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## **ALBA Synchrotron: Overview and opportunities**

ENURS2023

21<sup>st</sup> of June 2023

# ALBA synchrotron in a nutshell



1<sup>st</sup>

SCIENCE FACILITY  
IN SOUTH-WEST EUROPE

240

STAFF  
(~30% INTERNATIONAL)

2400

RESEARCHERS PER YEAR

450

EXPERIMENTS PER  
YEAR

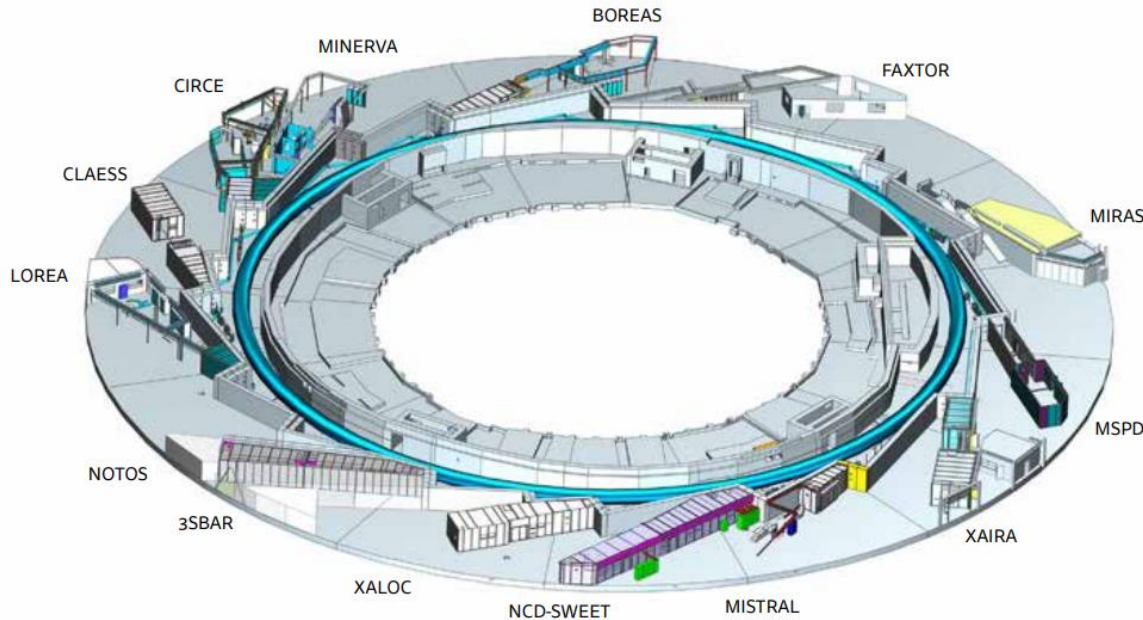
300

Publications per  
Year

National public institution funded by Ministerio de Ciencia e Innovación (50%) and GenCat Department de Recerca i Universitats (50%)

National and international (28%) staff and National and international (40%) users

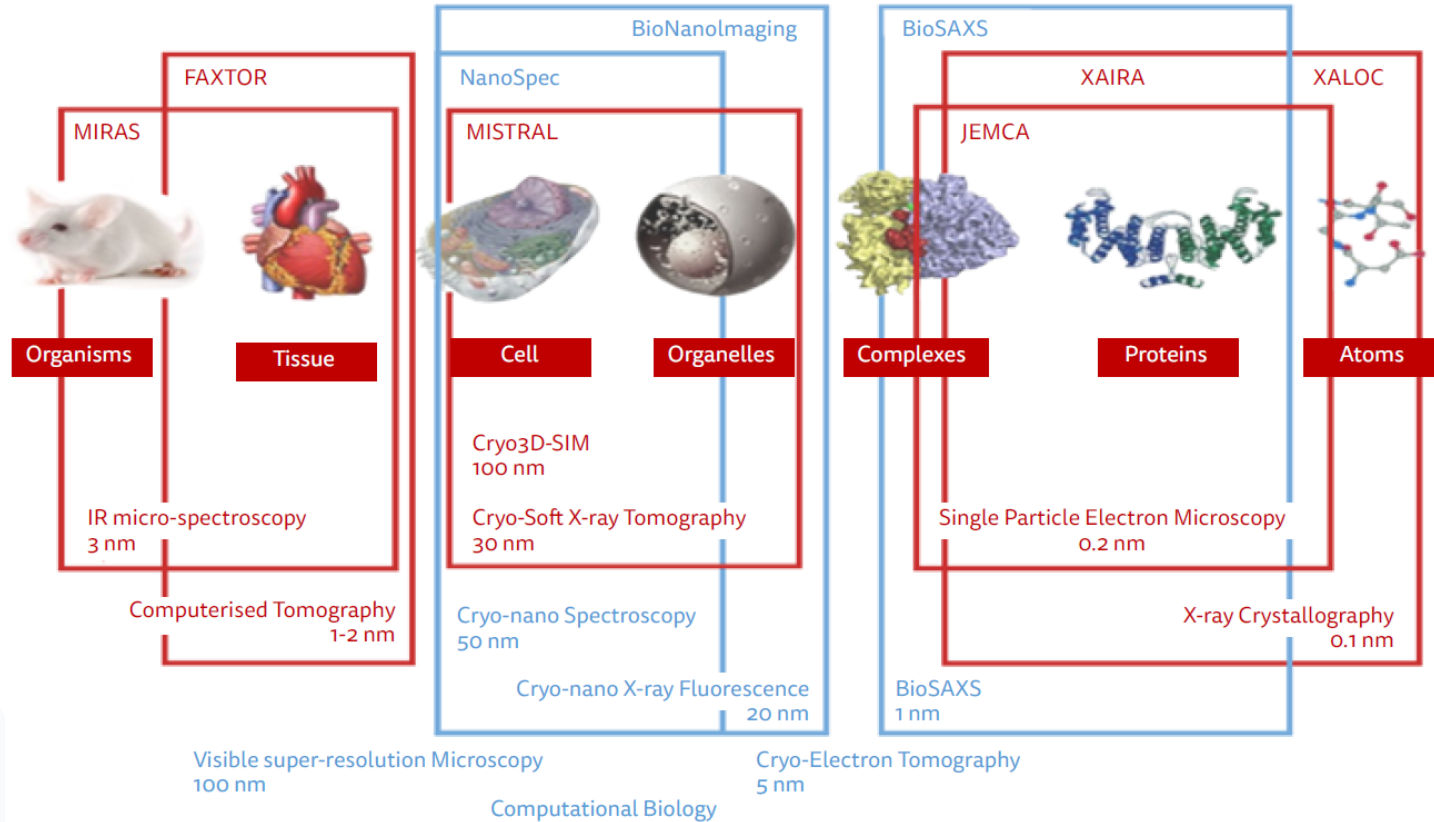
National and international collaborations



