ALBA Synchrotron - FCT joint meeting Chemistry and Material Science Section

By F. FAUTH (Section Head)

17.12.2020

Instruments:

Since 2011

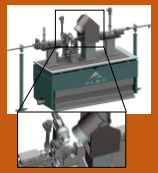
MSPD

Powder diffraction (PD) and micro-PD -BL

CLÆSS

Absorption & emission spectroscopy BL

NOTOS



PD & absorption-BL

The Section:

In 2021

Staff :

MSPD: 3 scientists + 1 PostDoc	CLÆSS: 2 scientists + 2PostDocs	NOTOS: 3 scientists + 1 PostDoc
F. Fauth (LBS) + section head *battery, instrumentation, strongly correlated* C. Popescu (BS) High Pressure A. Missiul (BS) *crystallography, porous material* Open PD R. Houdeville (ST DOCFAM -> 6/2021)	*L. Simonelli. (LBS)* battery, environmental science, *strongly correlated* C. Marini (BS) catalysis, *strongly correlated* V. Martin-Diaconescu (PD) J. Gorni (PD) N Manivannan (ST DOCFAM -> 10/2021)	*G. Agostini (LBS)* *catalysis* C. Escudero (BS) catalysis O. Vallcorba (BS) crystallography, MicroDiffraction PD to come

+ Controls, Mechnical, Electronics engineer + Technician

Lab infrastructure:

Chemistry Lab
(service lab to all ALBA not exclusive to the section, Glove Box, Furnace, Fume Hood)
High Pressure Lab
(HP–MSPD users exclusively so far, MIRAS used it : Lgasket drilling, pressure calibration)

Scientific Productivity:

Peer Reviewed Publications:

() InHouse beamtime

https://www.albasynchrotron.es/en/science-at-alba/albapub

Year	MSPD	CLÆSS
2020	**55 (6)**	**41(2)**
2019	60 (6)	28 (7)
2018	66 (13)	17 (3)
2017	41 (7)	15 (3)
2016	45 (11)	17 (6)
2015	27 (3)	8
2014	17 (5)	7
2013	5 (1)	1
2012	1 (0)	0

Achieved target of
1 pub per proposal

Allocated proposals:

Year	MSPD		CLÆSS	
	#Exp	#Req	#Exp	#Req
2020	**27/24**	**50/51**	**17/18**	**53/59**
2019	25/29	42/55	17/17	40/46
2018	25/22	32/**26**	17/17	30/42
2017	22/26	**25**/39	17/16	31/42
2016	22/29	29/35	13/16	28/29
2015	23/22	37/24	13/12	32/27
2014	29	49	18	38
2013	29	37	15	28
2012	17	26	7	22

Overbooking
2 MSPD / 3 CLÆSS
before NOTOS

ALBA highlights 2020

MSPD 6 / CLAESS 2 / Both 1

https://www.albasynchrotron.es/en/science-at-alba/science-highlights

MSPD beamline :

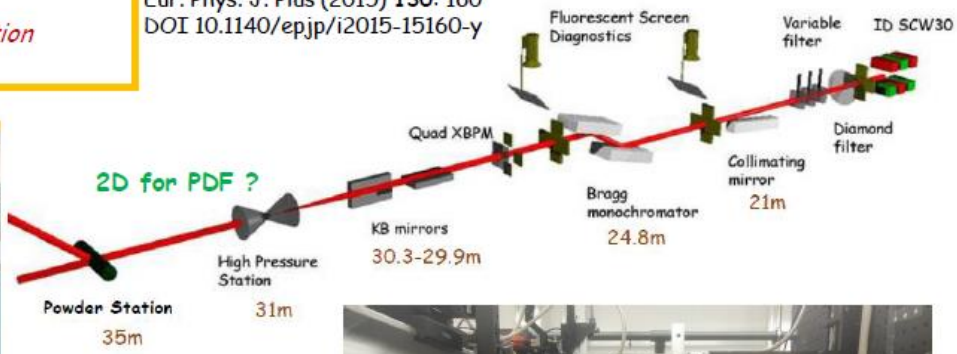
MultiAnalyzerDetector (8/10- 50 keV)

