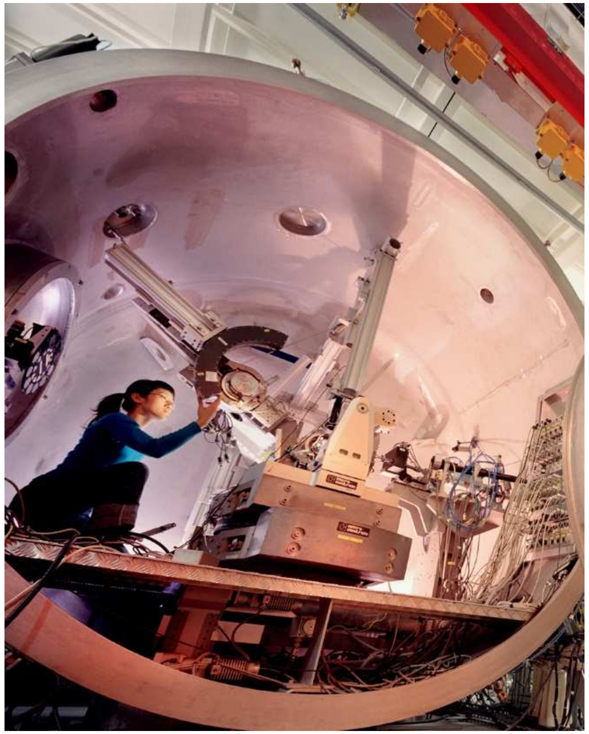
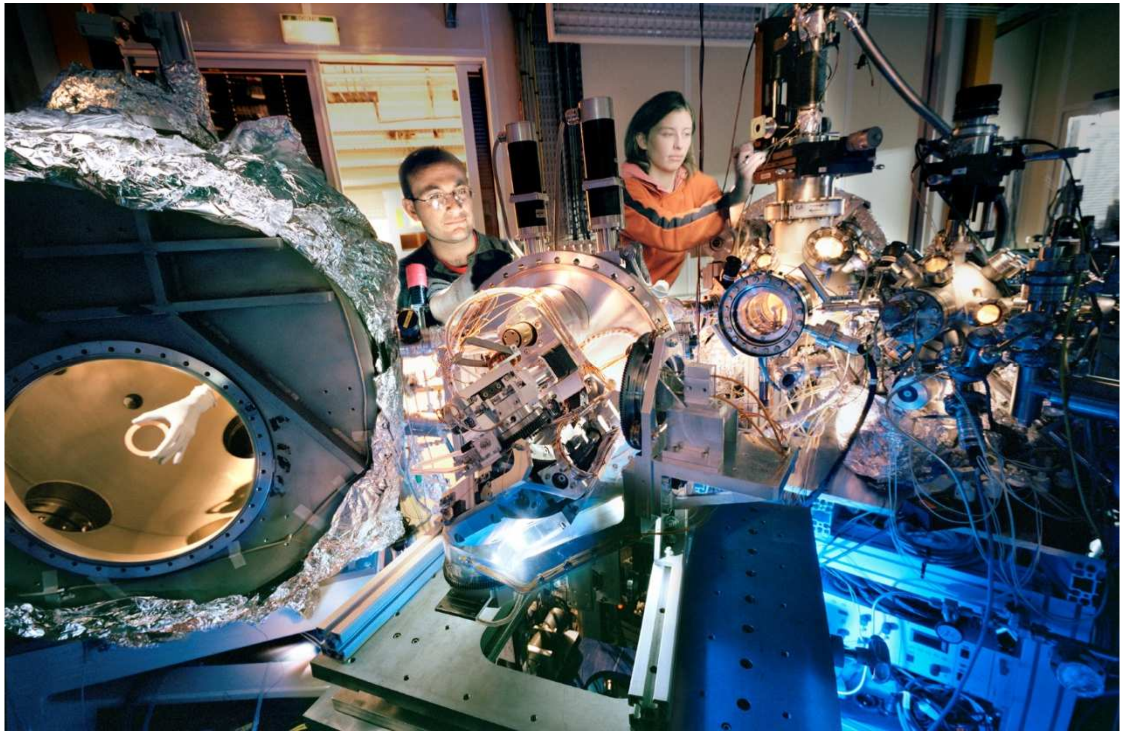
ESRF | The European Synchrotron

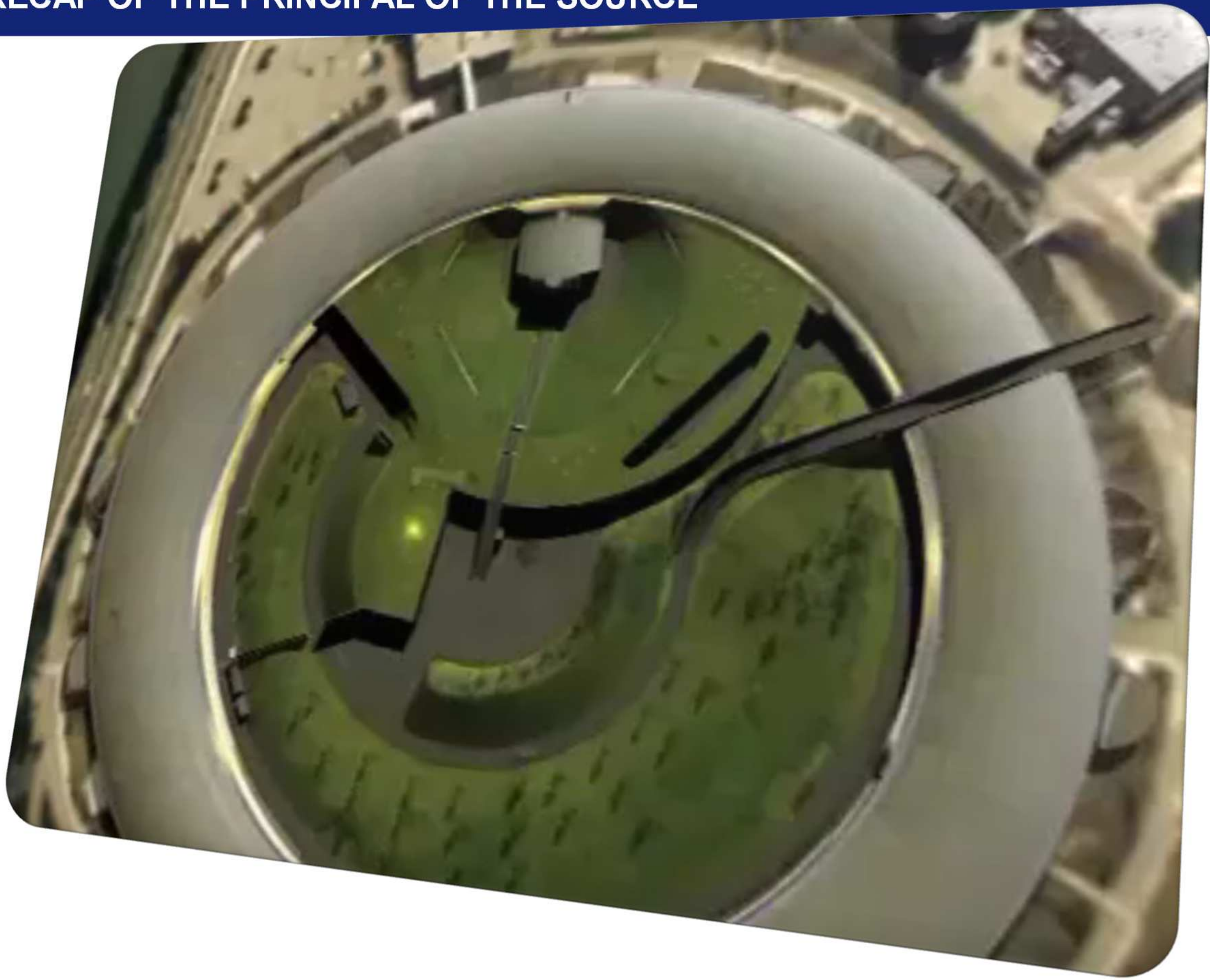
X-RAY INSTRUMENTATION



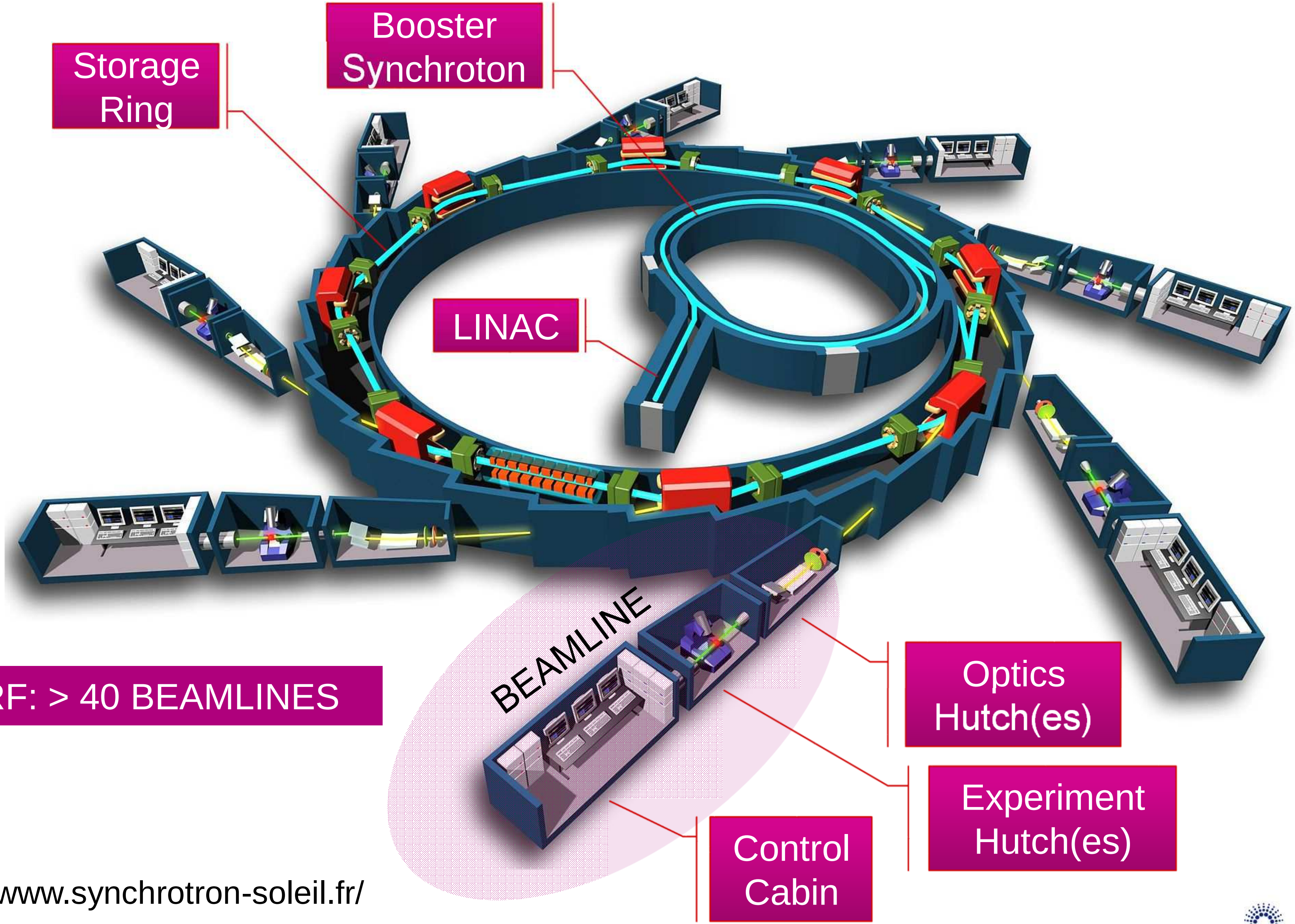


R. Barrett
X-ray Optics Group Leader
Instrumentation Services & Development Division
ESRF

RECAP OF THE PRINCIPAL OF THE SOURCE



SCHEMATIC OF A SYNCHROTRON RADIATION (SR) LIGHT SOURCE



ESRF: > 40 BEAMLINES

http://www.synchrotron-soleil.fr/

A TYPICAL BEAMLINE LAYOUT

Lead Radiation shielding

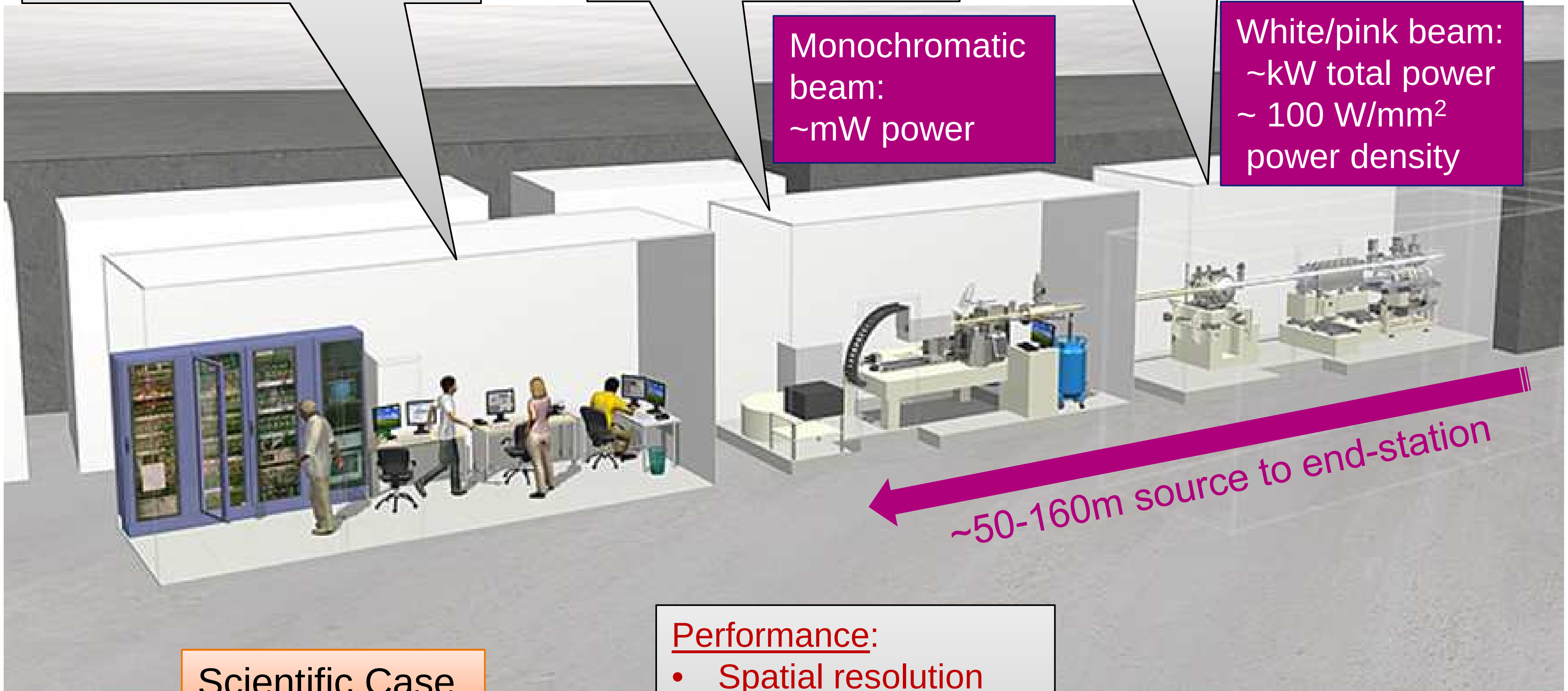
Data & control cabin

Experiment hutch

Optics hutch

Monochromatic beam:
~mW power

White/pink beam:
~kW total power
~ 100 W/mm^2
power density



~50-160m source to end-station

Scientific Case

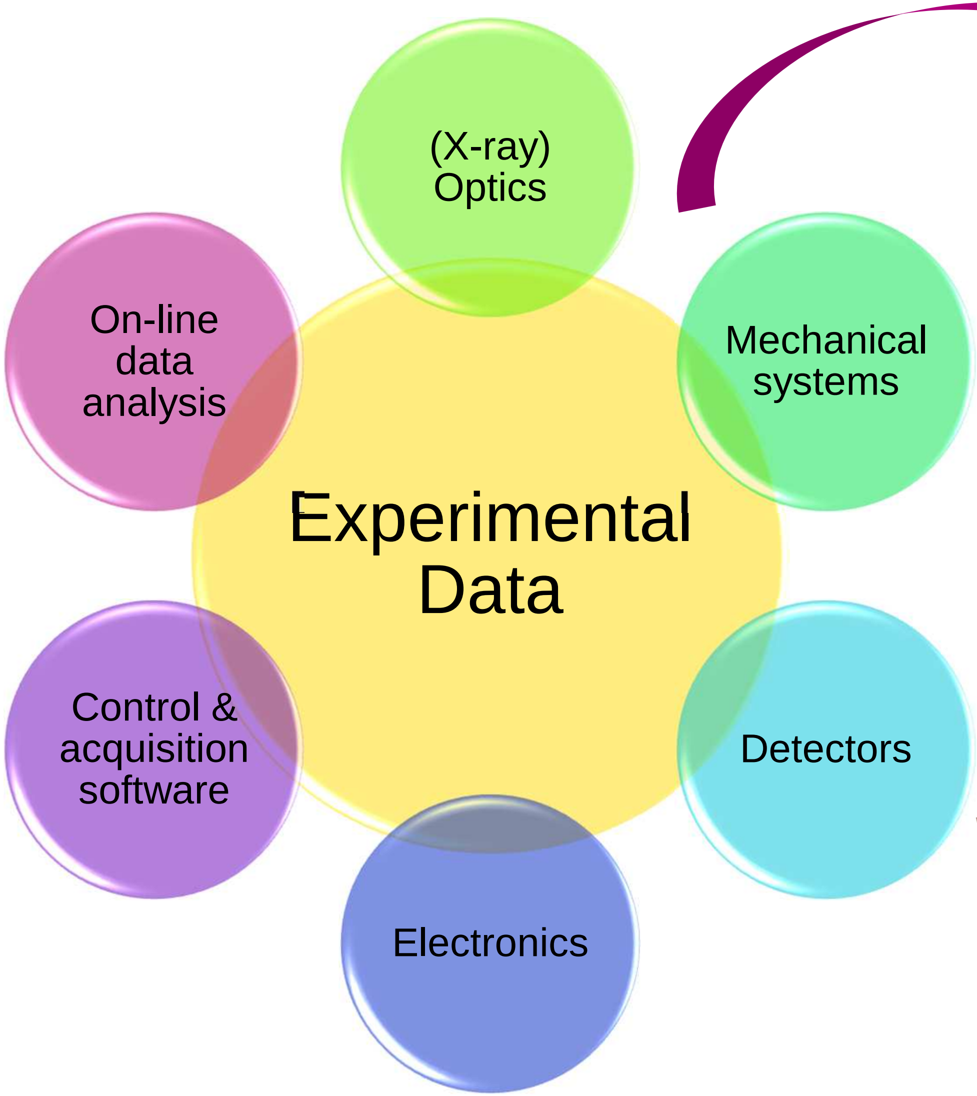
Performance:

- Spatial resolution
- Spectral resolution
- Time resolution
- 1D→ 2D→ 3D → 4D
- High throughput

X-RAY BEAMS AT 3RD GENERATION SR SOURCES

- Beam size
  - Unfocused: few mm to few cm (source is weakly divergent)
  - Focused beam: < 100 nm to ~10's µm
- Energy range/tunability
  - 0.1eV < E < 0.5 MeV but mostly 3-100 keV
- Energy bandwidth ($\Delta E/E$):
  - 10^{-2} to 10^{-8} at sample, typically ΔE ~ few eV @ 20keV
- Polarized radiation
  - 100% linear or circular or elliptical
- Pulsed radiation
  - 50 ps pulses every ns
- Power
  - several kW total power, several 100 W/mm^2 power density (white beam)
- High degree of coherence
- Photon Flux
  - Brilliance: 10^{22} ph/sec/mrad^2/mm^2 (10^{11} higher than conventional sources)
    $\Rightarrow$ photon flux (@ $\Delta E/E = 10^{-4}$): 10^9-10^{14} ph/s
  - Extremely variable photon rates on detectors (< 1 ph/s to full beam flux)

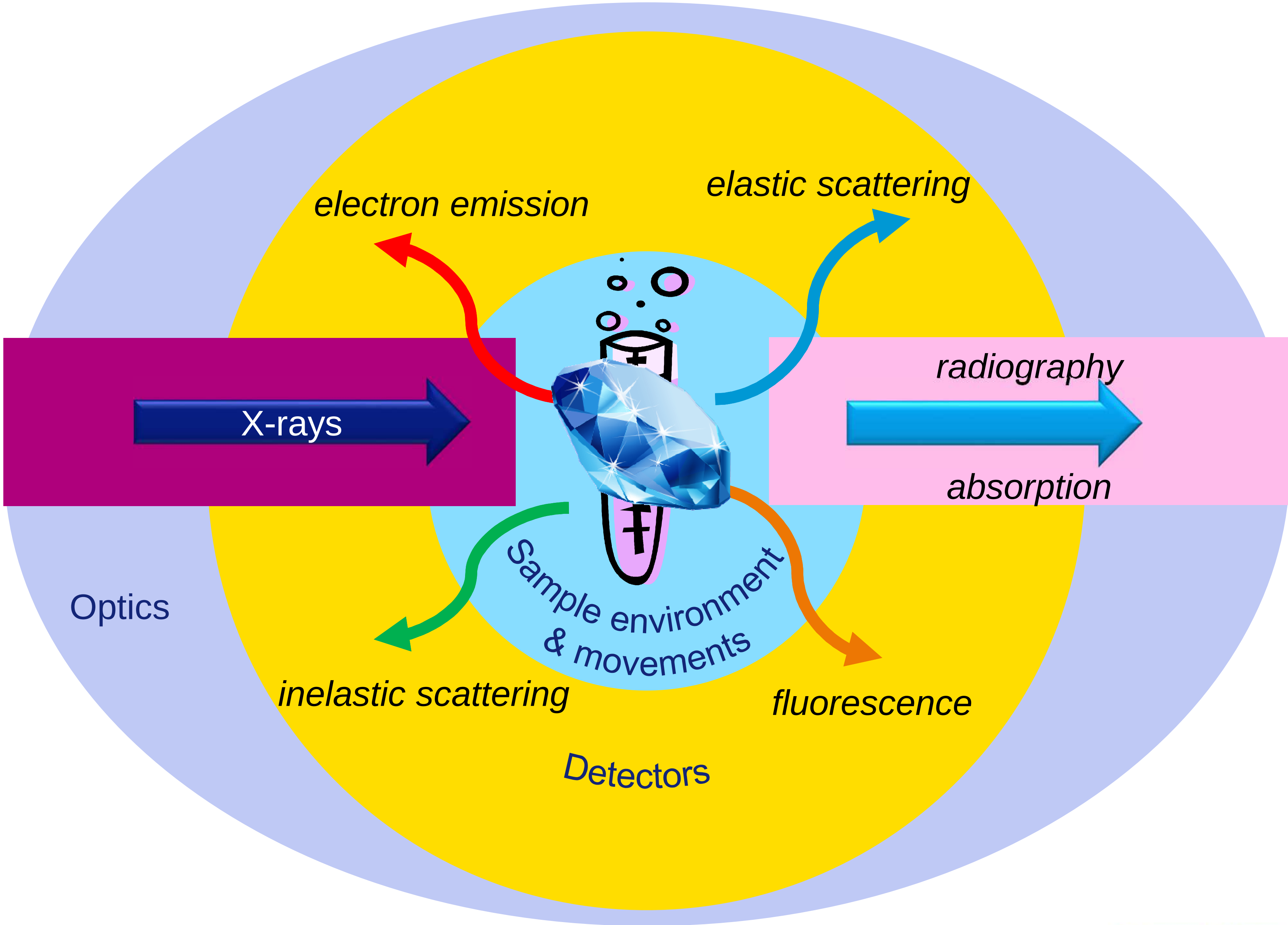
WHAT DO WE MEAN BY X-RAY INSTRUMENTATION AT A SR SOURCE?



Interpretation

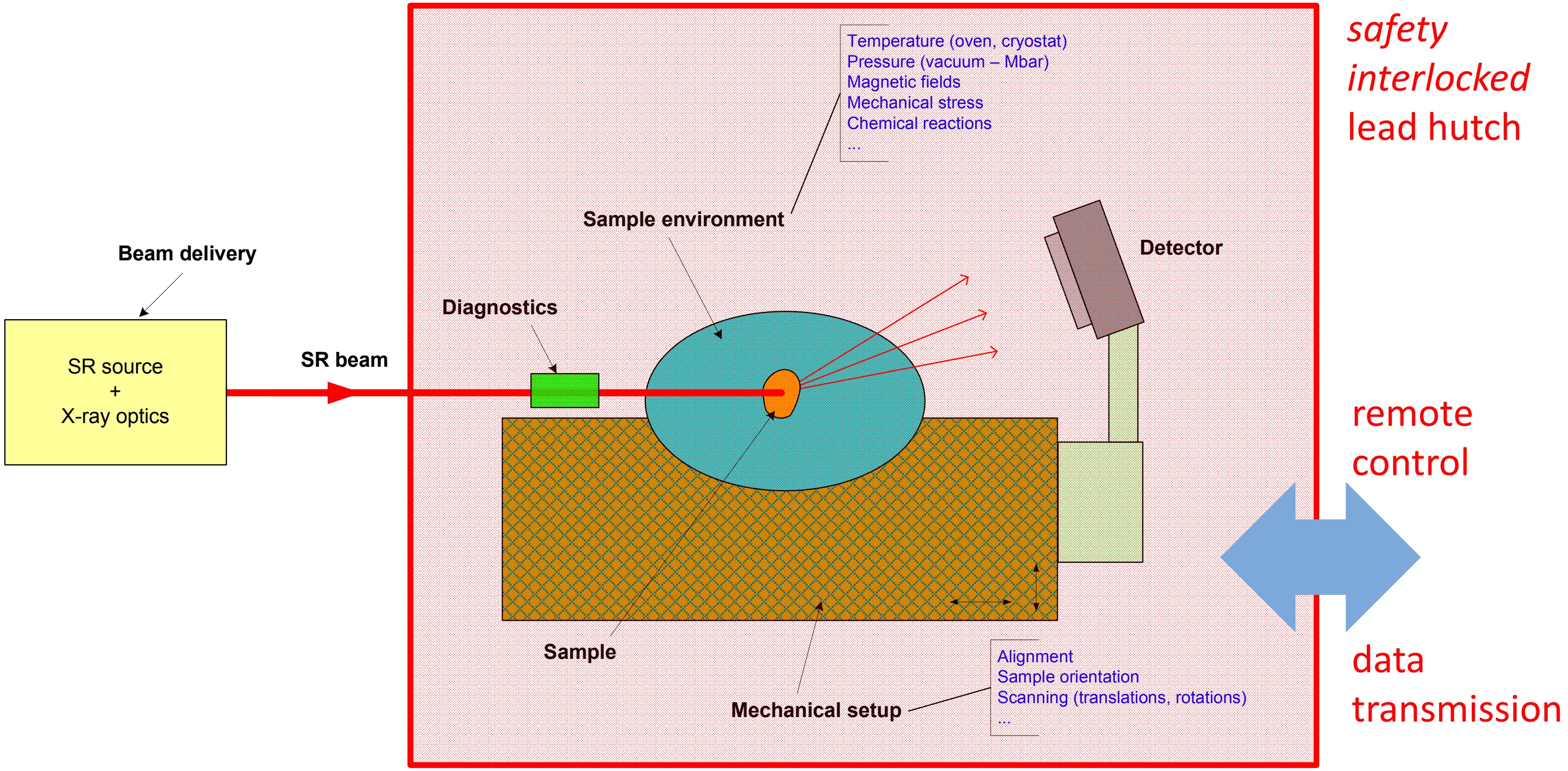
Publication/ Exploitation

A TYPICAL EXPERIMENT



experiments are built around the samples to be measured

⇒ importance of sample environment (temperature; pressure; E and B fields…)
need to physically manipulate sample during measurements (position, rotation…)

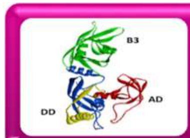


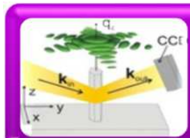
rapid turnover of samples and experiments

sample(s) in beam for minutes to hours
experiments typically last a few days

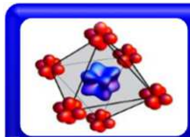
Courtesy: John Morse

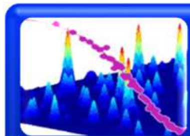
The ESRF groups its 40+ beamlines according to scientific application:

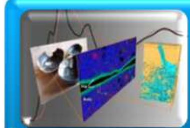

Structural biology

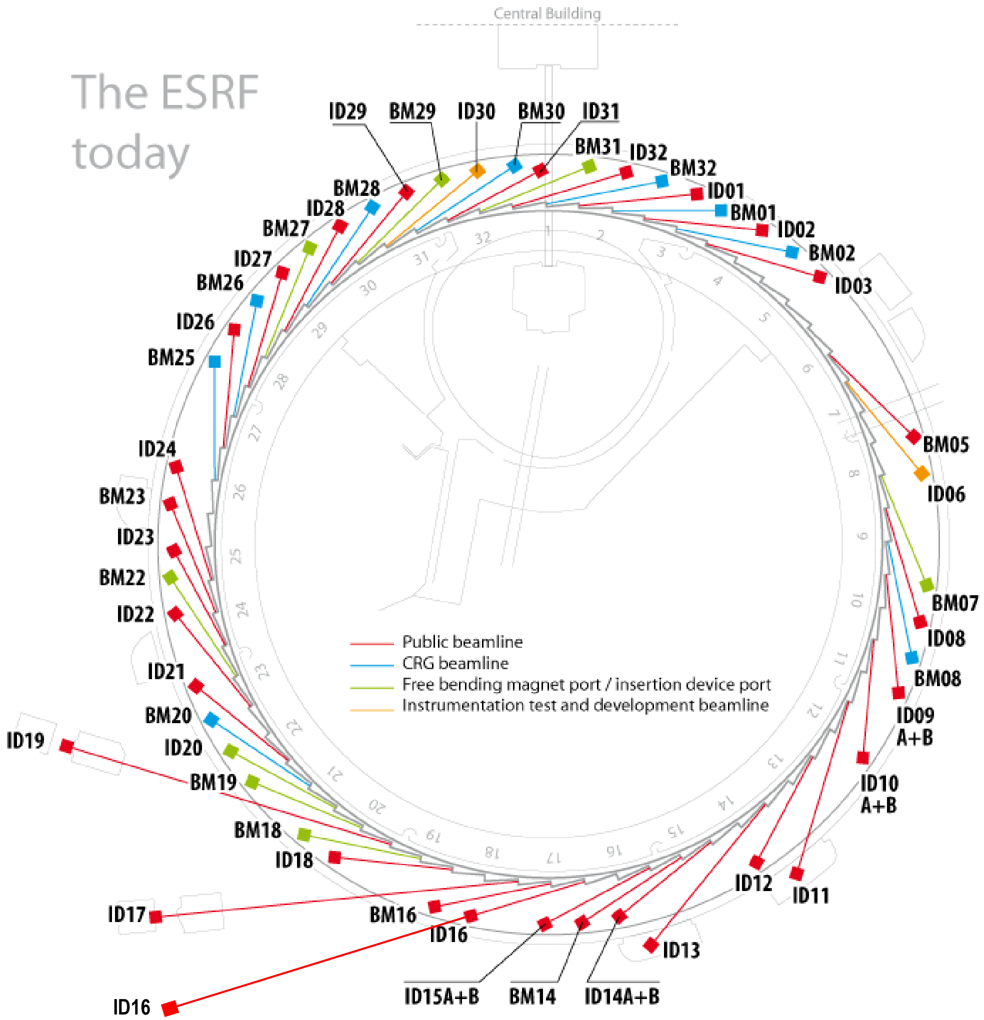

Structure of materials


Electronic structure & magnetism


Dynamics & extreme conditions


Structure of soft matter


X-ray imaging



A COMPLETE SUITE OF TECHNIQUES

X-Ray Fluorescence

- Composition
- Quantification
- Trace element mapping

X-ray Diffraction & scattering

- Long range structure
- Crystal orientation mapping
- Stress/strain/texture mapping

Specific

Sample environments

Phase contrast X-ray imaging

- 2D/3D Morphology
- High resolution
- Density mapping

Infrared FTIR-spectroscopy

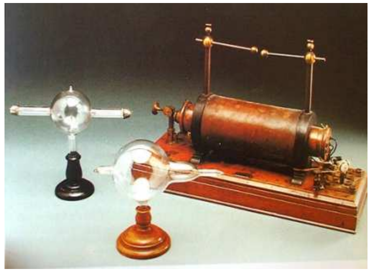
- Molecular groups & structure
- High S/N for spectroscopy
- Functional group mapping

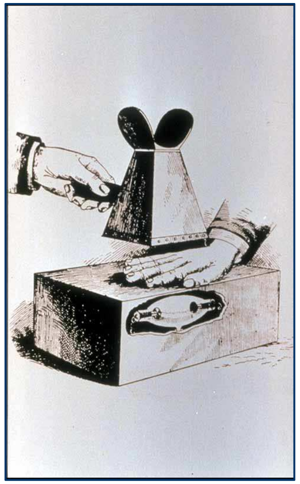
X-ray spectroscopy

- Short range structure
- Electronic structure
- Oxidation/speciation mapping

X-RAY INSTRUMENTATION: RÖNTGEN'S ORIGINAL WORK (1895)

after W.C. Röntgen
Über eine neue art von Strahlen.
Phys.-Med. Ges., Würzburg, 137, (1895)
English translation in Nature 53, (1996)





- "... The refractive index.... cannot be more than 1.05 at most.... X-rays cannot be concentrated by lenses…."
- "... Photographic plates and film are "susceptible to x-rays", providing a valuable means of recording the effects..."
- "... Detection of interference phenomena has been tried without success, perhaps only because of their feeble intensity..."

"... The refractive index.... cannot be more than 1.05 at most....
....X-rays cannot be concentrated by lenses..."

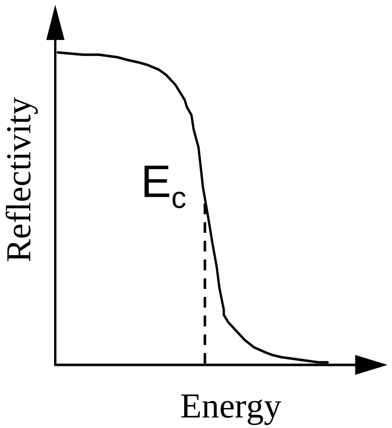
W.C. Röntgen
Über eine neue art von Strahlen.
Phys.-Med. Ges., Würzburg, **137**, p. 41, (1895)
*English translation in Nature **53**, p. 274*

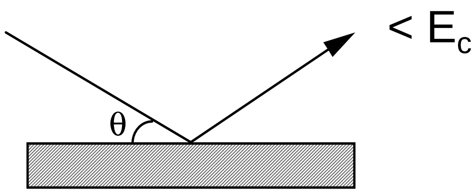
$n=1-\delta+i\beta$ with $\delta, \beta <<< 1$

δ (phase-shift), β (absorption), materials (and energy) dependent optical constants

- Very weak refraction
- Quite high absorption

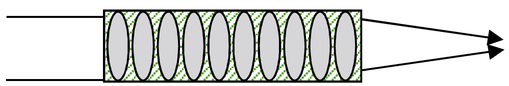
REFLECTION





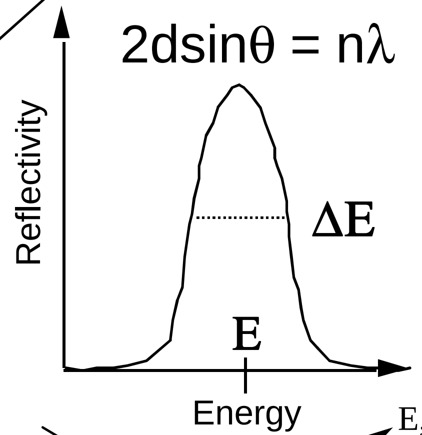
- X-ray mirrors
- Capillaries
- Waveguides

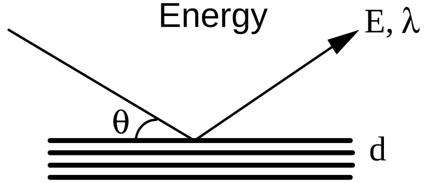
REFRACTION



- Refractive lenses

DIFFRACTION

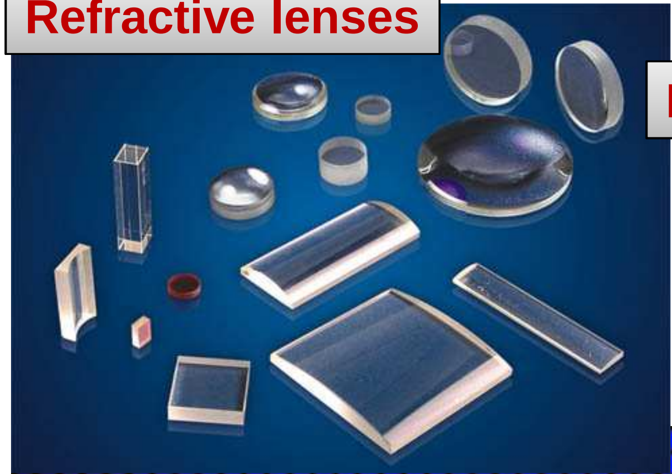




- Crystals & multilayers
- X-ray gratings
- Fresnel zone plates
- Bragg-Fresnel lens

