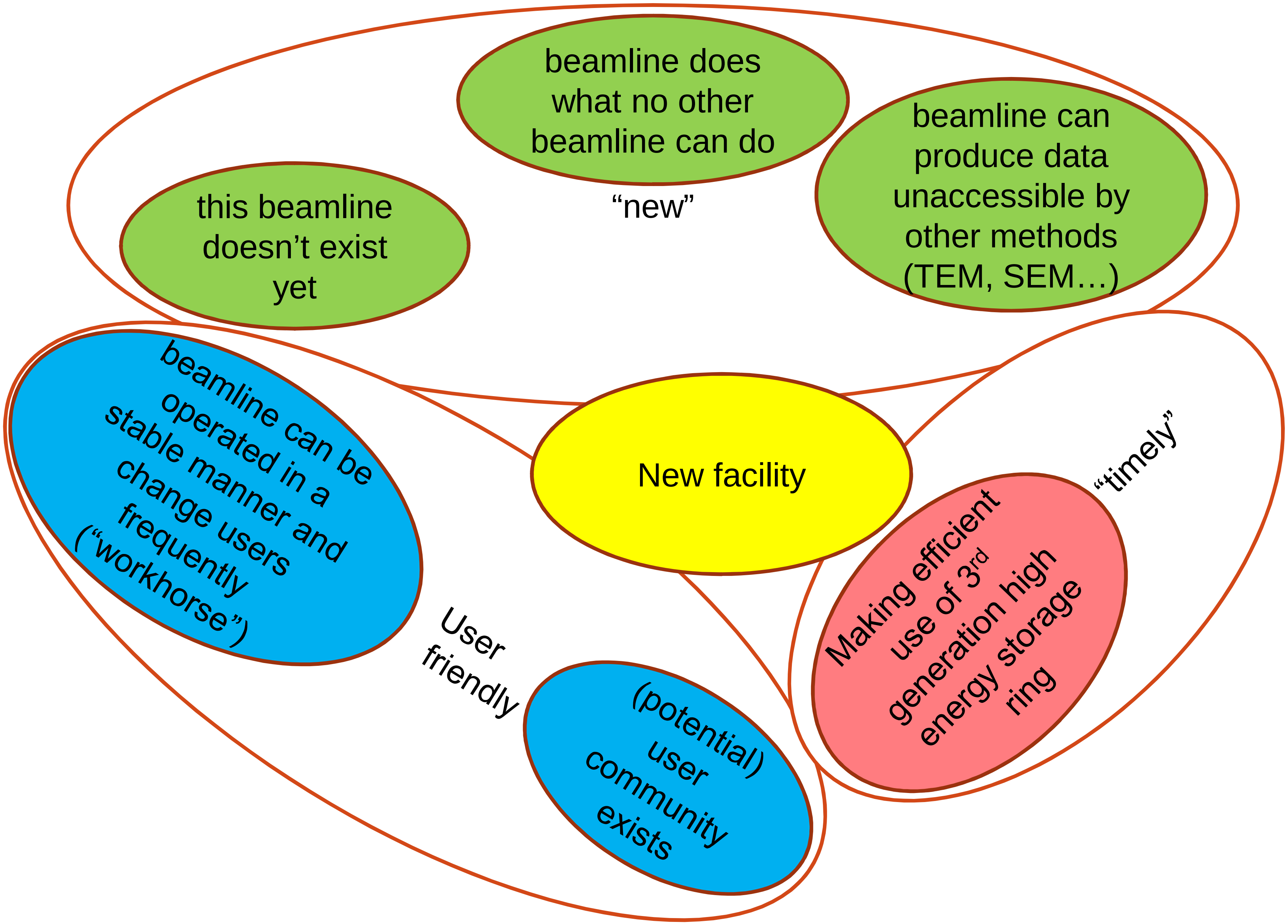
New facilities for applied surface science at 3rd generation synchrotron sources

..how new is new ?
A beamline scientists' point of view

T. U. Schülli APS workshop January 2012


beamline does what no other beamline can do
beamline can produce data unaccessible by other methods (TEM, SEM…)
this beamline doesn't exist yet
"new"
New facility
beamline can be operated in a stable manner and change users frequently ("workhorse")
User friendly
(potential) user community exists
"timely"
Making efficient use of 3rd generation high energy storage ring

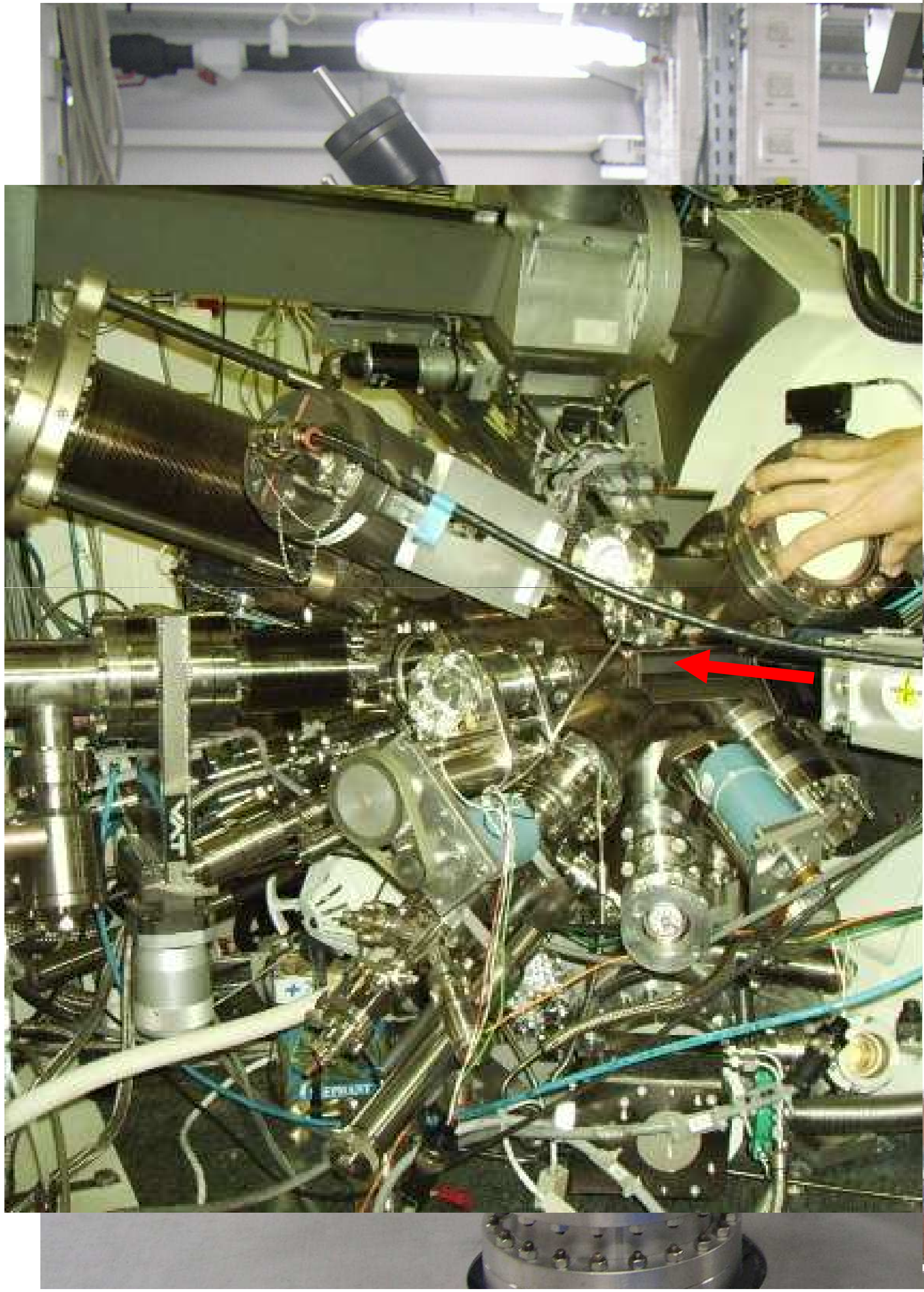
Outline

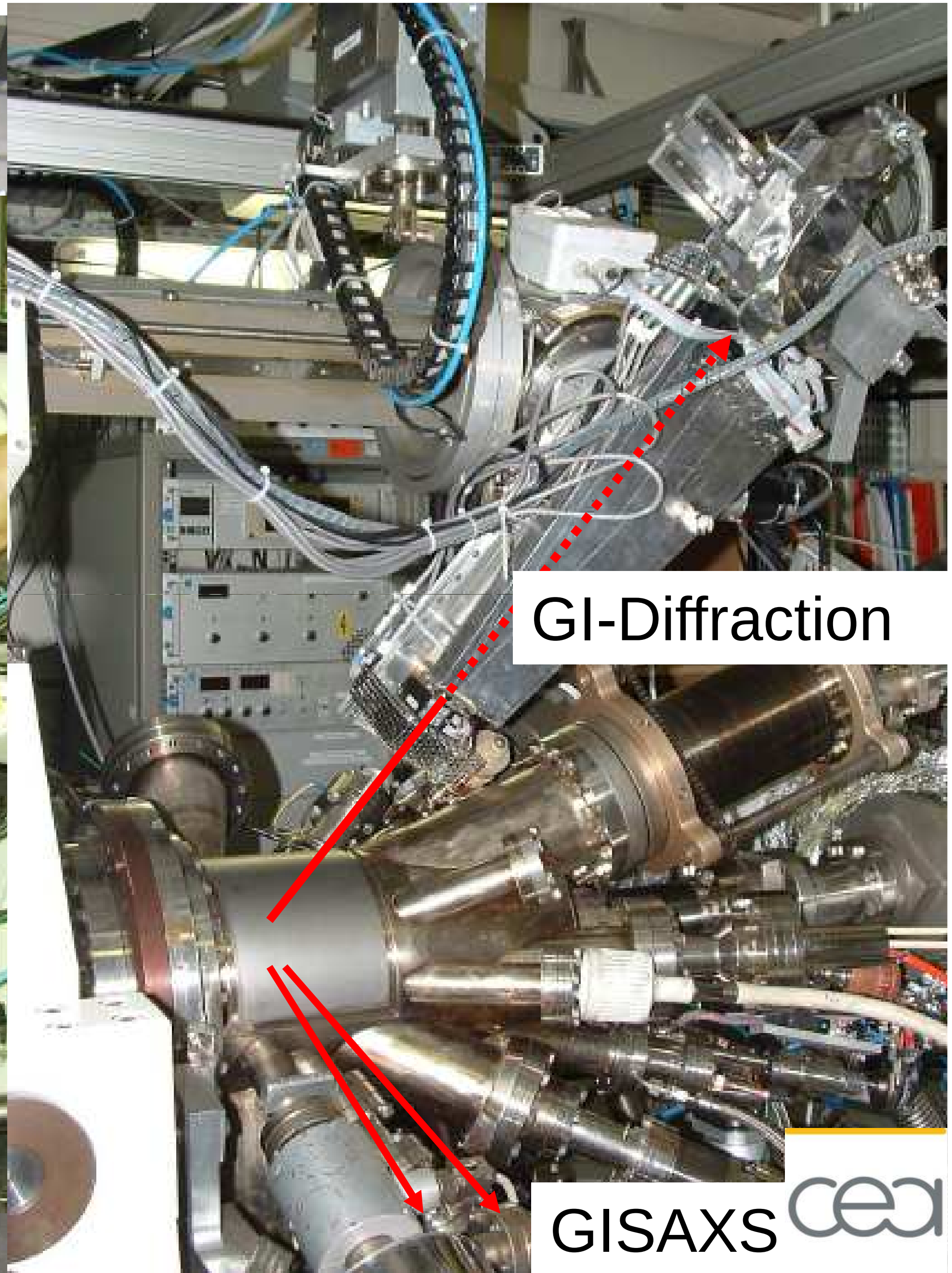
Observation of growth processes: Interface induced supercooling: An X-ray view on liquid – solid interaction

