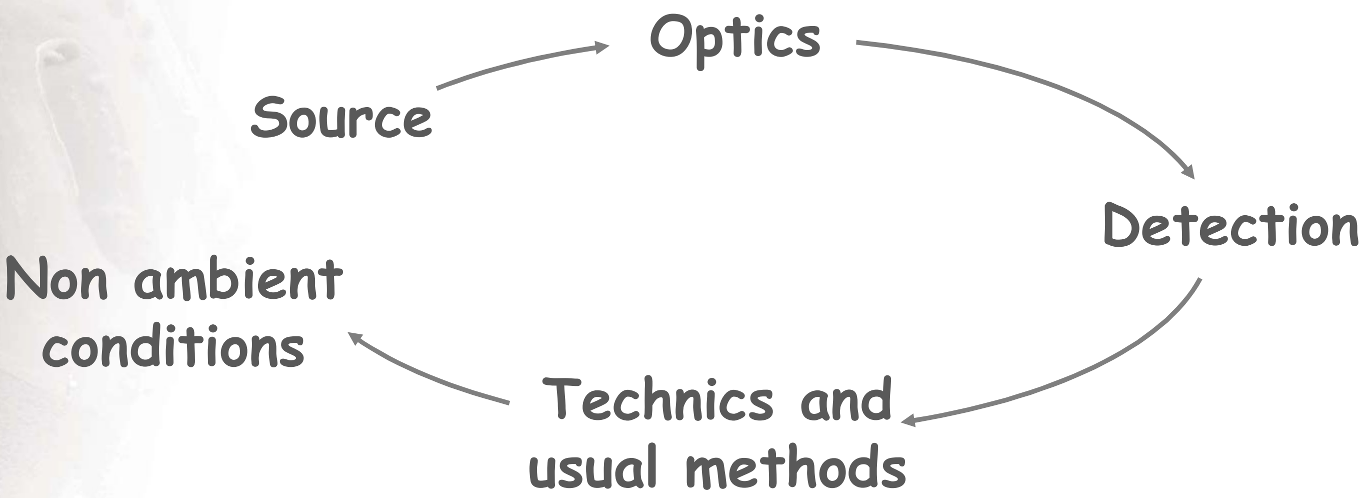
SR Instrumentation

Pierre Fertey

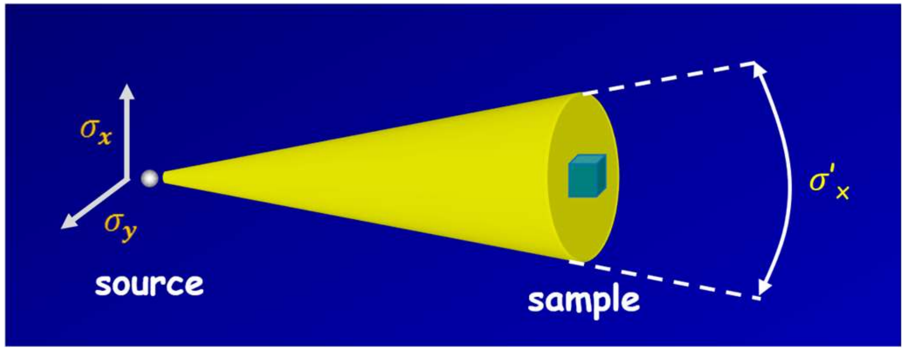
How to perform a (SR) x-ray diffraction experiment?



Large Facilities X-ray sources: synchrotrons

		Scale	Cost	Access
Lab. source		0.3 m		
Synchrotron		100 m		
X-FEL		1000 m		

How to compare X-ray sources?



- Intensity
- Energy (keV) or Wavelength (Å)
- Divergence s'_x, s'_y (mrad) *(1 mrad ~ 0.06°)*
- Source size s_x, s_y (mm)
- Homogeneity
- Shape

Brightness:

Photon flux based on the source divergence per $\Delta E/E$ = 0.1% bandwidth

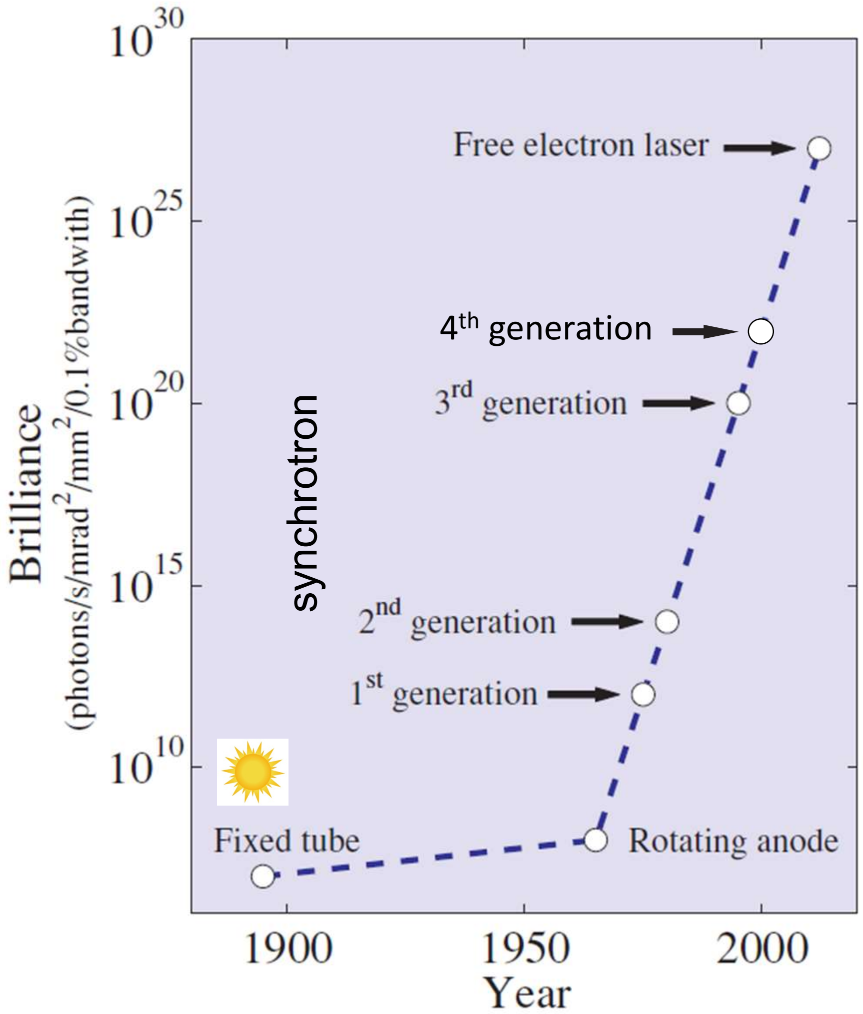
ph/s/mrad²/0.1%BW

Brillance:

Photon flux based on the source size and divergence per $\Delta E/E$ = 0.1% bandwidth

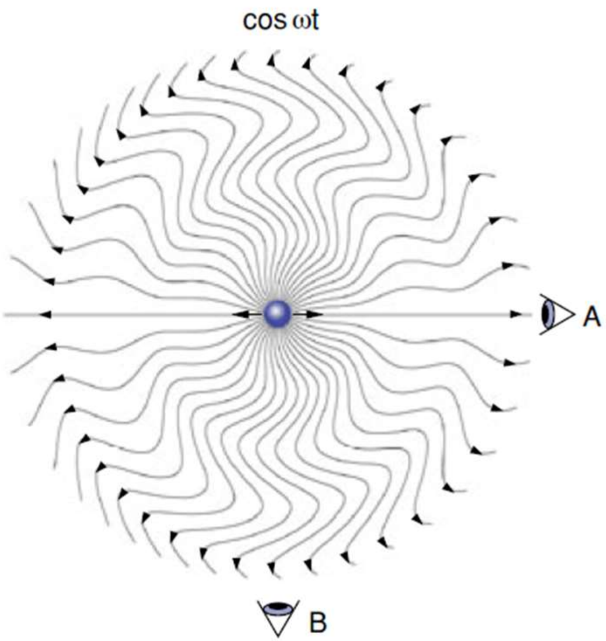
ph/s/mrad²/mm²/0.1%BW

Average Brillance



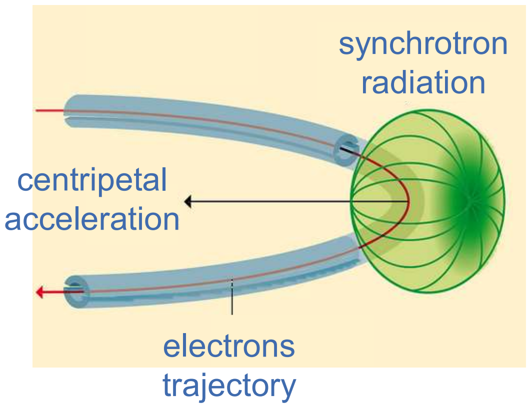
Synchrotron Radiation

Principle: generation of electromagnetic radiation through the acceleration of a charged particle