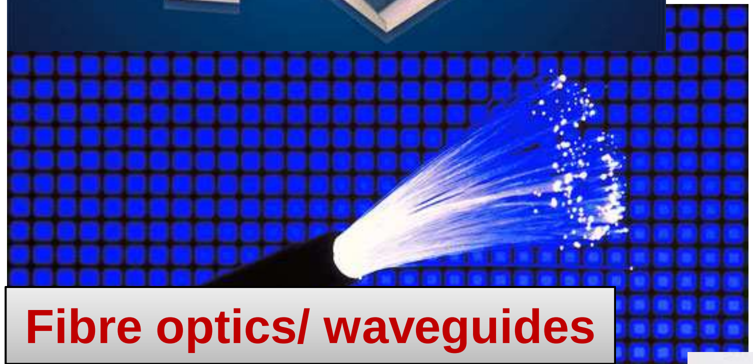
VISIBLE LIGHT OPTICS

Refractive lenses

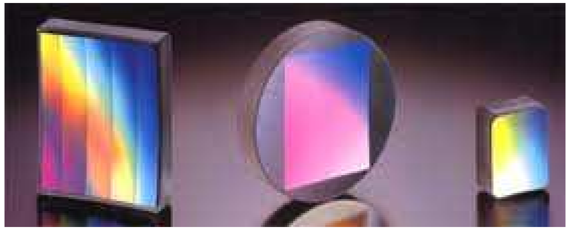


Polarising Optics

Fresnel lenses



Diffractive optics

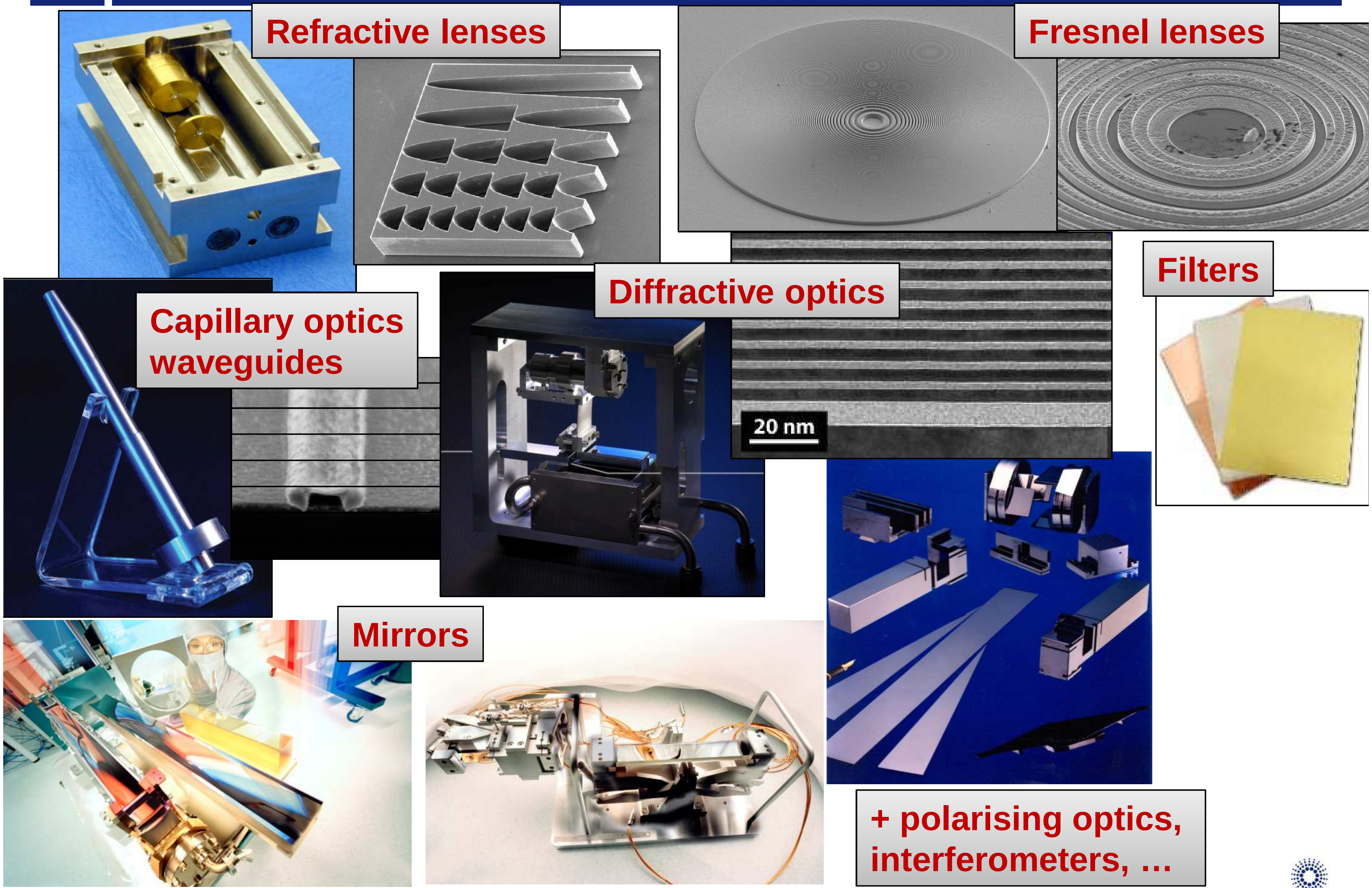


Fibre optics/ waveguides

Mirrors

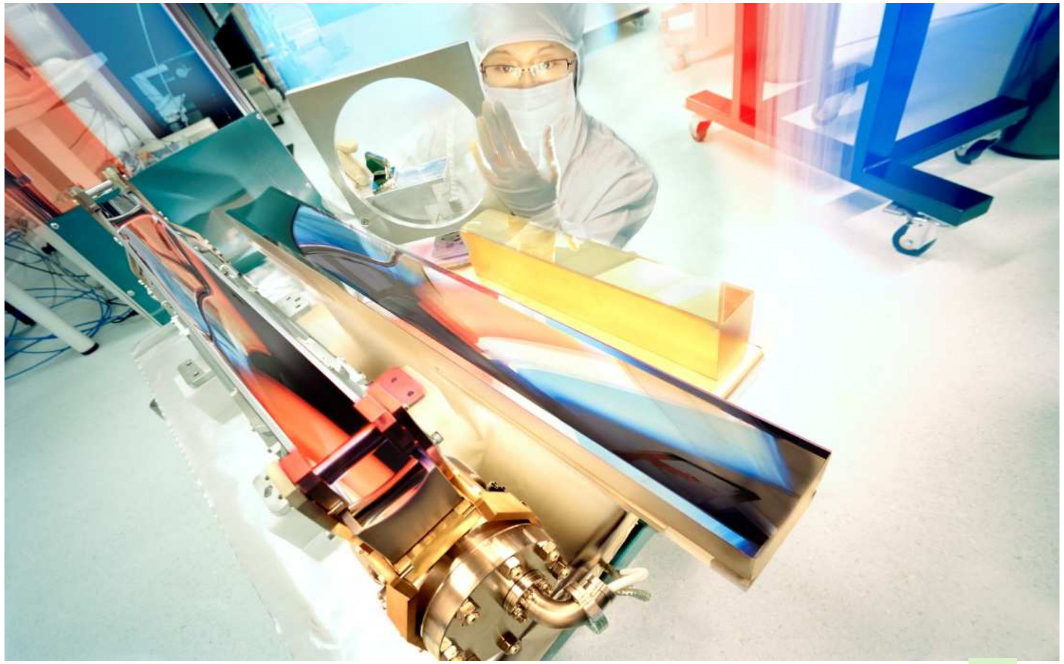
Filters

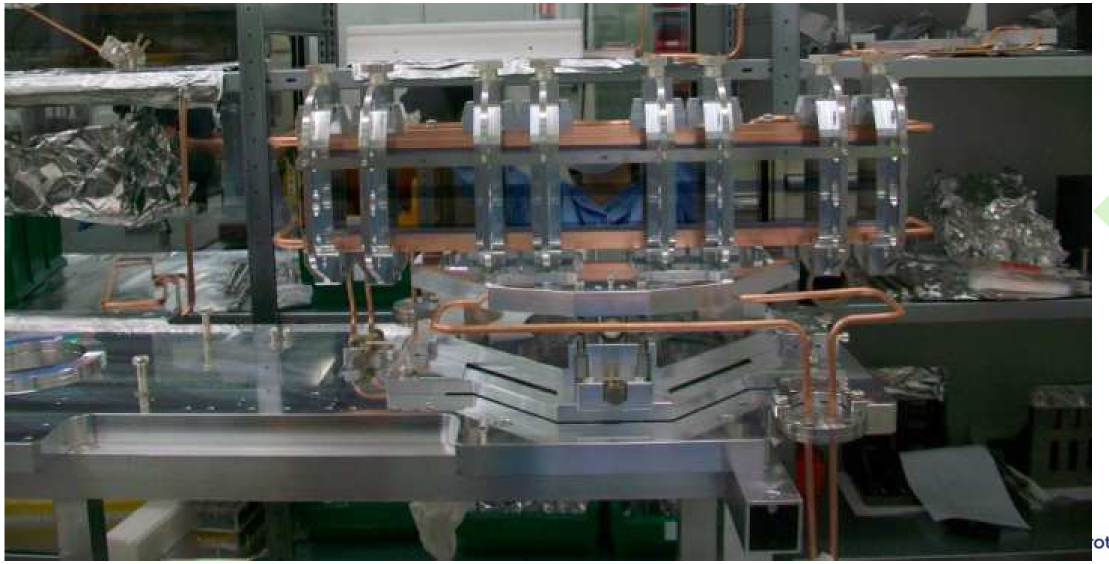
+ interferometers, …



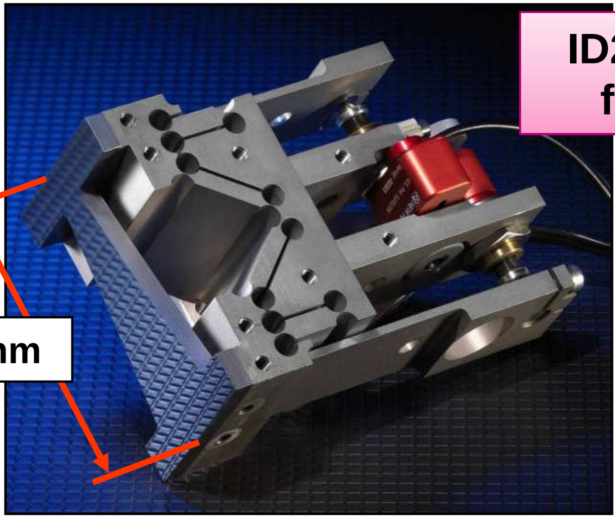
Refractive lenses
Fresnel lenses
Capillary optics waveguides
Diffractive optics
20 nm
Filters
Mirrors
+ polarising optics, interferometers, …

SILICON OPTICS – X-RAY MIRRORS



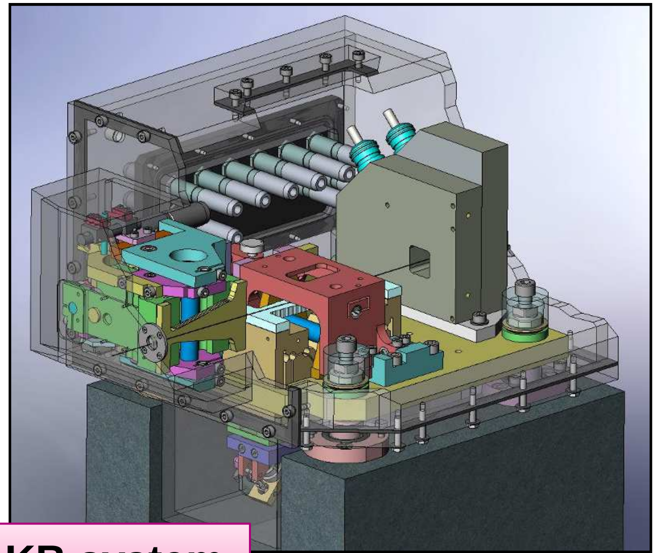


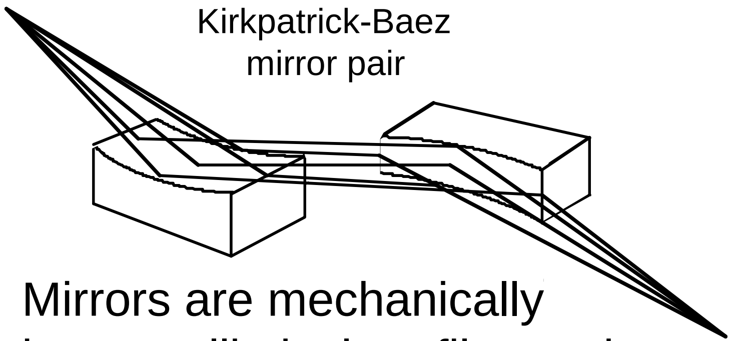
ESRF NANOFOCUSING 'KIRKPATRICK-BAEZ' MIRROR SYSTEM