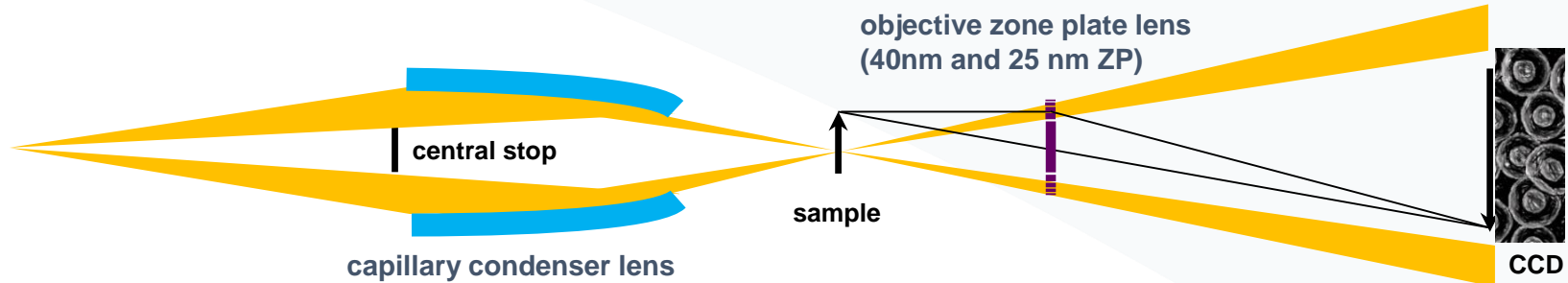
- ✓ 10 BLs in OPERATION
- ✓ 2 BLs in COMMISSIONING
- ✓ 2 BLs in CONSTRUCTION
- ✓ + advanced EM Center

**CHARACTERISTICS:**  
*3 GeV e- synchrotron*  
*270 m circumference*  
*250 mA operating current*  
*> 98% availability*

Beamline	Scientific Section	Main Technique
<b>BL01 - MIRAS</b>	Life Science	<i>Infrared Spectroscopy &amp; Microscopy – in operation</i>
<b>BL06 - XAIRA</b>	Life Science	<i>Microfocus for Macromolecular Crystallography -in commissioning; in operation in 2023</i>
<b>BL09 - MISTRAL</b>	Life Science	<i>Soft X-ray Microscopy - in operation</i>
<b>BL13 - XALOC</b>	Life Science	<i>Macromolecular Crystallography - in operation</i>
<b>BL31 - FAXTOR</b>	Life Science	<i>Fast X-ray Tomography and Radioscopy Beamline - being installed; starting operation in 2024</i>
<b>BL20 - LOREA</b>	Electronic & Magnetic Structure of Matter	<i>Angle Resolved Photoemission Spectroscopy – in operation</i>
<b>BL24 - CIRCE</b>	Electronic & Magnetic Structure of Matter	<i>Photoemission Spectroscopy and Near Ambient Pressure Photoemission – in operation</i>
<b>BL29 - BOREAS</b>	Electronic & Magnetic Structure of Matter	<i>Resonant Absorption and Scattering – in operation</i>
<b>BL04 - MSPD</b>	Chemistry & Material Science	<i>Materials Science and Powder Diffraction – in operation</i>
<b>BL11 - NCD-SWEET</b>	Chemistry & Material Science	<i>Non-Crystalline Diffraction Beamline – SAXS-WAXS Experimental Techniques – in operation</i>
<b>BL15 - 3SBAR</b>	Chemistry & Material Science	<i>Surface Spectroscopy and Structure at 1 bar – in construction; starting operation in 2026</i>
<b>BL16 - NOTOS</b>	Chemistry & Material Science	<i>Absorption, Diffraction, Instrumentation innovation and development – in operation</i>
<b>BL22 - CLÆSS</b>	Chemistry & Material Science	<i>Core Level Absorption &amp; Emission Spectroscopies – in operation</i>
<b>BL25 - MINERVA</b>	Instrumentation & optics	<i>Metrology and instrumentation – in operation in 2023</i>



# BL09-MISTRAL: Transmission X-ray Microscope



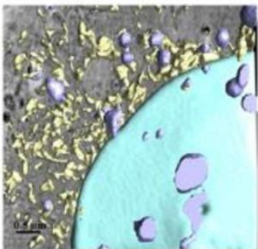
## TXM (Transmission X-ray Microscope) capability :

- 2D Measured Spatial Resolution  $\approx 30$  nm half pitch
- Detector effective Pixel size 16 nm – 8 nm, Field of view  $8 \times 8 \mu\text{m}^2$  -  $16 \times 16 \mu\text{m}^2$
- Cryogenic sample environment ( $\approx 110$  K)
- On-line visible light epifluorescence microscope for correlative low resolution 2D imaging

## Available techniques for bio-samples (with 30 nm spatial resolution):

- **Cryo-Soft X-ray Tomography** ( $\pm 70^\circ$  max angular range, typical acquisition time  $\approx 5$  -10 min)
- **Cryo-X-ray Spectromicroscopy** (from calcium L-edge)

# BL09-MISTRAL: Cell Biology applications



**Cancer research**  
 Chemotherapy  
 hyperthermia in tumor cells  
 Mitochondrial dysfunction  
 Chromatin expression

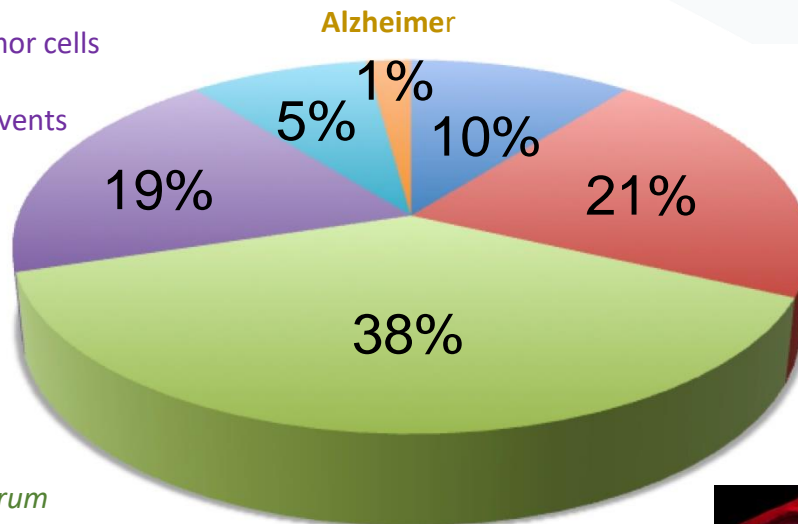
**Immune systems**  
 Cholesterol  
 T-Cell Migration  
 T-Cell interaction with pathogens  
 Primary T-cells

**Nanoparticle:**

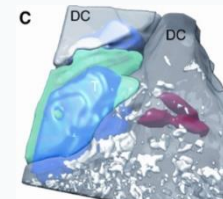
Hyperthermia in tumor cells  
 Gold coated siRNA  
 Anti-inflammatory events  
 Drug delivery  
 Nano liposomes

**Pathogens**

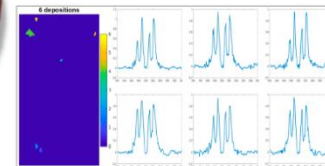
*HCV*  
*Zika*  
*Mycoplasma*  
*SARS-CoV-2*  
*Plasmodium Falciparum*  
*Babesia divergens*  
*Herpes*  
*Torovirus*  
*Vaccinia*



