ESRF

A Light for Science

Frontiers of Characterization and Metrology for Nanoelectronics
May 13th, 2009, University of Albany, NY

Synchrotron-based nanocharacterization

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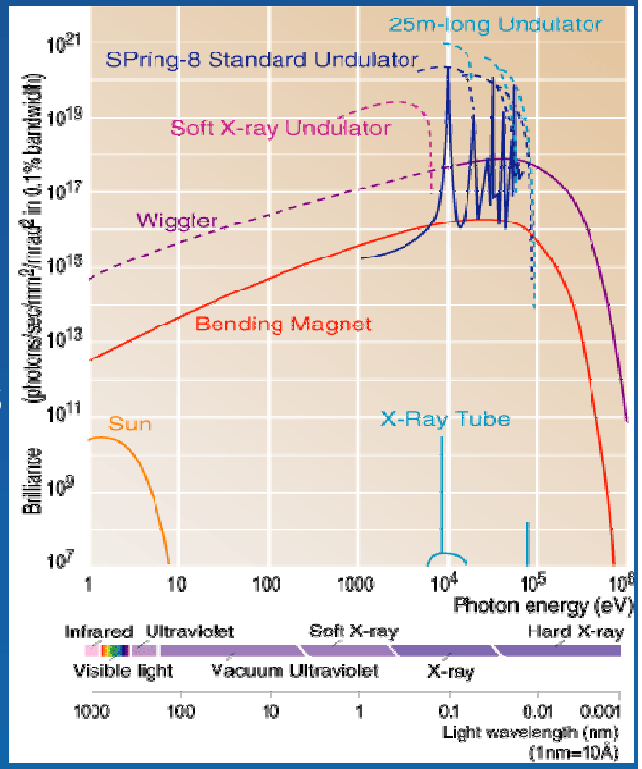
Outline

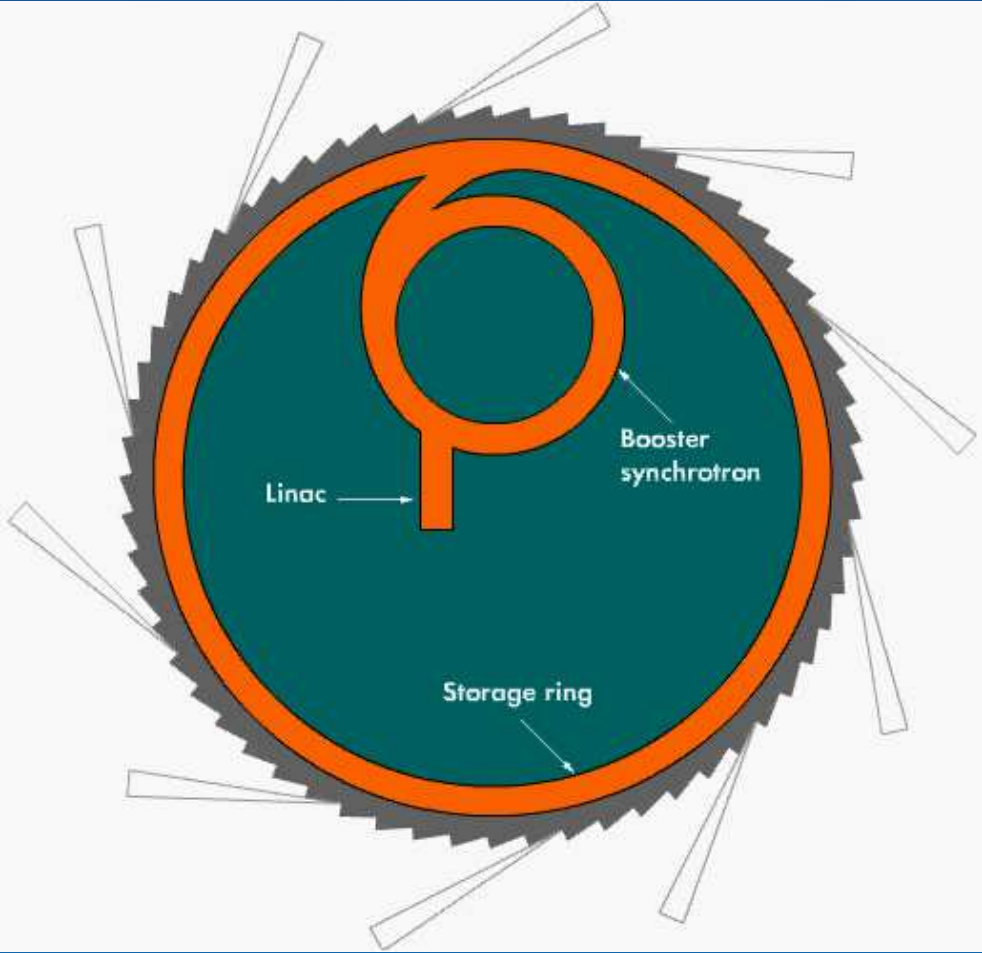
- **Introduction, Context, Background**
- **Some recent highlights**
  - Three-dimensional & multimodal imaging
  - Coherent diffraction imaging
  - Spectroscopy
  - Strain studies
- **Perspectives: ESRF upgrade**

Why this talk ?

- **Synchrotron usage**
  - Was limited to macro and micro applications
- **Advent of nanosciences**
  - Efforts to improve resolution with x-rays
  - More resolution=More photons in Smaller pixels
  - Higher brilliance
- **Go to synchrotrons !**



How does it work ?

