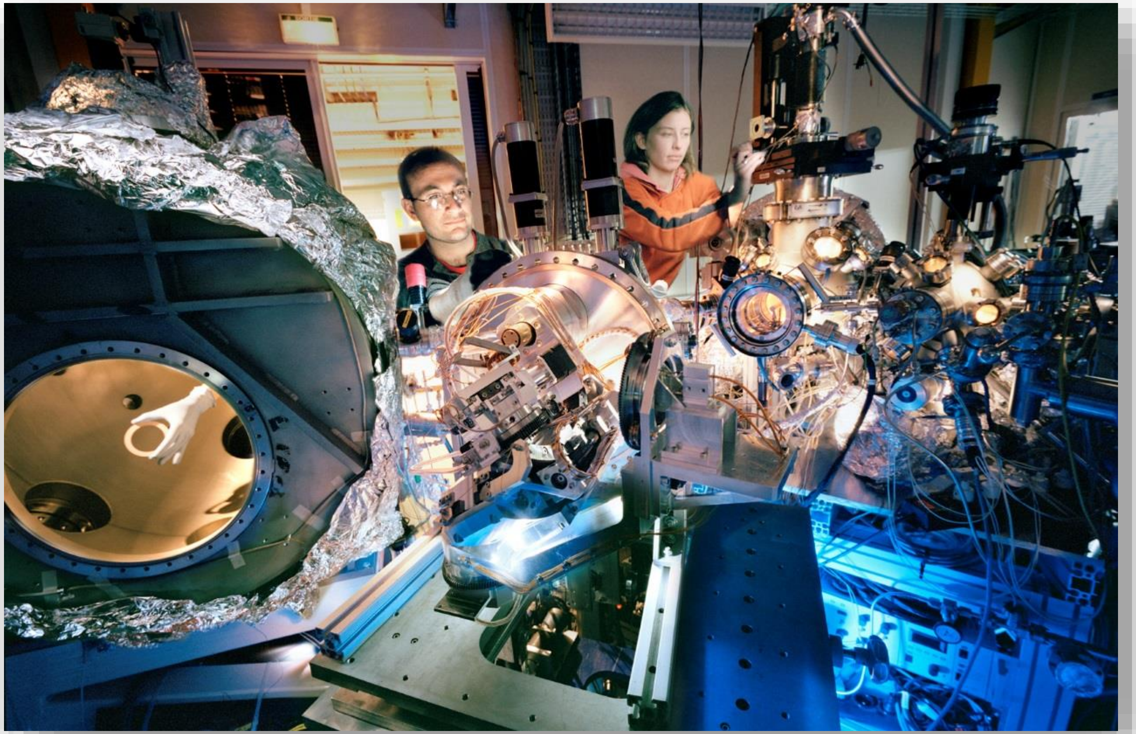
ESRF | The European Synchrotron

X-RAY INSTRUMENTATION

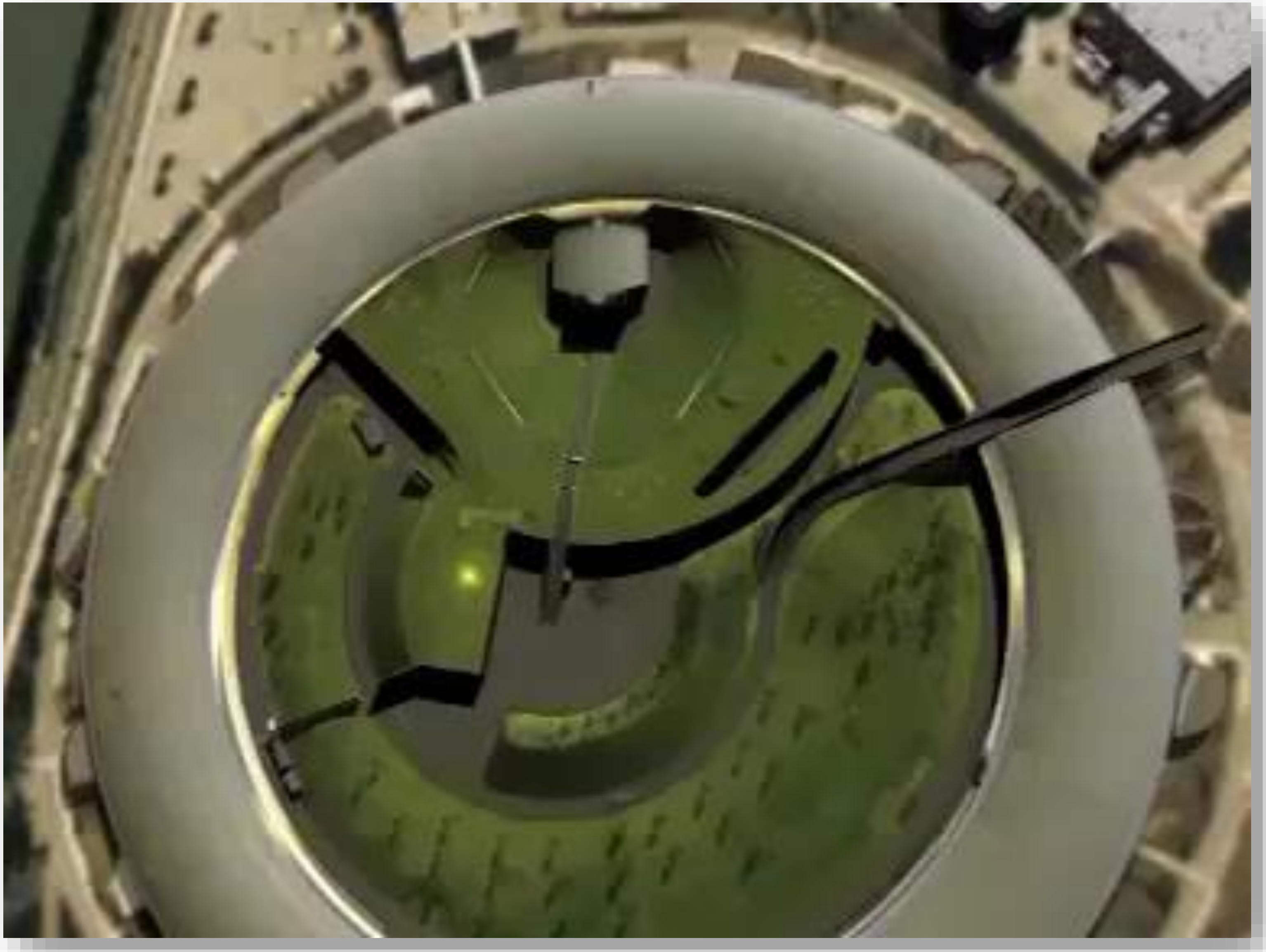


Outline:

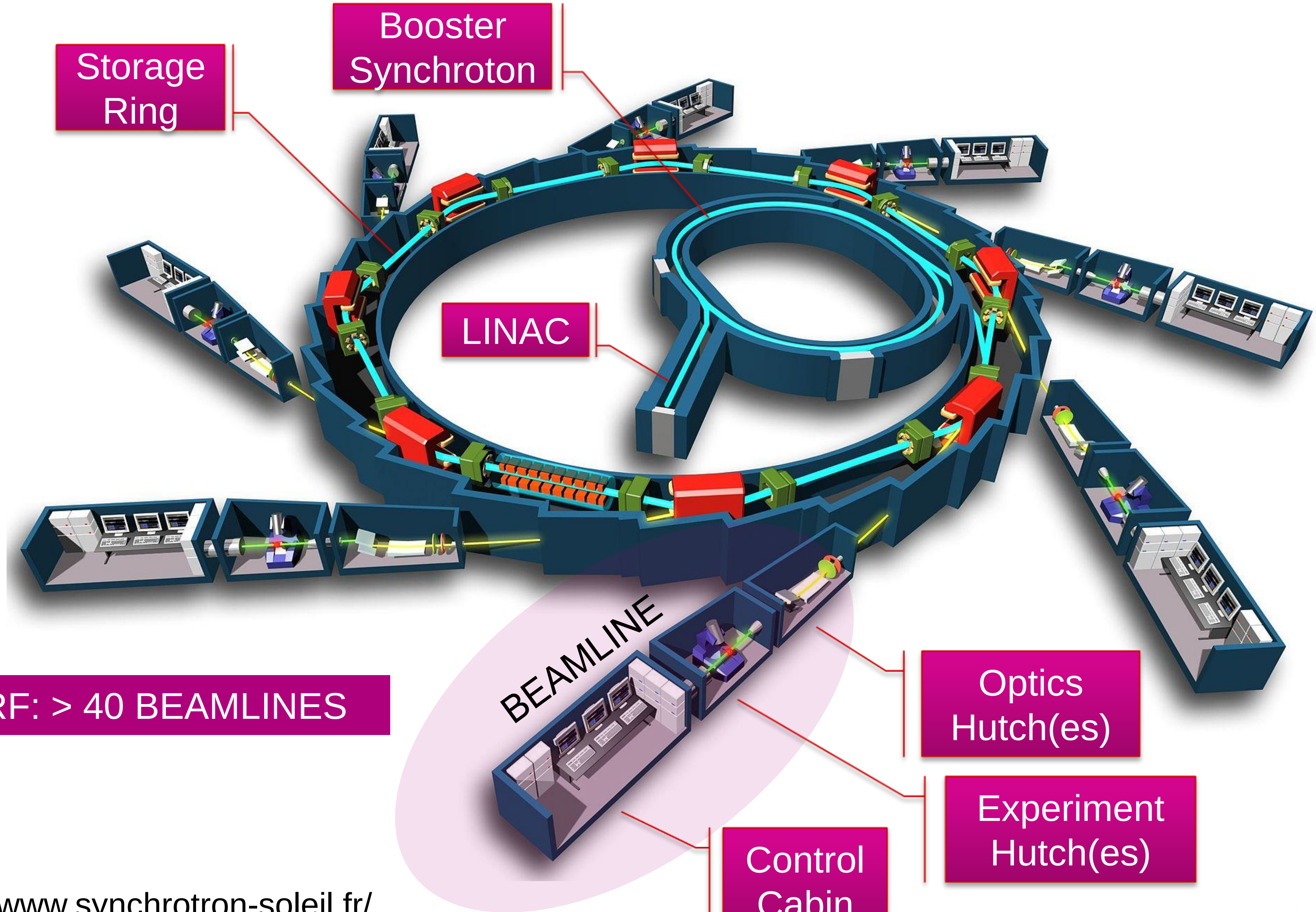
- Beamlines
- X-ray optics
- End-stations
- Sample environments
- Detectors

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

RECAP OF THE SYNCHROTRON SOURCE



SCHEMATIC OF A SYNCHROTRON RADIATION (SR) LIGHT SOURCE

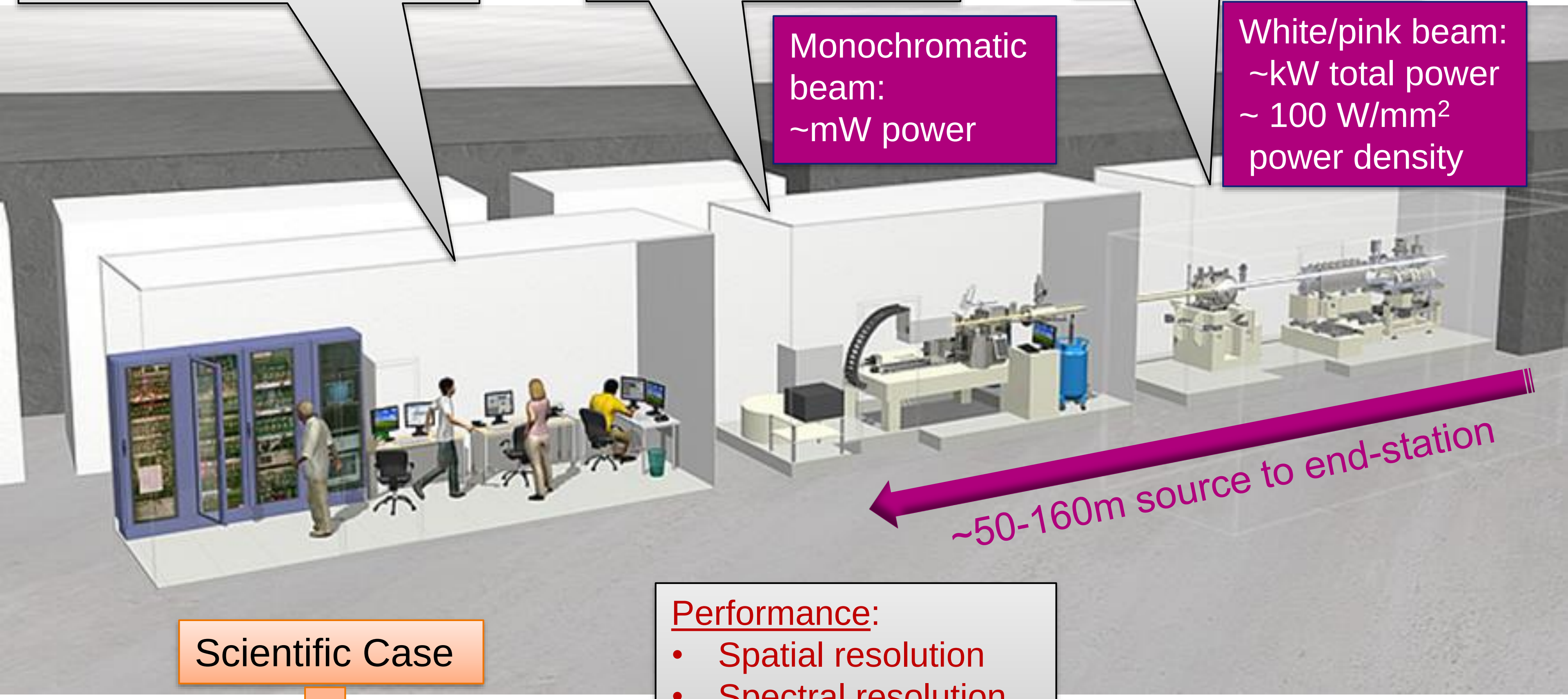


ESRF: > 40 BEAMLINES

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

A TYPICAL BEAMLINE LAYOUT

Lead Radiation shielding



Scientific Case

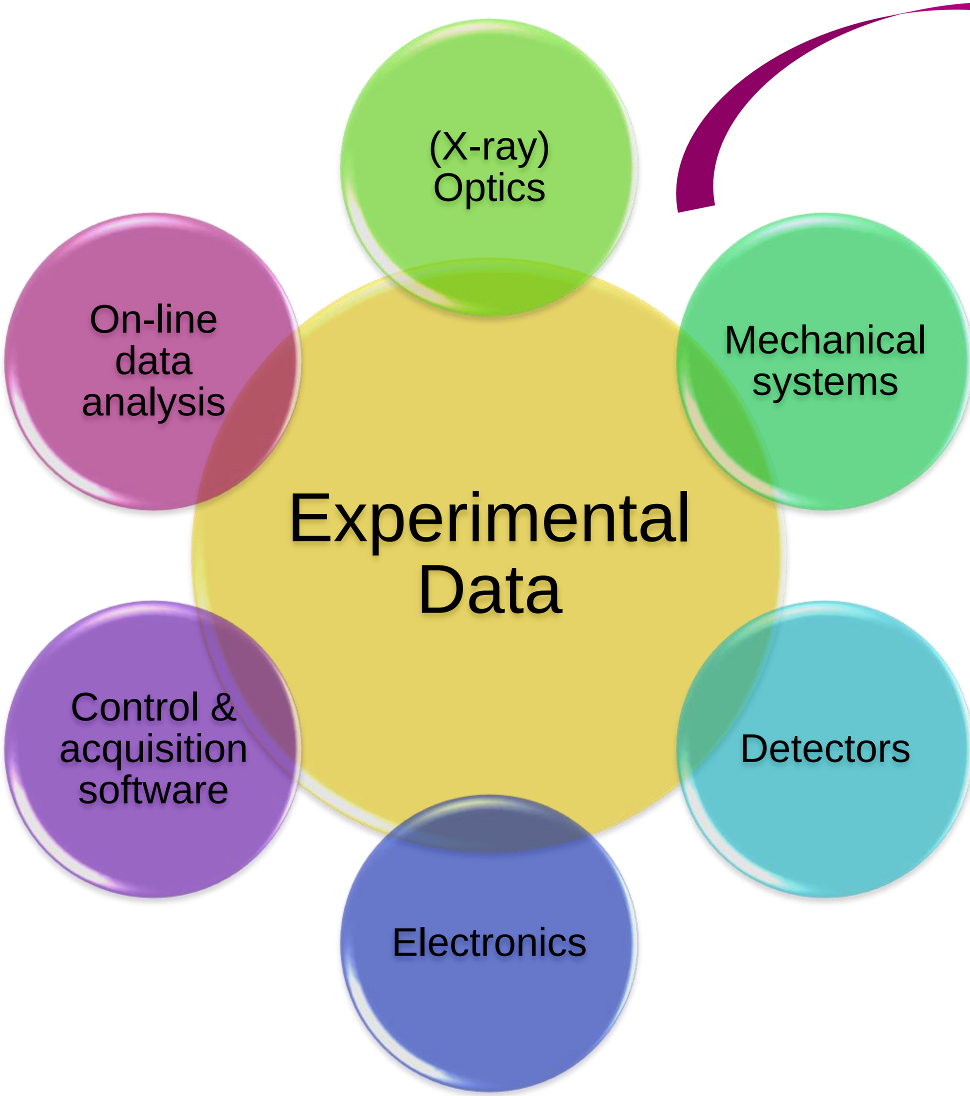
Performance:

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

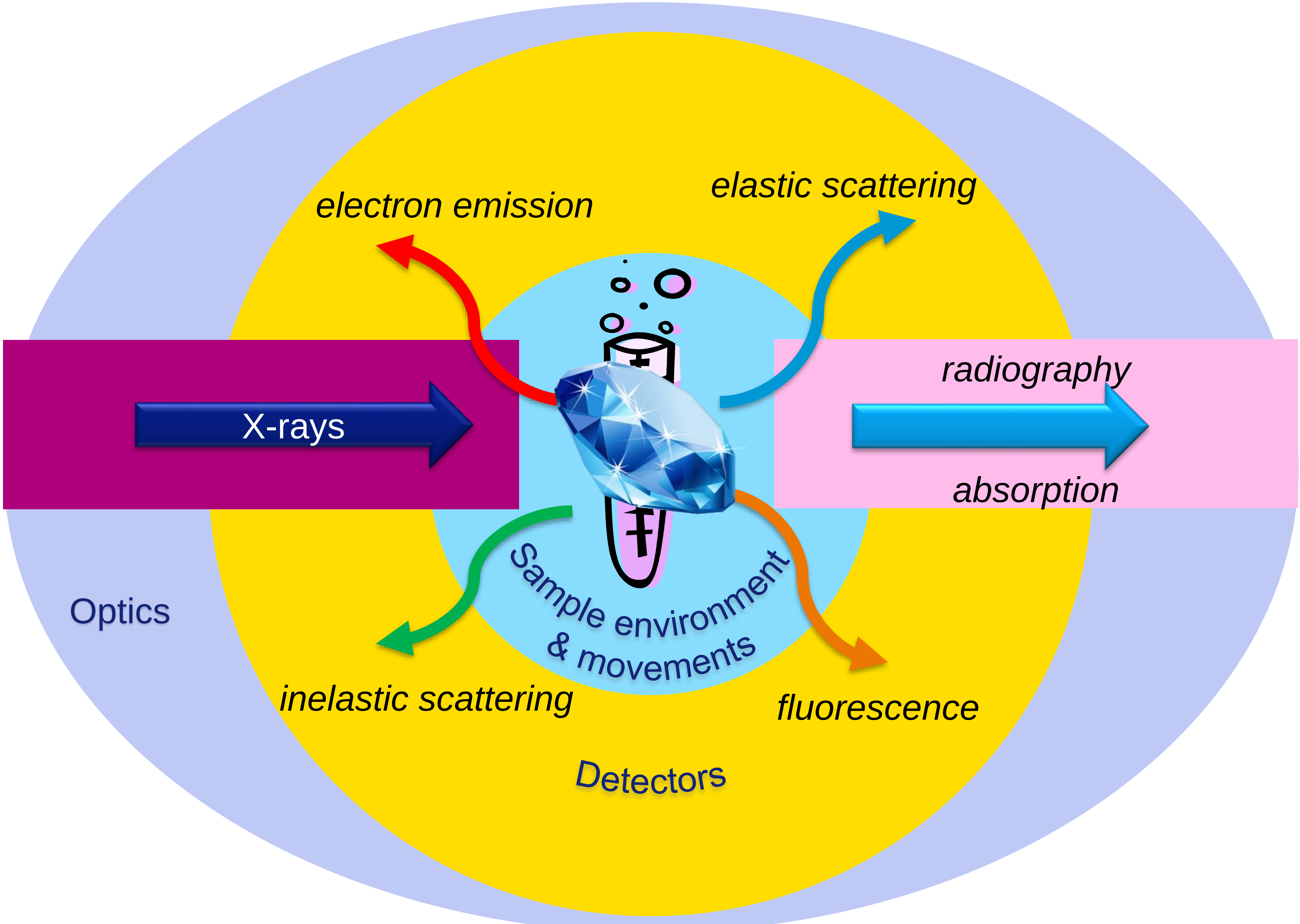
X-RAY BEAMS AT 3RD/4TH GENERATION SR SOURCES

- Beam size
  - Unfocused: few mm to few cm (source is weakly divergent)
  - Focused beam: < 100 nm to ~10's μm
- Energy range/tunability
  - 0.1 eV < E < 0.5 MeV (at ESRF mostly 3-100 keV ≈ 4-0.125 Å)
- Energy bandwidth (ΔE/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^2/0.1%bw (10^{11} higher than conventional sources) ⇒ photon flux (@ ΔE/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?



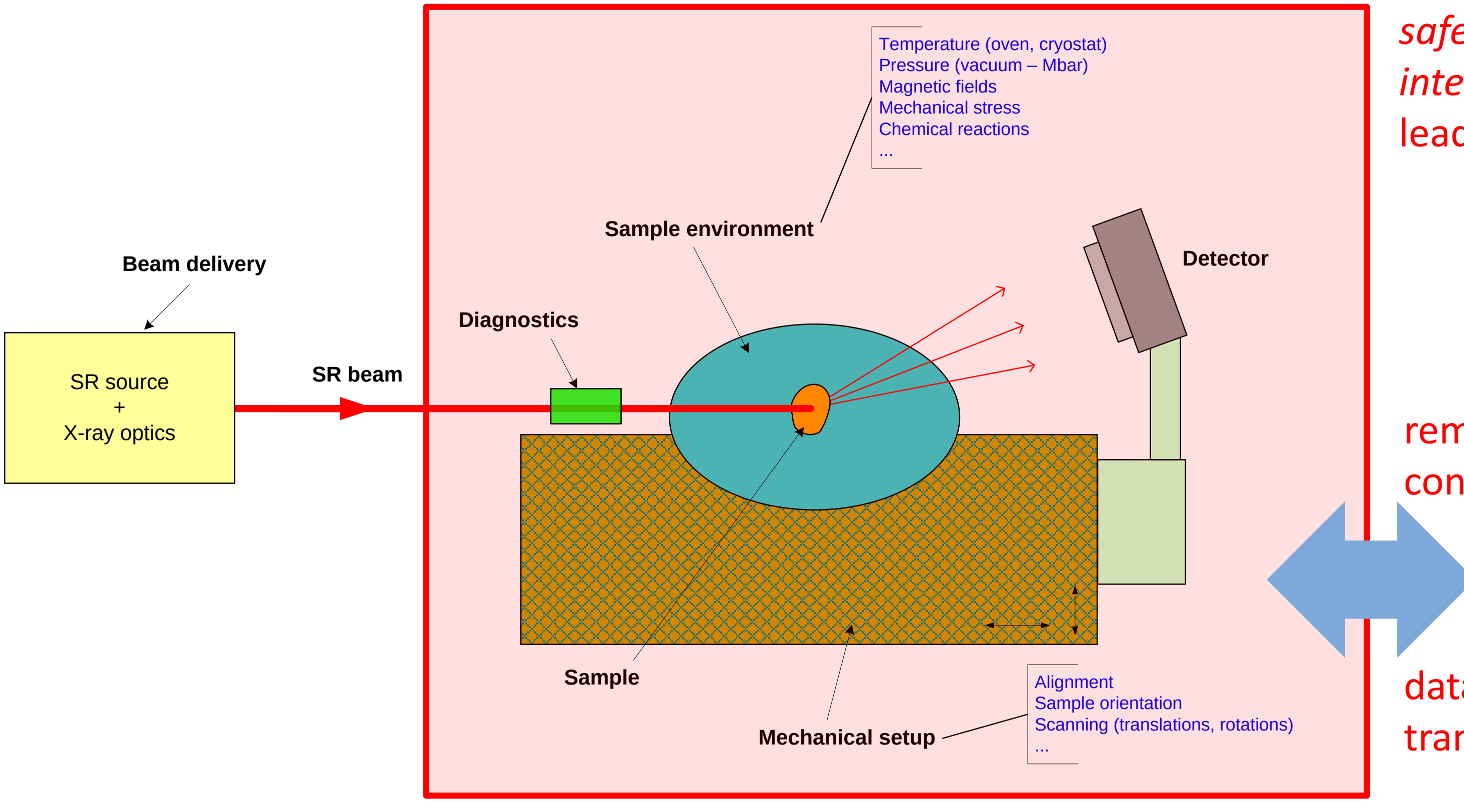
A TYPICAL EXPERIMENT



experiments are built around the samples to be measured

$\Rightarrow$ importance of sample environment (temperature; pressure; E and B fields…)

need to physically manipulate sample during measurements (position, rotation…)



safety interlocked lead hutch

remote control

data transmission

rapid turnover of samples and experiments

sample(s) in beam for minutes to hours

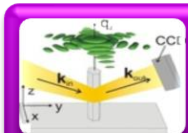
experiments typically last a few days

Courtesy: John Morse

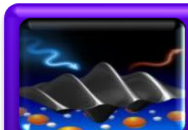
DIVERSITY OF APPLICATIONS ⇒ WIDE RANGE OF INSTRUMENTATION

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

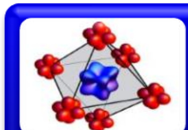
Structural biology



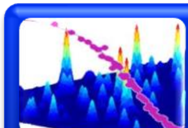
Structure of materials



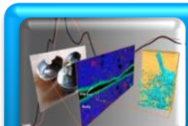
Electronic structure, magnetism & dynamics



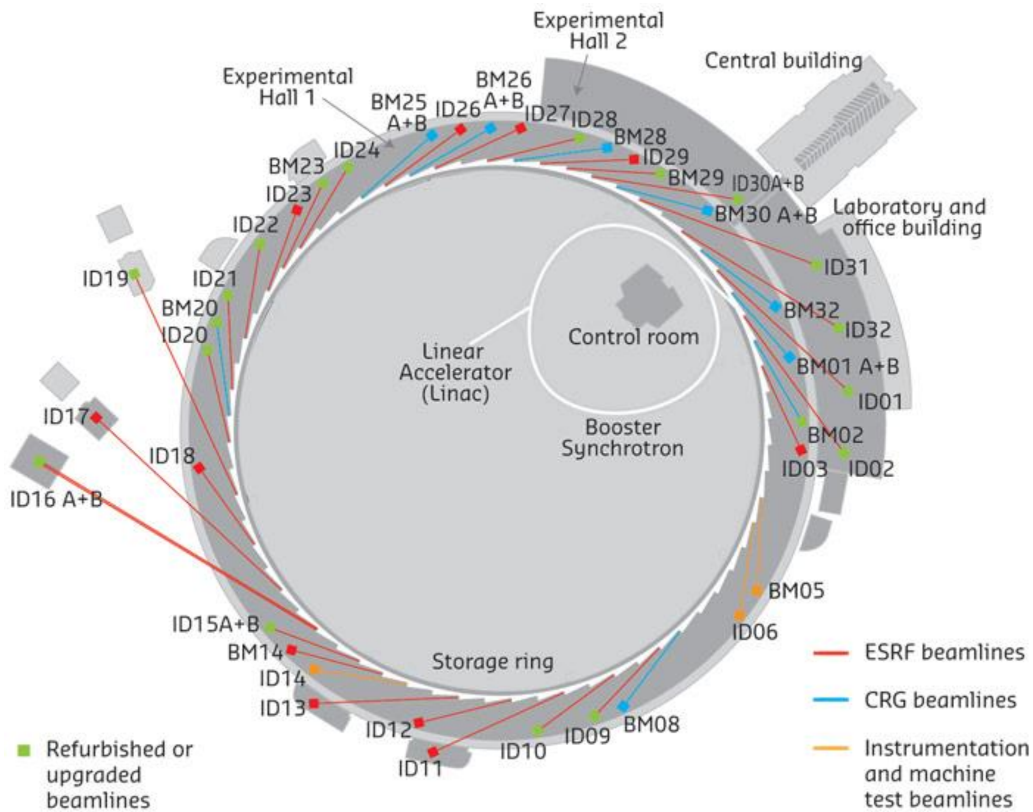
Matter at extremes



Complex systems & biomedical sciences



Multimodal-analytical nanoprobes



A COMPLETE SUITE OF TECHNIQUES

Specific

Sample environments

X-Ray fluorescence

- Composition
- Quantification
- Trace element mapping

X-ray diffraction & scattering

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

X-ray imaging

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

Infrared FTIR-spectroscopy

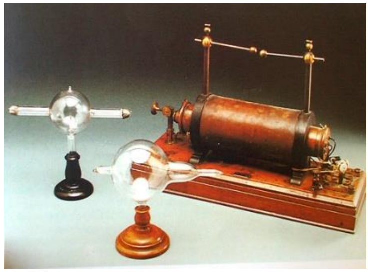
- Molecular groups & structure
- High S/N for spectroscopy
- Functional group mapping

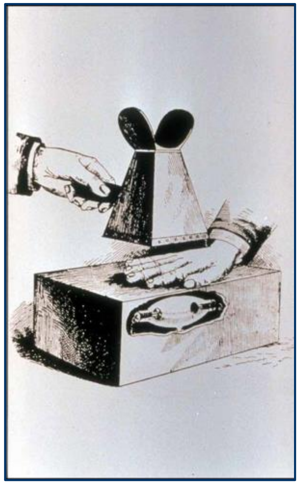
X-ray spectroscopy

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

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)



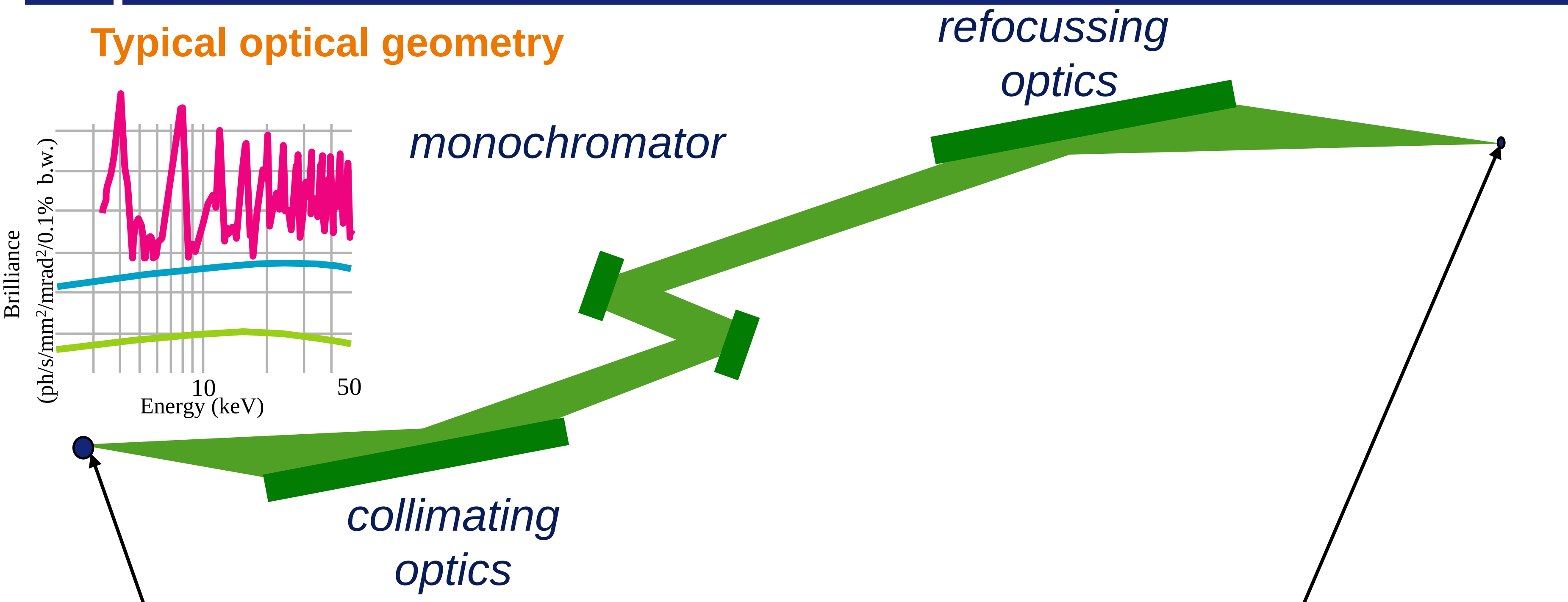


- “... The refractive index.... cannot be more than 1.05 at most.... X-rays cannot be concentrated by lenses….”