*Rotavirus*  
*Trypanosome Brucei*  
*Influenza*  
*E. Coli*  
*Cytomegalovirus*

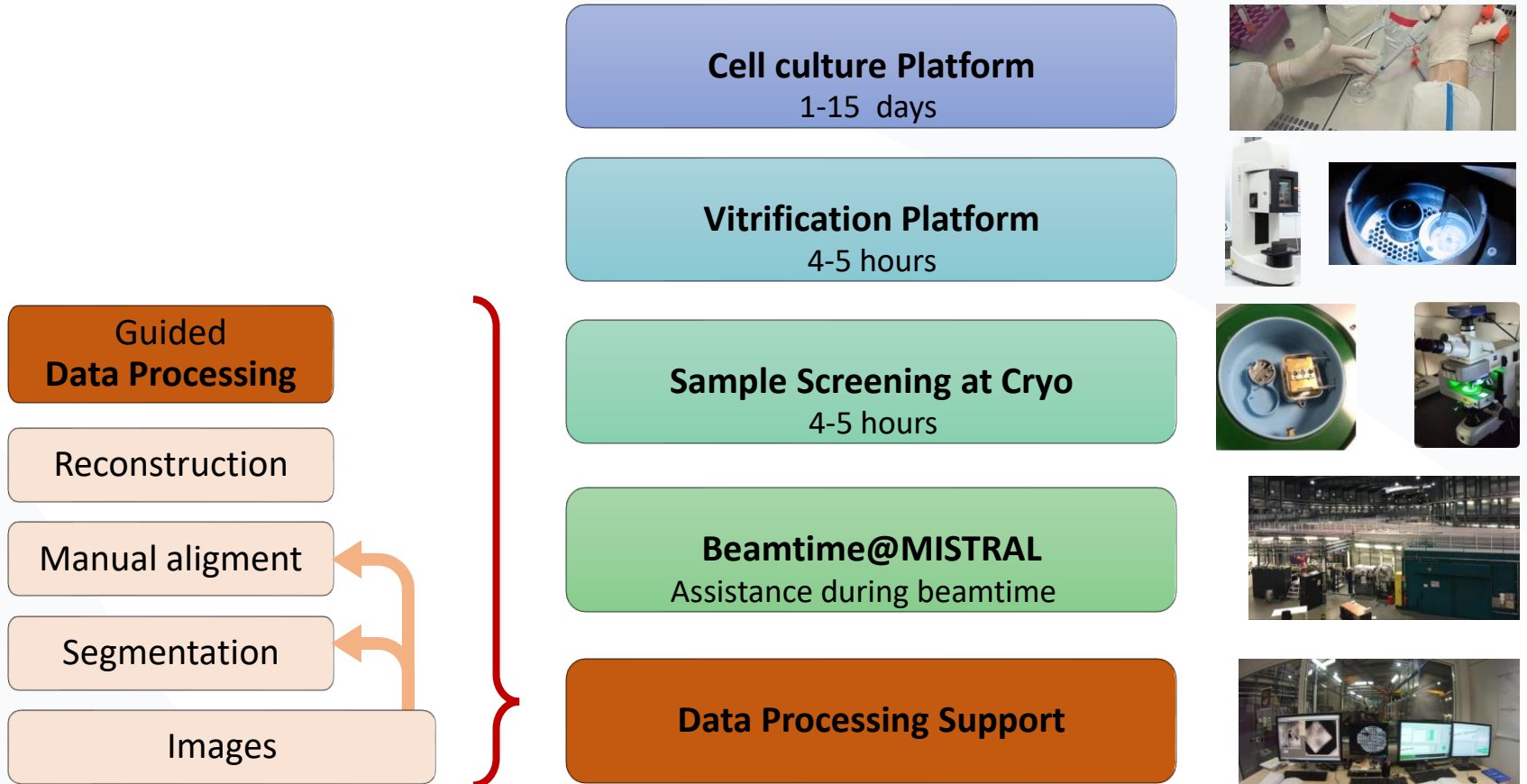


**Bio mineralization**  
 Cholesterol  
 Calcium  
 Hydroxyapatite



**Gene Therapy**

# BL09-MISTRAL: experiment pipeline





# BL09-MISTRAL: correlative SXT and 3D-XRF



**Correlative microscopy** is not only key to identify the individual organelles and their activities but also allows is the enabler technology to **multi-length scale imaging**

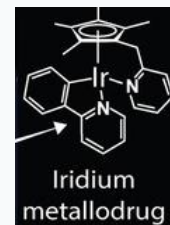
**Study case:** Functionality of a new iridium-based cancer drug (*breast cancer MCF7 cells*) with promising relatively low side effects

## Tools:

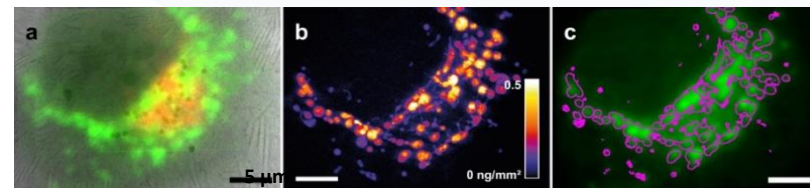
- Soft X-ray full field microscopy:
  - cell morphology.
- Epifluorescence signal
  - identification of mitochondria (green)
- 2D XRF (nano probe): iridium distribution

TXM image and epifluorescence from mitochondria (green)

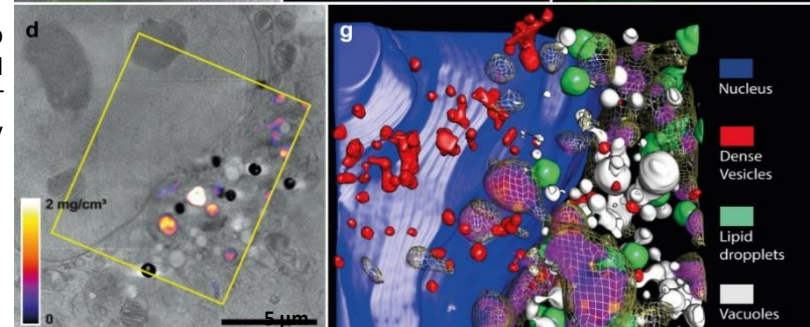
Iridium distribution from 2D XRF



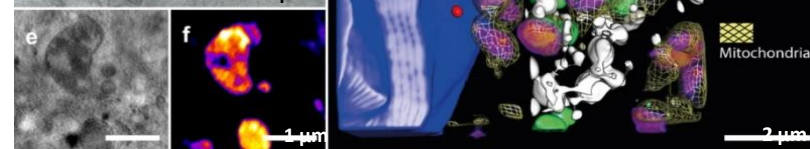
a+b



In selected area, overlap of two reconstructed slices from the cryo-SXT and the XRF tomography



Slices across the mitochondria from the cryo-SXT and the XRF

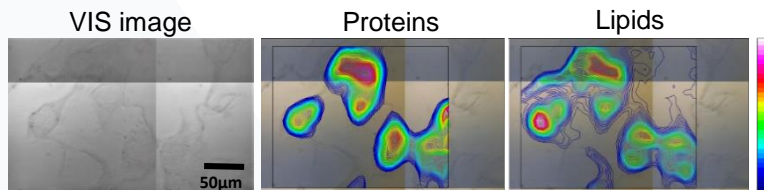


*For material sciences, biosciences, cultural heritage, food, environment.*

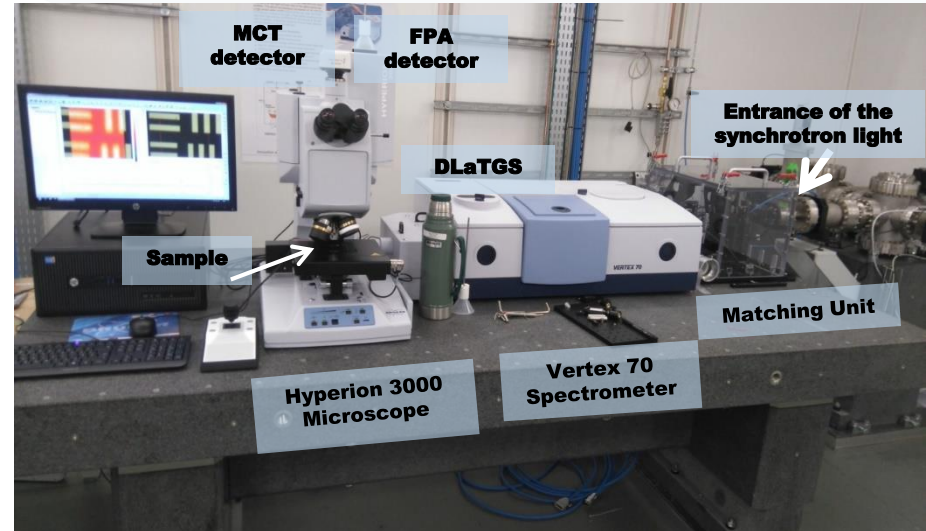
- Chemical and biochemical composition.
- Resolution up to  $3 \times 3 \mu\text{m}^2$ .
- Photon energy:  $\sim 1.2 \mu\text{m}$  to  $100 \mu\text{m}$ .

