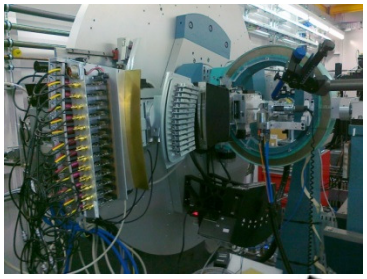
BL04 - MSPD Beamline

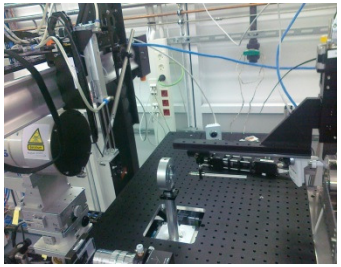
Catalin Popescu, ***cpopescu@cells.es***

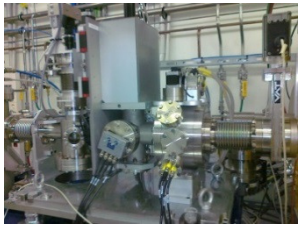
BL Postdoctoral Research Fellow of MSPD
Experiments Division

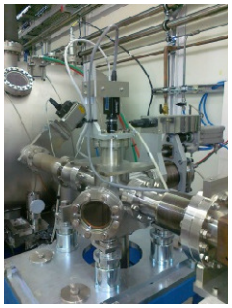
CELLS - ALBA http://www.cells.es
Carretera BP 1413 Km 3.3
08290 Cerdanyola del Vallès, Barcelona (Spain)

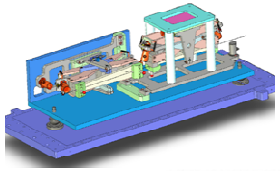
MSPD beamline overview

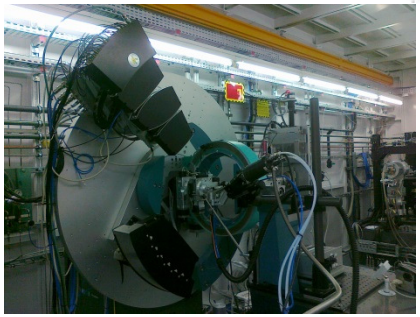


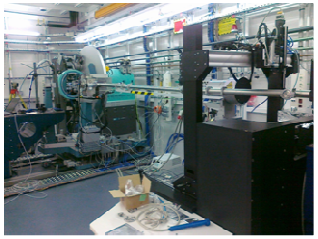


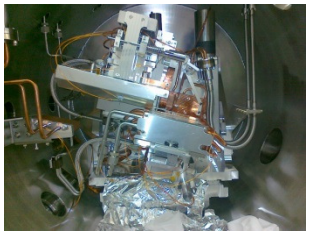


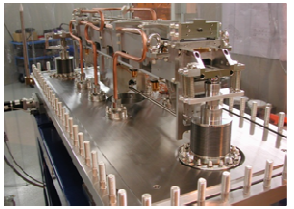


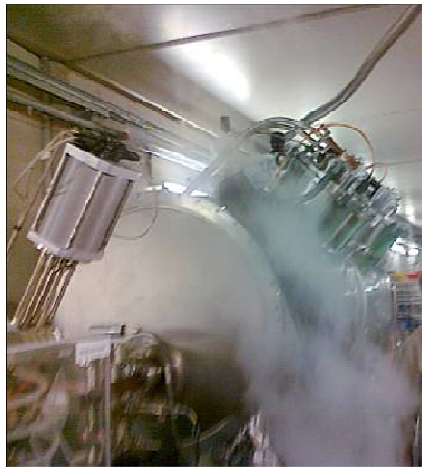












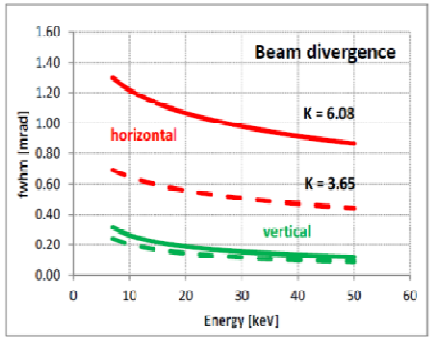
Oct 2011 : Wiggler installation and first cool down
(150 LN_2 and 900 LHe required)

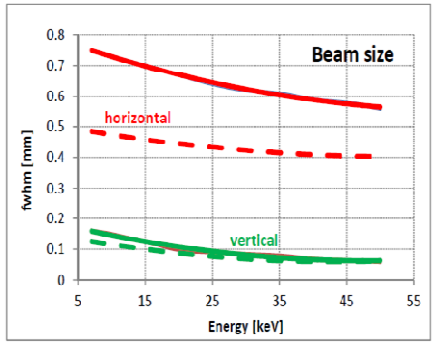
MSPD : Superconducting Wiggler

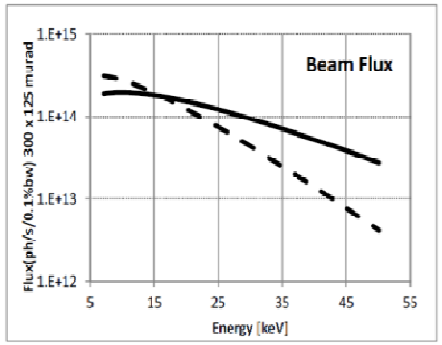
Budker Institute of Nuclear Physics (Novossibirsk, Russia)

B_{max} 2.1 T, 31 mm period, 60.5 periods

@200mA -> B= 2.1 T , ***K=6.08***, E_c = 12.5 keV, P_{max} 11.8 kW
@400mA -> B= 1.26 T, ***K=3.65***, E_c = 7.5 keV, P_{max} 6.8 kW







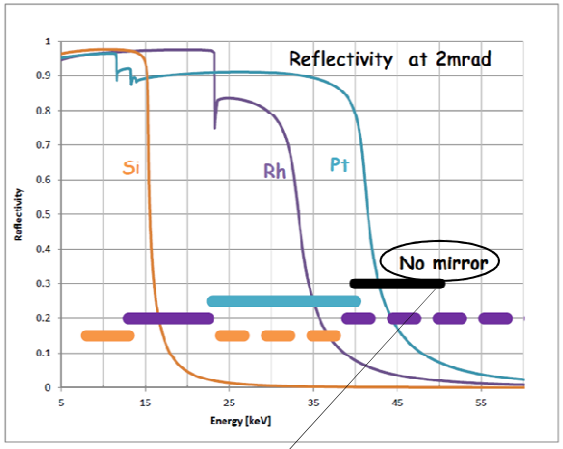
MSPD beamline optics : collimating mirror