- “... 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...”

Typical optical geometry



OPTICS

?

Source

- spectrum (ΔE/E)
- emittance (size x divergence)
- degree of spatial coherence
- brilliance (ph/s/mm^2/mrad^2/0.1%bw)
- polarisation (linear, circular, elliptic)

Sample

- beam size (µm)
- beam divergence (µrad)
- flux (ph/s)
- temporal coherence: ΔE/E
- spatial coherence
- polarisation

X-RAY OPTICS: MANY POSSIBLE APPROACHES

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

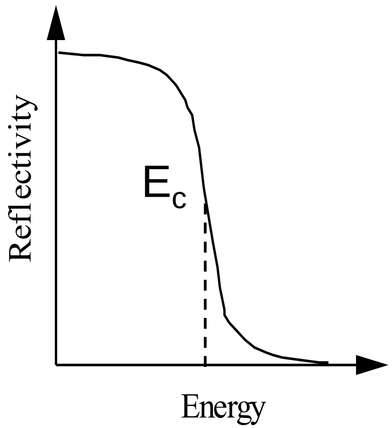
W.C. Röntgen
Über eine neue art von Strahlen.
Phys.-Med. Ges., Würzburg, **137**, p. 41, (1895)
English translation in Nature ***53****, p. 274*

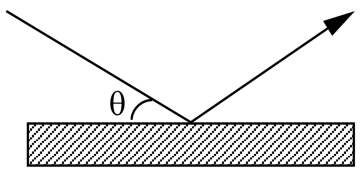
$n=1-\delta+i\beta$ with $\delta, \beta <<< 1$

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

- Very weak refraction
- Quite high absorption

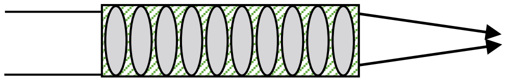
REFLECTION





- X-ray mirrors
- Capillaries
- Waveguides

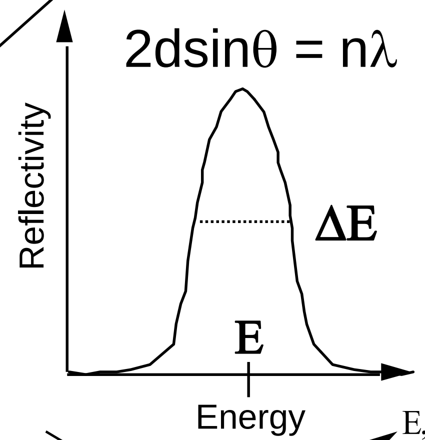
REFRACTION

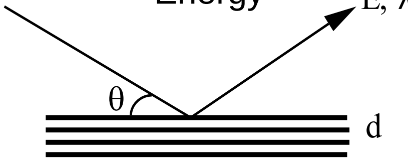


- Refractive lenses

DIFFRACTION

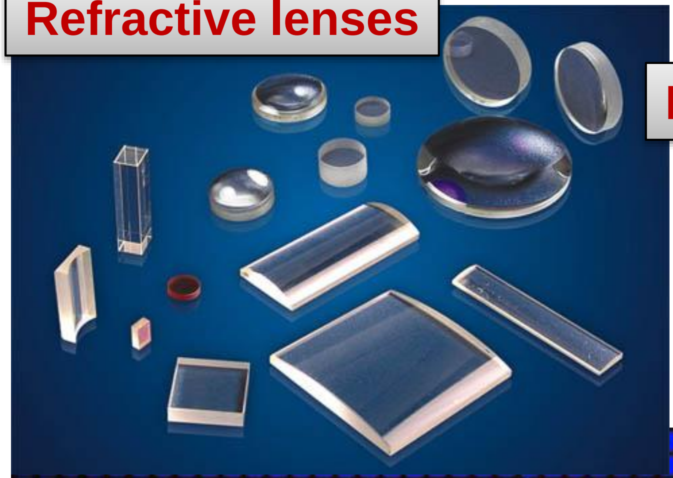
$$2d\sin\theta = n\lambda$$



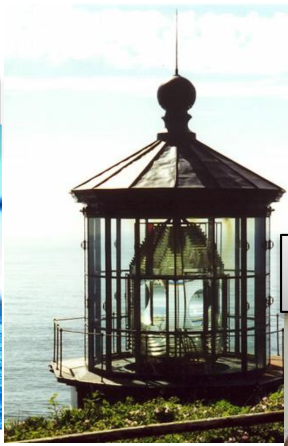


- Crystals & multilayers
- X-ray gratings
- Fresnel zone plates
- Bragg-Fresnel lens

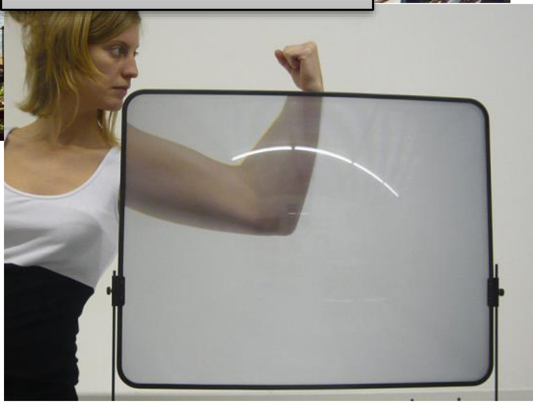
Refractive lenses

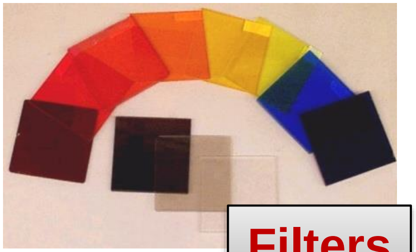


Polarising Optics



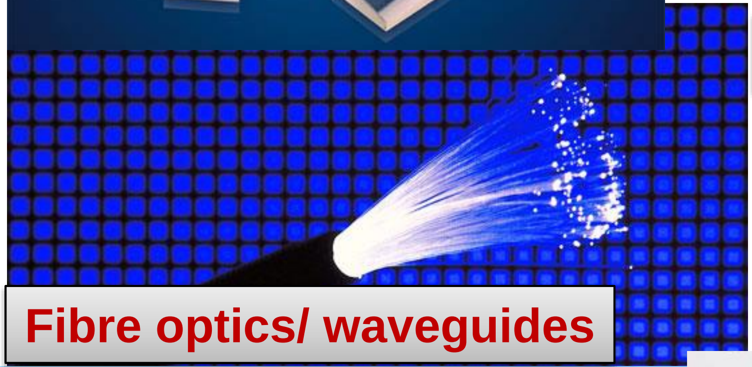
Fresnel lenses





Diffractive optics

Fibre optics/ waveguides

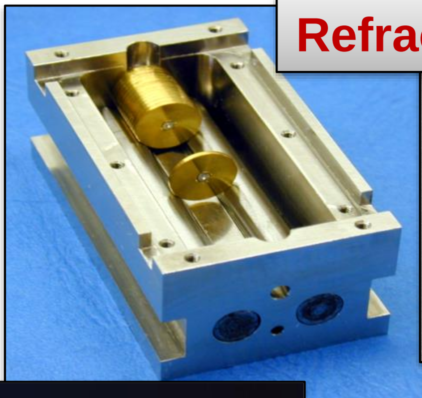
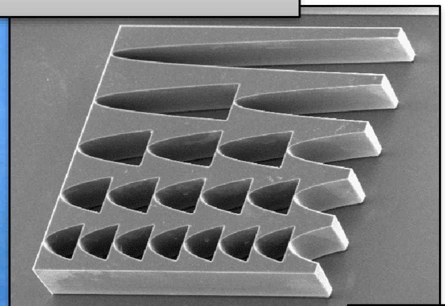


Mirrors

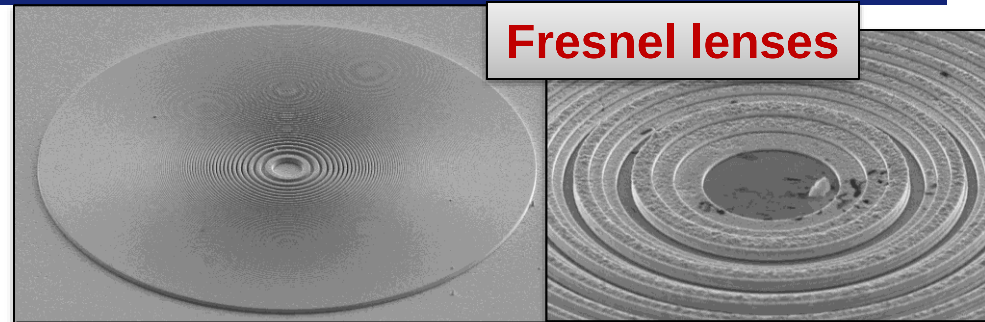
Filters

+ interferometers, …

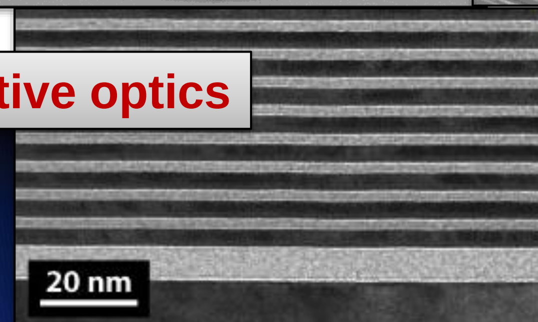
X-RAY OPTICS

Refractive lenses

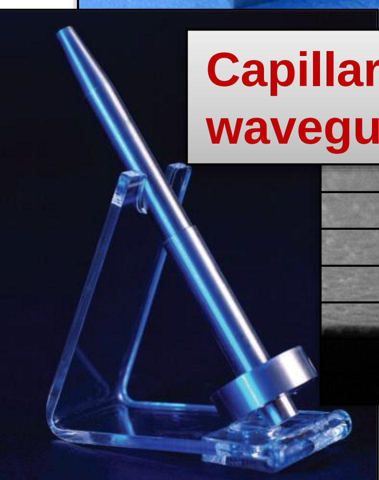
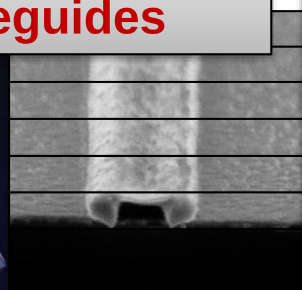


Fresnel lenses

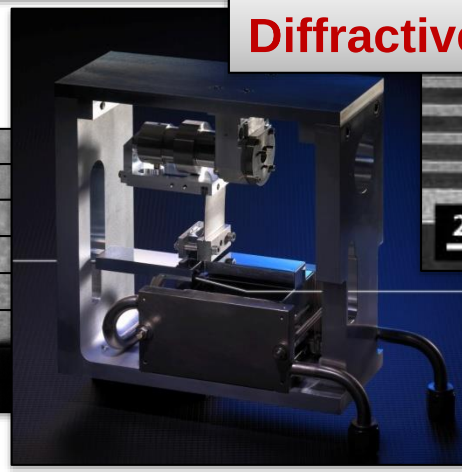


Filters

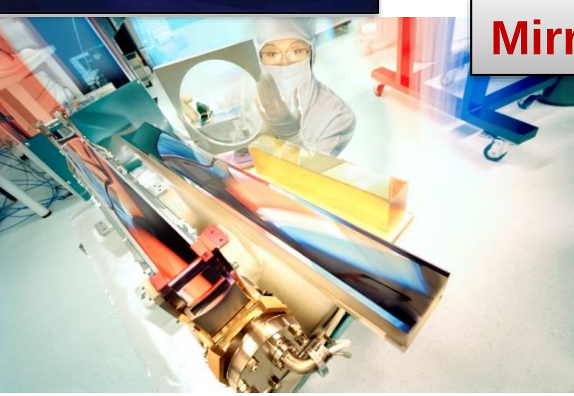
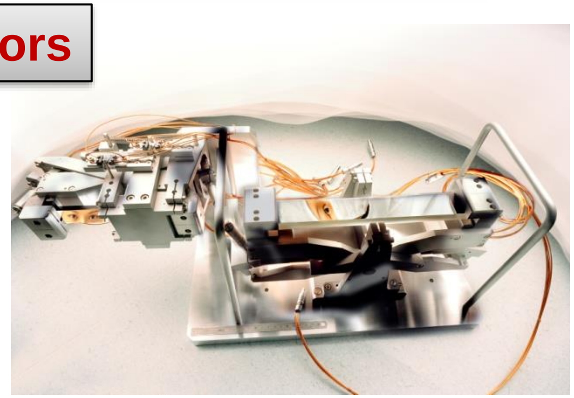
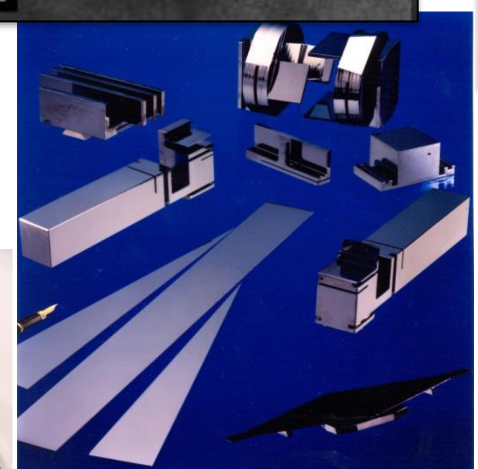
Capillary optics
waveguides