Surfaces studied with submicronic beams

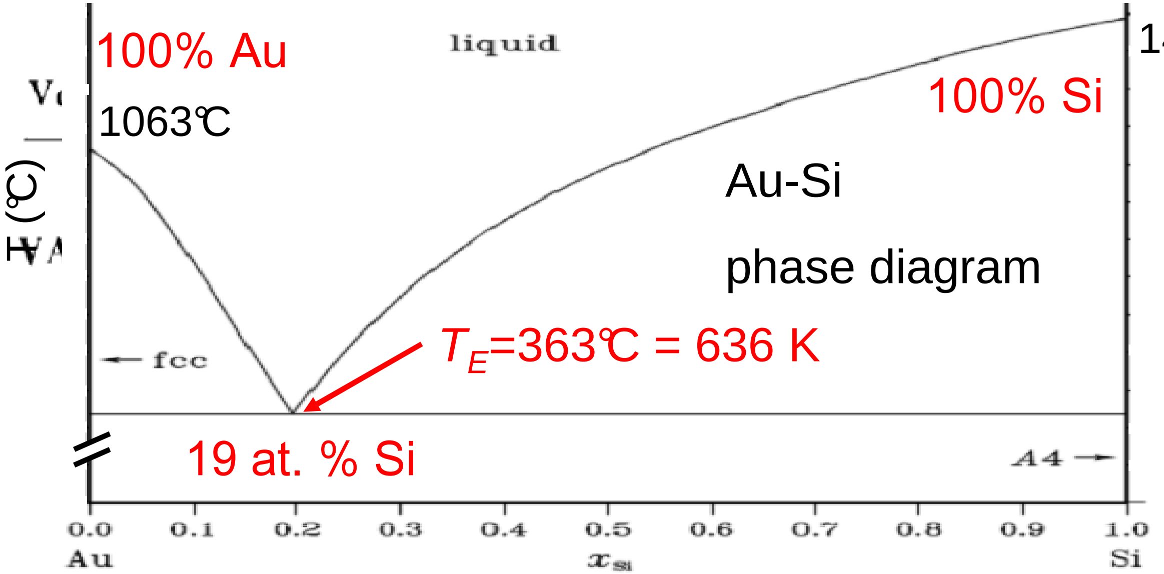

2003-2009
BM32
IF
Anomalous Scattering Beamline
ID1
ESRF

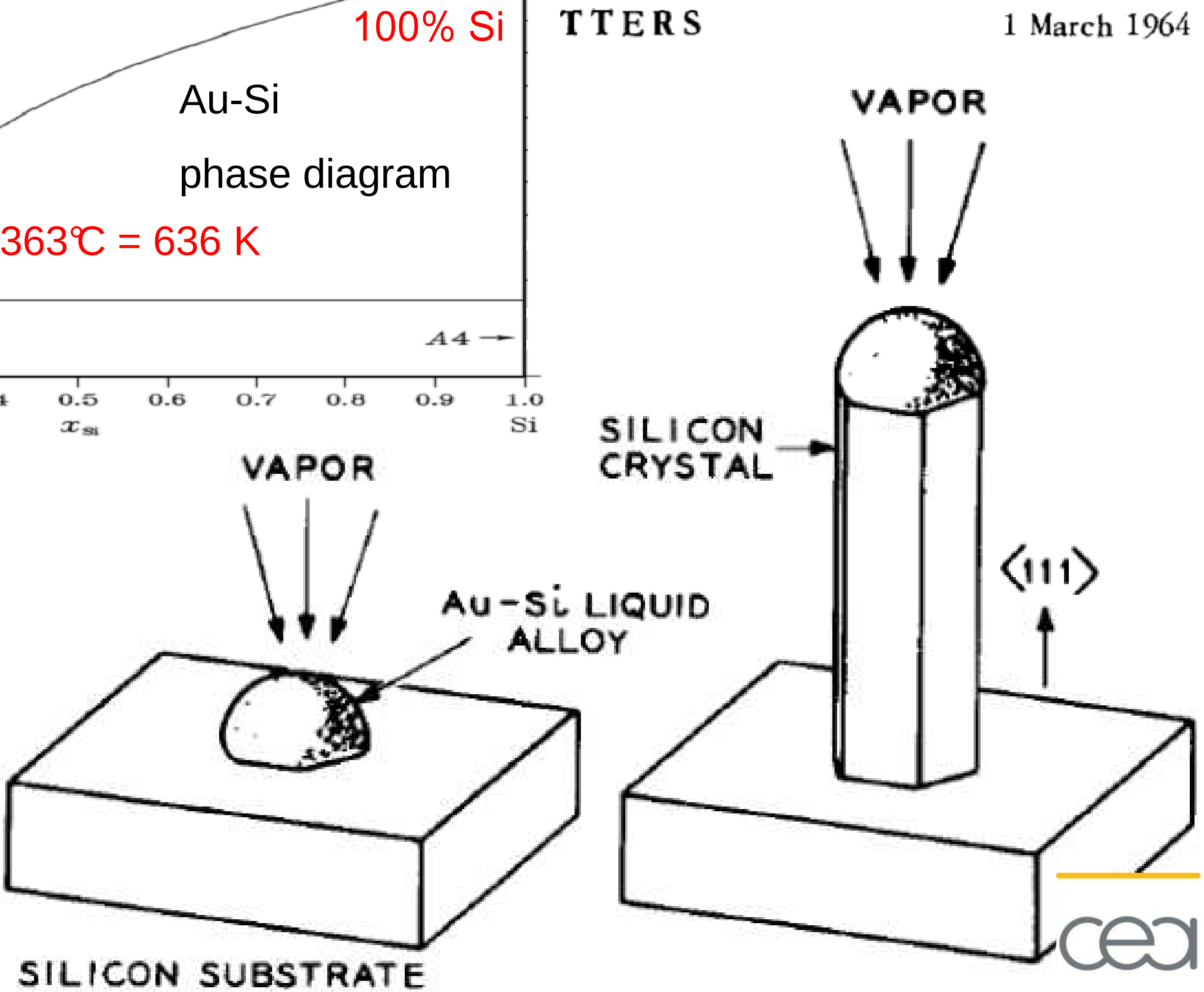
BM32 MBE in situ growth chamber





Vapour-Liquid-Solid growth

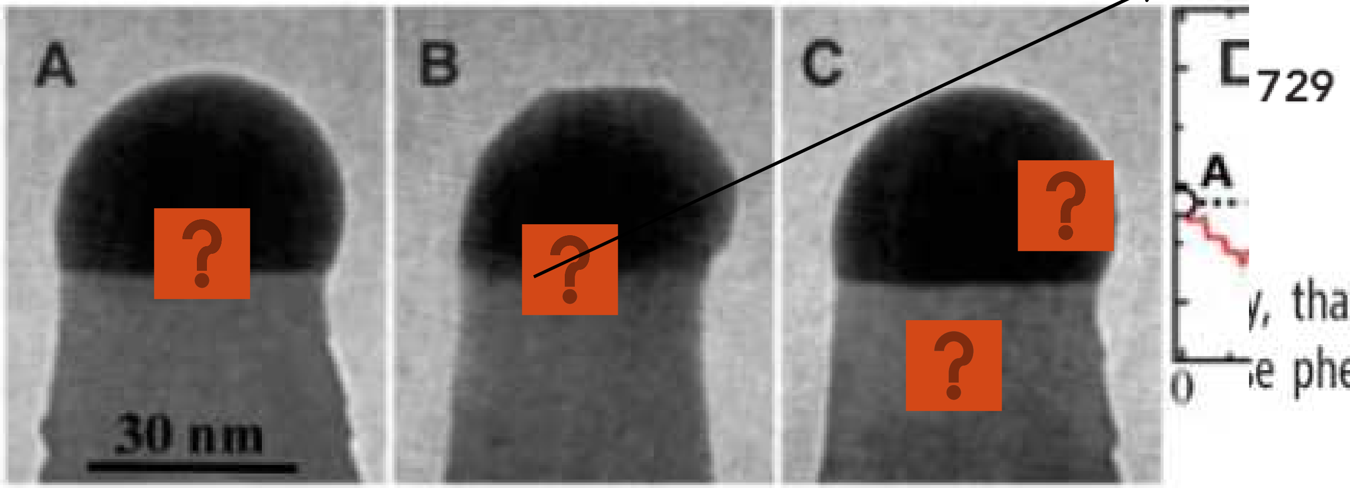




Germanium Nanowire Growth Below the Eutectic Temperature

S. Kodambaka,* J. Tersoff, M. C. Reuter, F. M. Ross†

“Supercooling”



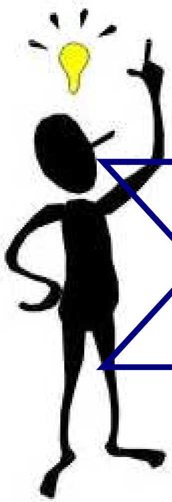
either liquid
state depend

y, that the catalyst
e phenomena may

Why add a gas-source?

CVD is the most applied growth mode in silicon and SiGe technology and beyond (SiC, nanowires, Graphene, Diamond…)

2005:

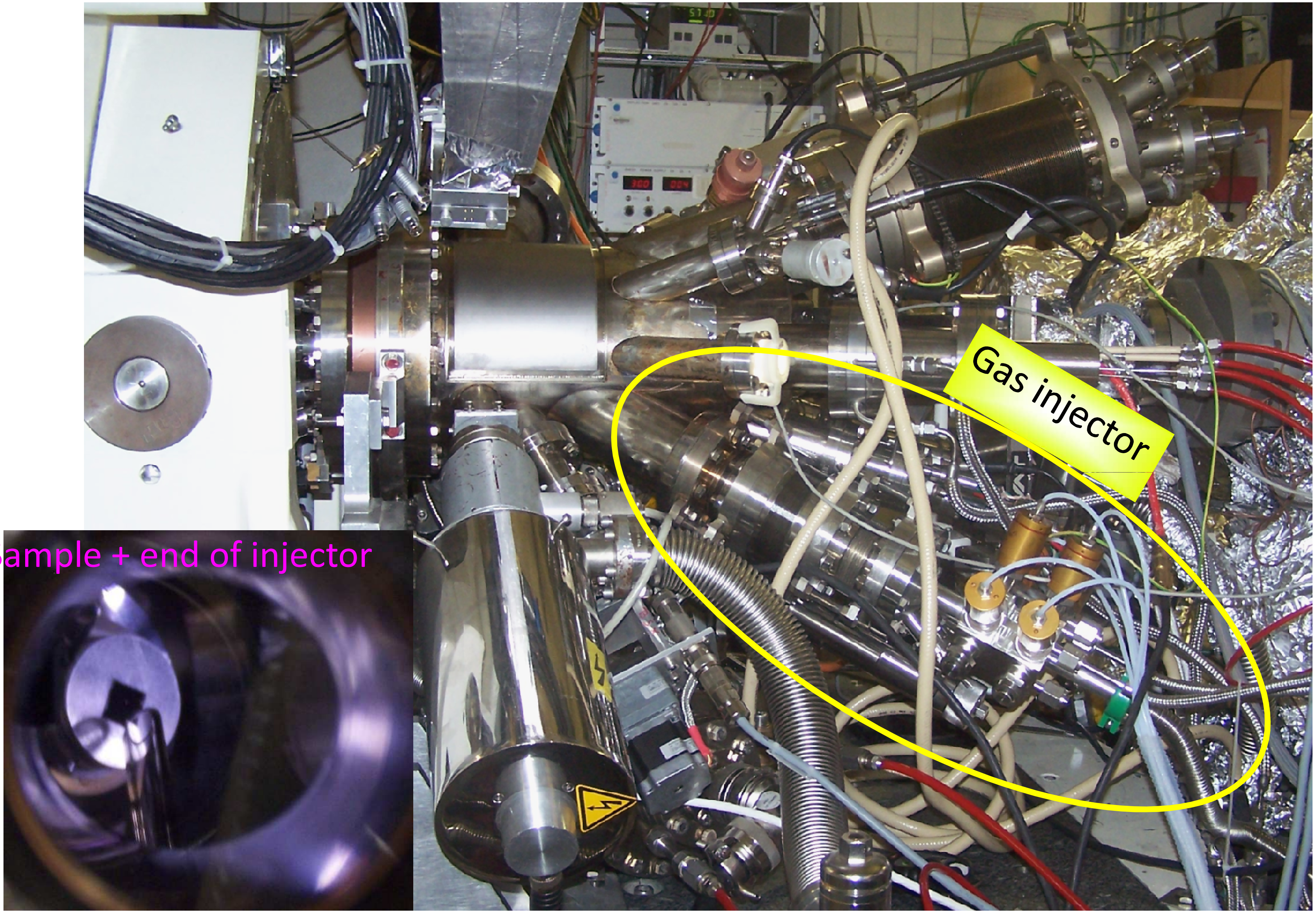


First approach to safety engineers

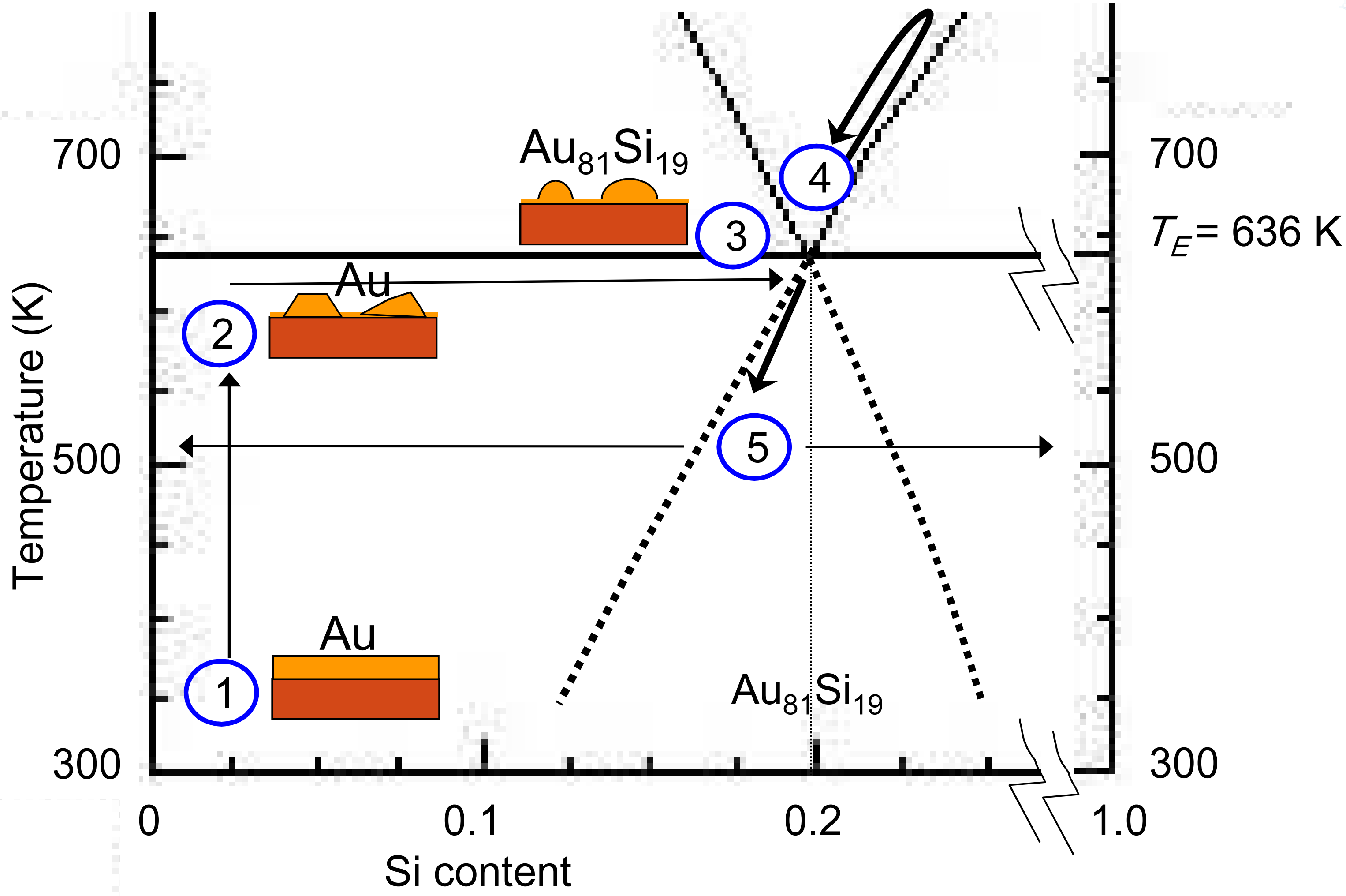
Final safety validation 04/2009

2007: Funding obtained by nanoSCIENCES FONDATION

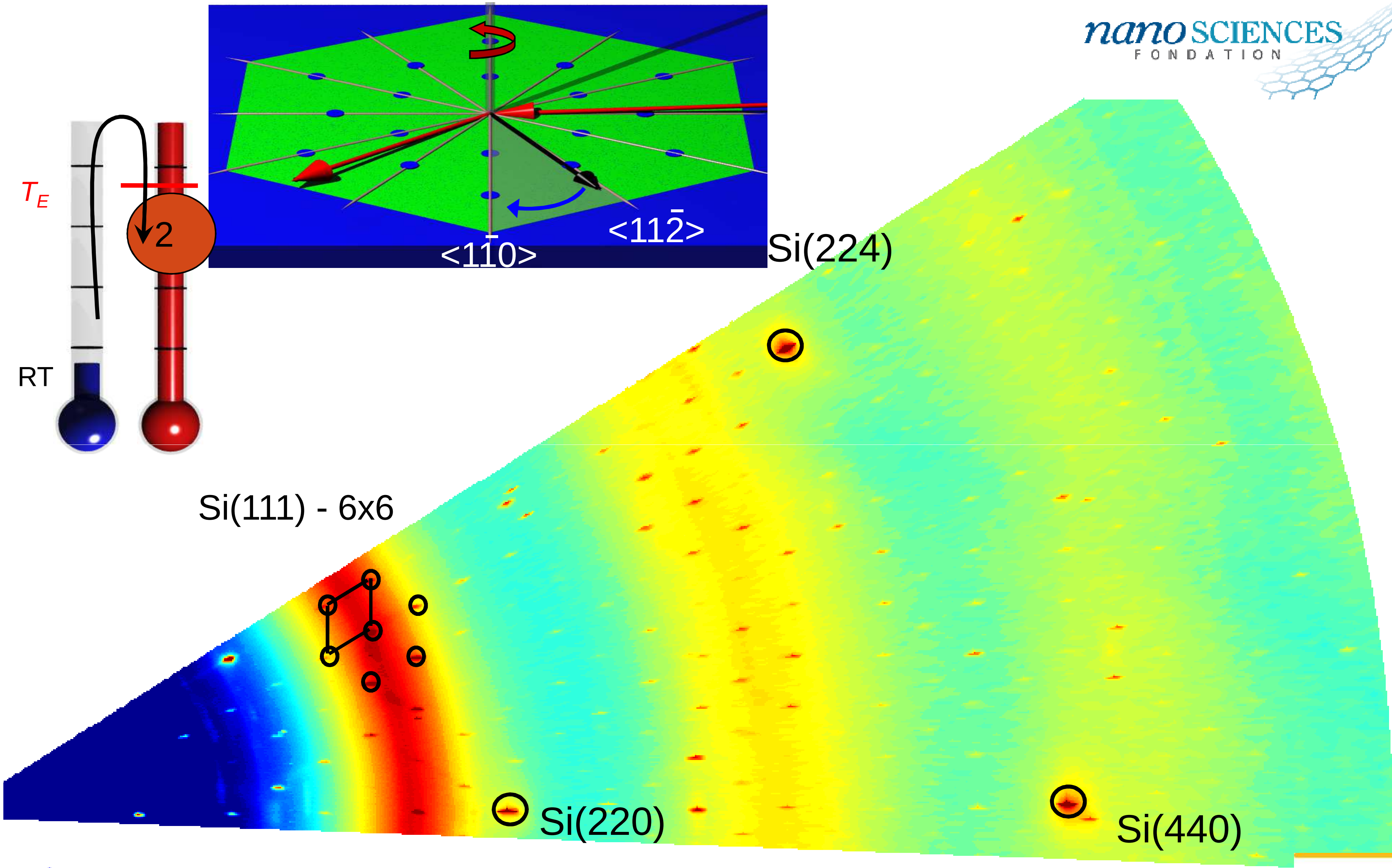
Injector tested, Gas-bottles installed, first wires grown in 2011