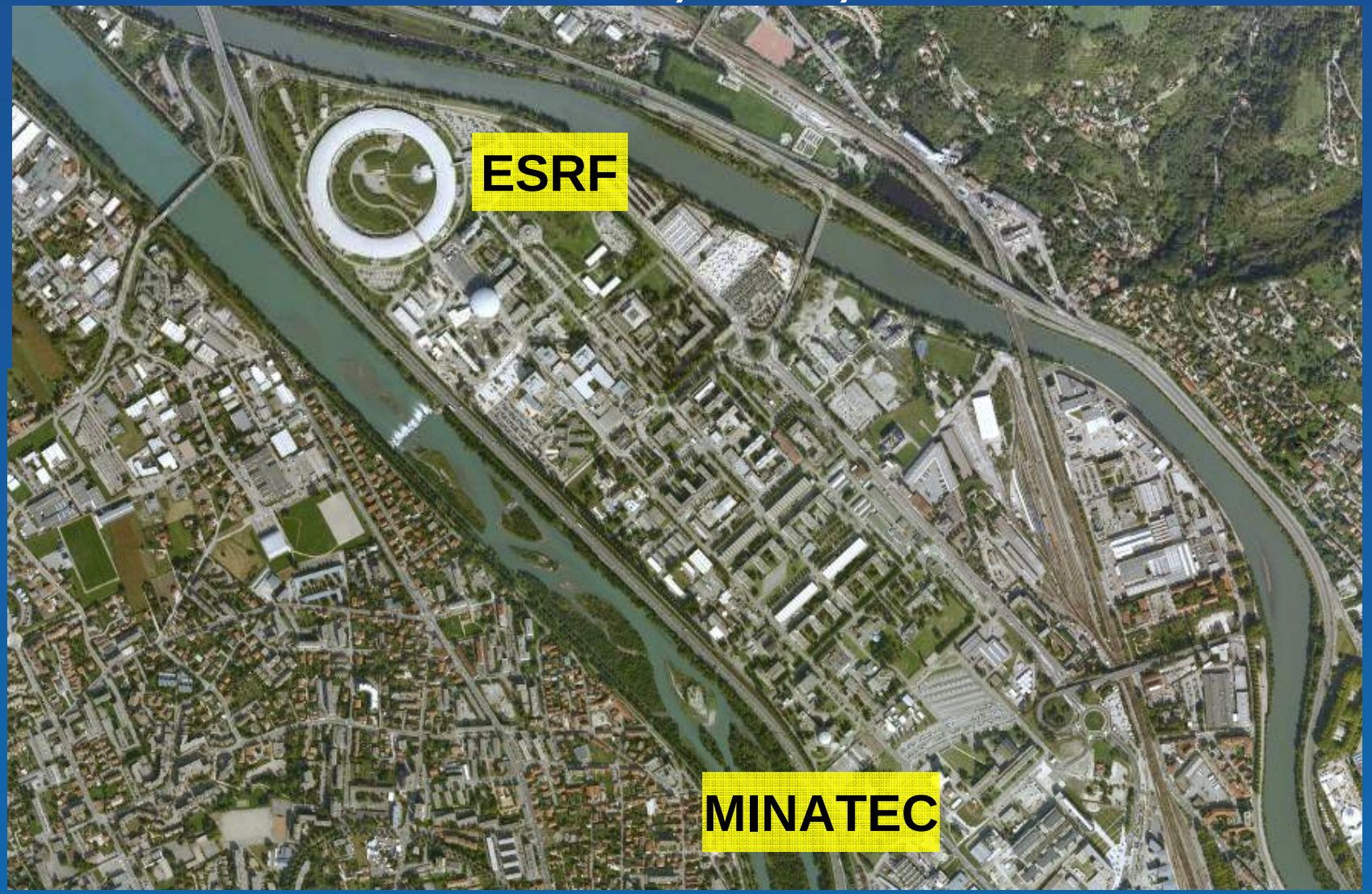


From http://www.esrf.eu

Where are synchrotrons hidden ?

Image from http://www.srs.dl.ac.uk/SRWORLD/index.html

The ESRF and CEA, LETI, MINATEC labs



What can a synchrotron give ?

X-ray fluorescence
- Composition
- Quantification
- Trace elements mapping

X-ray diffraction
- Long range structure
- Crystal orientation
- Strain, stress and texture mappings

Phase contrast
- 2D/3D morphology
- High resolution
- Density mapping

X-ray spectroscopy
- Short range structure
- Electronic structure
- Oxidation state mapping

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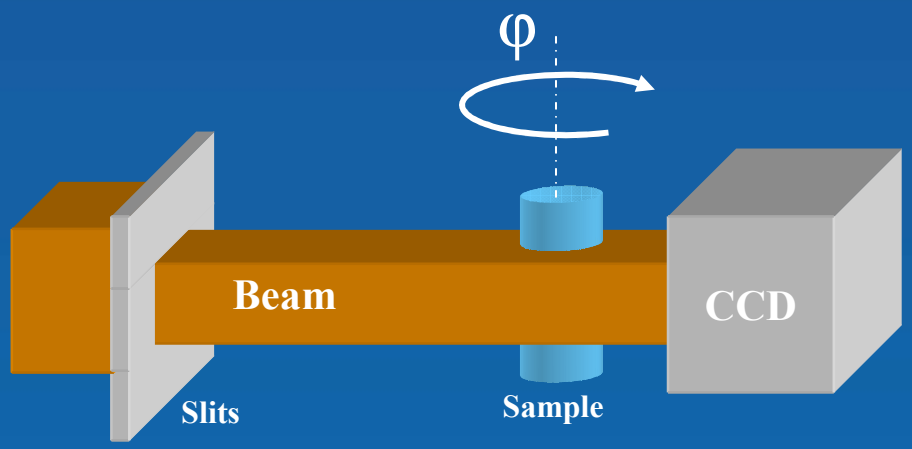
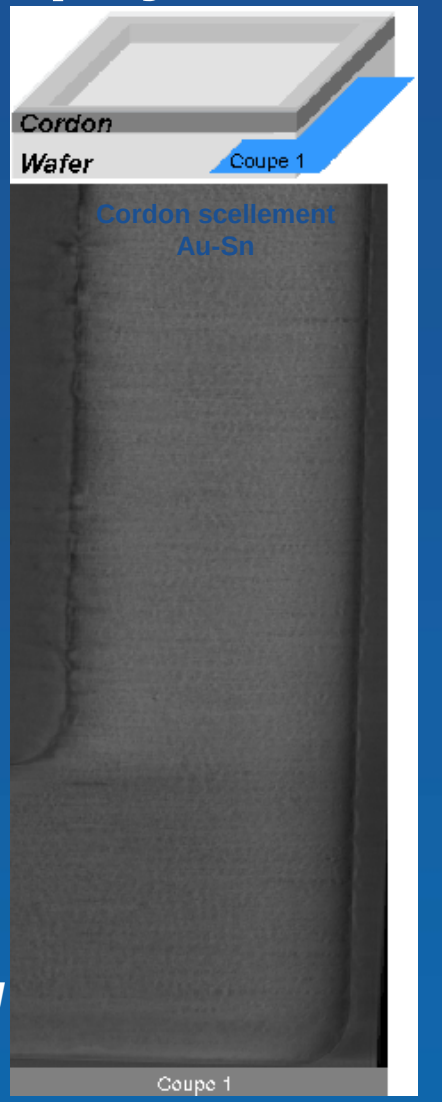
A routine technique : Microtomography

- **How it works**
  - Parallel synchrotron beam (no focusing)
  - Collection of radiographs while rotating the sample
  - Scanning time [1s, 45min]
  - Resolution ~1μm³ after 3D reconstruction





