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Characterization tools @ IRIG

CARAC 2020 7th edition

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NANOELEC.

Our Institute

- IRIG= Interdisciplinary Research Institute of Grenoble (IRIG)

- About 1000 people involved in fundamental research, with subjects ranging from health, biology, chemistry, cryogenics to physics.

- 5 departments and 10 labs.

- The MEM lab (within the Physics Department) groups the characterization teams (mostly in the field of material sciences)

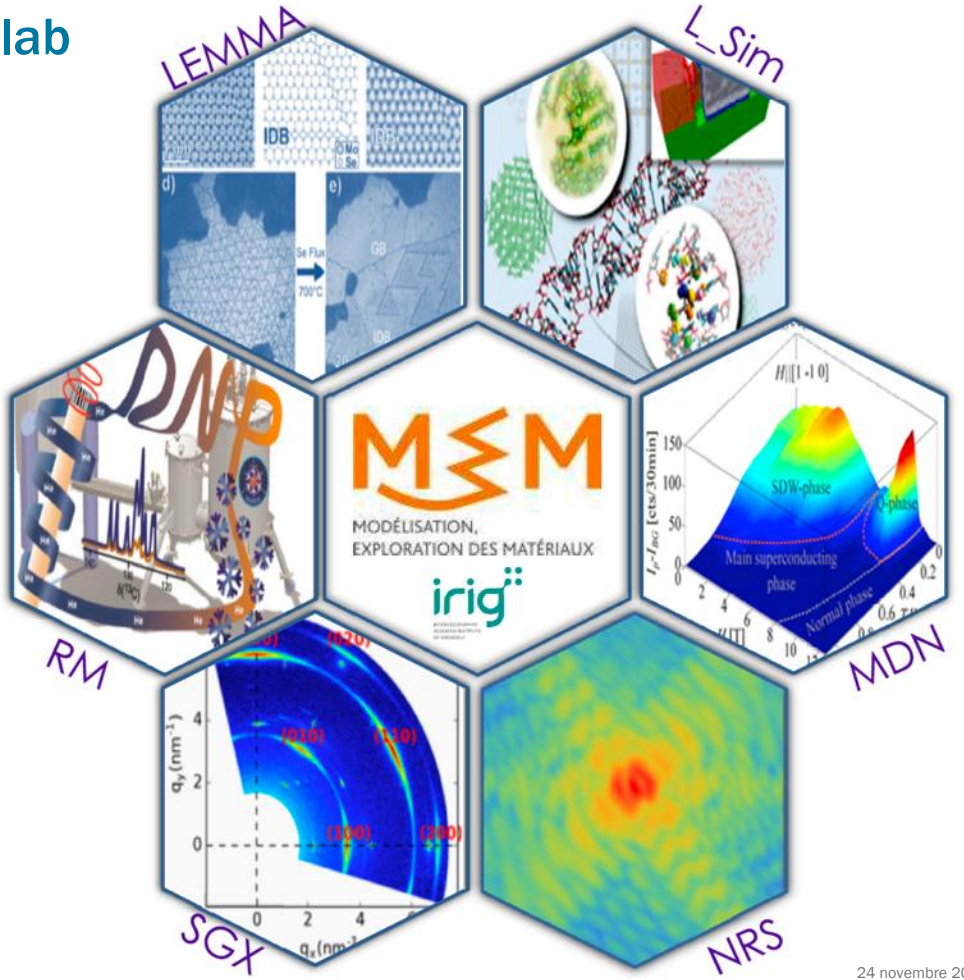
Key figures of the MEM lab

60p in 6 teams: experts in different techniques for materials characterization

- *Ab initio simulation (LSIM)*
- *Electron microscopy team (LEMMA)*
- *Magnetic resonance (RM)*

- *X-ray scattering (powder, single crystal, reflectivity (SGX)*

Synchrotron and neutron @ CRG beamlines
- *NRS: Synchrotron (grazing angle techniques, reflectivity) Strain measurements*
- *MDN: Neutrons (inelastic and magnetic scattering)*



Focus on 2 techniques where performance gains are in sight.