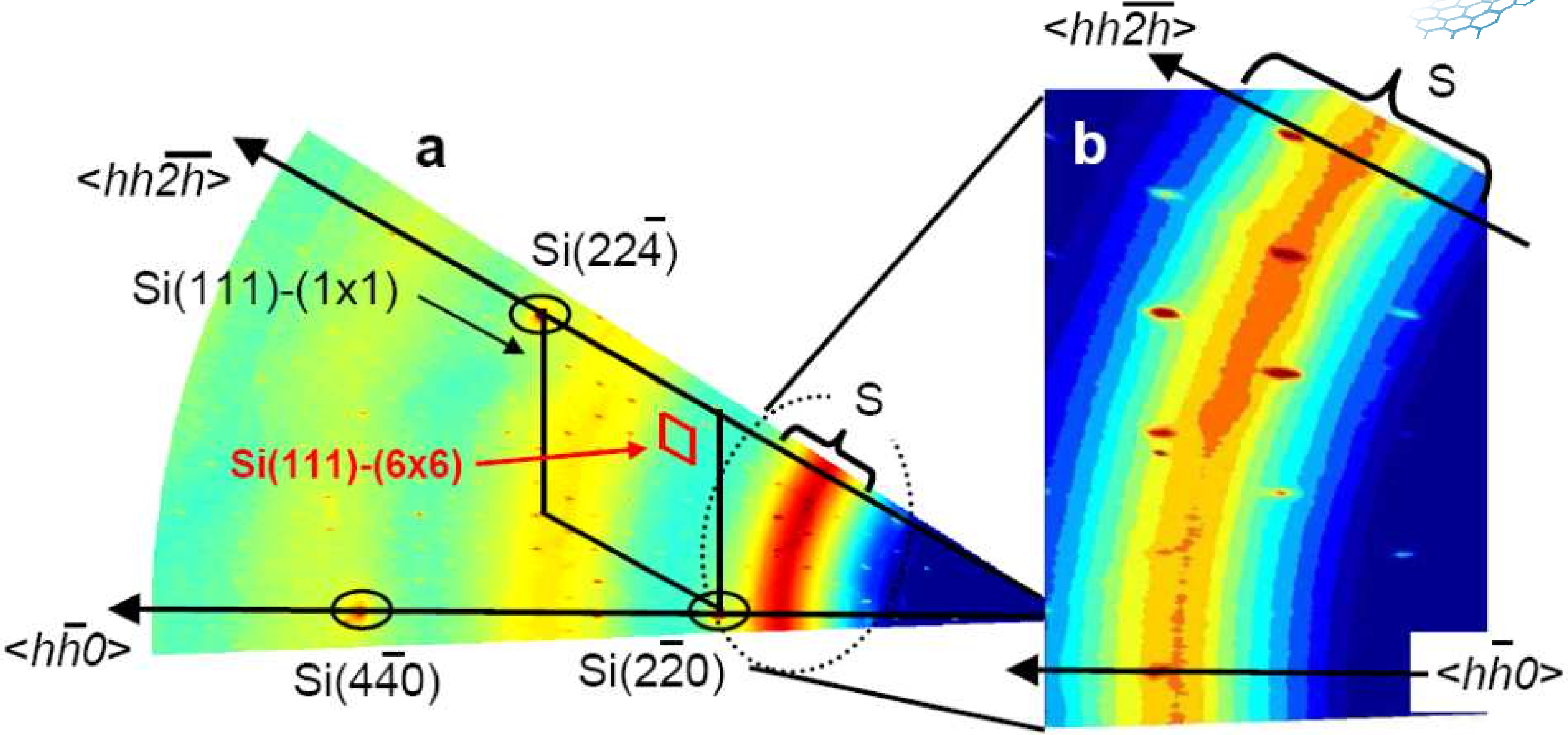


Nucleation and Solid-Liquid Transition of AuSi Nanowire Catalysts on Si(111)



Systematic X-ray measurements performed in all the above different states of the phase diagram.

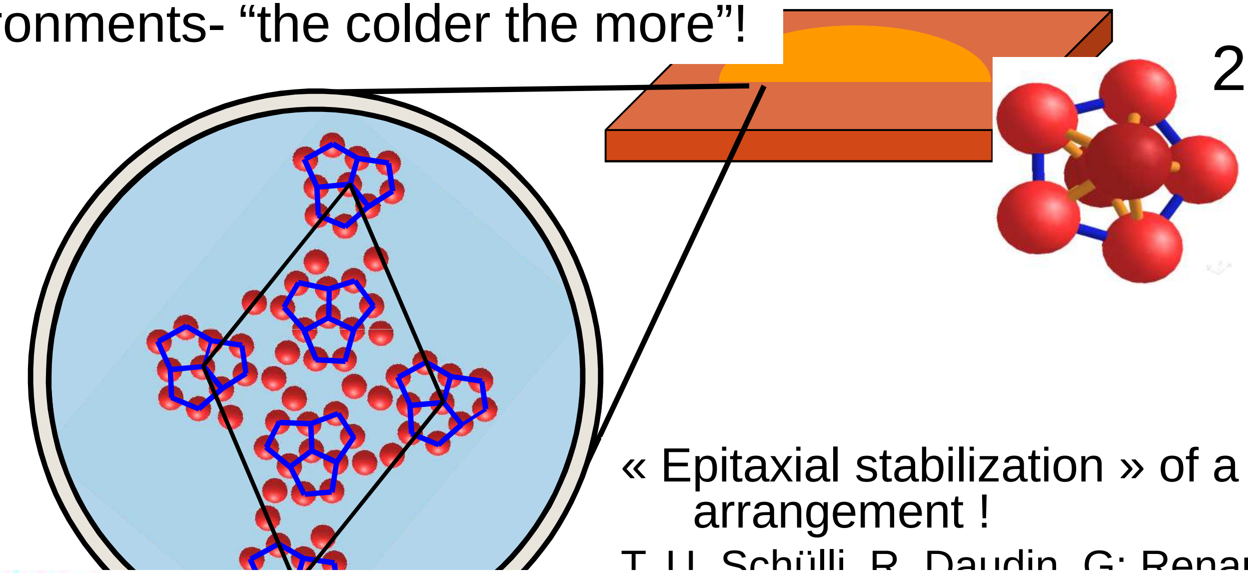

T_E
RT
2
<1$\bar{1}$0>
<11$\bar{2}$>
Si(224)
Si(111) - 6x6
Si(220)
Si(440)



There must be a structural similarity between the 6x6 structure and the short range order of the adjacent liquid layers

T. U. Schülli, R. Daudin, G: Renaud, A. Vaysset, O. Geaymond, A. Pasturel, Nature 464, (2010).

Molecular dynamics simulations of the "free liquid": ~30-40% of the atoms are in icosahedral environments- "the colder the more"!



2.84 Å

Surface Bond length and geometry close to clusters present in the simulated liquid

« Epitaxial stabilization » of a liquid by pentagonal arrangement !

T. U. Schülli, R. Daudin, G: Renaud, A. Vaysset, O.

`Nano Lett 11, 44 (2011).

2.9 Å)

NANO LETTERS

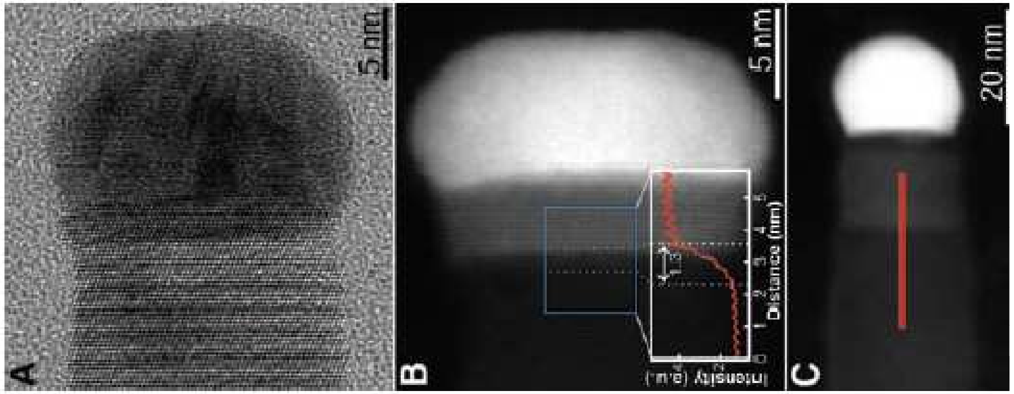
pubs.acs.org/NanoLett

Formation of Wurtzite InP Nanowires Explained by Liquid-Ordering

Rienk E. Algra,[†,‡,§] Vedran Vonk,[§] Didier Wermeille,[||] Wiesiek J. Szweryn,[§] Marcel A. Verheijen,[†] Willem J. P. van Enckevort,[§] Arno A. C. Bode,[§] Wim L. Noorduin,[§] Erik Tancini,[||] Aryan E. F. de Jong,[§] Erik P. A. M. Bakkers,[†,⊥] and Elias Vlieg[*,§]

Conclusion on *in situ* UHV-CVD

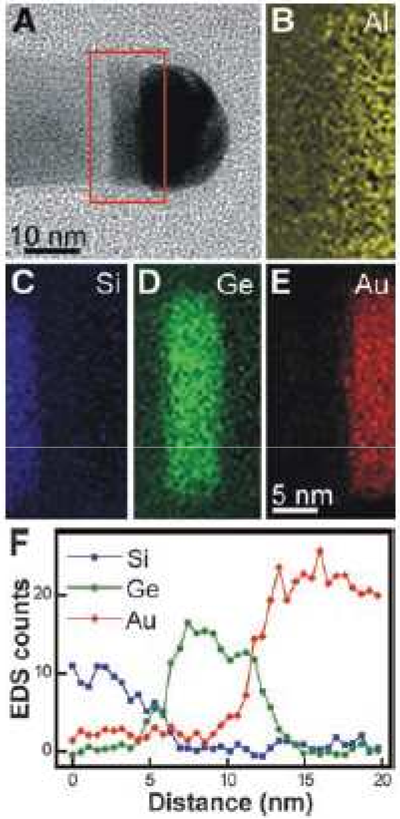
Facility is producing NW using CVD, but NW field is very competitive; in particular NWs are good samples for in situ SE



SCIENCE VOL 326 27 NOVEMBER 2009

Formation of Compositionally Abrupt Axial Heterojunctions in Silicon-Germanium Nanowires

C.-Y. Wen,[1] M. C. Reuter,[2] J. Bruley,[2] J. Tersoff,[2] S. Kodambaka,[3] E. A. Stach,[1] F. M. Ross[2]*



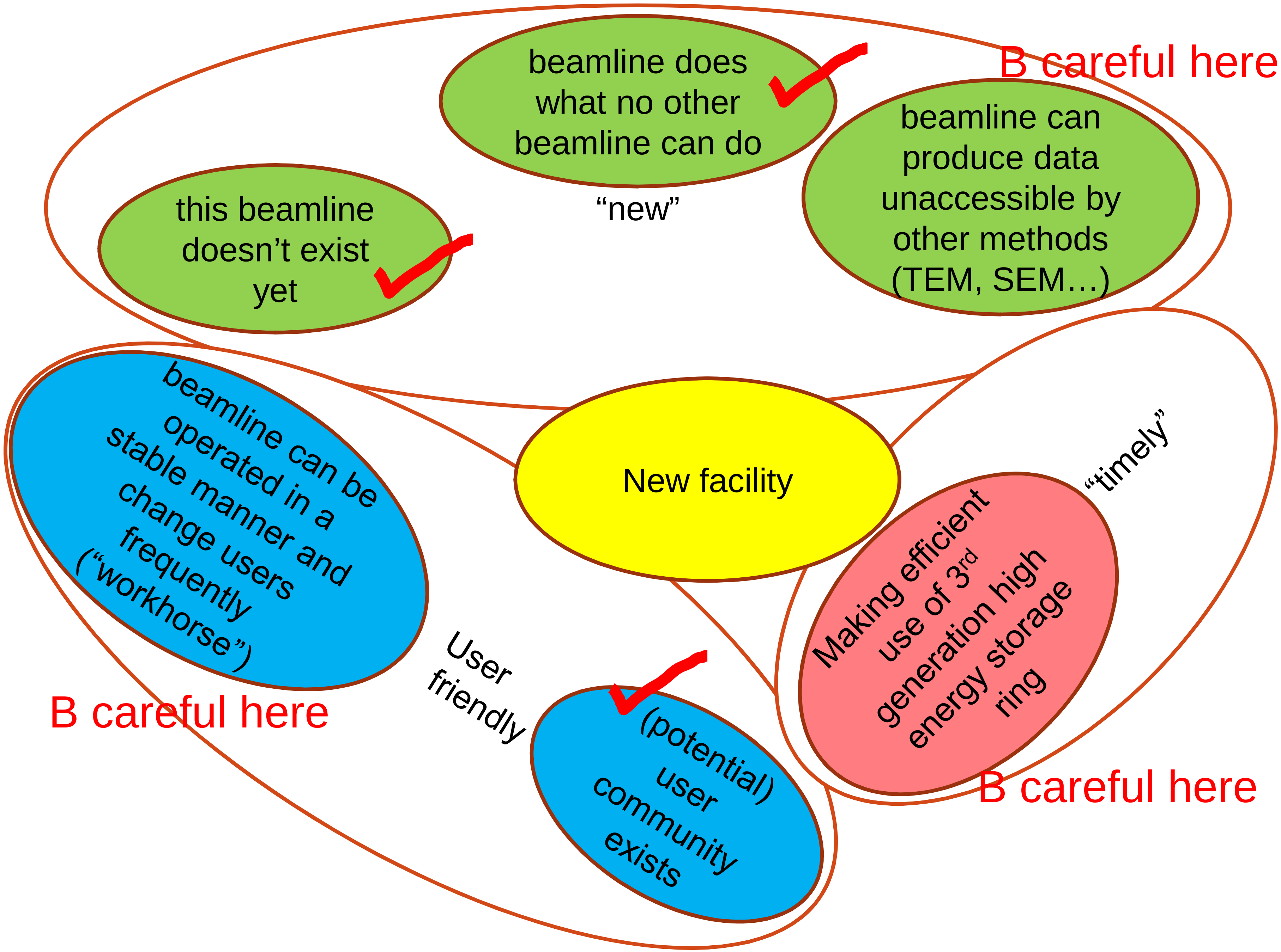
Definitely gas environments are of interest.

Installed on BM32

Above 1 mbar: electron techniques are out of competition

-> catalysis, working fuel cells,…

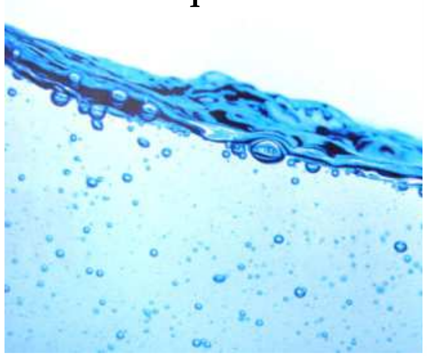
-> a lot to do beyond UHV (simpler sample environment, less dedicated BL!)



