Report on BL04-MSPD

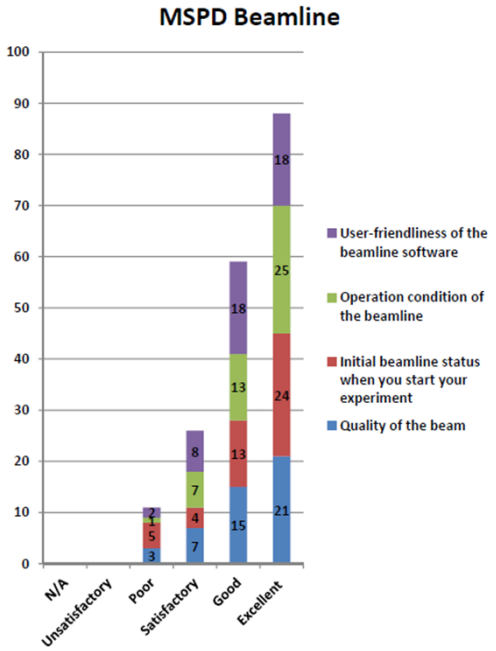
Staff	
Scientists	Francois Fauth (electrochemistry, strongly correlated electron materials, instrumentation)
	Inma Peral (catalysis, pdf measurements, microdiffraction)
PostDoc	Catalin Popescu (Feb 2012) (high pressure IH projects + collaborations, HP setup development (pressure calibration, membrane cell))
	Oriol Vallcorba (Jan 2014) (inorganic structure determination, amorphous phase quantification, 'grains' microdiffraction)
Technician	Francesc Farre (exp div -> technical developments)
Lab Technician	Robert Oliete (exp div ->chemistry lab support)
Engineer	Edmundo Fraga (exp div), Joaquin Gonzalez (Jan 2014)
Controls	Zbigniew Reszela (-> May 2014), Daniel Roldan
Electronics	Xavi Serra

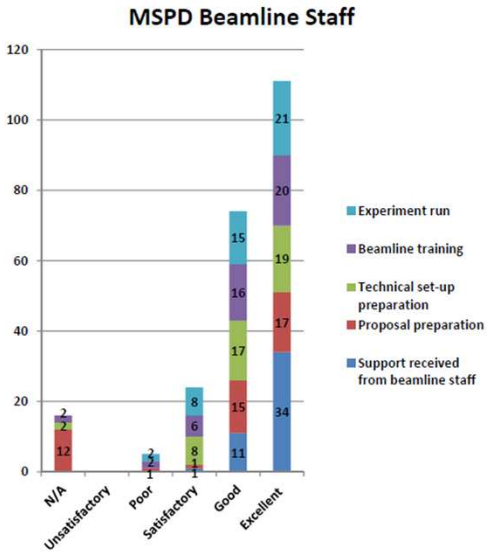
Acknowledgement to all ALBA staff and users

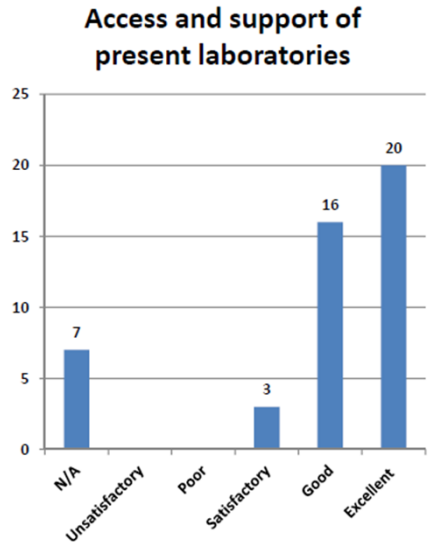
How is performing the beamline ?

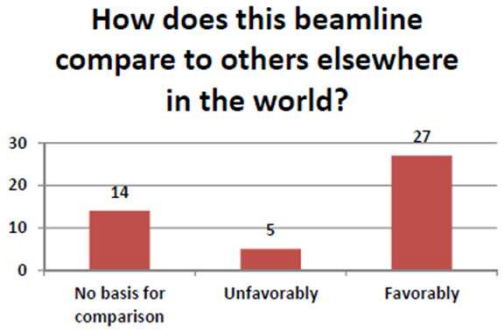
Users point of view : Hard to decipher users' feedback …

at least to my opinion !









Truth is in details

- *What was wrong ?*
- *Which experiment ?*
- *Comparison to which other beamline ?*
- *One should consider time evolution*

How is performing the beamline ?

MSPD staff point of view : The beamline is perfoming well !

- ***No problem with SC wiggler source (one power supply failure in Oct 2012).***
  Still operating with initial lHe refill (mid 2011)

- ***No technical problems with optical components***
  MONO : May 2013 one water cooling pipe changed -> 2nd Xtal vert translation encoder down
  Dec 2013 slight vacuum increase -> tighten cryocooling pipes (but a 3d experiment cancelled)
  MOCO to commission (if necessary)
  MIRROR : live with initial 'galistan' droplet spills on surface
  COOLING : OK at 120mA, thought flow below initial requirement (under engineering study)

- ***Experimental stations components + detectors running smoothly***
  MOTORS solved IcePap power overload problems on pd_oc/pd_mc (led to beamtime loss)
  CONTROLS got 90% of desired functionalites, 10% remaining in the pipe

- ***25 users experiment in 2013 / 29 (13 done) users experiments in 2014***
  (65% / 35 % PD/HP-microdiffraction, 2-3 days expt on average)

- ***11 (6 HP) paper published (+ 3 accepted) from MSPD data since opening in June 2012***
  8 (+2) with MSPD staff coauthorship
  (JACS, PRB, InorgChem, ChemMat, JPhys CondMatC, CementConcreteRes, PowderDiffraction, MatResBull, JPhysChemC)

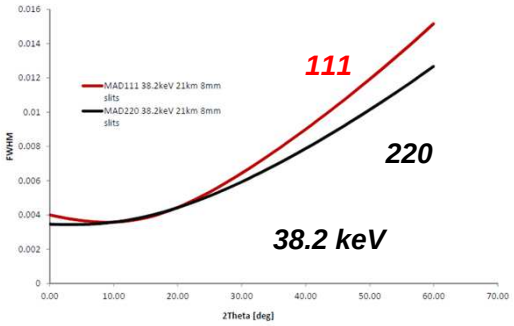
- ***Industrial beamtime sold to 6 different companies since July 2013***
  + 1 new to come (July 2014) + 1 new contact + industrial related research project
  (51 hours over 10 sessions, 4 users came back)

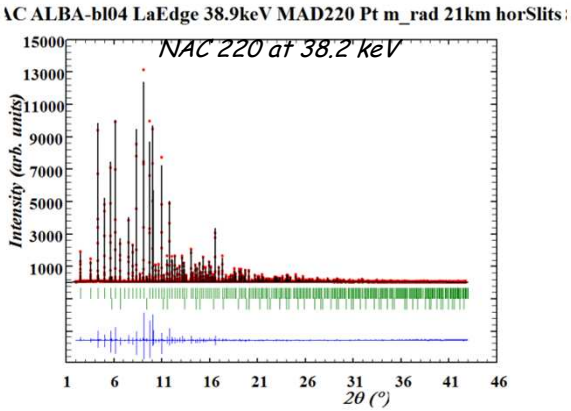
PD : high angular resolution (figure of merit)

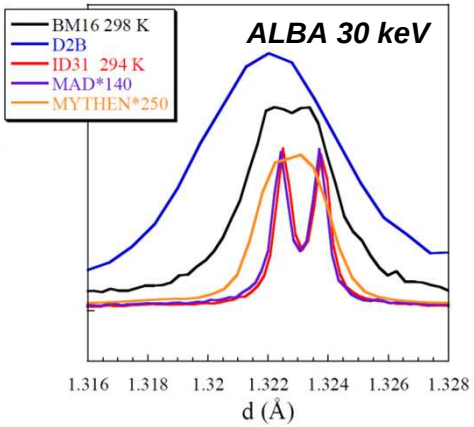
Activity on MAD :

- exchange central Si_{111} Xtal – intrinsic width was broader - with outer one (i13)

- commissioned the Si_{220} Xtal set





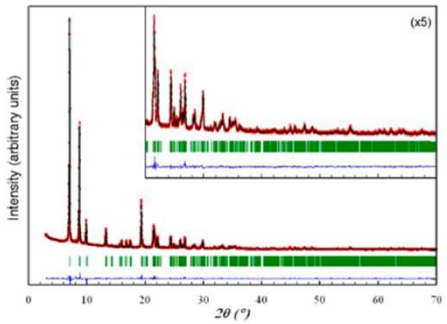


$YNiO_3$
90.0754 ° monoclinic distortion
Alonso et al, PRL (1999) 3871

Why high resolution needed ?

-> small distortions

-> overlap in structure determination



Xray lab

-> Le Bail Fit orthorhombic S.G

$a = 17.937$ Å, $b = 17.516$ Å, and $c = 12.375$ Å

-> extinction : Ima2m,I2cm, Imam, Imcm

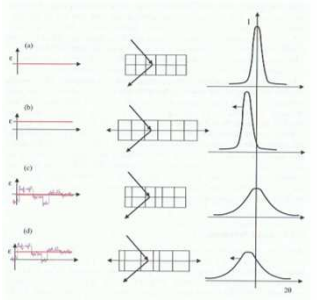
-> no reasonable structure

MAD data @MSPD

$a = 17.511$ Å, $b = 17.907$ Å, $c = 12.367$ Å, and $\beta = 90.22°$

I12/m1 S.G -> correct structure 10 Si , 19 O

PD : strain scanning experiments (Feb 2014)



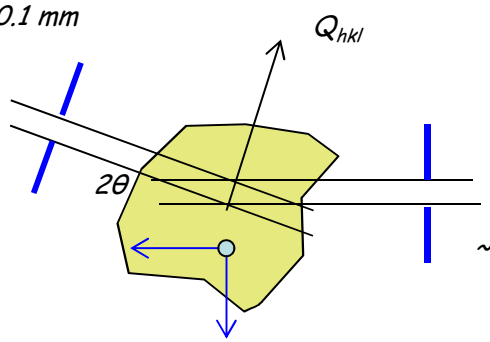
Basics

No strain

Macrostrain (e.g. tensile strain)