## Study case in live cells:

Metabolic compounds in brain glioblastoma cell line after Riluzole treatment



*Ducic et al, Anal. Chem. 1932, 2021*

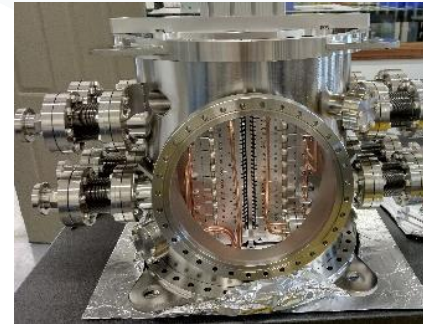


- Live cells setup allows the real time observation of the biochemical rearrangements undergone in living cells upon treatment.

**BL in construction**

## Fast Hard X-ray micro-Computed Tomography and Radioscopy beamline

- Fast micro-Computed tomography
- Monochromatic/filtered white beam
- Absorption/phase-contrast imaging
- Energy range: 8-70 keV
- 0.7 – 15  $\mu\text{m}$  pixel size
- First users by end-2024

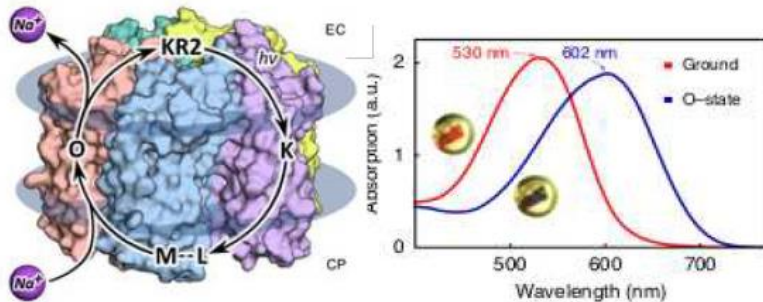


### Possible case studies:

- Small organs: rat brains, rat livers, ...
- Cartilage
- Spinal cords

Recently upgraded with a  
PILATUS3 6M (100Hz) - Dectris

A SSX structure of the intermediate O-state obtained by time-resolved laser photoactivation at BL13-XALOC<sup>5</sup>, helped elucidating the active-light driven Na<sup>+</sup> transport mechanism of *K. eikastus* rhodopsin 2



Kovalev et al., Nat Comm 11:2137 (2020)

## High Viscosity Extrusion (HVE) injector implemented at XALOC for jet-based SSX



- A 45° jet orientation
- 5-20 μm minimal size
- 12 Hz
- 50x30 μm beam

Serial MX at ALBA Synchrotron  
J. Synchrotron Rad. 29, 1130 (2022)

**SSX already available**  
request it in the next call for proposals !!!

**BL in construction**

## BL Instrumentation

### Beam features

1×1 μm beam @ 1 Å

Low Photon Energy  
4-14 keV

Low background  
He chamber, beam tailoring

μMX

Elemental  
Determination

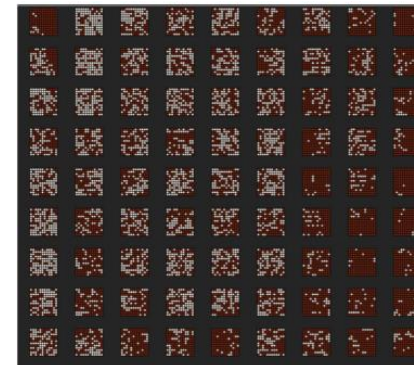
Optimal  
native  
Phasing  
of μ-crystals  
(std mount)

Fixed-target  
serial crystallography

Optimal fixed-target SX  
Time-resolved experiments

*Serial native-phasing crystallography*

- **Fast pixel array detector:** Eiger2 XE 9M (frame rates up to 1000Hz)
- **Automated sample mounting system ISARA2** (29 cryo-pucks+3 RT pucks)
- **First users by end-2023**



Chip for fixed-target SSX

Owen *et al.* (2017)  
*Acta Cryst. D73:373*

## **Tuning Electronic and Chemical Properties by Controlling Atomic Structure of Functional Materials**

### **Scientific topics:**

- Energy storage
- Hydrogen circular economy
- Carbon sequestration

### **Techniques**

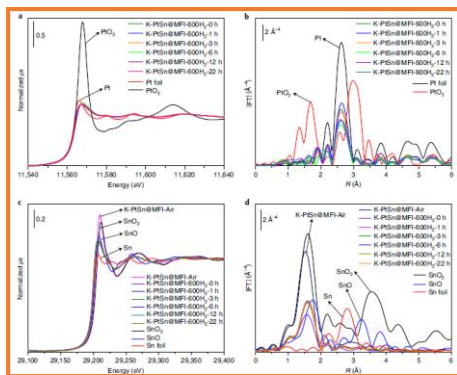
- High throughput ensemble averaging probes including IR, XAFS, and diffraction.
- X-ray microscopy in scanning and full field mode with absorption and phase contrast
- TEM
- Multi-modal approach combination of ex-situ, in-situ, and operando experiments.

### **Services**

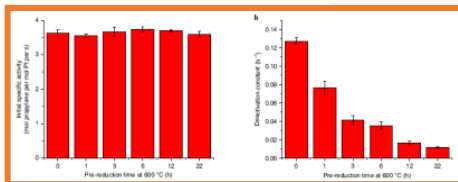
- Sample preparation tools allowing remote combinatorial approaches.
- multi-modal pipelines and access modes for providing solutions for main focus area applications.
- Big data gateway for data mining community.
- data analytics tools to identify structure-function correlations in multi-modal experiments.