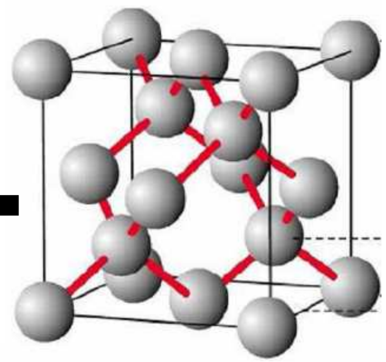
beamline does what no other beamline can do
this beamline doesn't exist yet
beamline can produce data unaccessible by other methods (TEM, SEM…)
"new"
B careful here
New facility
beamline can be operated in a stable manner and change users frequently ("workhorse")
User friendly
(potential) user community exists
B careful here
"timely"
Making efficient use of 3rd generation high energy storage ring
B careful here

Complexity!

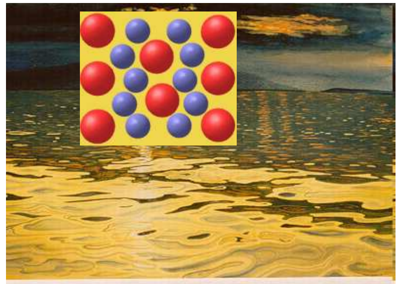
liquid



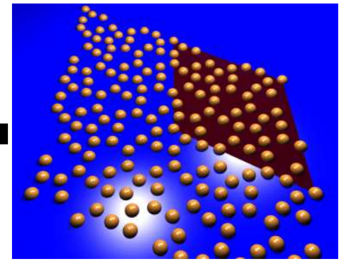
3D solid



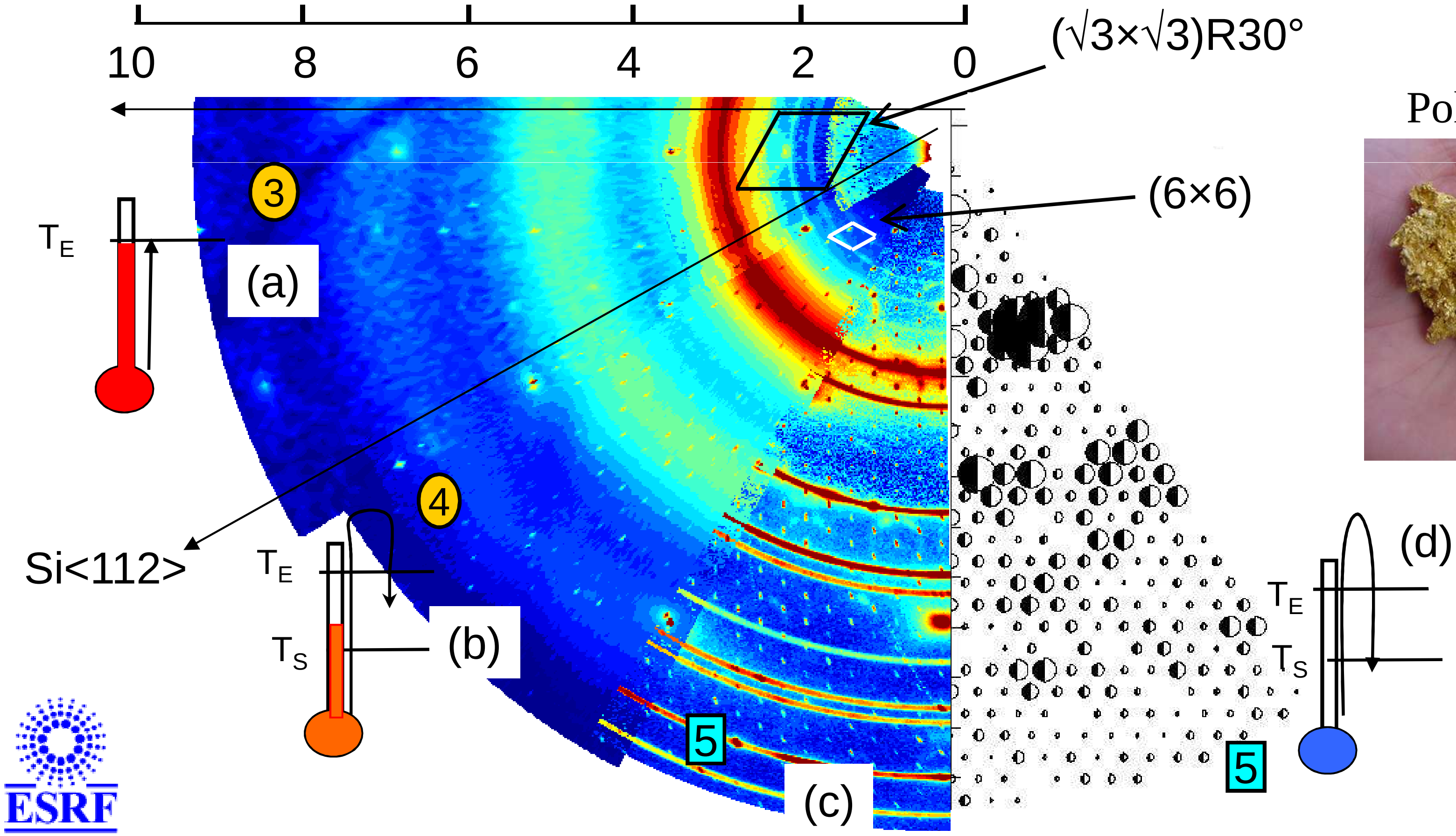
2D AuSi phases floating on liquid AuSi (*Shpyrko et al., Science 2007*)



2D interfacial structures



Polycrystalline Gold



ESRF

Complexity, -from surface science to device structures

More complex growth processes, or reactions involving heterogeneous structures
Open the field for sub-micronic beams

This is one technique where 3rd generation high energy storage rings meet requirements to generate new knowledge in surface and nano science.

Small beams are good to be used
1. When heterogeneity does not allow conclusive measurements with big beams
2. *in operando* experiments require "individual" measurements (electromigration breakdown phenomena in devices, mechanical testing of nanostructures/ wires).

ID01 in the ESRF UPGRADE: Small Beams, Diffraction, Coherence

-Recent activities at **ID01 (since 2000)**: Anomalous and diffuse scattering from nanostructures, mainly grazing incidence techniques, studies of shape, strain and composition.

Since 2007 use of "small beams" for single object properties, exploitation of coherence

-

ESRF+ID01 Upgrade 2008-2017

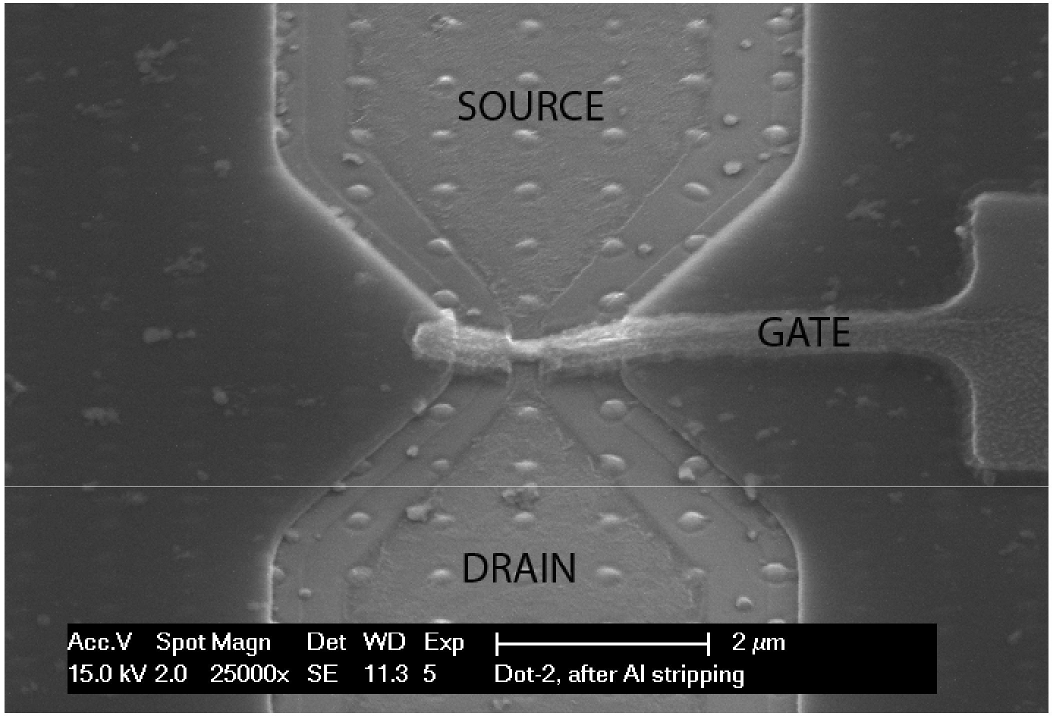
No dedicated diffractometer at that time,
Triggered idea of the upgrade

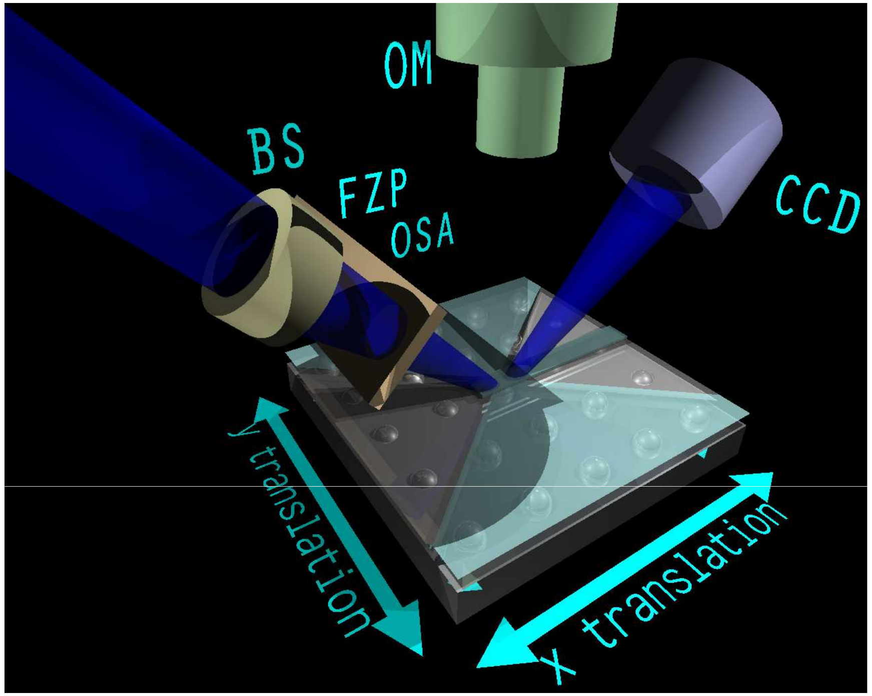
PHYSICAL REVIEW B 77, 245425 (2008)

Beyond the ensemble average: X-ray microdiffraction analysis of single SiGe islands

C. Mocuta,[1] J. Stangl,[2] K. Mundboth,[1,2] T. H. Metzger,[1] G. Bauer,[2] I. A. Vartanyants,[3] M. Schmidbauer,[4] and T. Boeck[4]

Diffraction as a local tool, study of devices:
Shedding light onto the heart of a transistor



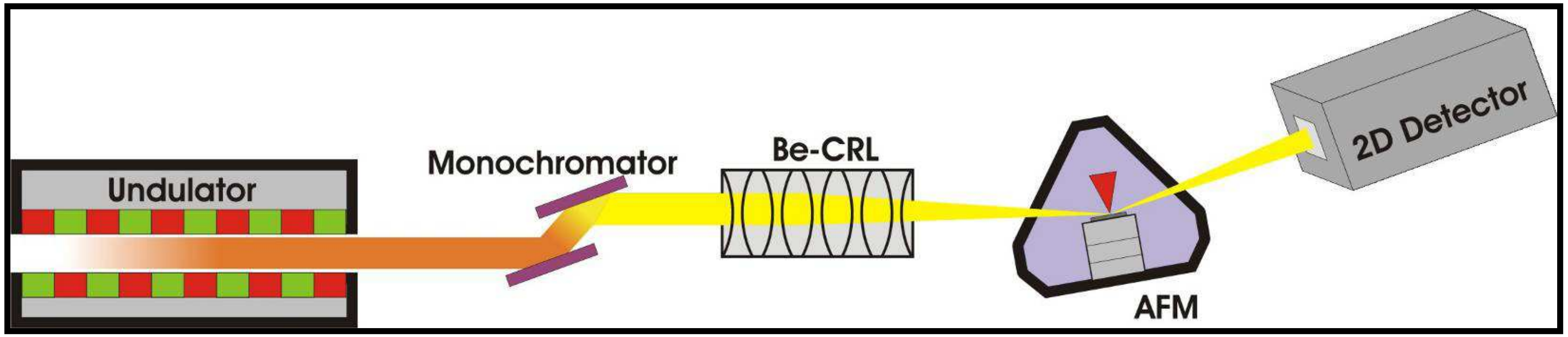


Strain determination in the Si channel above a single SiGe island inside a field effect transistor using nanobeam x-ray diffraction

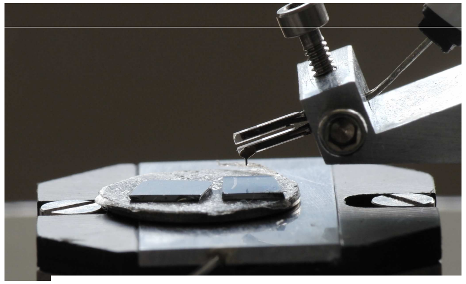
N. Hrauda et al. *Nano Letters* 2011, **11**, 2875

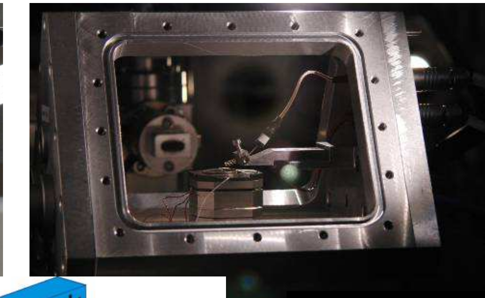
Experimental setup for elastic studies on nanoscale

Combination of *in-situ* AFM and microfocused XRD

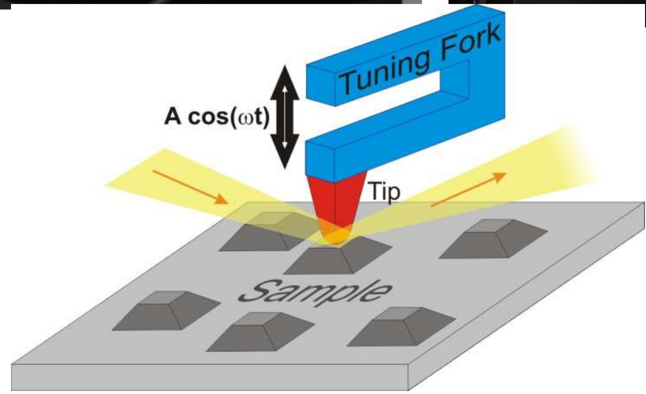


- X-ray beam focused to 1.5 x 3 mm^2 by Be-CRLs
- AFM constructed by Small Infinity™
- AFM used both to image and to manipulate single nanostructures