Diffractive optics

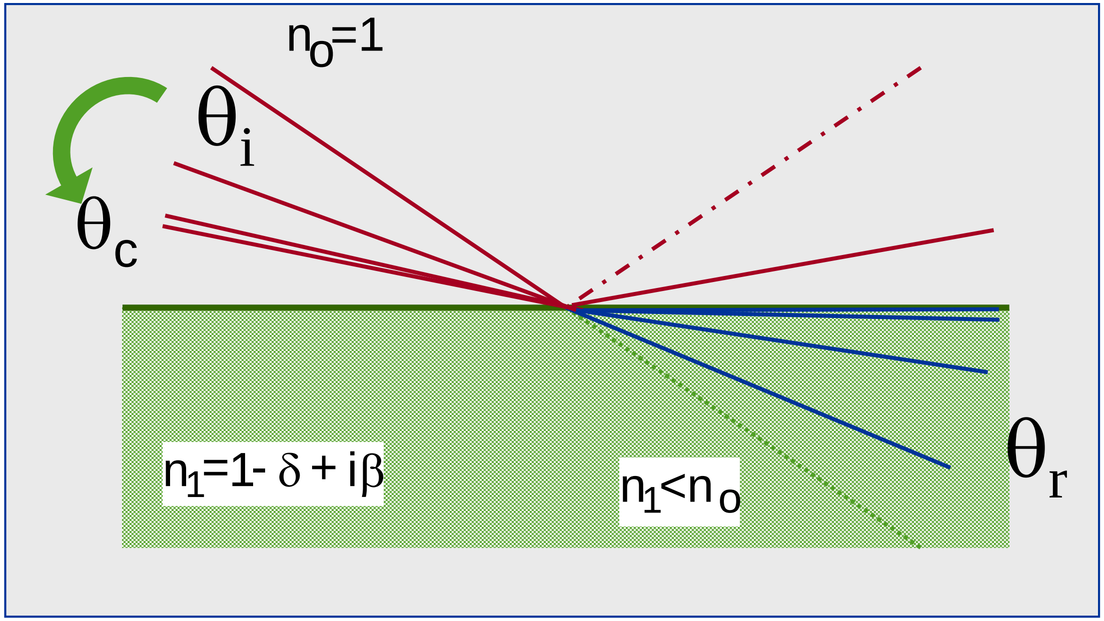


Mirrors

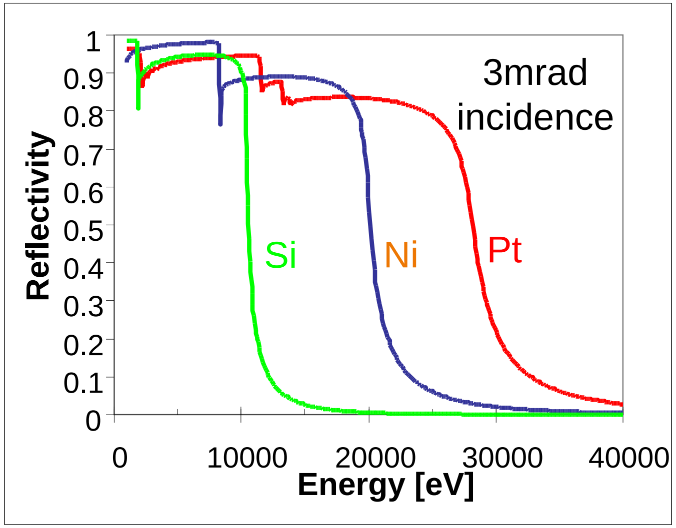




+ polarising optics,
interferometers, …

X-RAY MIRRORS: TOTAL EXTERNAL REFLECTION



'real' materials



Snell's Law:

$$n_o \cos\theta_i = n_1 \cos\theta_r$$

for δ << 1 and β << δ

$$\theta_c \approx \sqrt{2\delta} \propto \lambda\sqrt{Z}$$

The critical angle for total external reflection.

$$\theta_{c_{[mrad]}} E_{c_{[keV]}} = 19.83\sqrt{\rho_{[g/cm^3]}}$$

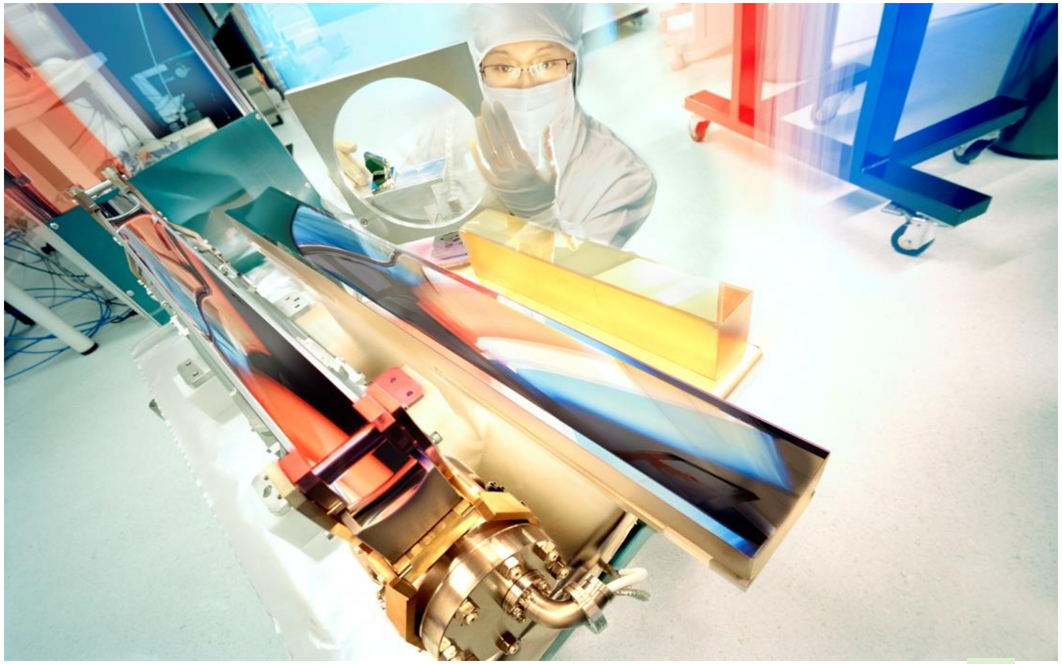
E=10keV

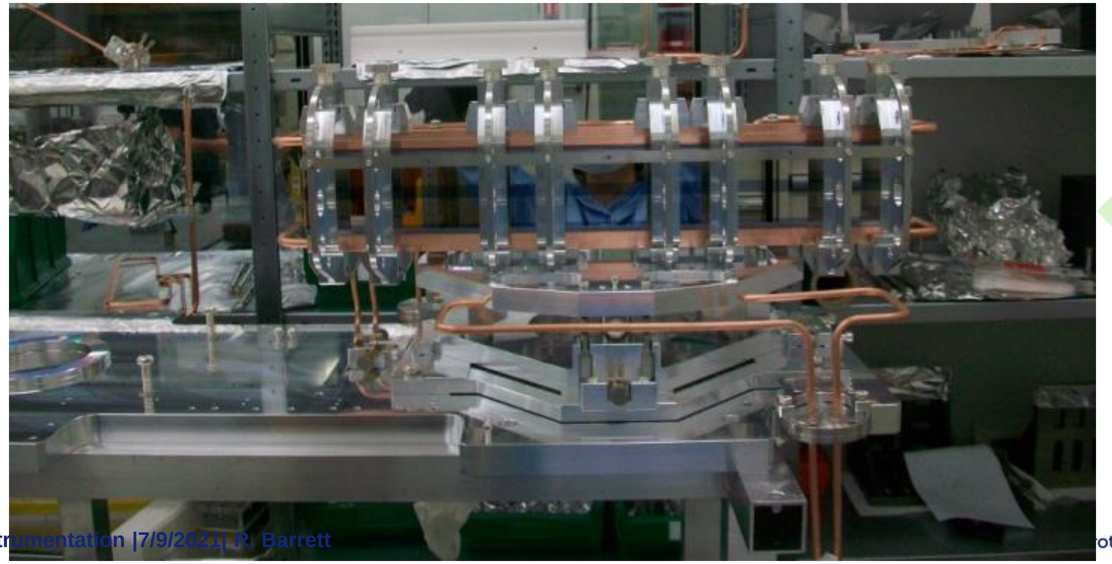
- Gold 9 mrad
- Nickel 6 mrad
- Silicon 3 mrad

- Grazing incidence ⇒ often long (gravity sag)
- Most SR mirrors manufactured from Si
- One or several coatings applied after polishing

See also: http://www.coe.berkeley.edu/AST/sxreuv/

SILICON OPTICS – X-RAY MIRRORS





X-RAY MIRRORS : MAIN FUNCTIONS

- ***Deflection***

  beam steering (different experiments, Bremsstrahlung)

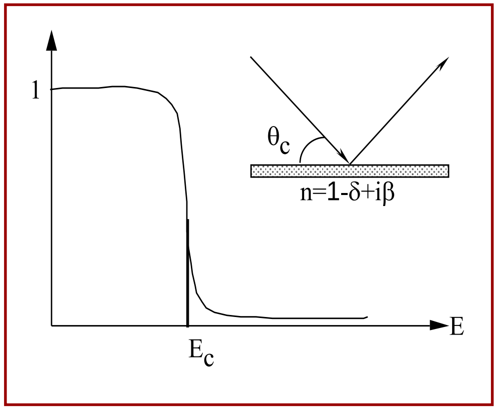
- ***Power filter***

  lower incident power on sensitive optical components

- ***Spectral shaper***

  energy low-pass filter (harmonic rejection)

  mirror+filter = spectral window



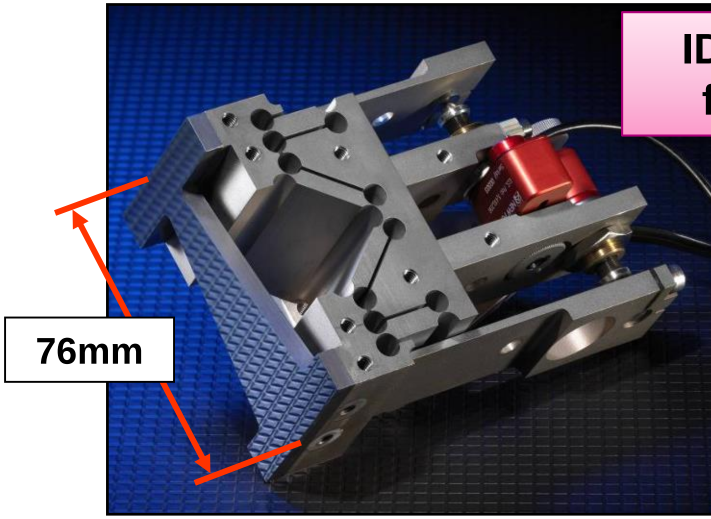
- ***Collimation or focusing – use of curved surfaces***

  wiggler & bending magnet : spherical, cylindrical, and toroidal mirrors

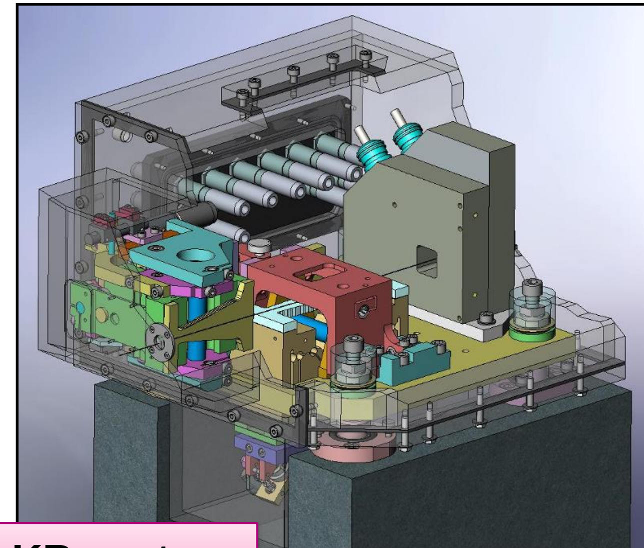
  microscopy & microprobe : source demagnification (ellipsoidal mirror, Kirkpatrick-Baez mirrors…)

J. Susini, Optical Engineering, 34(2), (1995)

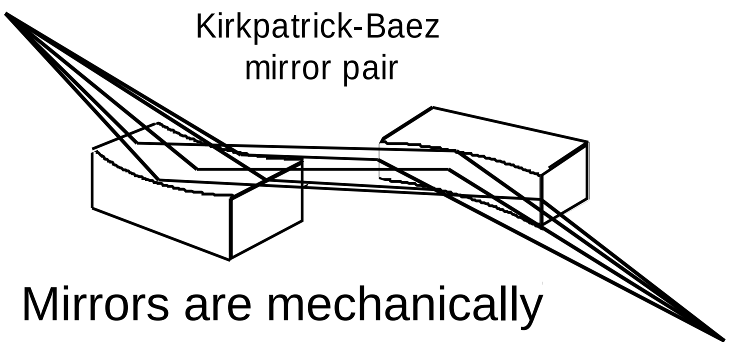
ESRF NANOFOCUSING 'KIRKPATRICK-BAEZ' MIRROR SYSTEM



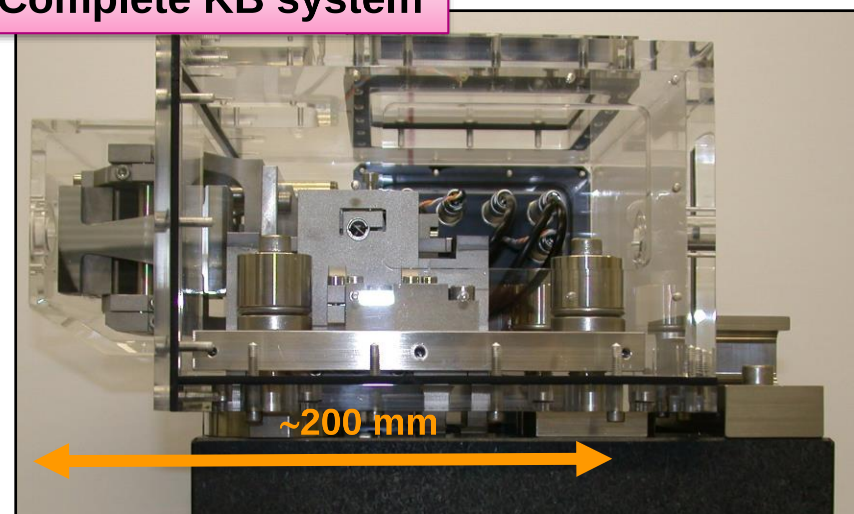
ID16B horizontally focusing bender



Complete KB system



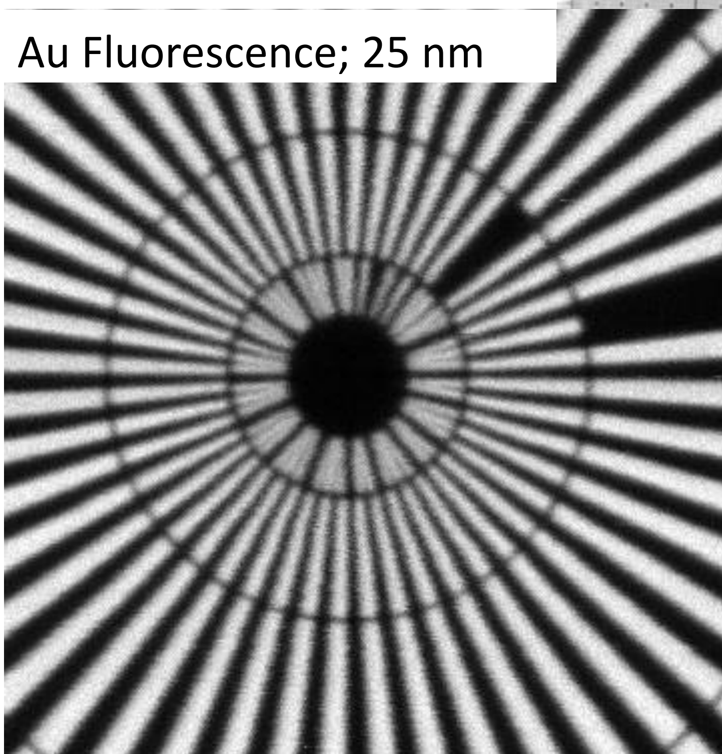
Mirrors are mechanically bent to elliptical profiles and focus X-rays