Microstrain

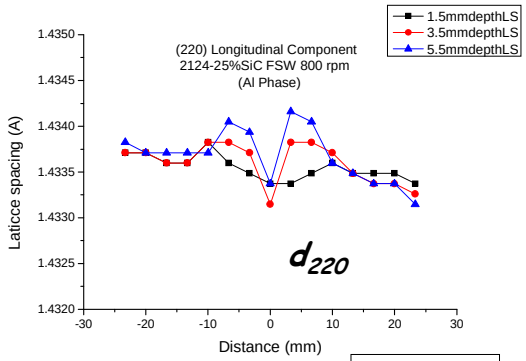
Macrostrain + Microstrain

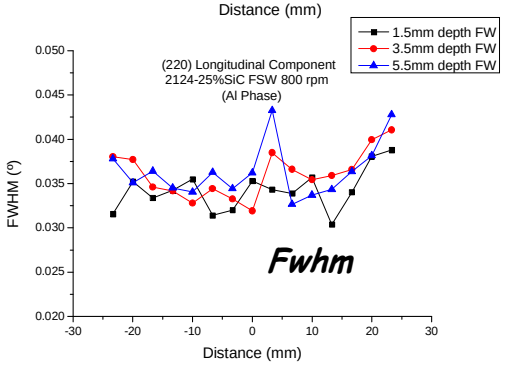


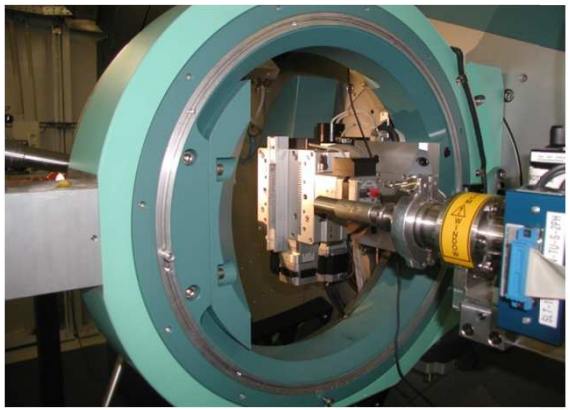
Experiment performed March 2014
E = 40 keV, mirror focusing

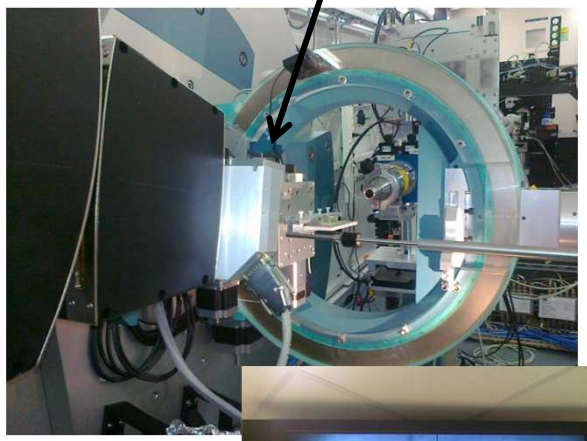
Slits in front of central channel of MAD

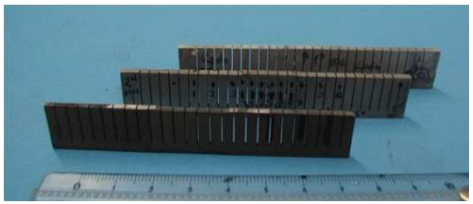
2124Al-25vol%SiCp

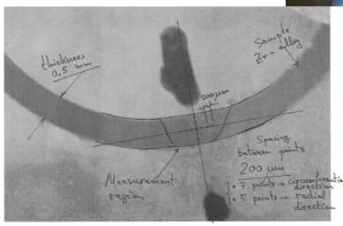


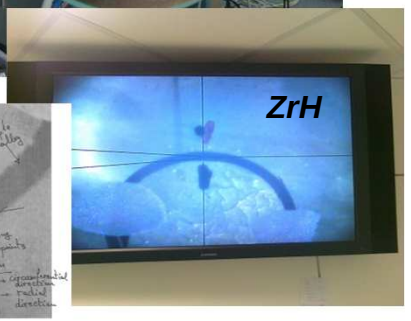










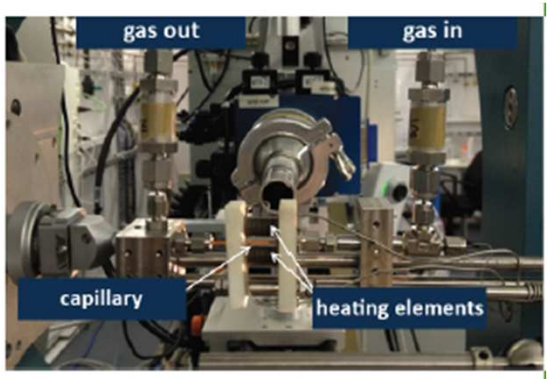


Determination of the microscopic residual stress in friction stir welds
Gaspar González Doncel, Ricardo Fernández Serrano, Florencia Cioffi
Dept. of Physical Metallurgy, CENIM, C.S.I.C.

+ J. Hervias et al
Departamento de Ciencia de Materiales ETSI Caminos. Universidad Politécnica de Madrid

PD : catalysis/gas flow experiments (Feb 2014)

The new ICP-ITQ-ICIQ-ALBA Capillary flow cell performed its first experiment



- The construction and commissioning of the cell has been the result of the collaboration of a group of researchers of the Spanish community: Laura Barrio (from EQS group at ICP), Fernando Rey's group (from ITQ) and Atsushi Urakawa's group (from ICIQ) with the scientific staff of the MSPD beamline. The design is based on the design by Peter Chupas et. al.J. Appl. Cryst. (2008). 41, 822–824

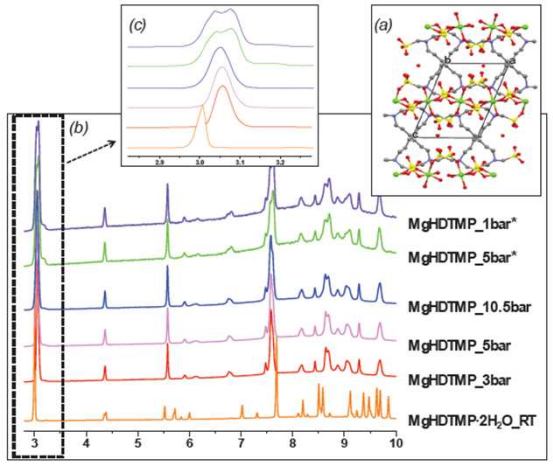


Figure 14. (a) View of the crystal structure of MgHDTMP·2H_2O along the b-axis. (b) X-ray diffraction patterns at different CO_2 pressures. The inset (c) shows the evolution of the peaks during the CO_2 adsorption-desorption. (*desorption).

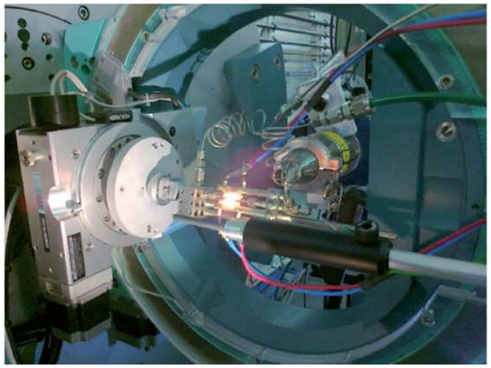
MYHTEN
20 keV
Cabeza et al ,
Dept Inorganic Chemistry
Uni Malaga

Inma Peral

Drawback :
share equipment with CLAESS
(gas mixing cabinet)
-> users expt scheduling problem

Adopt same protection system as in CLAESS (€ € €) if we accept selected gases

MAD 30 keV, L. Barrio et al, CSIC Madrid



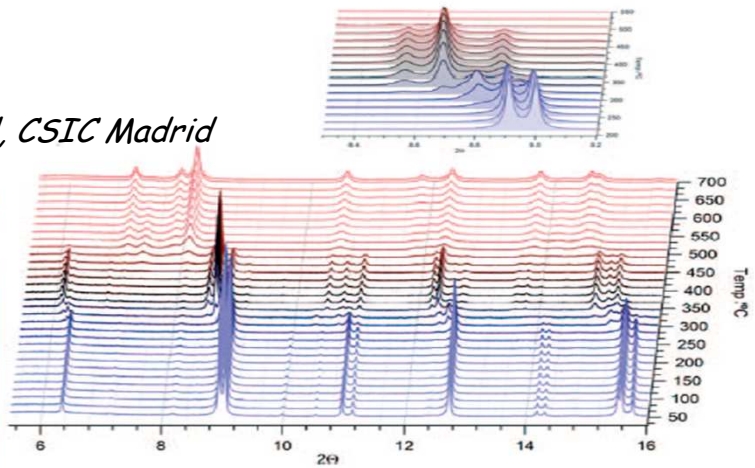
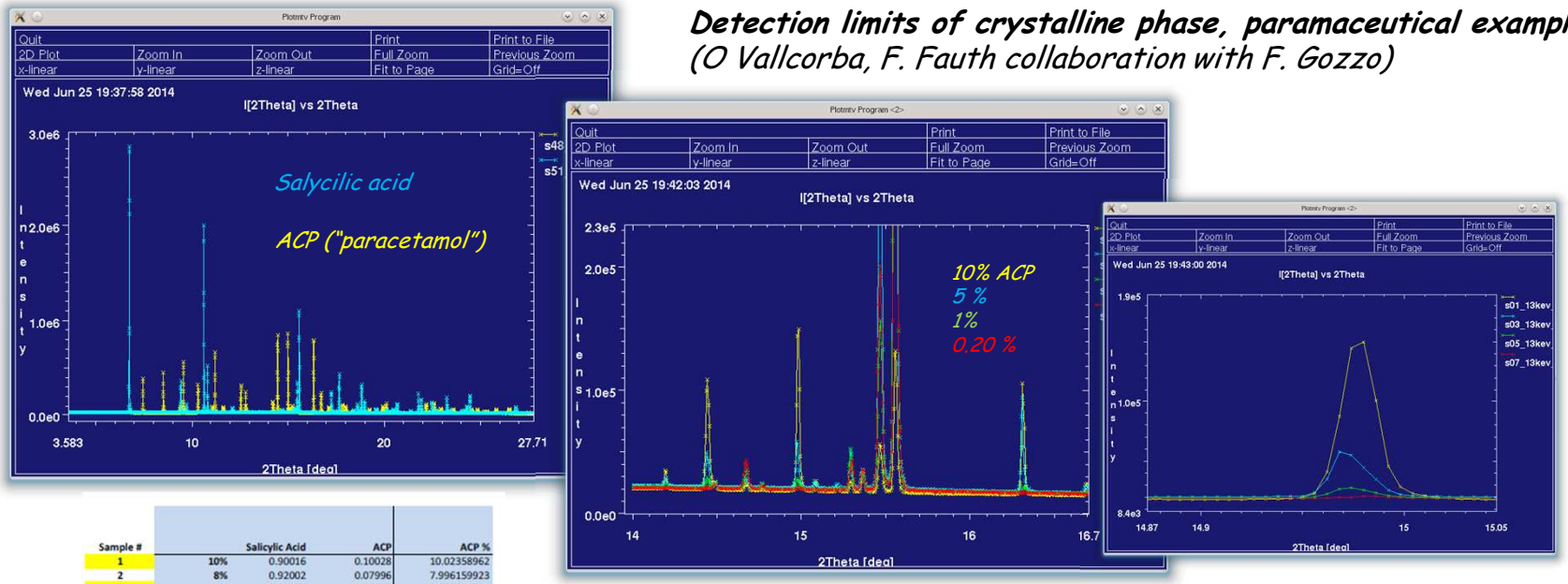