76mm

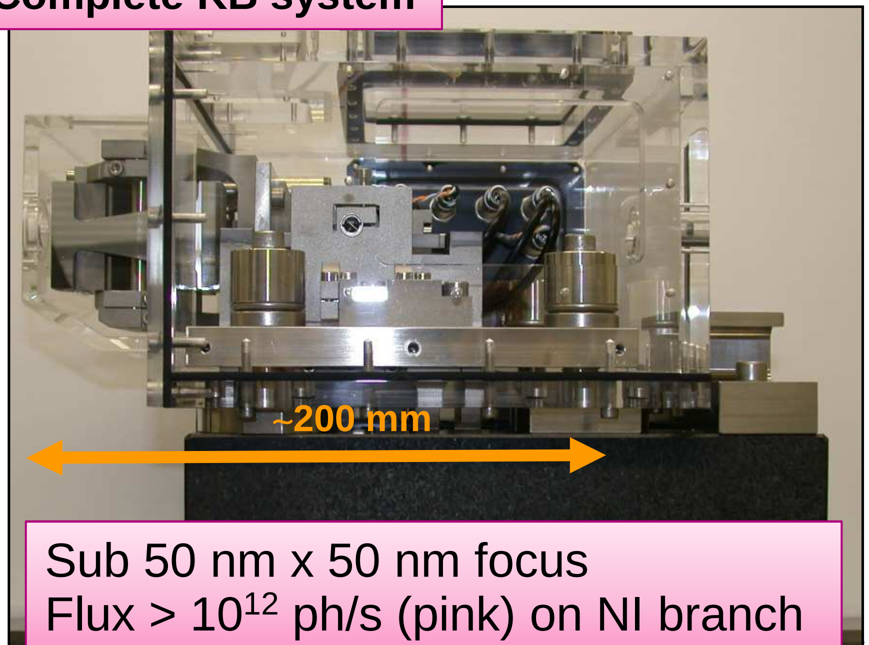
ID22-NI horizontally focusing bender





Mirrors are mechanically bent to elliptical profiles and focus X-rays

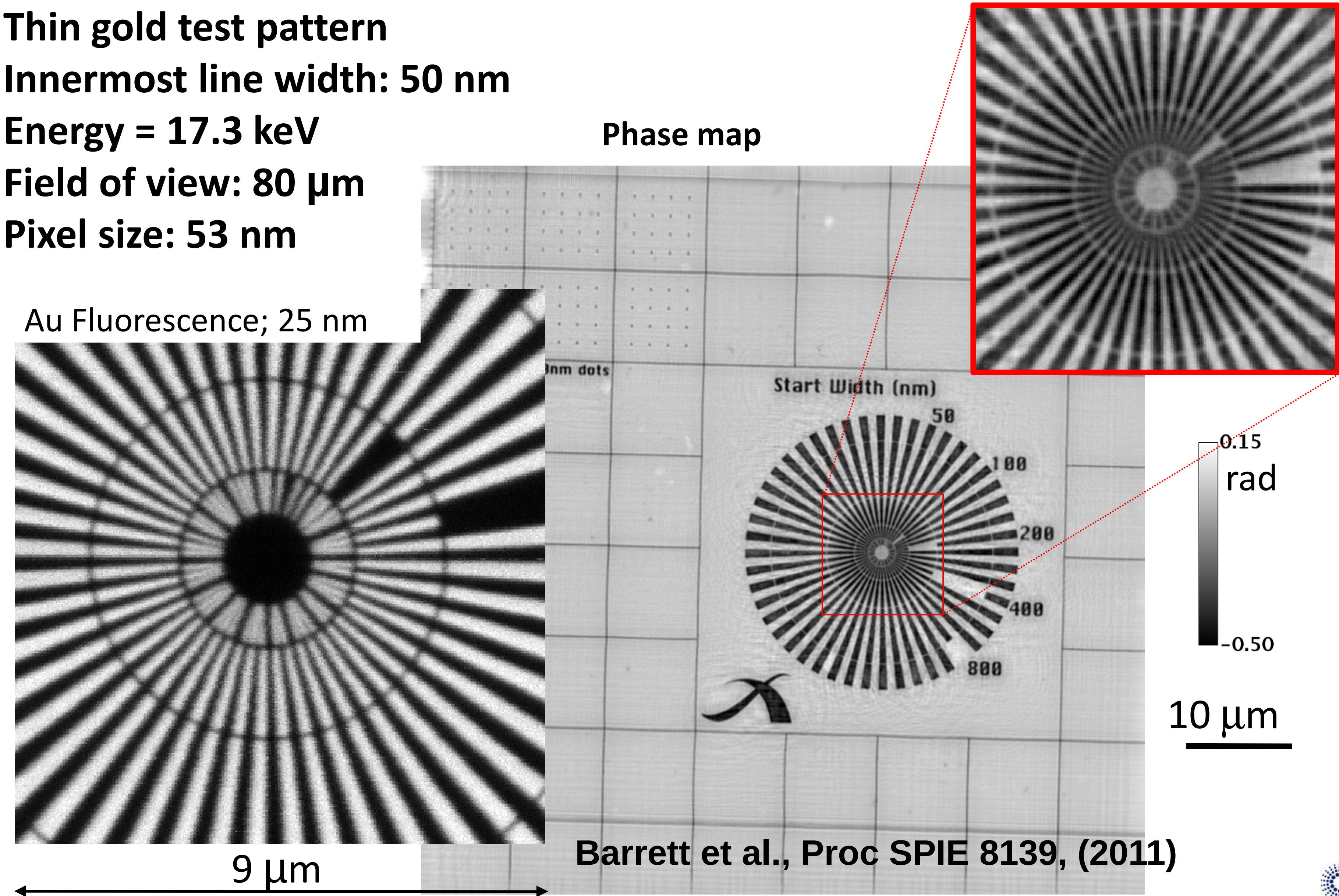
Complete KB system



Sub 50 nm x 50 nm focus
Flux > 10^{12} ph/s (pink) on NI branch

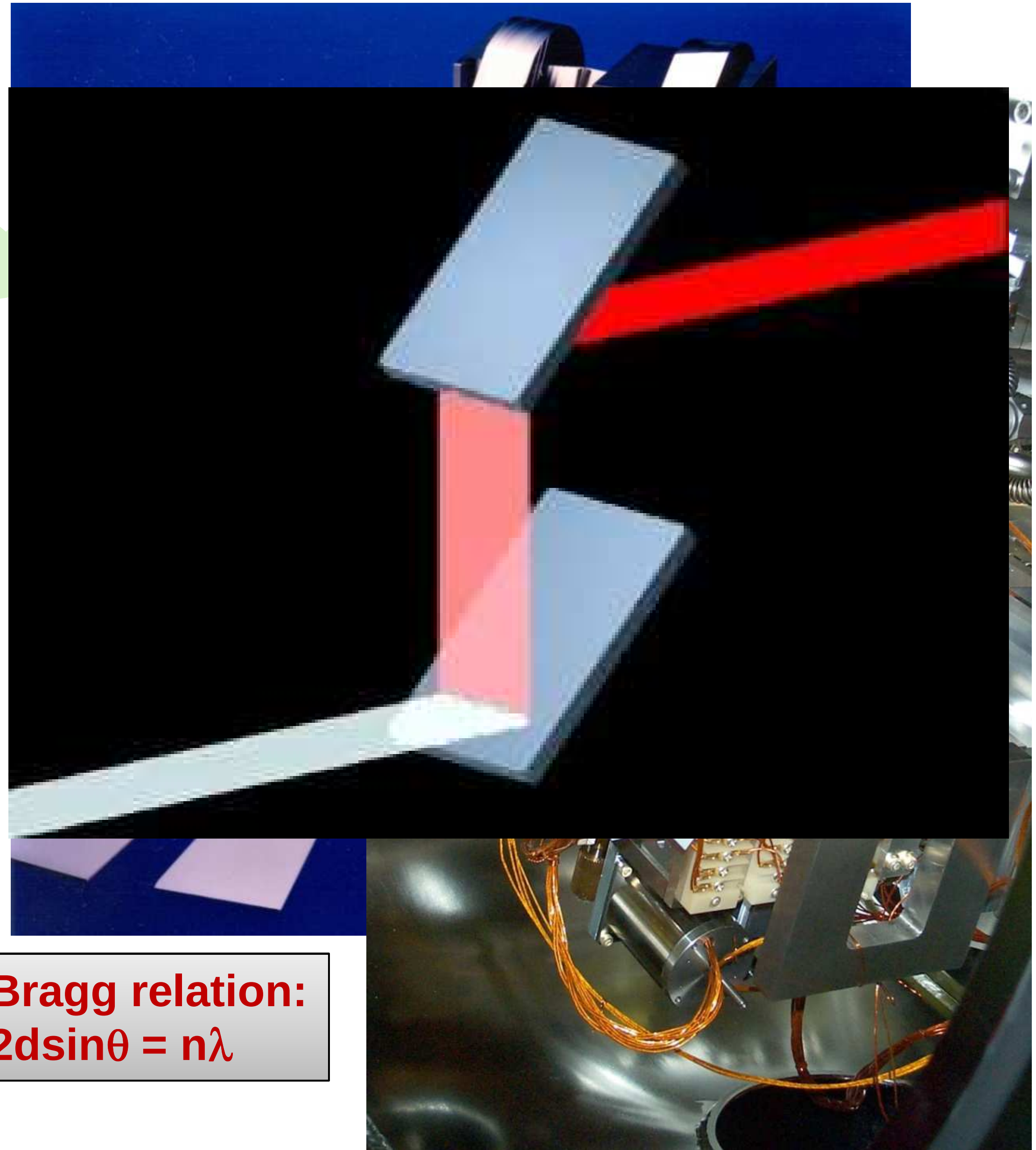
PROJECTION MICROSCOPY USING KB OPTICS

Thin gold test pattern
Innermost line width: 50 nm
Energy = 17.3 keV
Field of view: 80 µm
Pixel size: 53 nm



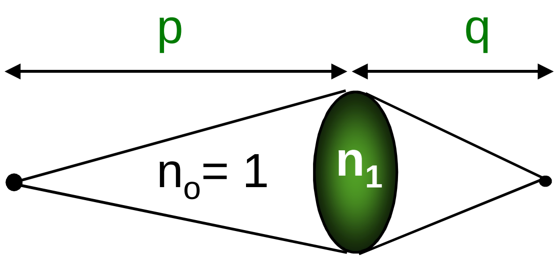
Barrett et al., Proc SPIE 8139, (2011)

SILICON OPTICS -MONOCHROMATORS



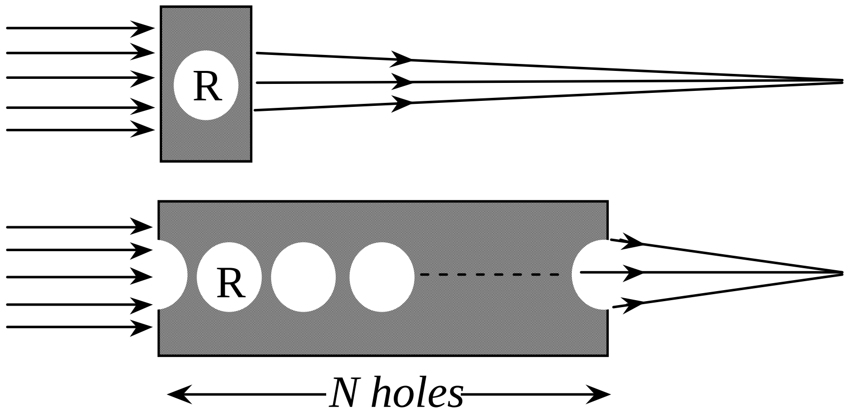
Bragg relation:
$2d\sin\theta = n\lambda$

COMPOUND REFRACTIVE LENSES



Gaussian lens equation : $\frac{1}{f} = \frac{2(n_1 - 1)}{R}$

Thin lens equation : $\frac{1}{f} = \frac{1}{p} + \frac{1}{q}$



$$\frac{1}{f} = \frac{2\delta}{R}$$

$$\frac{1}{f} = N\frac{2\delta}{R}$$

X-rays : $n = 1 - \delta + i\beta$

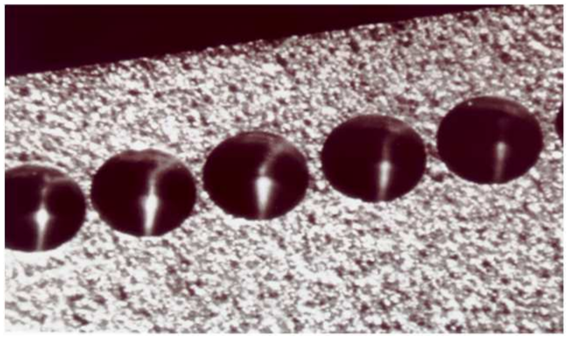
$n_1 < 1$: concave lens

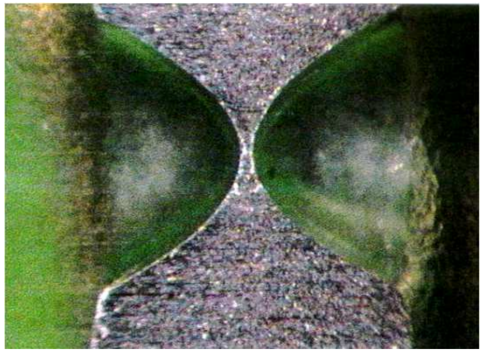
Typically Be or Al lenses – e.g.

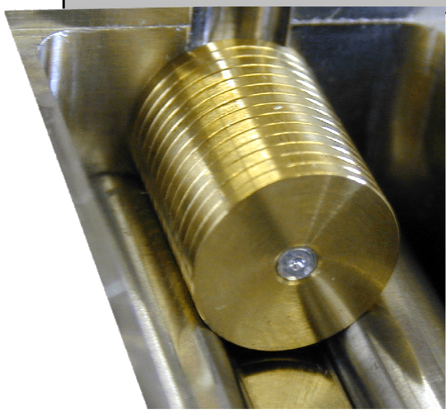
Aluminium @ 10keV $\delta = 5.5\ 10^{-6}$

1 hole 100 µm radius : f = 9 m

15 holes 100 µm radius: f = 60 cm

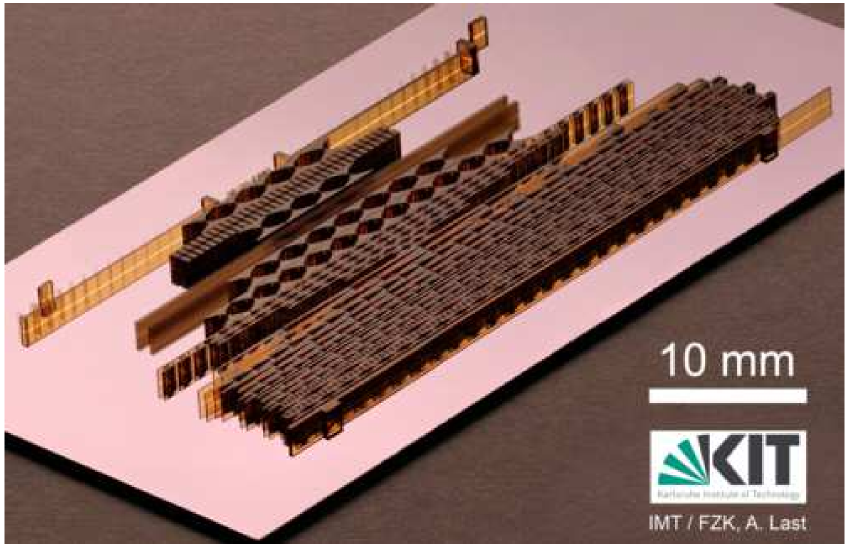


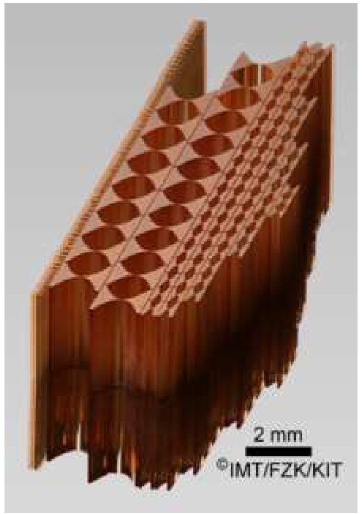




A. Snigirev et al. Nature, 384 (1996)

LIGA-process



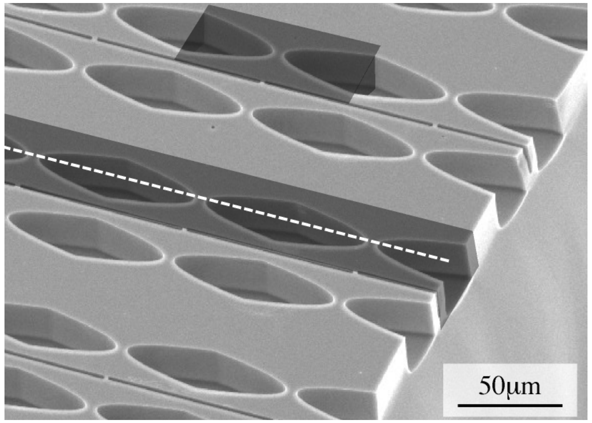


- Polymers (SU-8)
- Nickel

V. Nazmov & V. Saile
Microstructure Technology (IMT)
FZK, Germany

V. Nazmov *et al.*, NIM A 582 (2007)

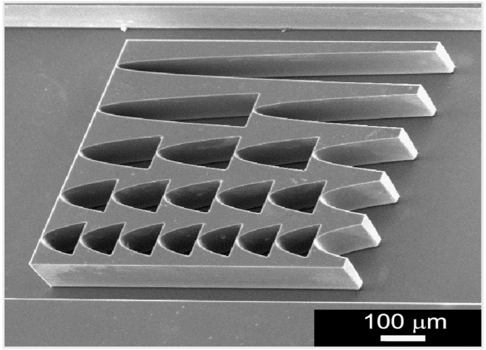
Reactive ion etching (RIE)



Silicon

C. G. Schroer, Institut für Strukturphysik
Dresden, Germany

C. G. Schroer *et al.*, APL 87 (2005)

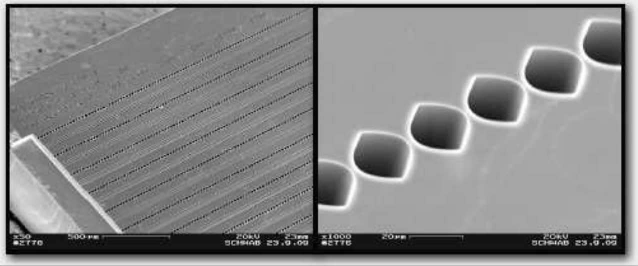


Diamond

C. David
PSI, Villigen, Switzerland

B. Nöhammer *et al.* JSR 10 (2003)

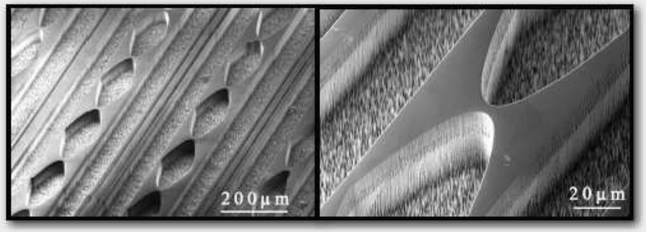
COMPOUND REFRACTIVE LENSES



new NFLs of highest quality

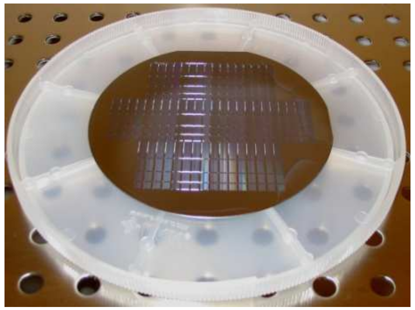
- new lens design ensures isotropic etching conditions
- reduced slope of sidewalls: <0.01 rad
- reduced roughness of surface: <20 nm rms

→ ready for AFL fabrication!

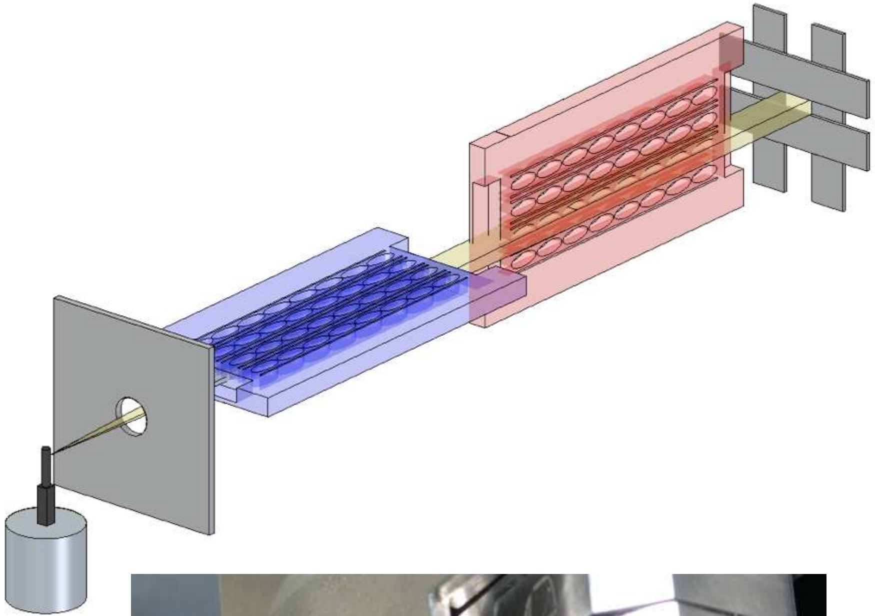


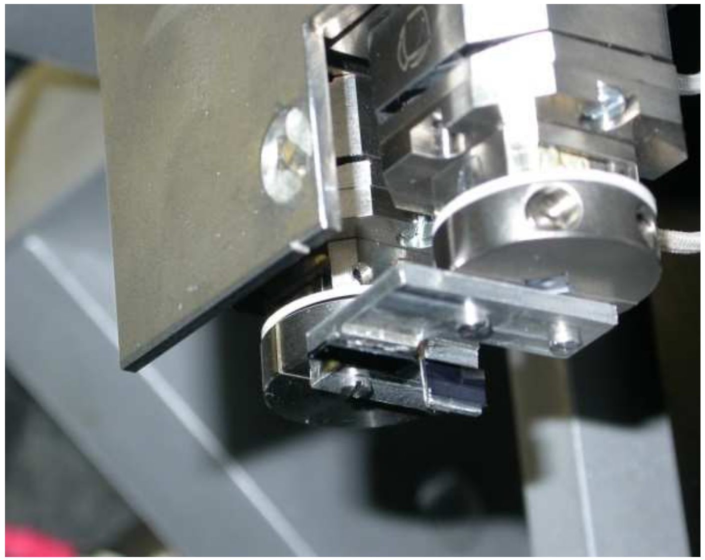
diamond lenses

- high lens shape precision
- improved deepness of structures (but still not deep enough)
- still a problem: roughness
- successfully tested in an experiment at the ESRF

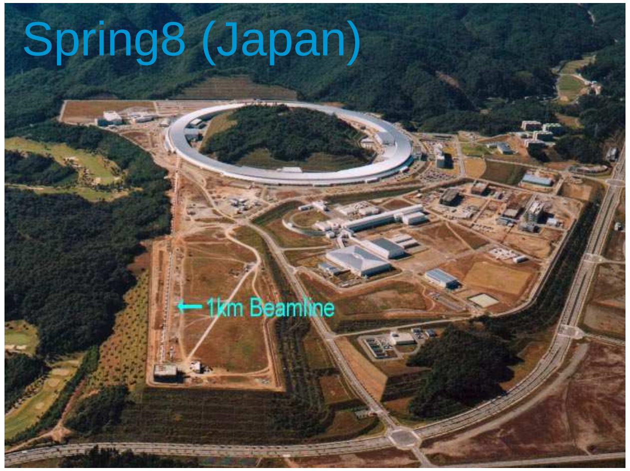


3136 Silicon NFLs on wafer
about 600000 single lenses



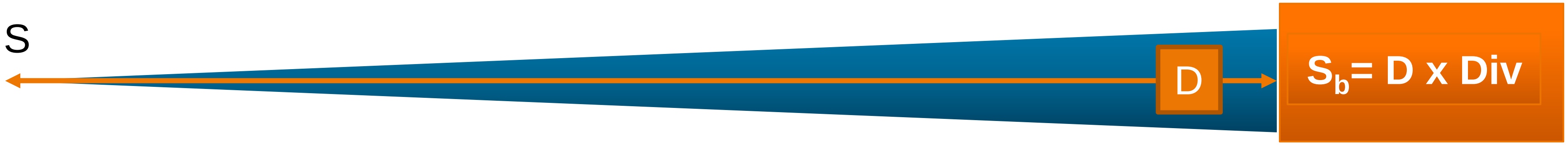


WHY LONG BEAMLINES?