Mirror (CINEL, Italia)

21 m from the source, 4 m before monochromator

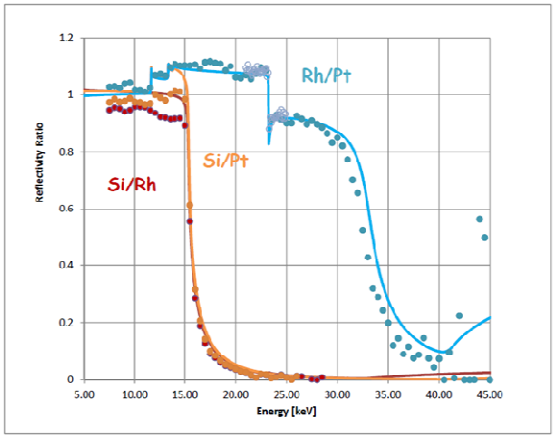
1.2 m long, 2 mrad incident angle, 3 stripes (Si, Pt, Rh), water cooled
1.5 μrad slope error specified

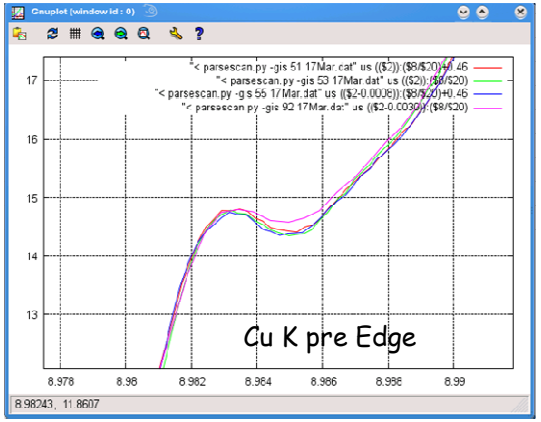
- reduce heat load on monochromator
- attenuate 3rd harmonics after monochromator
- collimate the beam (Radius = 21 km),
  - -> residual vertical divergence under heat load~ φ_{M1}~10μrad)
  - -> but striation effects
- focus beam on MYTHEN detector (Radius = 9 km)



adjustable height
for all downstream components
(up to 56mm at PD station)

No mirror 40-50 keV





Energy [keV]

Why collimation ? -> improve energy resolution

Why collimation for Powder Diffraction ?
-> improve angular resolution

$$\frac{\Delta E}{E} = \sqrt{\omega_s^{\,2} + \varphi_{M1}^{2}} \cdot ctg(\theta_B)$$

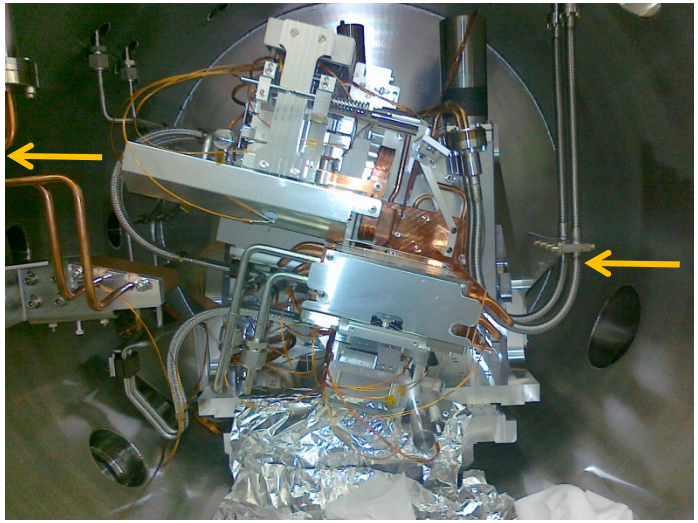
-> 1.3-3.3 10^{-4} (8-50 keV)

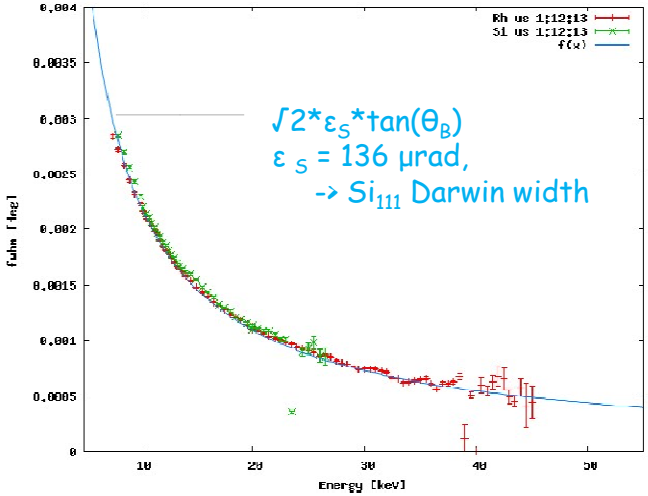
MSPD Double crystal monochromator
(Accel/Bruker, Germany)

-1st and 2nd silicon crystals cryogenically cooled,
-Inside mechanics (crystals support, shields, rotation,….) thermalized by secondary water circuit
-7 motor axes : dcm_bragg, dcm_pitch2, dcm_piezo2, dcm_t2, dcm_roll1, dcm_roll2, dcm_vert

Si_{111} Bragg reflection, fixed exit offset of 20 mm

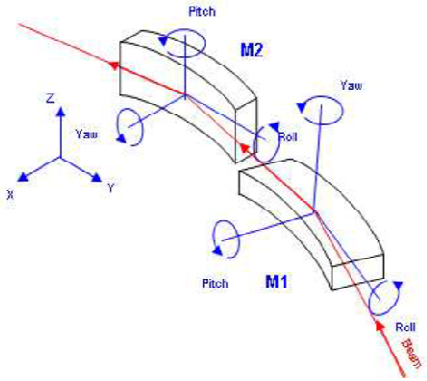
MOCO : Monochromotor 2nd Xtal pitch regulation (either in intensity or in position)