Figure 2. *Reduction of $LaCoO_3$ to La_2O_3 + Co under 5 % H_2/He gas flow. The transition is passing through a brownmillerite intermediate. Each temperature scan was collected in 8 minutes.*

PD : using MYTHEN for inorganic compounds



Detection limits of crystalline phase, paramaceutical examples
(O Vallcorba, F. Fauth collaboration with F. Gozzo)

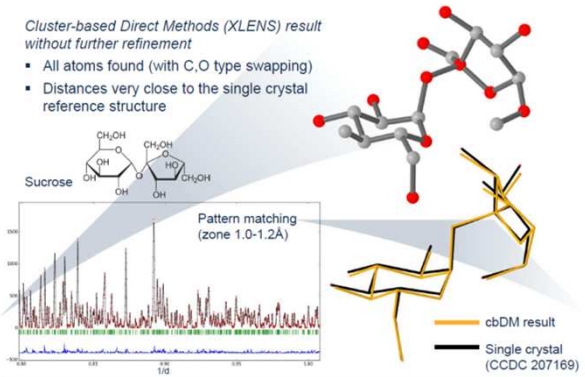
Sample #		Salicylic Acid	ACP	ACP %
1	10%	0.90016	0.10028	10.02358962
2	8%	0.92002	0.07996	7.996159923
3	5%	0.94976	0.05057	5.055331741
4	3%	0.96871	0.02968	2.972786186
5	1%	0.99004	0.01004	1.003919686
6	0.50%	0.99502	0.00497	0.49700497
7	0.20%	0.99802	0.00195	0.19500585
8	0.10%	0.99887	0.00106	0.106007421
9	0.05%	2.99836	0.00145	0.048336395
10	0.01%	2.9998	0.00031	0.010332954

Detection/quantification limit ~0.5 %

Industrial related projects in quantitative determination of amorphous/crystalline phase (O Vallcorba with a Local Pharmaceutical company)

Application of Direct Methods to MYTHEN data

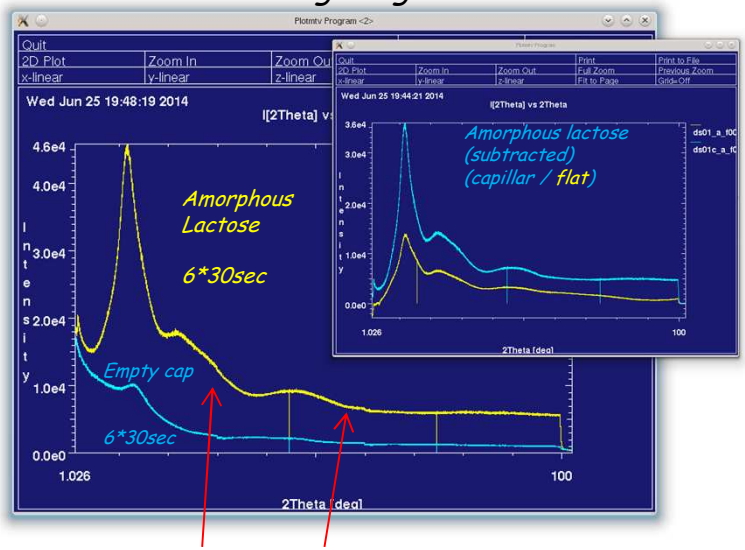
(O Vallcorbaa)

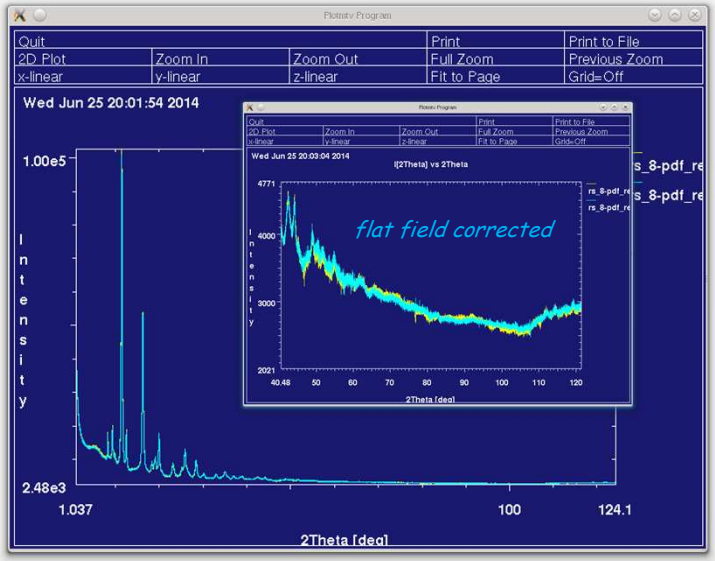


PD : inline/offline MYTHEN data processing

Hardware/software development for MYTHEN data acquisition/processing

- real beam monitor + real accurate position reading for each MYTHEN position
(PS ongoing project to integrate similar beam monitoring for the HP station as well)
- software for merging multiple positions from MYTHEN raw data inline/offline
(Bad channels option / flat field file option / merging selected position / produce converted file for each position, binning option,….)
- Protocol for fast flat field data collection (1-2 hours) either scanning a Silicon peak or glass sample data collection at high angle



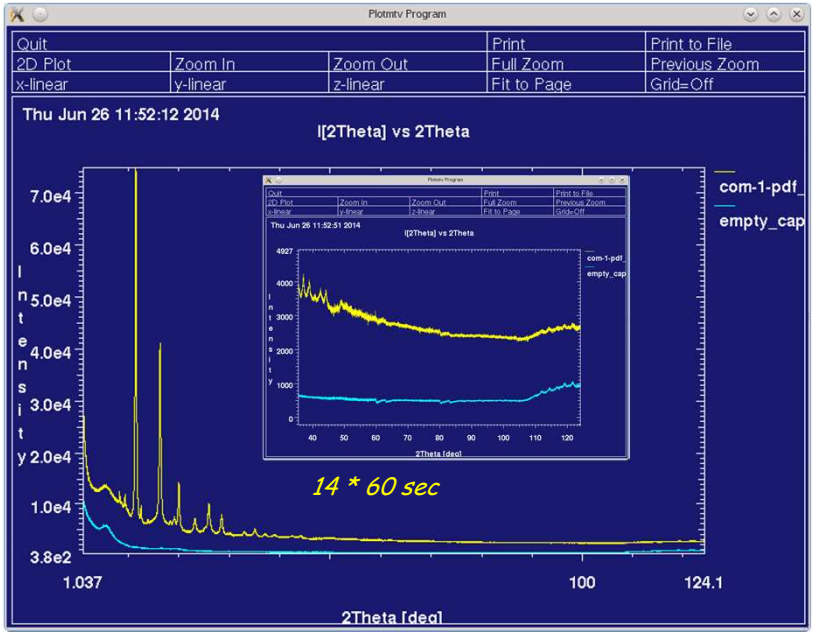


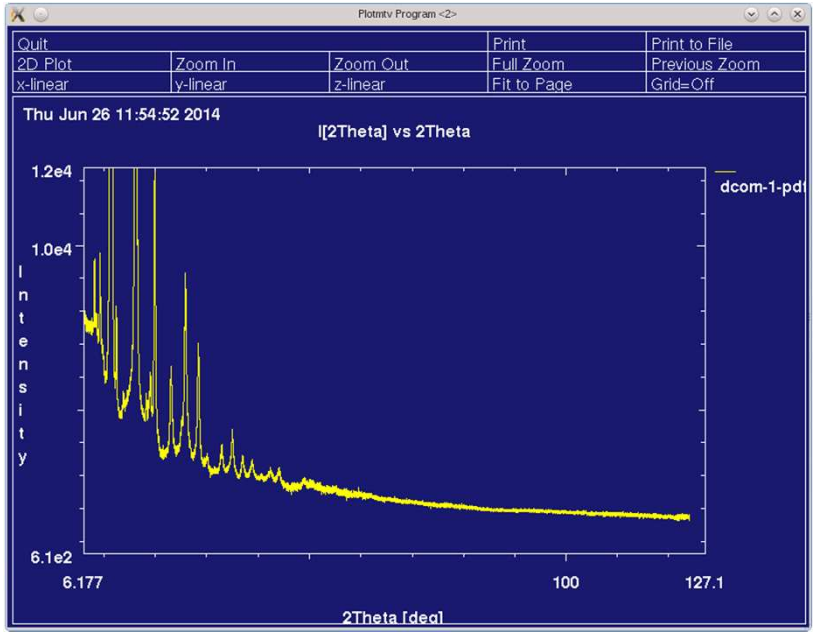
Steps at starting position of aquisition

What about continuous scan with MYTHEN ? (Briefly raised during last SAC meeting)

Motor speed + integration time + angular detector resolution (5 mdeg) are limiting factors
80 deg pattern -> 40 deg scanning (reminder 40deg angualr coverage), at 5 deg/min => 8minutes
but 5 deg/min -> 30ms integration over 2.5mdeg range
-> 16000 raw data files (7680points) to process

