CRG-IF BM 32 @ ESRF

French beamline dedicated to **surfaces** and **interfaces**

250 days of beamtime per year for science and knowledge

100 Users/year, collaborators, in-house

- *Explore and determine structure by X-ray scattering techniques inside materials and at surfaces*
- *Multiscale and Multitechniques facility*
- *In situ & operando for fundamental mechanisms and technological issues*

Expertise in Physics & Chemistry and X-ray scattering techniques
M. De Santis, G. Renaud, O. Robach, S. Tardif

High technologies and advanced instrumentation
O. Geaymond, J.-S. Micha (resp.), O. Ulrich

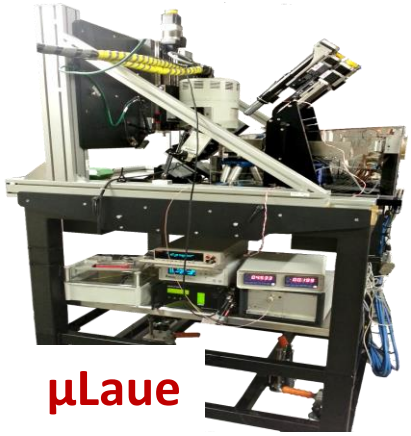
CRG-IF BM 32 @ ESRF

Nano – sciences / nano & micro-technologies
Structure / Morphology / Composition *ex situ* / *in situ* / *operando*
***of* Nanostructures / Surfaces / Interfaces**
Structure & mechanics of materials at mesoscale

GMT & µLaue

In situ
operando
ex situ

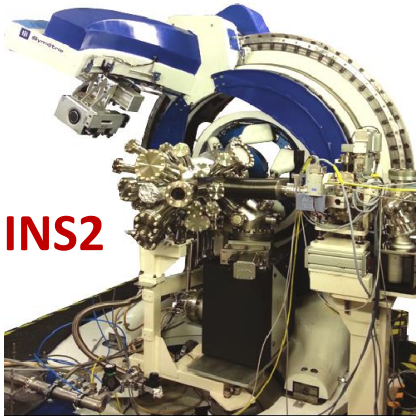
GMT



µLaue

INS2

In situ
UHV &
partial pressure



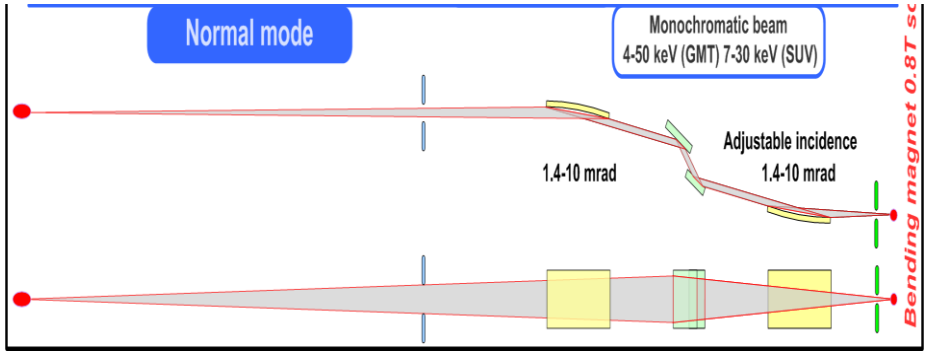
INS2

Fundamental
↔
Applied materials

Optics

2006-2018 : ESRF BM32 (0.8 T) X-ray source size : 250µm (H) x 25 µm (V)

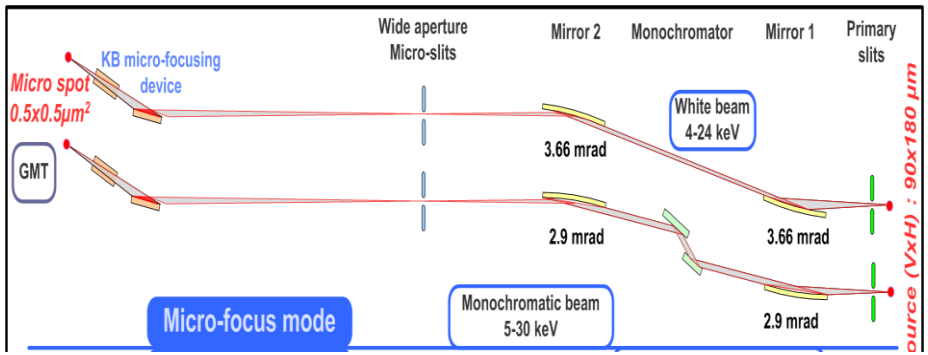
INS2 GMT



Monochromatic Mode

Beamsize 600 (H) x 300 (V) µm², 5-50 keV
5x10¹¹ ph/s, ΔE/E = 1.6 x 10-4
Divergence 1mrad (H) x 0.1 mrad (V)

µLaue



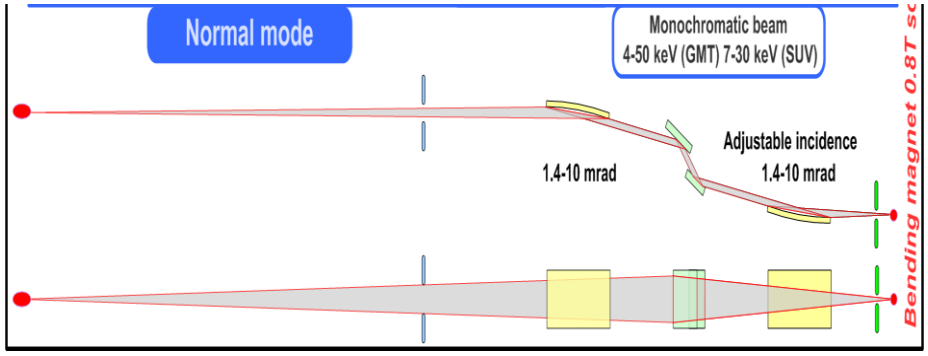
Microfocus & Polychromatic Mode

Beamsize 500 (H) x 500 (V) nm², 5 – 23 keV
10^6 - 10^7 ph/s/few eV
Divergence 0.5 mrad (H) x 1.5 mrad (V)
+ complementary Monochromatic beam

Optics

2020 - … : ESRF SBM32 (0.8 T) X-ray source size : 50 µm (H) x 5 µm (V)