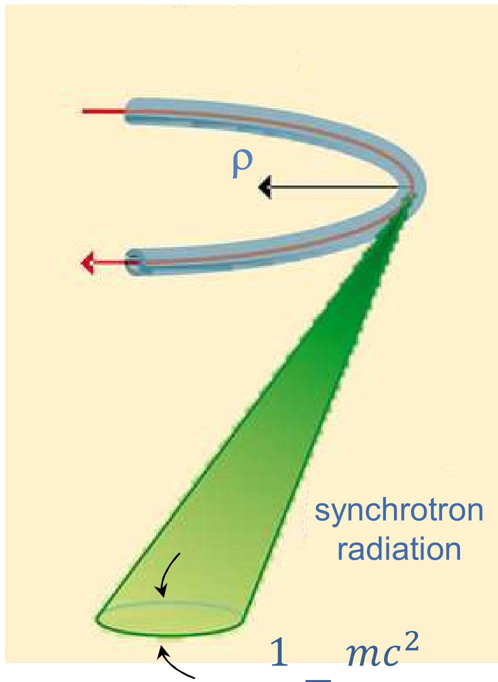
non relativistic electrons

$\beta = v/c << 1$



relativistic electrons

$\beta = v/c \sim 1 \quad \gamma = \frac{1}{\sqrt{1-\beta^2}}$



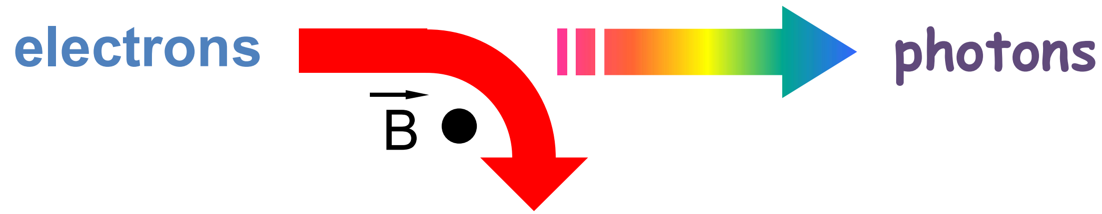
$$\sim \frac{1}{\gamma} = \frac{mc^2}{\mathbb{E}}$$

$$P_{ray} \sim \frac{\mathbb{E}^4}{m^4 \rho^2}$$

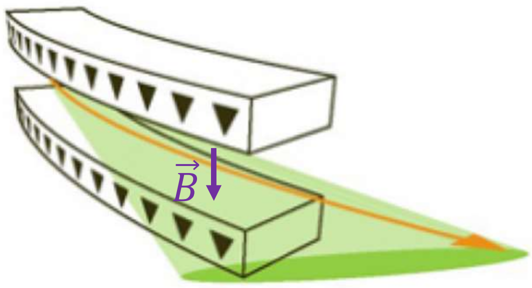
➡ e^- or e^+

➡ accelerate e^- for light radiation: curved trajectory
relativistic regim (v ~ c) : collimation

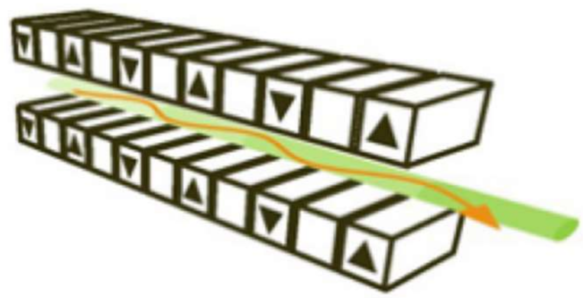
Synchrotron Radiation



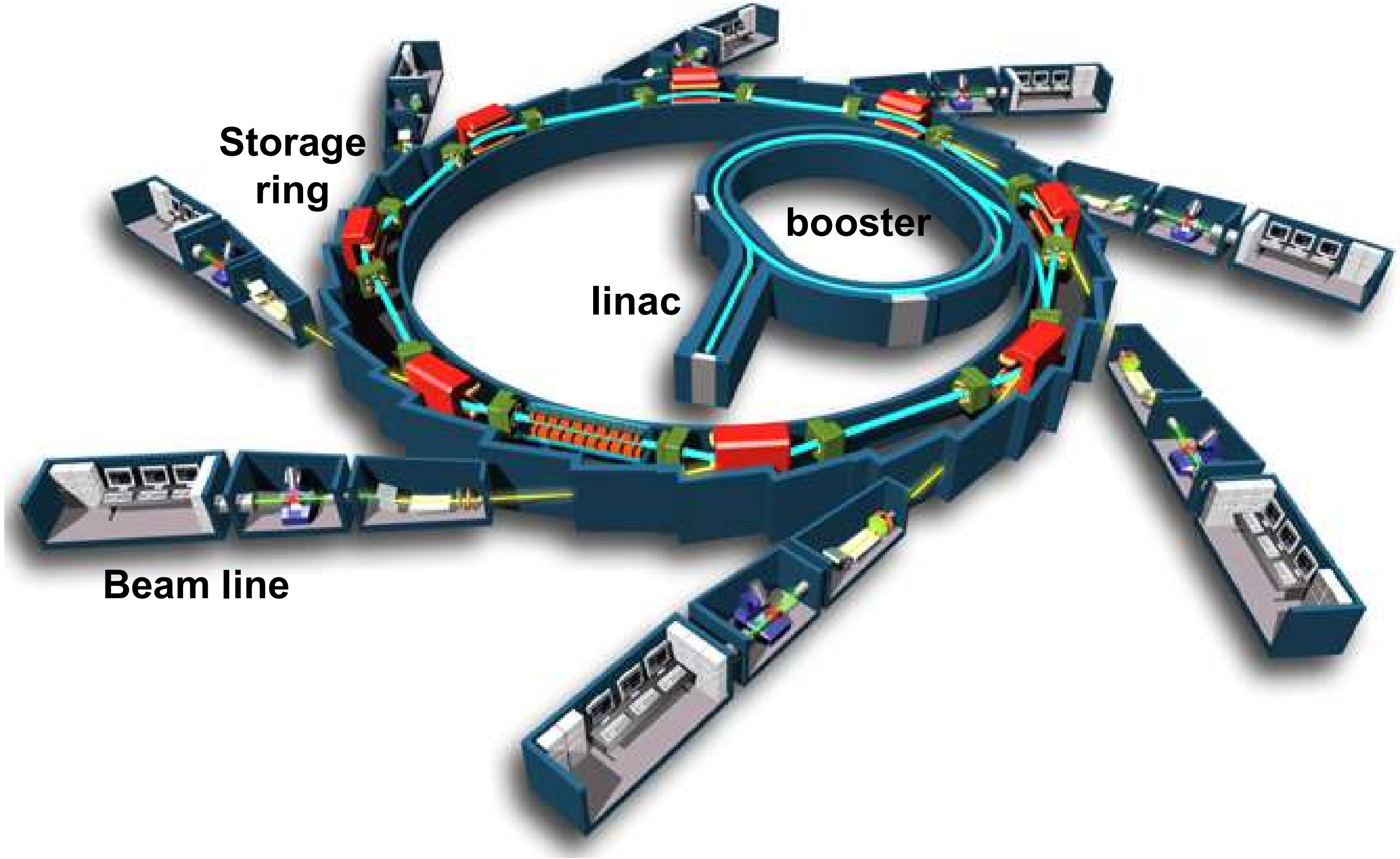
Bending magnet (dipole source)