- 13 channels with 1.5 deg pitch
- Si_{111} or Si_{220} Bragg reflection
- YAP scintillator + PMT

-> 0.005° angular resolution
-> scanning speed 1°/min

Powder Diffraction Vol. 28, No.S2, EPDIC13 Proceeding,
doi:10.1017/S0885715613000900

Eur. Phys. J. Plus (2015) **130**: 160
DOI 10.1140/epjp/i2015-15160-y



Mythen detector (8-30 keV)

- Si position sensitive detector
- 6 modules (1280 channels, 50 μm pitch)
- ms time resolution
- Full pattern acquisition :

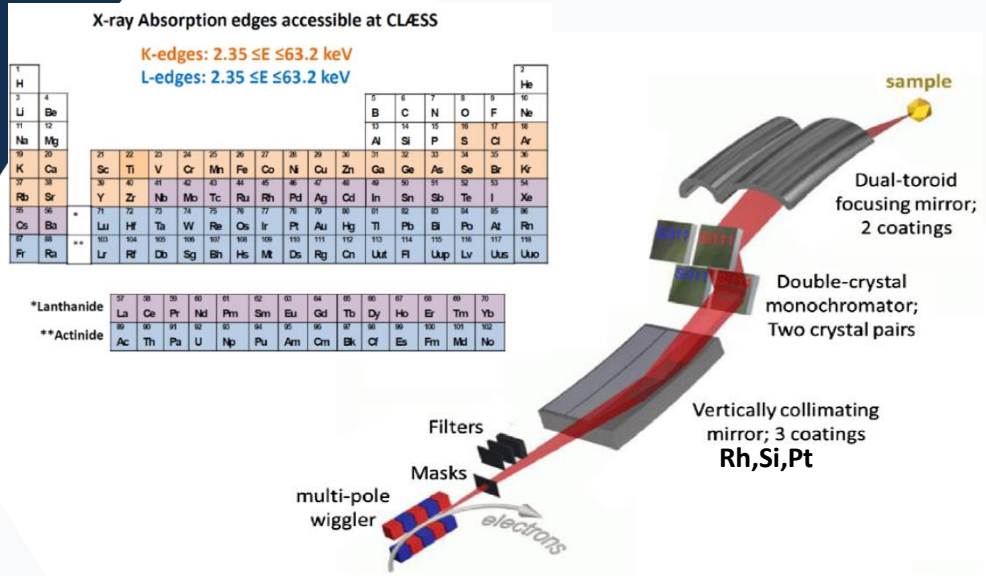
ms : 1 position / trigger mode (stroboscopic)
1-5 sec : 1 position, ~40° range
1 - 10 min :5 positions , ~60° range
-~40min : 72 positions, ~130° range (PDF)
-> ~40° in 0.005 deg pitch
-> 0.02° angular resolution

HP-MD station (20-50 keV)

- 2D Rayonix CCD detector ø162mm
- Lorentzian shape focus spot 15x15 μm
- 150-500mm sample-detector distance

-> 0.05-0.08° angular resolution

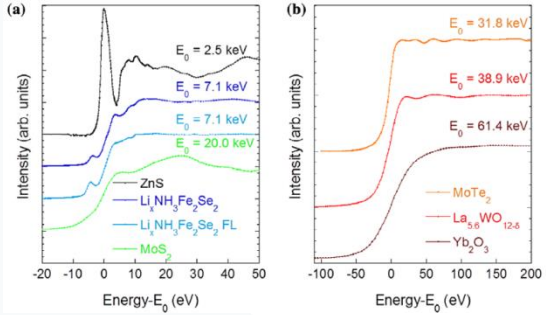
CLÆSS beamline :

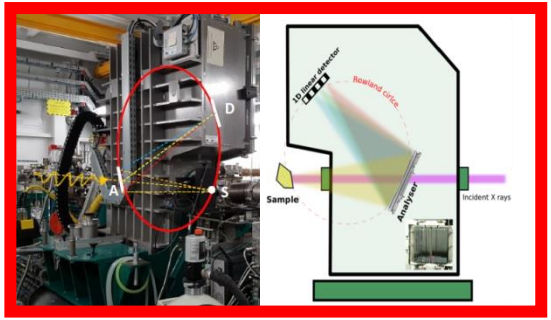


5 elements (65 mm^2)
fluorescent detector
=> 30-60min acquisition time
in fluorescence mode
(several passes of 3-10min)
=> higher dilution (0.5-1 ppm)