PD : pdf using MYTHEN

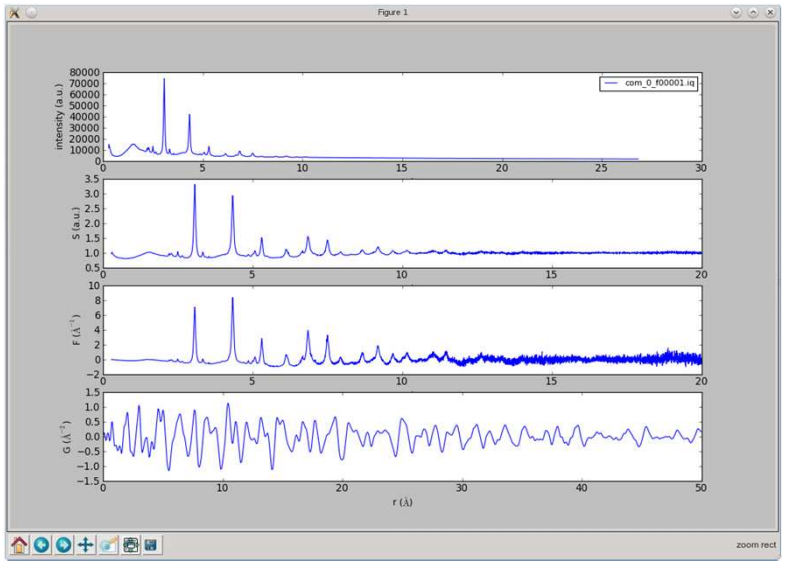




MYTHEN data collection at 30keV
14 positions of 1min
-> q_{max} ~ 25 1/Ang

Real users' sample (June 25th)

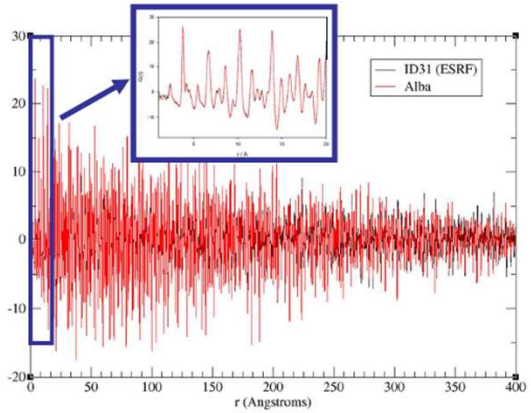
In Top Up mode !!!
BTW : no problem with Top Up on MSPD



PD : pdf on BL04 I. Peral

$Ce_{0.625}Sm_{0.375}O_{1.8125}$ was measured in ID31 (ESRF) and MSPD (ALBA)
Mauro Coduri (Lecco CNR IENI, Italy) and Inma Peral (Alba synchrotron)

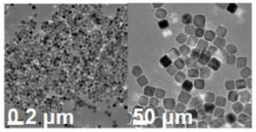
-Experimental PDFs present almost the same structural information
-Alba data presents slightly "less" signal of PDF at high r with "fast" acquisitio times (~20 minutes). MythenII detector at Alba offers "fast" acquisition and hig angular resolution (but not as high as multicrystal analyzers which explains the reduced signal at high r values).



- *high energy :* E ~30 keV to reduce truncation error in Fourier Transformation
- *Good quality data for Rietveld, profile analysis and PDF studies*
- *high angular resolution: exploiting PDF on a wide r-range*

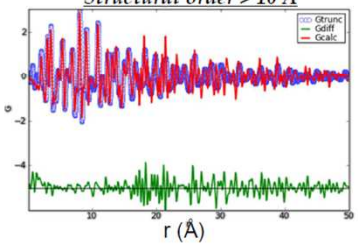
Example of Pair Distribution Function Study: Structural study on bi-magnetic core/shell magnetic nanoparticles

Josep Nogués's group (CIN2) and Inma Peral (Alba synchrotron)



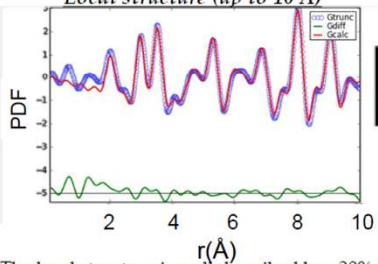
- ✓ The defects, vacancies and interface mismatch of the samples seem to have a direct influence in the overall magnetic properties of the system but little of this "disorder" is known.
- ✓ The PDF can probe different length scales

Structural order > 10 Å



- Magetite and wustite phases do not reproduce the PDF signal
- Evidence of strong interphase mismatch and strain within the nanoparticles: atomic distances correlation is lost at ~5nm

Local structure (up to 10 Å)



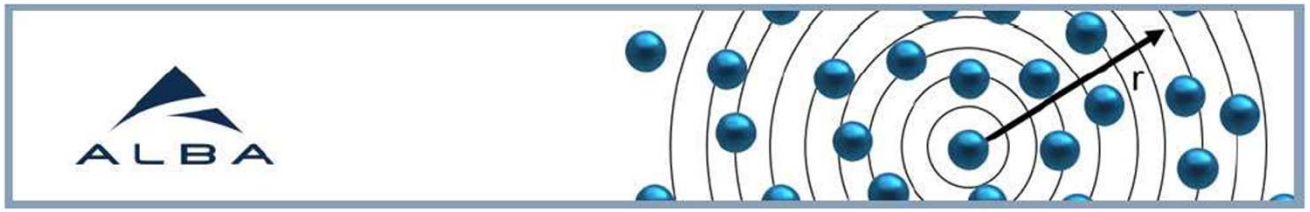
The local structure is well described by ~30% of wustite (FeO) and ~70% of magnetite (Fe_3O_4).
In agreement with the study of the Fe oxidation states by EELS (Electron Energy Loss Spectroscopy)

+ 2 'expert' users experiments to come (Coduri + Barrio)

Master research work on

Pair Distribution Function on Nanostructures

Albert Castellví Toledo

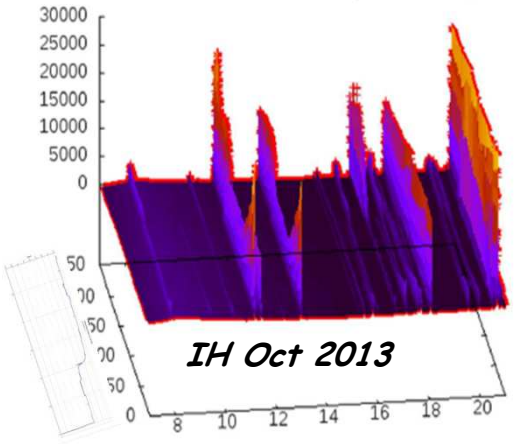


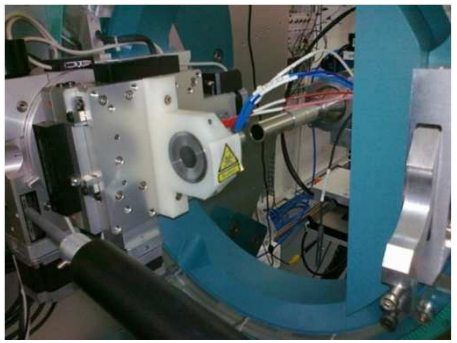
Hands-on course on the Pair Distribution Function method

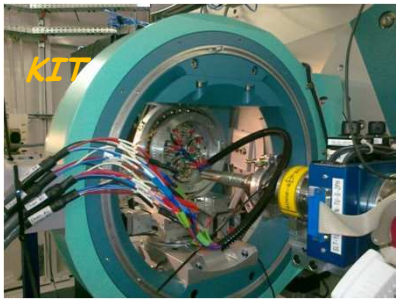
12-14 November 2014
ALBA Synchrotron
UTC timezone

Electrochemistry using the MYTHEN detector

Bianchini, Suard (Grenoble) Croguennec (Bordeaux), Masquelier (Amiens), Palacin (Barcelona), Fauth (Alba)







CHEMISTRY OF MATERIALS *just accepted*

Subscriber access provided by UNIV AUTONOMA DE BARCELONA

Article

Na3V2(PO4)2F3 revisited : a high-resolution diffraction study

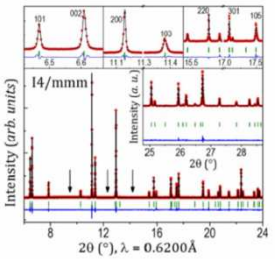
Matteo Bianchini, Nicolas Brisset, François Fauth, François Weill, Erik Elkaim, Emmanuelle Suard, Christian Masquelier, and Laurence Croguennec

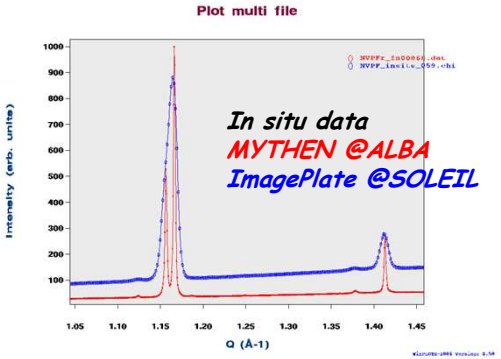
Chem. Mater., **Just Accepted Manuscript** • Publication Date (Web): 19 Jun 2014

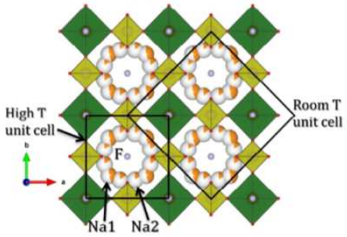
Downloaded from http://pubs.acs.org on June 24, 2014

ALBA MAD data
HT and LT
(down to 100K)

RT at SOLEIL







In 2014 :

Bianchini 3 days, Palacin 3 days, (Following preliminary IH test)
Karlsruhe Institut Technology group (M. Knapp) : 5d + 4d + 4d (oct)

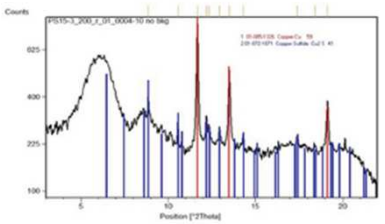
-> all will likely apply for 2015-I round