2nd crystal rocking curve vs. Energy

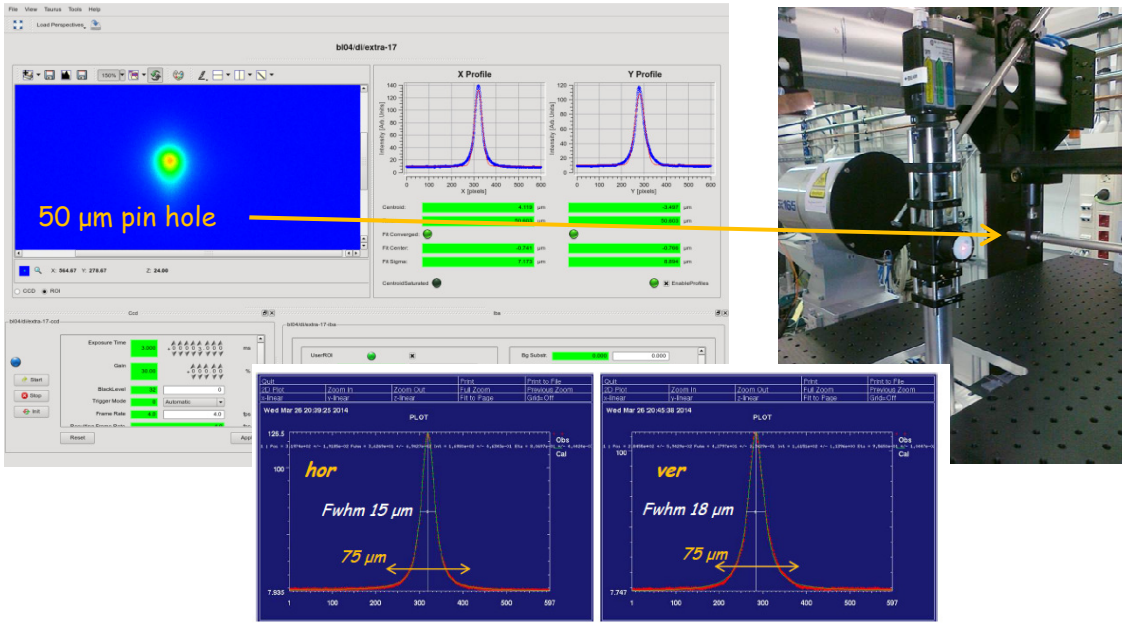
Kirkpatrick-Beatz focusing optics – Irrelec France (ESRF licence)



- 30m form the source, 1.1/0,7 m before HP sample
- Elliptical bendable,
- Grading [W/Si] multilayer :
  - Vertical p/q= 29.9/1.1, $[W/Si]_{100}$, d~3nm, slope error < 1 µrad
  - Horizontal p/q= 30.3.9/0.7, $[W/Si]_{150}$, d~2.6nm, slope error < 1.5 µrad
- Focus spot of 12 µm (H) x 7 µm (V) at sample position over 20-50 keV

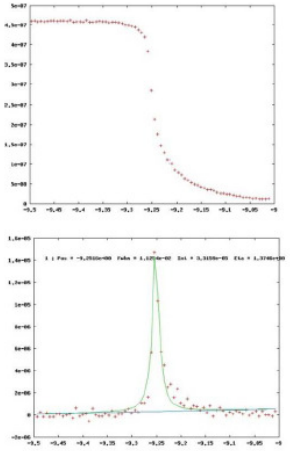
KB optimized to focus at the HP station, but designed to focus at the PD station as well

Direct spot size illumination



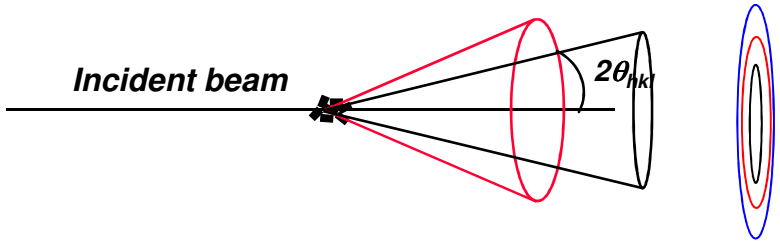
0.7 mm x 0.7 mm aperture in FE and before KB, Ø 50 µm pinhole