XAS in total electron yield mode
1-5min (0.5-2min per repeat

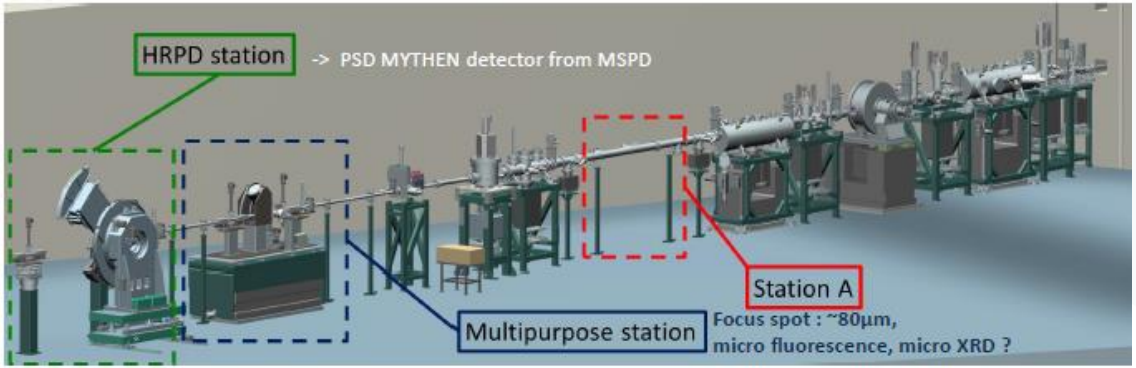
3 ionisation chambers
1-10min full EXAFS
(several passes of 0.5-2 min)





CLEAR spectrometer XES
20-30 min typical acquistion
up to 23 keV
Kα, Kβ emission lines
10min (Kα), 30-40min (Kβ1,3), 1-4h

NOTOS beamline :

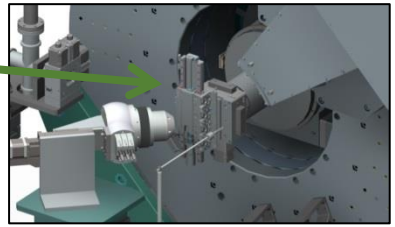


-> strengthen hard XAS capability : focus on catalysis (gas system)
AND electrochemistry using complementary PD and XAS techniques

-> bending BL at ALBA with **operating range 5-25(30) keV** (Ti-Kedge to Rh-K edge)
so **most TM used in actual batteries** accessible

QUASI-simultaneous In Situ PD and XAS data collection on Operando batteries or Catalysis reaction

Pending to sort out/design most appropriate cells and data collection geometry for both techniques



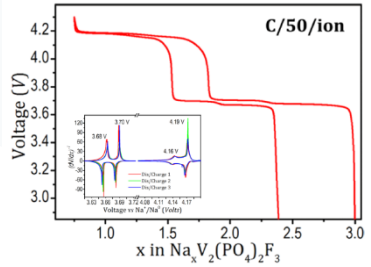
Scientific example :

Full reversible extraction/insertion of 2 Na+ ions with 3/4 characteristic Voltages

$Na_3V_2(PO_4)_2F_3$ -> $Na_1V_2(PO_4)_2F_3$

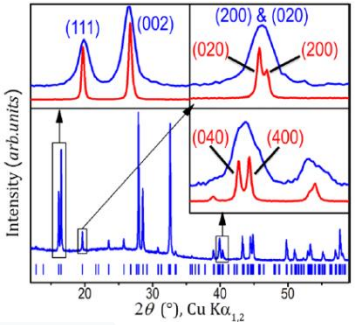
V^{3+} / V^{3+} -> V^{4+} / V^{4+} (or V^{3+} / V^{5+})