INS2 GMT

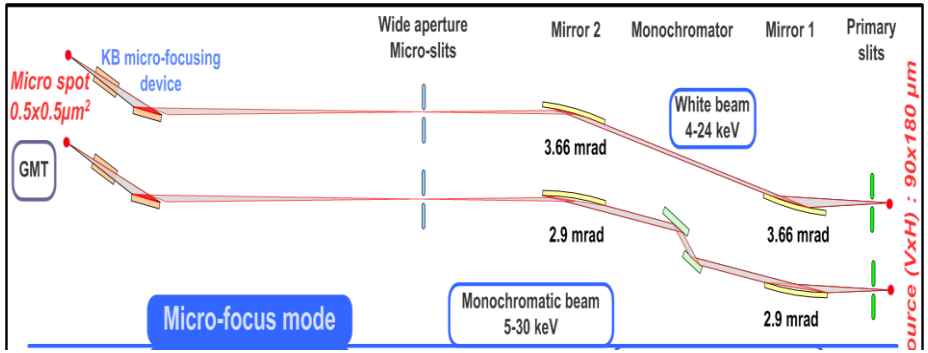


Monochromatic Mode

Beamsize 100 (H) x 100 (V) µm², 5-50 keV
$1x10^{12}$ ph/s, ΔE/E = 1.6 x 10-4
Divergence 1mrad (H) x 0.1 mrad (V)

New Ge/Si Double Crystals Monochromator => flux X 3
New horizontal 2nd crystal monochromator focusing scheme
State of the art polishing mirrors

µLaue



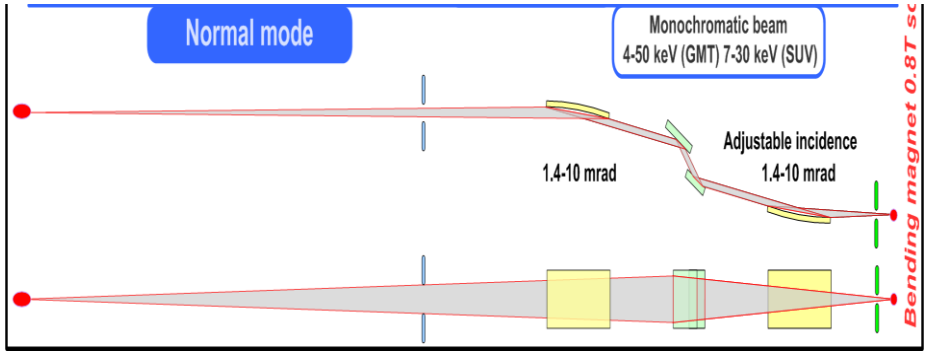
Microfocus & Polychromatic Mode

Beamsize 500 (H) x 500 (V) nm², 5 – 23 keV
10^6 - 10^7 ph/s/few eV
Divergence 0.5 mrad (H) x 1.5 mrad (V)
+ complementary Monochromatic beam

Optics

2020 - … : ESRF SBM32 (0.8 T) X-ray source size : 50 µm (H) x 5 µm (V)

INS2 GMT

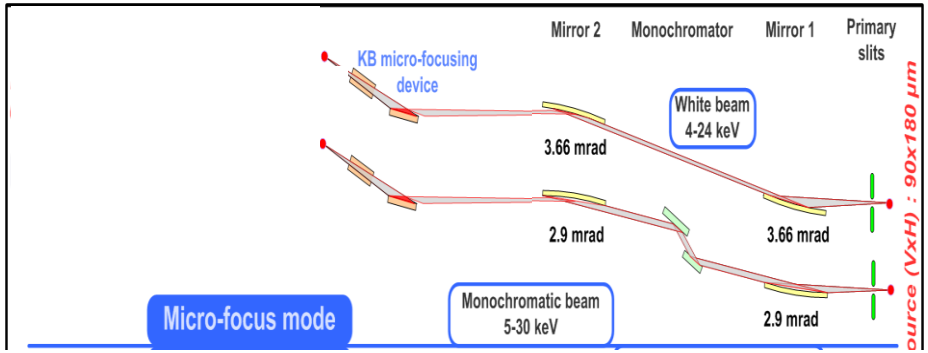


Monochromatic Mode

Beamsize 100 (H) x 100 (V) µm², 5-50 keV
$1x10^{12}$ ph/s, ΔE/E = 1.6 x 10-4
Divergence 1mrad (H) x 0.1 mrad (V)

Relocation of µLaue closer to the source => flux X 30
State of the art polishing mirrors and KB mirrors