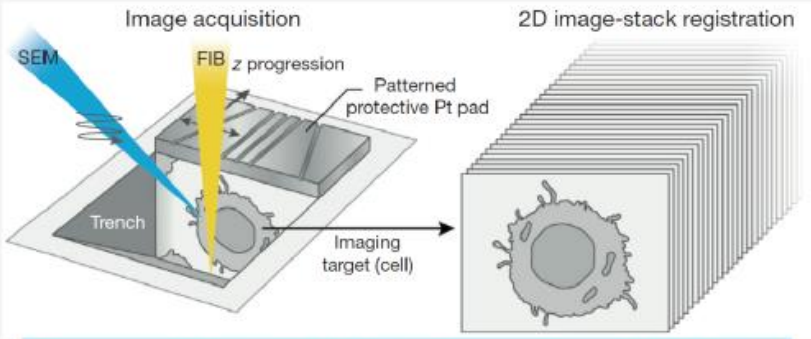
1 - FIB-SEM nano- tomography

from LEMMA team
@ PFNC (coll. LETI, LITEN and CMTC)
(PH Jouneau and coll.)

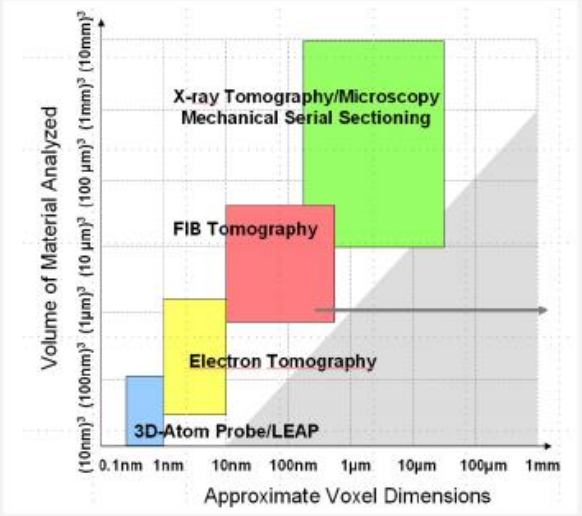
2 - Scanning MicroLaue diffraction

from NRS team
@ ESRF on a BM - CRG beamline (coll. CNRS)
(JS Micha, O. Robach, S. Tardif, O. Ulrich and coll.)

1- FIB-SEM nano-tomography: principle



The sample is cut slice by slice, and each slice is imaged, automatically



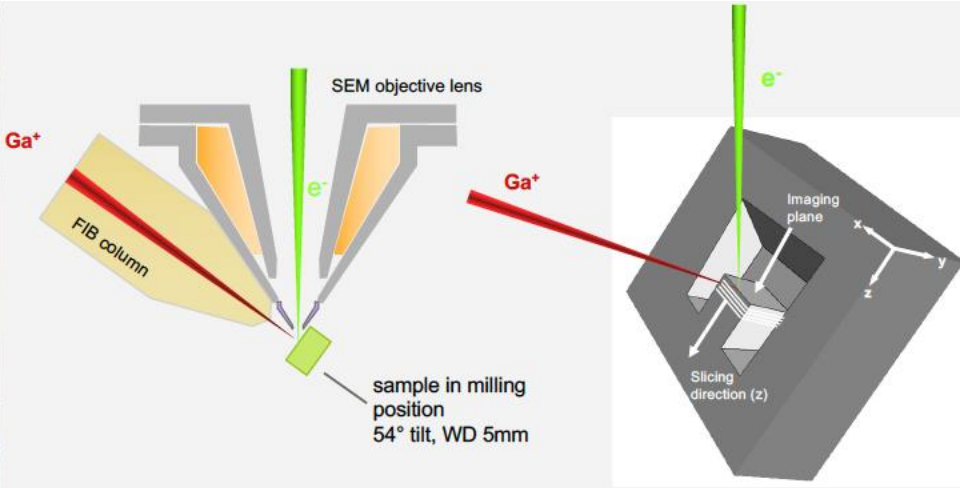
- **Acquisition conditions**
  FIB : Ga^+, 30 kV, milling current 700 pA
  SEM : **SE & BSE images at low voltage (~ 1.5 kV)** : no charging effect, good spatial resolution in BSE

- **Ultimate spatial resolution**
  x, y : **~ 2 nm** resolution in secondary or backscattered electron images
  z : Thinnest possible slice **~ 2 to 3 nm** (<< beam size)
  → 3D volumes with with iso-voxels

- **Acquisition time** : **SNR-limited collection time**
  ~ 1'-2' for each slice → **5 to 60 hours for a volume** (± automatically !)