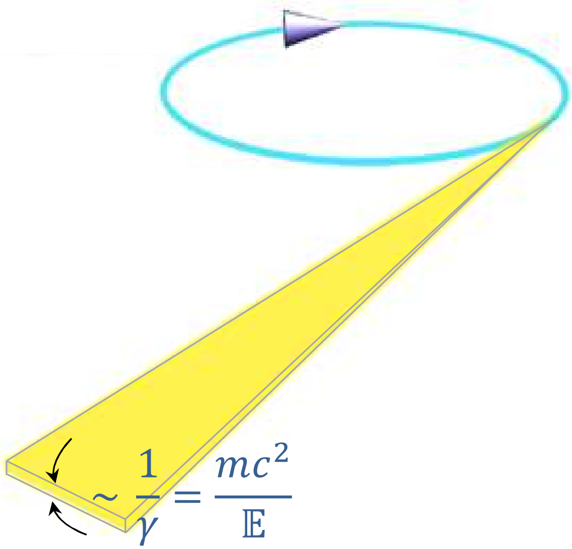
Undulator/Wiggler (insertion device)



Synchrotron Radiation: the Machine



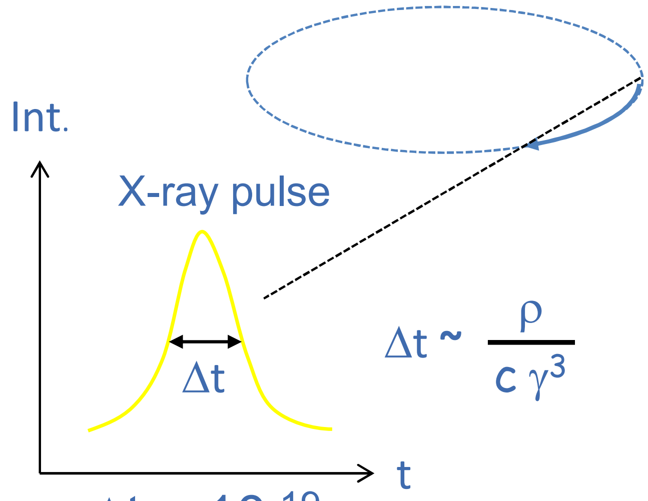
Synchrotron Radiation



relativistic electrons

$\beta = v/c \sim 1$

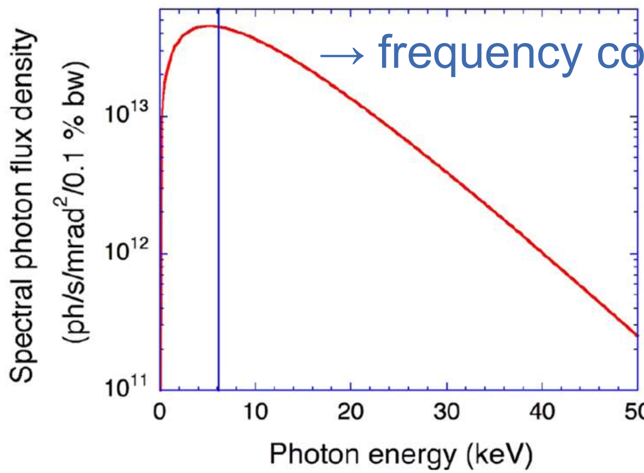
$\gamma = \frac{1}{\sqrt{1-\beta^2}}$



$\Delta t \sim 10^{-19}$ s

	Vert. Div.
Lab Source	'5' mrad
Bending Magnet	0.2 mrad
Insertion Device	0.01 mrad

(1 mrad ~ 0.06°)

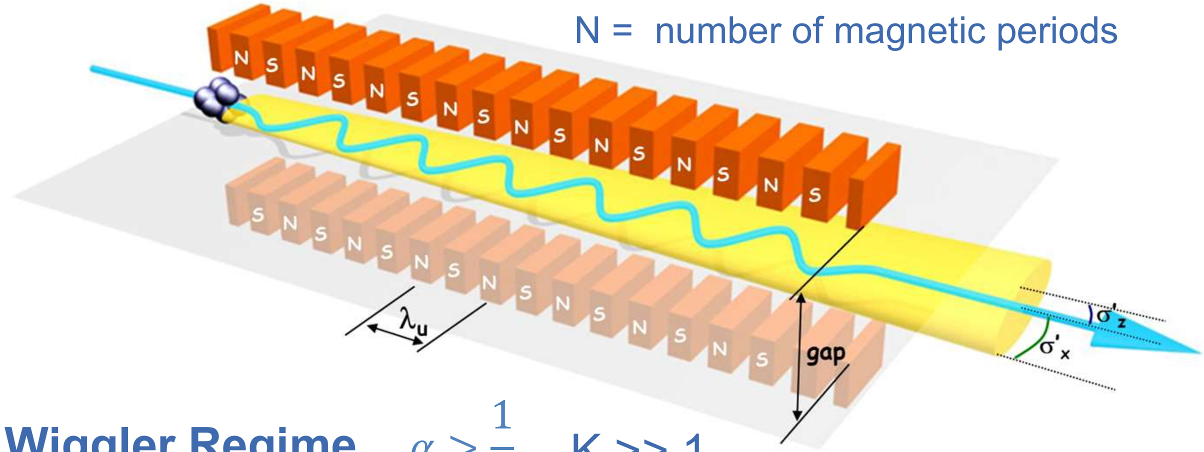


→ frequency components up to 1/Δt

white beam

Insertion device: Wiggler and Undulator

N = number of magnetic periods



amplitude of oscillations

↔ K ~ λ_u [cm] B_0 [T]

↔ gap

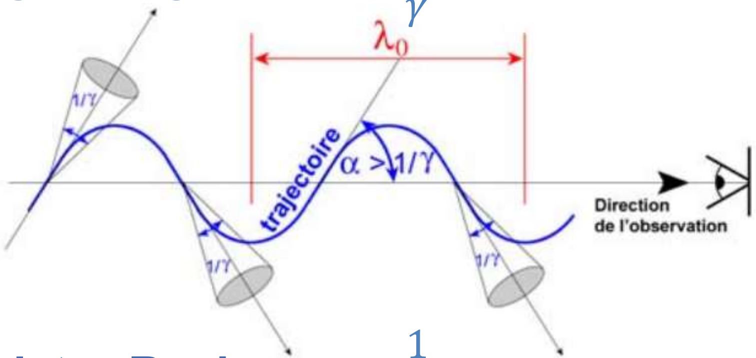
↔ $\alpha = \frac{K}{\gamma}$

$\gamma = \frac{1}{\sqrt{1-\beta^2}}$

E_{Soleil}= 2.75GeV

γ(Soleil) ~ 5400

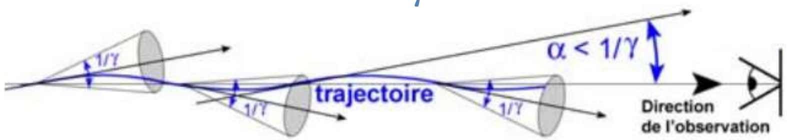
Wiggler Regime $\alpha > \frac{1}{\gamma}$ K >> 1



In the wiggler regime, the observer sees a train of distinct light pulses, each of them similar to that observable from a bending magnet with the same magnetic field: **the pulses add incoherently**

I ~ 2N I(wiggle)

Undulator Regime $\alpha < \frac{1}{\gamma}$ K ~ 1

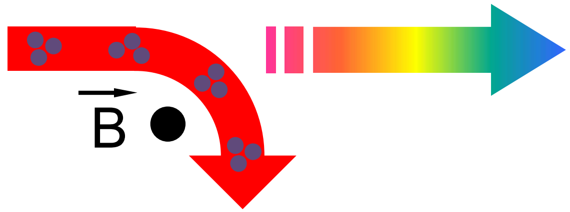


In the undulator regime the angle and the transverse displacement of the electron are so small that the observer can see the electron during the full length of the ID and therefore a much longer time interval → much thinner spectrum around privileged photon energies = undulator harmonics: **the pulses add coherently**

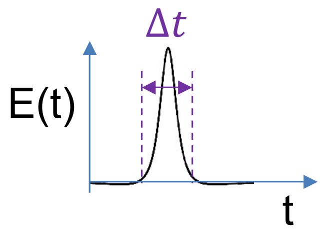
I ~ N^2 I(wiggle)