D=1km

D=150-180m

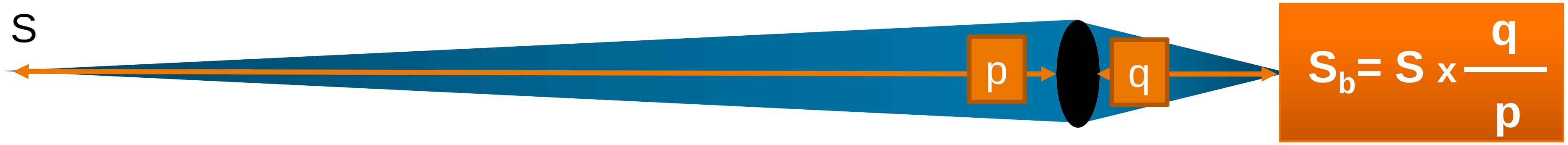


mm-cm

Tiny source

Low divergence beam

Beam size



µm-nm

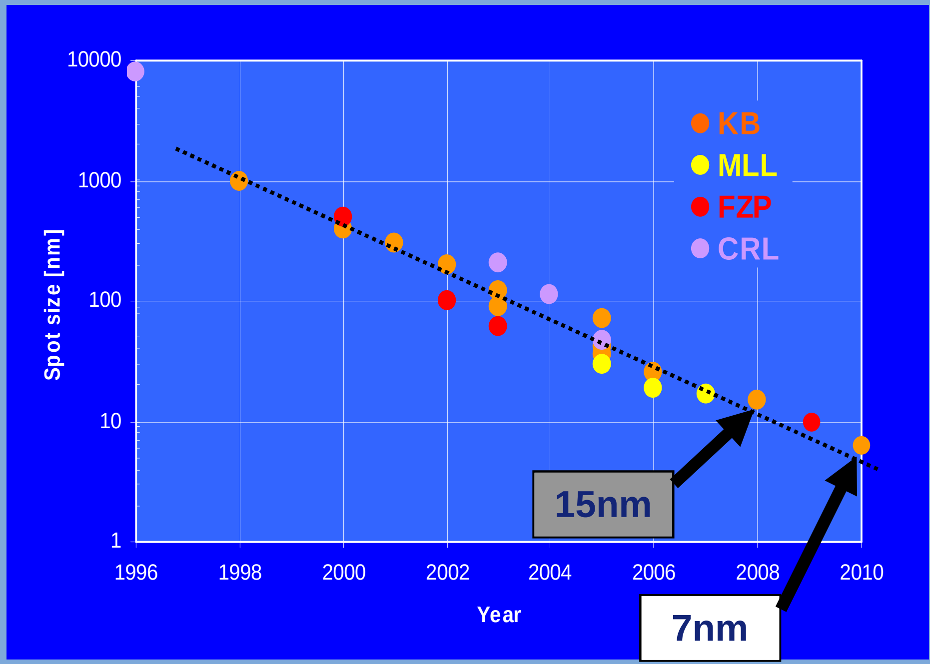
Moore's law adapted to the X-ray world:

ESRF Red Book (1987): very few beamline projects aiming even for 10 micron sized beams

Now optics exist for 10nm beams

Routine application of sub-micron beams still complicated

Also many engineering issues in implementing stable, reliable X-ray nanofocusing systems



Historical evolution of the measured spot size for different hard x-ray focusing elements *(courtesy C. Morawe)*

- H. Mimura *et al. Nature Physics*, **6**, 122-125 (2010).
- J. Vila-Comamala et al., Ultramicroscopy,**109**,1360–1364 (2009)

Best focus Experiments

- H. Kang *et al., Physical Review Letters*, **96**:127401 (2006)
- C. Schroer *et al., Physical Review Letters*, **94**:054802 (2005)

Ultimate resolution Theory

adapted to one or more techniques…

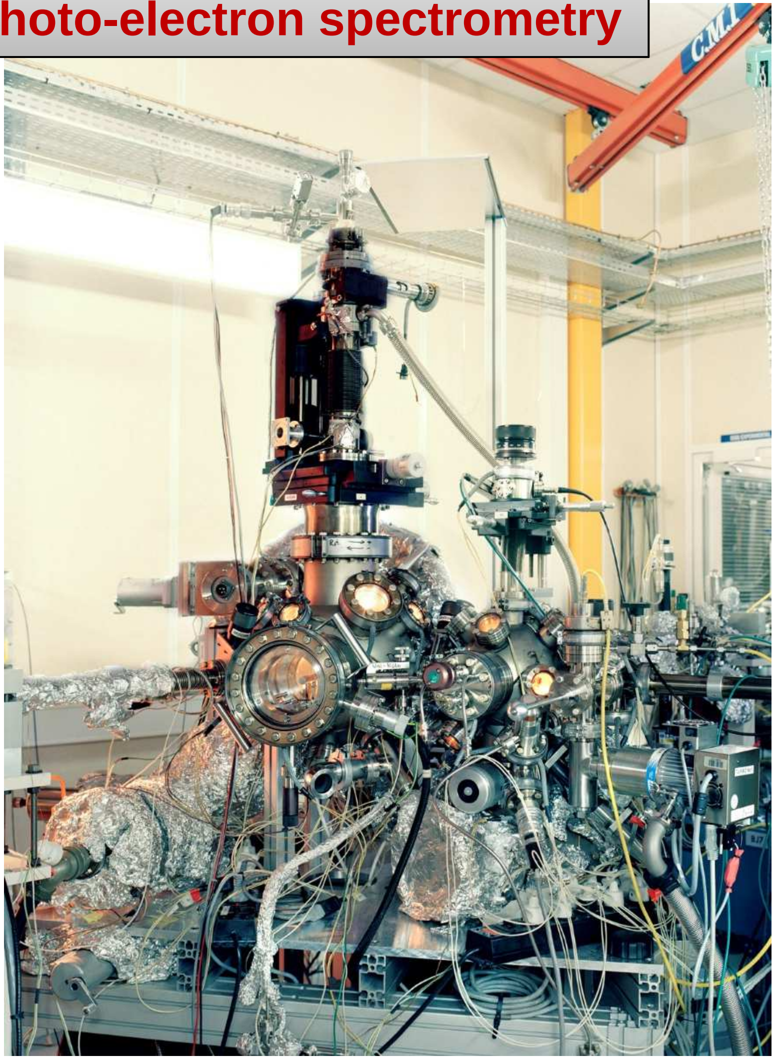
- X-ray Diffraction & Scattering
- X-ray Spectroscopy
- X-ray Fluorescence
- X-ray Imaging …

… on samples of varying types

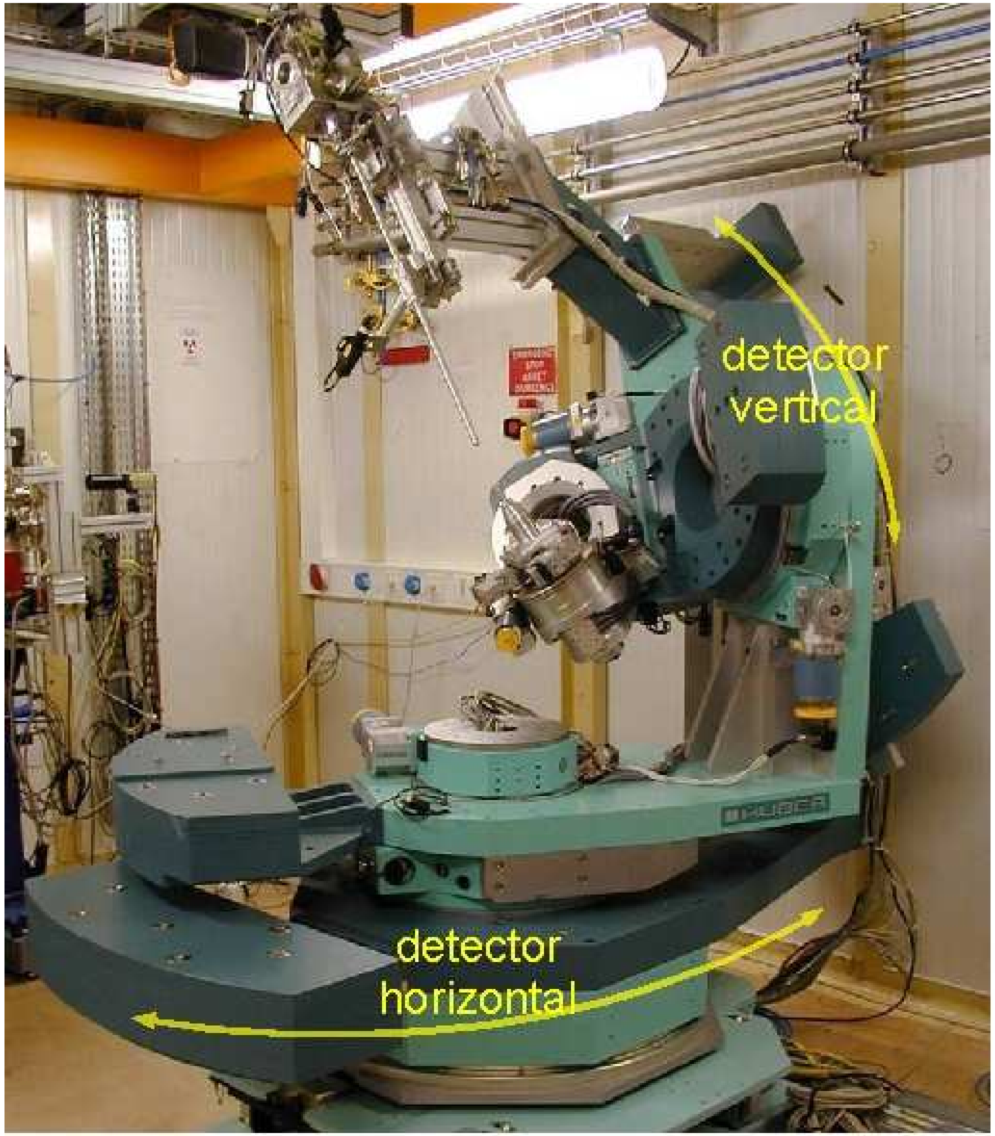
- Inorganic/organic crystals
- Colloidal solutions
- Fossils
- Cells
- Industrial materials ...

… and different sample environments

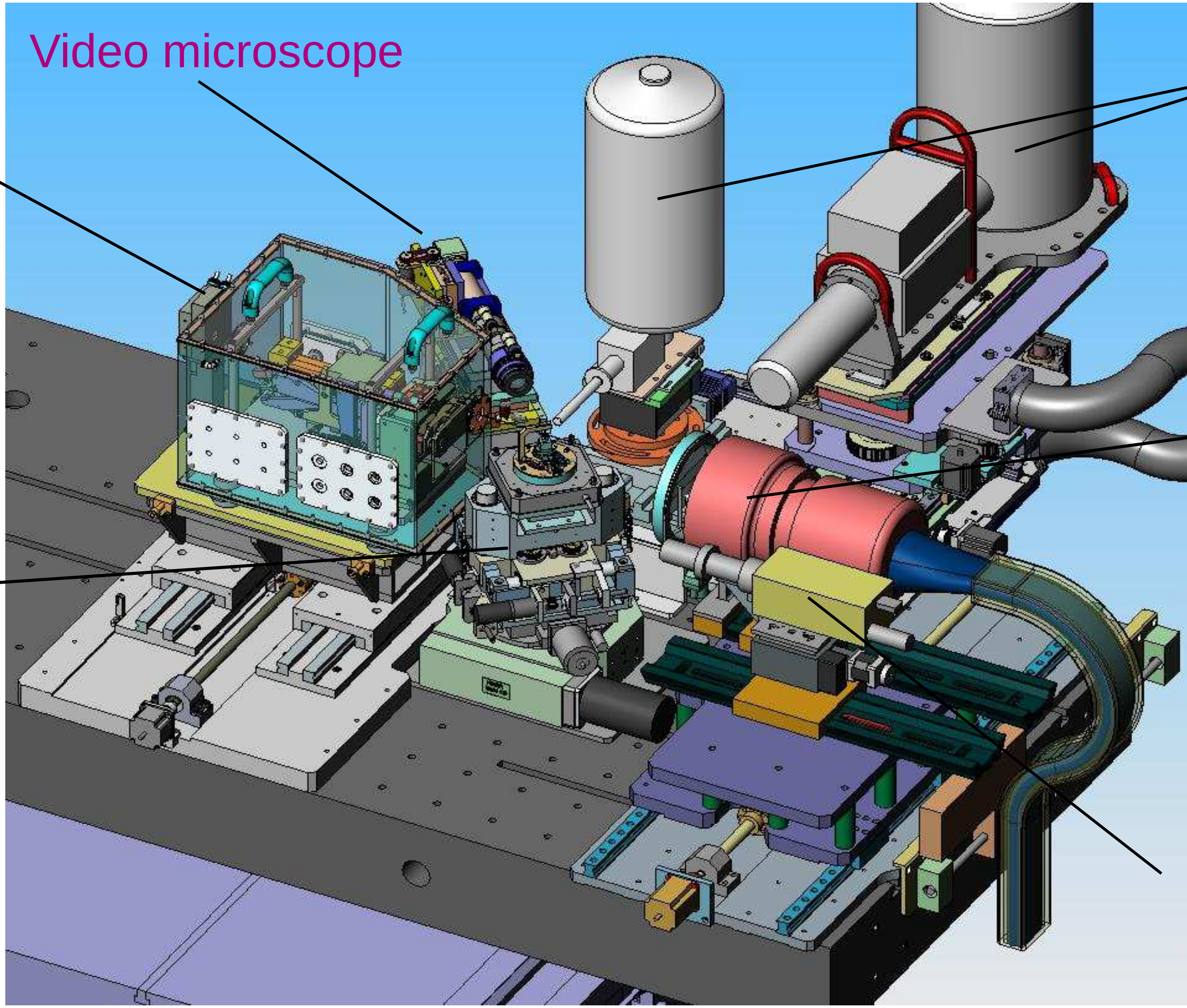
ID08: experimental station - Photo-electron spectrometry



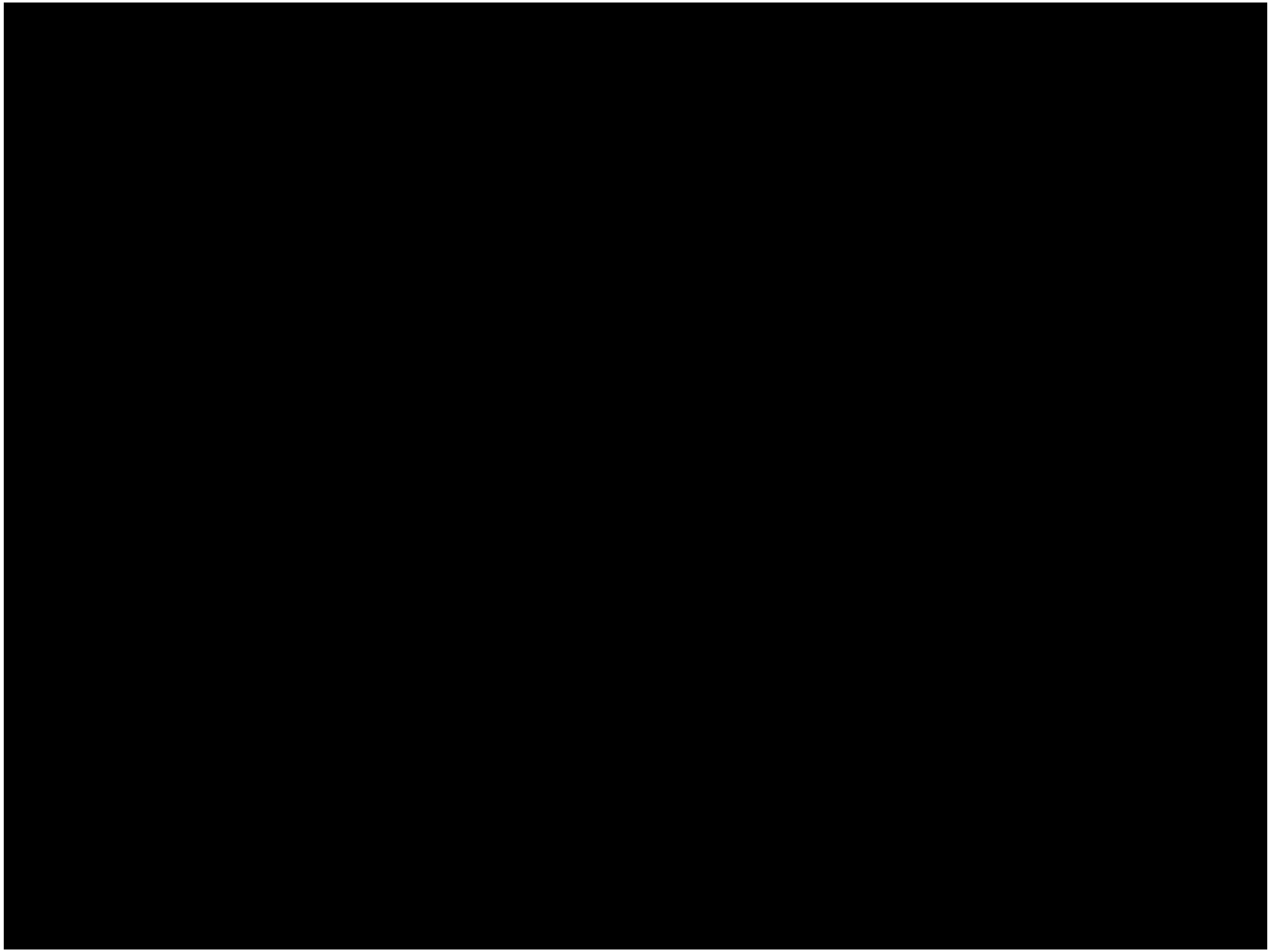
X-RAY DIFFRACTOMETER



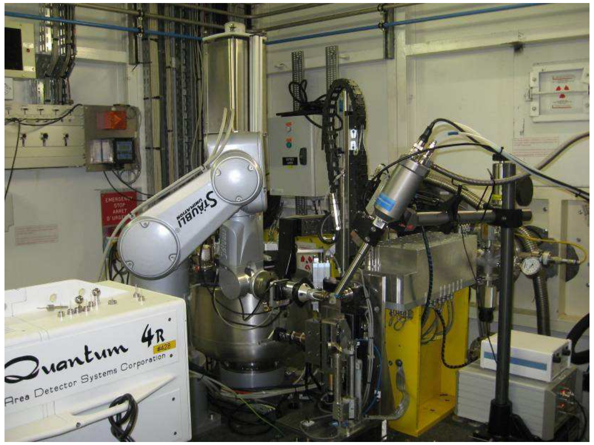
HARD (5-70KEV) X-RAY MICROPROBE (ESRF-ID22)