Sub 50 nm x 50 nm focus
Flux > 10^{12} ph/s (pink) on ID16B

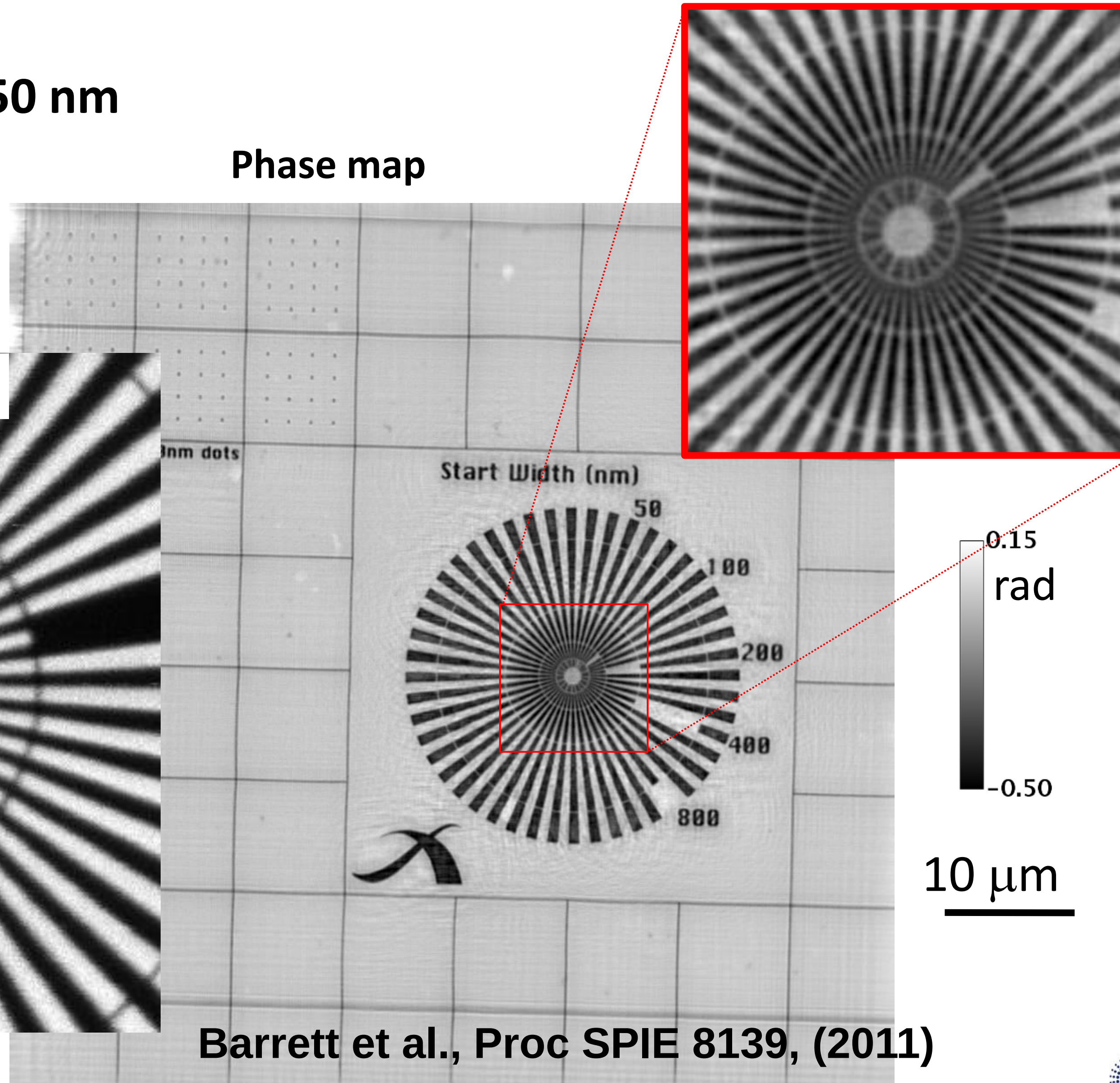
Thin gold test pattern
Innermost line width: 50 nm
Energy = 17.3 keV
Field of view: 80 µm
Pixel size: 53 nm

Au Fluorescence; 25 nm



9 µm

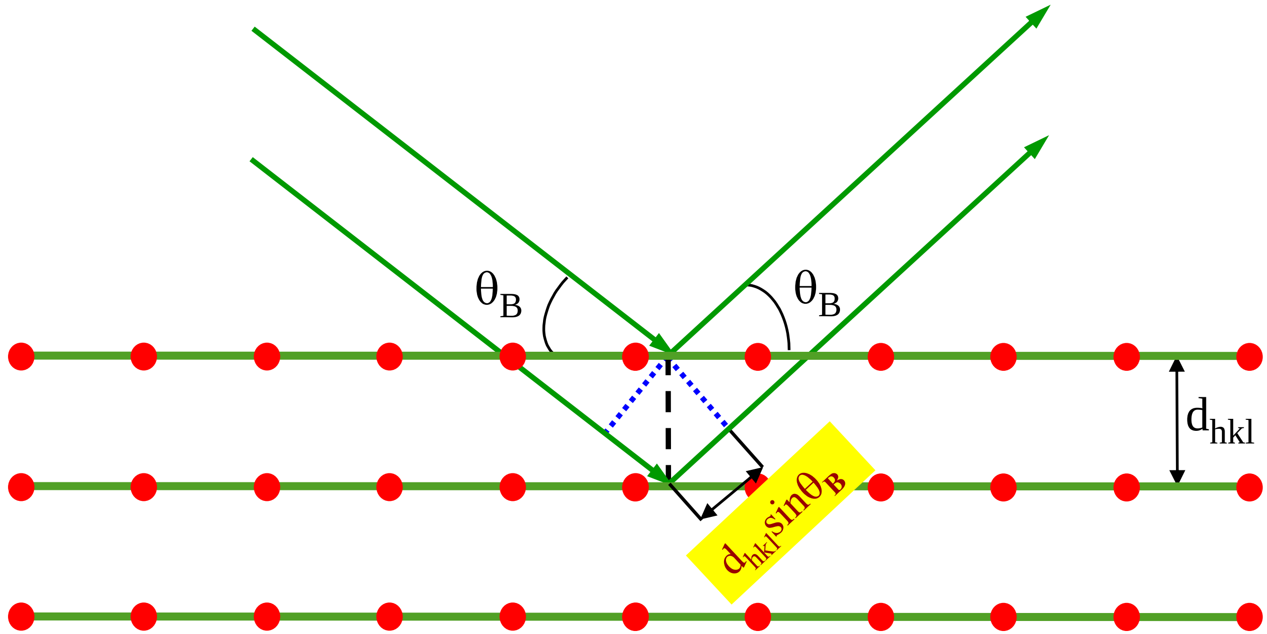
Phase map



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

X-RAY DIFFRACTION

X-ray diffraction results from elastic scattering of X-rays from structures with long-range order. For X-ray optics generally concerned with highly perfect single crystals *cf* neutron mosaic crystals

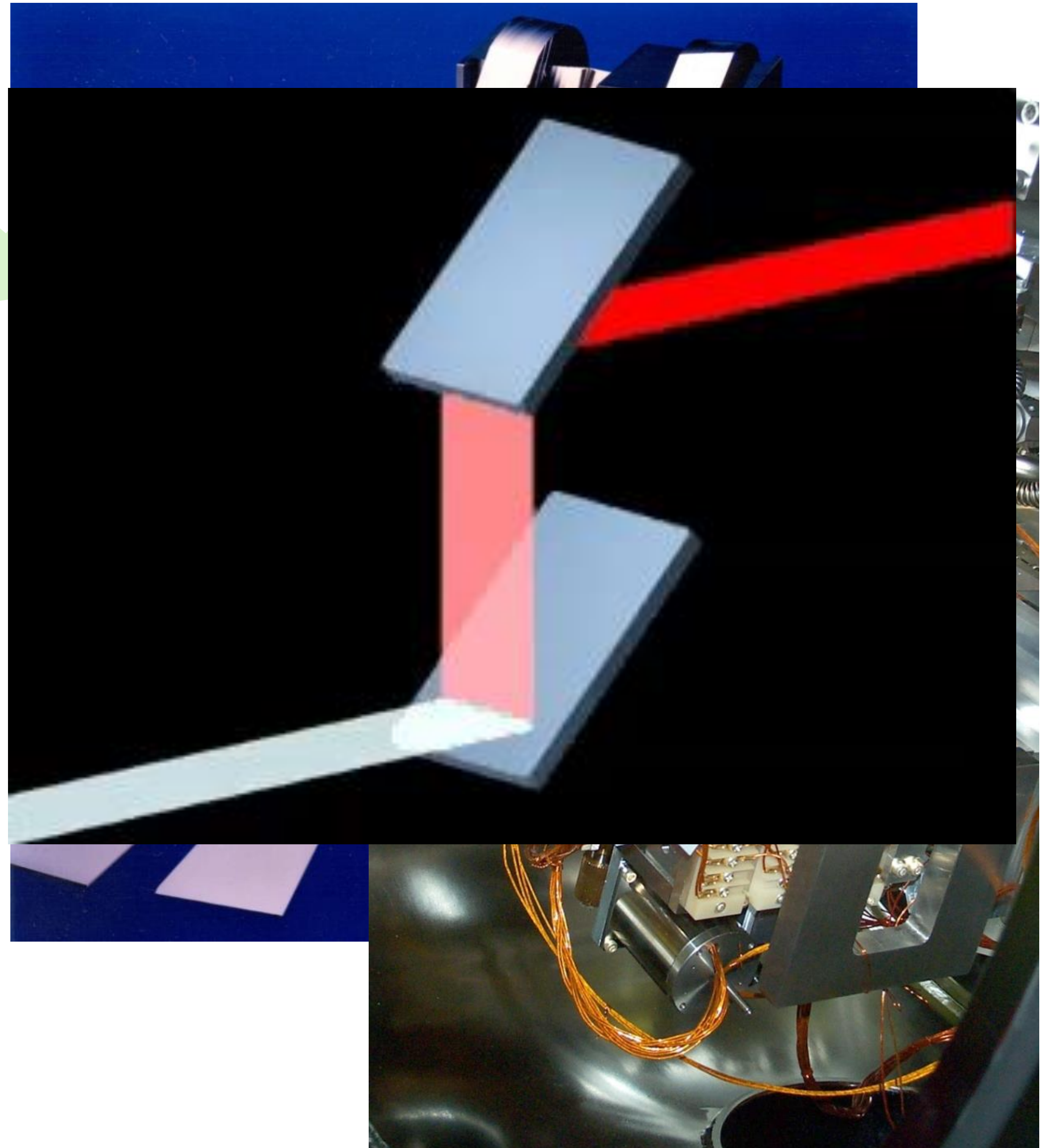


Bragg equation: $2d_{hkl}\sin\theta_B = n\lambda$

- Incident X-rays are “reflected” at atomic planes in the crystal lattice
- Path difference of the rays $2d_{hkl}\sin\theta_B$
- Constructive interference if the path difference amounts to λ ($n\lambda$?)

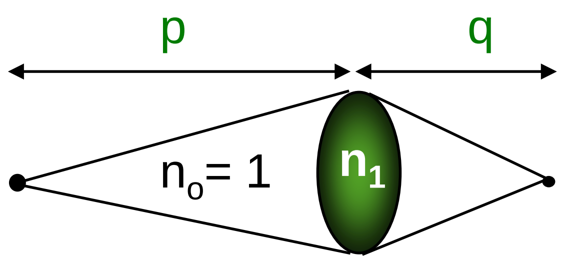
h k l are usually used, (e.g. 1 1 1, 3 3 3, 4 4 4), these are not Miller indices, but Laue indices, or “general Miller indices”.

SILICON OPTICS -MONOCHROMATORS



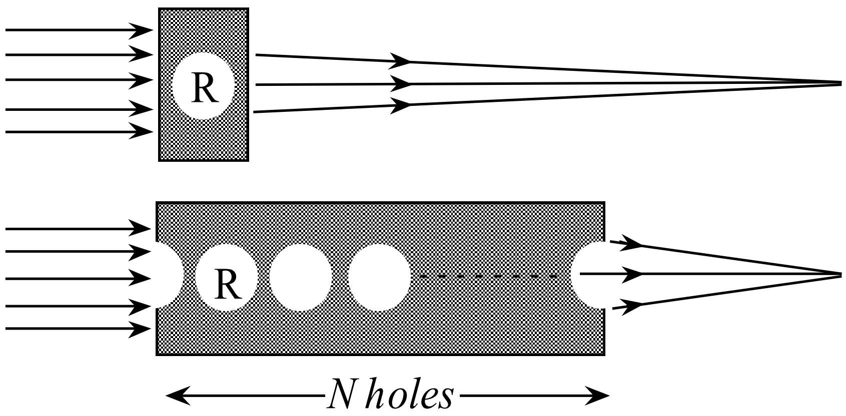
Bragg relation:
$2d\sin\theta = n\lambda$

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

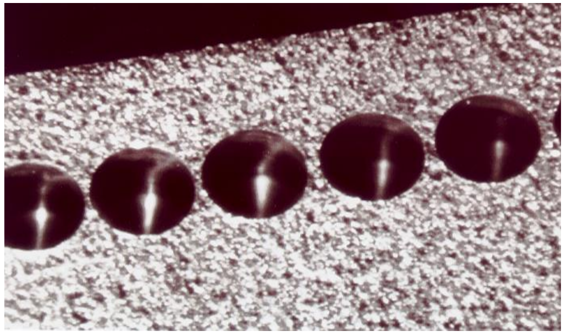
$n_1 < 1$: concave lens

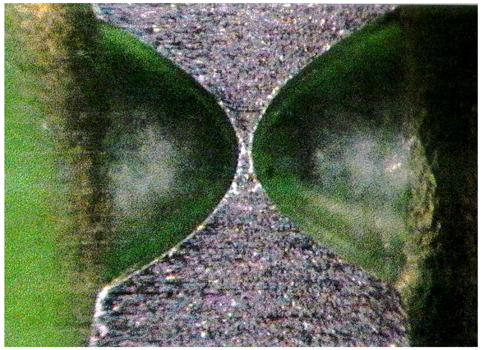
Typically Be or Al lenses – e.g.