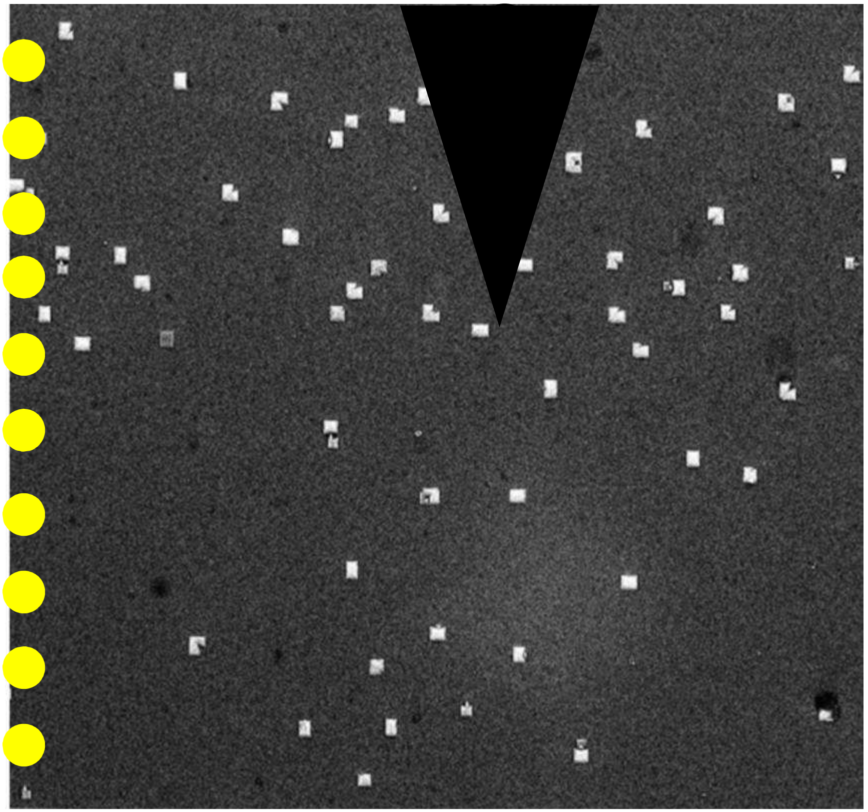


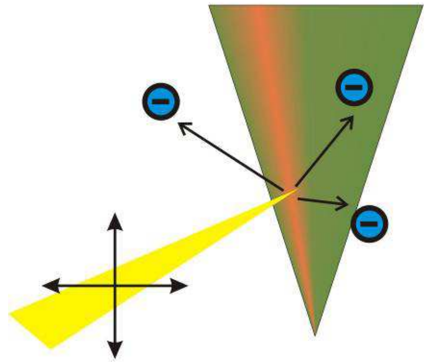


Small Infinity



Sample alignment

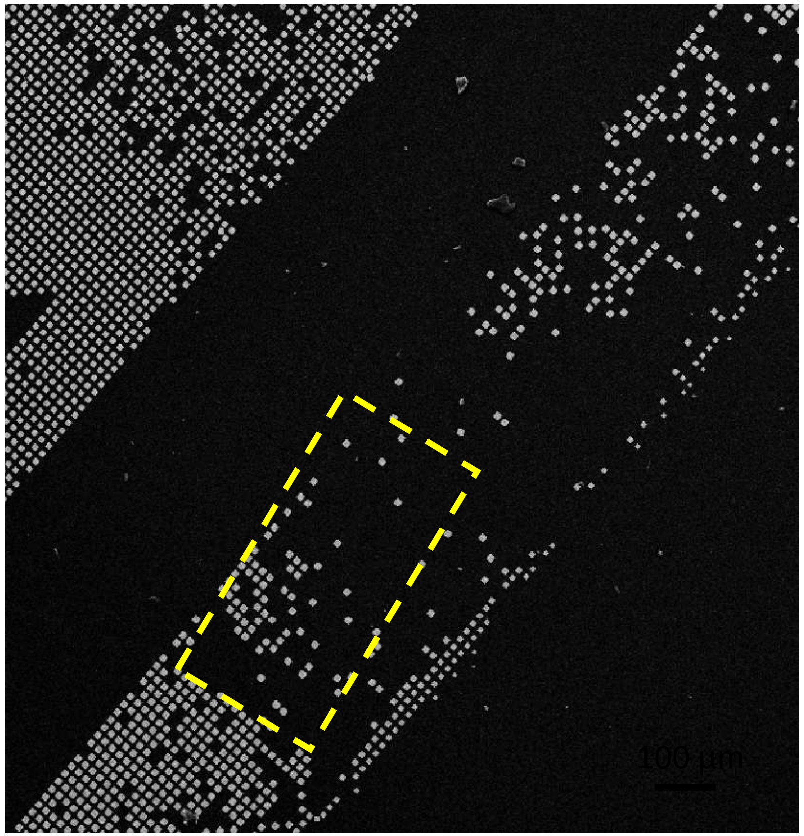




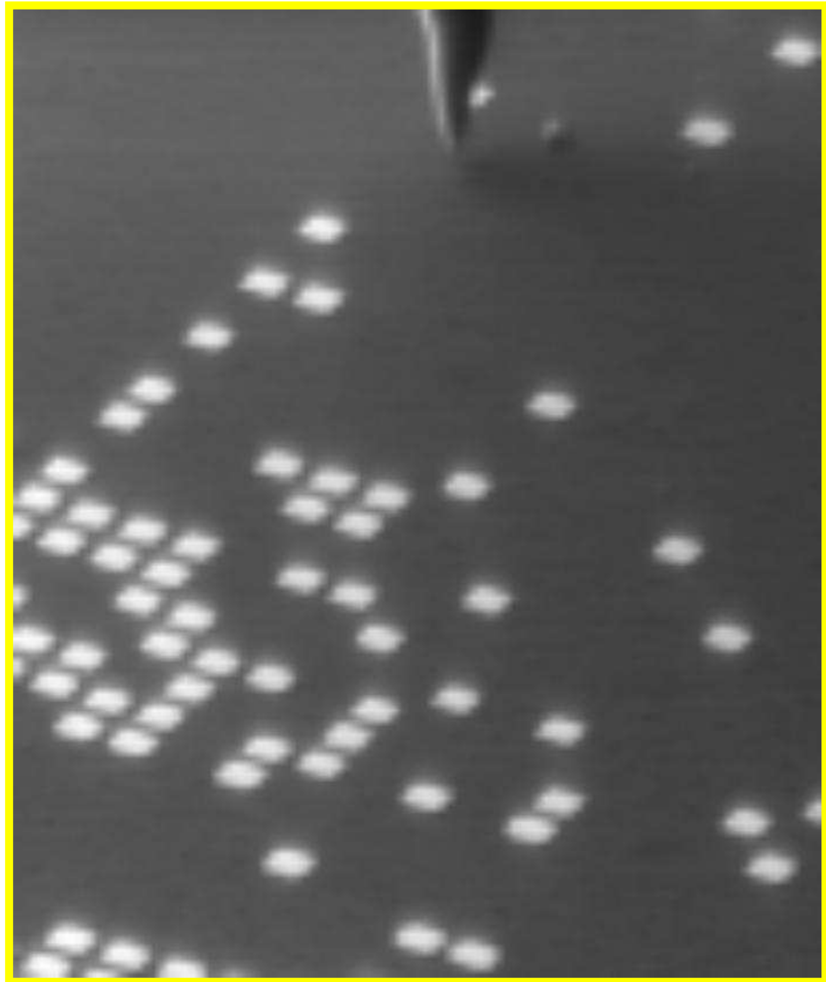
X-ray excited **photoelectrons** can be used to **image** AFM tip

Simultaneously to photocurrent acquisition, **scattered X-rays** recorded

Sample alignment

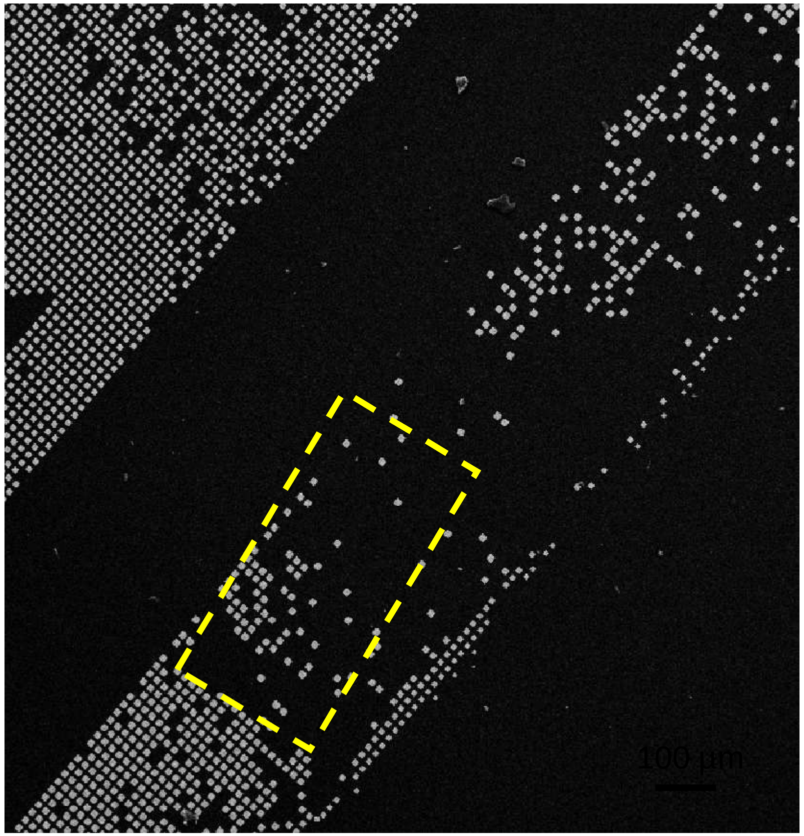


SEM



Photocurrent imaging (PI)

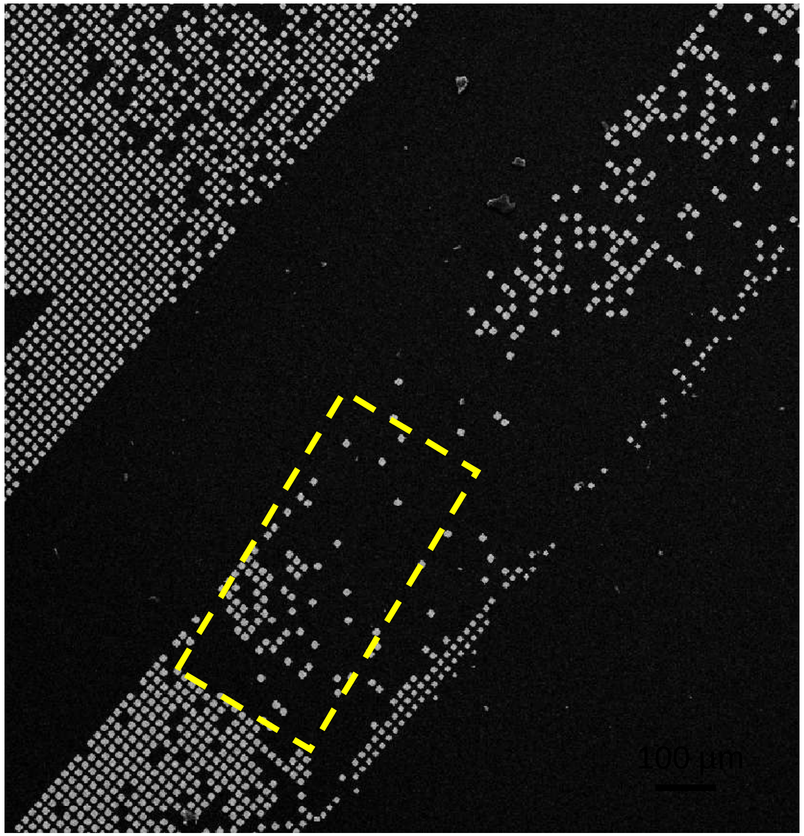
polycrystalline Bi islands with grain size ~500 nm