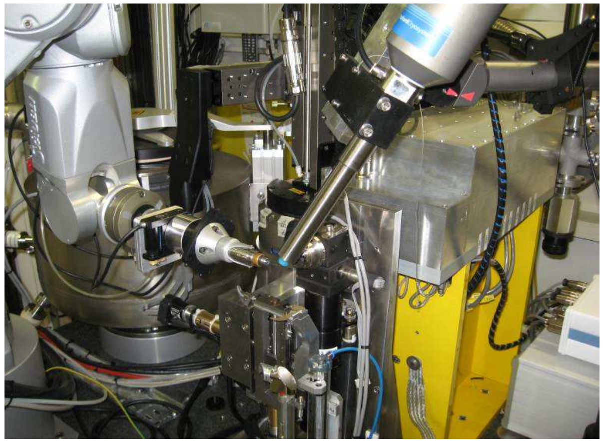


HIGH THROUGHPUT CRYSTALLOGRAPHY

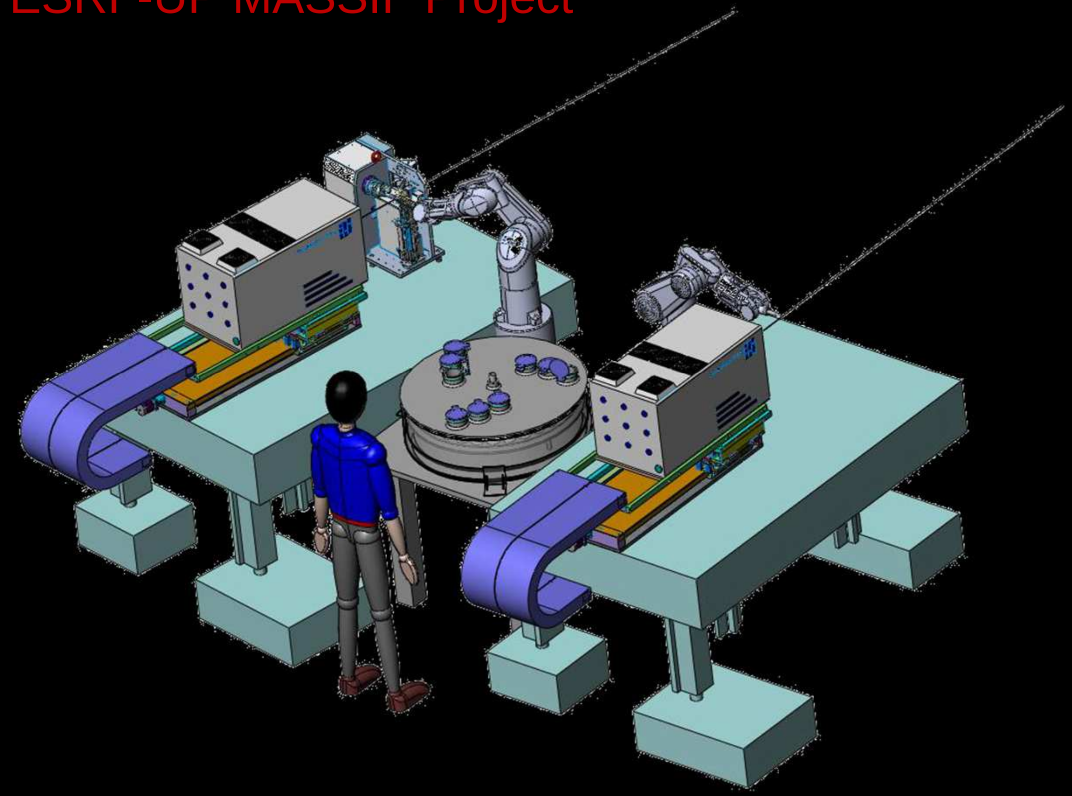


AUTOMATION AND HIGH THROUGHPUT





ESRF-UP MASSIF Project



Capacity per Station: 1000 crystals per day

3 stations …. 600 000 crystals per year….

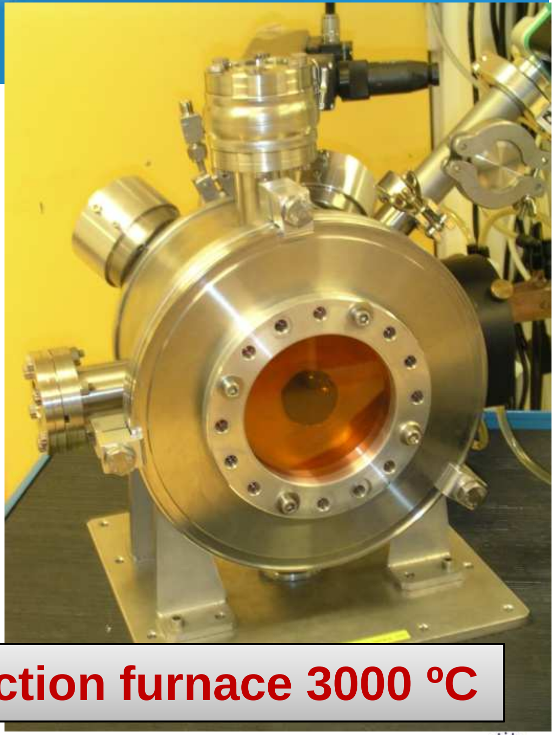
Courtesy S. McSweeney

Particularly for:

- High temperature (furnaces)
- Low temperature (cryostat)
- Magnetic field
- Electric field
- Pressure application
- Controlled gas atmospheres
- Pump-probe experiments

Also to limit sample damage due to photon absorption (e.g. protein crystallography experiments) …

Mini flow cryostat: 2 Kelvin



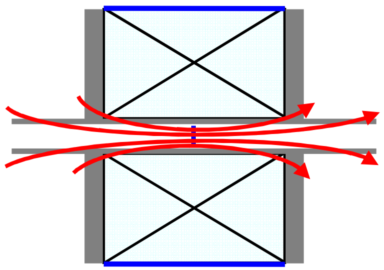
Induction furnace 3000 ºC

Courtesy: Peter van der Linden

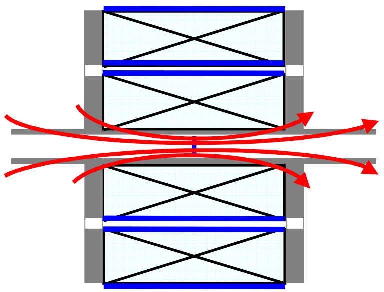
MINI PULSED MAGNETIC FIELD

High duty cycle minicoil

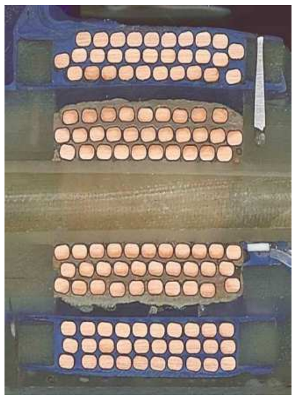
• monolithic



• slit coil



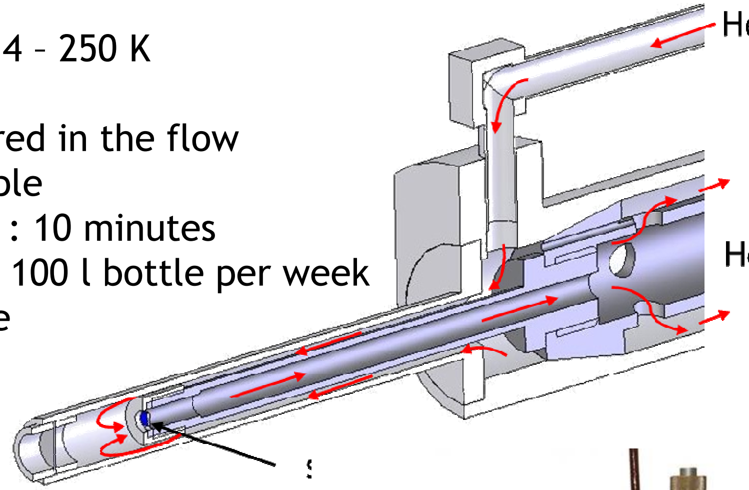
cooling surface

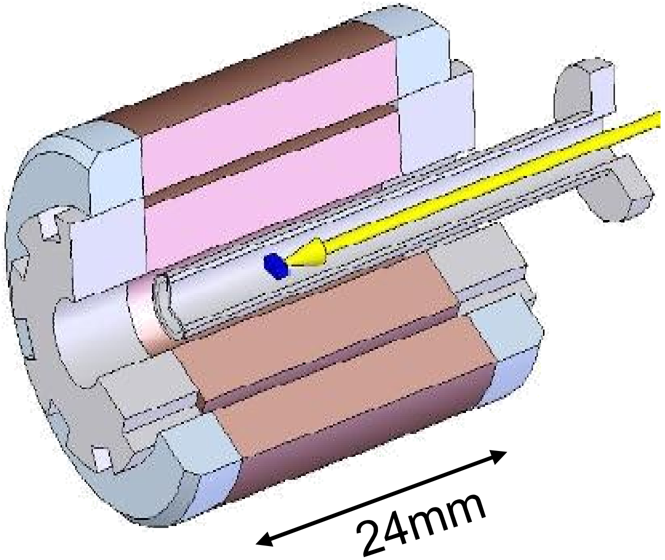


5 mm

Power supply
C = 1 mF
U = 3000 V
Imax 32000 A
Coil
I = 12 000 A
L = 35 μH

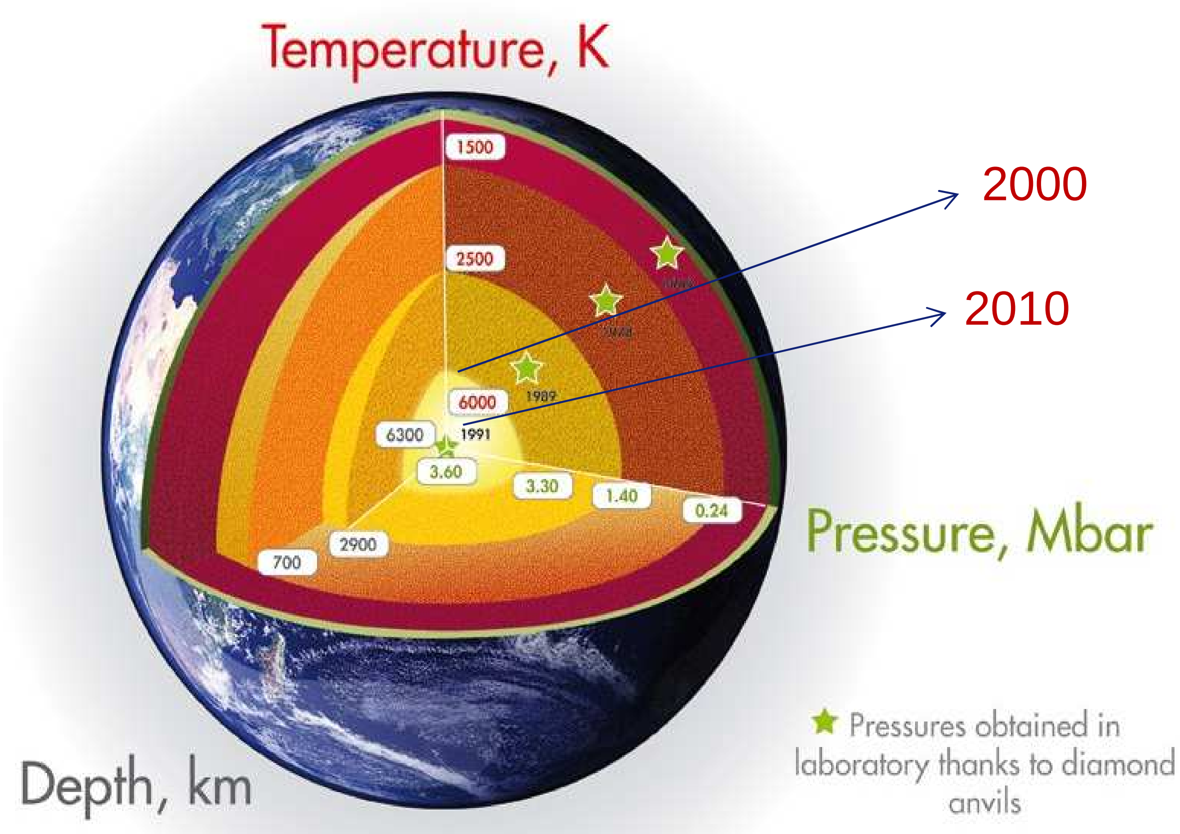
• Temperature range: 4 – 250 K
• Sample in He flow
• Temperature measured in the flow right after the sample
• Stabilization at 10 K : 10 minutes
• Consumption (10 K): 100 l bottle per week
• Quick sample change





Duty cycle: $1 \cdot 10^{-4}$ rep. rate: 6/min
B = 30 (38) T at working T: 120K

EXTREME CONDITIONS (T, P)



Diamond Anvil Cell (DAC)

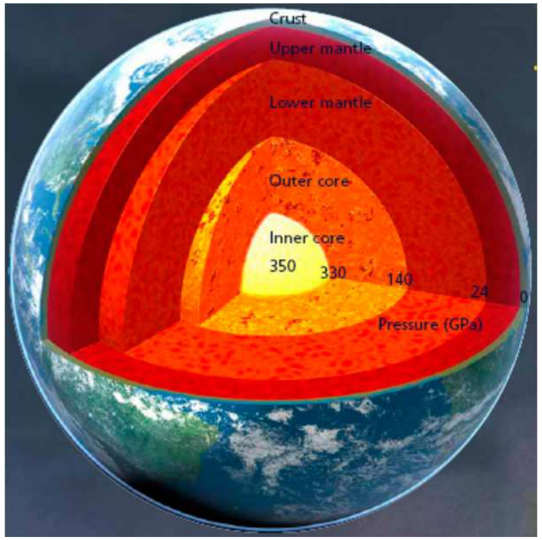
1 million atmospheres (Bars) =100 Billion Pascals (GPa))

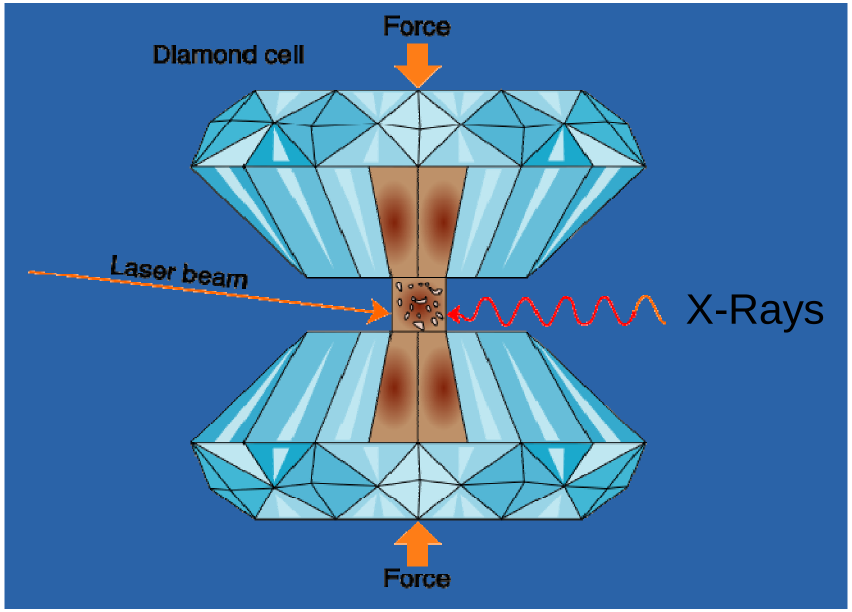
Record Pressure: 650 GPa

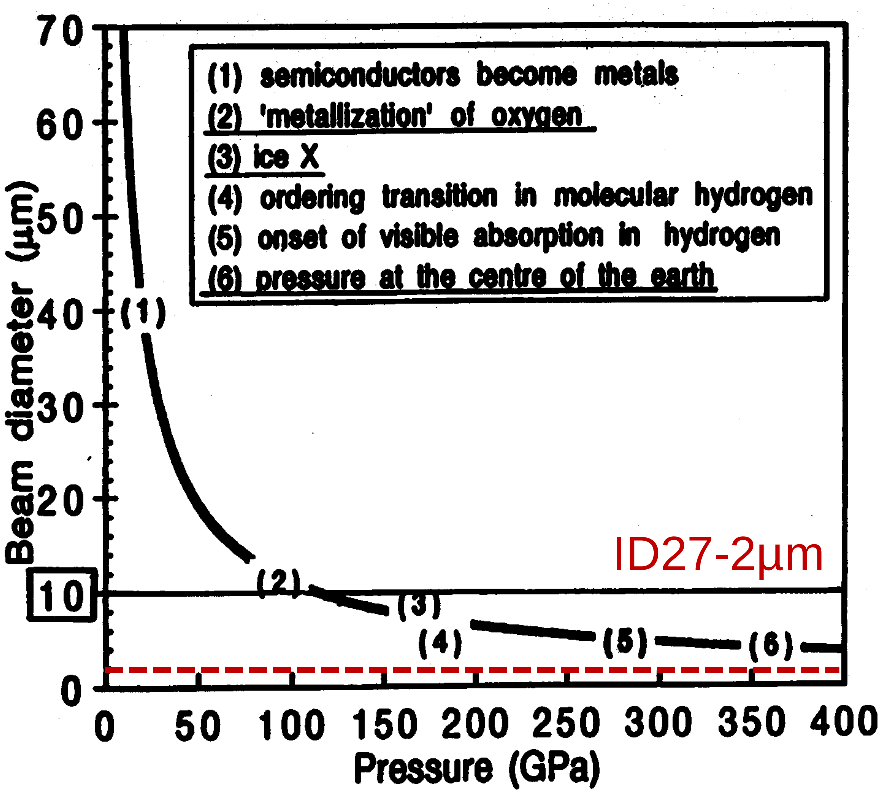
L. Dubrovinsky et al., Nature Commun. 3, 1163, 2012.