µLaue

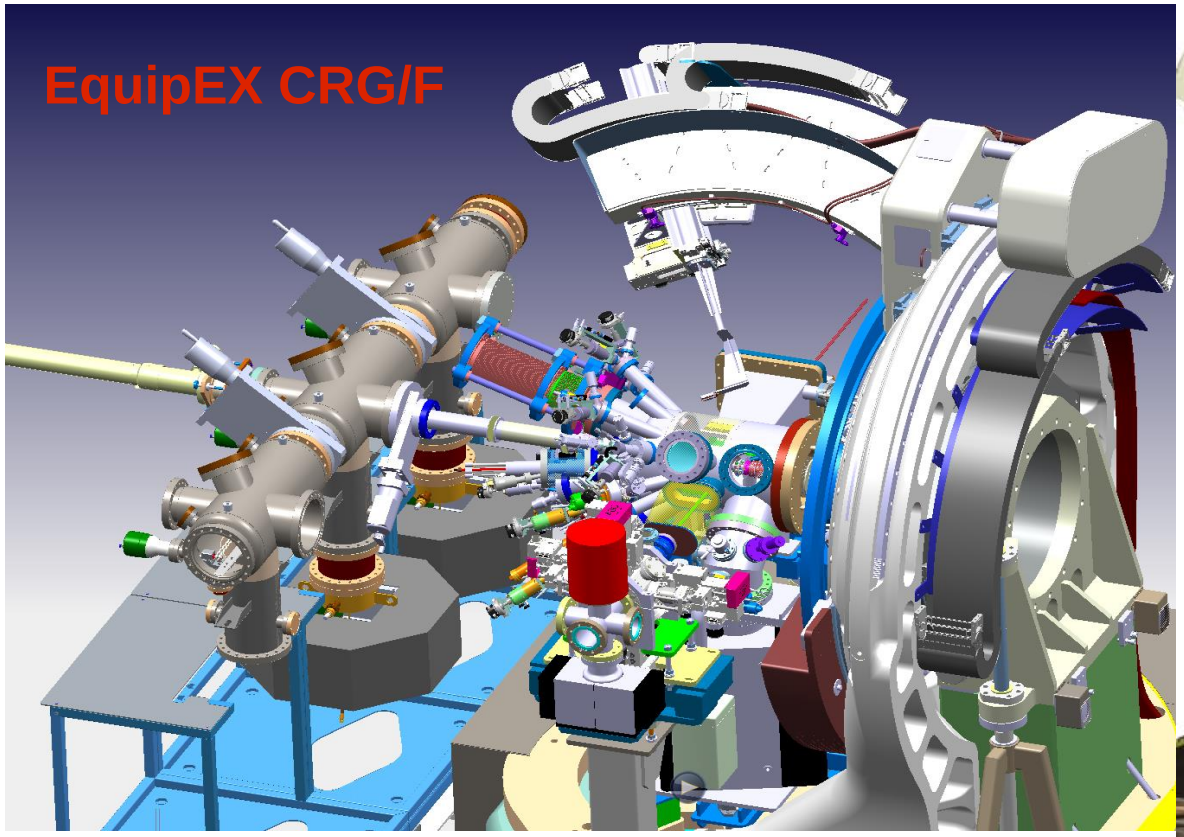


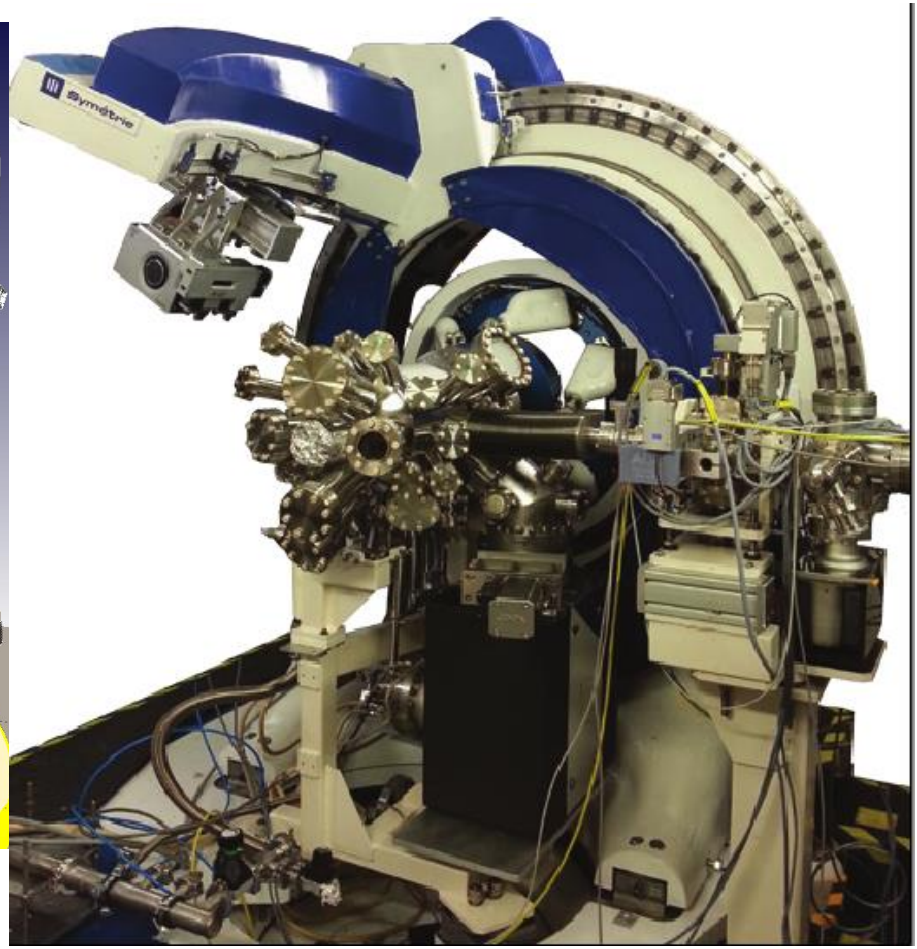
Microfocus & Polychromatic Mode

Beamsize 100 (H) x 100 (V) nm², 5 – 23 keV
$3\ 10^7 – 3\ 10^8$ ph/s/few eV
Divergence 0.5 mrad (H) x 1.5 mrad (V)
+ complementary Monochromatic beam

INS 2

New instrument for **structure**, **morphology** and **growth** under **Ultra-High-Vacuum**





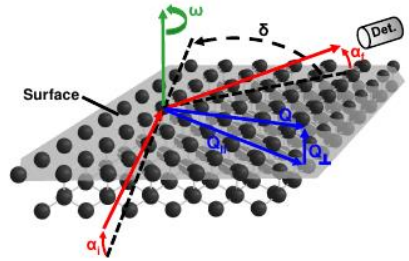
Gas injection and MBE

Open to users & collaborations: gilles.renaud@cea.fr

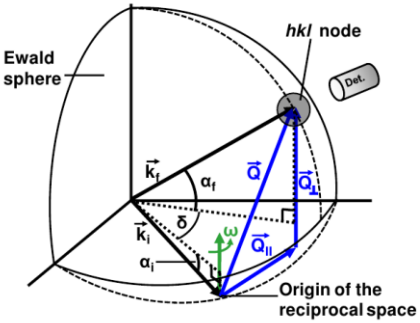
INS 2

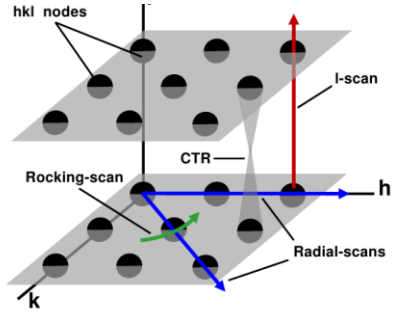
Flexible et **capabilities**:
- 10 sources + gaz: Ar, O_2,C_2H_6,...
- MBE + CVD (Si_2H_6, Ge_2H_6, H_2S)

Multitechniques:
- (MA)GIXD, (MA)SXRD, (MA)GISAXS, XRR
- Coupled GISAXS+WAXS
- in situ



Advanced Instrumentation
- 2D pixel detector
- Fast and accurate Diffractometer
- Low scattering background





Structure and growth : Graphene, clusters, complex materials, 2D materials

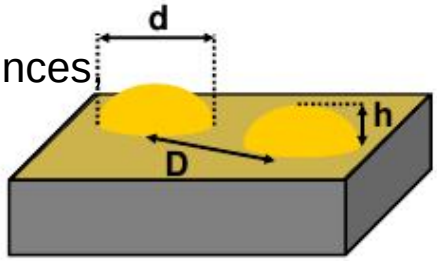
Nanoobjects: Nanowire, islands, dots

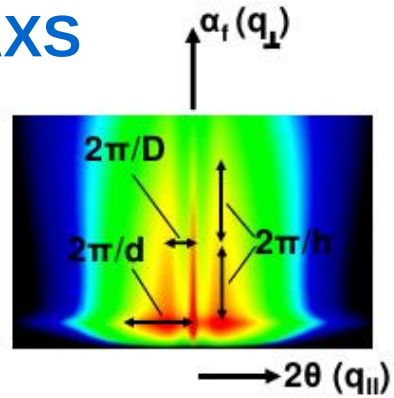
Surfaces-Interfaces: model catalysis, thin filmscouches minces, magnetic oxide, molecules / surface,

