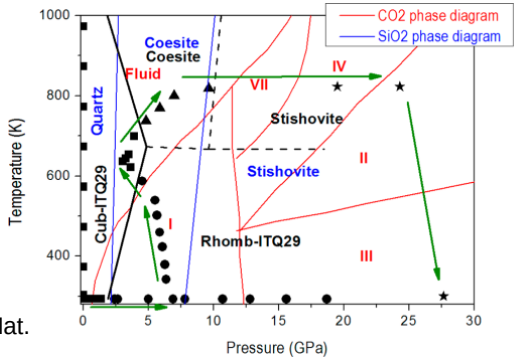


where we are

HP-XRD:Functional porous, molecular and framework materials: CO_2 filled zeolite @HP-HT

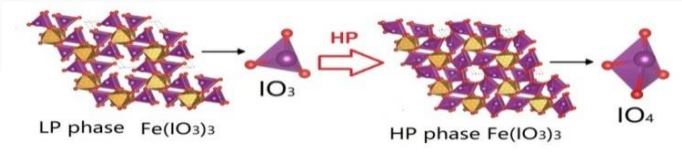


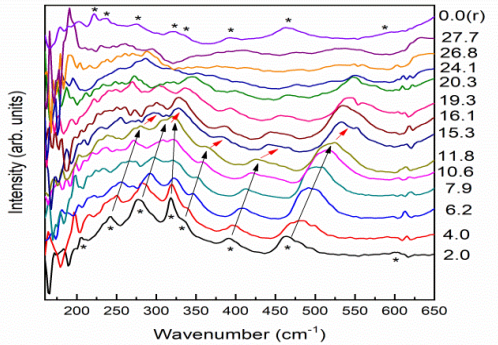




SantaMaria et al. 2017, Chem. Mat.

HP-IR: first order isostructural phase transition $Fe(IO_3)_3$





Liang et al. 2020, J.Phys.Chem.C

earth science, functional materials, superconductors, thermoelectrics, barocaloric, vdW materials, …

Support services

HP X-ray diffraction @MSPD
High productivity | driving force HP program
Big beam for HP experiments (wiggler source)
Upgrade detector (obsolete)
Online visualization, automate data collection

Far-IR spectroscopy @MIRAS
Vertical geometry & working distance limiting SE
Automate data collection (automate pneumatic drive)

HP X-ray absorption @CLAESS
Flux, missing focusing optics
nano polycrystalline 💎 (glitch free)

High Pressure laboratory
limiting supporting infrastructure
Sample environment pool
shared between BLs

SWOT

Strengths

- high productivity HP publications
  1 publication/experiment
- stable and wide user community with over 47 groups
- wide temperature range (MSPD)

Weaknesses

- limiting supporting infrastructure (HP lab)
- small earth science community
- missing automatic data collection
- obsolete instrumentation: detector (MSPD)
- limiting geometry of IR program (MIRAS)
- no BAG-like proposals for consortium groups

Threats

- competitive HP programs at other facilities
- competing with other programs in Alba serving wide user community
- no laser heating

Opportunities

- growing the HP laboratory to serve the characterization capabilities of Alba
- enlarge international user community by customer oriented program
  - dedicated HP beamline (matching Alba II)
  - Alba workshop (Oct 28th 2021)

Gap Analysis

where we are

- limiting support laboratory
- limited availability of multimodal techniques (XRD, FTIR, XAS, XES & tomography)
- beam size limitation for HP-XRD hampers users at MSPD
- limited availability of sample environment (IR – missing gas loading capability)
- no time resolution capabilities

where we want to be

- ✓ Adjust staffing and add suitable infrastructure
- ✓ develop multimodal data analysis methodologies
- ✓ providing microXRD based on undulator concept (4th generation upgrade)
- ✓ Enhancing characterization capabilities
  - develop Cassegrain microscope (IR)
  - gas loading system (all applications)
- ✓ integrate time resolution experiments by providing suitable data collection and sample environment

HP laboratory

HP Lab role:

- Service HP BLs| loan pool and development
- size of HP lab: limit factor of the HP program (1pers)

HP Lab equipment:

- Microscopes for DAC loading
- Indentation ‘stage’
- offline PRL for ruby fluorescence
- Instrumental development
- Laser drilling machine (soon)

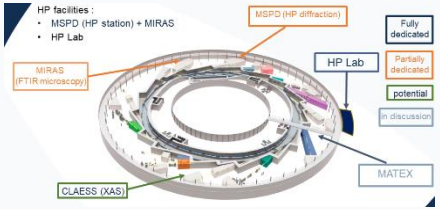
HP Lab extension:

- gas loading machine
- inert atmosphere DAC handling
- off-line characterization

Adjust staffing

1 dedicated technician

1 beamline scientist +



HP multimodal approach

Beamline

spatial resolution (5μm) for HP X-ray diffraction
Undulator source at **HP-MSPD** (15-30 keV+)
Upgrade detector (high energy efficiency)
Complementing with dynamic compression (piezo actuators DAC) & multichannel collimator (low Z elements)

Far-IR spectroscopy program (5μm)
develop home made Cassegrain microscope to move to horizontal geometry

spatial resolution (5μm) for HP X-ray absorption
Undulator source at **CLAESS** (5-30 keV+)
KB mirrors as focusing optics
nano polycrystalline 💎 (glitch free)

High Pressure laboratory
Sample environment shared between BLs
Laser heating (pulsed/flash 6000K)
Internal resistive heaters (2000 K)
gas loading machine

X-ray Emission Spectroscopy
Evaluate dedicated beamline or mini XES spectrometer for existing BL

X-ray Imaging
Evaluate tomography in large volume cell for existing BL (Faxtor)

Computing

Multimodal data analysis
Online visualization
Data base access

HP microXRD @ALBA

Beam size limitation for XRD at MSPD

MSPD upgrade
Undulator source at **HP-MSPD** (15-30 keV+)
Upgrade detector

Scenario 1

Microfocus PD BL: HP program + microXRD (5µm)
Undulator source (15-30 keV +)
High energy detector

Scenario 2

MATEX beamline (5µm) for HP XRD
Undulator source (20-40 keV)
KB mirrors as focusing optics
High energy detector