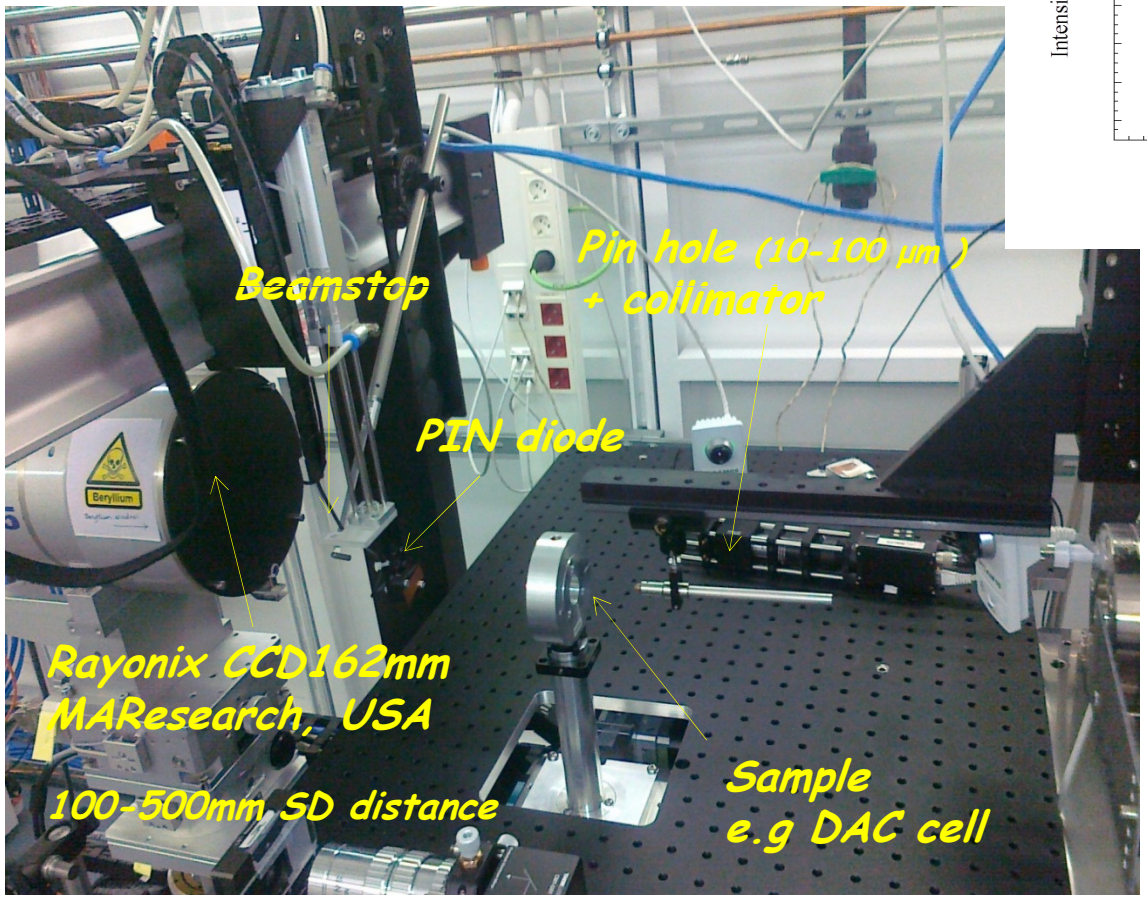
Knife edge scans



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

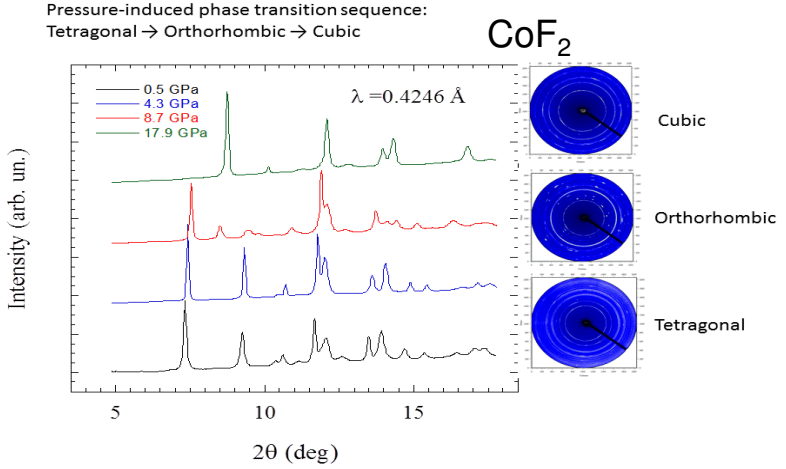
MSPD beamline : HP station



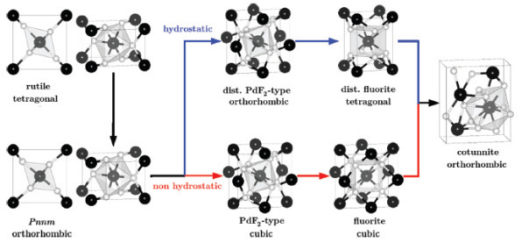


Powder diffraction patterns versus pressure

Pressure-induced phase transition sequence:
Tetragonal → Orthorhombic → Cubic

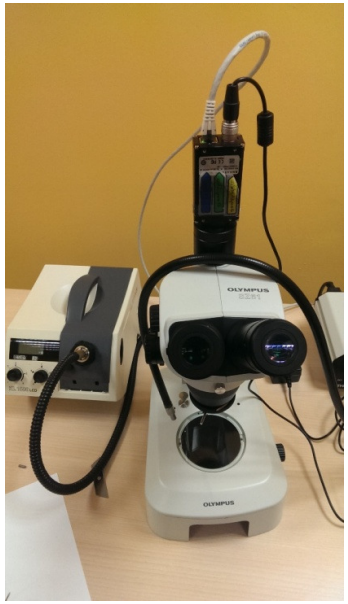


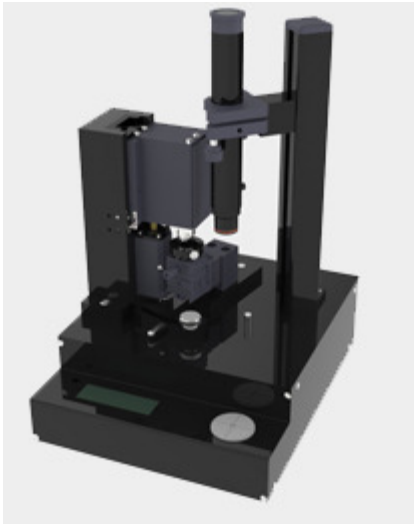
Density Function Theory of structural phase transition sequence in CoF_2 under ideal hydrostatic and non hydrostatic conditions



