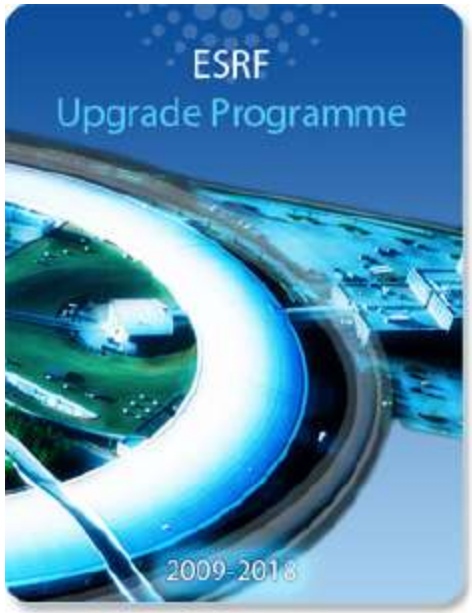
SPECTROSCOPY DETECTOR NEEDS FOR X-RAY NANOPROBES WITHIN THE ESRF UPGRADE



P. Cloetens

P. Bleuet[2], S. Bohic[3,1], F. Fus[1], M. Hubert[1,2], G. Martinez-Criado[1], A. Pacureanu[1], J.C. da Silva[1], R. Tucoulou[1], Y. Yang[1]

[1]ESRF, Grenoble
[2]CEA, F-38054 Grenoble, France
[3]INSERM U-836 Grenoble, France

Special thanks to J. Morse, C. Cohen and T. Martin for NINA ED Detectors
to A. Mirone for PyMCA fitting software

ESRF Upgrade Programme
Phase I and Phase II

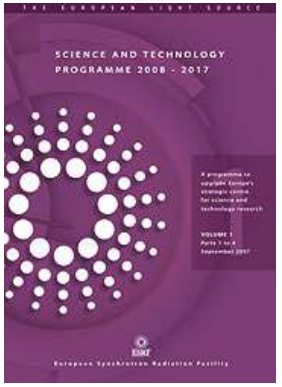
Phase I

19 upgraded or refurbished BLs

Accelerator and source upgrade

Construction programme

2009 — 2015



Today — 2015 — 2022

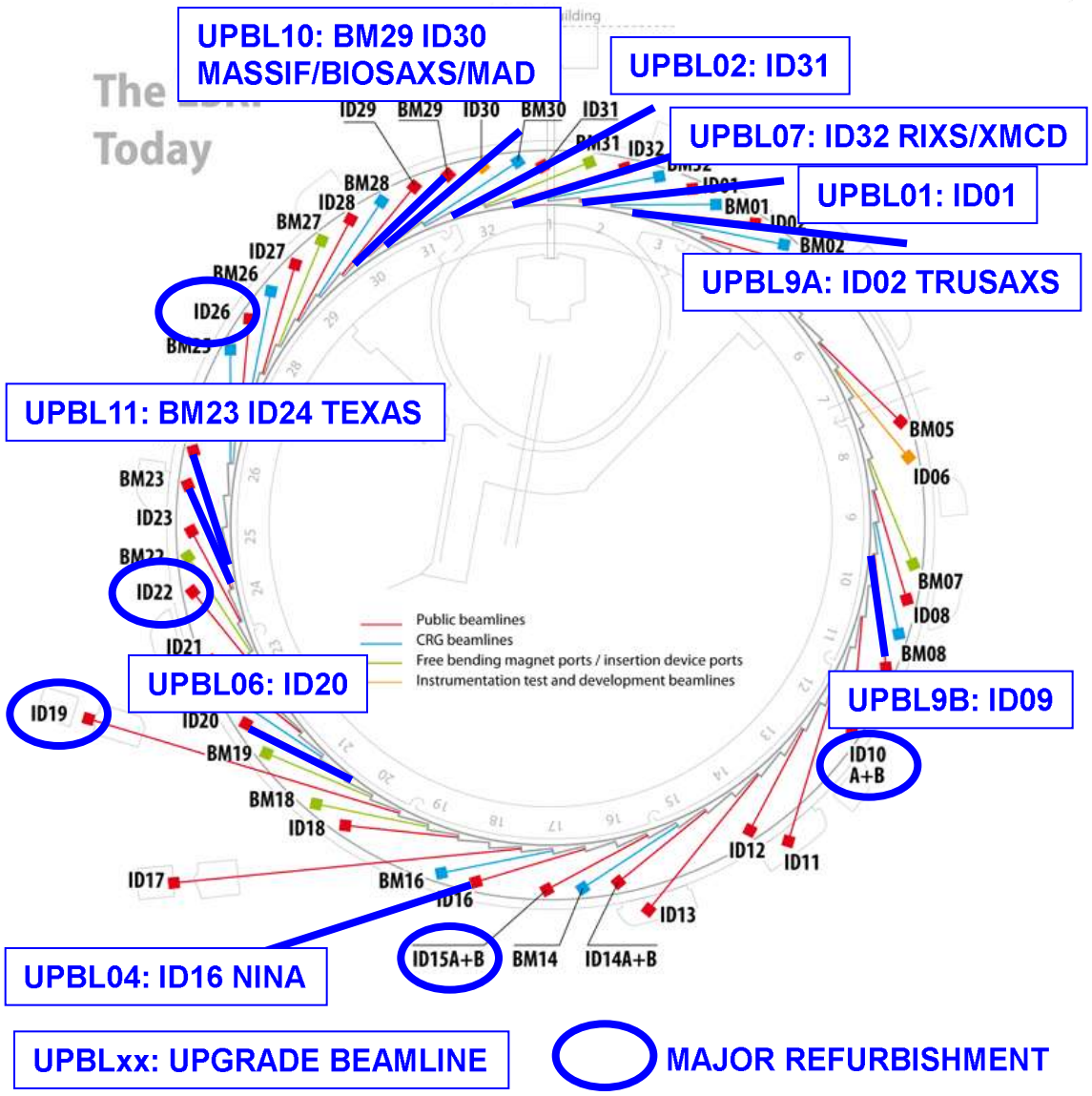
Phase II

New storage ring

4 new BLs

Enabling technology (Detectors)