Scenario 3

Enhancing characterization capabilities

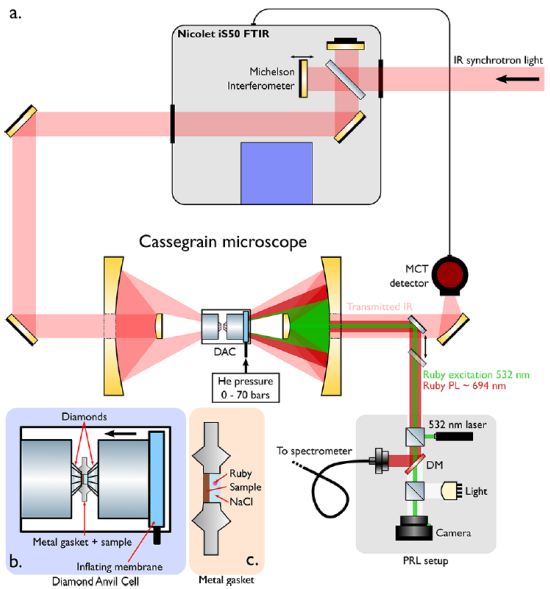
MIRAS: vertical microscope geometry

- limiting working distance > missing sample environment

horizontal microscope geometry

home-made Cassegrain microscope

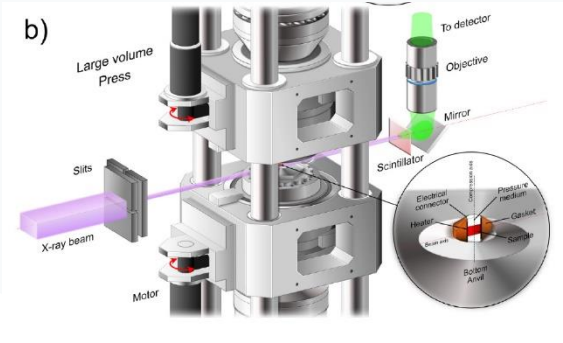
not available commercially



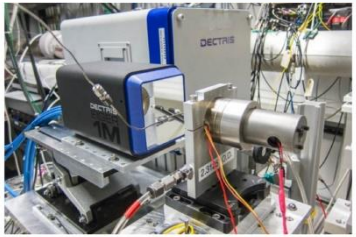
SMIS – Soleil Synchrotron
Livache et al. 2019, J.Phys.Chem.C

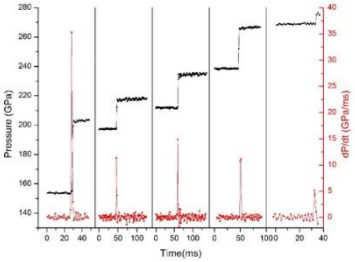
Time resolution @HP

- Time resolution @HP: dynamic processes, kinetics of chemical reactions
- Evaluate tomography in Large Volume Cell (Paris-Edinburgh or RoToPEc) in **FAXTOR BL** (@20-70 keV)
  - P ~ 20 GPa & T<1800K |volume sample 2-5 mm^3
  - larger beam to LVC and images directly to 2D detector
  - FoV: 1-10 mm | resolution ~ 1 μm
- XRD in dynamic DAC: piezo-actuators
- computing: develop pipeline and data analytics



Guignot et al. 2020, J.Appl. Phys.





HPCAT 16-ID-B beamline @APS

Matter AT EXtremes (MATEX) beamline

main proposer: Juan Ángel **Sans** (UPV & BL design)

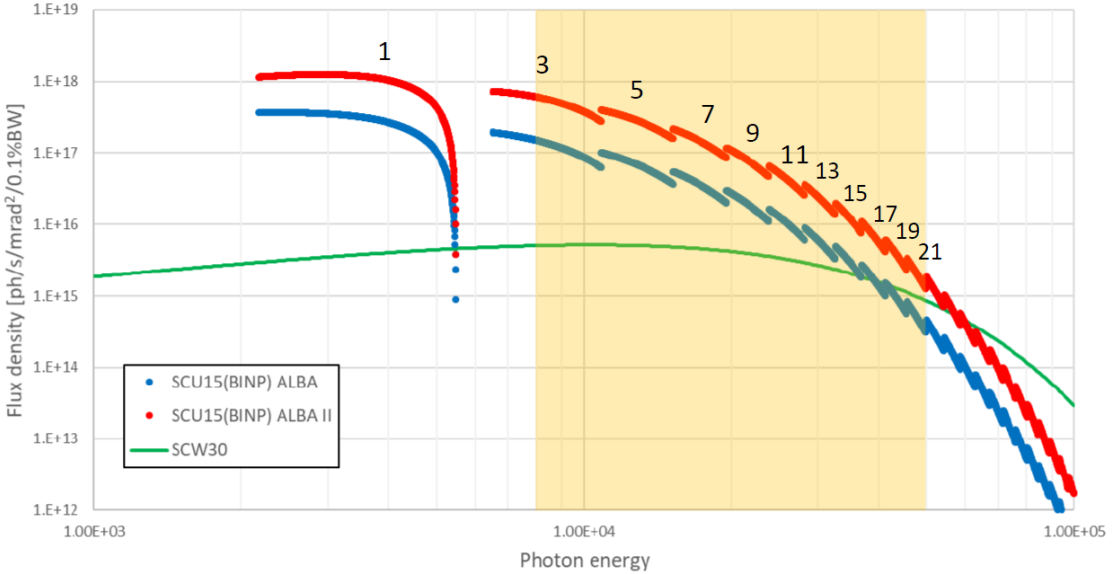
co-proposers: Jose Antonio **Alonso** (ICMM-CSIC & scientific case), Vera **Cuartero** (UNIZAR & scientific case), Daniel **Errandonea** (UV & BL design), Virginia **Monteseguro** (UCAN & scientific case), Miriam **Peña-Alvarez** (U. Edinburgh & scientific case), Catalin **Popescu** (ALBA & BL design)