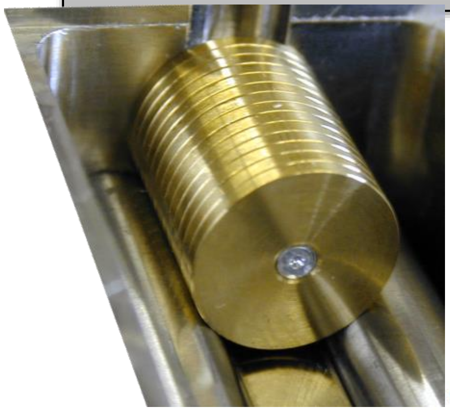
Aluminium @ 10keV $\delta = 5.5\ 10^{-6}$

1 hole 100 µm radius : f = 9 m

15 holes 100 µm radius: f = 60 cm







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

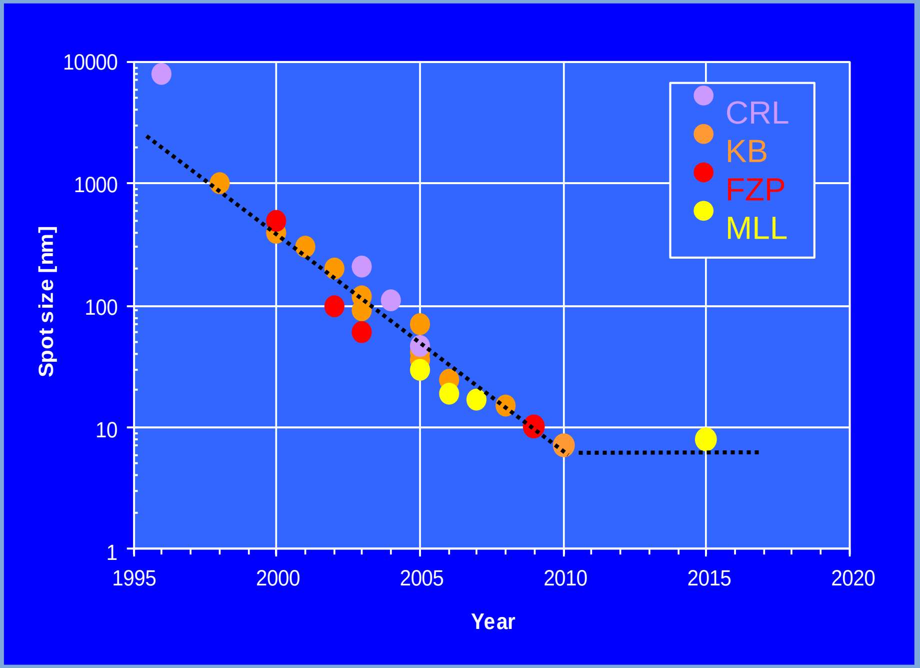
Moore's law adapted to the X-ray world:

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)*

Best focus Experiments:

- A. Morgan et al. Scientific Reports, 5, 9892 (2015)
- H. Mimura *et al. Nature Physics*, **6**, 122-125 (2010).
- J. Vila-Comamala et al., Ultramicroscopy,**109**,1360–1364 (2009)

Ultimate resolution Theory:

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

HIGHLY SPECIALIZED EXPERIMENTAL STATIONS

adapted to one or more techniques…

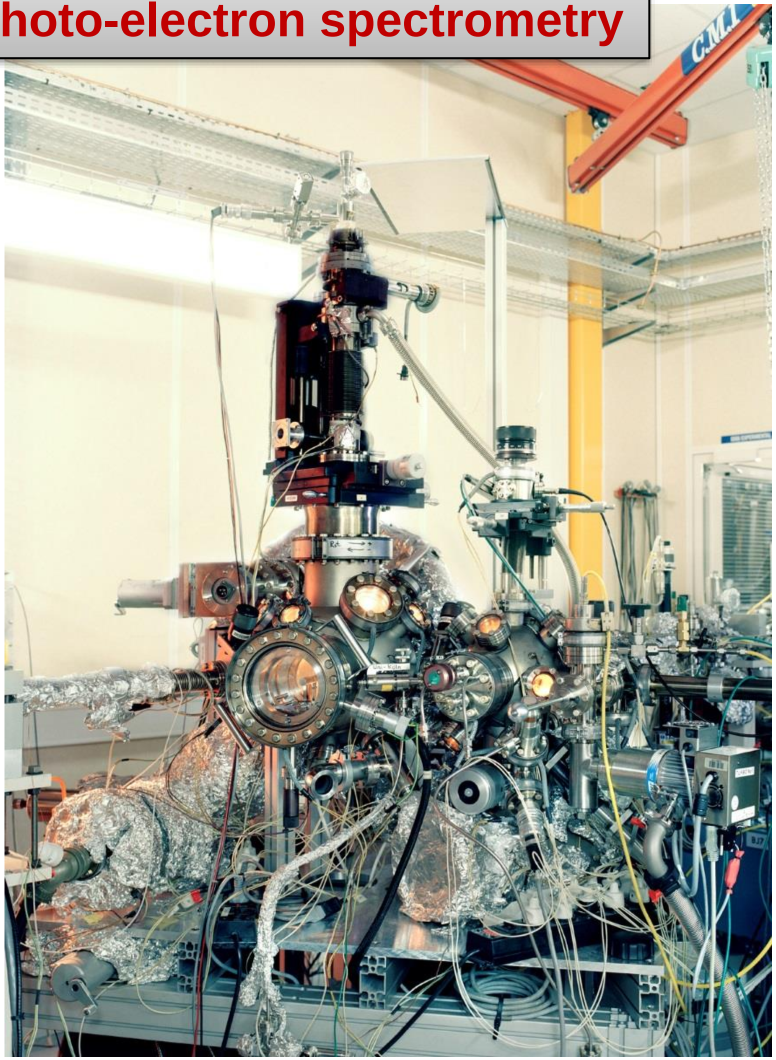
- X-ray Diffraction & Scattering
- X-ray Spectroscopy
- X-ray Fluorescence
- X-ray Imaging …

… on samples of varying types

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

… and different sample environments

ID32: experimental station - Photo-electron spectrometry

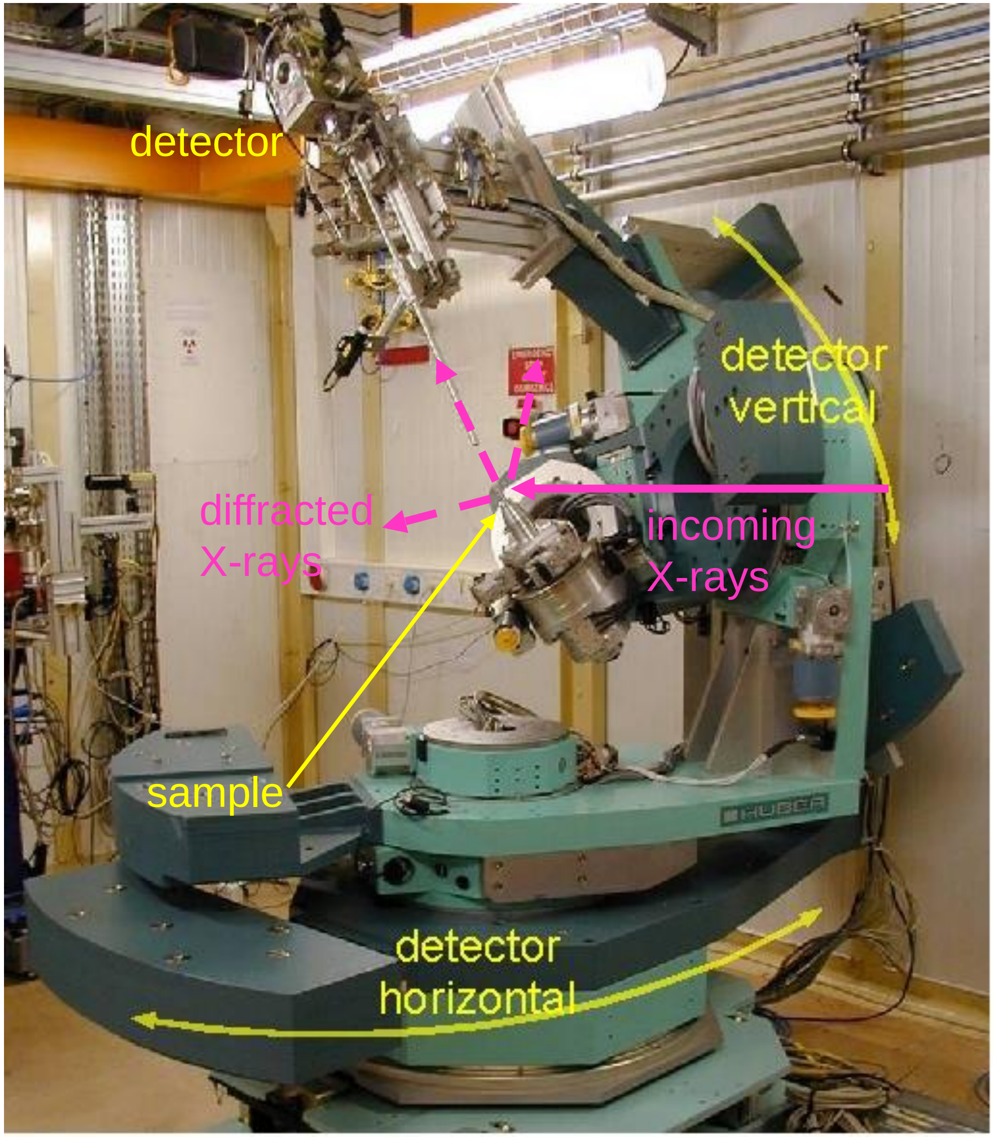


X-RAY DIFFRACTOMETER

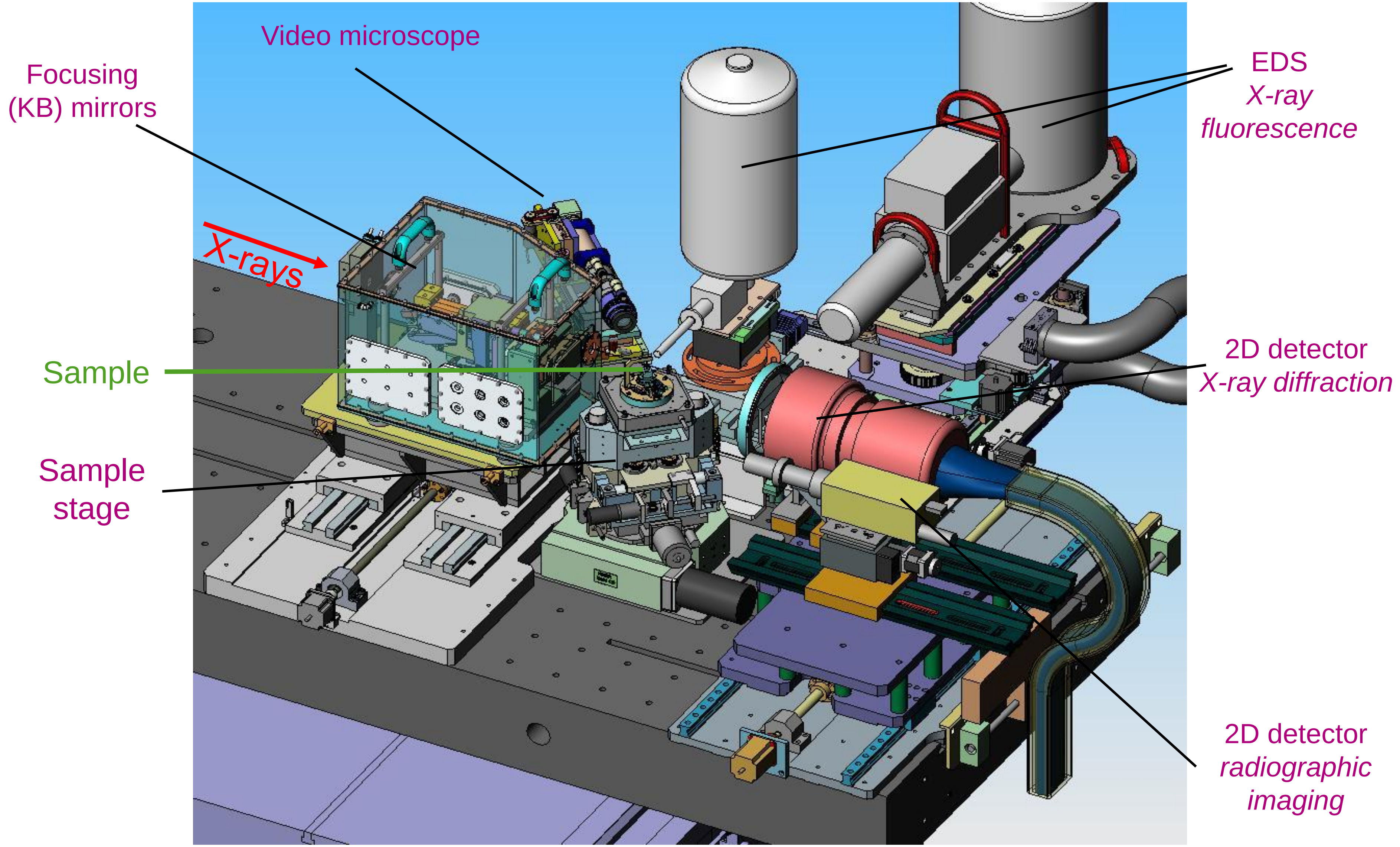
X-ray diffraction experiments require precise positioning and orientation of the sample and detector relative to the X-ray beam.

Diffractometers provide versatile sample rotation around multiple axes intersecting at single point.

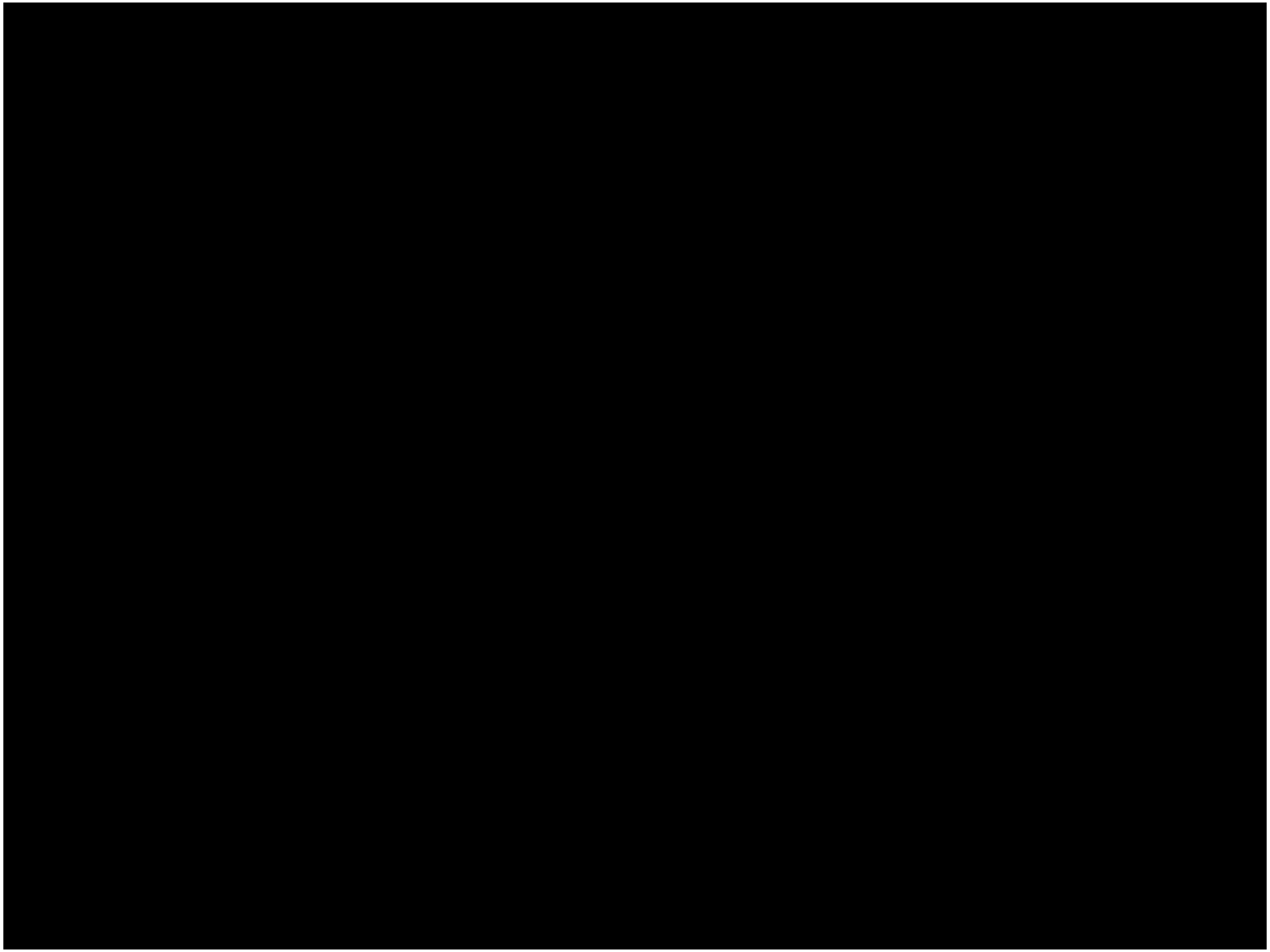
Sample remains stable within a ‘Sphere of Confusion’ typically < 100 µm to minimise drifts relative to X-ray beam