Shedding light to historical glaze decorations

BL04-MSPD and BL22-CLAESS

T Pradell, G Molina Universitat Politecnica de Catalunya
J Pla, J Molera, Universitat de Vic

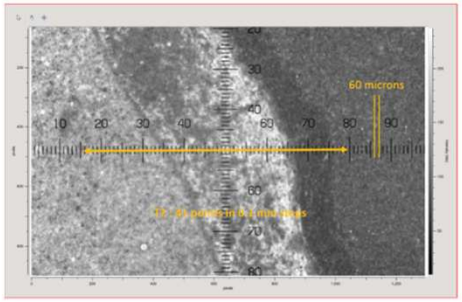
Cultural Heritage

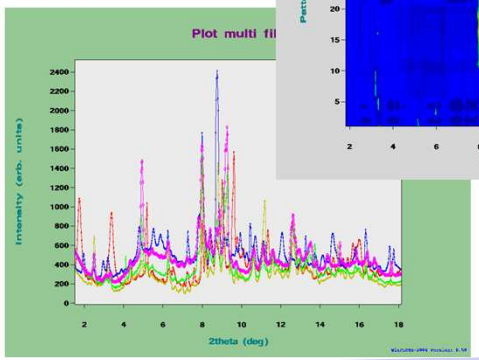


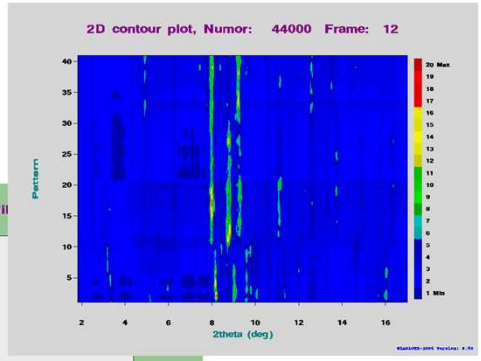
This group focused their research on the micro-and nano-crystalline compounds responsible for the colors and decorations in historic glass and glazed ceramics. The nature of these decorations depends on the procedures followed to obtain them. Two main sets of historic materials were studied: decorated stained glass fragments from various periods and cathedrals in Spain (14th to 16th AD) to discover materials and methods used and early opaque Islamic glazes from Syria and Egypt (7th and 8th AD) to identify the connection with opaque glass technology.

2012010267 - Mineral quantitative determination on the interface of cementitious bi-layers by synchrotron microdiffraction

X. Turillas, UPM-CSIS - ALBA



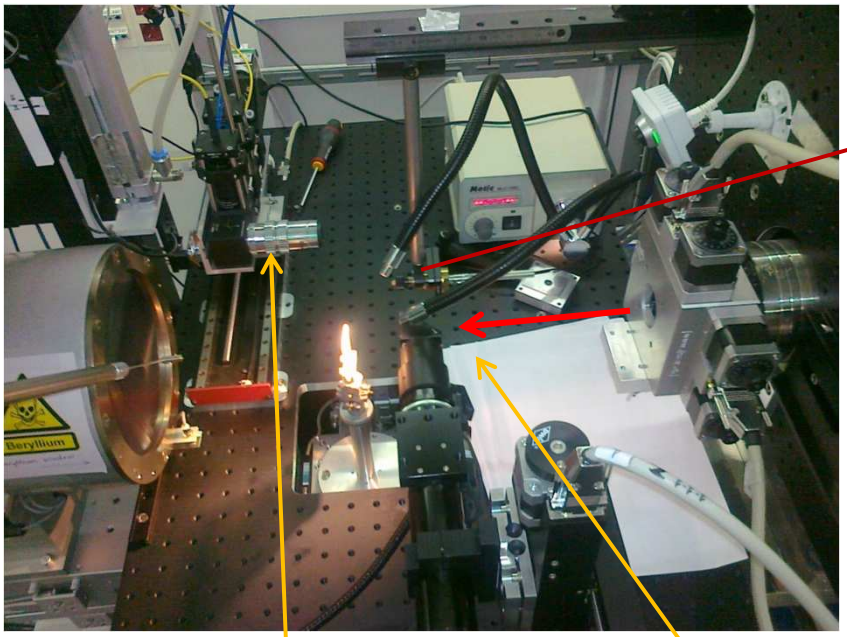




3 micro diffraction users' experiments so far

3 to come in 2014-II

HP : technical development for micrdiffraction



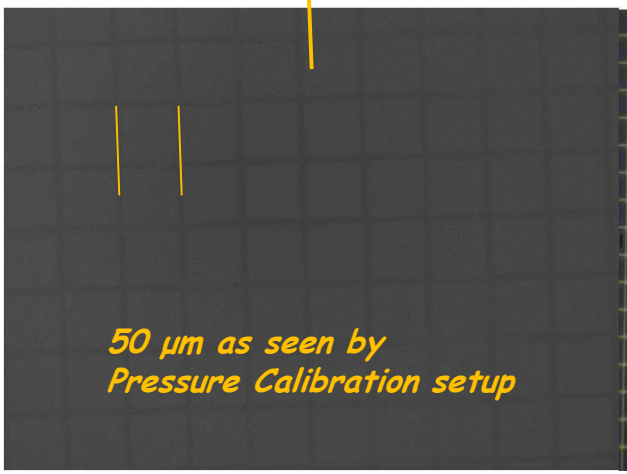
On axis visualisation (I Sics development) -shared with CLAESS

OAV-pinhole interference

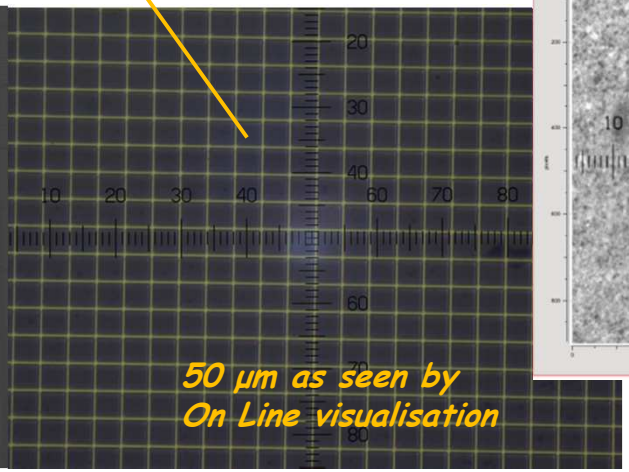
xyz motorized translation, zoom/focus motorization

…but lack of magnification and proper GUI interface :
frame grabber, 3-click centering, 'drawing 'objects (line, circle, distances….)

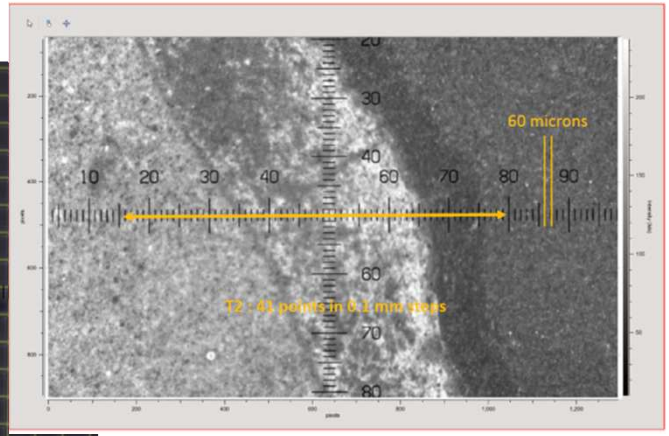
Such a GUI as well needed for the HP calibration setup



50 µm as seen by Pressure Calibration setup



50 µm as seen by On Line visualisation



HP : technical development for pressure calibration

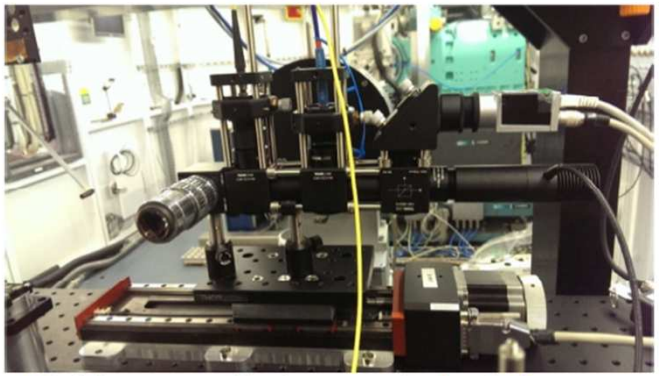
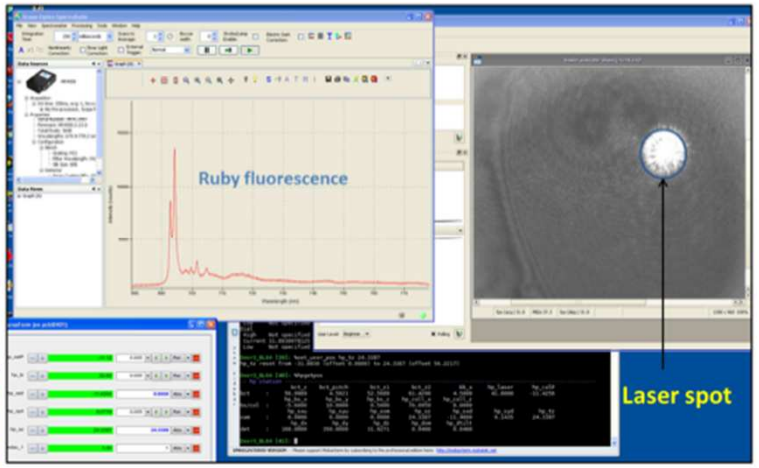
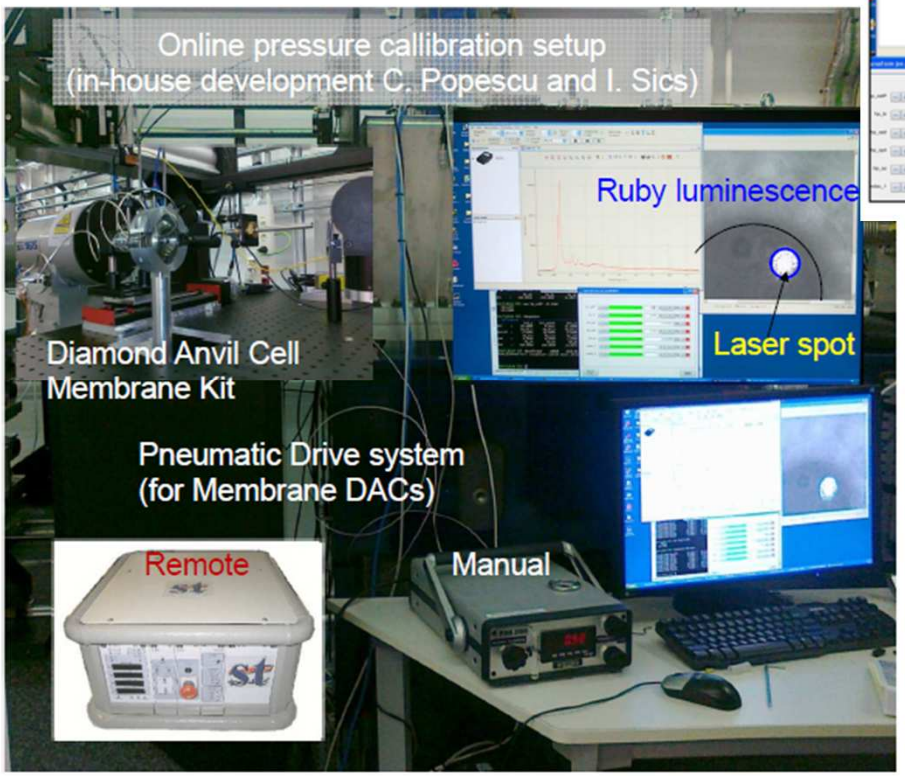