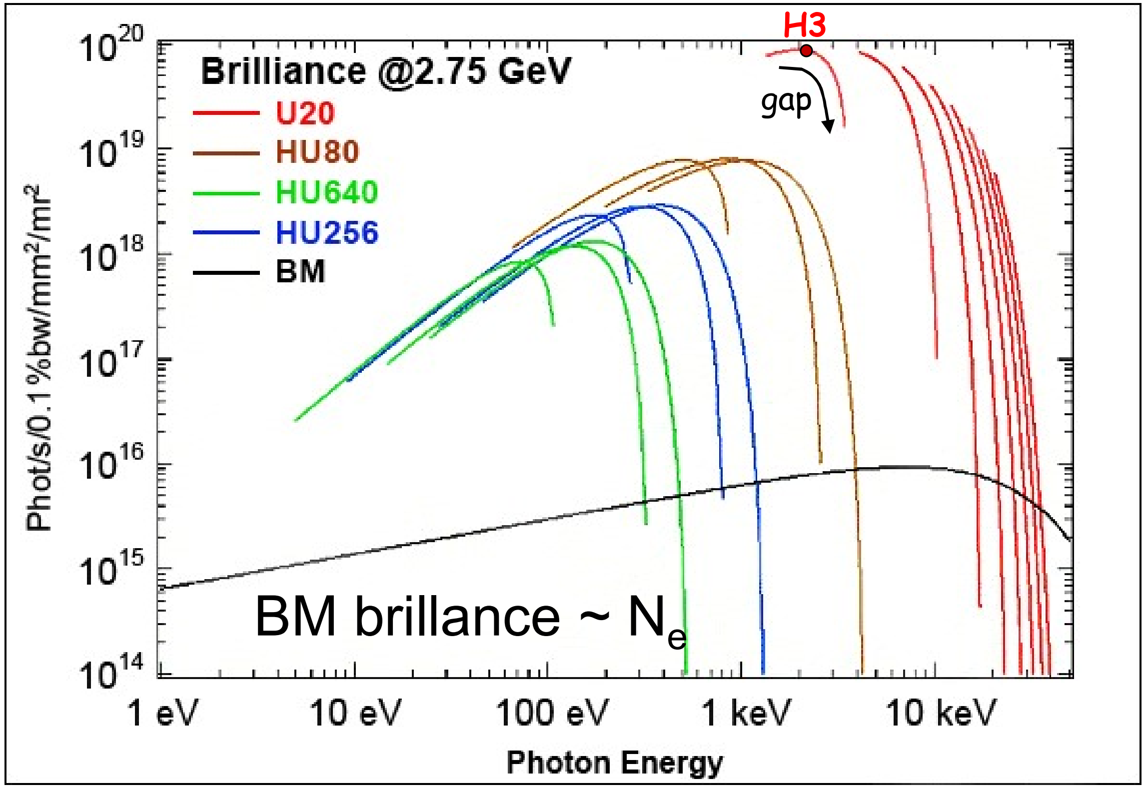
Synchrotron Radiation

bunches of electrons

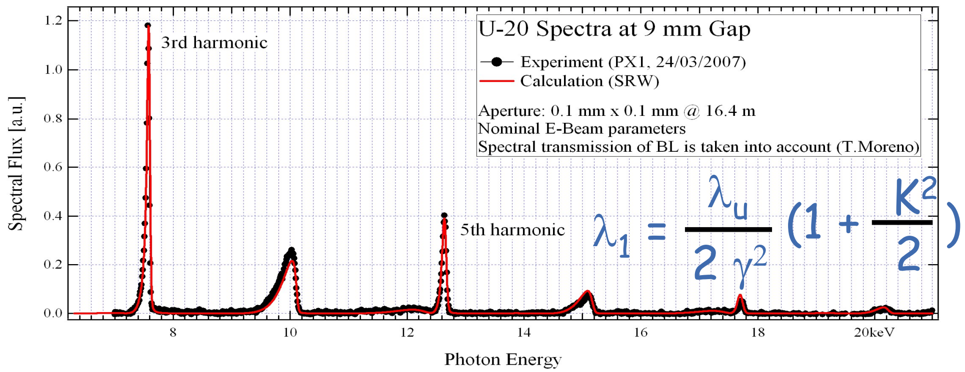


pulsed light



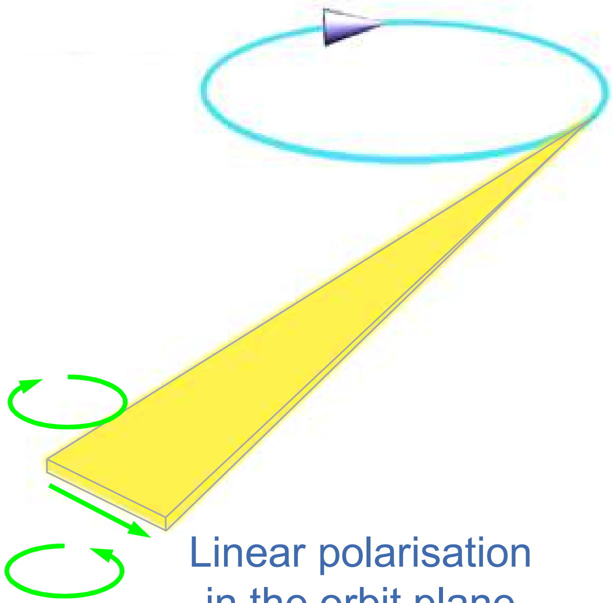


Undulator: brillance $\propto N_e N_p^2$



Wiggler: brillance $\propto N_e N_p$

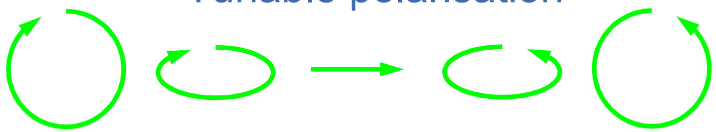
Polarized beam



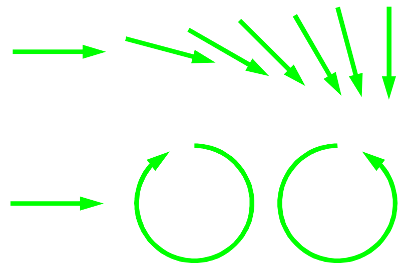
Linear polarisation
in the orbit plane

Undulator with Bz + Bx componentrs

Tunable polarisation



Undulator + « phase plate »



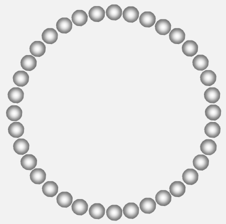
⇨ Magnetic diffraction

ex. magnetic domains,
magnetic structures

Pulsed beam

Storage ring filling modes: (e.g. SOLEIL):