PHASE I BEAMLINES RELATED TO ANALYSIS / IMAGING



UPBLxx: UPGRADE BEAMLINE

MAJOR REFURBISHMENT

Phase I Pilot Projects

ID11 Materials Science

ID13 Micro and Nano Diffraction

Refurbished Beamlines

ID10 Soft Interfaces and Coherent Scattering

ID21 X-ray Microscopy

ID19 Full-field Hard X-ray Imaging

New Beamlines

ID24/BM23 'Texas' X-ray Absorption Spectroscopy

ID16A/ID16B 'NINA" Nano-Imaging / Nano-Analysis

Upcoming:

ID01 Diffraction Imaging for Nano-Analysis

ID31 High Energy: buried interfaces and materials processing

NINA ON ID16

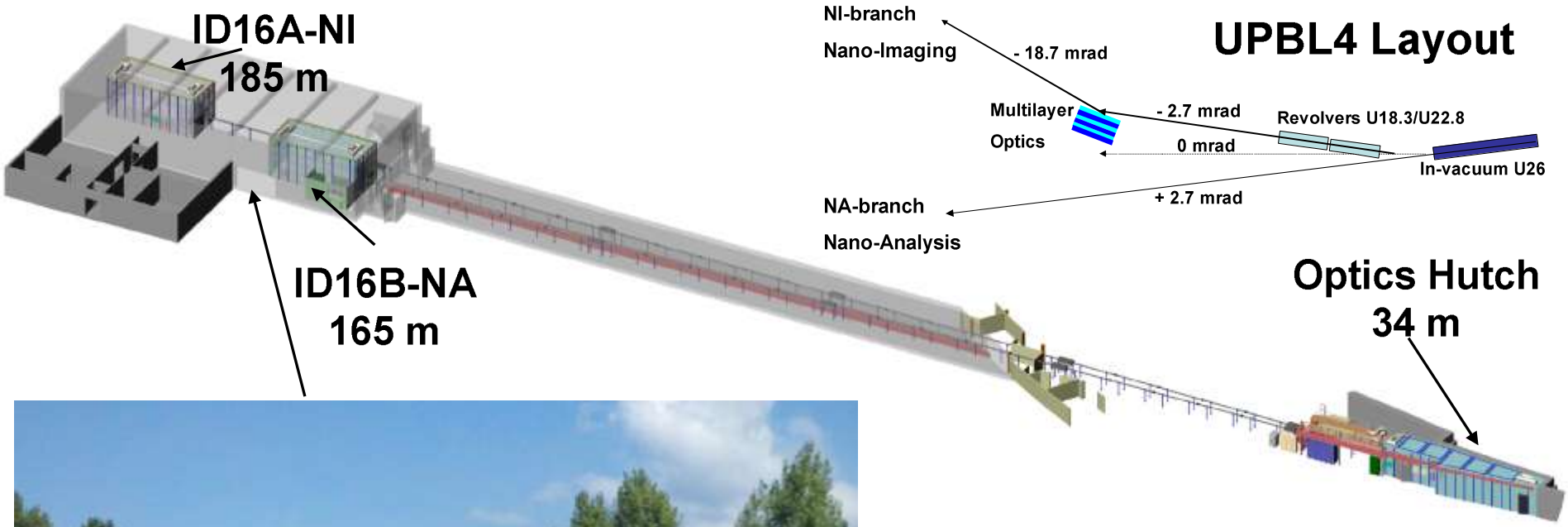
NINA consists of two independently operating beamlines:

- **ID16A-NI: ultimate pink beam focus for imaging and XRF**
- **ID16B-NA: nanofocus monochromatic beam for spectroscopy**

X-ray ultra-microscopy and nano-spectroscopy

	NI	NA
Length	185 meters	165 meters
Spatial Res.	10 – 100 nm	50 nm -1 μm
ΔE/E (%)	1	0.01
Energy range	Discrete 11 – 17 – 33 keV	Scanning 5 → 70 keV
Main goals	XRF, coherent XRI-2D/3D Cryo environment	XAS, XRD, XRF, XRI-2D/3D *in-situ* experiments
Main fields	Biology & Life Sciences Nanotechnology & Nanomedicine	Biology, environmental sciences, geoscience, materials sciences, ...