AuSn packaging
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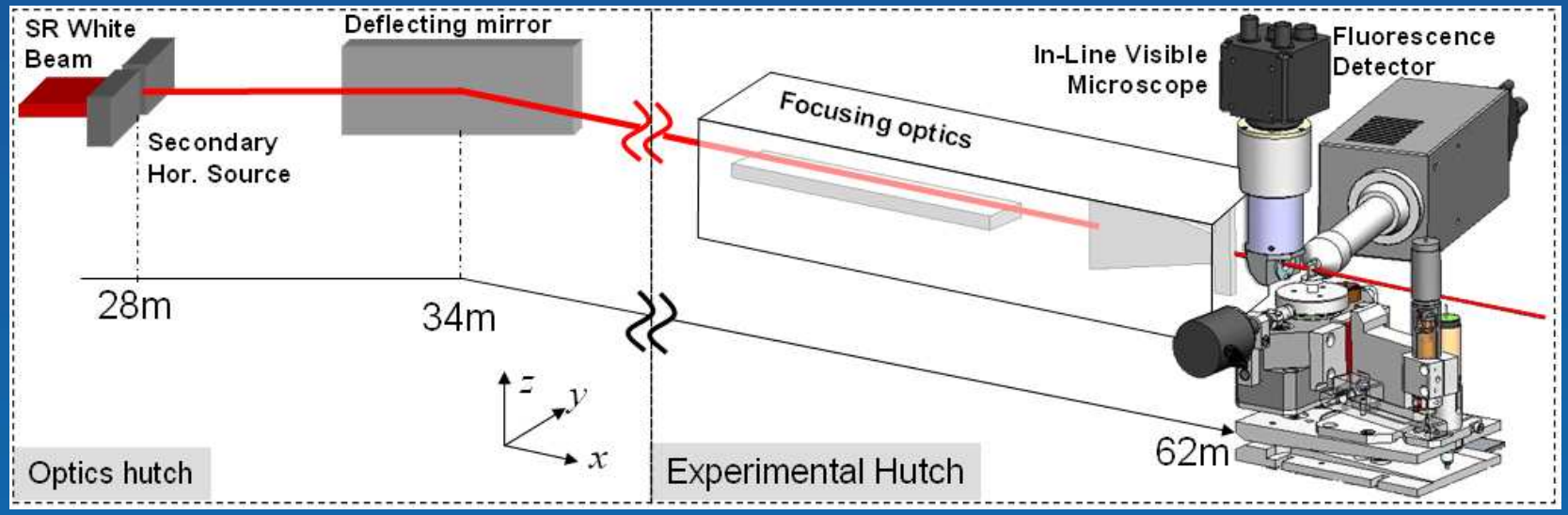
An emerging technique : nanotomography

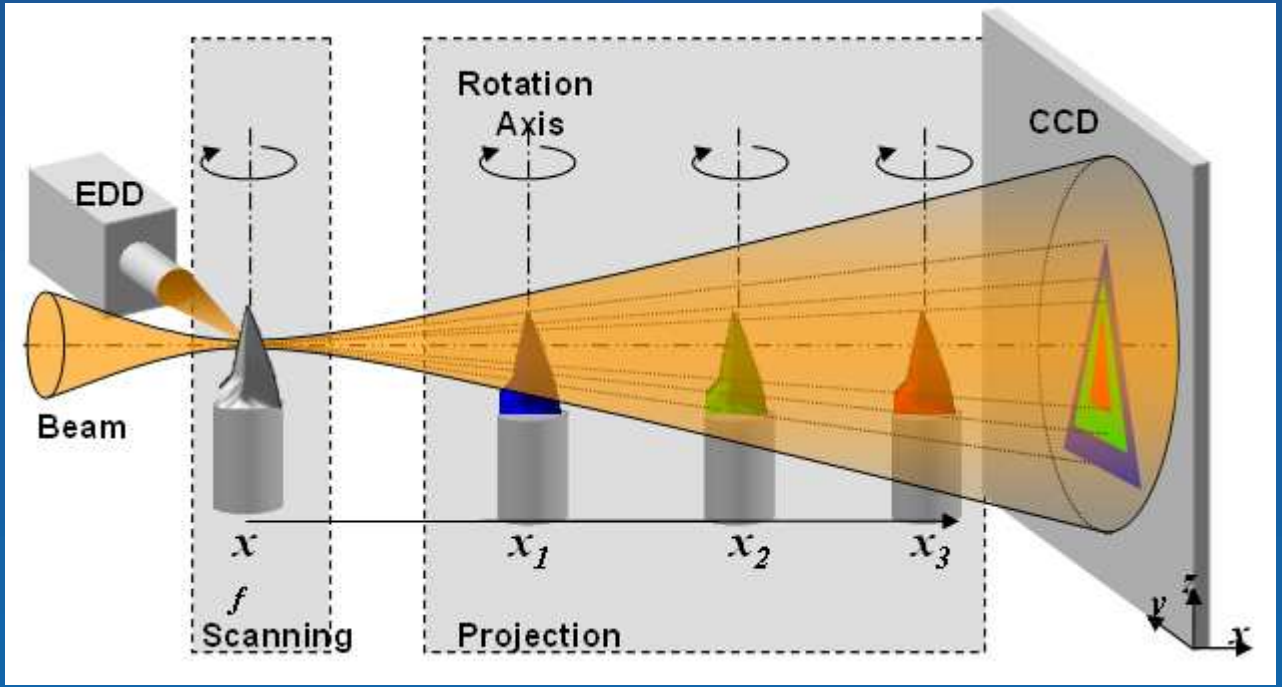
- **Goal**
  - **3D *direct* imaging at 10-20nm resolution**
  - **Hard x-rays (around 20keV)**
  - **Morphology, structural and chemical imaging**
- **Application fields**
  - **Nanodevices**
  - **Biology : cell imaging**
  - **Materials science**



Bleuet et al. App. Phys. Lett. **92**, 21311 (2008)
Bleuet et al. Nature Mat. **7**, 468-72 (2008)

An emerging technique : nanotomography

- Two kinds of microscope: scanning & projection
- Tomography → Rotation of the sample



Bleuet et al. Rev. Sci. Instr., May 2009.

An emerging technique : nanotomography

- **Assessment of the technique on a well-known object**
  - AFM (Pt, Ir) tip

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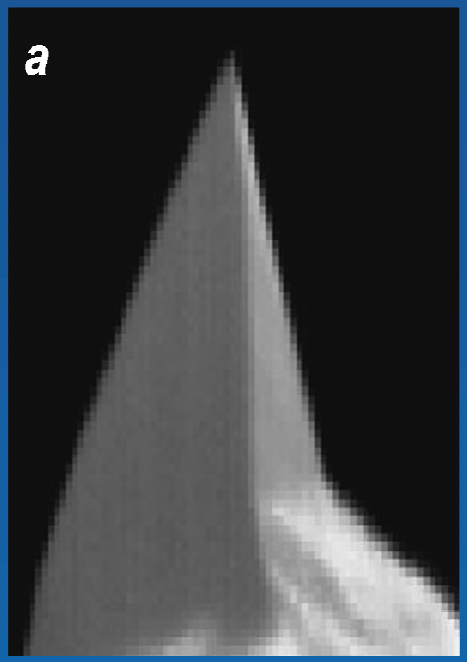


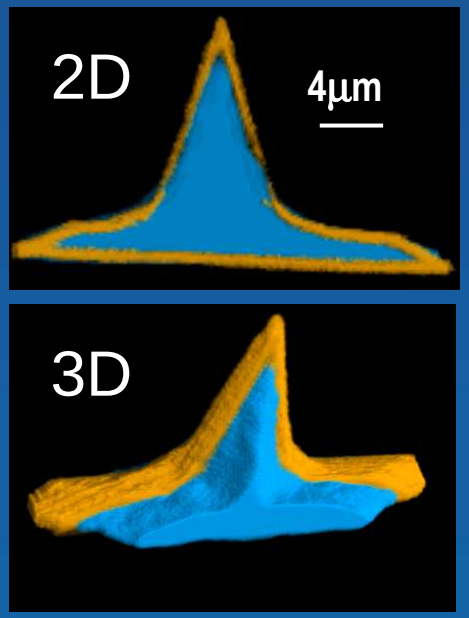
2D Fluorescence mapping (Pt signal)

SEM micrograph (provided by Nanosensor Company)

Bleuet et al. Rev. Sci. Instr., May 2009.