Uniform: 416 bunches



I_{ring} = 416*1.2 mA = 500 mA

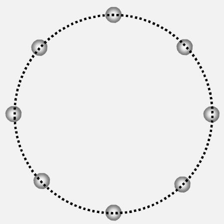
1 pulse (~ 40 ps) every ~ 3 ns

Hybrid: 312 + 1 bunches

I_{ring} = 312*1.42*1*5 = 450 mA

1 pulse ~ 60 ps every ~ 1.2 µs

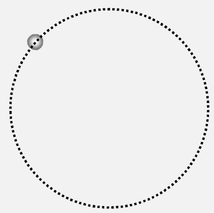
8 bunches



I_{ring} = 8*12.5mA = 100 mA

~ 90 ps every ~ 150 ns

1 bunch



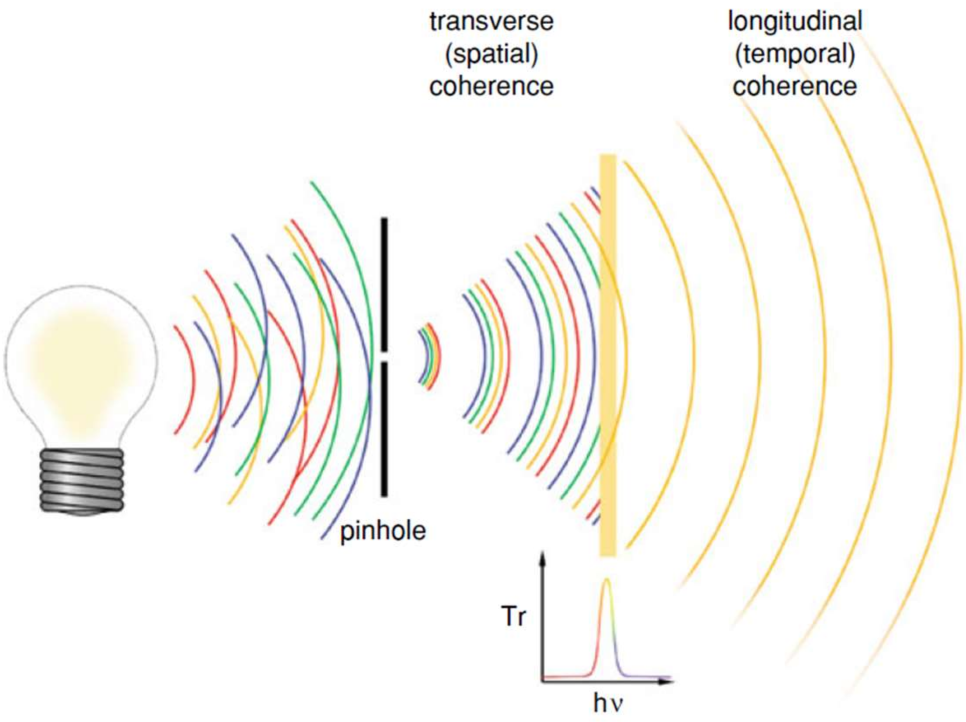
I_{ring} = 1*20 mA

~ 100 ps ~ 1.2 µs

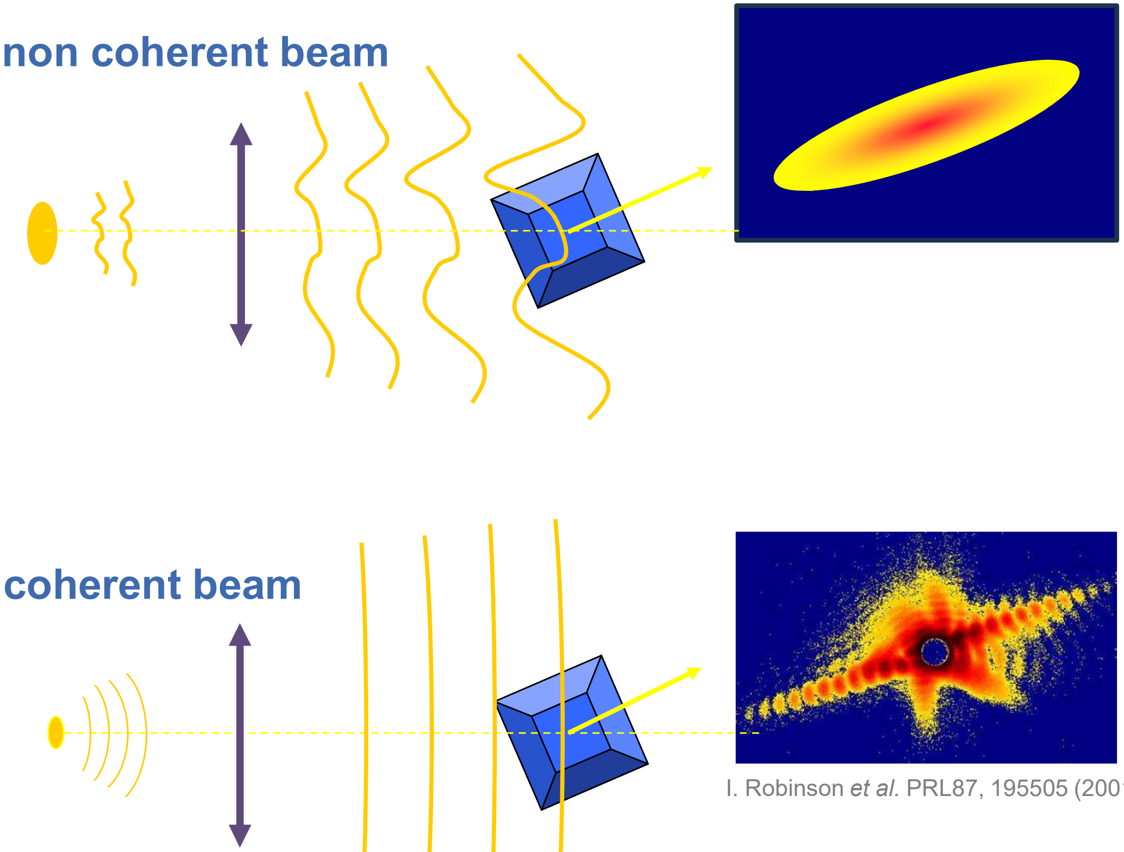
⇨ **Time resolved experiments**

cf. lecture of C. Laulhé on Friday

Coherent beam



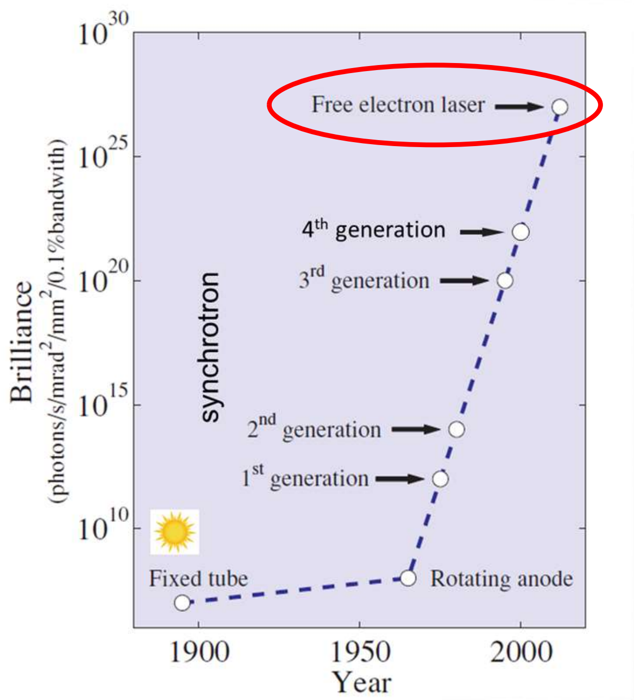
(cf. P Willmott in Springer Proceedings in Physics 262)



I. Robinson *et al.* PRL87, 195505 (2001)

⇨ **Coherent Diffraction Imaging**

cf. lecture of V. Jacques on Friday
+ talk V. Chamard on Wednesday evening

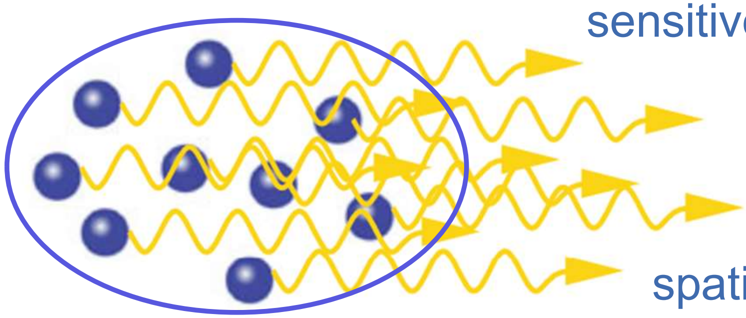


Large Facilities X-ray sources: X-FELs

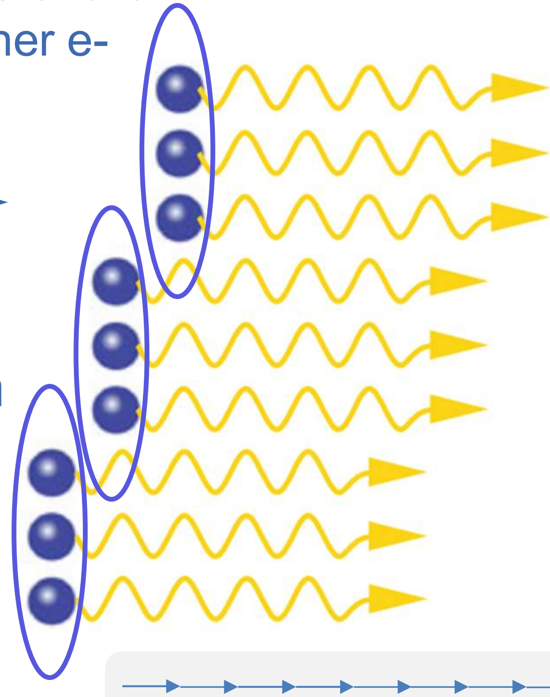
X-ray Free Electron Laser

after a certain distance in an undulator, the e- are sensitive to the electric field created by other e-

(e.g. SASE1@European XFEL: 175 m)

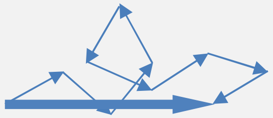


spatial modulation of the e- distribution



Self **A**mplified **S**timulated **E**mission

(free e- laser)