First HP User experiment (June 27, 2012), Cantabria University Group

Phys Rev B 88 (2013) 214108

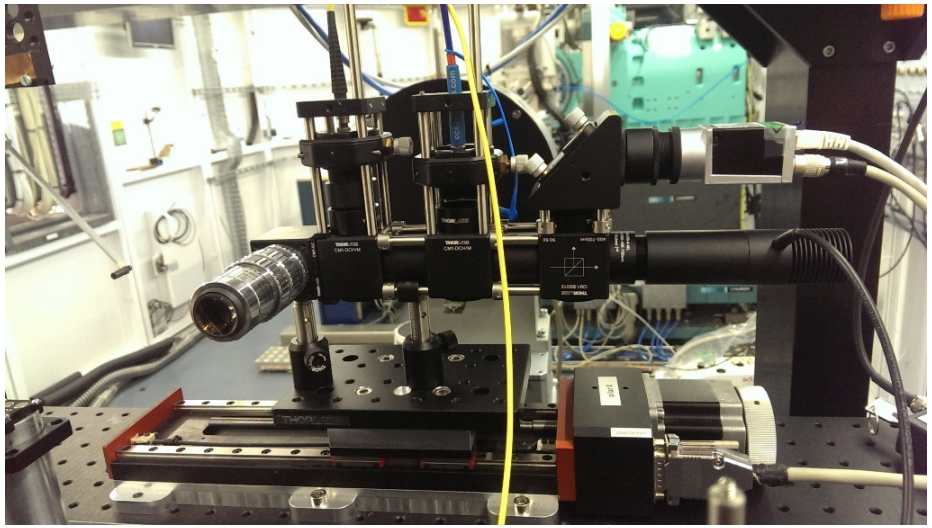

OLYMPUS
OLYMPUS

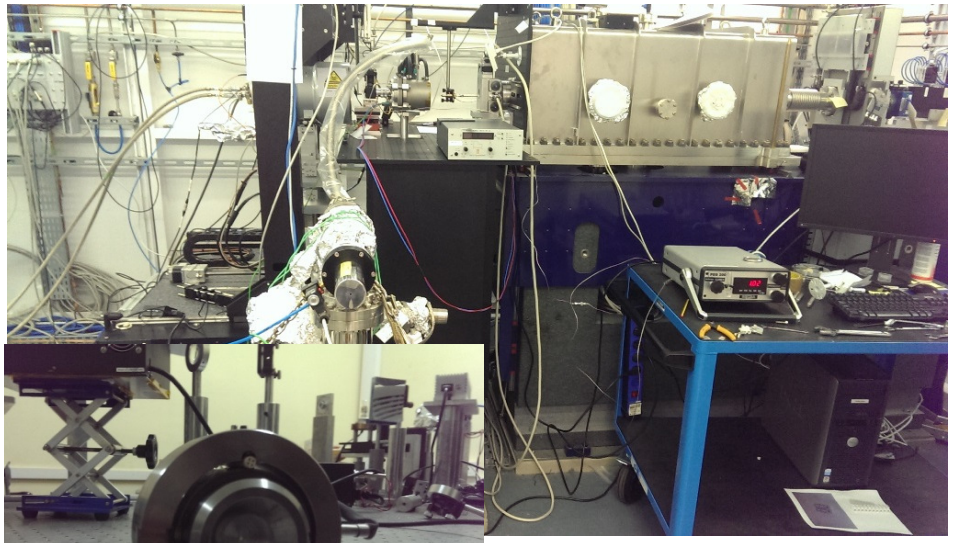


Alba award for technical development
Inline pressure calibration device

HT – HP experiments

- limited to 700 K
- 10% difference in temperature
- vacuum chamber to be designed
- increase the Tmax



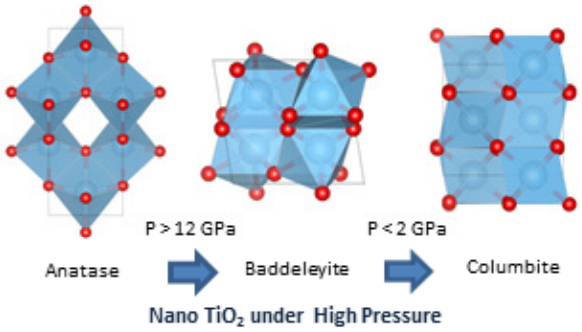


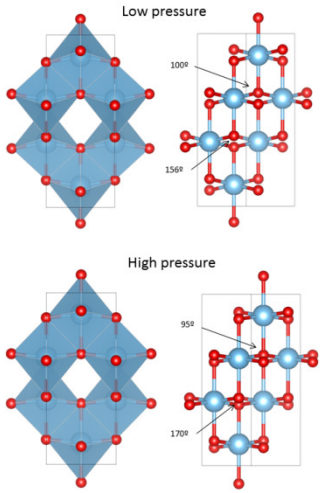
Inorganic Chemistry
Article
pubs.acs.org/IC

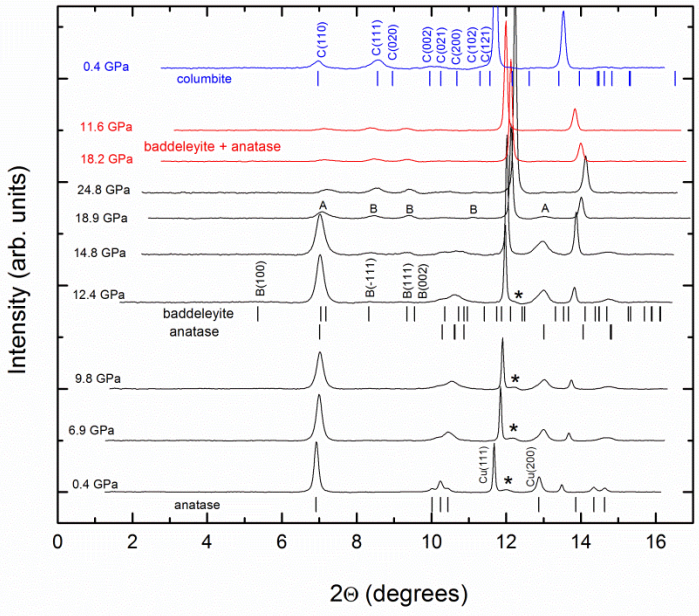
Compressibility and Structural Stability of Nanocrystalline TiO_2 Anatase Synthesized from Freeze-Dried Precursors

Catalin Popescu,*,† Juan Angel Sans,‡ Daniel Errandonea,§ Alfredo Segura,§ Regina Villanueva,∥ and Fernando Sapiña∥

†CELLS-ALBA Synchrotron Light Facility, 08290 Cerdanyola, Barcelona, Spain
‡Instituto de Diseño para la Fabricación y Producción Automatizada, MALTA Consolider Team, Universitat Politècnica de València, 46022 València, Spain
§ICMUV-Departamento de Física Aplicada, MALTA Consolider Team, Universitat de València, 46100 Burjassot, Spain
∥Institut de Ciencia dels Materials, Universitat de València, Apartado de Correos 22085, E-46071 València, Spain







HP data @MSPD:

Rietveld fit > structure solved
Effect on pressure on atomic positions, bond distances and bond angles >> increase the distortion of TiO_6 octahedra >> anatase more planar with pressure >> trigger the phase transition

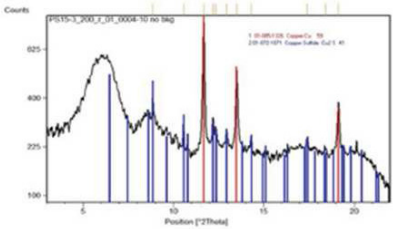
Shedding light to historical glaze decorations

BL04-MSPD and BL22-CLAESS

Cultural Heritage

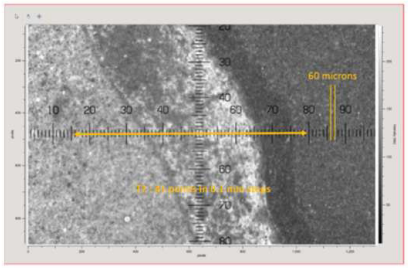
T Pradell, G Molina Universitat Politecnica de Catalunya
J Pla, J Molera Universitat de Vic

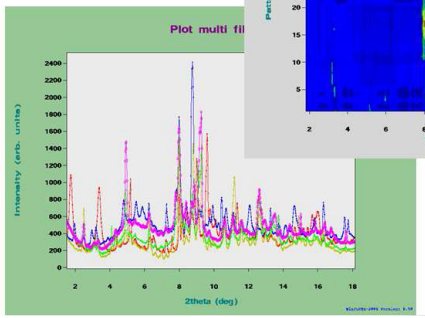
- This group focused their research on the micro-and nano-crystalline compounds responsible for the colors and decorations in historic glass and glazed ceramics. The nature of these decorations depends on the procedures followed to obtain them. Two main sets of historic materials were studied: decorated 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.