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



HIGH THROUGHPUT CRYSTALLOGRAPHY

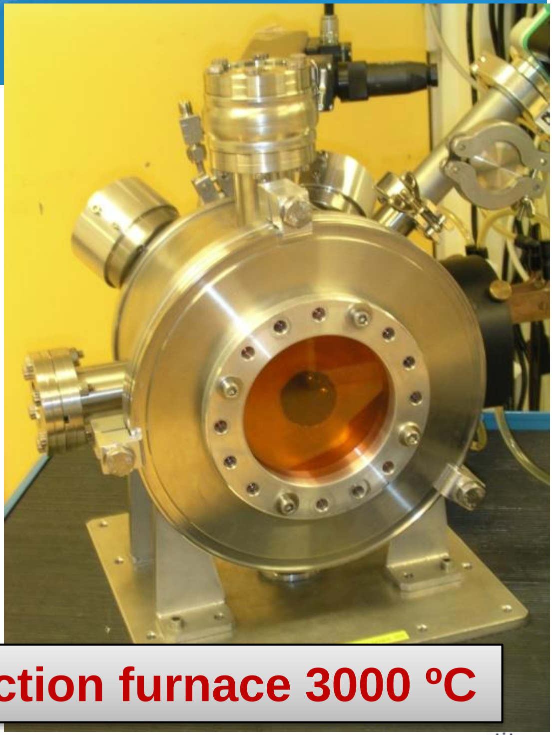


Mini flow cryostat: 2 Kelvin

Particularly for:

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

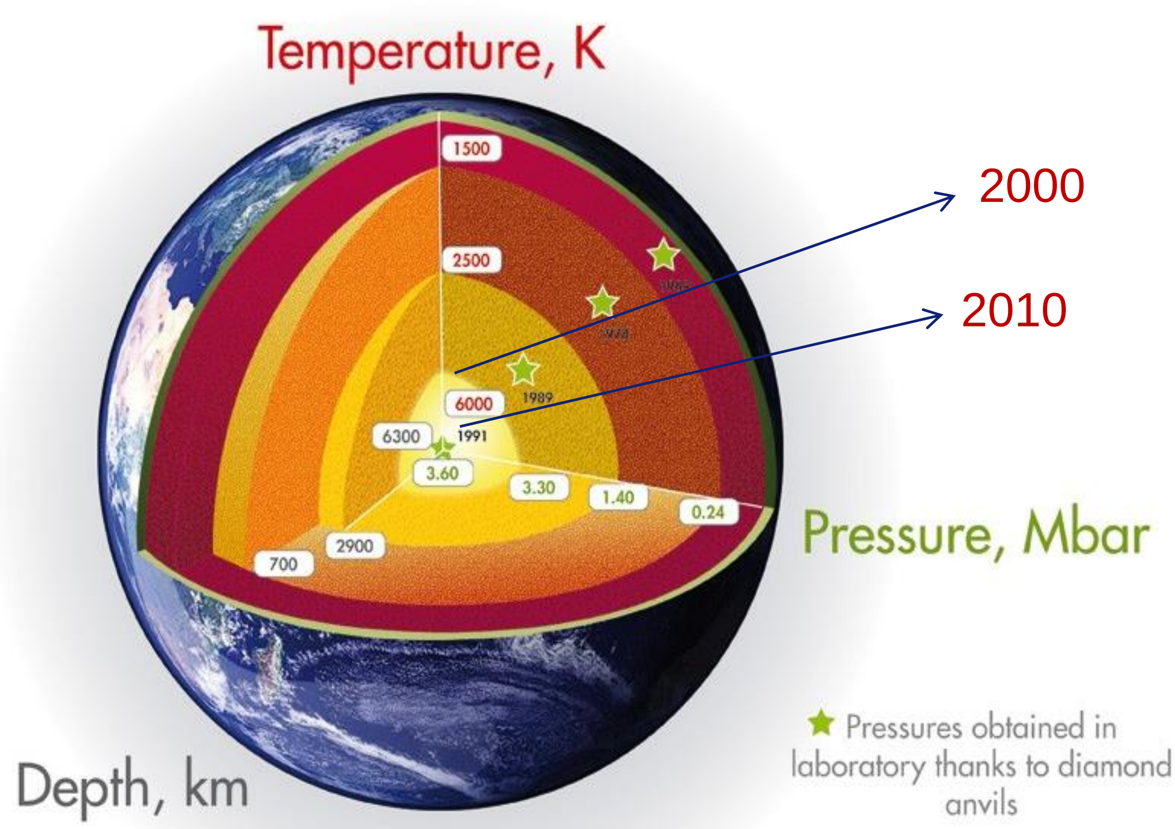
Also to limit sample damage due to photon absorption (e.g. protein crystallography experiments) …



Induction furnace 3000 ºC

Courtesy: Peter van der Linden

EXTREME CONDITIONS (T, P)



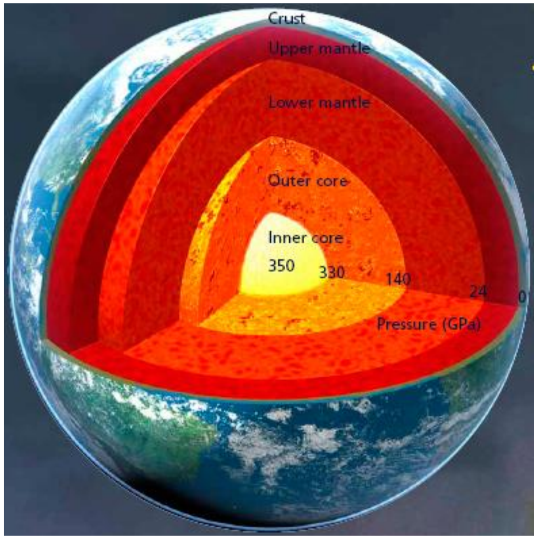
Diamond Anvil Cell (DAC)

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

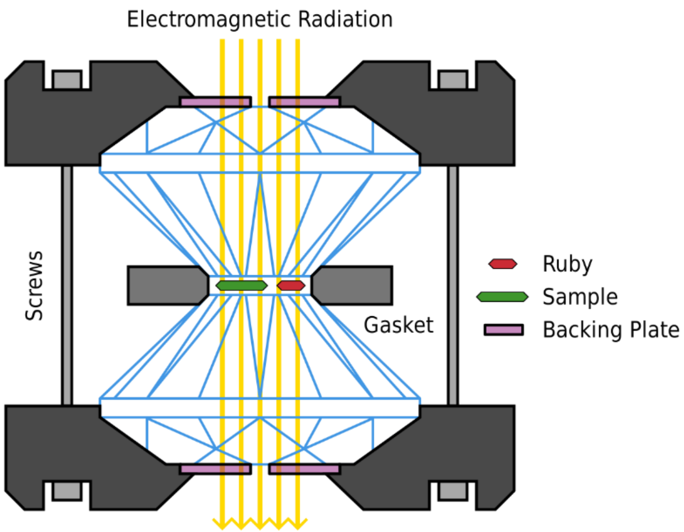
Record Pressure: 650 GPa

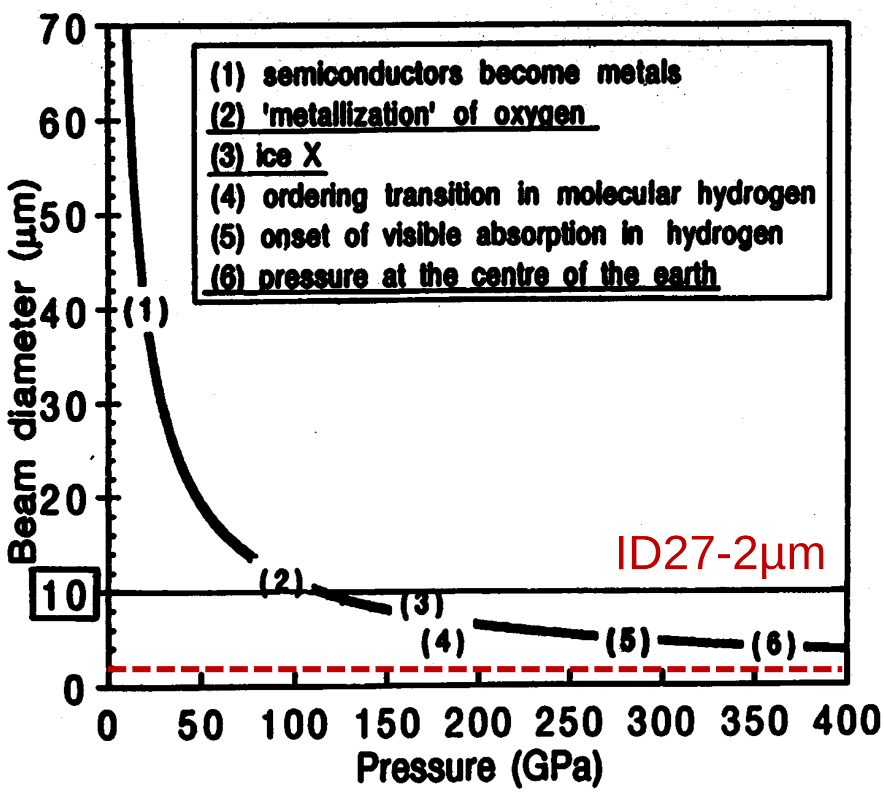
L. Dubrovinsky et al., Nature Commun. 3, 1163, 2012.

Courtesy M. Mezouar (ESRF-ID27)



Diamond Anvil Cell

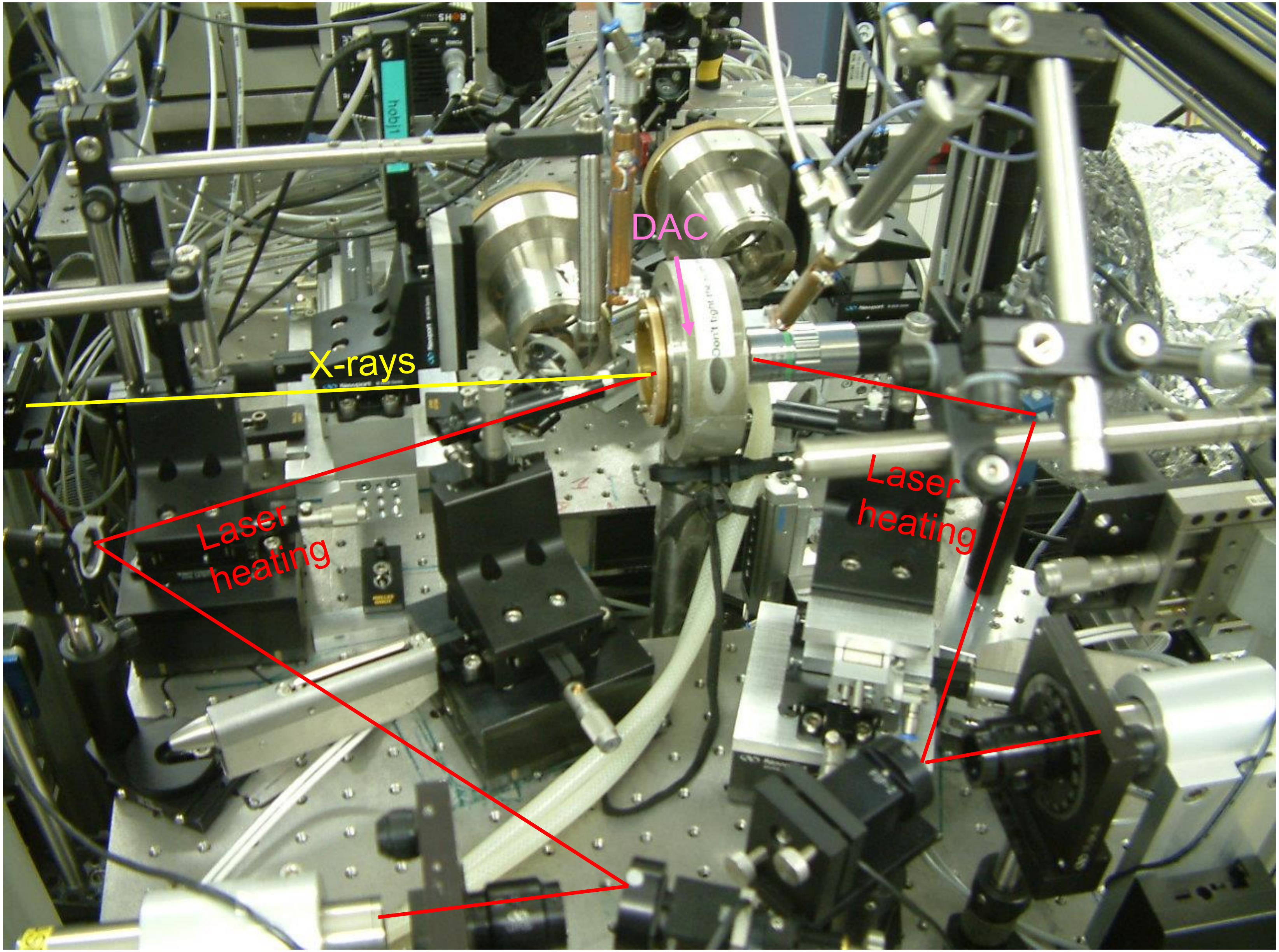




Transparent diamonds allow simultaneous laser heating of sample

Courtesy M. Mezouar (ESRF-ID27)

EXTREME CONDITIONS (T, P) – ESRF - ID27



Courtesy M. Mezouar (ESRF-ID27)

X-RAY DETECTORS

Measure X-ray intensity (sometimes energy too)

– selection depends upon various factors e.g.

- **Need for spatial distribution 0-D, 1-D, 2-D**
- **Spatial, energy resolution**
- **Efficiency (Energy), dynamic range**
- **Signal intensity: photon counting, integrating – max count rate**
- **Event time-stamping, time resolution**
- **Robustness**
- **Price (most advanced detectors can cost >1M€)**

X-RAY CONVERSION PROCESS/SENSORS

Types of conversion sensors in X-ray detectors:

semiconductors	X-ray → electron-hole pairs
scintillators	X-ray → visible light ; light sensor
photocathodes	X-ray → photoelectrons
gas	X-ray → ions
microbolometers	X-ray → phonons ; precision thermometer (TES)
superconductors	X-ray → charged quasiparticles

The complete conversion process: X-ray → e-

In some cases: amplification in the conversion process:
high electric field → charge multiplication

Earliest detector: Film – still used occasionally
Direct detection

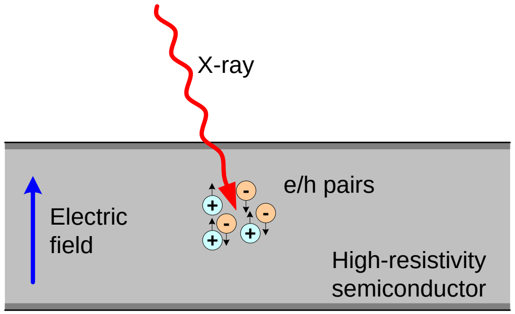
- Absorbed X-rays directly generate electrical signal e.g. photodiodes, pixel-detectors, silicon drift-diodes

Indirect detection

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

SEMICONDUCTOR (DIRECT DETECTION)

Generation of electron-hole pairs e.g. Silicon PIN diode

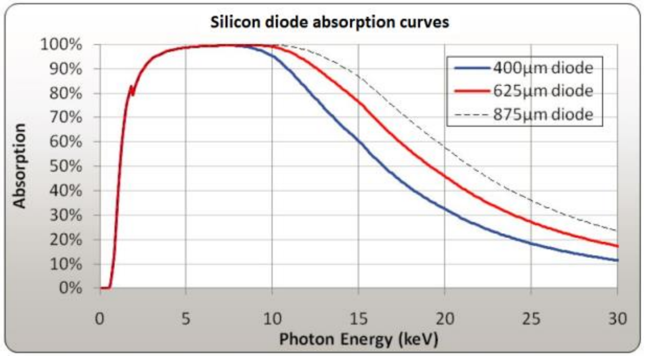


Efficient charge collection:

High electric field (depleted volume)
High resistivity semiconductor
Minimise dark current (cooling)