UPBL4 NINA ON ID16

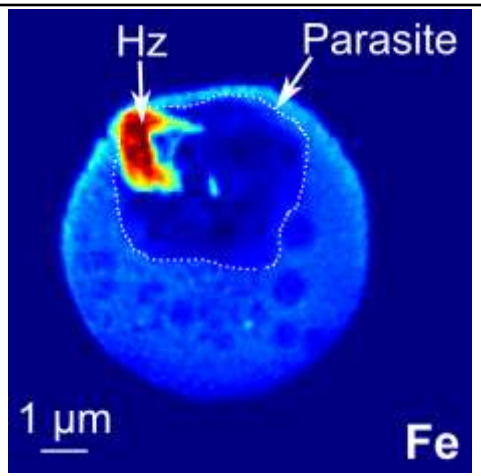


UPBL4 Layout

ID16 Satellite Building

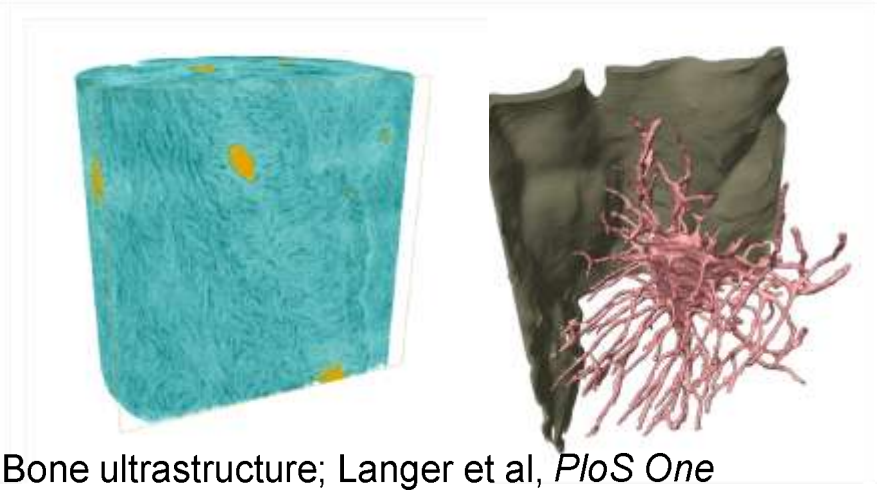
Scientific drivers

Biomedical Research: Sub-cellular processes



Anti-malarian drugs
Dubar et al, *Chem. Commun.*

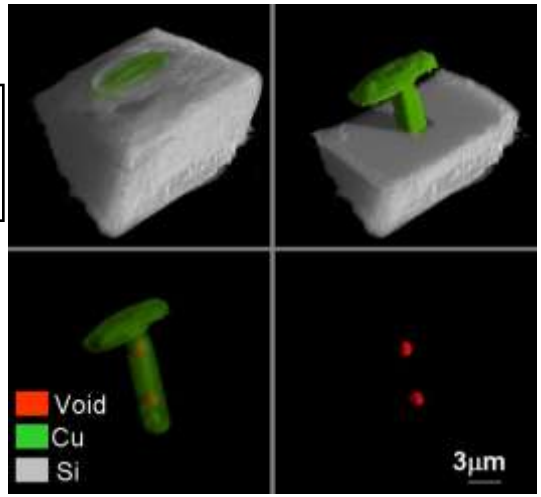
Biomedical Engineering: Tissue-level



Bone ultrastructure; Langer et al, *PloS One*

ID16A-NI

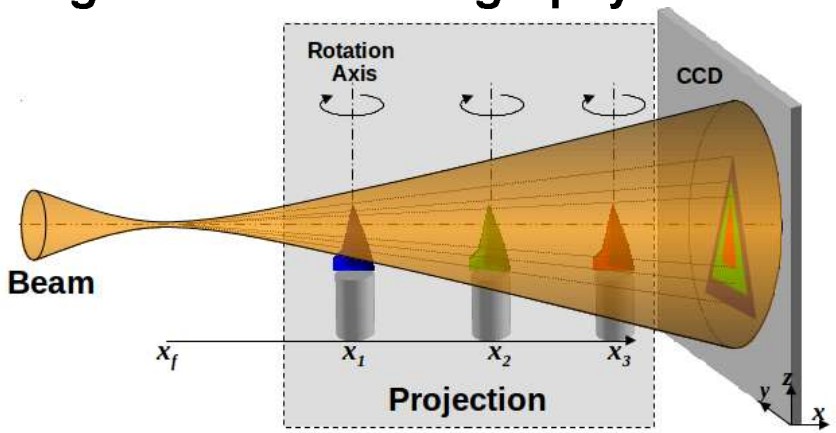
Nano/Micro-Technology: 3D Integration



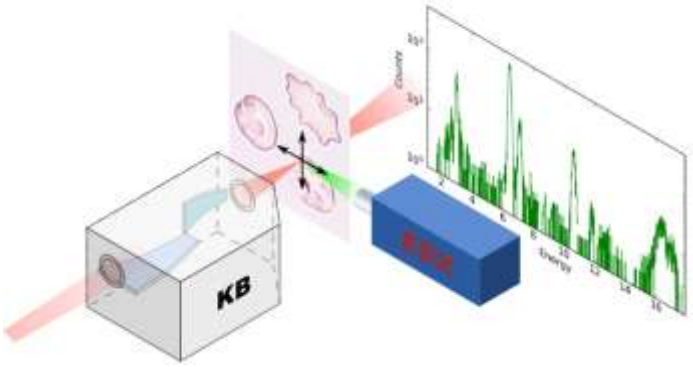
Voids in Through-Si-via
Bleuet et al (CEA-Leti)