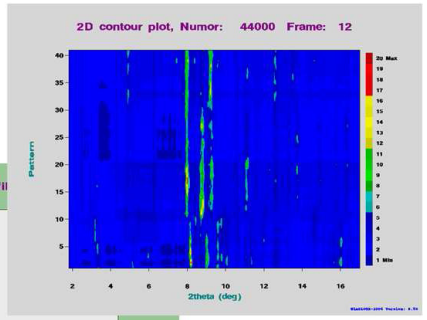


2012010267 - Mineral quantitative determination on the interface of cementitious bi-layers by synchrotron microdiffraction

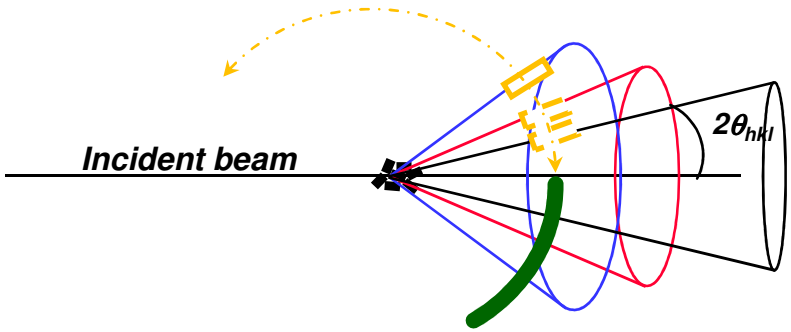
X. Turillas, UPM-CSIC – ALBA

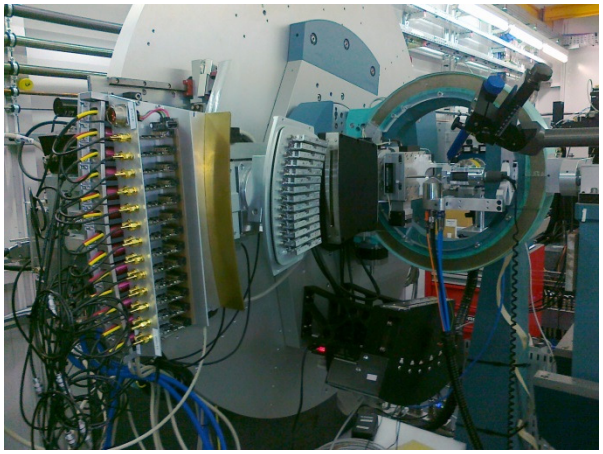


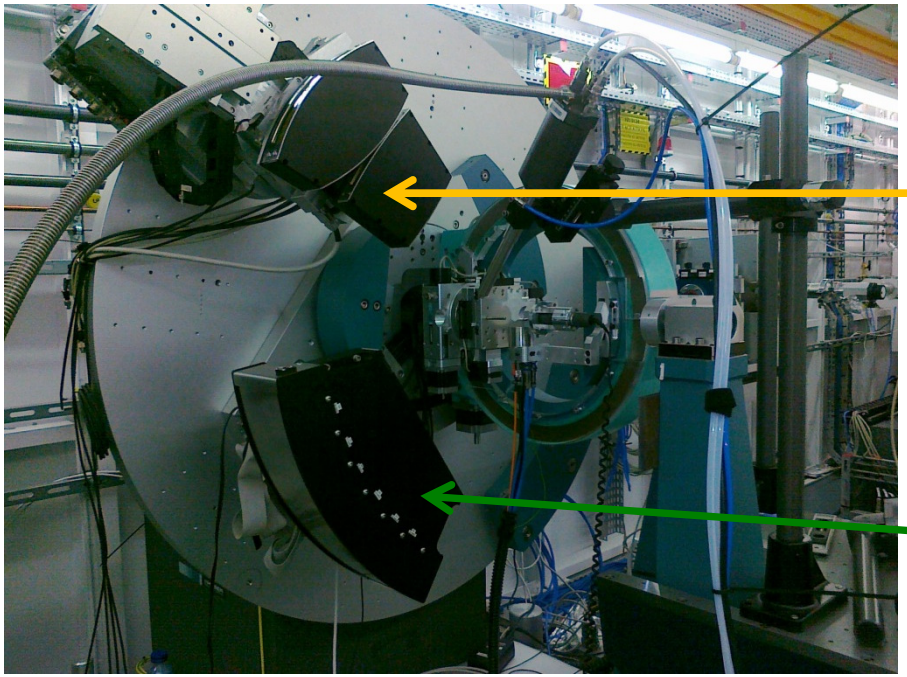




MSPD beamline : PD station







Multi Analyzer Detector (8 – 50 keV)

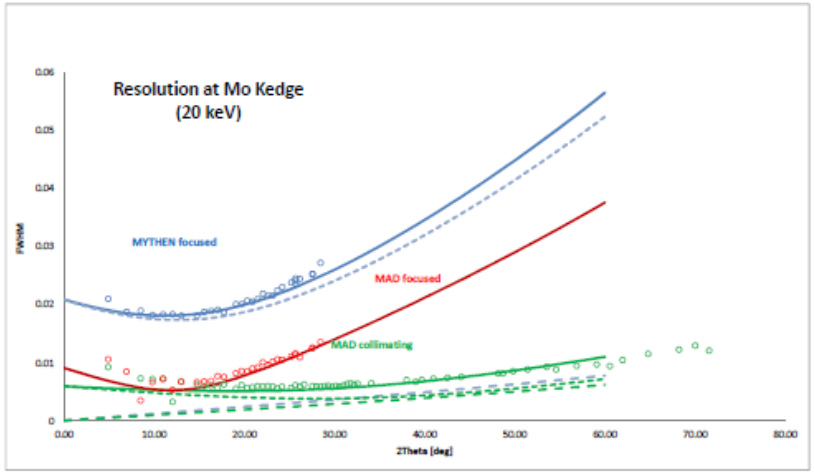
- 13 channels with 1.5 deg pitch
- Si_{111} or Si_{220} Bragg reflection
- YAP scintillator + PMT

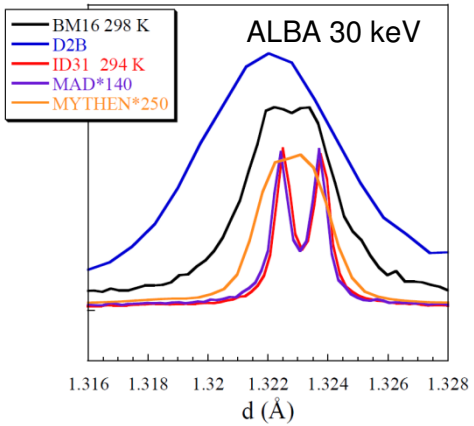
Mythen detector (7-30 keV)