An emerging technique : nanotomography

- **Assessment of the technique on a well-known object**
  - AFM (Pt, Ir) tip

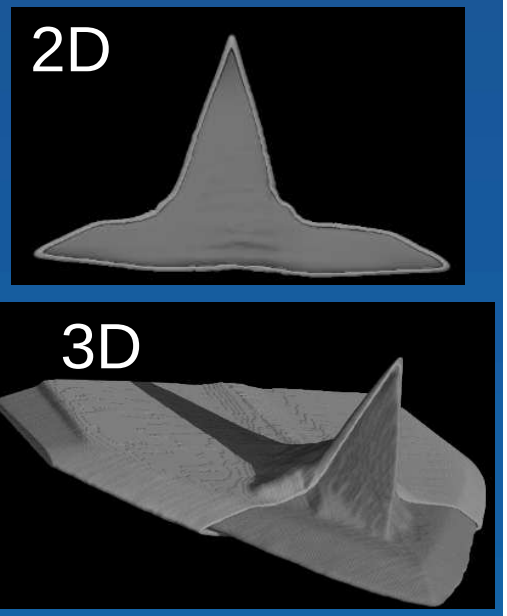


Si

Pt

Low density

High density



Fluorescence tomography
(scanning tomography)
Chemically sensitive

Holotomography
(projection tomography)
Density sensitive

Bleuet et al. Rev. Sci. Instr., May 2009.

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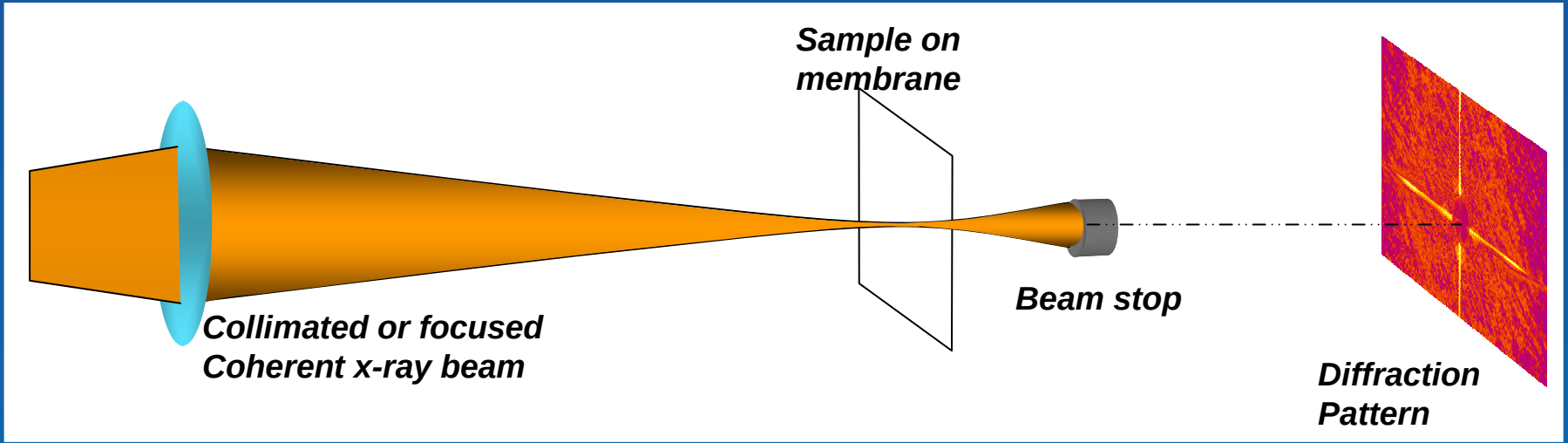
- **Perspectives: ESRF upgrade**

Pushing the spatial resolution limits

- **High resolution *direct* imaging method**
  - Are all based on x-ray optics
    - Resolution limited by aberration and diffraction to d>0.61λ/ NA
- **Switch to lensless imaging using coherent diffraction**

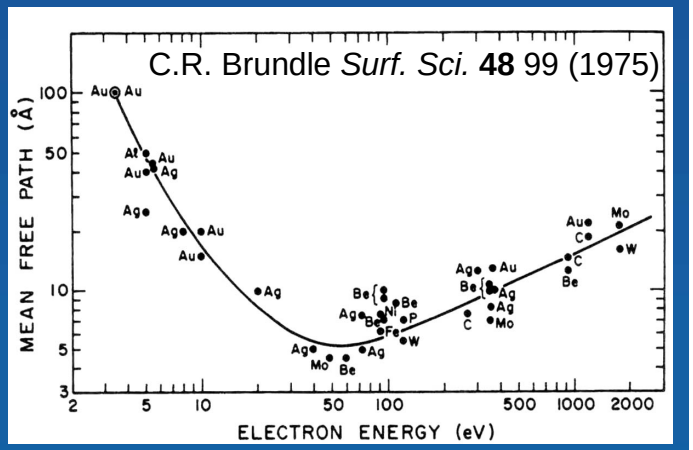


- **Inverse problem: find back the phase and perform inverse Fourier Transforms**
  - Ends up with “radiographs”

Back to 2D surface analysis

- **Exciting with X-rays and measuring outgoing x-rays**
  - Depth-resolved imaging
- **Exciting with x-rays and measuring outgoing photoelectrons**
  - Surface imaging (first 10nm)

→**X-ray Photoelectron Spectroscopy (XPS)**
  - Low Z elemental composition
  - Chemical state/binding
  - Performances
    - Detection limit (in size): 5nm
    - Spatial resolution : 450nm



- **Poster WE-025**
  - "*X-Ray Photoelectron Spectromicroscopy of Doped Silicon Patterns*"