Time scale t ~ 1 sec ,

Surface diffraction & Reciprocal Map

GISAXS





GMT

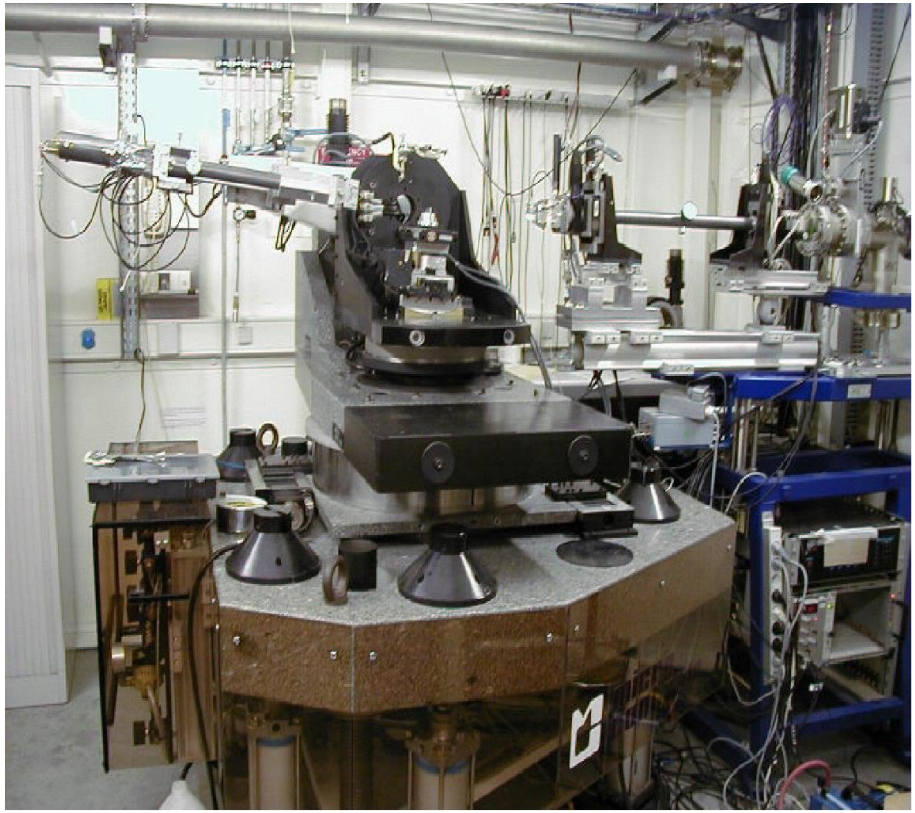
- 2+2 diffractometer with large sample environment space

- material sciences, hard condensed matter, soft condensed matter, catalysis.

- emphasis on the use of hard x-rays **(> 20 keV)** => study of buried interfaces, encapsulated samples

- a rather high amount of **technology-related** problems (Grenoble area)

- techniques panel:
Reflectivity, GISAXS, WAXS, Surface Diffraction, RSM



GMT

Solid Surfaces

Strain measurement

- in implanted materials (CEA-cadarache, INAC/LETI)
- in patterns for lithography (ST, ASML)
- semiconducting surfaces (CEA/DOPT)

Solid/Solid interfaces

- Mechanics of bonding interface: influence of roughness wavelength, terraces on bonding strength (INAC/Leti)
- Fluid penetration at the bonding interface(INAC/Leti)
- Fluid imbibition in nanochannels

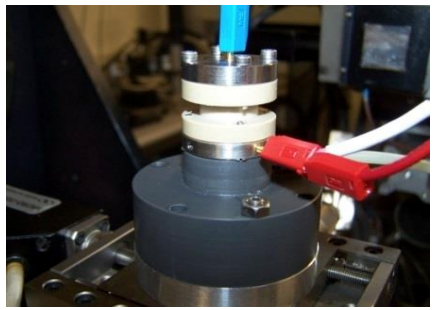
(Univ. Orléans, INAC/Leti)

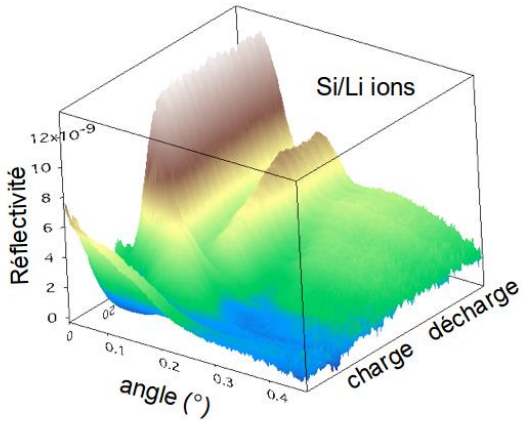
Solid/liquid interfaces

- Li batteries

FP7 Project BACCARA,
H2020 Sintbat

- Mineral surfaces (ISTerre)





GMT

Available online at www.sciencedirect.com
ScienceDirect
Procedia Earth and Planetary Science 17 (2017) 682 – 685
Procedia Earth and Planetary Science

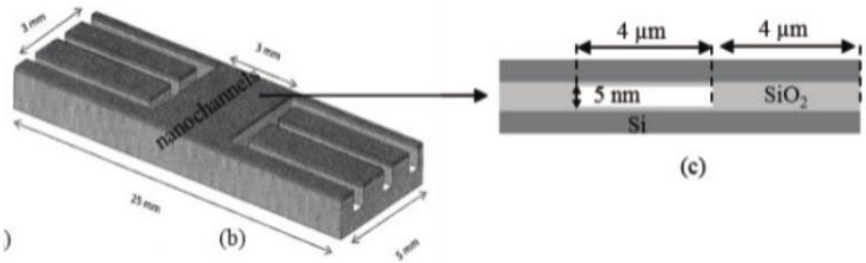
X-ray reflectivity

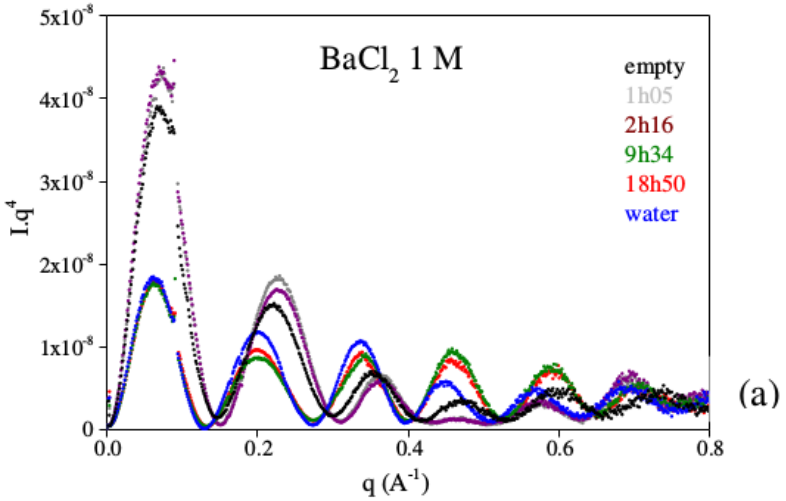
15th Water-Rock Interaction International Symposium, WRI-15