Experimental techniques

Magnified HoloTomography

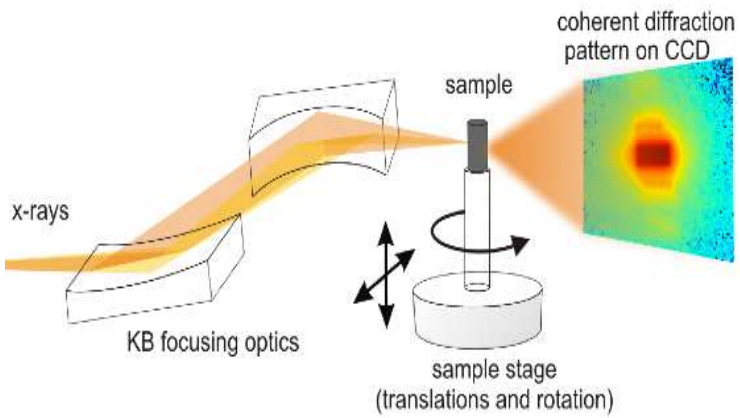


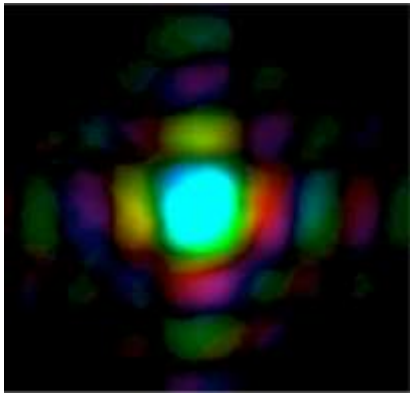
X-ray Fluorescence Microscopy (2D/3D)



Electron Density distribution ← **ID16A-NI** → **(Trace) Element distributions**

Ptychographic Nano-Tomography (ultimate resolution)





Probe

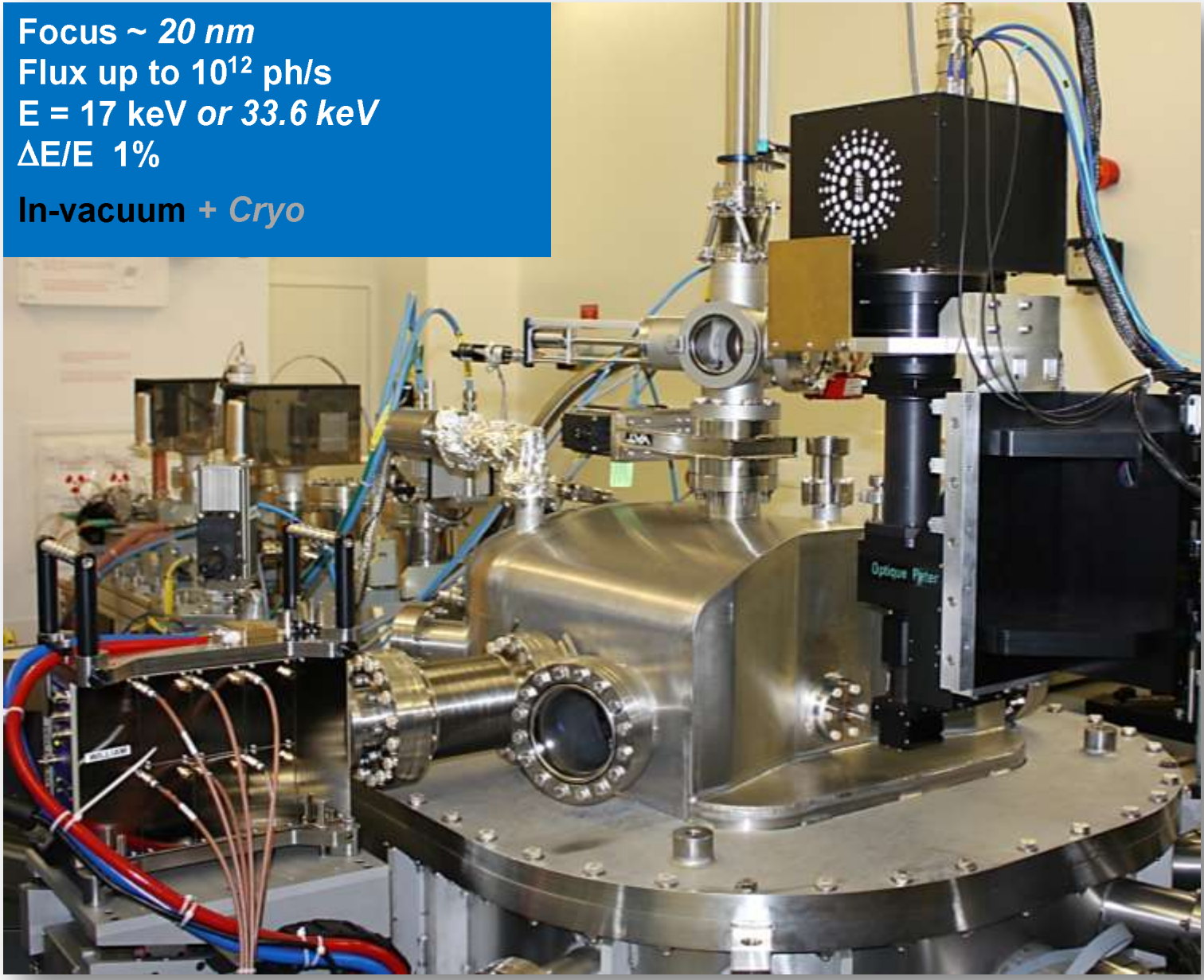
Object

in collaboration with TUM group and UCL
P. Thibault et al.