1 - FIB–SEM nano-tomography: Instruments

Zeiss XB 550



Focused Ion Beam / Scanning Electron Microscopy

- SEM : surface imaging
- FIB : surface milling

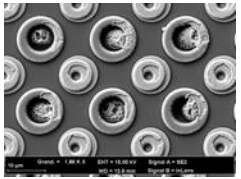
Automated iterative cycles

Several instruments @ PFNC: 3 FIB-SEM currently running

1 - FIB-SEM nano-tomography:
Applications, recent developments and perspectives @PFNC

Applications

- *Semiconductor doped layer*
- *Interconnects*
- *PV and Optoelectronic devices*



IR photodiode pixel array

Developments/perspectives

- Cryogenic sample holder (2019): Quorum sample stage
  - Reduced damage at low T
- Compressed sensing (2020)
  - Faster acquisition /Easier data handling
- Replacement of one Ga-FIB by Gallium-free sample preparation FIB (Plasma FIB?) (2021) (in collaboration with CMTC)

  Less sample damage (Ga contamination / alloying)

  Faster ion milling

2 - Scanning MicroLaue setup at ESRF

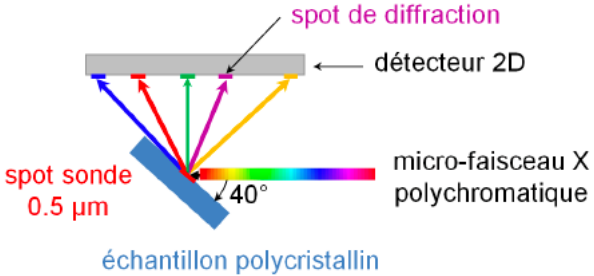
CEA-IRIG and CNRS run 5 CRG beamlines at ESRF

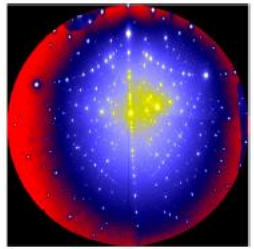
- BM or Wiggler beamlines

- Several instruments to perform X-ray scattering
or X-ray spectroscopy (XANES,EXAFS, ...)
- ESRF-> allow the use of high energy X-rays suited to
  - buried system
  - real samples, or in-situ/operando measurements with sample environment.

2 - MicroLaue setup on the ESRF CRG–IF BM32 beamline

- Principle: shine a micro(nano) multi-wavelength beam to the sample

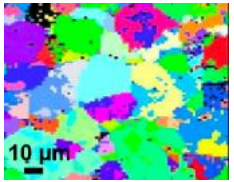




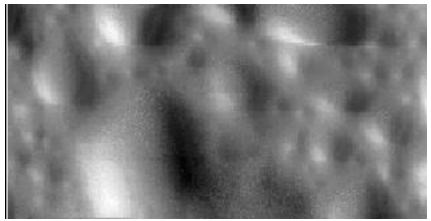
- Full use of the whole spectrum from BM-> **flux**
- No need to rotate the sample, control the rotation angle etc… -> **fast**
- Full info in one (short) exposure (full image -> all components of the shear strain tensor in one shot at one place)

2 - MicroLaue setup on the ESRF CRG–IF BM32 beamline: examples

Scanning beam/sample-> imaging ->

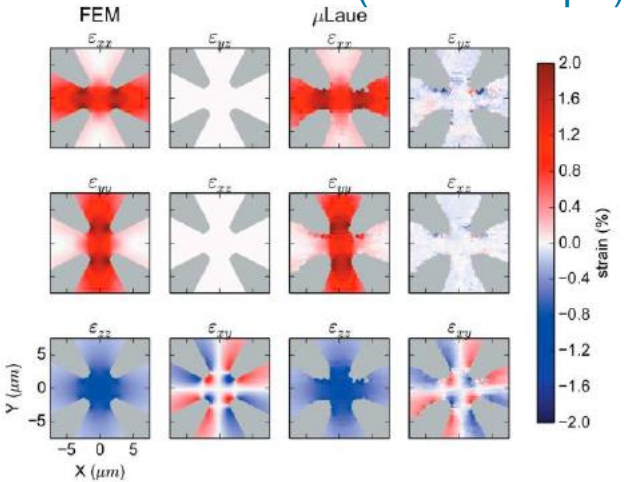


100*50µm image of microcracks (contrast: ε_x)