Figure 1 The inline pressure calibration system using the ruby fluorescence method.

C Popescu, I Sics
ALBA awards for technical development





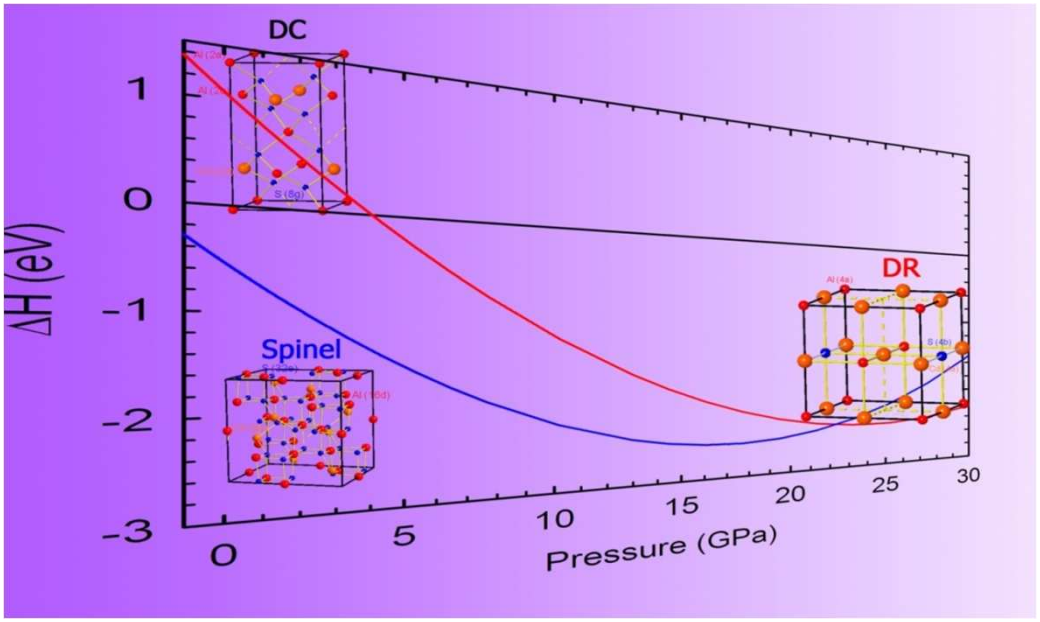
Pressure determination ?

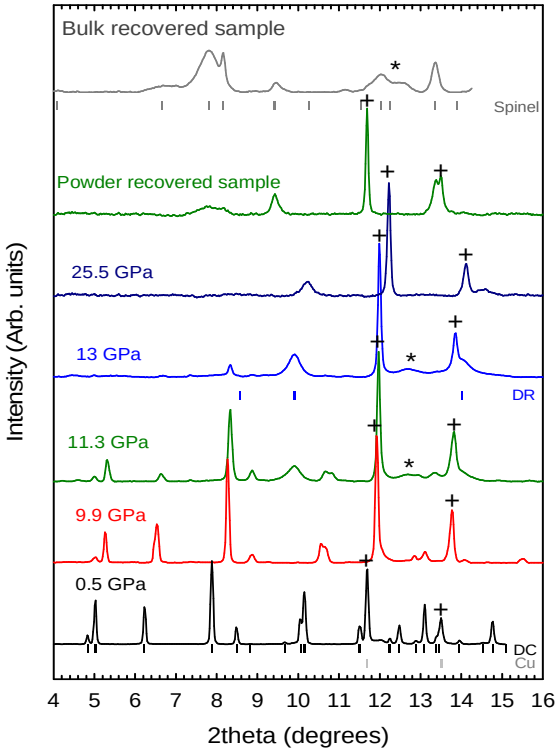
Put an internal standrad (e.g. Cu, Au…)
EoS known : $d_{111\ spacing}$ <-> internal pressure
(drawback : possible overlap with other peaks)

Ruby fluorescence technique :
$Al_2O_3:Cr^{3+}$ crystals illuminated by a laser (blue) -> fluorescence signal
-> peak position <-> internal pressure

$CdAl_2S_4$ under high pressure

Defect chalcopyrite ↗ *Disordered Rocksalt* ↘ *Cubic Spinel*





THE JOURNAL OF PHYSICAL CHEMISTRY C

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Article

Structural and Vibrational Properties of CdAl2S4 under High Pressure: Experimental and Theoretical Approach

Juan Angel Sans, David Santamaría-Pérez, Catalin Popescu, Oscar Gomis, Francisco J. Manjon, Rosario Vilaplana, Alfonso Munoz, Placida Rodriguez-Hernandez, Veaceslav V. Ursaki, and Ion Tiginyanu

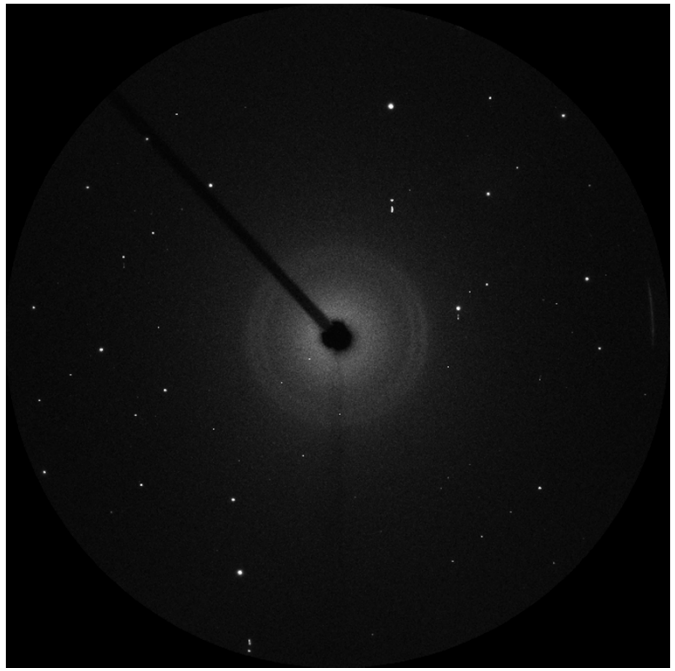
J. Phys. Chem. C, **Just Accepted Manuscript** • Publication Date (Web): 24 Jun 2014

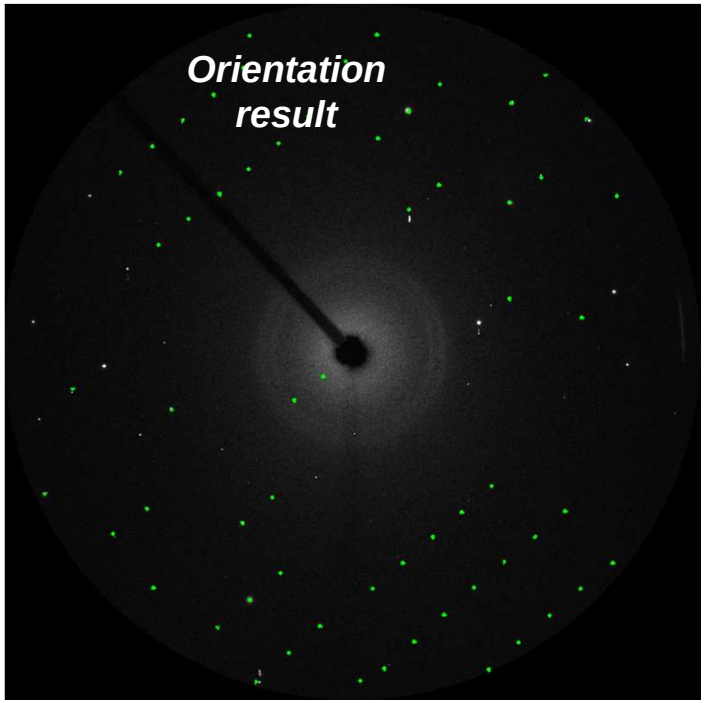
See Poster

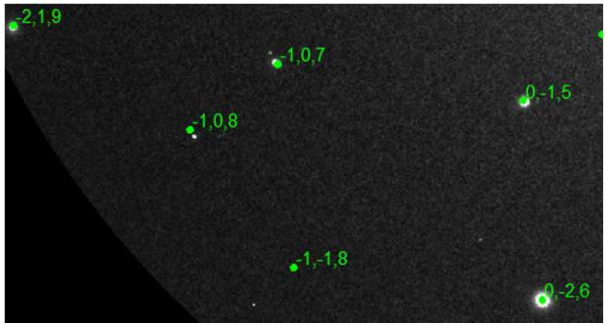
(O Vallcorba, J Rius, I Peral)