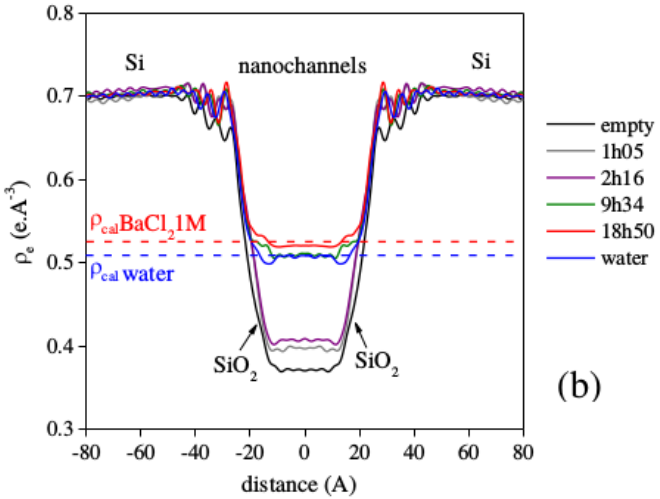
X-Ray Reflectivity analysis of SiO_2 nanochannels filled with water and ions: a new method for the determination of the spatial distribution of ions inside confined media

M. Baum[a,1], D. Rébiscoul[a], S. Tardif[b], N. Tas[c], L. Mercury[d], F Rieutord[b]

[a]CEA, ICSM – UMR 5257 CEA-CNRS-UM-ENSCM, 30207 Bagnols-sur-Cèze Cedex, France
[a]CEA, INAC, Minatec Campus, 38000 Grenoble, France
[c]a Transducers Science and Technology Group, MESA+ Institute for Nanotechnology, University of Twente, 7500 AE Enschede, The Netherlands







e- density, layer thickness, roughness

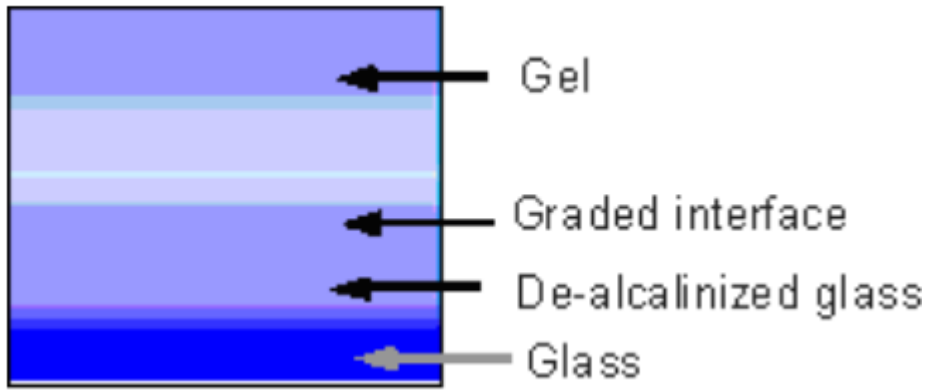
GMT

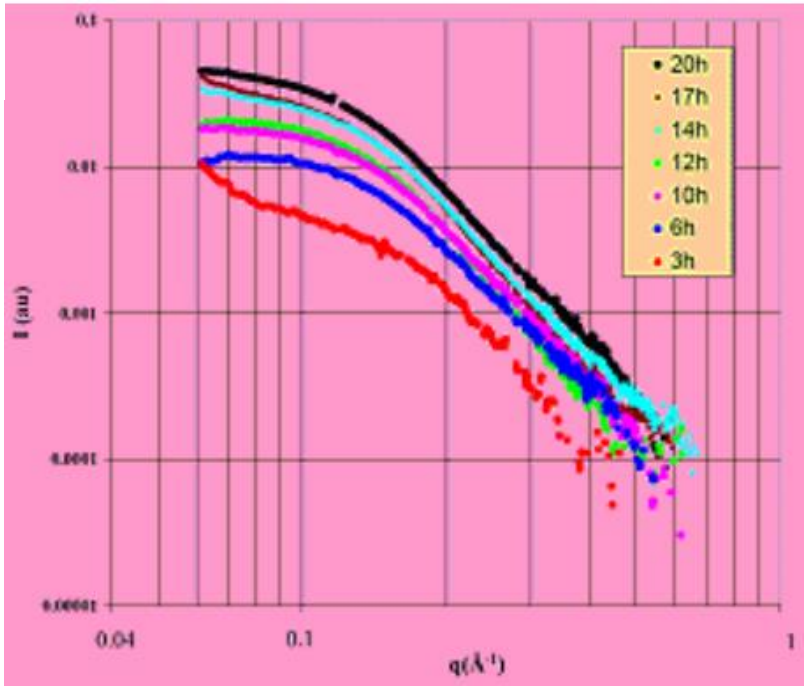
Journal of Non-Crystalline Solids
Volumes 345–346, 15 October 2004, Pages 230-233

GISAXS

A First contribution of the grazing incidence small angle X-ray scattering to the study of the corrosion of glass monoliths

L. Sicard [a], O. Spalla [a], P. Barboux [b], F. Ne [c], O. Tache [a]

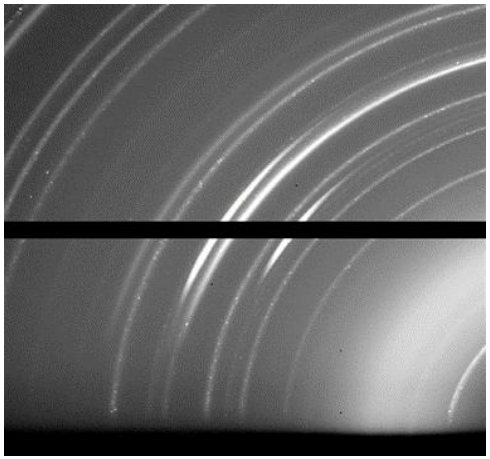




Porosity, morphology

What's next

- Scattering techniques with **hard x-rays (20-50 keV)**
  - Reflectivity
  - GISAXS, WAXS, anomalous diffraction
  - Surface diffraction, thin film



- **Large area 2D and fast detector**
  . Fast and continuous reciprocal space map
  . Small beam footprint
  . Weak signal

- **In situ & operando**
  - Environnement échantillons (T, méca., contacts e-, ...)
  - Pilotages: performants et souples, synchronisation communication
  - Visualisation/traitement de données

Interfaces solid/solid

Interfaces liquid/solid

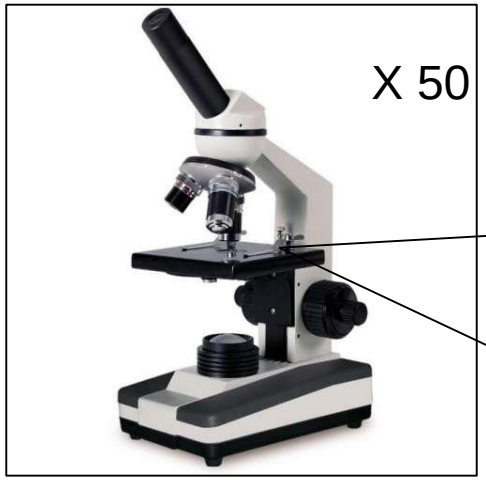
Technological materials and devices: µelectronics, energy, ...