## XAS

Local structure around selected element  
Chemical/electronic state



## Synthesis of bimetallic particles for optimized catalytic reactions (H<sub>2</sub>, O<sub>2</sub>) conversion



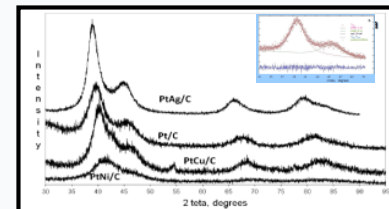
## Laboratory

Synthesis, calcination, pre treatment (oxidation/reduction)  
Sample environment

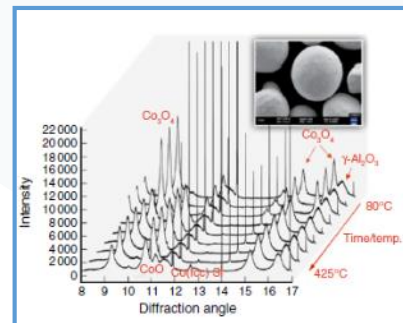
## Diffraction

Long/short range structure

Nano size



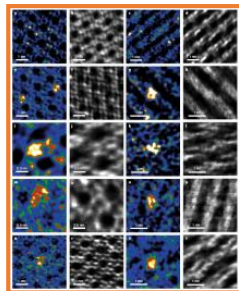
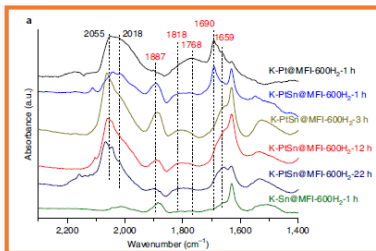
InSitu XRD under 5% H<sub>2</sub> flow



XRD-CT PDF-CT

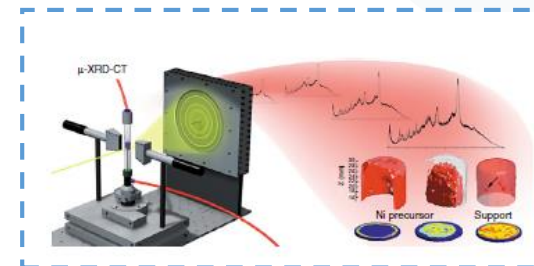
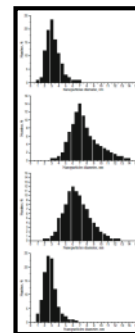
## Infra Red (CO Probe)

Interaction with molecule



## TEM / STEM

Localisation of atoms arrangement  
Nanoparticle size distribution

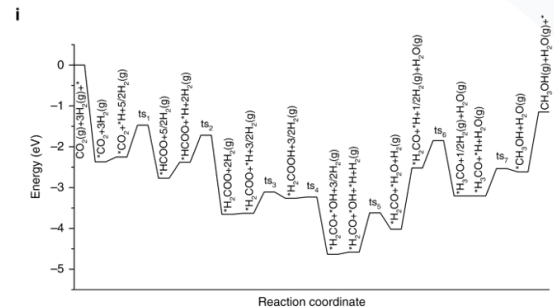
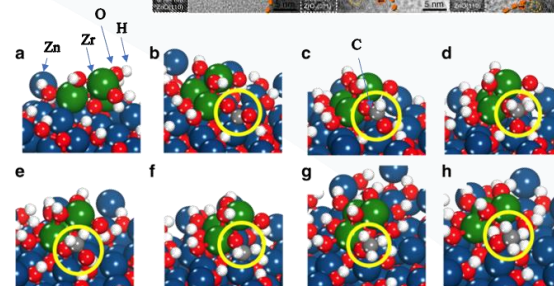
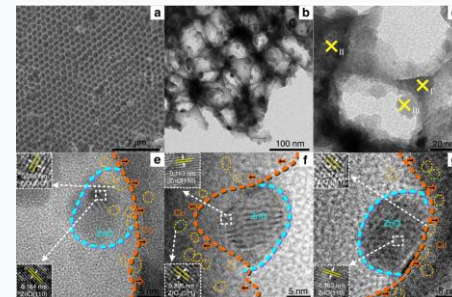


Multimodal approach under operando conditions combining imaging on the different length scales with ensemble averaging spectroscopic tools is key to “understand” how catalysts work.

## Study case:

Macroporous Cu-ZnO-ZrO<sub>2</sub> (CZZ) catalysts for methanol production from CO<sub>2</sub>:

- Simple cartoon to explain the steps of the reaction pathway.
- TEM showing the morphology and different interfaces of the catalyst.
- Reaction pathway and corresponding structural models showing the complex changes during the reaction based on DFT calculations.

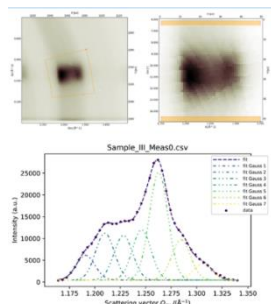
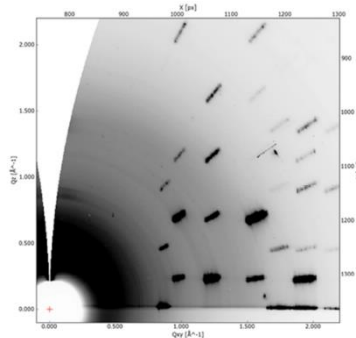
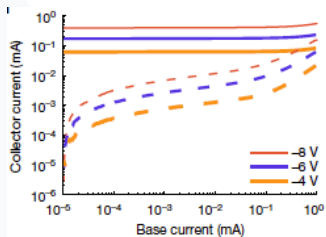
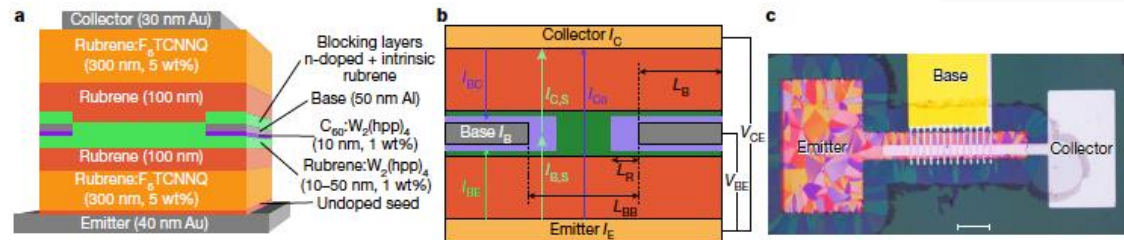


<https://doi.org/10.1016/j.chempr.2019.10.023>

<https://doi.org/10.1038/s41467-019-09072-6>

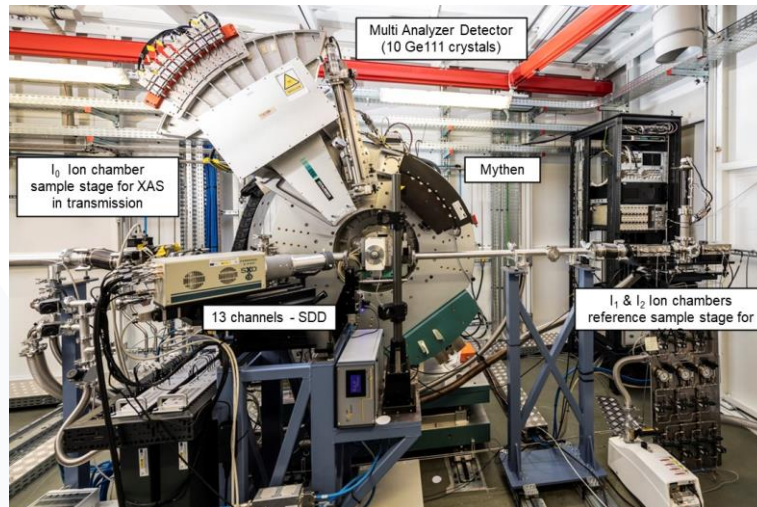


## BL11-NCD-SWEET: Novel High performance Organic Thin Film Bi-Polar Transistor



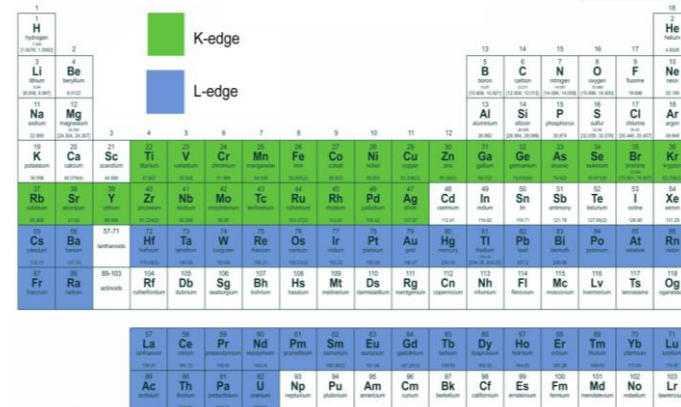
- Development of organic thin film transistor technology based on rubrene shows high gain (larger than 100).
- Performance depends on crystal structure of the different organic thin layers which are strongly impacted by the doping concentration
- GI-WAXS was used to determine crystal structure, orientation, and broadening ultimately guiding the doping concentration.