Intensity extraction from random oriented grains

Epidote mineral, $Ca_2Al_{2.25}Fe_{0.75}(Si_2O_7)(SiO_4)O(OH)$

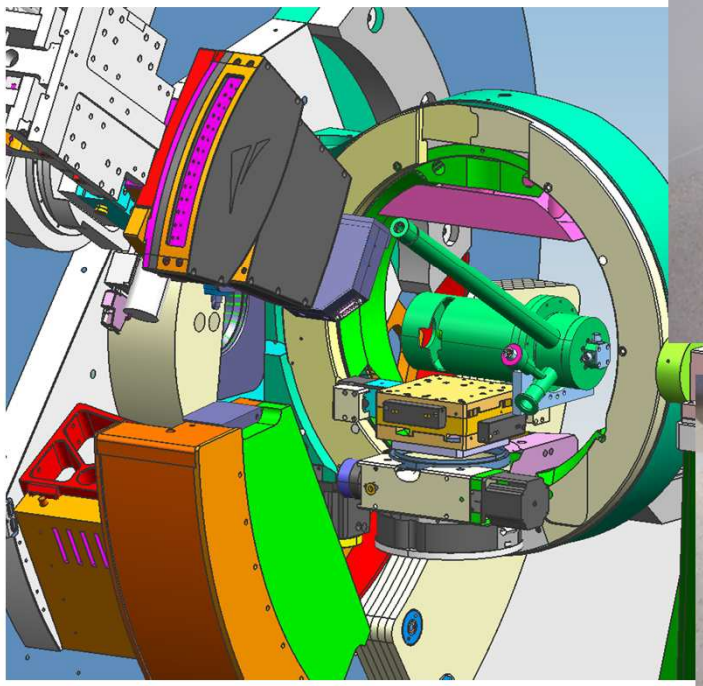


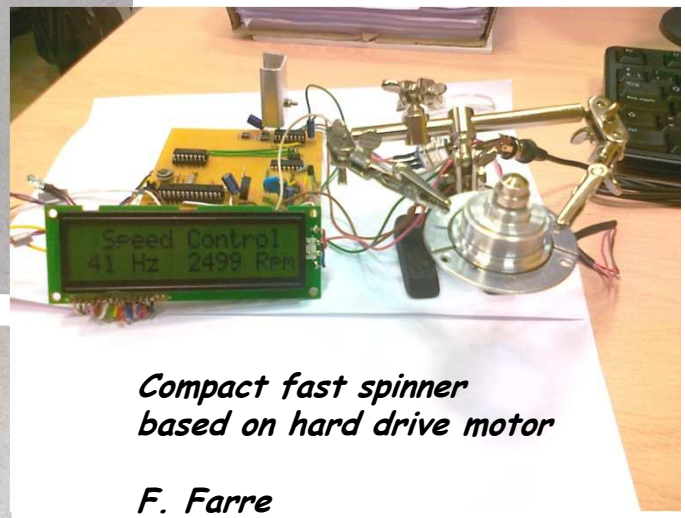




Requirements:
- Image with the contribution of a reduced number of grains
- Cell parameters
- Slight oscillation of the sample (more reflections, reliable intensities)

PD : Sample environment projects





Compact fast spinner based on hard drive motor

F. Farre

lHe continuous flow cryostat – to be shared with CLAESS-
order to the ESRF (expected delivery end 2014)

Ancillary equipment to be ordered
transfer line, controller, pump,
dewars if local lHe supply, organize lHe supply/recovery ?)

Engineering design for integration and sample spinning
E Fraga, ExpDiv transverse

- ***-> 1000 °C furnace (2 proposals required for it last round)***

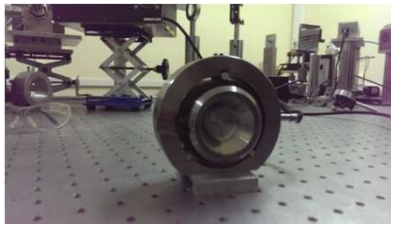
commercial STOE chamber ?

halogen lamp furnace like at ESRF ?
-> in house technical development
but technical/engineering ressources

WARNING : shared equipment -> users scheduling, limit number experiments

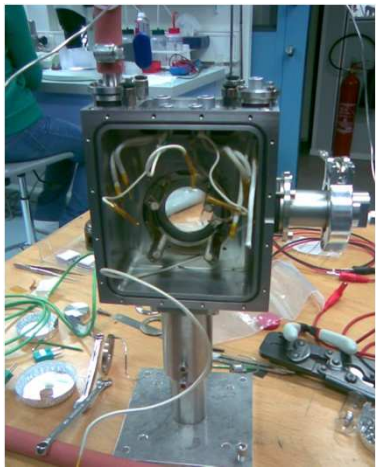
HP : Sample environment projects

- ***First HT-HP experiment performed in Dec2013***
  Errandonea, McLeod, McMahon
- ***Isothermal pressure dependencce in Ti***
  (EoS : up to 250 C, 30 GPa)
- ***-> continuation at higher T in Dec 2014***
  more poweful ALBA power supply + internal heaters
- ***Using own equipment, agree to transfer technology***



Commercial solution (BETSA)
- *compatible with our HT ready membrane cell*
- *limited to 400° C in air,*
- *? 600° C in vacuum (chamber to be designed)*

bullky ,
specific cells and heaters required



ESRF design
Purely based on internal microheaters (up to 1200 °C)

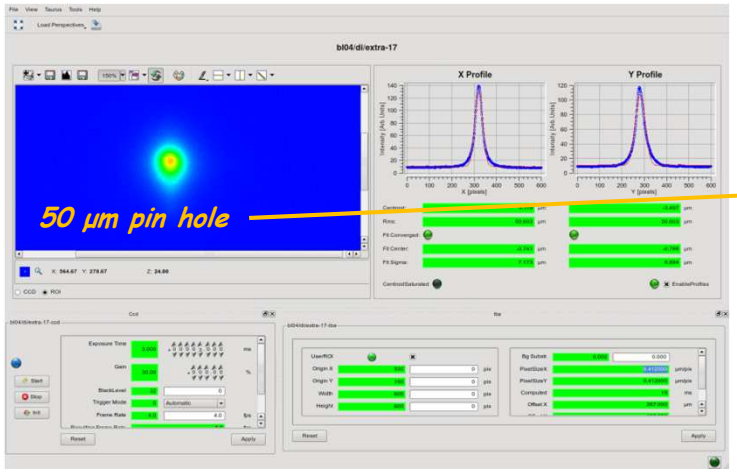
HP instrumental developments are delicate, time consuming, high level of engineering/technical skill

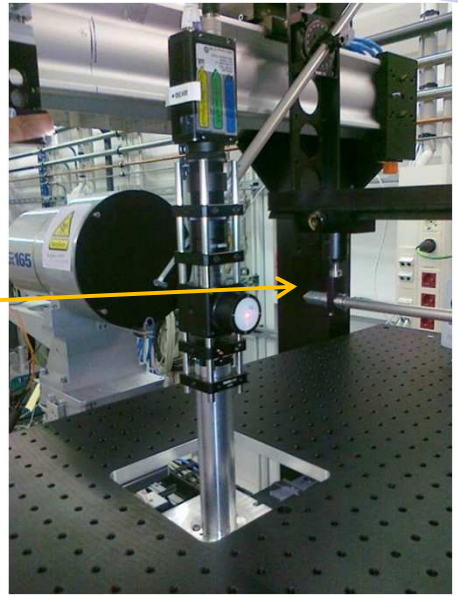
HP demand for development might increase if CLAESS and MIRAS jump in

HP : beam size as limiting factor

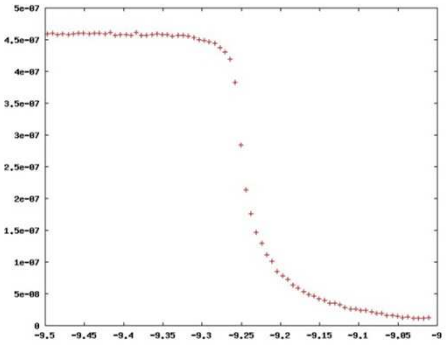
Beam size at HP
-> limit measurements
to Ø 80 µm gaskets
~ 50 GPa