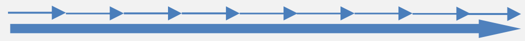


$\sqrt{n_e}$

incoherent bunch

$I \propto n_e$

- **about 10^{12} ph/pulse**
- **short x-ray pulses (a few fs)**
- **coherent beam**



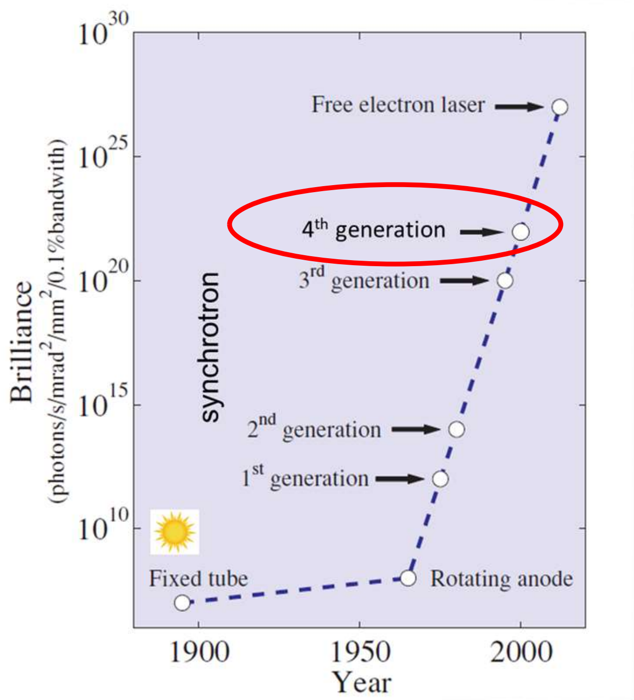
n_e

coherent bunch

$I \propto n_e^2$

⇨ **Ultra fast time resolved experiments**

cf. lecture of C. Laulhé on Friday

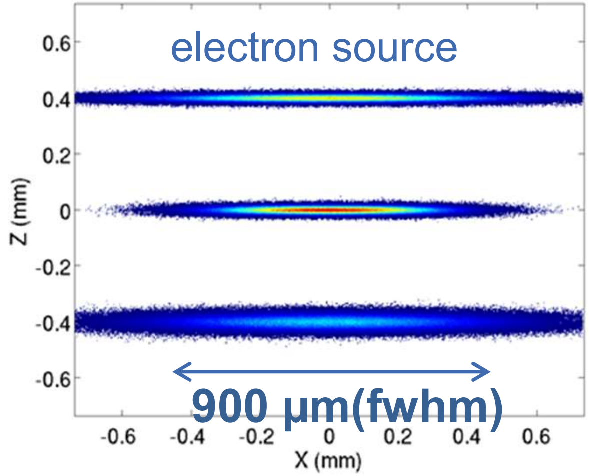


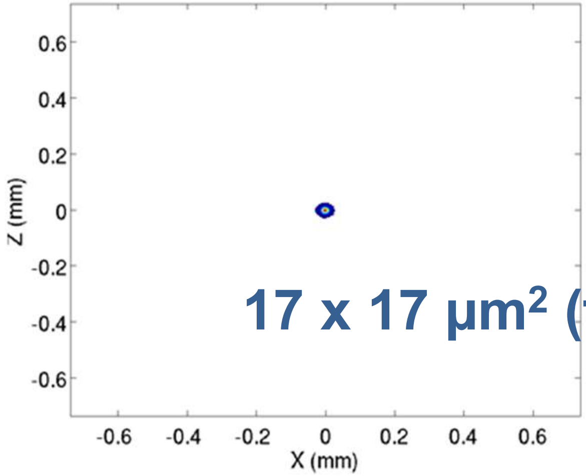
4th generation of synchrotron rings

4th generation of synchrotron rings

MAX IV (Sweden)
SIRIUS (Brazil)
ESRF-EBS (Grenoble)

SOLEIL SYNCHROTRON → SOLEIL II SYNCHROTRON





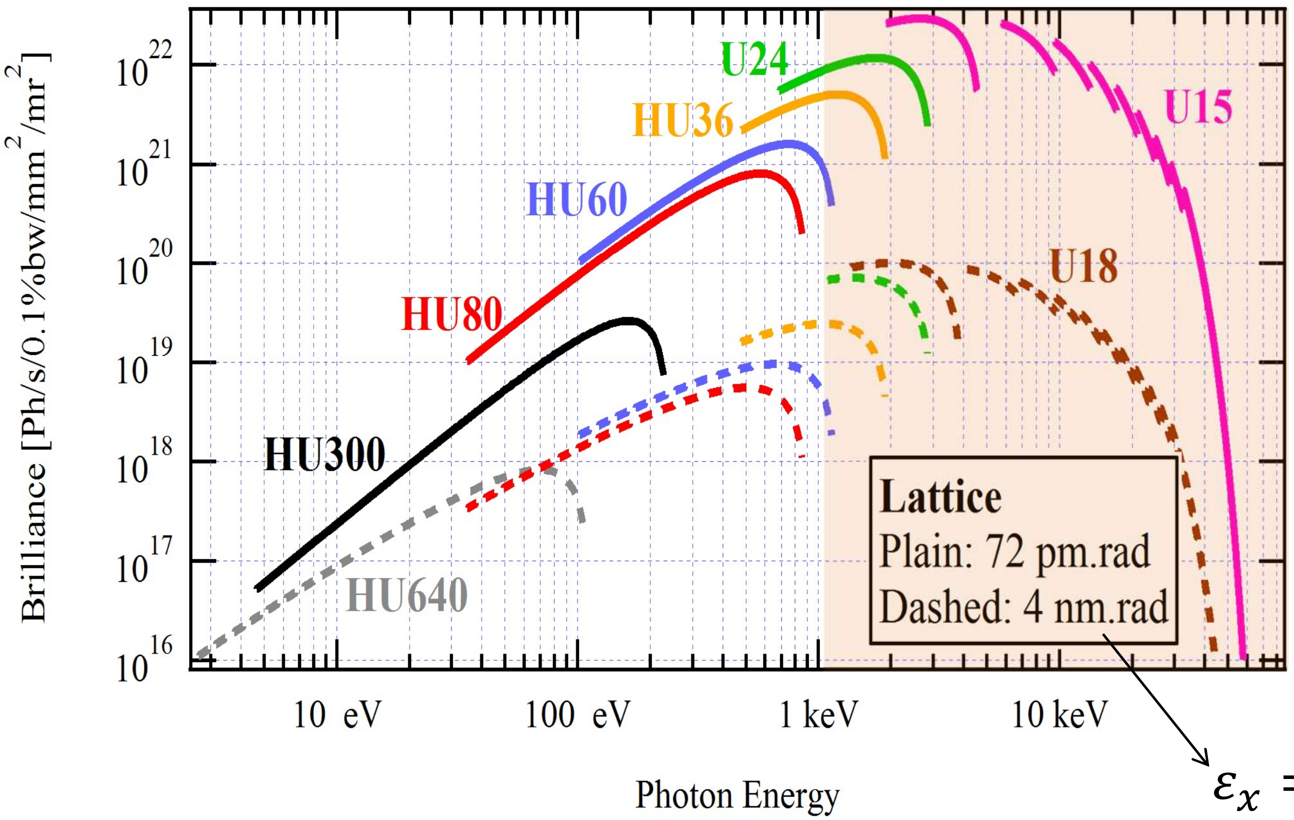
17 x 17 µm^2 (fwhm)