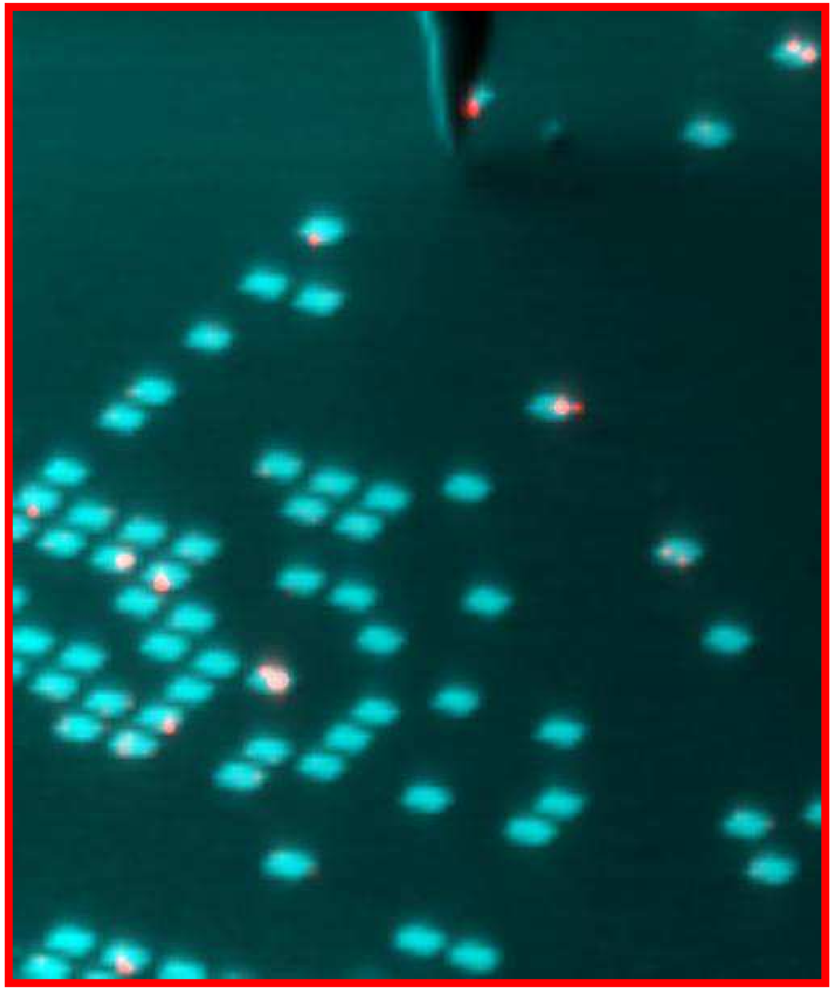
SEM

XRD

polycrystalline Bi islands with grain size ~500 nm



SEM



PI + XRD

polycrystalline Bi islands with grain size ~500 nm

New diffractometer required

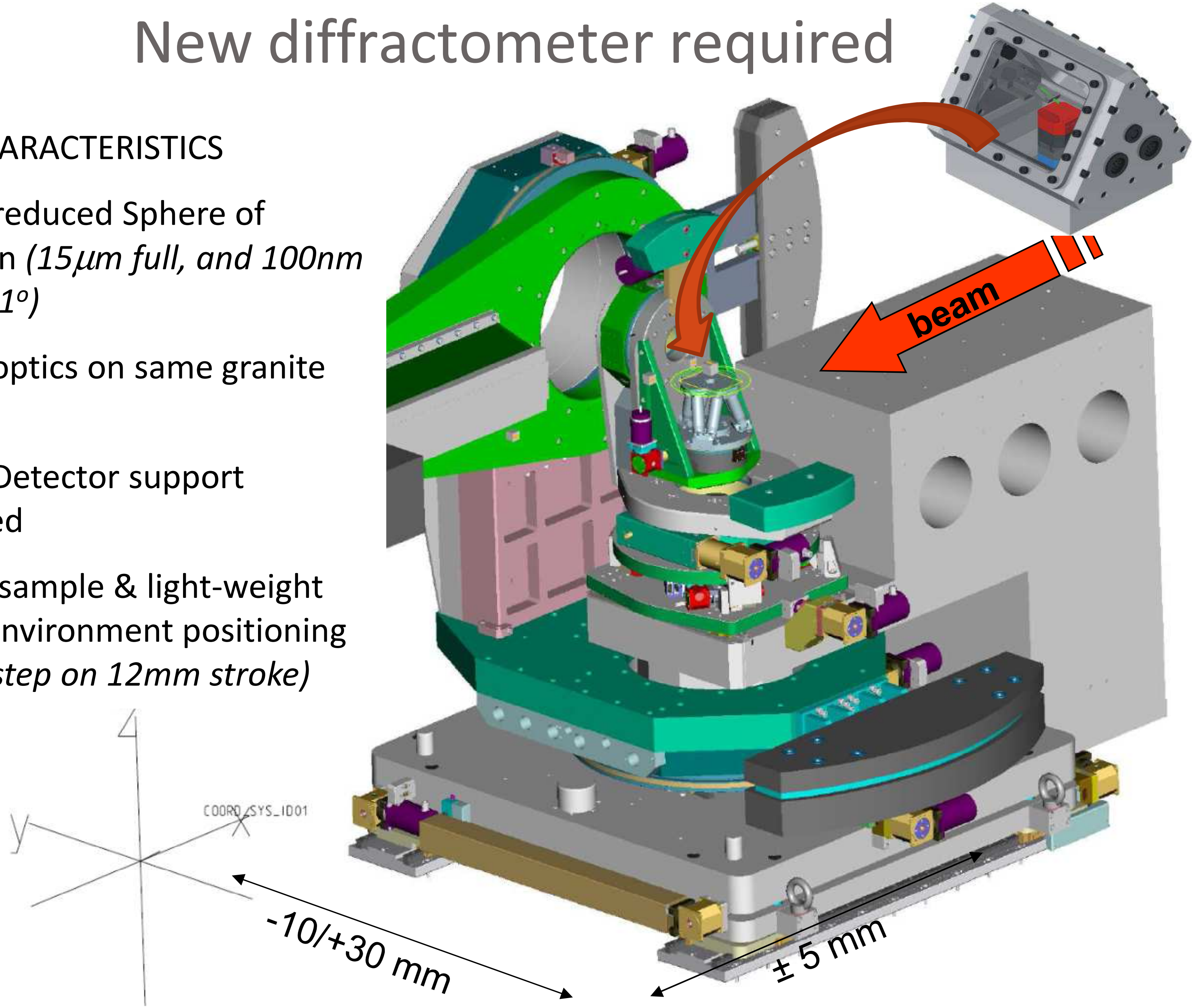
CHARACTERISTICS

3+2 circles - reduced Sphere of Confusion *(15μm full, and 100nm within +-1^o)*

Sample and optics on same granite table

Sample and Detector support decoupled

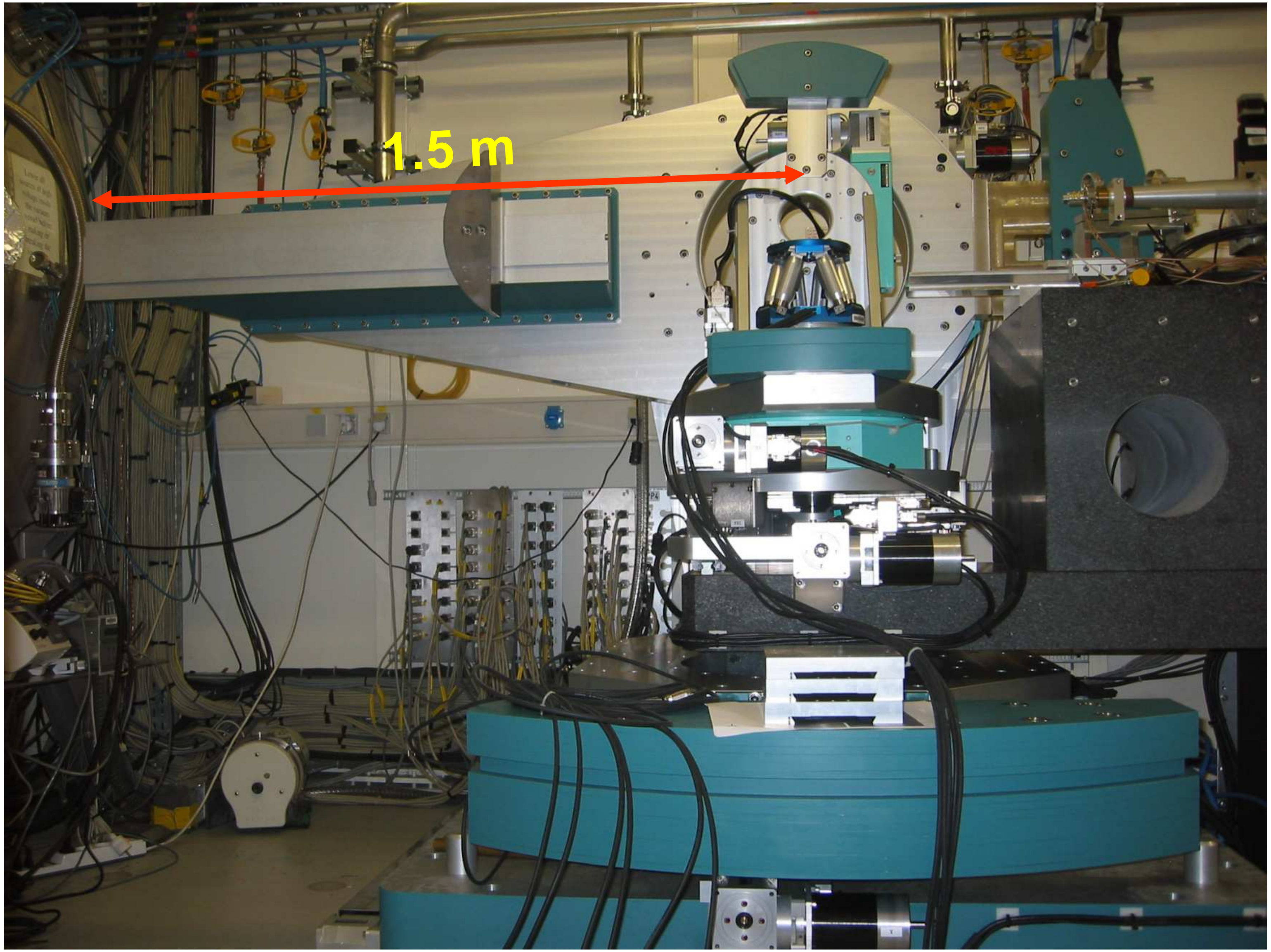
Hexapod for sample & light-weight sample environment positioning *(100nm step on 12mm stroke)*



New Huber diffractometer

Arrival x-mas 2010 installation January 2011

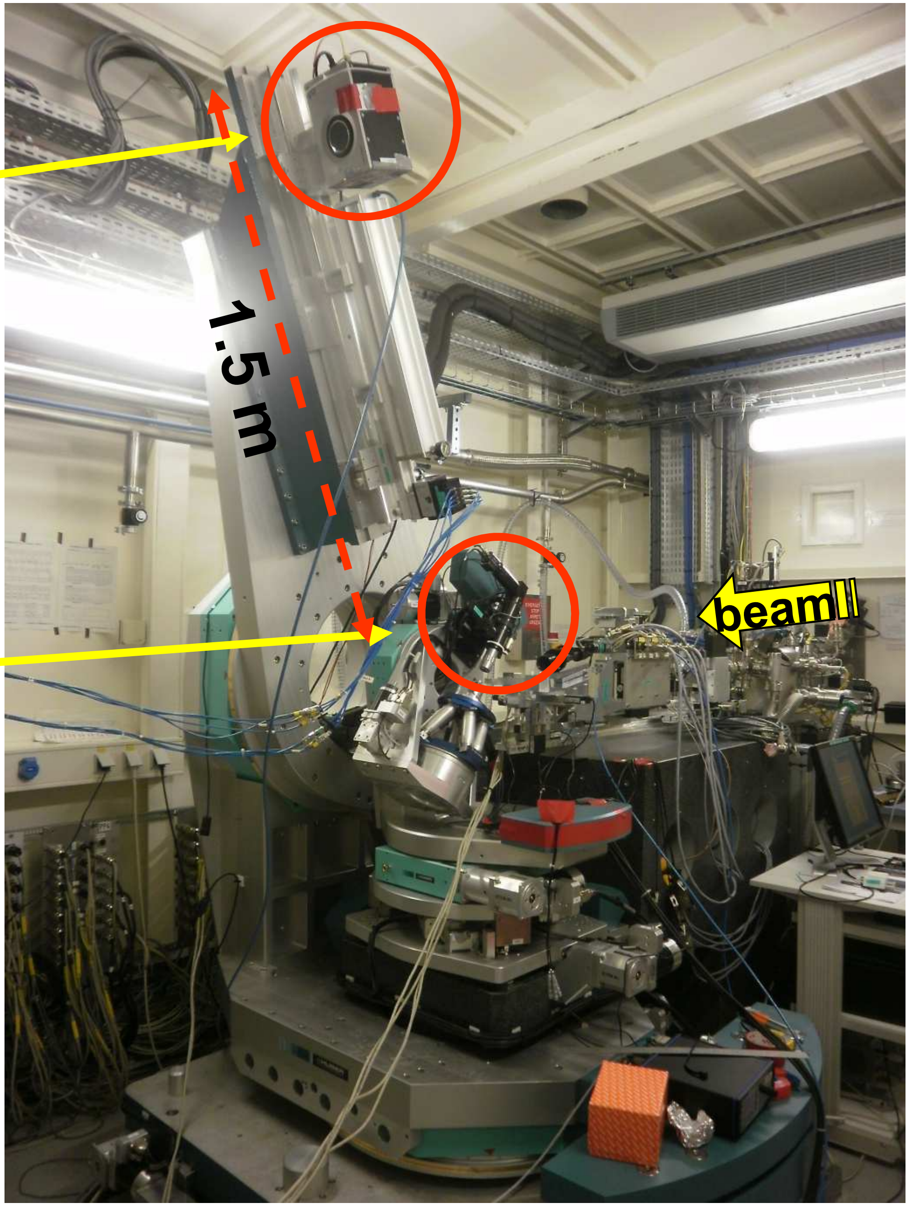
New Huber diffractometer



new Maxipix
with 4 chips
(512 x 512 pixels)

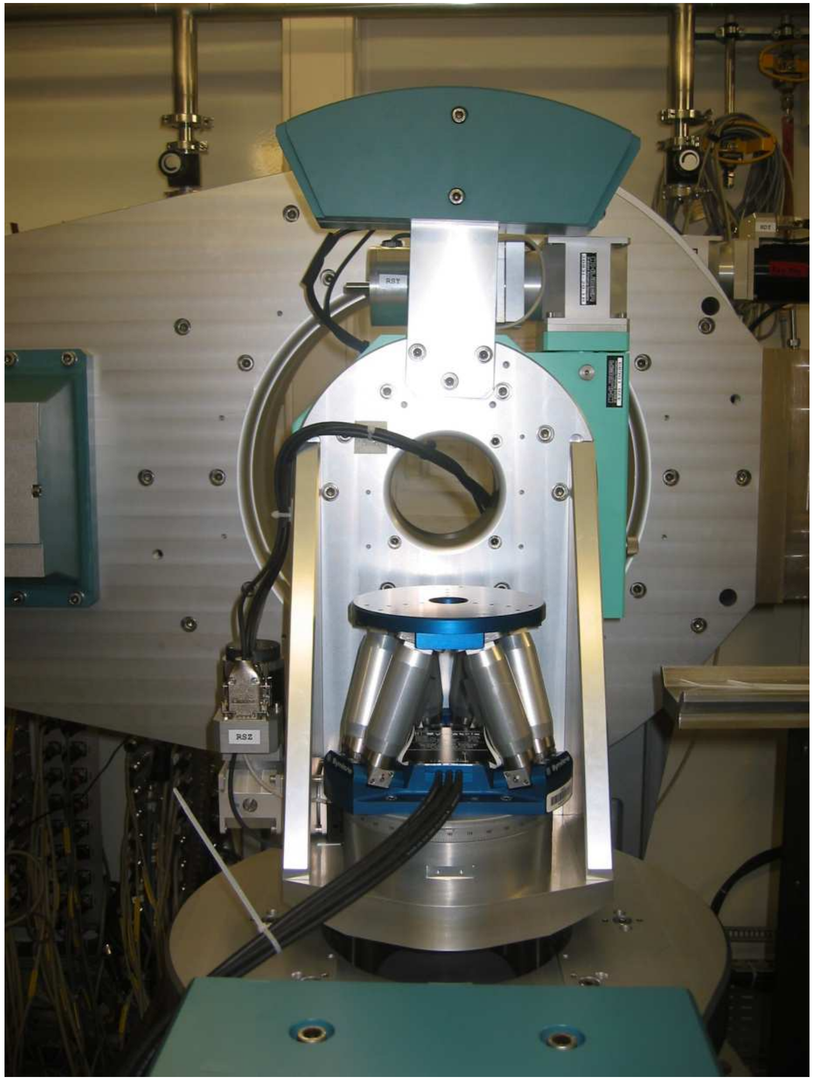
allows image
accumulation

new optical
microscope
(resolution: 500 nm)



detector arm and
sample stage
mechanically
decoupled

Symétrie hexapod



translation
x, y, z = ± 5 mm
Δx, Δy, Δz = 100 nm

rotation
$\Omega = \pm 5°$

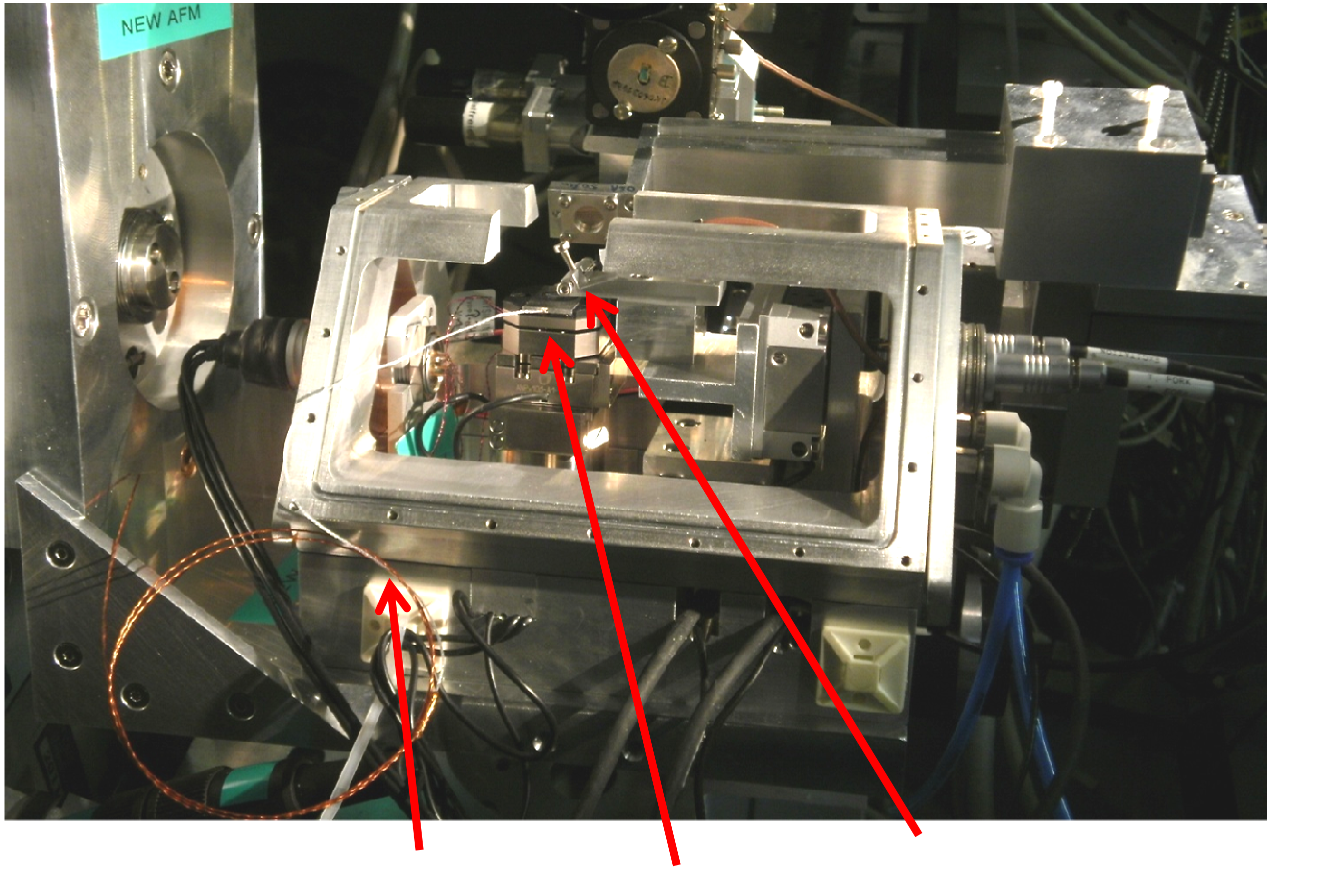
goniometer
$\alpha, \beta = \pm 8°$
$\Delta\alpha, \Delta\beta = 2.5$ µrad

supportable weight
2 – 5 kg

Sample scanner+environments

Keep things as lightweight as possible:
-low elastic deformation during angular change
-use of piezo scanners together with sample environments