- Si position sensitive detector
- 6 modules (1280 channels, 50 µm pitch)
- Dist Sample-Det = 550mm
- ms time resolution

-> ~40deg in 0.005 deg pitch

PD station : high angular resolution





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

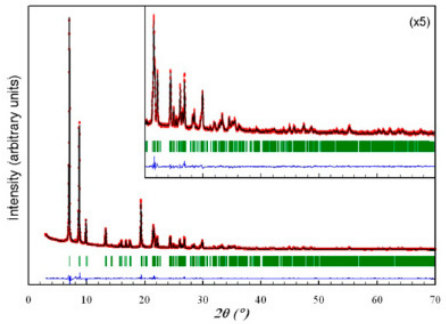
J|A|C|S JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
Communication
pubs.acs.org/JACS

A New Microporous Zeolitic Silicoborate (ITQ-52) with Interconnected Small and Medium Pores

Raquel Simancas,[†] Jose L. Jordá,[†] Fernando Rey,*[,†] Avelino Corma,*[,†] Angel Cantín,[†] Inma Peral,[‡] and Catalin Popescu[‡]

[†]Instituto de Tecnología Química (UPV-CSIC), Universidad Politécnica de Valencia−Consejo Superior de Investigaciones Científicas, Av. de los Naranjos s/n, 46022 Valencia, Spain
[‡]ALBA Light Source, Cerdanyola del Vallés, Barcelona, Spain

Supporting Information dx.doi.org/10.1021/ja411915c | J. Am. Chem. Soc. 2014, 136, 3342−3345



Why high resolution needed ?
-> small distortions
-> overlap in structure determination

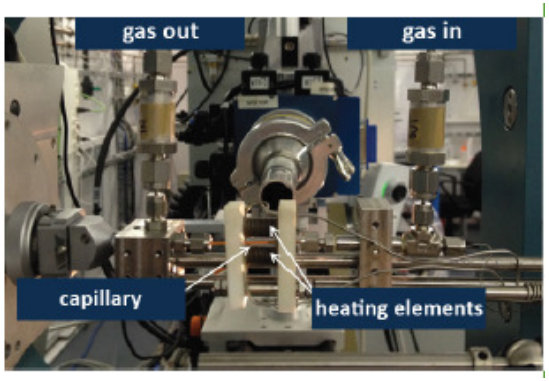
Xray lab
-> Le Bail Fit orthorhombic S.G
$a = 17.937$ Å, $b = 17.516$ Å, and $c = 12.375$ Å
-> extinction : Ima2m,I2cm, Imam, Imcm
-> no reasonable structure

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

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

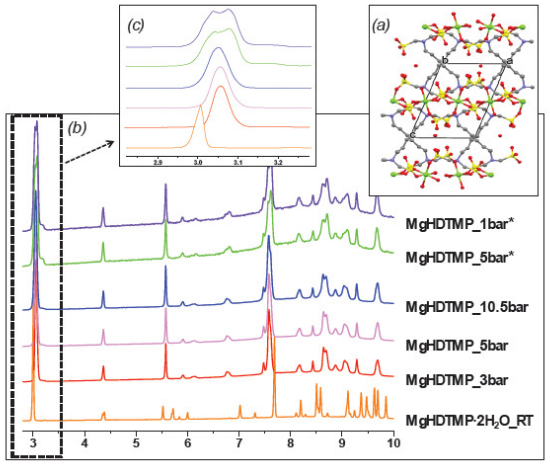
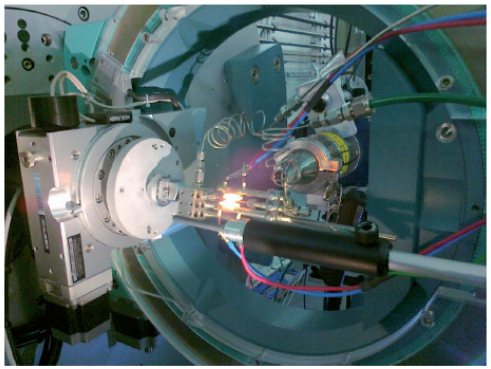


Figure 14. (a) View of the crystal structure of $MgHDTMP{\cdot}2H_2O$ along the b-axis. (b) X-ray diffraction patterns at different CO_2 pressures. The inset (c) shows the evolution of the peaks during the CO_2 adsorption-desorption. (*desorption).

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

Inma Peral

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



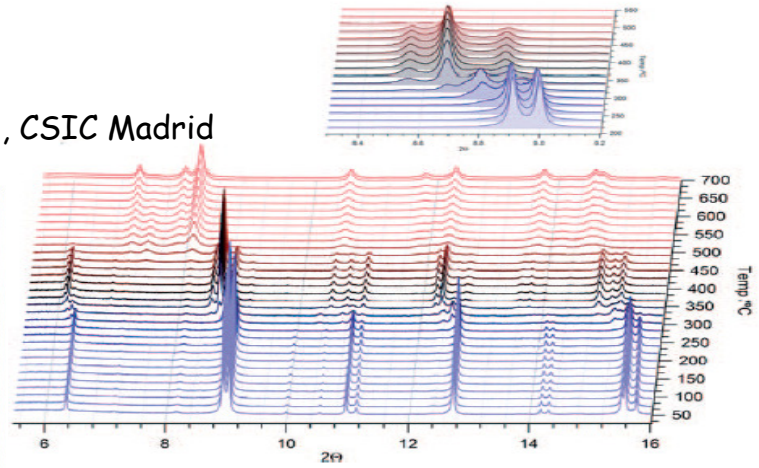
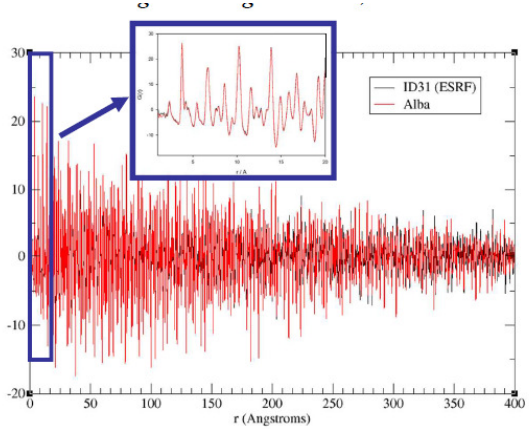


Figure 2. *Reduction of $LaCoO_3$ to La_2O_3 + Co under 5 % H_2/He gas flow. The transition is passing through a brownmillerite intermediate. Each temperature scan was collected in 8 minutes.*

$Ce_{0.625}Sm_{0.375}O_{1.8125}$ *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" acquisition times (~20 minutes). MythenII detector at Alba offers "fast" acquisition and high angular resolution (but not as high as multicrystal analyzers which explains the reduced signal at high r values).