## HRPD&XAS endstation fully operative



### Main features:

- Energy range: 4.7 – 27 keV (XAS) / 30 keV (XRD)
- Minimum photon flux:  $10^{11}$ (ph/s)
- Spot properties on sample at HRPD & XAS station: min 400x400  $\mu\text{m}^2$
- Reactive gas system available at ambient pressure
- Capillary setup for operando experiments



Legend:

- Green square: K-edge
- Blue square: L-edge

1																	18																				
1	H																	2																			
	He																																				
3	Li	4	Be													10	Ne																				
	B	6	C	7	N	8	O	9	F	11	Na	12	Mg	13	Al	14	Si	15	P	16	S	17	Cl	18	Ar												
19	K	20	Ca	21	Sc	22	Ti	23	V	24	Cr	25	Mn	26	Fe	27	Co	28	Ni	29	Cu	30	Zn	31	Ga	32	Ge	33	As	34	Se	35	Br	36	Kr		
37	Rb	38	Sr	39	Y	40	Zr	41	Nb	42	Mo	43	Tc	44	Ru	45	Rh	46	Pd	47	Ag	48	Cd	49	In	50	Sn	51	Sb	52	Te	53	I	54	Xe		
55	Cs	56	Ba	57-71	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn																		
87	Fr	88	Ra	89-103	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Cn	Nh	Fl	Mc	Lv	Ts	Og																		
89	La	90	Ce	91	Pr	92	Nd	93	Pm	94	Sm	95	Eu	96	Gd	97	Tb	98	Dy	99	Ho	100	Er	101	Tm	102	Yb	103	Lu								
105	Ac	106	Th	107	Pa	108	U	109	Np	110	Pu	111	Am	112	Cm	113	Bk	114	Cf	115	Es	116	Fm	117	Md	118	No	119	Lr								

## **Resolving and Manipulating the Electronic and Magnetic States, and its Spatial and Dynamic Characteristics in Quantum Materials.**

### **Scientific topics:**

- Spintronics and spinorbitronics.
- Topological materials.
- Materials discover.
- Device physics.

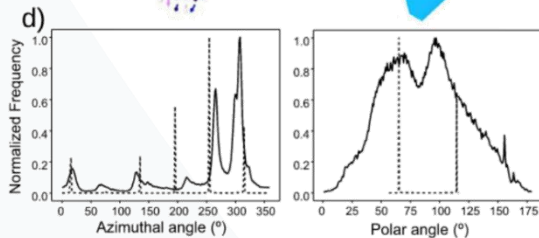
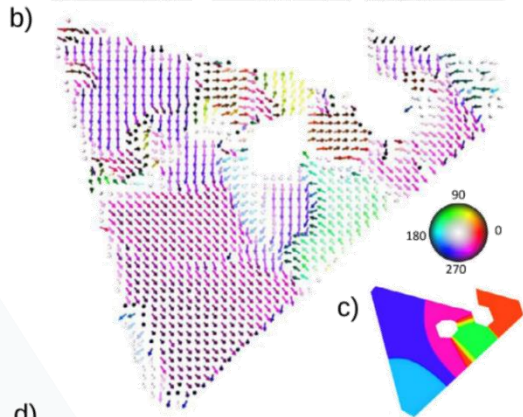
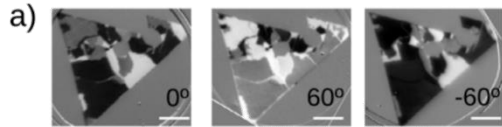
### **Techniques:**

- Materials growth and device fabrication.
- XAS, XMCD, XMLD, XLD, XRMS.
- ARPES (including Spin-ARPES).
- X-ray microscopy and coherent imaging with chemical, magnetic, spin/orbital- momentum contrast.
- TEM.
- Structural and functional characterization tools.

### **Services**

- Sample preparation tools allowing remote combinatorial approaches.
- Multi-modal pipelines and access modes for providing solutions for main focus area applications.
- Computational modeling tools for understanding impact of structure and environment on topological entanglement.

## Magnetic Domain Wall Pinning in Cobalt Ferrite Microstructures for Spintronics Applications

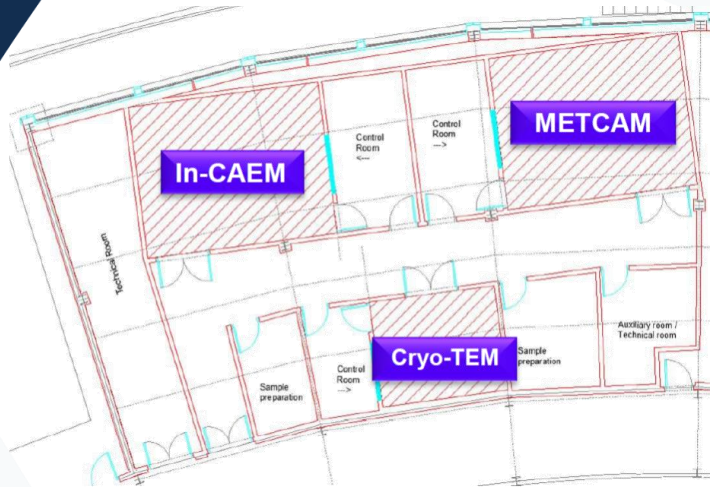


Correlative structural, magnetic and chemical analysis of (non-stoichiometric) cobalt ferrite micro-metric crystals by X-ray Magnetic Circular Dichroism (XMCD) combined with photoemission microscopy (PEEM), low-energy electron microscopy and atomic force microscopy

- The **magnetization vector** inside the islands was resolved at the nanoscale, as obtained from magnetic images taken at different x-ray incidence angles.
- The **location of the magnetic domain walls** was correlated with the presence of different types of defects as revealed by the different microscopies.
- **Micromagnetic simulations**, suggests that the main source of pinning in these microcrystals are linear structural defects induced in the spinel structure by the substrate steps underneath the islands.
- The defects responsible for the pinning of the domain walls are the substrate steps.