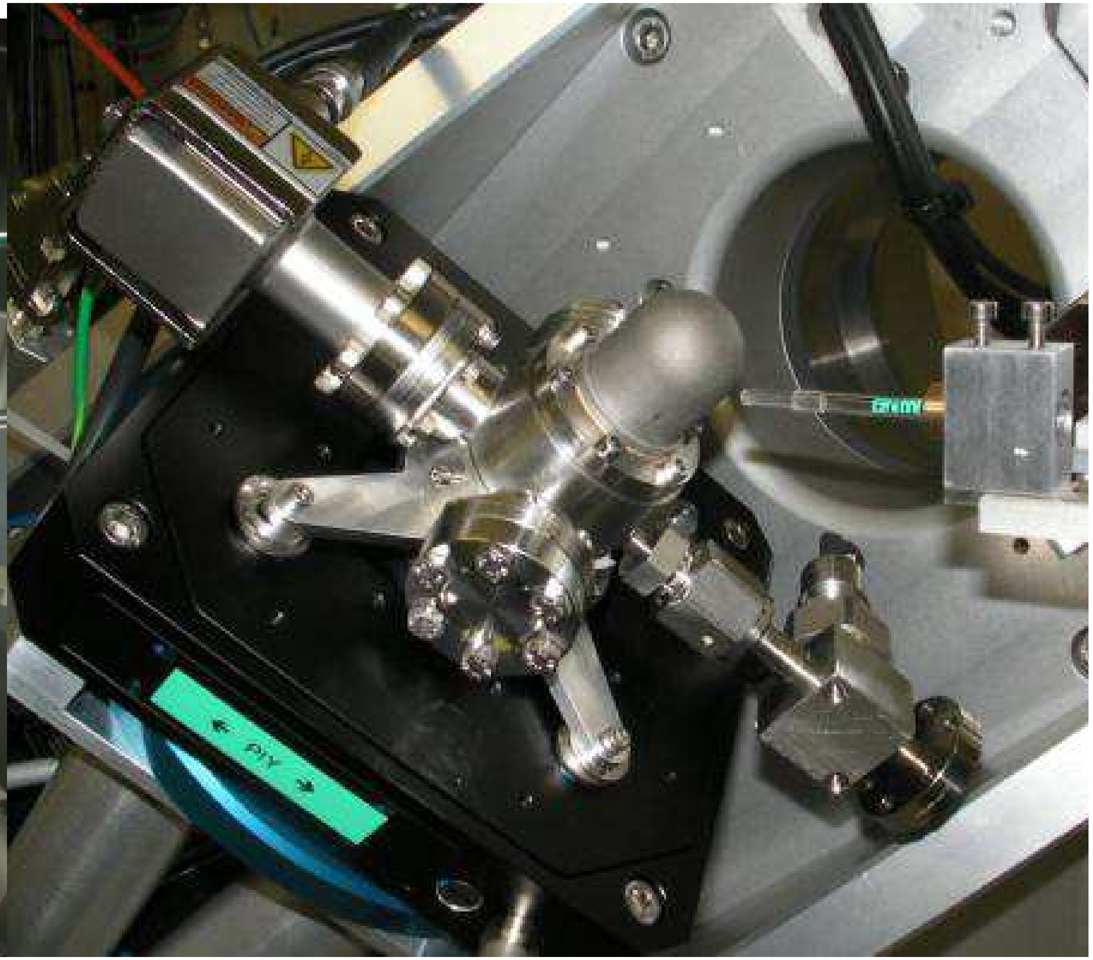
Like this: ….



AFM housing with sample and tip on piezo positioners

Sample scanner+environments

…or like this:



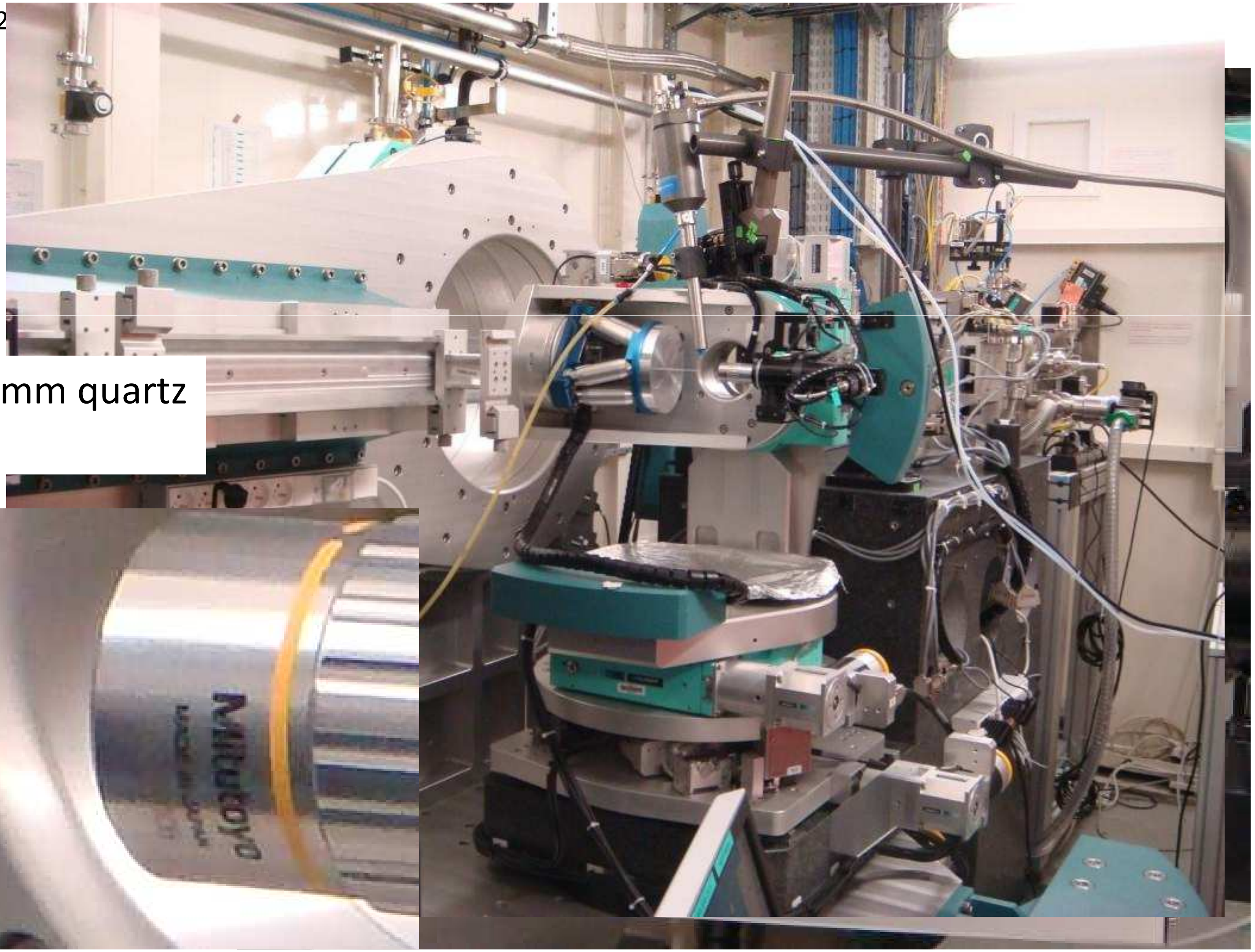
Highly compacted light weight AFM (400 g) inside vacuum chamber.
Ensemble is carried on a piezo positioner (black table).

HV chamber "the Kilo"

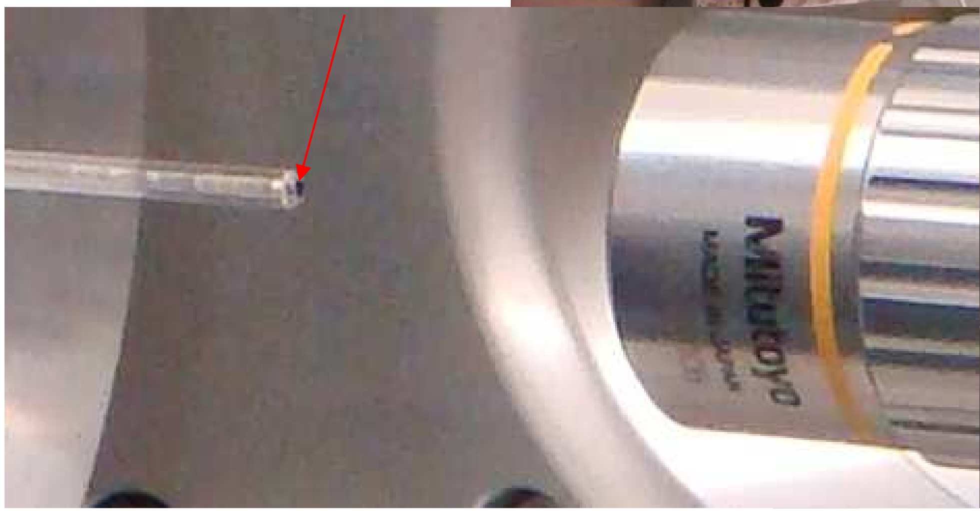
Grazing incidence diffraction from " difficult" samples

Grazing incidence diffraction (GID) from a "small" (5 mg) crystal:
e.g. facet < 1x 1 mm^2, incidence at critical angle of 0.2 º: efficient beam size: 3-4 μm

Focusing in the plane of incidence with 1D Be lenses (typically at 6.5-15 KeV), resulting beam size: 5x1000 μm^2

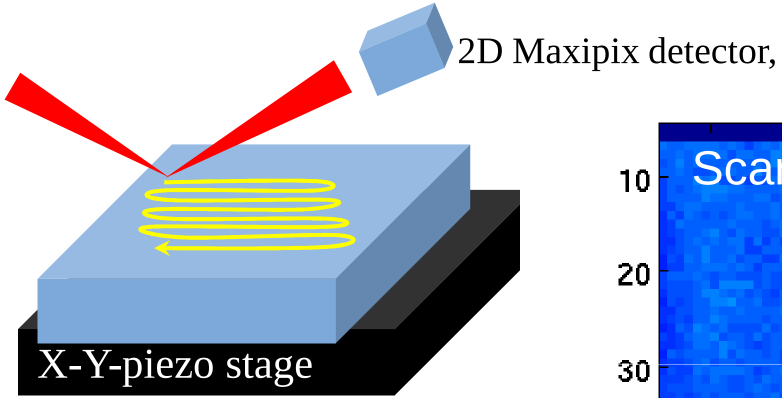


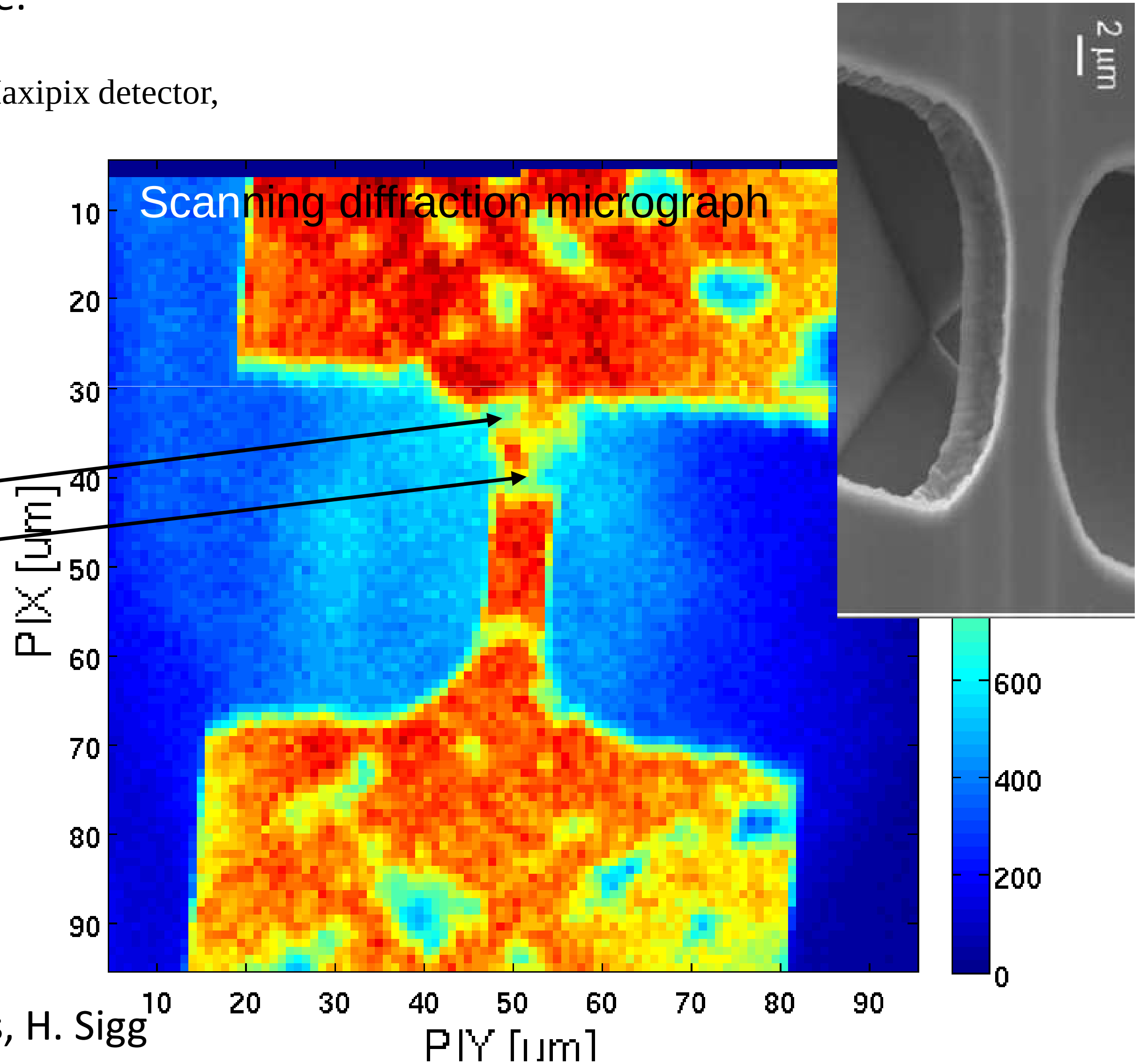
Small crystal fixed to a 3 mm quartz rod investigated in GID



scanning diffraction microscopy : strain mapping

Continuous motion of the piezo-stage and synchronized acquisition of images from 2D detector and position from stage.





100x100 points map with micrometer resolution: The low intensity parts are highly strained

Enormous gain in time

EX: 100x100 points

-SPEC> 100x100x(1s+2,7s)= 37000 s~10 hours

-FS=> 100x100x0.04=400s

Image courtesy: A. Diaz, M. Süss, H. Sigg

Hutch and diffractometer environment

Nano hutch checklist:

-silence !