Courtesy M. Mezouar (ESRF-ID27)

EXTREME CONDITIONS (T, P)

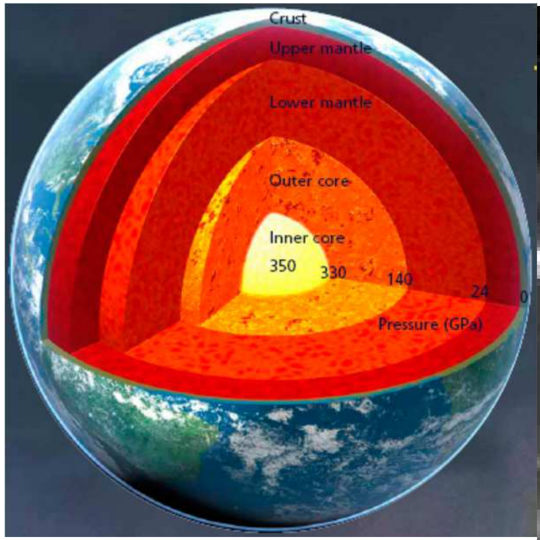


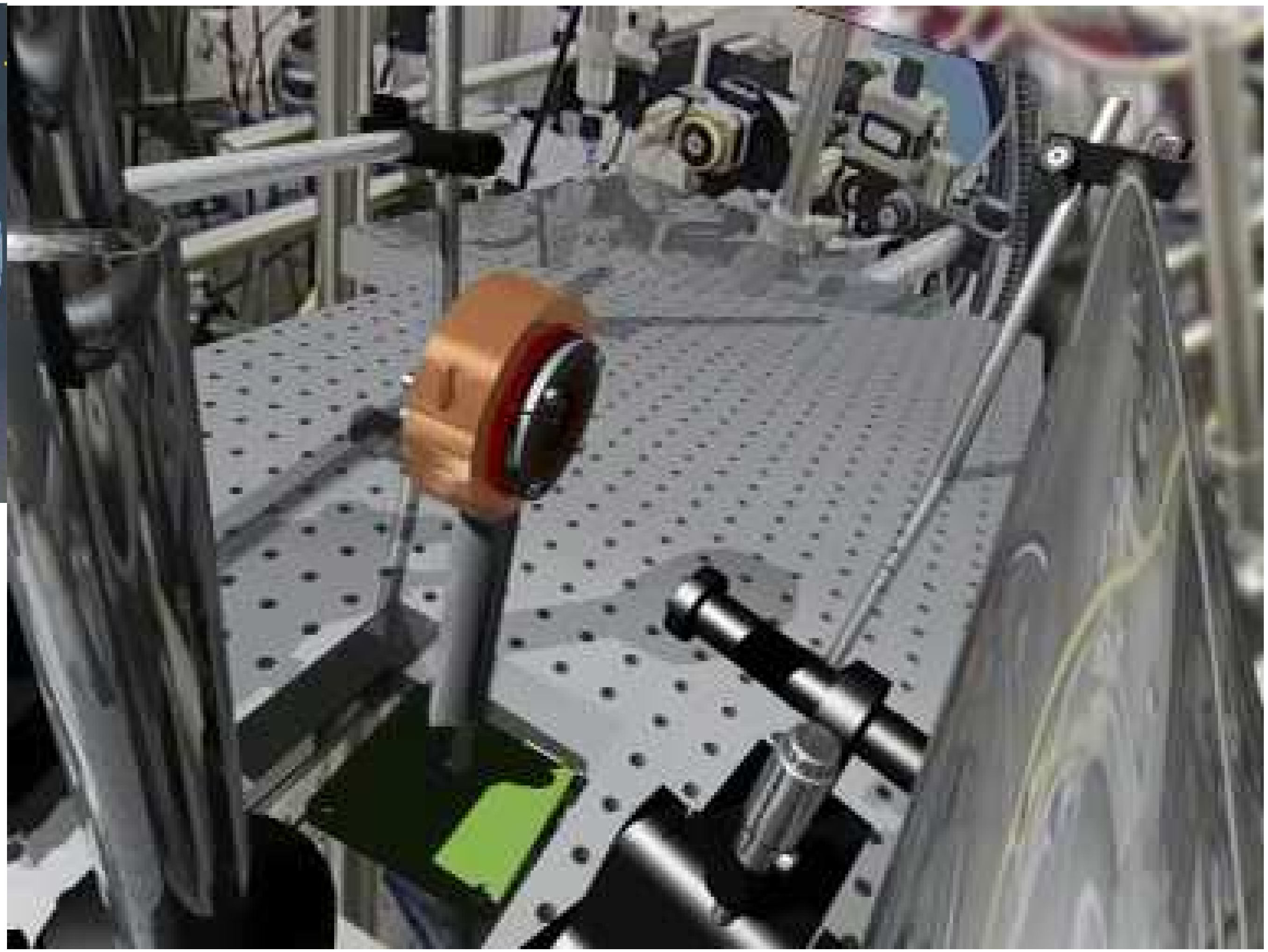




Courtesy M. Mezouar (ESRF-ID27)

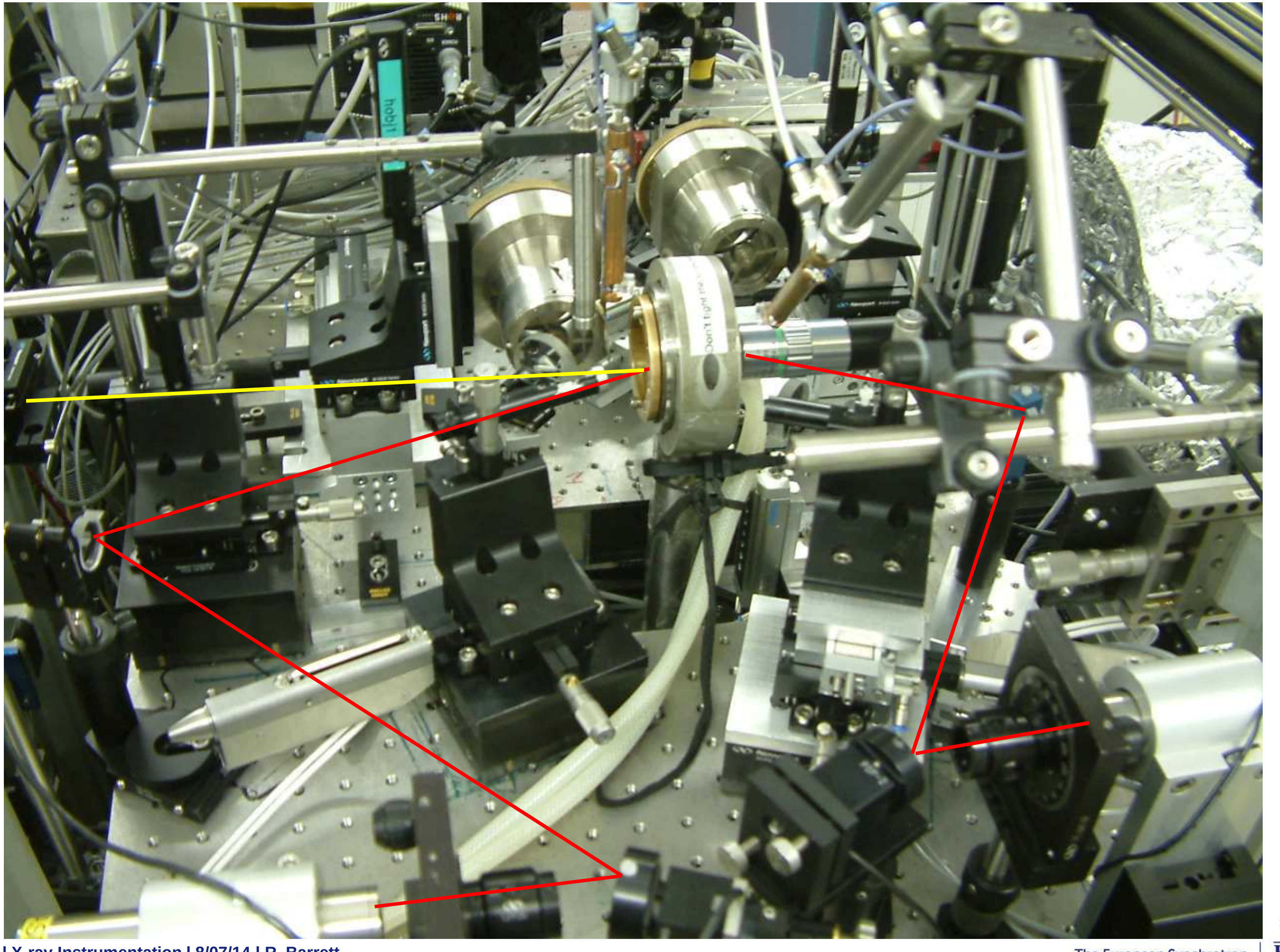
EXTREME CONDITIONS (T, P)





Courtesy M. Mezouar (ESRF-ID27)

EXTREME CONDITIONS (T, P) – ESRF - ID27



Courtesy M. Mezouar (ESRF-ID27)

Measure X-ray intensity (sometimes energy too) – selection depends upon various factors e.g.

- 0-D, 1-D, 2-D
- Spatial, energy resolution
- Efficiency (Energy), dynamic range
- Signal intensity: photon counting, integrating – max count rate
- Robustness
- Price

Earliest detector: Film – still used

Direct conversion

- Absorbed X-rays directly generate electrical signal e.g. photodiodes, pixel-detectors, silicon drift-diodes

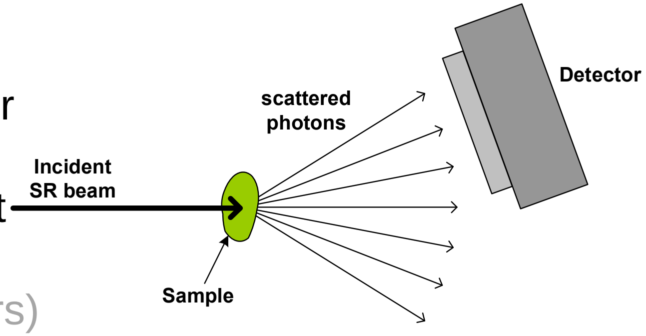
Indirect conversion

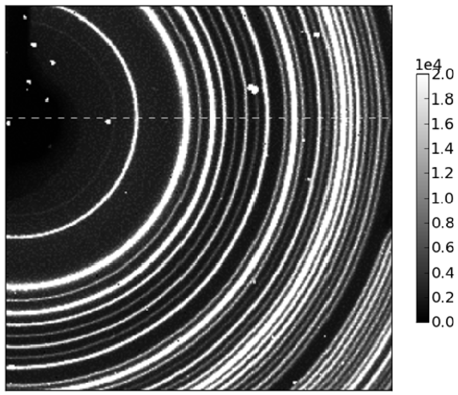
- X-rays absorbed by a conversion medium and secondary signal such as light, heat detected e.g. scintillator PMT, optically coupled CCD, bolometer, calorimeter

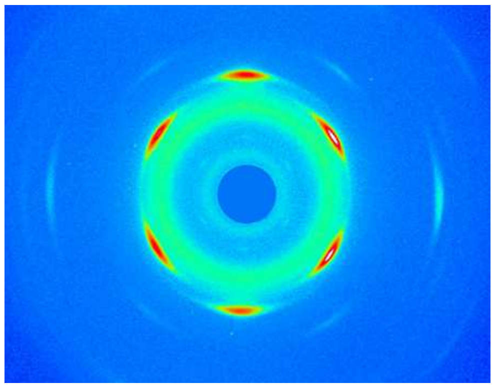
- Scattered photons *conserve their initial energy*, i.e. only momentum changed
  angular measurements are required, usually measured with 2D spatially resolving detectors
  $\Rightarrow$angular resolution can be varied by changing detector-sample distance
- Large dynamic range may be required (crystal diffraction can cover ~8 orders of magnitude!)

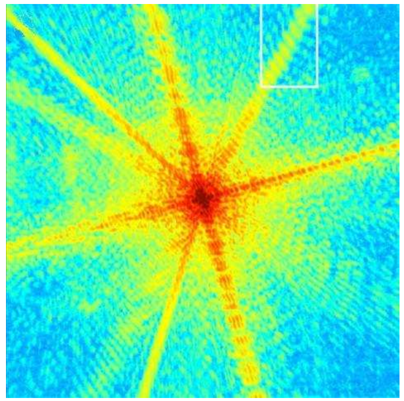
- detectors used:
  - scintillator-PMT; silicon-diode, -APD using diffractometer
  - solid state semiconductors (1D strip, 2D area PADs)
  - 'indirect detection' with flat panel a-Si or CMOS readout
    2D area scintillator-optic-CCD cameras

  (laser read-out phosphor image plates, MWPC gas detectors)





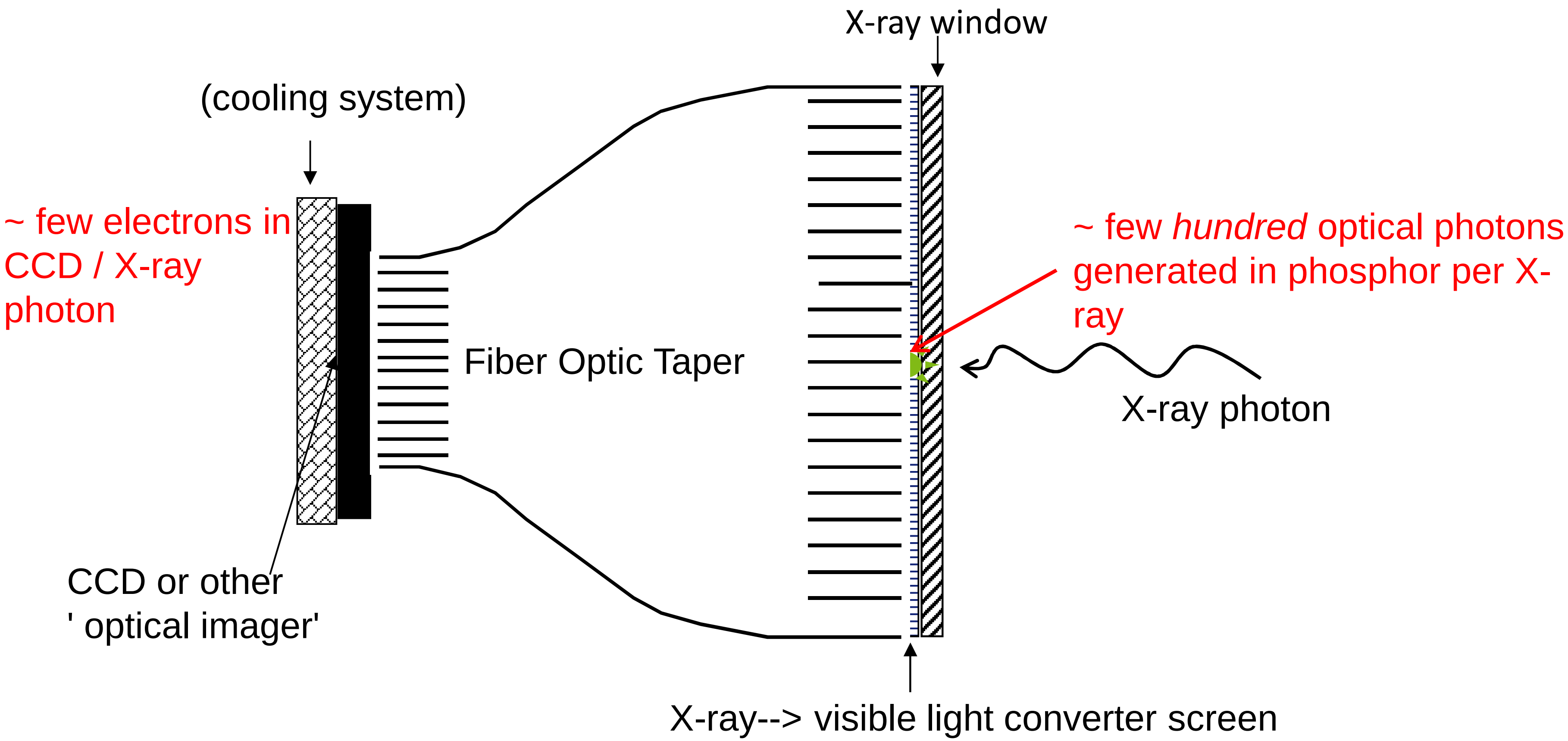




Courtesy: John Morse

'INDIRECT' X-RAY DETECTION: CCD CAMERA SYSTEMS

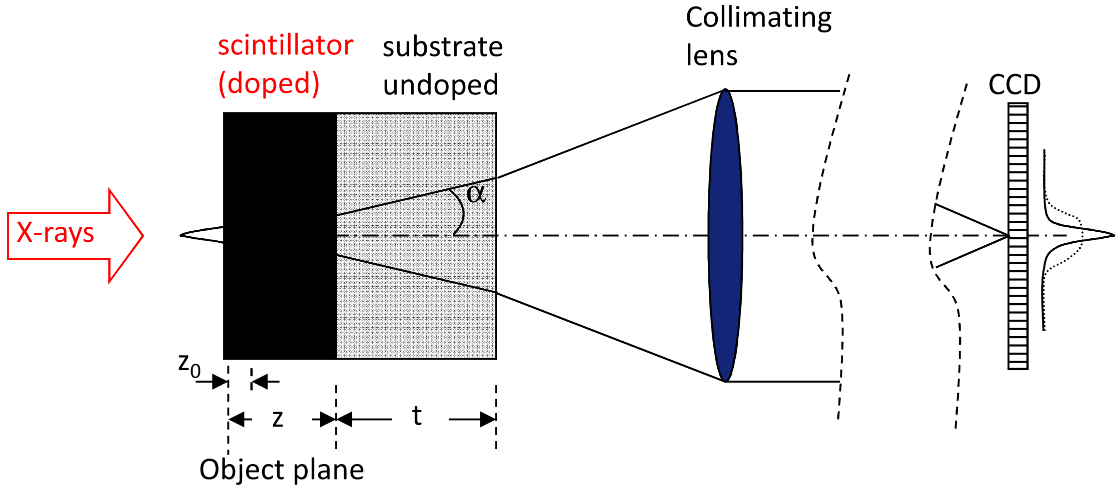
position-resolved 2D area detectors, especially high resolution X-ray imaging