NANO-TOMOGRAPHY AND NANO-FLUORESCENCE AT ID16A

Focus ~ *20 nm*
Flux up to 10^{12} ph/s
E = 17 keV *or 33.6 keV*
ΔE/E 1%

In-vacuum + *Cryo*

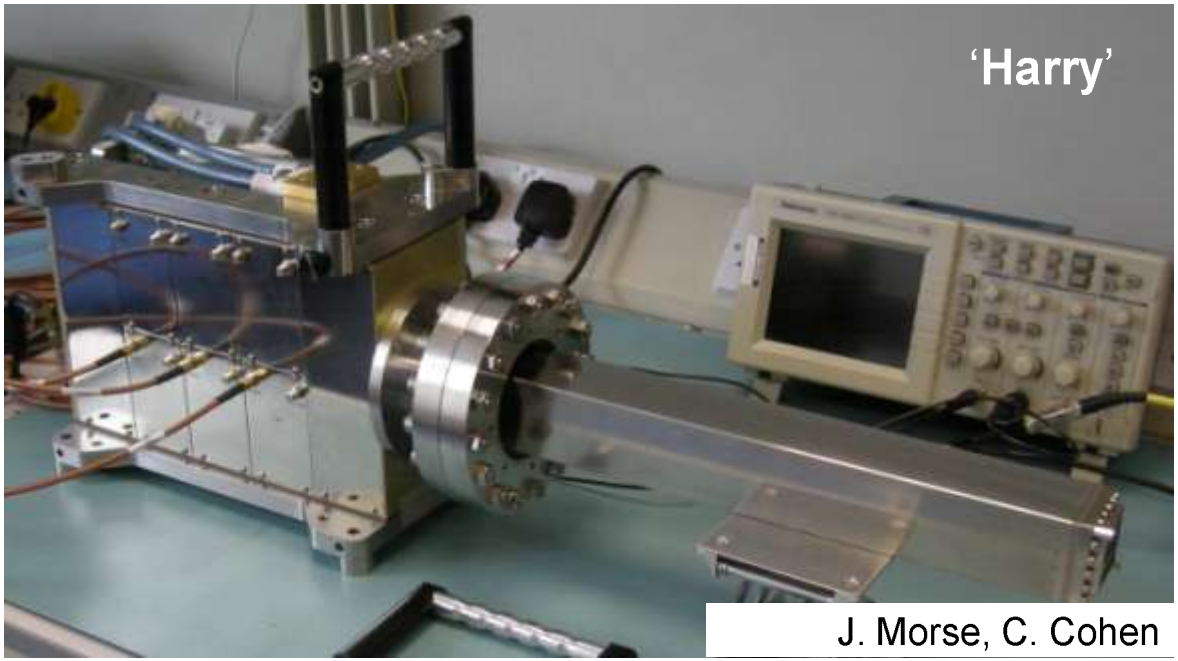


ID16A-NI: DETECTORS

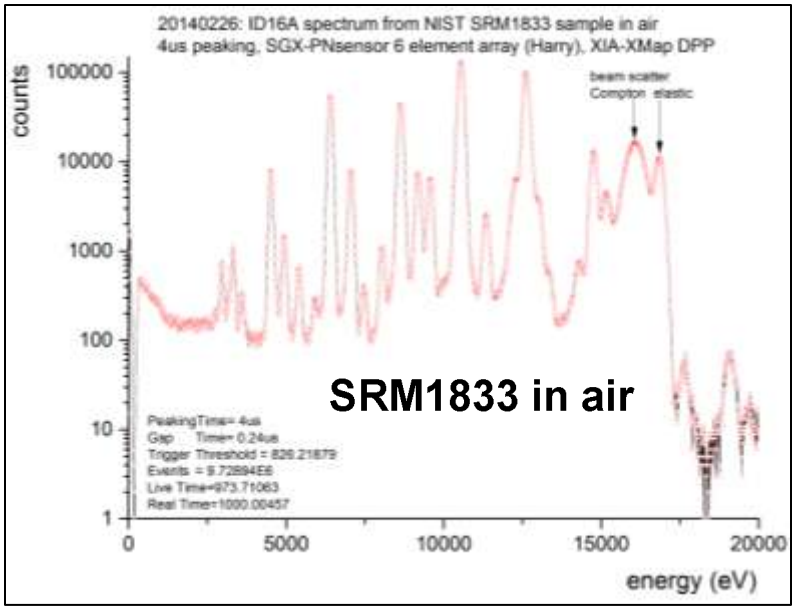
Energy dispersive detectors

- *Two identical detectors*: Harry & William
- 2 x PNdetector SDD 6 element arrays
  = **2 x 540mm²**
- **large detection solid angle**.
- *Operation in vacuum*, confined space
- Detectors produced by **SGX Sensortech**

- Global ***throughput*** **count rate to ~6Mcps**
- Fast spectra readout to 1kHz?