(coll. SOITEC)

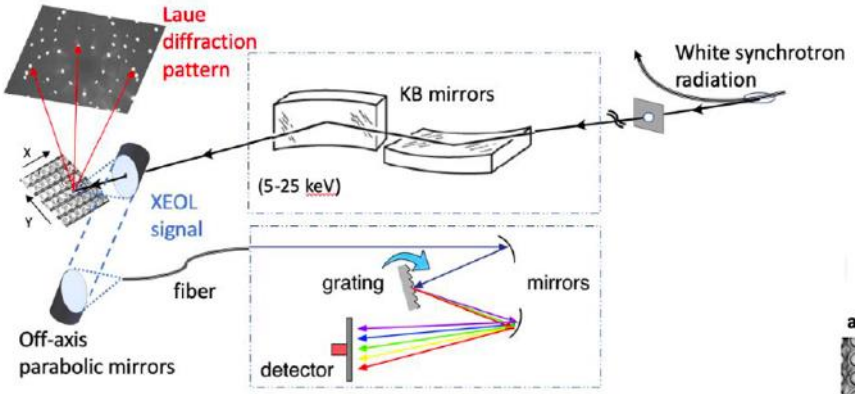
Strained Ge membrane (cross shape) of Ge



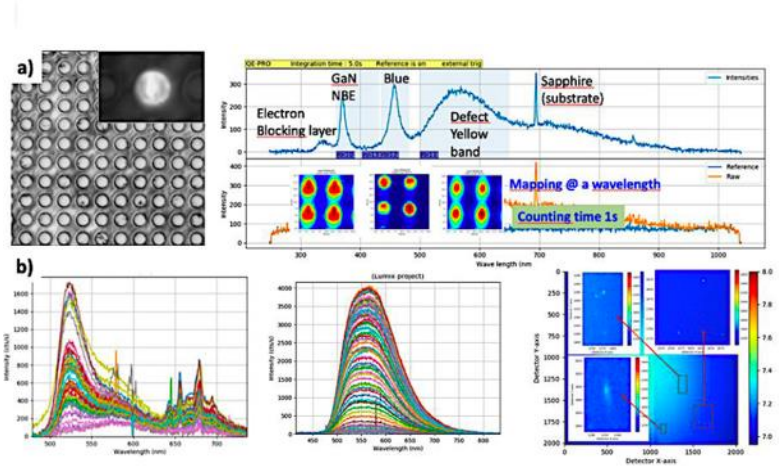
(coll. S.Tardif, V. Reboud, V. Calvo)

2 - Scanning Micro-Laue setup: example

Combine X-ray scattering with light collection



Array of GaN LED emitters



-> correlate material

structural information (strain, composition)

to

optical properties (light emission, XEOL)

(courtesy: J. Eymery)

2 - MicroLaue setup on the ESRF CRG–IF BM32 beamline

New developments:

- High speed scanning micro-Laue
- Possibility to combine with other techniques: fluorescence, XEOL, ..
- 3D mapping
- Improved resolution (down to 100x100nm) thanks to EBS
- Improved data treatment software(LaueTools, notebooks, etc..)

Thank you for your attention

Contacts:

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X-ray characterization (SGX) (single crystal, powder, SAXS etc..;)		*stephanie.pouget@cea.fr*
LSIM: Ab initio simulation DFT, etc		*luigi.genovese@cea.fr*
NRS Synchrotron : BM32. Jean-Sébastien Micha (MicroLaue)		*micha@esrf.fr* *joel.eymery@cea.fr*
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Whole LAB : Thierry Deutsch	*(thierry.deutsch@cea.fr) (Head of the MEM lab)*	
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Thank you for your attention

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