It should be thus possible to obtain structurally perfect ferrimagnetic crystals with domain sizes limited only by the substrate terrace size

# Joint Electron Microscopy Center at ALBA



**EM01-Cryo-TEM** ThermoFisher Scientific, Glacios 200 kV transmission electron microscope equipped with a Falcon 4 direct electron detector. Owned by IBMB<sup>11</sup> and shared among different partners, including ALBA. In operation since fall 2022

**EM02-METCAM** ThermoFisher Scientific Spectra 300kV monochromatic transmission (scanning) electron microscope with double aberration correction. Starting operation. Owned by ICN2<sup>12</sup> and shared among different partners, including ALBA. First users in 2023

**TEM** In construction, foreseen operation in 2025

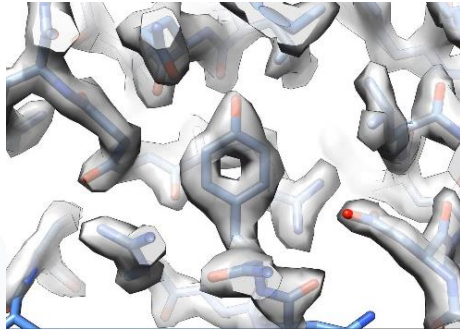
**AFM/STM** In construction, foreseen operation in 2025

## Materials Science

### 300 kV STEM Thermo Fisher Spectra

- double aberration corrected (probe and image)
- HR EELS detector and ULTRA X EDX detector





*Apoferritin map from cryoEM data collected at ALBA*

## Glacios cryo-EM 200kV X-FEG

- Autoloader robot to sample exchange
- Falcon 4-direct electron detector: 240 fps



- Partners have different beamtime shares
- ALBA share is of 15% and is offered to users on the same basis as synchrotron beamtime
- ... be attentive to calls opening at User Office Portal



## ***Sample preparation lab***

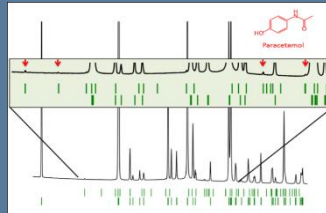
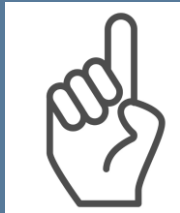
- Vitrobot Mark IV vitrification system
- GLoQube to grid treatment previous to vitrification
- Liquid N2 dry dewars to grid storage



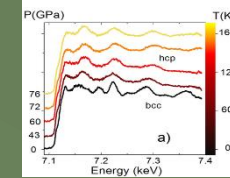
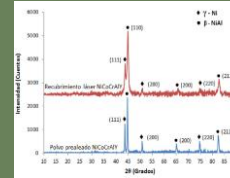
## ***Control room***

- PC to control the microscope
- EPU 2 software for sample screening and data acquisition
- RSync PC for data downloading, sftp
- GPU Workstation to carry out on-the-fly data processing

## Single service



## Long collaboration



Several techniques are required

## Joint service with partners



Challenge

Techniques  
& skills

Advanced  
techniques

## Funded EU projects



a hub for  
materials research



# Funding for Industry via European projects



- SPECIFIC PROGRAMME FOR SMEs (TamaTA-INNOV)
- FREE ACCESS to ALBA
- CALL OPEN: <https://wayforlight.eu/en/industries/>
- ALBA ILO ([industrialoffice@cells.es](mailto:industrialoffice@cells.es)) will help



<https://wayforlight.eu/en/industries/>



## INDUSTRIES

Your company may gather invaluable information about your product at the micro-scale by using advanced experiments at European infrastructures. Talk to us about your challenge and benefit from the current LEAPS-INNOV project support.

APPLY HERE ↓



What can Light Sources do for you?

English

### INDUSTRIAL LIAISON OFFICES

#### Contacts

SMEs are encouraged to contact directly to any of the following light sources to better prepare the industrial proposals

#### CELLS (ALBA SYNCHROTRON)

Barcelona, SPAIN

<http://www.albasynchrotron.es/en/industry/services>

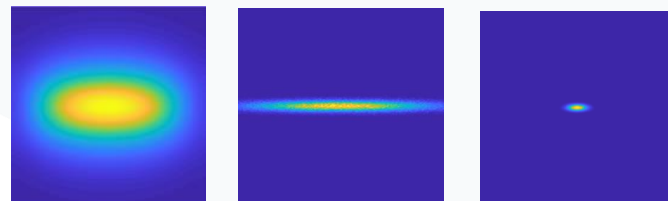
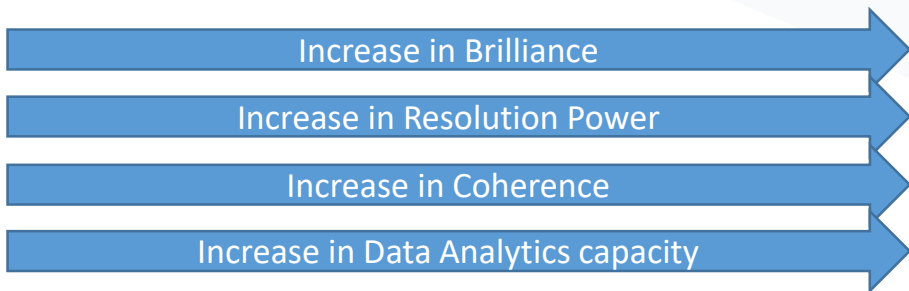
[industrialoffice@cells.es](mailto:industrialoffice@cells.es)

#### DESY

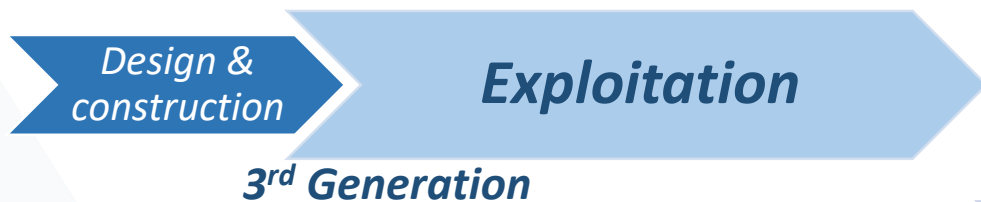
Hamburg, GERMANY

<https://innovation.desy.de>

# Evolution of ALBA to the 4<sup>th</sup> generation



*Representation of photon beam on sample*

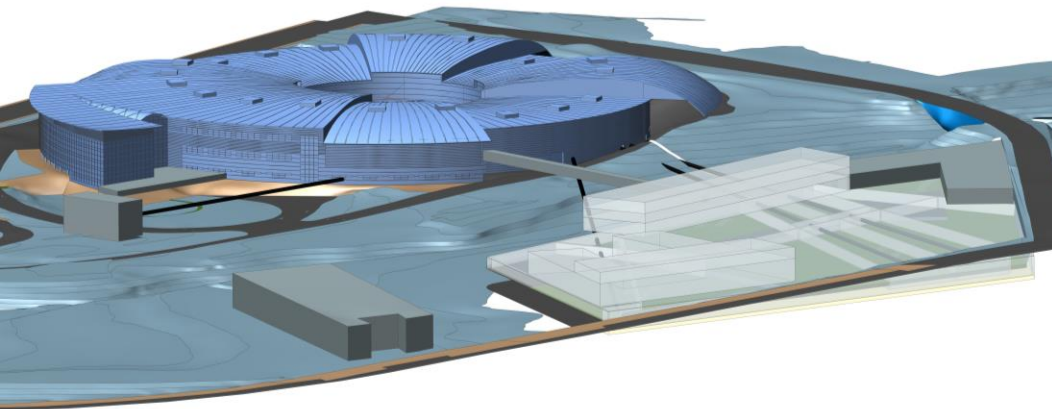


2003      2012      2022      2031      2050

# ALBA II design as of today

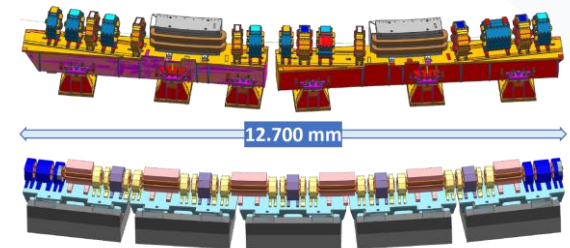


- Emittance 185 pm (25 times smaller than ALBA)
- Current 300 mA (+ 20% with respect to ALBA)
- Renovation of the accelerator structure
- Construction of long experimental lines
- Renewal of the current beamlines
- Development of the capabilities for data management
- Adding staging and laboratory capabilities (with partners from the community)
- Adjusting access policies and review criteria