- « round » beam
- brillance
- coherence

$$B = \frac{F[ph/s/(0.1\%\Delta E/E)]}{\sigma_x \sigma_z \sigma'_x \sigma'_z}$$

size *divergence*

Brillance @ SOLEIL II

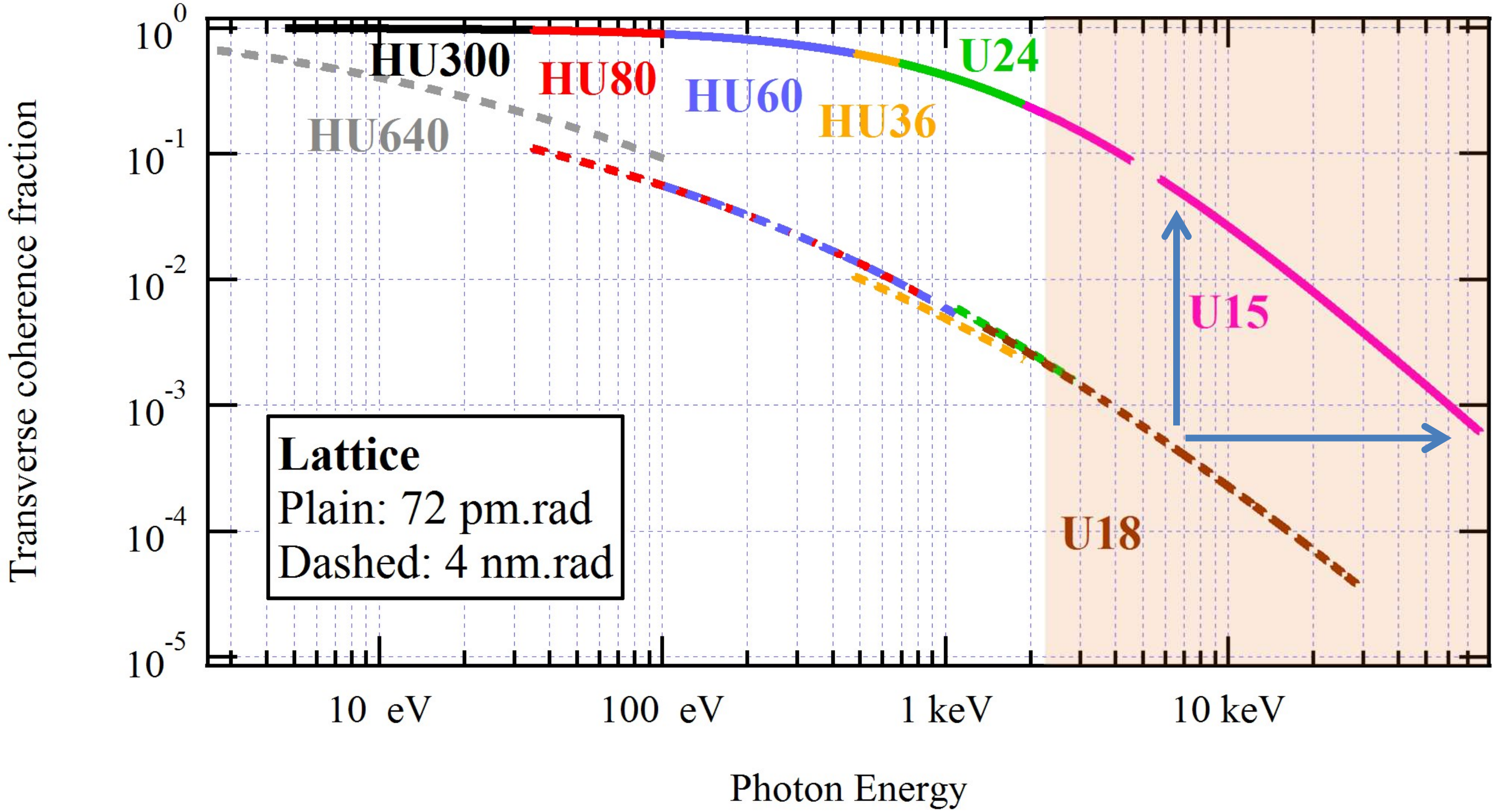


$\varepsilon_x = \sigma_x \sigma'_x$

$$\boldsymbol{B} = \frac{\boldsymbol{F}[ph/s/(0.1\%\Delta E/E)]}{\boldsymbol{\sigma_x \sigma_z \sigma'_x \sigma'_z}}$$

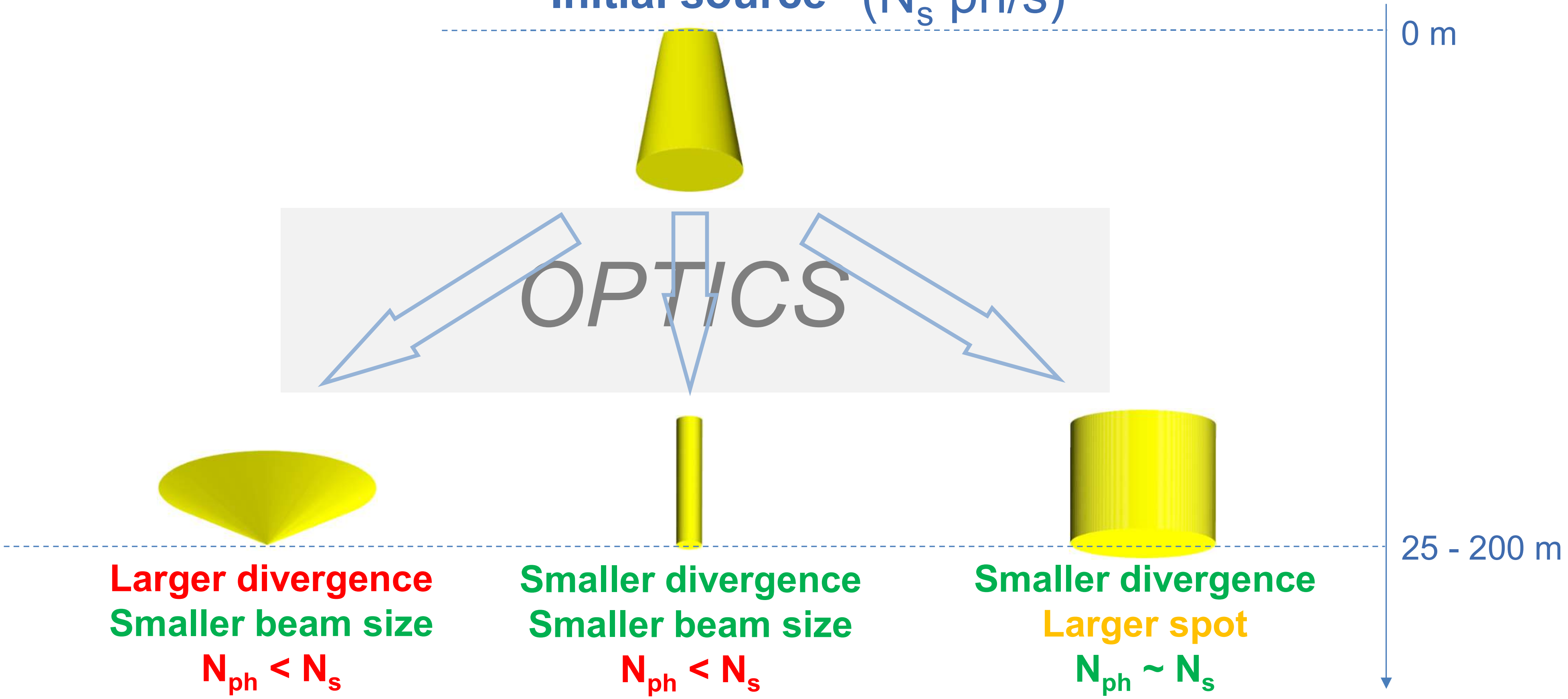
taille *divergence*

Coherent flux @ SOLEIL II



Optics

Initial source (N_s ph/s)



Larger divergence Smaller beam size $N_{ph} < N_s$	Smaller divergence Smaller beam size $N_{ph} < N_s$	Smaller divergence Larger spot $N_{ph} \sim N_s$

X-ray absorption spectroscopy

X-ray diffraction

Optics

Initial source (N_s ph/s)

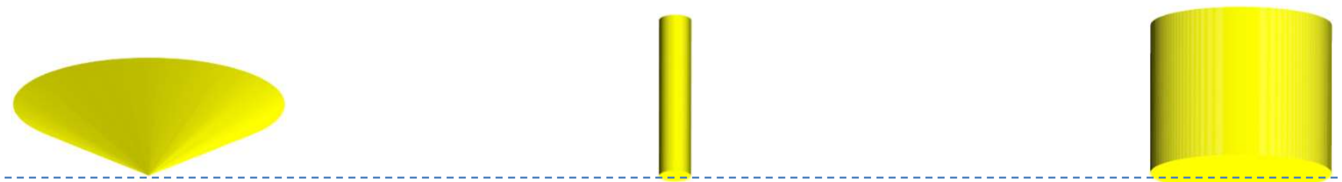
0 m

OPTICS

Wavelength selection:
→ diffraction based optics: monochromators
→ reflection based optics: mirrors