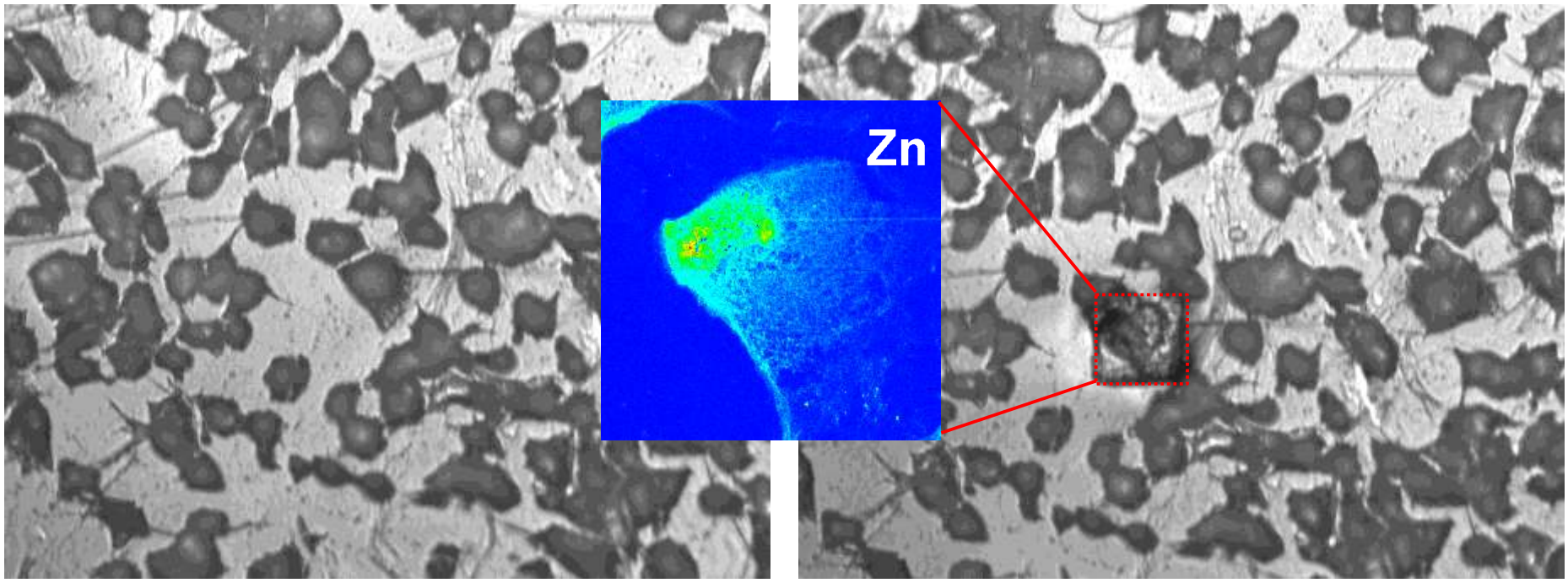
J. Morse, C. Cohen



DETECTION EFFICIENCY: RADIATION DAMAGE

Before

After



ID22NI measurement

- **Large collection angle** is critical

Push integrated detection scheme further
multiple single detectors or detector array
preserve detector collimation

- Multiple fast low dose acquisitions
- **Cryo** to be integrated in 2015
- **Main limitation towards 3D**

DETECTOR SPEED: FAST NANO XRF

Preserve large FOV (e.g. complete cell)
at high spatial resolution

Example:
Today:
50 nm step size and 10 μm area (200 x 200 pixels)
100 ms / pt → **1.1 hour**
Future:
5 nm step size and 10 μm area (2000 x 2000 pixels)
100 ms / pt → **5 days**
1 ms / pt → **1.1 hour**

Practical limitation with XIA-Xmap DPP for 12 elements *at ESRF*: **50 ms/pt**
Fast Nano-positioning is also an issue