## *Accelerator design in progress*

Cell Architecture



# Evolution of ALBA to the 4<sup>th</sup> generation



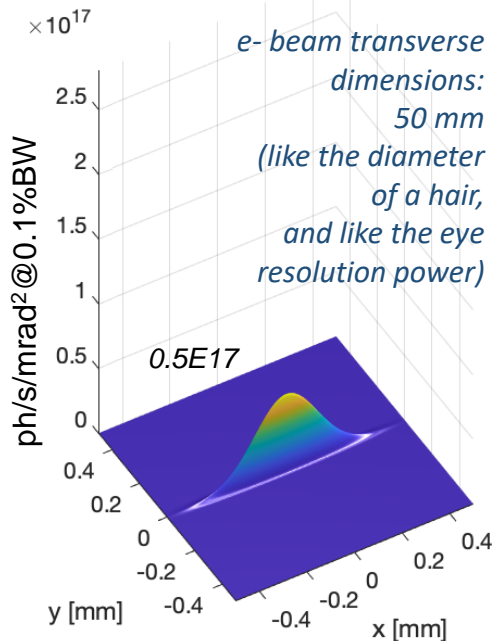
## Flux density on sample hard x-ray undulator

For hard x-Ray BLs the emission cone is dominated by the electron beam size.

Although the integrated flux does not change, it is concentrated in a narrower cone.

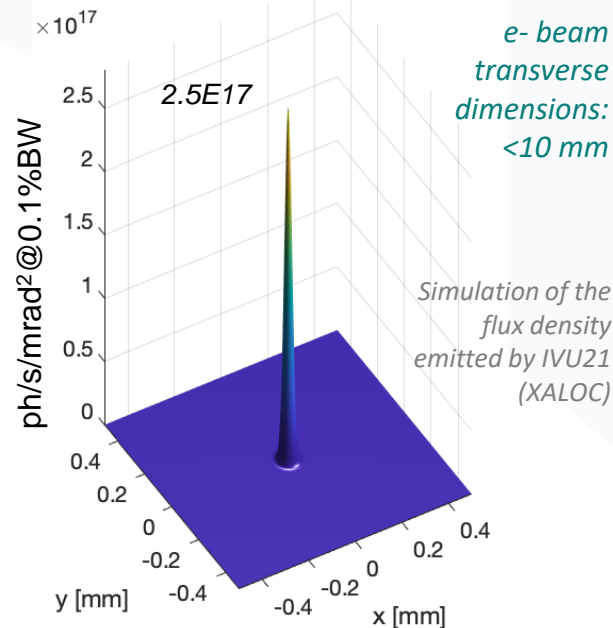
### ALBA

Flux Distr. E=12654 eV

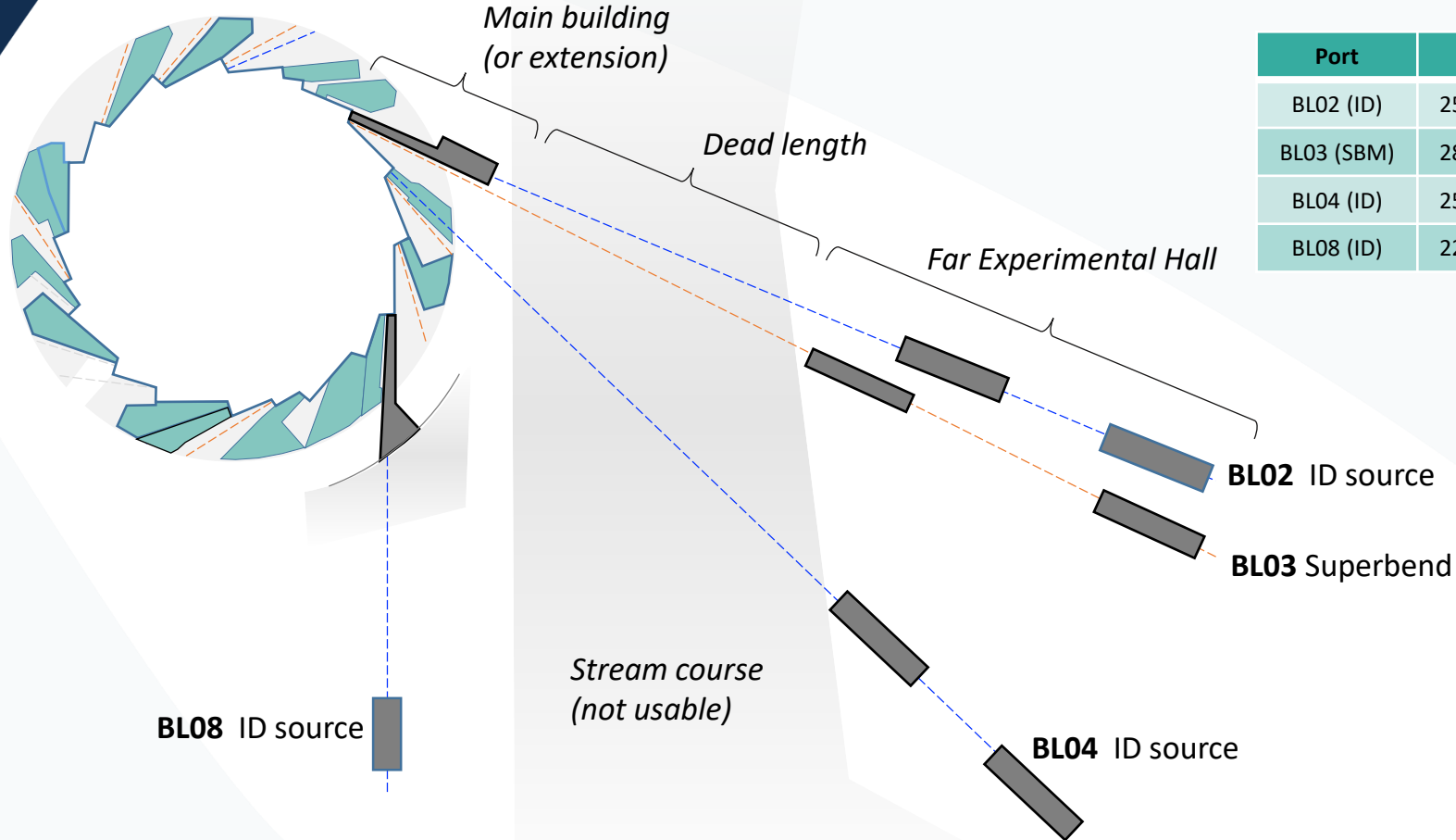


### ALBA II

Flux Distr. E=12654 eV



# Long Beamlines using the new plots



Port	EH	FEH
BL02 (ID)	25-42 m	134-249 m
BL03 (SBM)	28-41 m	141-255 m
BL04 (ID)	25-42 m	173-292 m
BL08 (ID)	22-42 m	85-125 m

# The Science enabled by ALBA II



ALBA II combines the excellence and availability of the user program of ALBA with the development of full characterization suites for characterizing multi-lengthscale problems

- Enhanced **microscopy** capabilities
- **Multi-modal methodologies** to address complex development tasks
- **High-throughput capabilities** and big-data connectivity for fast innovation
- **Optimized operando environments** to optimize functional materials and devices

[bcalisto@cells.es](mailto:bcalisto@cells.es)

**THANK YOU!**