maximal theoretical capacity of 192.4 mAh/g



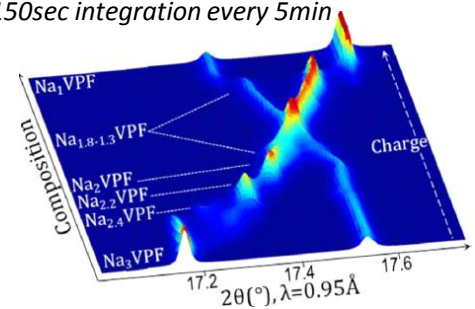
Chem. Mater. 2014, 26, 4238−4247
Chem. Mater. 2015, 27, 3009−3020
Chem. Mater. 2016, 28, 7683−7692
J. Phys. Chem. C 2017, 121, 4103−4111
Small Methods **2018**, 1800215

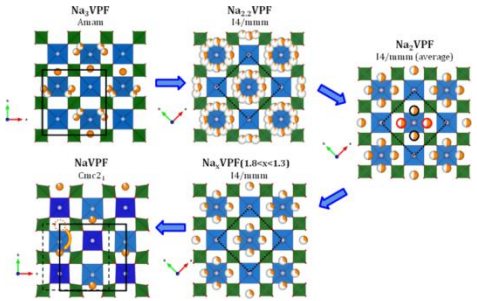
Angular resolution



Fast acquisition

150sec integration every 5min

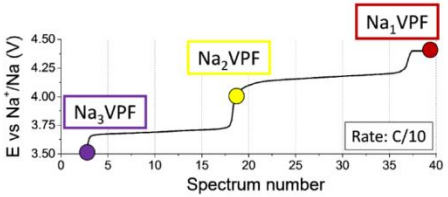


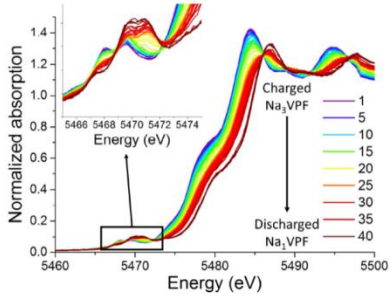


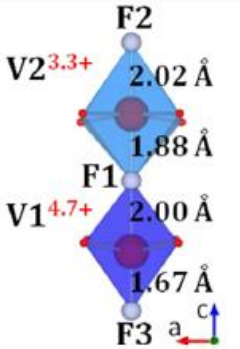
Structure refinement

- *Phase transition*
- *Na occupancy*
- *Bond distances*

Complementary XANES -> V^{3+} / V^{5+} @ Na1

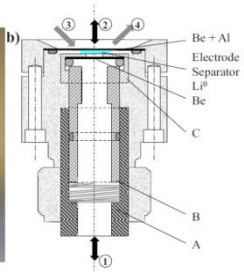


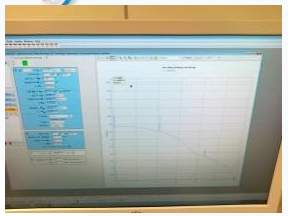




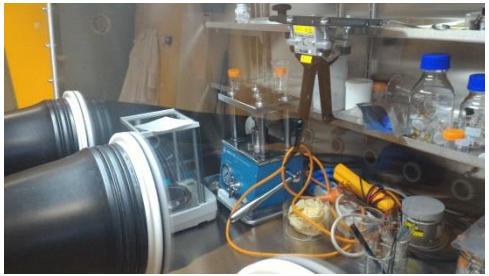
Prerequisite for In Situ Operando:

…electrochemical cells, support and potentiostats





…but very important, !!!
a properly equipped –easy accessible- Ar-filled Glove Box



Available at ALBA !

Conclusion :

Chemistry and Material Science section is well equipped for conducting collaborative projects in the strategic scientific fields

energy related materials, catalysis, environmental science

using either X-ray absorption spectroscopy, Powder Diffraction, or joined

in terms of instruments, sample environment and laboratory infarstructure

Scientific staff is always open to collaboration.
ALBA technical services (mechnical engineering, controls, electronics) is an asset for developping ad hoc sample environment, data processing,…

Now is time for exchange and propostions