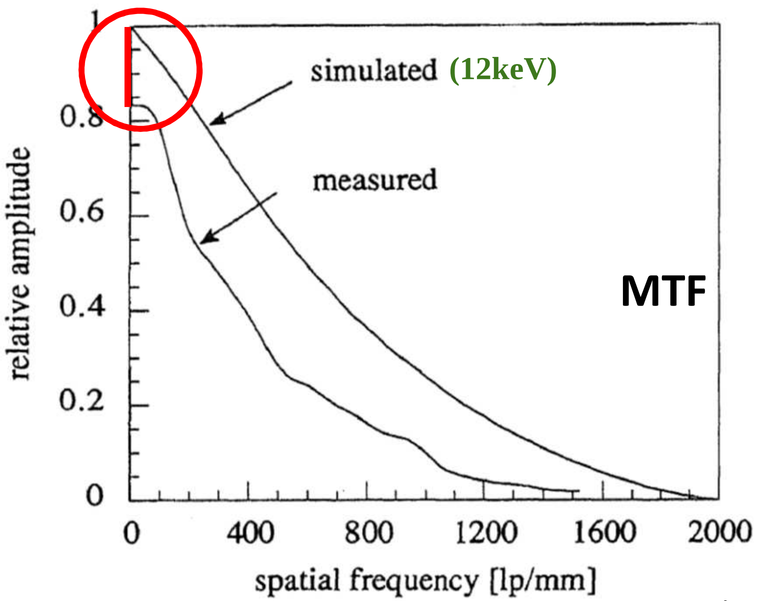
for detector areas above ~1cm^2, fibre optic gives more efficient optical coupling to CCD

Courtesy: John Morse

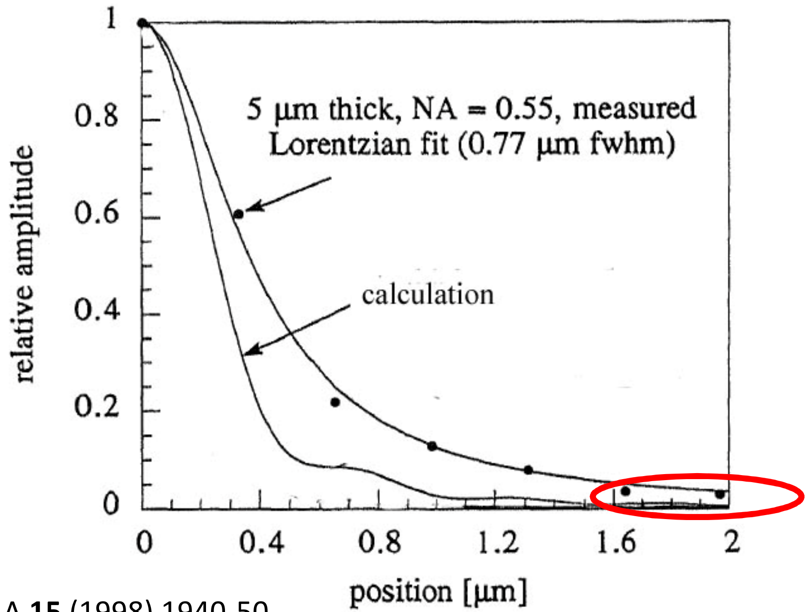
HIGH RESOLUTION CRYSTAL SCREEN-LENS-CCD SYSTEMS



e.g. doped YAG:Ce by liquid epitaxy on undoped 170µm YAG substrate,
X-rays, 100x magnification optics
CCD 24 µm pixel sampling



MTF → **LSF**

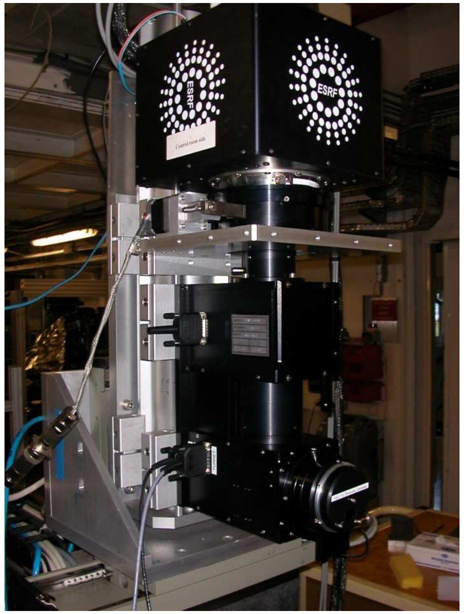


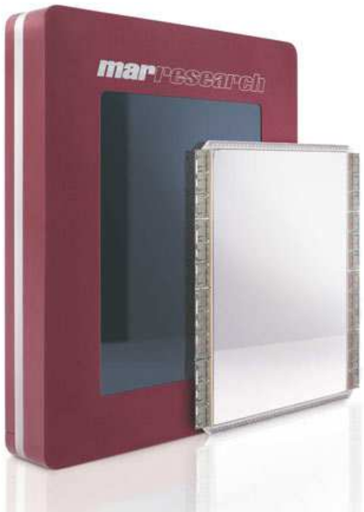
Koch et al, J. Opt. Soc. Am. A **15** (1998) 1940-50

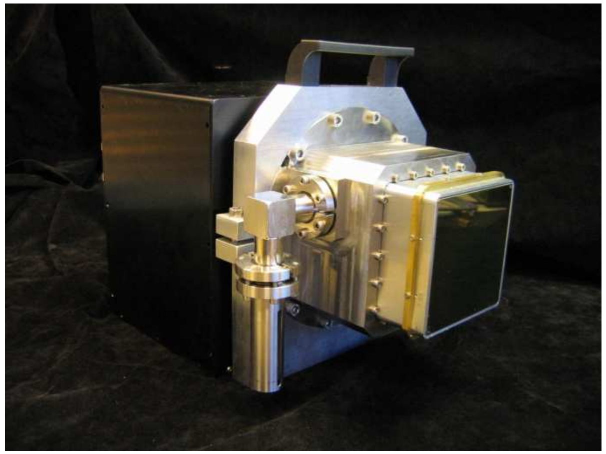
Courtesy: John Morse

2D X-RAY DETECTORS

Roentgen: "... Photographic plates and film are "susceptible to x-rays", providing a valuable means of recording the effects..."

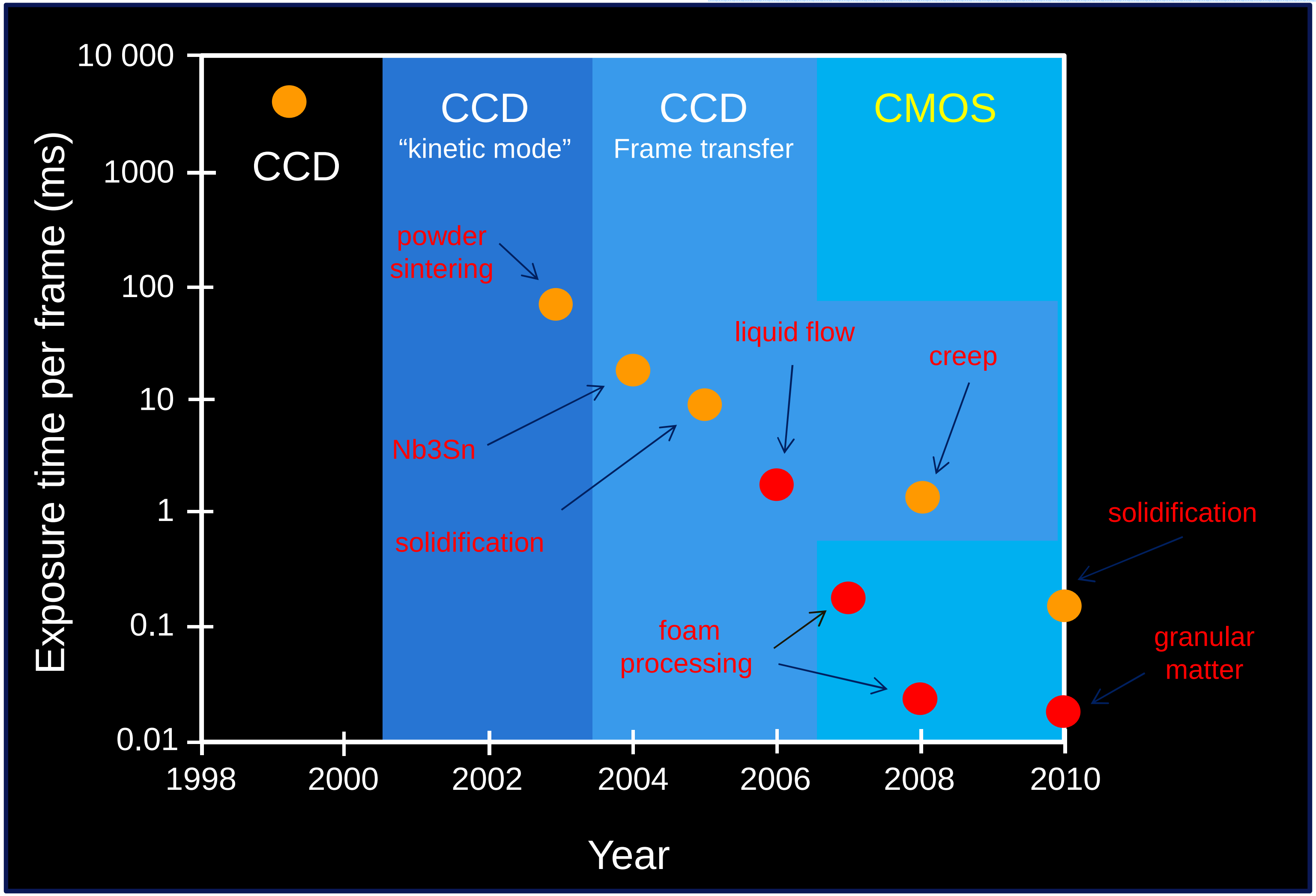






CHRONOLOGICAL EVOLUTION OF FAST IMAGING (ESRF)

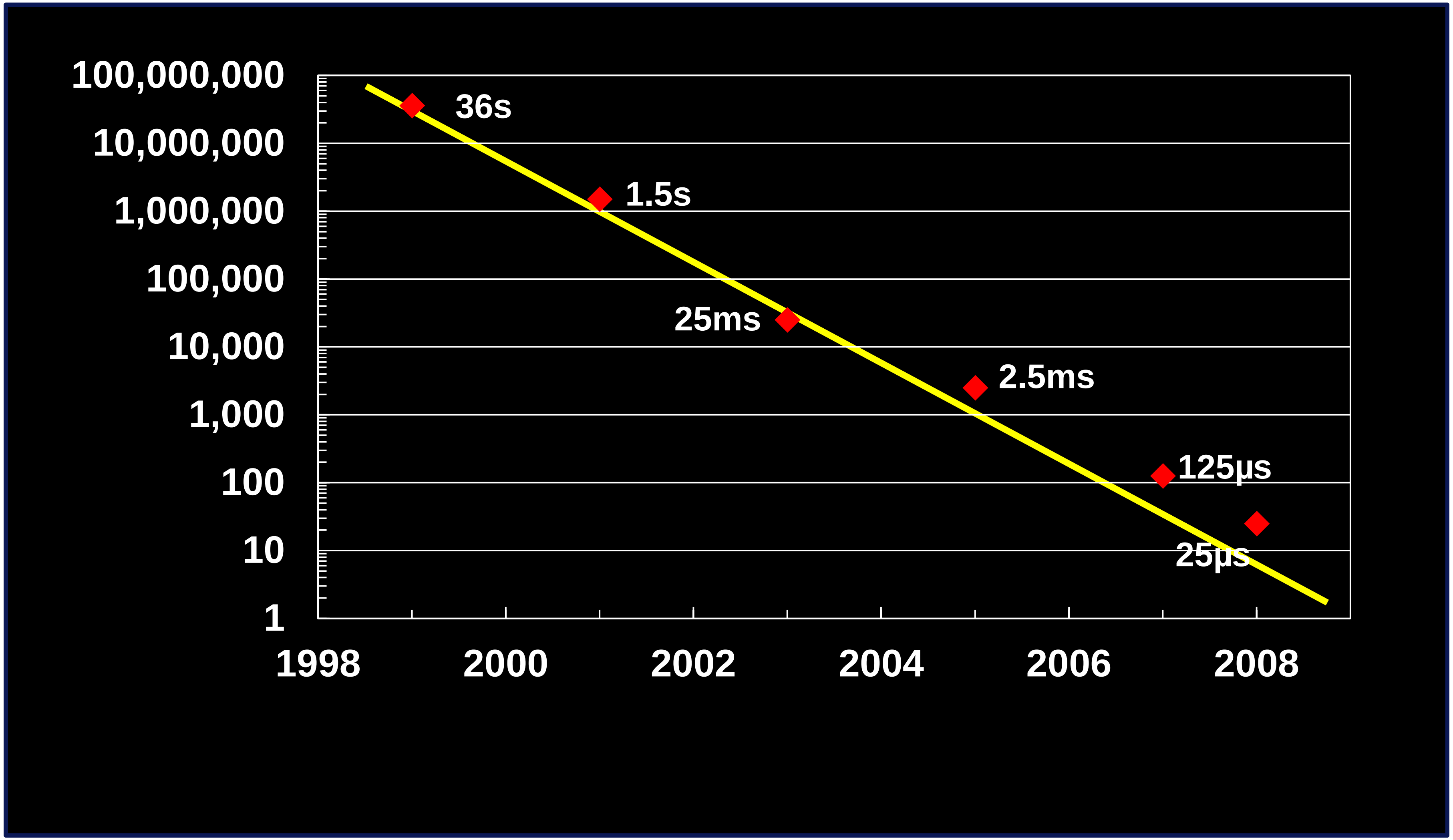
- Orange points: spatial resolution 1-2µm
- Red points: spatial resolution 10-20µm



Data courtesy: M. Di Michiel ID15-ESRF

EVOLUTION OF FAST HARD X-RAY IMAGING

FROM 100 FRAMES/1h TO 100FRAMES/2ms



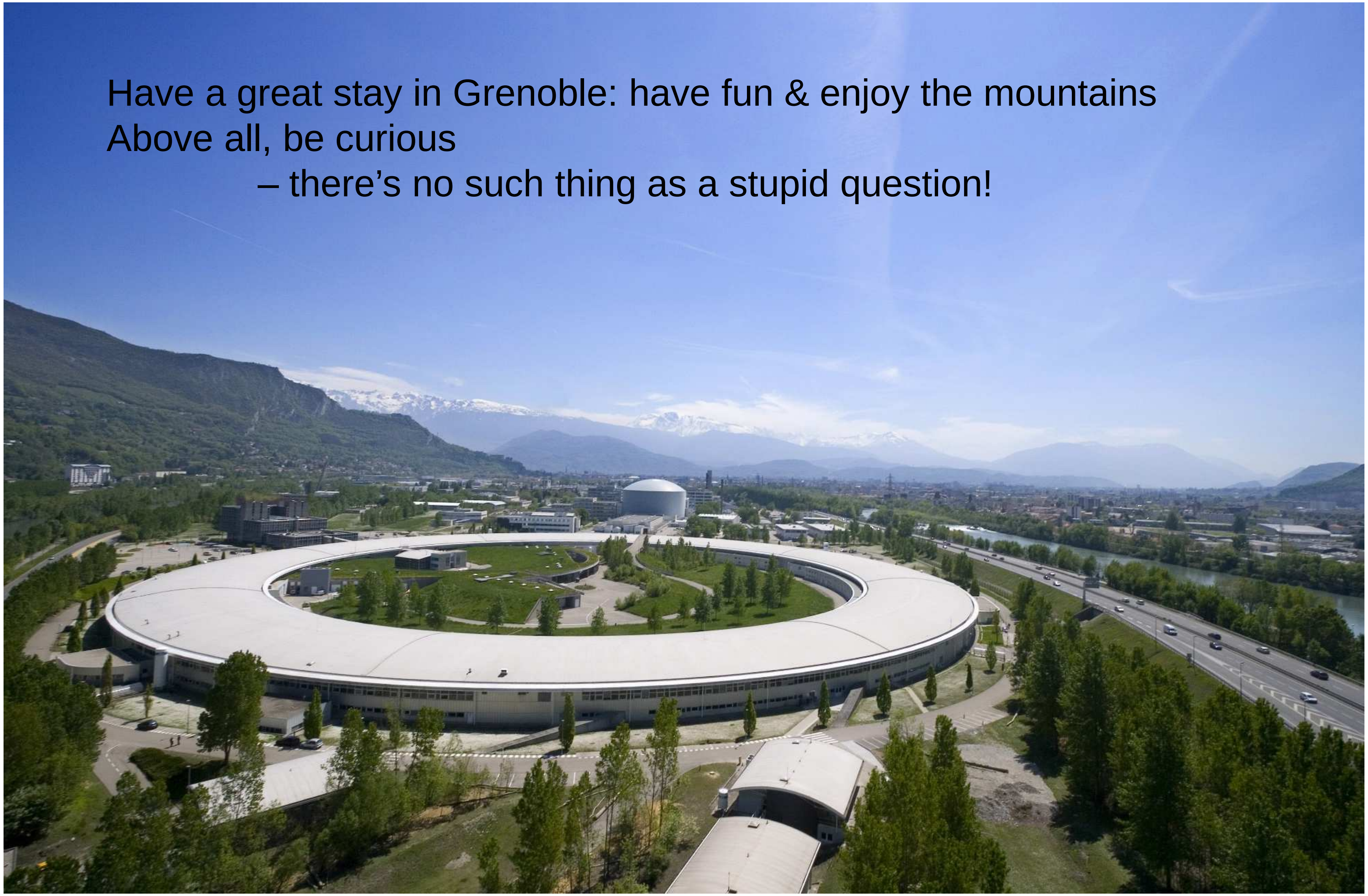
Challenge: Data processing and storage: 3000Gb / Day

ID15- ESRF

CONCLUSIONS

- **Present day synchrotron radiation sources offer a unique tool for probing the interior of matter over length scales ranging from the few cm to sub-atomic dimensions**
- **The full potential of the continually improving sources can only be exploited by parallel developments in appropriate X-ray instrumentation**
  - **X-ray optics/ optomechanics**
  - **Sample alignment systems**
  - **Sample environments**
  - **X-ray Detectors**
- **The new capabilities of instrumentation in these fields mean that increasingly sample throughput is limited by:**
  - **Sample exchange**
  - **Evaluation of data quality**
  - **Instrument control (optimised data collection)**
  - **Data handling and archiving**
- **ESRF Phase II Upgrade will include an ambitious instrumentation development program addressing many of these issues**

THANK YOU


Have a great stay in Grenoble: have fun & enjoy the mountains
Above all, be curious
– there's no such thing as a stupid question!