X-Ray Photoelectron Spectroscopy principle

X-ray source

Laboratory source @ MINATEC

Synchrotron Radiation

$$E_B = E_0 - E_k$$

Spectrometer

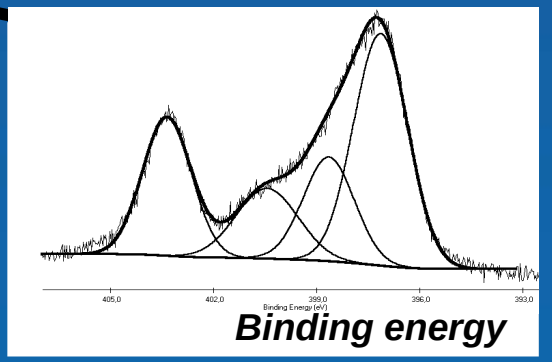
Incoming monochromatic beam E_0

Photoelectrons E_k

10nm

Sample

Few 100µm to few mm



Binding energy

Extending XPS to X-ray Photoelectron Emission Microscopy

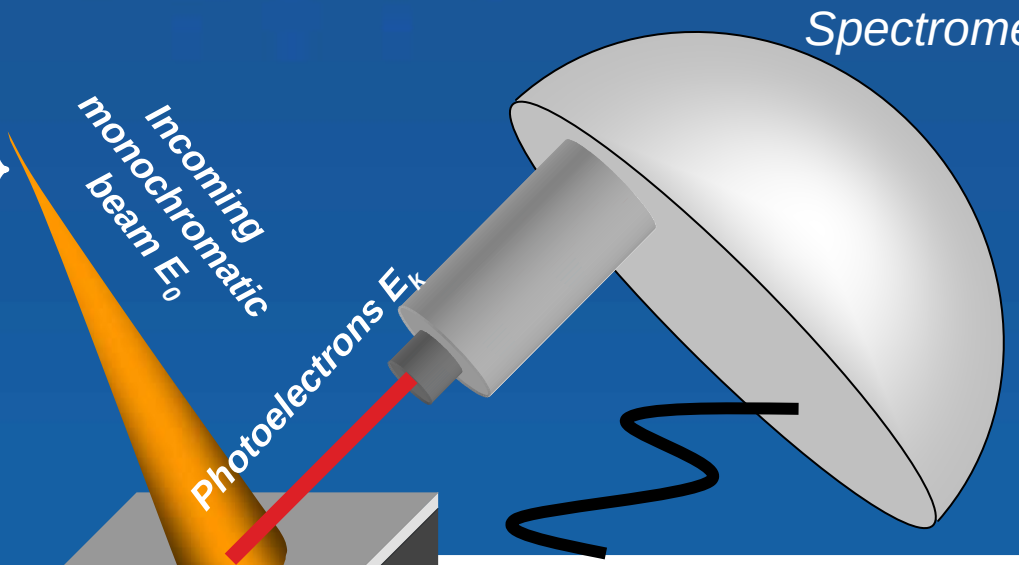
X-ray source

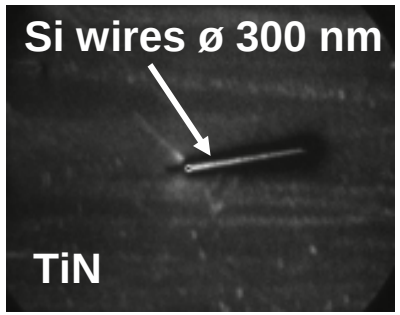
Laboratory source @ MINATEC

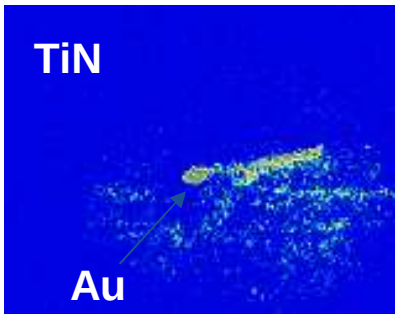
Synchrotron Radiation

XPEEM

Coupling a microscope to the spectrometer







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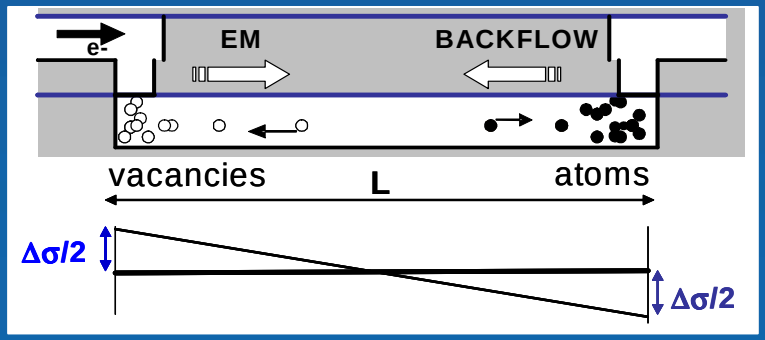
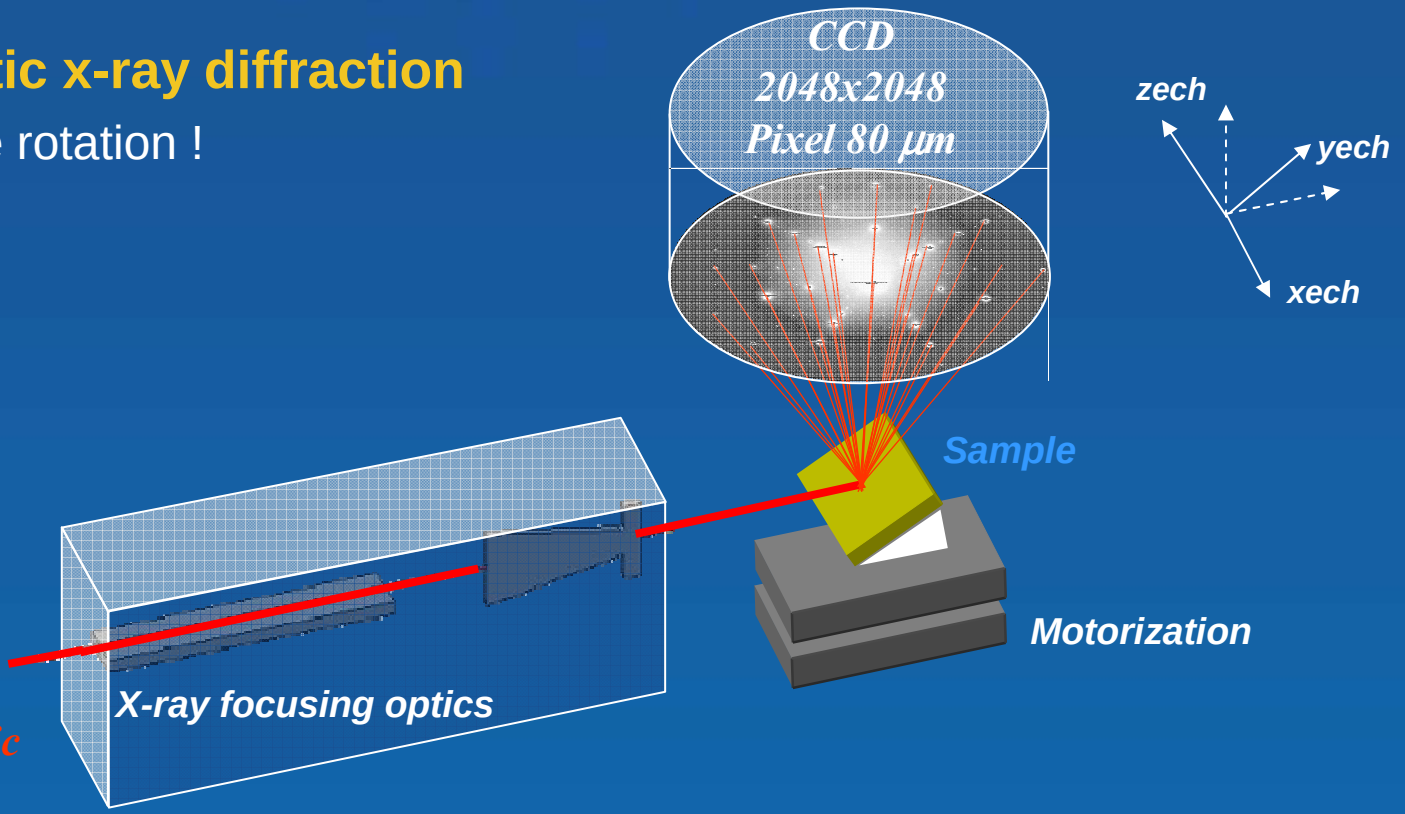
Goal & Applications

- **Determine the local strain state**
  - Sub-micrometer scale
  - Non-destructive testing
  - Sample as fixed as possible
- **Laüe diffraction with polychromatic x-rays**
- **Potential applications**
  - Electromigration in copper lines
  - Ferroelectric materials



Laüe microdiffraction

- **Grain size ~ Beam size**
- **Polychromatic x-ray diffraction**
  - No sample rotation !