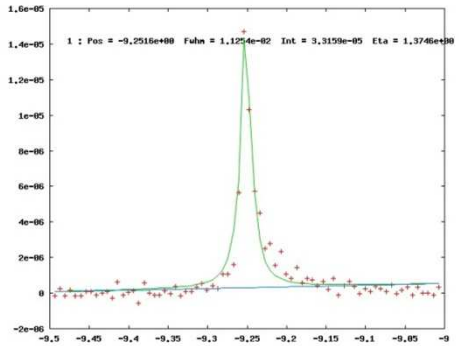
Direct spot size illumination

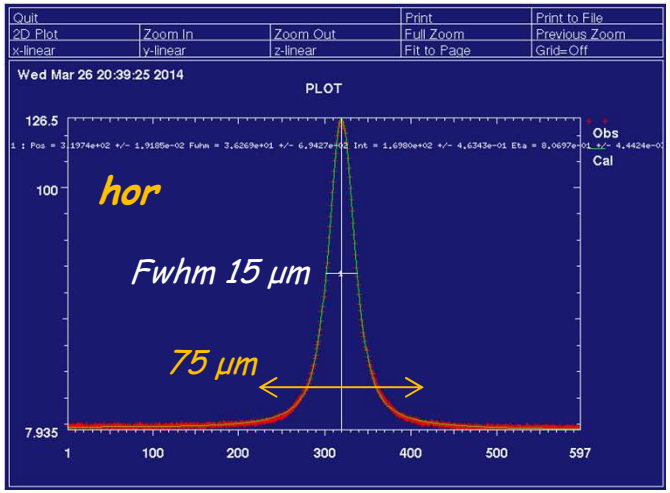


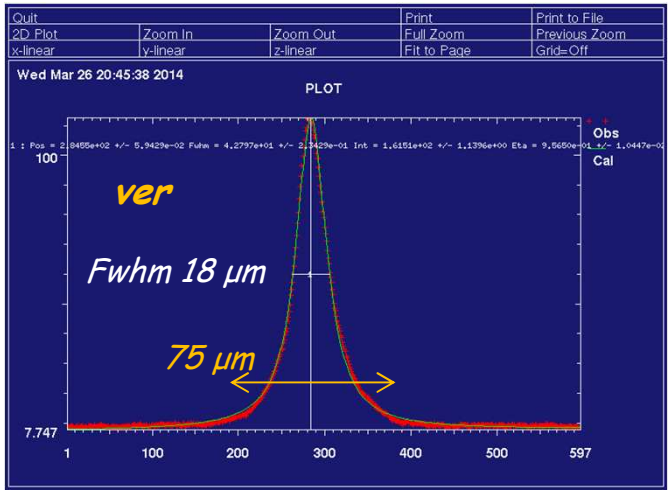


Knife edge scans







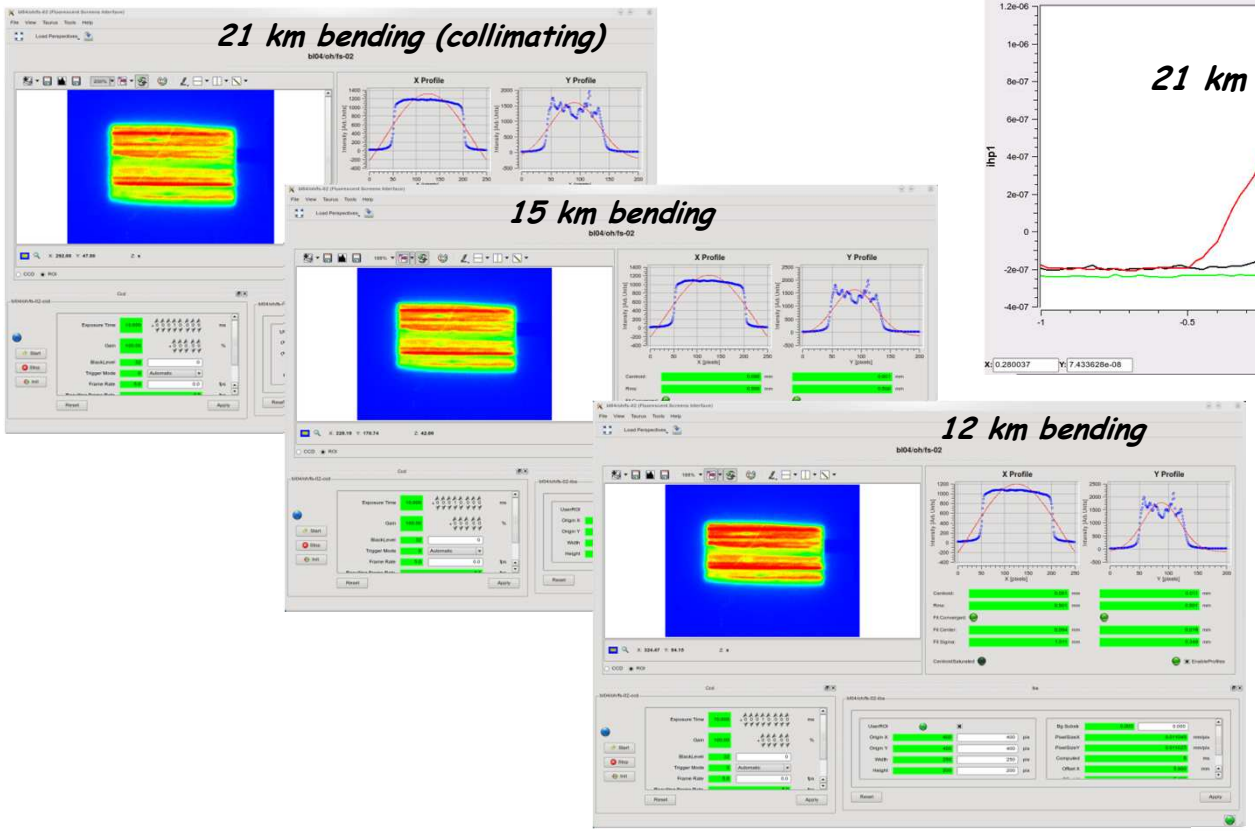


0.7 mm x 0.7 mm aperture in FE and before KB, Ø 50 µm pinhole

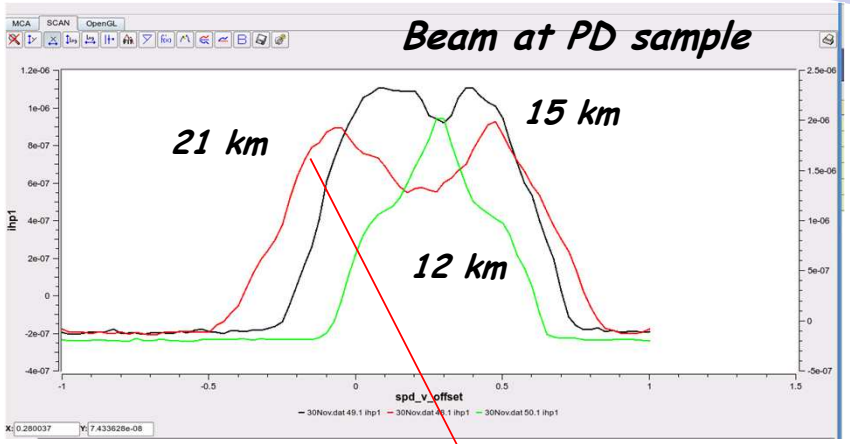
PD Striation effect on 1st mirror

Signorato et al , SPIE 3152, Moreno et al , SPIE 5921

Beam after mono

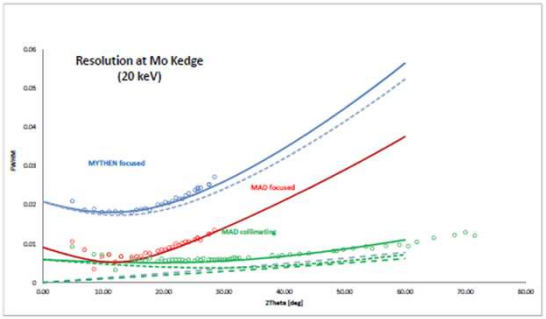


Beam at PD sample



Peak structured reflected in MYTHEN not in MAD

-> focus at MYTHEN (9.5 km bending) to have symmetric – non structured - peak shape
-> small vertical beam (~0.7mm height), hard to operate with capillars (organics)…but good angular resolution
-> not the same bending setting for MAD and MYTHEN, limit simultaneous data collection



BL04 : long term 'unreasonable ?' projects

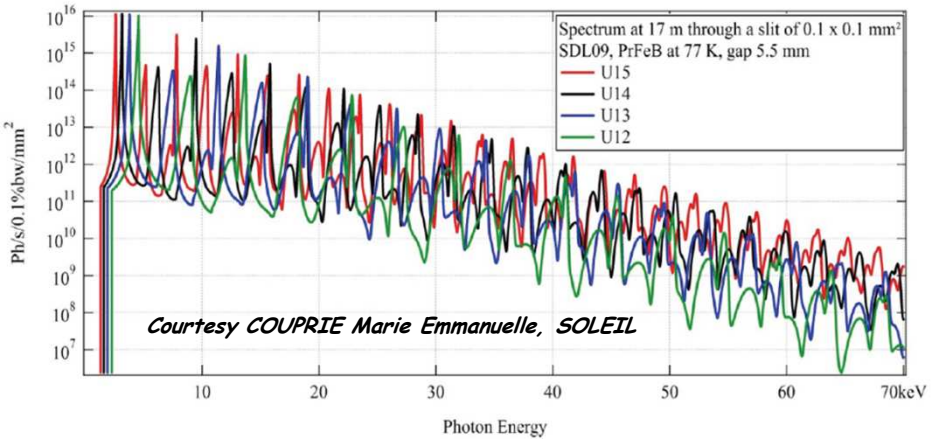
HP : a smaller source is desired for beam size < 10 µm
PD-MYTHEN : get rid of mirror to eliminate striation structure effects
but keep a reasonable vertical divergence
PD-MAD : keep reasonable vertical divergence without mirror
for preserving angular resolution

-> change the wiggler by lN_2 cryogenic undulator and operate without 1st mirror
(BL04 designed for operating in unmirrored, check heatload !)

MSPD : SC Wiggler
@200mA -> B= 2.1 T , ***K=6.08***, P_{max} 11.8 kW
mono designed fro 800W locally

Done at SLS-MS beamline (2.3 geV machine, 5-40 keV energy range):
Nd-Dy-Fe_B alloy at 150K 14mm periode, 2m length, 3.6mm min magn gap

Development at SOLEIL
Pr-Fe-B alloy eg 15 mm periode 5,5 magn gap, 5.5 x 2 mm pole size -> B= 0,93 T K=1,303



Of course low flux at high E …but to compare Wiggler/Undulator numbers with our small FE aperture

BL04 : long term 'more reasonable ?' projects

Commission KB mode to PD station (20-50keV)
(No time, no high demand for it so far… may be for strain scanning)

PDF using focused beam and 2D image plate detector to acquire (Perkin Elmer)

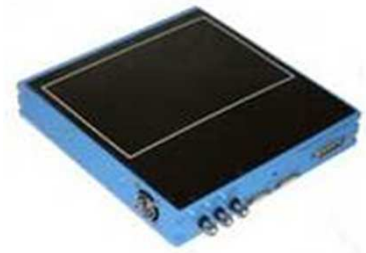
SNBL @ ESRF

Relatively cheap option but afterglow problem

~~Dexela~~

Now Perkin Elmer

2923 CMOS X-ray detector
29 x 23 cm



Pixel size	74.8µm	MTF at 6 lp/mm [1]	>20%
Sensitive area	290.8 mm x 229.8 mm	DQE [2]	> 0.7 at 0.5 lp/mm
Resolution	3888 x 3072 pixels	Saturation charge	1.4 Me⁻ (high saturation mode) 0.36 Me⁻ (high sensitivity mode)
Sensor type	CMOS active pixel sensor	ADC resolution	14 bits
Chest wall to active area	3.3 mm	Detector dynamic range	typ. 70dB (high saturation mode) typ. 66dB (high sensitivity mode)
Pixel binning	1x1, 1x2, 1x4, 2x1, 2x2, 2x4, 4x1, 4x2, 4x4	Dark current	8000 e⁻/s (typical at 40C)

HP technical development (in collaboration with Valencia group + CLAESS + MIRAS ?)
Low Temperature cryostat
He gas cell loading equipment
High temperature
Inhouse design/production of membrane

HP Workshop likely to be organized in 2015 at ALBA

I am giving the floor to SAC

Thanks for your attention !