MALTA-Team (20 Spanish groups), EHPRG (European HP community) and AIRAPT (International HP community) supported this proposal

Superconducting undulator for ALBA II

New design based on SCU & replace SCW30 of MSPD

Main parameters:	
Magnetic field, T	1.2
Period, mm	15.6
Maximum K	1.75
Magnetic gap, mm	8
Period number	119
Magnetic length, mm	~2000
Current,A	250
SC wire diameter, mm	0.55
Turn number in layer	12/11
Layer number	7
Width of coil, mm	146
SC wire critical current 7T	250
Ratio Cu/NbTi	0.42



courtesy of Jordi Marcos (Alba)

Overview

- ❑ adjust support HP laboratory staffing and add suitable infrastructure
- ❑ HP multimodal approach and develop data analysis methodologies
- ❑ providing microXRD based on undulator

- ▪ Enhancing characterization capabilities
  - ▪ Develop Cassegrain microscope (IR)
  - ❑ Gas loading system

- • Integrate time resolution experiments by providing suitable data collection and sample environment

- ❑ essential
- ▪ desirable ++
- • desirable +

Thank you for your attention !!!

Any Questions 

MATEX BL Concept

Name beamline: **MatterATEXtremeconditions Beamline (MATEX)**

Beamline technique(s): High energy Diffraction in DAC (1 phase)

- Focus and optimize for high performance BL: **High energy Diffraction** (XRD in DAC main)
- beamsize: 3x3 μm^2
- BL outline: undulator beamline (U20) + DCM + KB mirrors set + sample environment platform DAC (laser heating) + 2D detector
- Sample environment: LT (He cryostat) + HT: resistive heating (2000K) + laser heating (6000K) + gas loading machine
- Partnerships: MALTA consortium ready to co-fund technologies at MATEX BL (laser heating platform: laser and man power for a given amount of time & in-situ Raman).
- PhD stays at ALBA from different national groups from MALTA

Energy range (soft, tender hard) : Hard (20 – 40 keV)

Beamline type (short ID, BM, long ID): Undulator U20 with a period of 19,9 mm and 2,3 m length (calculations with gap 5.2 mm by J.Campmany).

MATEX BL Concept

International competition (at which other sources there are similar instruments):

MATEC BL will be competitive and will rival other leading extreme conditions BL at ESRF, APS, DESY, Diamond

Criteria	ID27 ESRF 6 GeV	13 IDD APS 7 GeV	ID15B (ESRF)	P.02.2 Desy 6 GeV	I15 3 GeV Diamond	Psyché Soleil 2.75GeV	MATEC ALBA
E (KeV)	20-80	10-42	30	20-80	20-80	15-100	20-40
Typical E	33	37	30	30	30	30	30
beam (µm)	2x3	2x2	5-30	20x20	20x20	10x10	3x3
source	2 U23	2 U	U20	Long U23	SWig 3.5T	Wig 2.1T	U20

Potential or existing user community (communities or also institutes):

- wide and stable user community (45 groups | 67% Spain, 16% EU, 17% non EU)
- Institut for Geowissenschaften Potsdam, Bayreuth Geoinstitut, Institute of Geology and Mineralogy Koln, Institut fuer Anorganische Chemie Wurzburg, Laboratoire Magmas et Volcans Clermont, IMPMC Paris, Univ. Lyon 1, ICG Montpellier, Univ de Lille, Department of Earth Science Firenze & Milano, Dept. GeoSciences University of Padova, Dept. of Earth Sciences ETH Zurich, Univ. Bristol (UK), Aristotle Univ. Thessaloniki, Univ. Uppsala, Univ. Prague.

ALBA HP workshop: October 28th 2021