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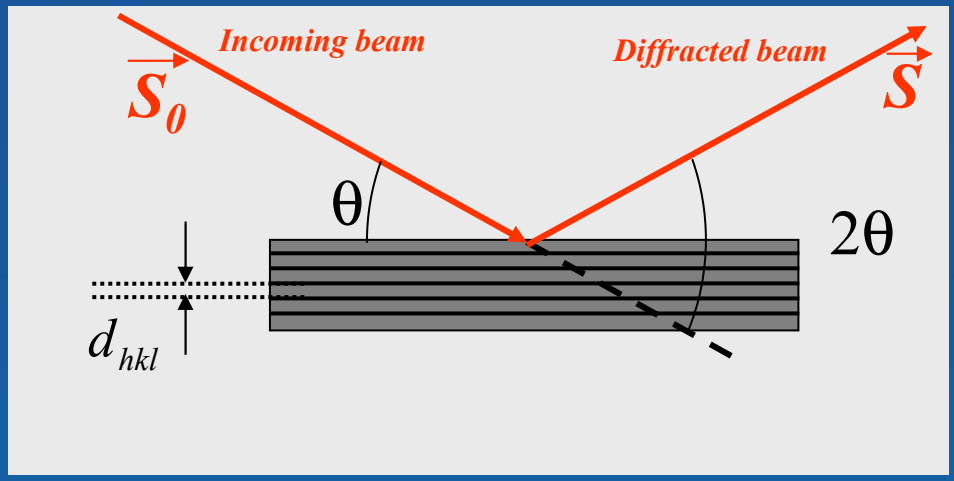
Strain determination

- **Bragg law**

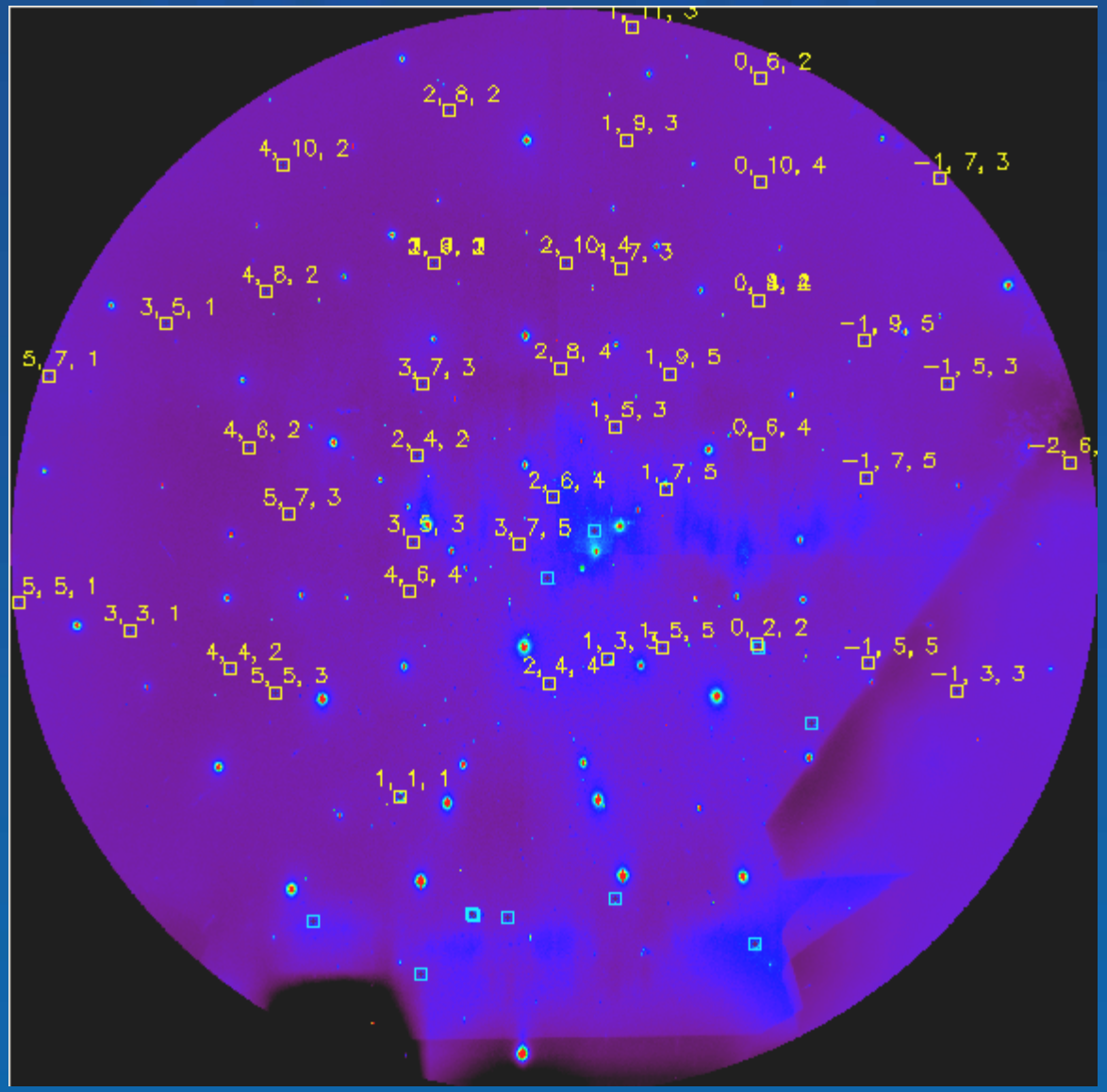
$$\lambda = 2d_{hkl}\sin\theta$$

- **Strain**

$$\varepsilon = \frac{d - d_0}{d_0} = \frac{\sin\theta_0 - \sin\theta}{\sin\theta}$$



- **Measuring the shifts of white beam diffraction spots**
  - Access to the (deviatoric) strain !


0, 6, 2
2, 8, 2
1, 9, 3
4, 10, 2
0, 10, 4
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4, 8, 2
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−1, 9, 5
5, 7, 1
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3, 7, 3
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4, 6, 2
2, 4, 2
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−1, 7, 5
2, 6, 4
1, 7, 5
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3, 3, 1
0, 2, 2
−1, 5, 5
4, 4, 2
5, 5, 3
2, 4, 4
−1, 3, 3
1, 1, 1