Processes: mechanisms at surface & interfaces:
Ion-implantation, water imbibition, corrosion, adhesion & bonding, ...

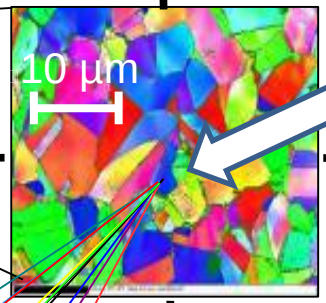
What for geosciences ?

µLaue

X-ray Microscopy



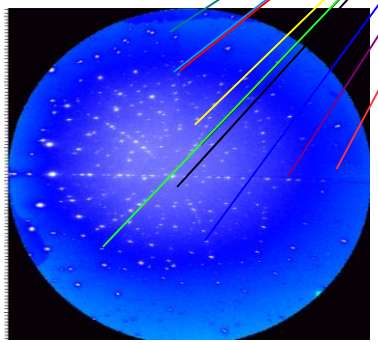
Optical microscope



µ-focused White beam

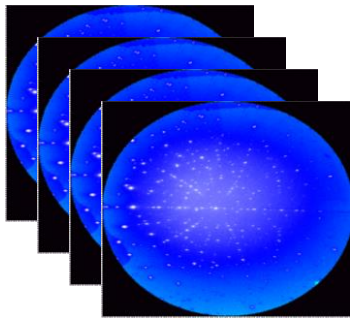
Intense
Flat spectrum
x-ray souce

scattered beams



Laue pattern

Scanning mode



Images Dataset

Analysis → **local**

- *Structure*: phase, orientation, strain
- *Morphology*: Grain position & shape
- *Crystal defects:* location & nature

µLaue

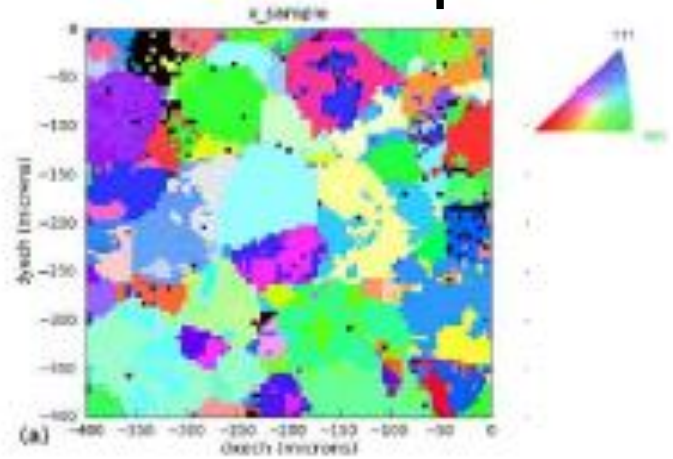
Unique in Europe

Spatially-resolved single crystal Laue diffraction

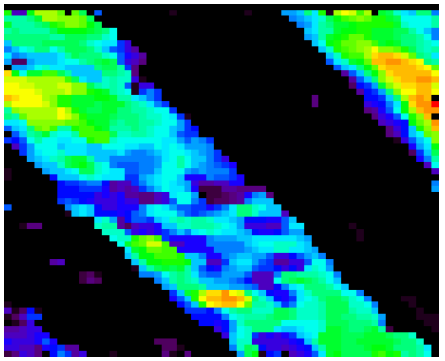
in situ &

- **2D (3D) mapping**
  Polycrystals orsingle objet
- **Selection** of single objet
- ***Ex situ and in situ***
- ***Non destructive***
- ***No preparation***
- ***EBSD-like technique***

Orientation maps



Stress/strain maps



+ LaueTools dédicated open source analysis software

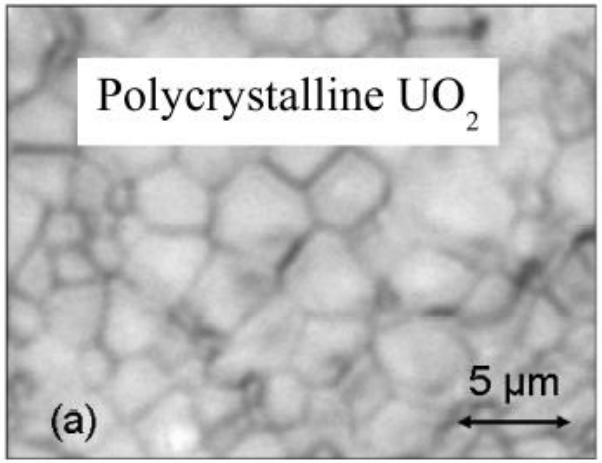
- **reliability/control and design of materials**
  micro-electronics, energy, ...
- **Mechanical properties**
  elasticity, plasticity, defect, grain boundary…

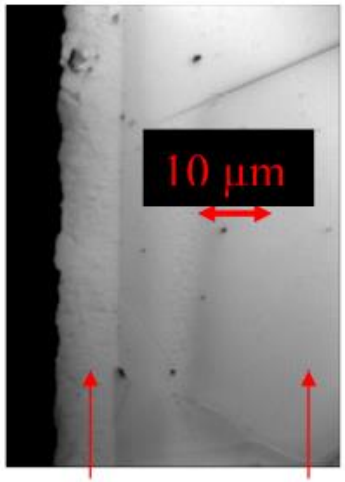
Easy mapping:
Always diffraction signal even for unknown orientation or highly deformed structure