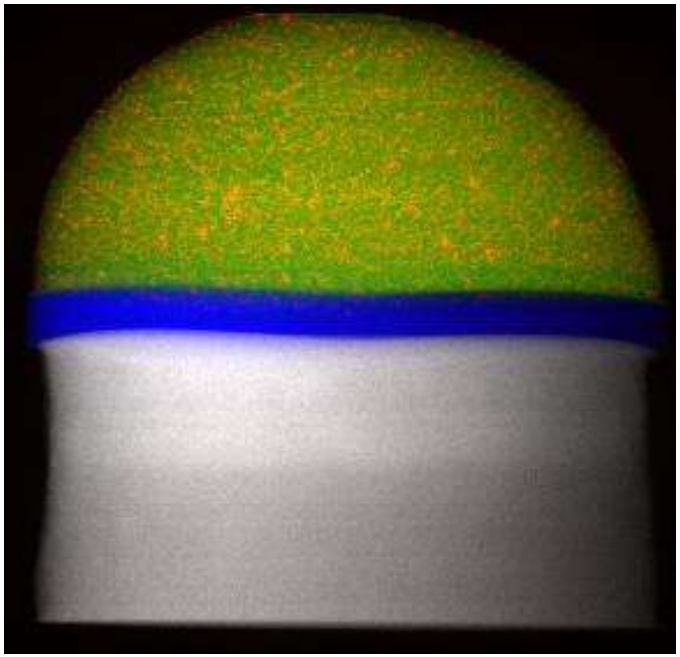
HIGH ENERGY NANOCT AND FLUORESCENCE CT

ID22NI measurement

3D integration for CMOS technology
3D non-destructive imaging of Copper pillars

Energy = 29.6 keV → Transmission and excitation K-edge Sn

2D XRF maps → elemental distributions



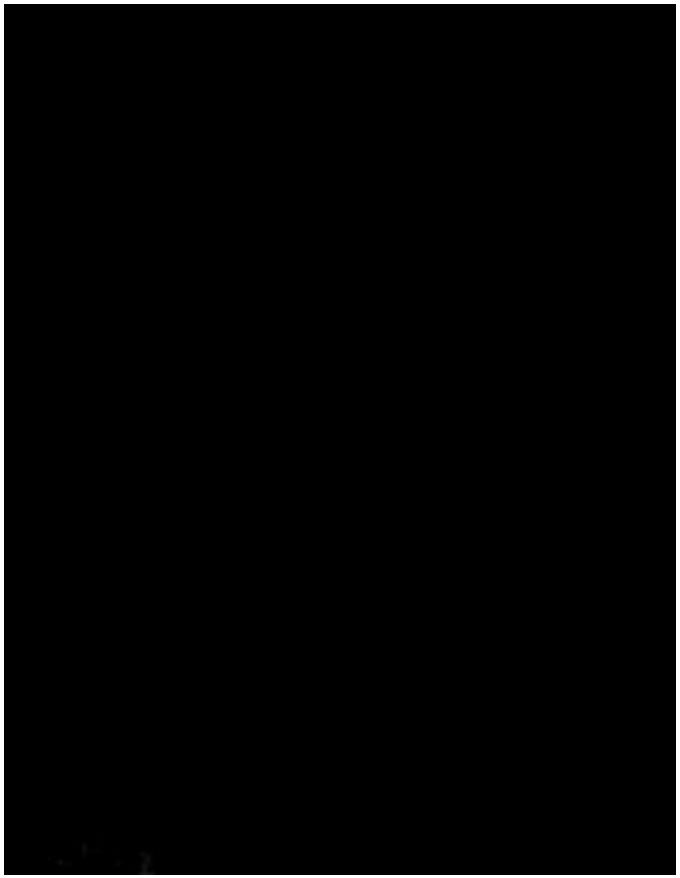
Bleuet, P. et al., SPIE Developments in x-ray tomography IX, 2014, San Diego

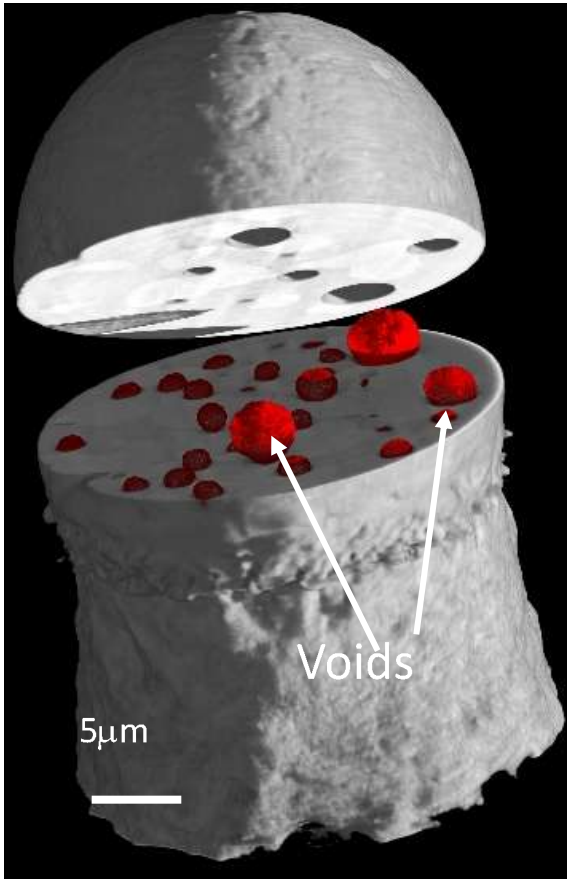
HIGH ENERGY NANOCT AND FLUORESCENCE CT

ID22NI measurement

3D integration for CMOS technology
3D non-destructive imaging of Copper pillars

NanoCT provides distribution of voids and phases





Bertheau, J. et al., Microelectronic Engineering 113, 123 (2014)

The European Synchrotron | ESRF

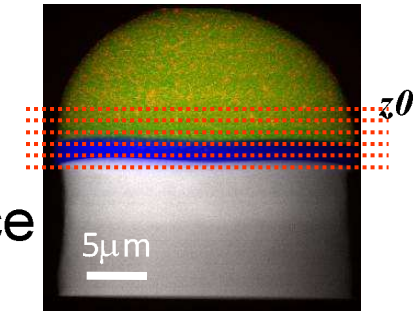
HIGH ENERGY NANOCT AND FLUORESCENCE CT

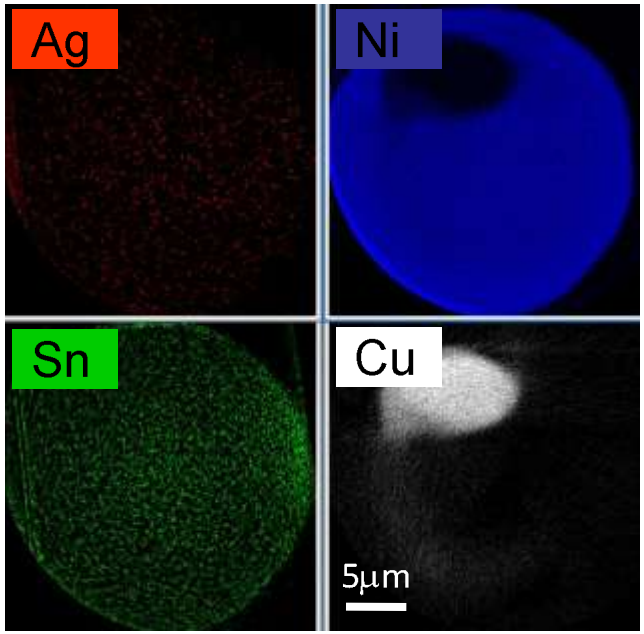
ID22NI measurement

3D integration for CMOS technology
3D non-destructive imaging of Copper pillars

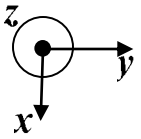
Fluorescence tomography slices at different heights

2D fluorescence mapping





Reconstructed slices at z=z0-0.9µm



2D fluorescence tomography

Throughput is critical to go to full 3D
Beam typically attenuated 2000 times !!!