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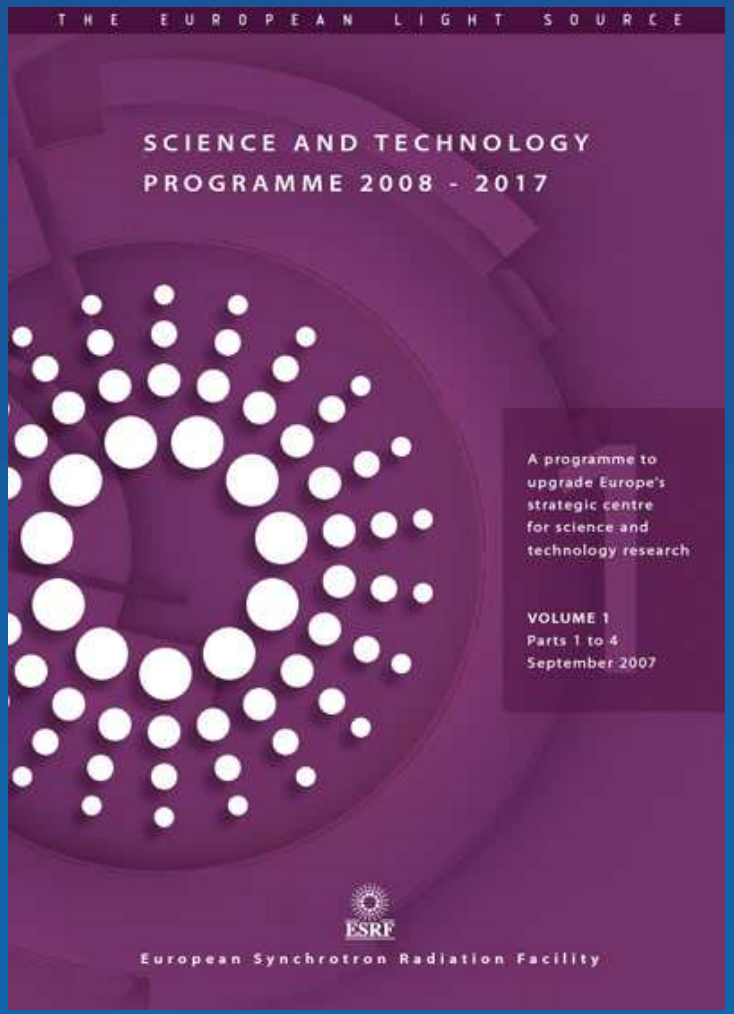
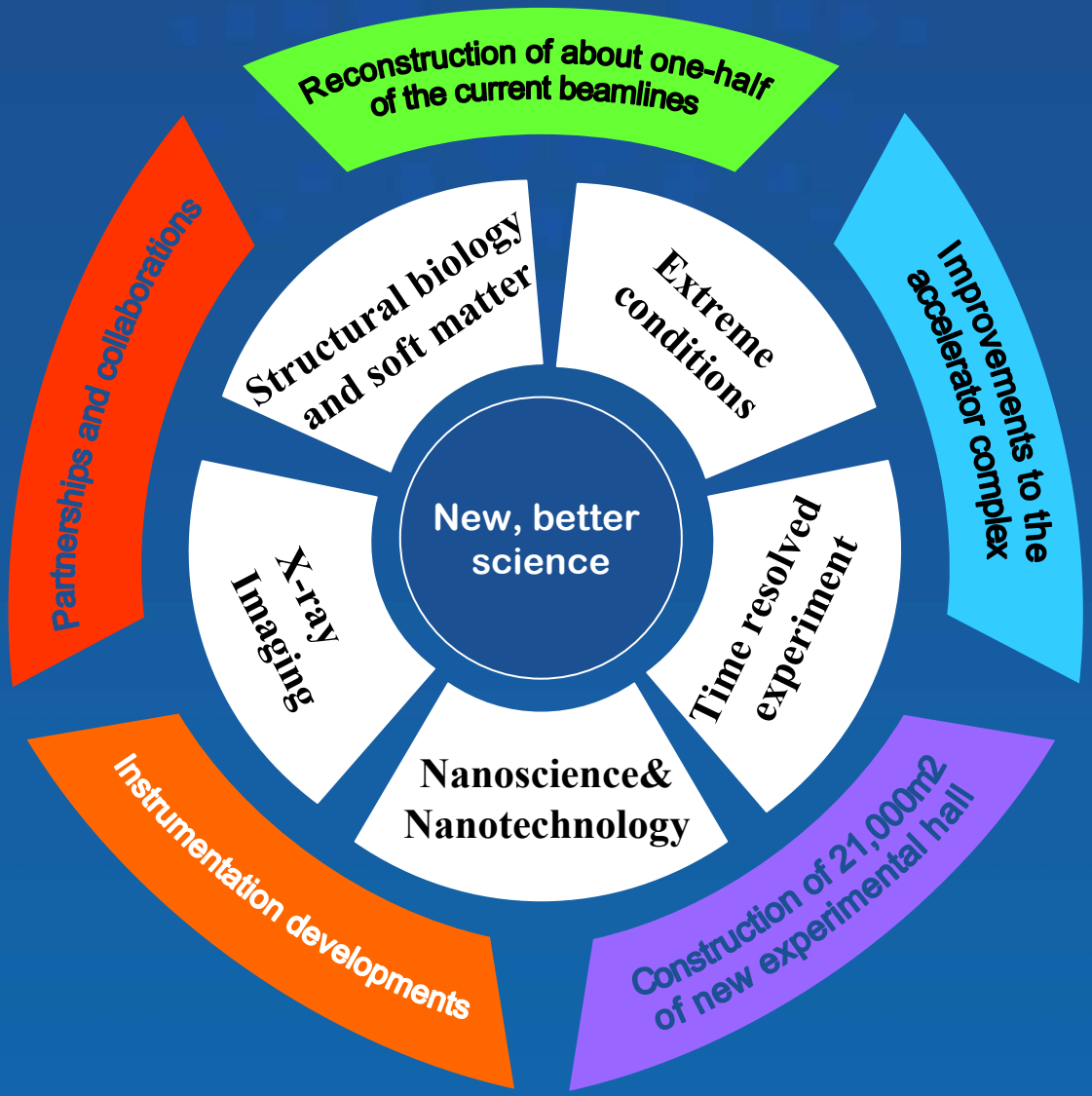
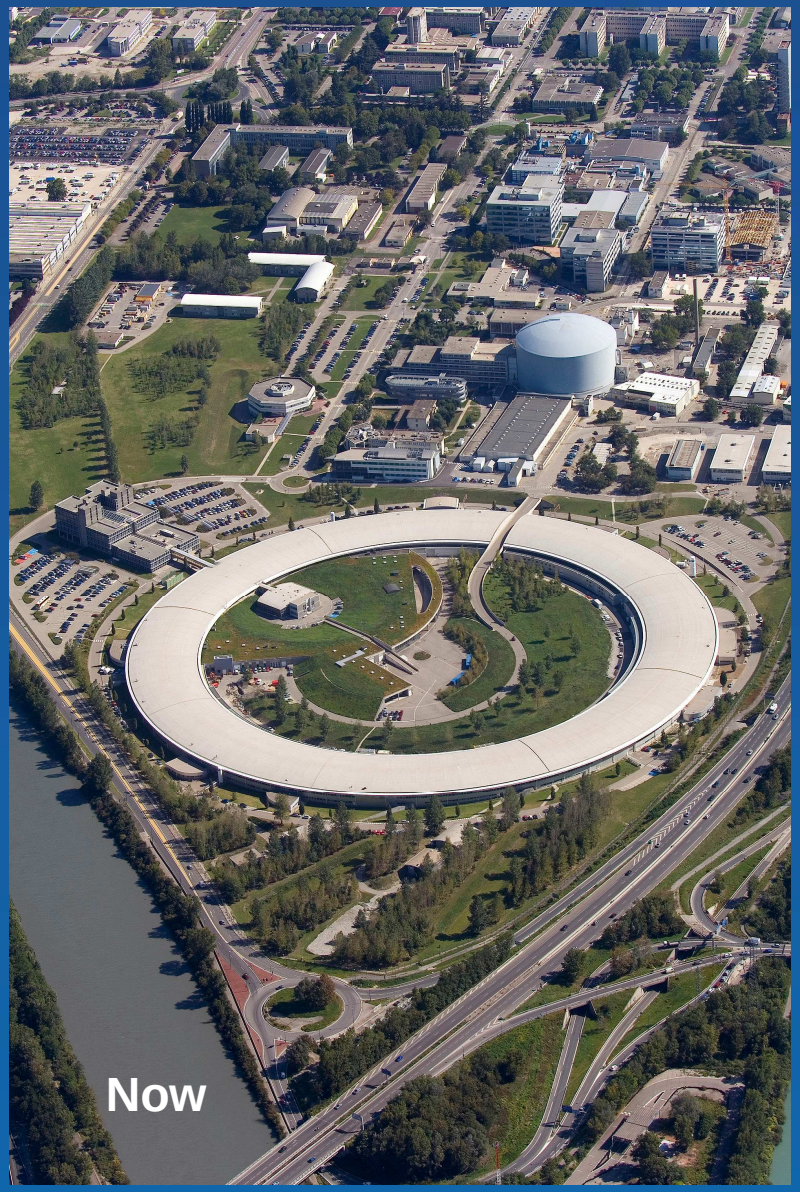
What's next ?