µLaue

Visible microscope

Field of view: 200X200 to 40X40 µm²

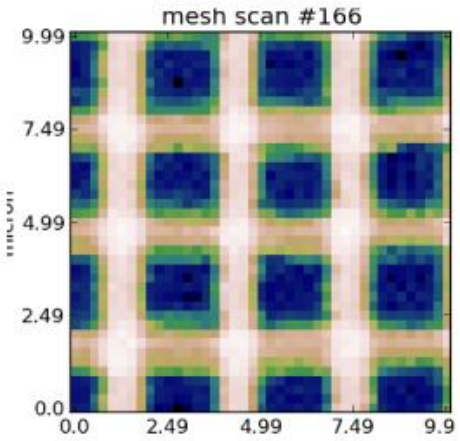




N-implanted Ni Ni

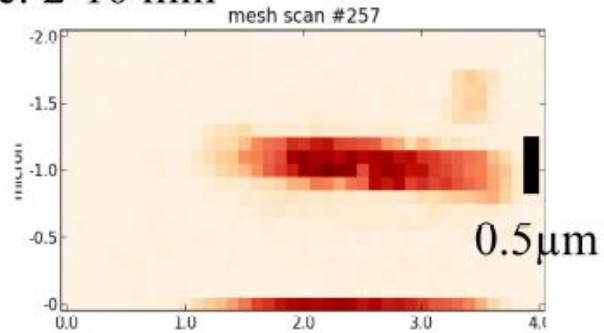
Fluorescence map

Duration time: 2-10 min

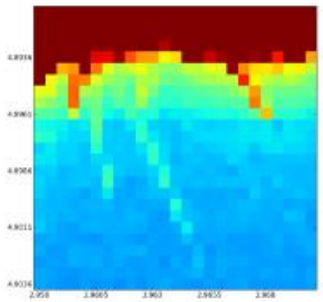


Cu pads/Si

2µm

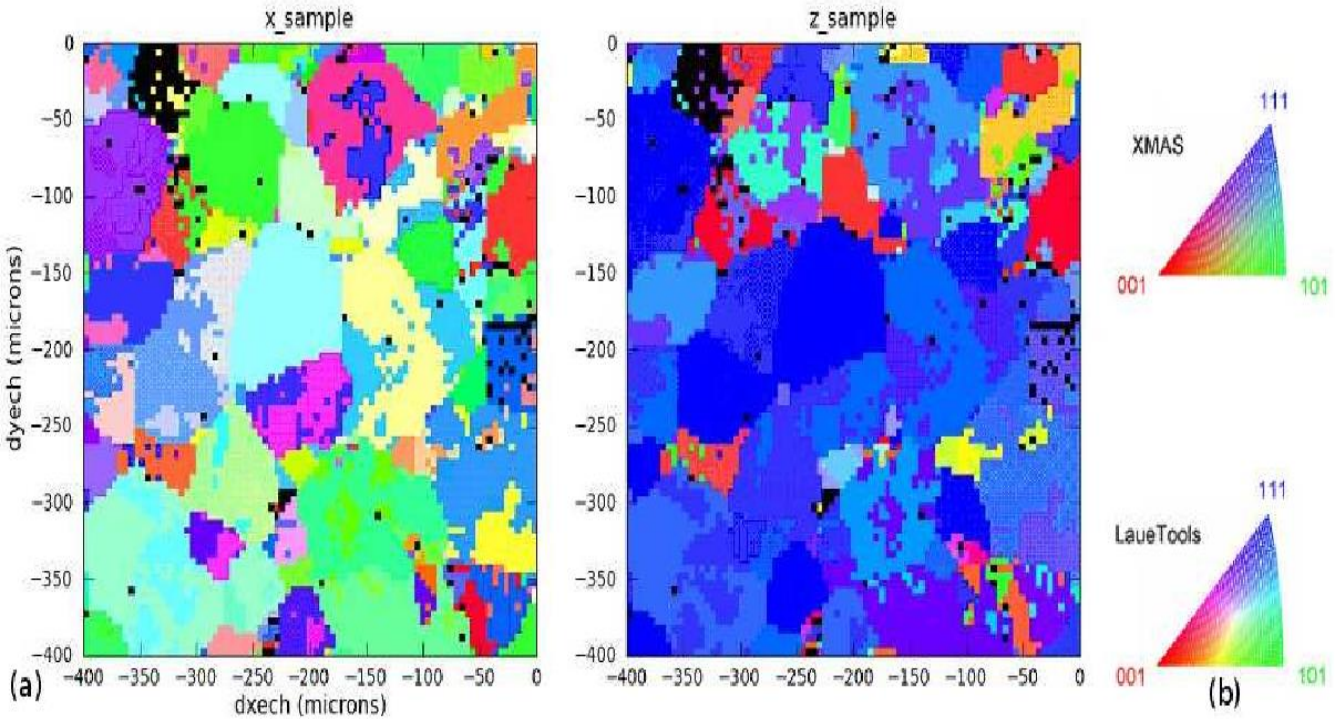


Au nanowires



- Find micro region in macro sample
- Select & memorize ROI for 1D or 2D scans

µLaue



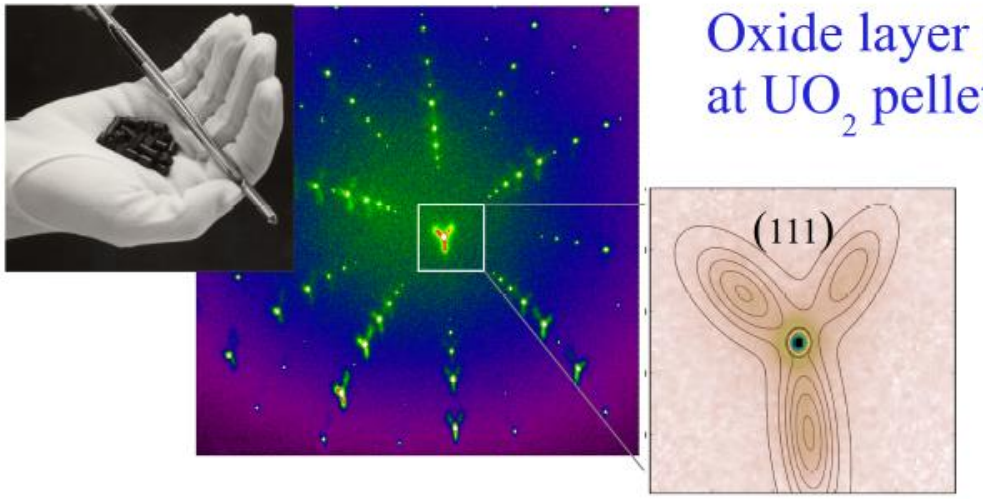
Orientation map

Strain map

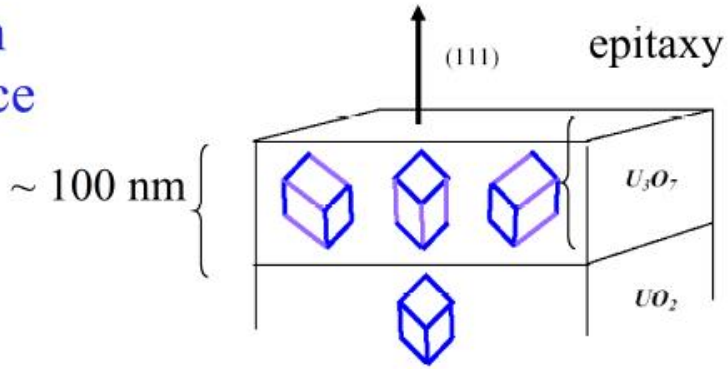
Useful to Correlate

- **scattering** – **spatial** informations
- Orientation, strain, texture, defects
- Grains size, shape, positions, phase