X-ray photon energy range:

lower limit: due 'entrance window' cut-off
higher limit: sensor transmission



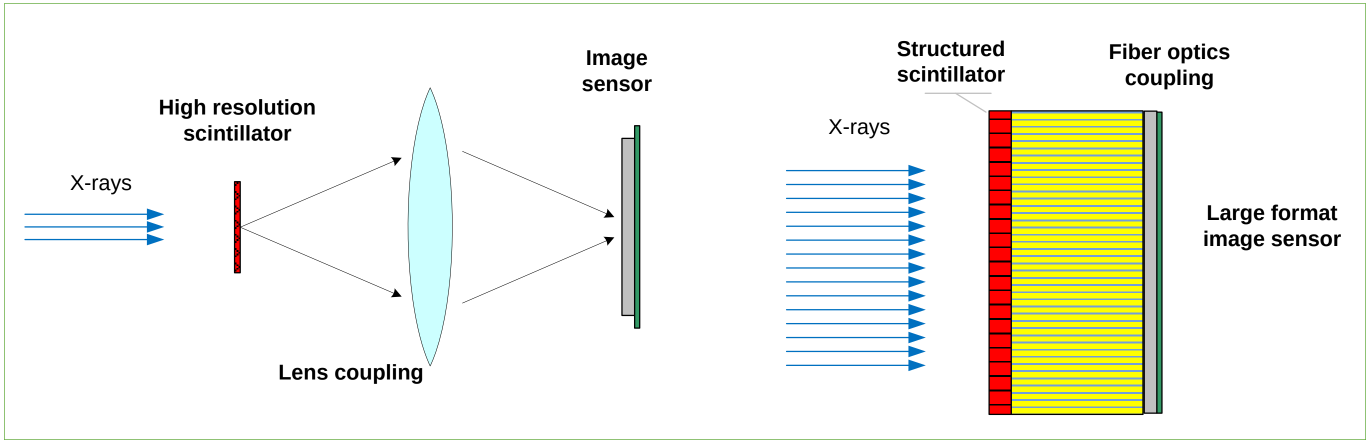
Silicon is the reference material, but:

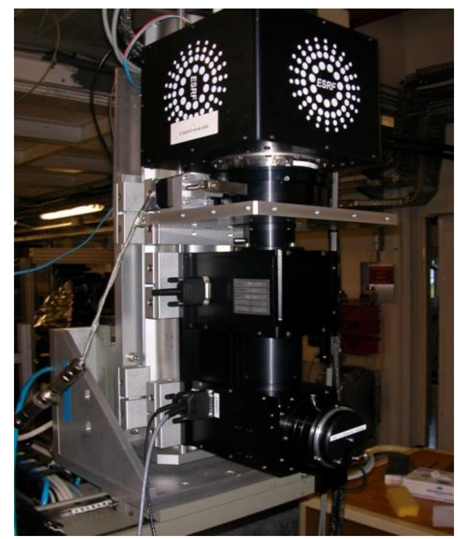
limited energy range up to 15-20 keV
devices have moderate radiation hardness

That is why other semiconductors are used (Ge, GaAs, CdTe, …)

Courtesy: Pablo Fajardo

SCINTILLATORS (INDIRECT DETECTION)





Main key points:

- Scintillators can be radiation hard (not always the optics)
- High spatial resolution is possible (Optical magnification)
- Efficient for high energy photons.

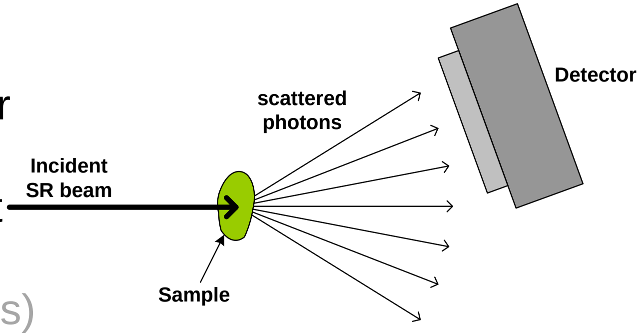
Courtesy: Pablo Fajardo

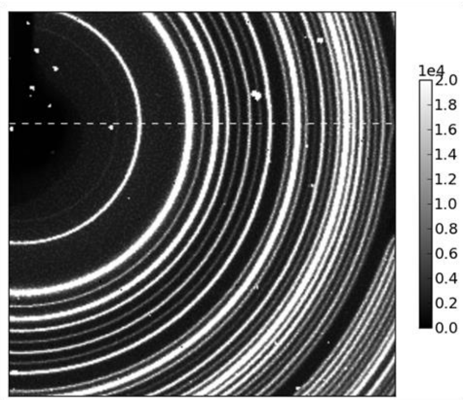
E.G. ELASTIC SCATTERING: DIFFRACTION, SMALL ANGLE SCATTERING…

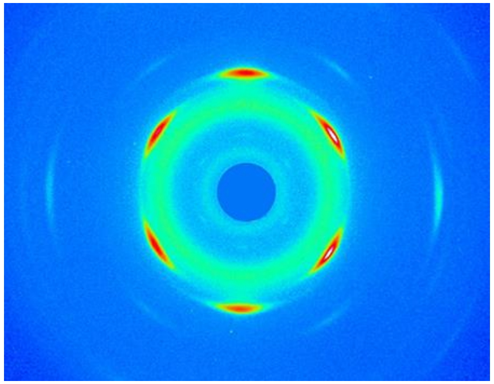
- Scattered photons *conserve their initial energy*, i.e. only momentum changed
  angular measurements are required, usually measured with 2D spatially resolving detectors
  $\Rightarrow$angular resolution can be varied by changing detector-sample distance
- Large dynamic range may be required (crystal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

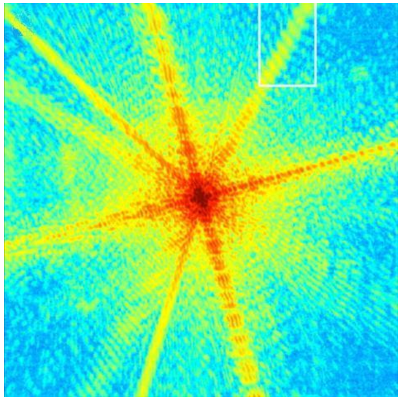
- detectors used:
  - scintillator-PMT; silicon-diode, -APD using diffractometer
  - solid state semiconductors (1D strip, 2D area PADs)
  - 'indirect detection' with flat panel a-Si or CMOS readout
    2D area scintillator-optic-CCD cameras

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





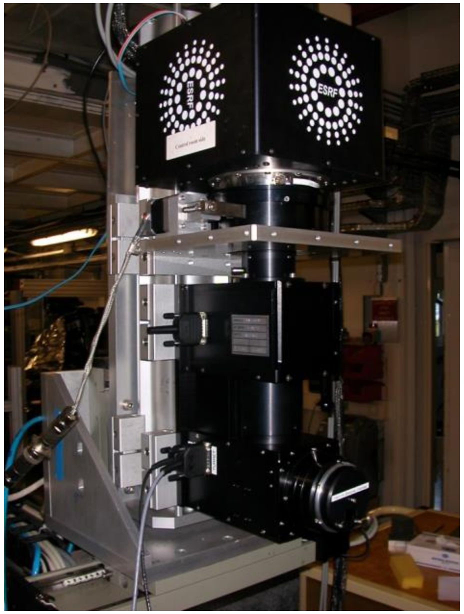


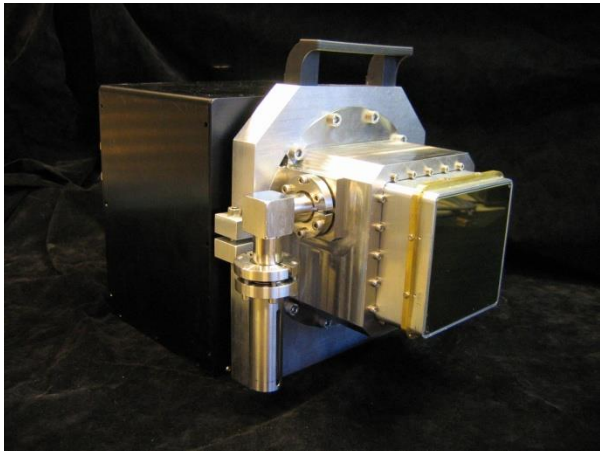


Courtesy: John Morse

2D X-RAY DETECTORS

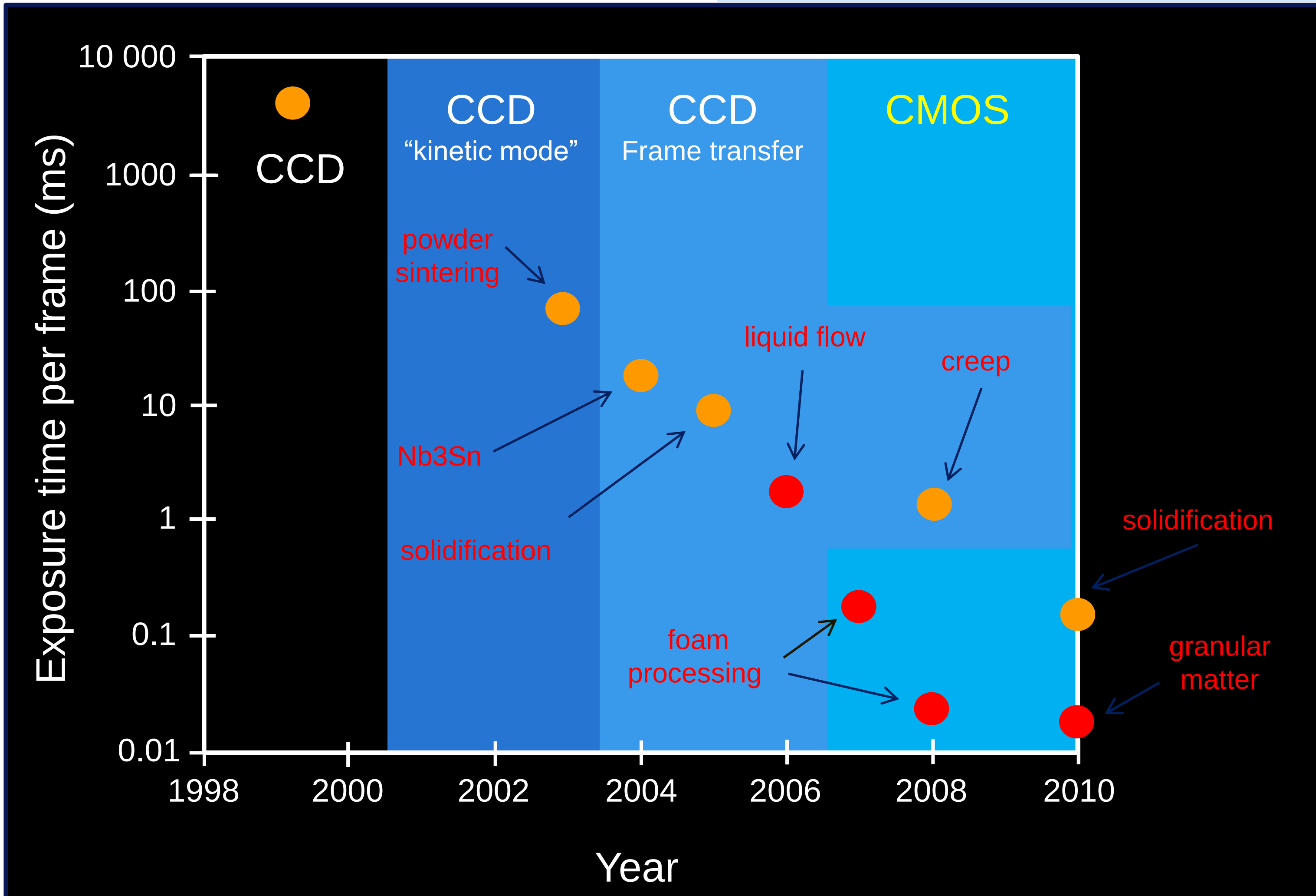
Roentgen: “... Photographic plates and film are ”susceptible to x-rays”, providing a valuable means of recording the effects...”





CHRONOLOGICAL EVOLUTION OF FAST IMAGING (ESRF)

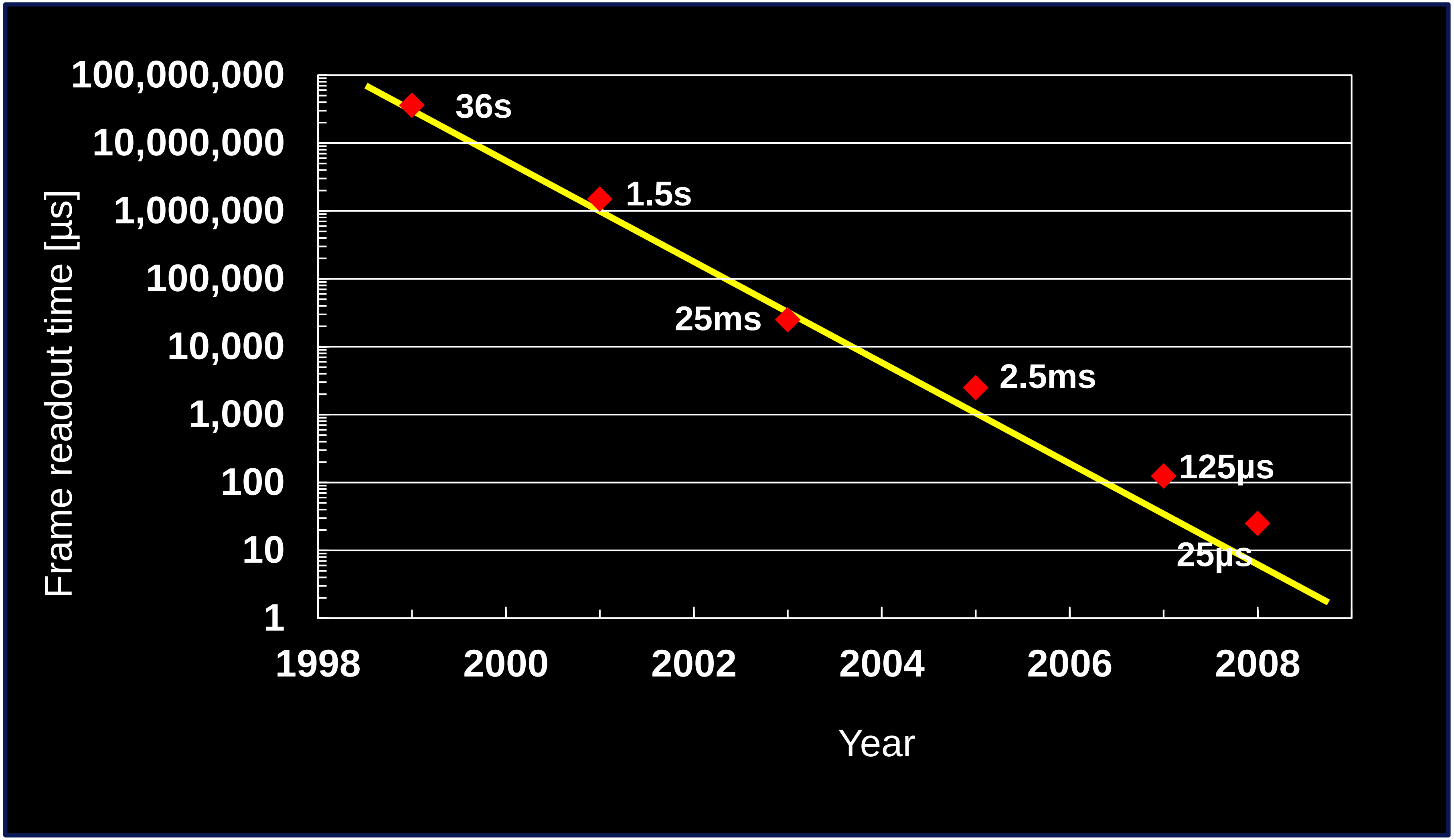
- Orange points: spatial resolution 1-2µm
- Red points: spatial resolution 10-20µm



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 EBS Upgrade includes an ambitious instrumentation development program addressing many of these issues

QUESTIONS?