Throw primary vacuum pumps out
watch over all sources of vibrations at low frequencies (fans of turbo pumps)

-thermal stability
Avoid unnecessary heat sources (Halogen lamps)

Separately ventilate electronics which have to be close to experiment

Thermally insulate hutch

-rapid achievement of thermal equilibrium

ID01 upgrade: Energy specifications and undulator combination

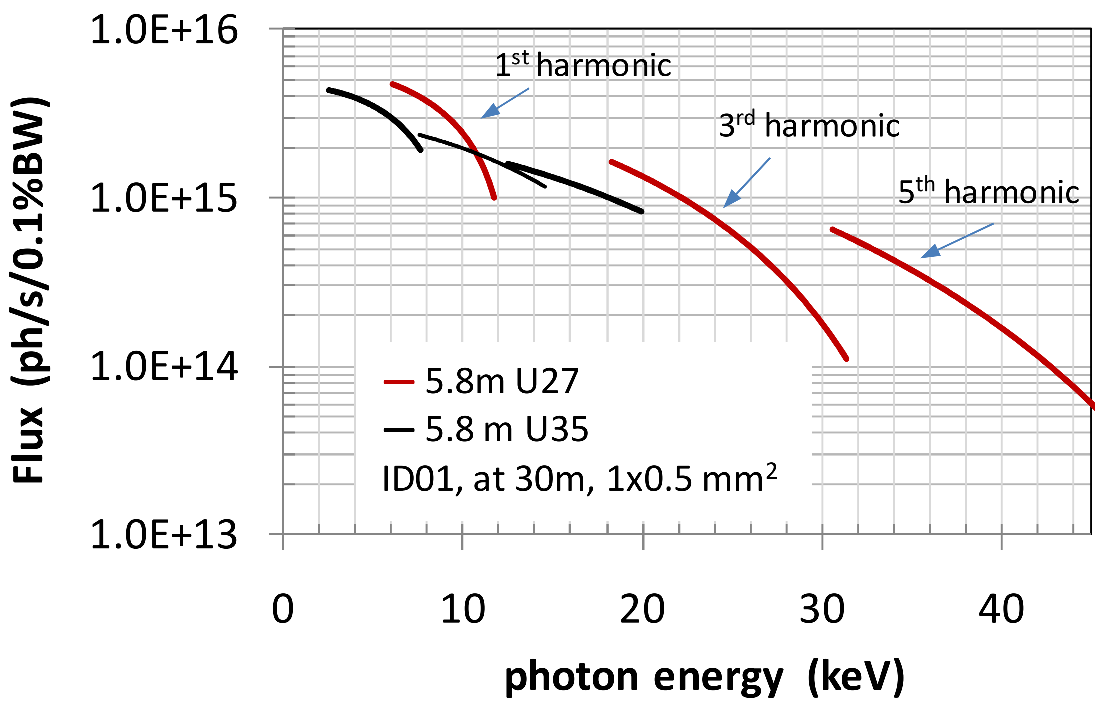
U35/ U27 (5.8 m) revolver setup to cover *complete energy ((2.1…4.2-42…60 keV)* range and optimize 3 energy regimes:

6-11 KeV: Coherence+Nanofocussing on 1st harmonic

18-25 KeV: Nanofocusing+ Diffraction

30-35 KeV: 1D/2D microfocusing/ transmission geometries

Higher energies ease the use of “beryllium-free” sample environments



New id01 endstation at 115 m:

Granit bench and precision diffractometer

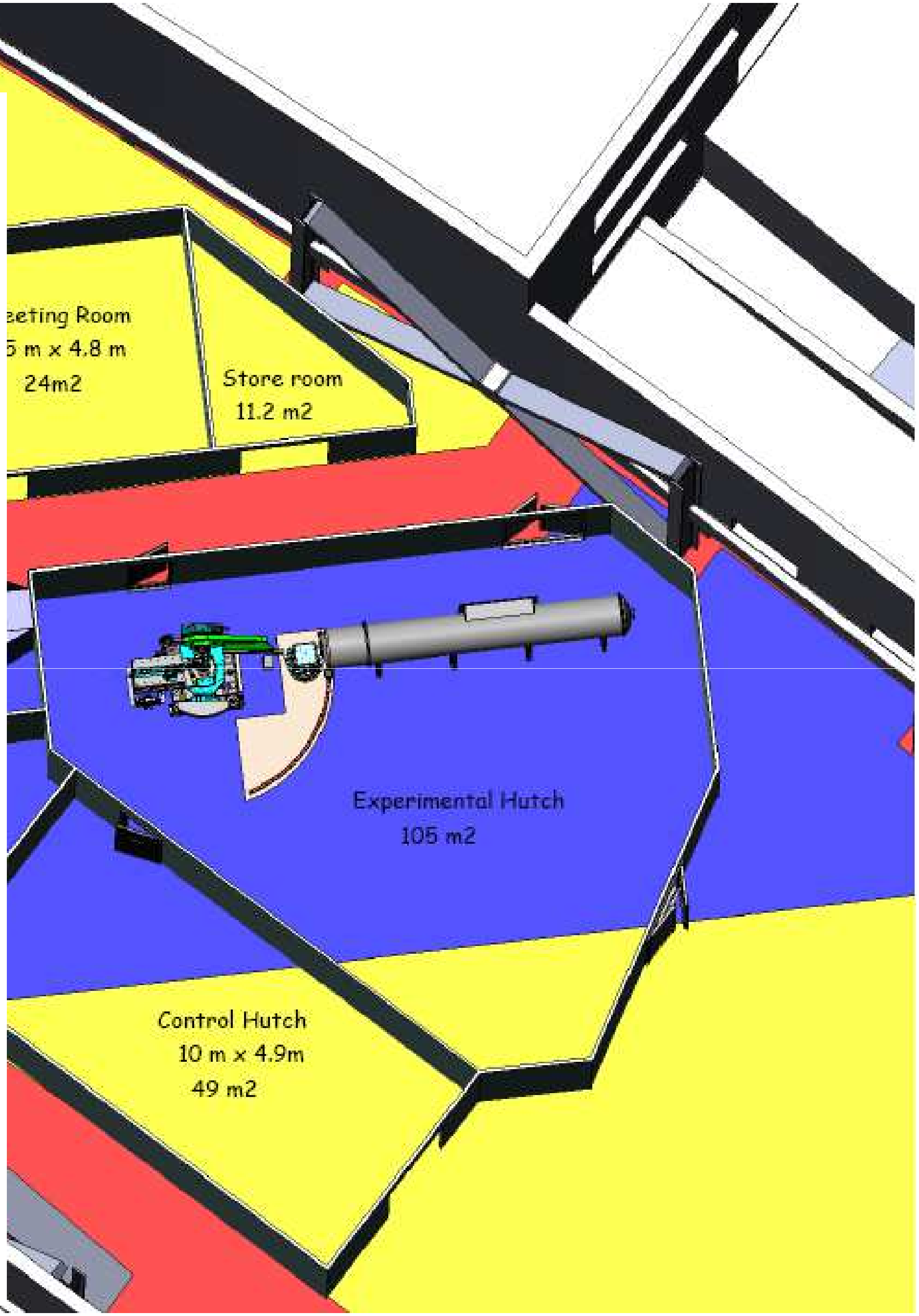
Easy choice of energy and focusing device
Beamsizes 50 nm to 5 mm.

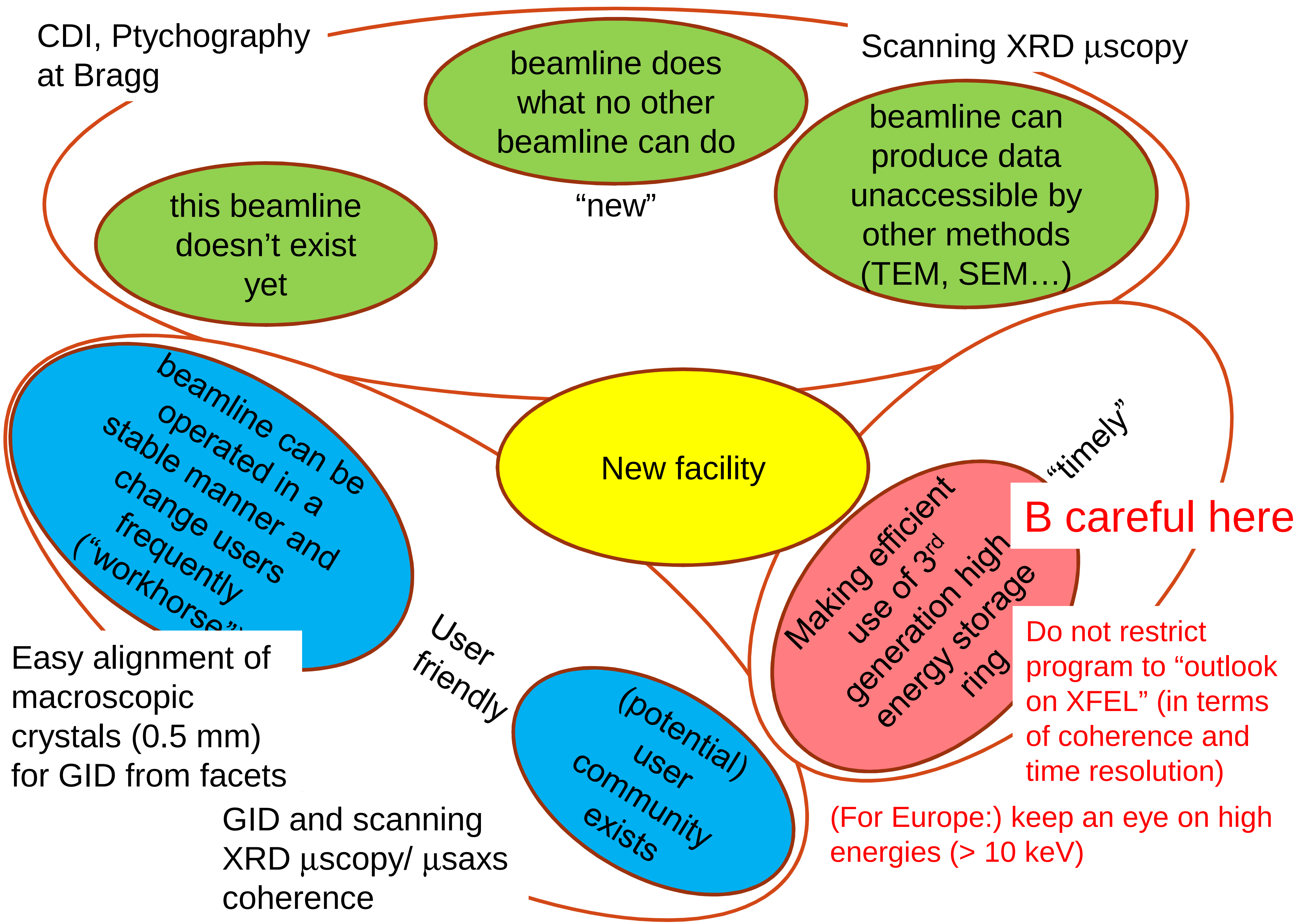
Long “saxs” tube available, also as 5 m detector arm for Coherence, Ptychography

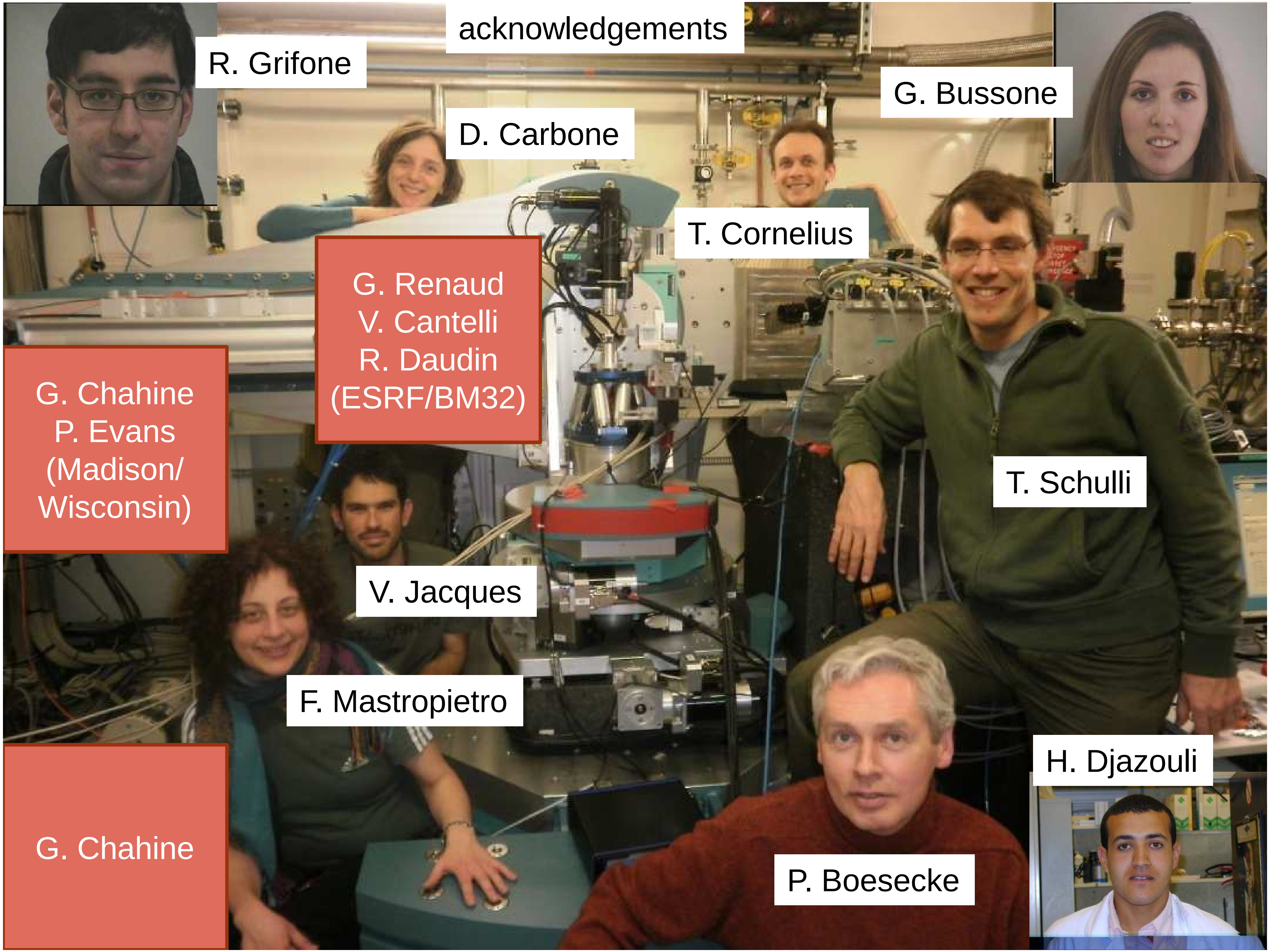
Nano-compatible, compact sample environments (HV, reaction chambers, cryostat) prepared in house.

Heavy stage available for up to 150 Kg user equipment (horizontal scattering geometry only)

-> applied surface and “nanostructure” science (devices with heterogeneity on 100 nm scale, ageing processes, process steps in fabrication…)




CDI, Ptychography at Bragg
beamline does what no other beamline can do
Scanning XRD µscopy
beamline can produce data unaccessible by other methods (TEM, SEM…)
this beamline doesn't exist yet
"new"
beamline can be operated in a stable manner and change users frequently ("workhorse")
New facility
"timely"
B careful here
Making efficient use of 3rd generation high energy storage ring
Do not restrict program to "outlook on XFEL" (in terms of coherence and time resolution)
User friendly
(potential) user community exists
Easy alignment of macroscopic crystals (0.5 mm) for GID from facets
GID and scanning XRD µscopy/ µsaxs coherence
(For Europe:) keep an eye on high energies (> 10 keV)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V. Cantelli
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P. Evans
(Madison/
Wisconsin)
T. Schulli
V. Jacques
F. Mastropietro
G. Chahine
H. Djazouli
P. Boesecke