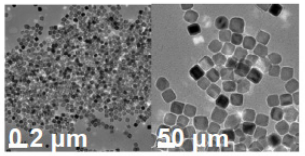
- *high energy :*
  E ~30 keV to reduce truncation error in Fourier Transformation

- *Good quality data for Rietveld, profile analysis and PDF studies*

- *high angular resolution:*
  exploiting PDF on a wide r-range

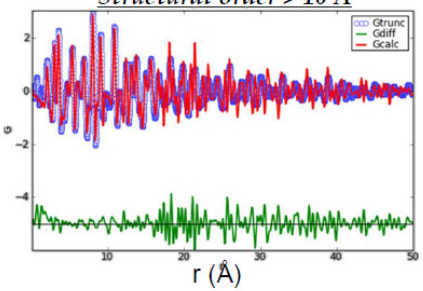
Example of Pair Distribution Function Study: Structural study on bi-magnetic core/shell magnetic nanoparticles

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



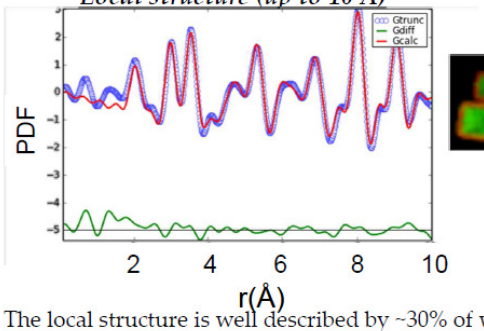
- ✓ The defects, vacancies and interface mismatch of the samples seem to have a direct influence in the overall magnetic properties of the system but little of this "disorder" is known.
- ✓ The PDF can probe different length scales

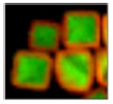
Structural order > 10 Å



- 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)

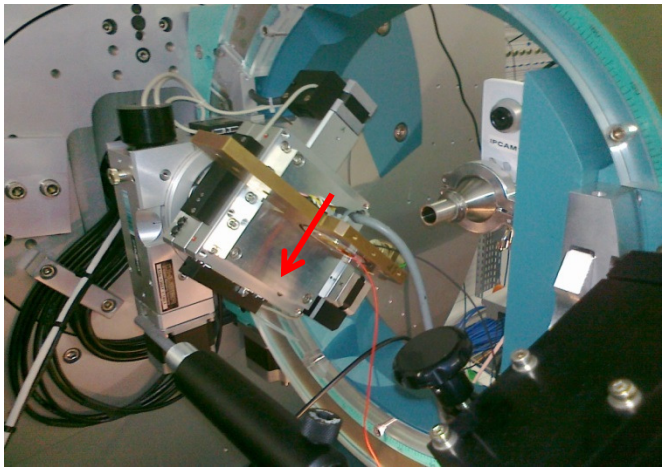




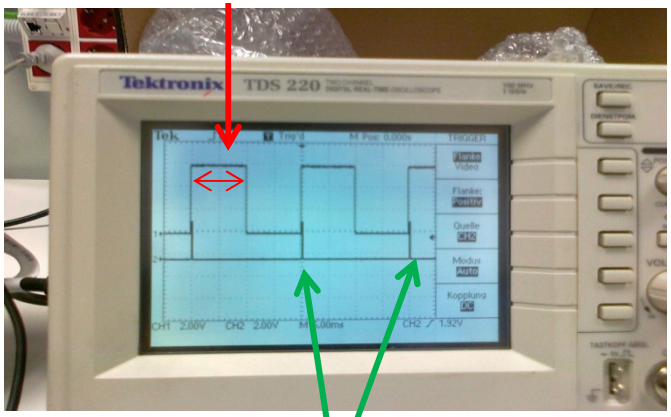
The local structure is well described by ~30% of wustite (FeO) and ~70% of magnetite (Fe_3O_4).
In agreement with the study of the Fe oxidation states by EELS (Electron Energy Loss Spectroscopy)

Stroboscopic/Pump probe experiment/fast data acquisition using the MYTHEN detector (Knapp et al, July 2103)

PZT (PbZrxTi1-xO3) Well known ferroelectric
MYTHEN 28 keV

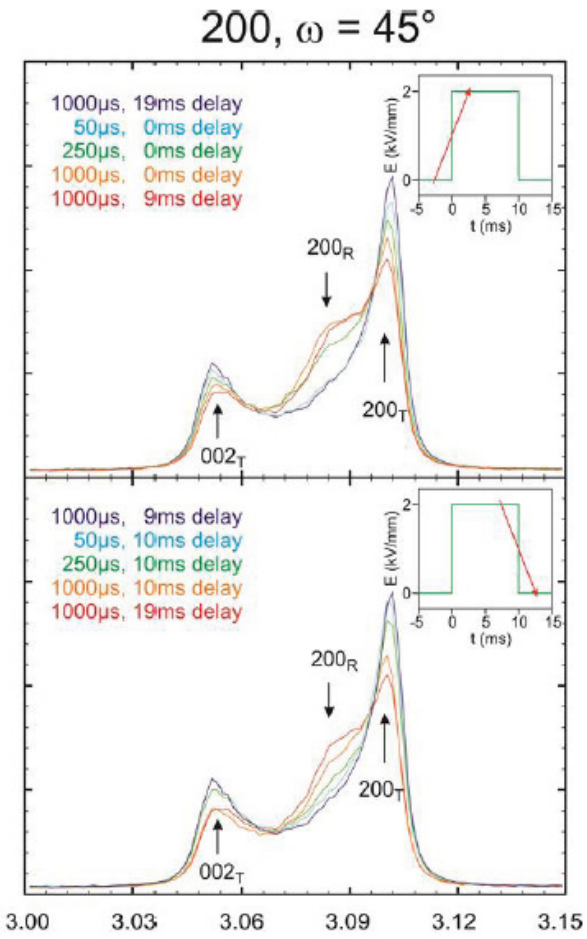


E field (10 ms gate) …
but whatever excitation possible (e.g. laser)



Data acquisition over 50-1000 µs, adjustable delay
-> statistics obtained by cumulating frames

a phase transition on the rising edge of the electric field in the range of 250µs (see the peak labeled 200R), while on the trailing edge several intermediate steps can be observed over a period of more than 1000µs. At the same time the tetragonal reflections (002T, 200T) show the opposite behaviour.



Thank you for
your attention!