Bleuet, P. et al., SPIE Developments in x-ray tomography IX, 2014, San Diego

The European Synchrotron | ESRF

DETECTOR NEEDS FOR NANOPROBES

	Required*	Achieved**
Energy Range	1-20 keV (1-70 keV)	
Energy Resolution	~ 140 eV	~ 140 eV
Integration Time	1 ms	50 ms
Total count rate	10^7-10^8 ph/s	10^6 ph/s @ 1µs PT
Solid angle coverage	> 1 steradian	0.5-0.9 sr
Price	?	Known

- Compatible with confined space and environment of nanoprobes
- Robust software well integrated with motion control

* From UPBL04 TDR
** On ID16A-NI

ESRF Upgrade Programme
Phase I and Phase II

Phase I

19 upgraded or refurbished BLs

Accelerator and source upgrade

Construction programme

2009 — 2015

Technical Design Study

2015 — 2022

10/2018: Start SR construction
9/2019: Restart/conditioning
6/2020: User operation

Phase II

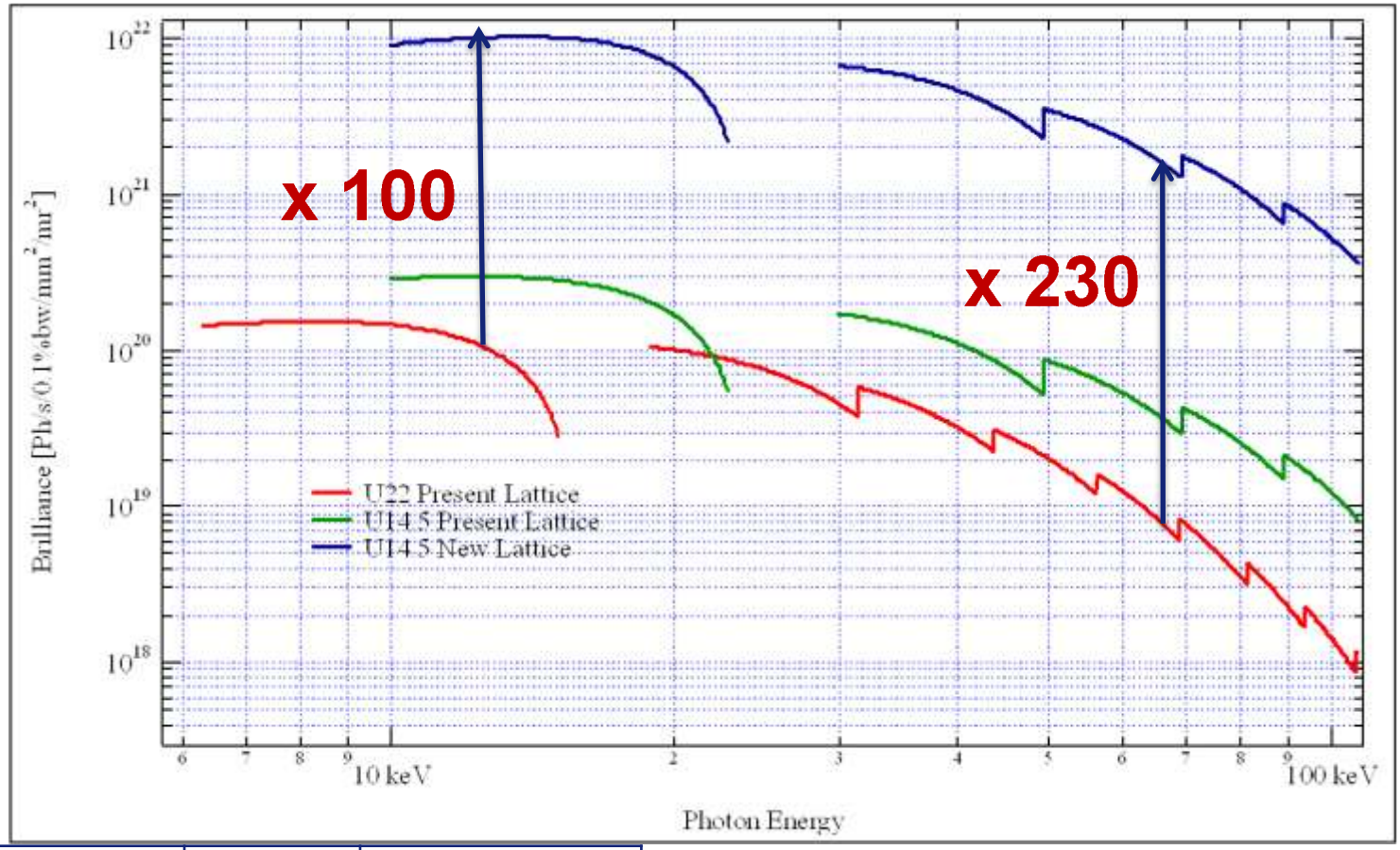
New storage ring

4 new BLs

Enabling technology (Detectors)

BRILLIANCE INCREASE AT LOWER HORIZONTAL EMITTANCE

With new short period cryogenic undulators



Hor. Emittance [nm]	4	0.15
Vert. Emittance [pm]	3	2
Energy spread [%]	0.1	0.09
β_x[m]/β_z [m]	37/3	3.4/2.8

U22 Min. gap 6 mm, K_{max}=1.7
U14.5, Min. gap 4 mm, K_{max}=1.7 (CPMU)


Thank you for your attention