- **Main synchrotron limitation for nanocharacterization**
  - Resolution, limited to tens of nanometers
- **Need to go further in synchrotron developments**
  - ESRF upgrade

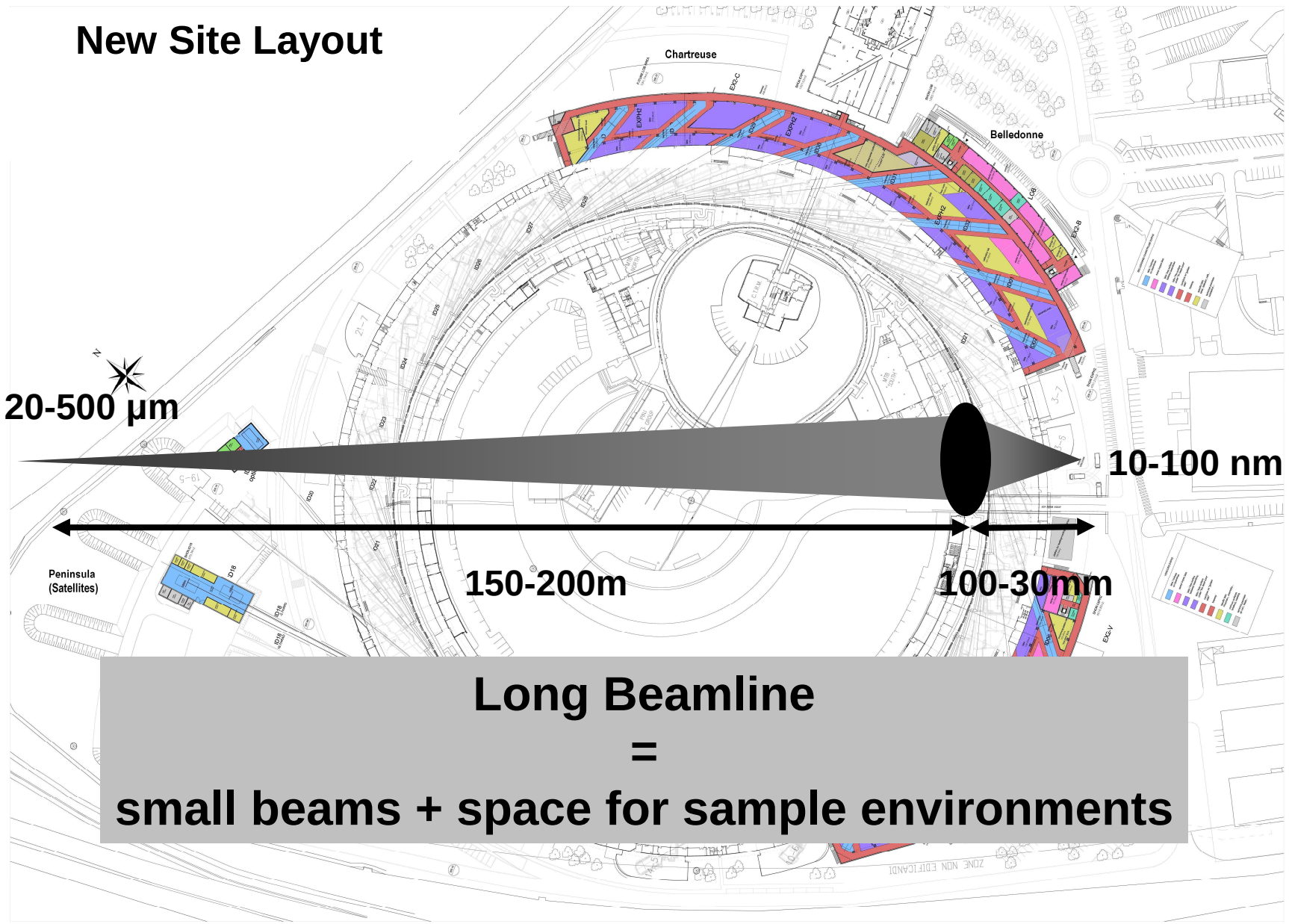



Reconstruction of about one-half of the current beamlines
Improvements to the accelerator complex
Construction of 21,000m2 of new experimental hall
Instrumentation developments
Partnerships and collaborations
Structural biology and soft matter
Extreme conditions
Time resolved experiment
Nanoscience& Nanotechnology
X-ray Imaging
New, better science


Now


2012

New Site Layout



Long Beamline
=
small beams + space for sample environments

Conclusion

- **Opportunity to have a synchrotron so closed to nanotech center !**
- **3D mentioned in many talks !**
  - 3D→penetration depth→ X-rays
  - Synchrotron is ‘spot-on’ for 3D nanocharacterization
  - …and also for 2D !
- **Synchrotron**
  - Combination of techniques, in-situ
  - Unprecedented way to observe 3D embedded micrometer-sized objects
- **Next step**
  - Access to $(10\text{-}20\text{nm})^3$ resolution with hard x-rays

Acknowledgments

- **Special thanks to**
  - FCMN organizers, A. Chabli, J. Susini, D. Mariolle, R. Tucoulou, P. Tafforeau, P. Gergaud, O. Renault, P. Leduc, A. Garnier, F. Souchon, M. Lavayssière, P. Lamontagne, B. Florin, H. Dansas, J-S. Micha, O. Ulrich, F. Rieutord

- **And to a number of people involved in this work among which**
  - ESRF ID21, ID22, BM32 beamline staff and support groups
  - CEA, INAC synchrotron team
  - CEA, LETI, MINATEC characterization team

- **Poster promotion**
  - Synchrotron related : WE-006, WE-025
  - Tomography related : TH-021
  - Others : WE-020, TH-014, TH-020

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