µLaue



Oxide layer growth at UO_2 pellet surface

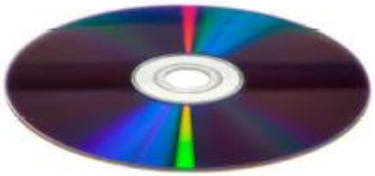


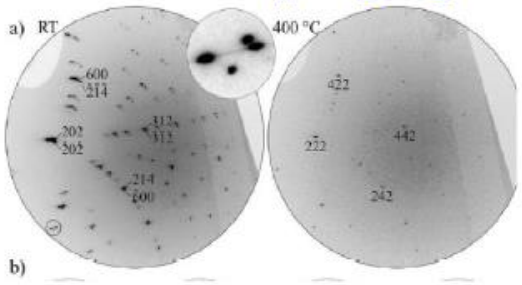
L. Desgranges *et al.*,J. Nucl. Mat. 402 p 167 (2010)

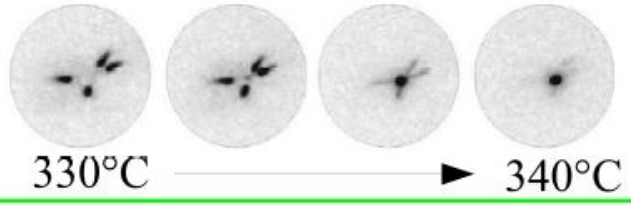
Single shot measurement :
3 variants with quadratic strain c/a ~ 1.03

In situ Phase transitions of $(GeTe)_nSb_2Te_3$ (6 <= n <= 15)

DVD





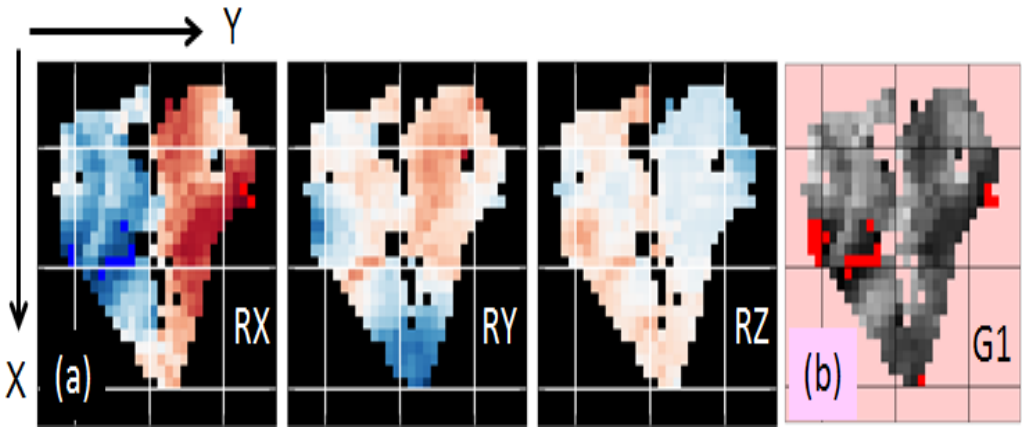


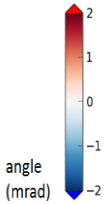
M. Schneider *et al*, Chem. Comm. 48 (16), 2192 (201

Spatially resolved study of
defects and distorted structures distribution

µLaue

Heterogeneous intra grain orientation in CdTe





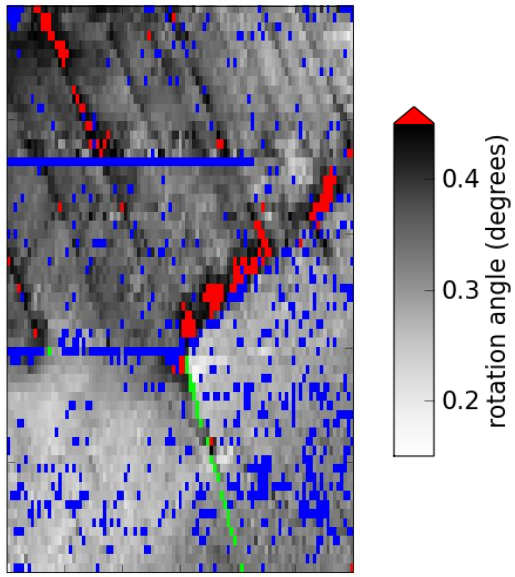
Lattice planes curvature due to defects and stress in grain or at GBs

V. Consonni *et al*, Phys. Rev. B 89 (2014) 035310

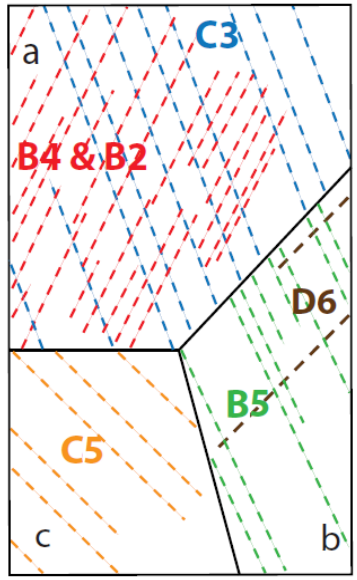
G. Daveau Thesis Ecole Centrale Paris (2012)

O. Robach *et al.*, in preparation

In situ tensile test on Cu tricrystal



Local rotation induced by 0.2% macroscopic compressive strain (two maps difference)



Slip traces as seen by optical microscope

Defects localization in **plastic** regime:
- at GB
- at dislocations accumulation

What's next

Flexible beamsize

100nm – 10 µm, poly and monochromatic beam

Combine Laue and monochromatic measurements

Sample environnement

Mechanical test, T = 77K -1700K, XEOL,…

3D µLaue

Sample depth resolution (DAXM)

Laue Tomography

Full stress tensors

Energy resolved methods, energy dispersive detectors

Coupling Laue with DIC, Laue-DIC

Local and macro strain fields, Enhance relative strain accuracy

What for geosciences ?

Conclusions

- CRG-IF and ISTerre are close

- Open to collaborations and co-developements

- 2017-2018: key years to design future beamline activities
    - higher **brilliance** ESRF-EBS
    - **opportunities** of dedicated instrumentation for **geosciences** with **EQUIPEX** to be submitted:
        - state-of-the-art **optics**
        - new diffractometer GMT + **detector**
        - μLaue **sample environment** + **variable beamsize**