Focusing:
→ reflection/réfraction based optics

$n=1-\delta+i\beta$, ($\delta \sim 10^{-5}$; $\beta \sim 10^{-6}$)
- **very weak refraction**
- **important absorption**

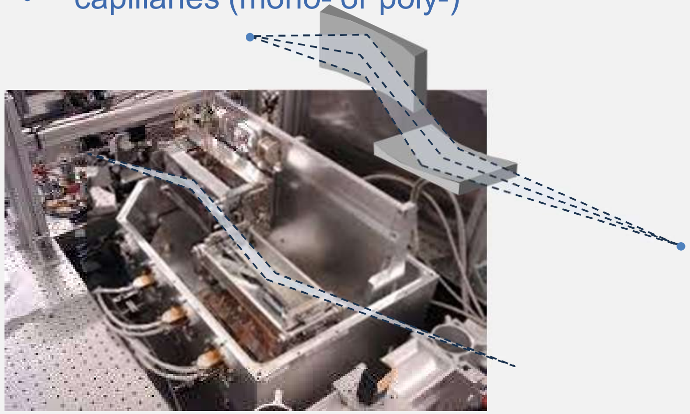


25 - 200 m

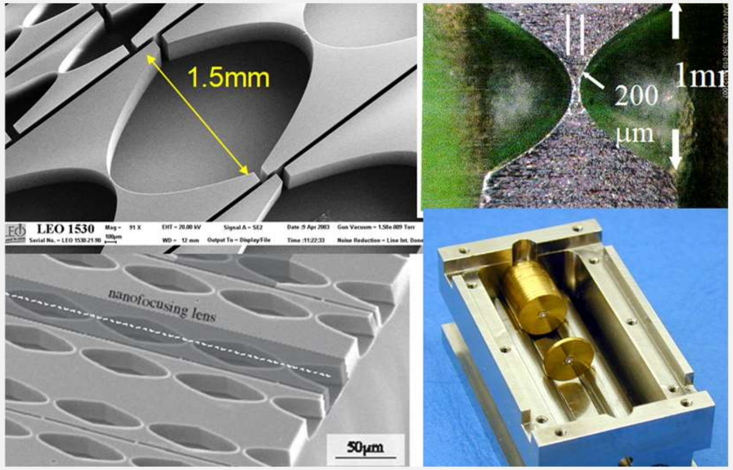
$N_{ph} < N_s$	$N_{ph} < N_s$	$N_{ph} \sim N_s$
Larger divergence	**Smaller divergence**	**Smaller divergence**
Smaller beam size	**Smaller beam size**	**Larger spot**

Optics: examples

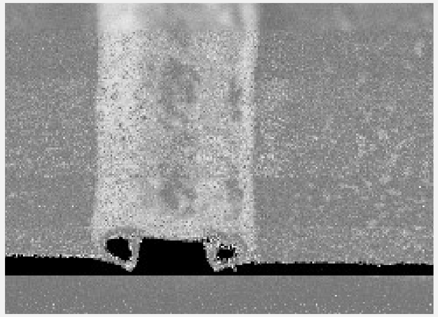
Reflective optics
- x-ray mirrors (curved, KB)
- capillaries (mono- or poly-)



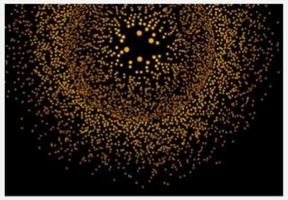
Refractive optics
- Compound Refractive Lenses
- Planar lenses
- Kinoform lenses

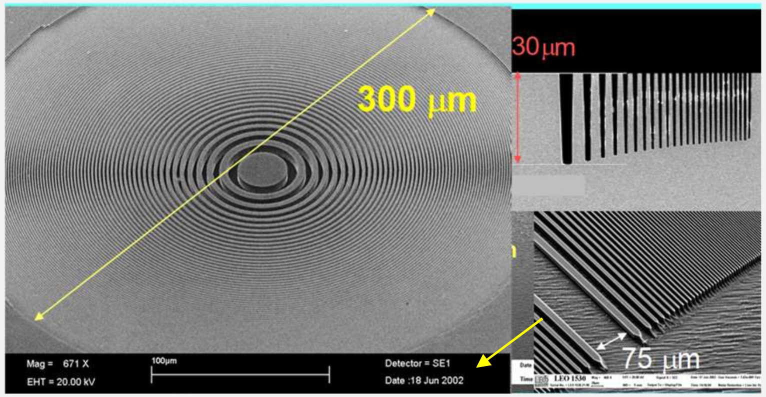


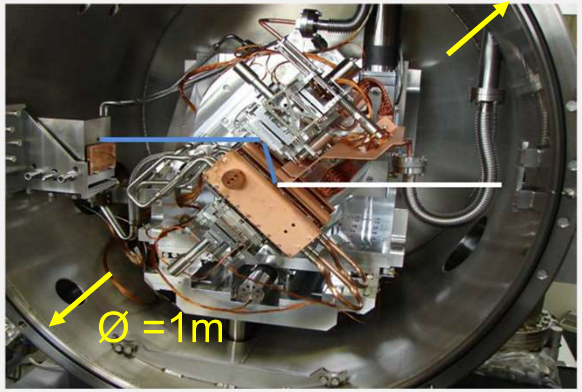
X-ray resonators
- waveguides



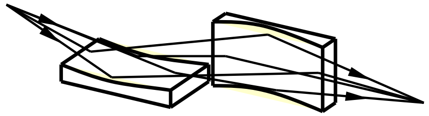
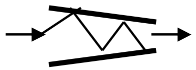
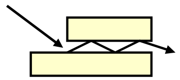
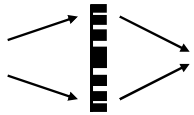
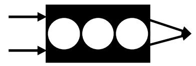
Diffractive optics
- crystals, multilayers, gratings
- Fresnel lenses (Bragg, Laue)
- Photon sieves, pin-holes







Focusing optics

	REFLECTIVE				***DIFFRACTIVE***	***REFRACTIVE***
	Kirkpatrick Baez systems		**Capillaries**	**Waveguides**	**Fresnel Zone plates**	**Refractive lenses**
	mirrors Kirkpatrick Baez, 1948	multilayers Underwood Barbee, 1986	Kreger 1948	Feng *et al.* 1993	Baez 1952	Snigirev *et al.*, 1996
						
E	< 30 keV	< 80 keV	< 20 keV	< 20 keV	< 30 keV (80)	<1 MeV
ΔE/E	wide band	10^{-2}	wide band	$10^{-2} – 10^{-3}$	$10^{-3} - 10^{-4}$	$10^{-3} - 10^{-4}$
min. spot size	**< 25 nm**	**~ 40 nm**	**50 nm**	**30 nm**	**30 nm**	**50 nm**
spot-size	**+++**	**+++**	**+++**	**+++**	**+++**	**+++**
flux	**+++**	**+++**	**---**	**---**	**++**	**+**
achromatic	**YES**	**NO**	**YES**	**NO**	**NO**	**NO *but* f(N,E)**
coherence	**+**	**+**	**+/-**	**+++**	**++**	**+/-**
in-line	**NO**	**NO**	**YES**	**YES**	**YES**	**YES**
long-f	**YES**	**YES**	**NO**	**NO**	**YES**	**YES**
easy to use	**+/-**	**+/-**	**++**	**+/-**	**++**	**++**
clean-spot	**+++**	**++**	**+++**	**+**	**+**	**++**

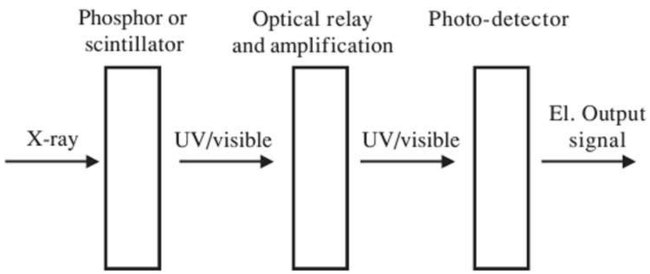
courtesy C. Mocuta

A.Snigirev et al., C.R.Physique ***9*** *(2008) 57*

Detectors

0D detectors

- Scintillation counters (e.g. NaI:TI, CsI:TI, Gd_2O_2S, YAG:Ce, $LaBr_3$:Ce…)
- Semi-conductor counters (e.g. Si diodes)



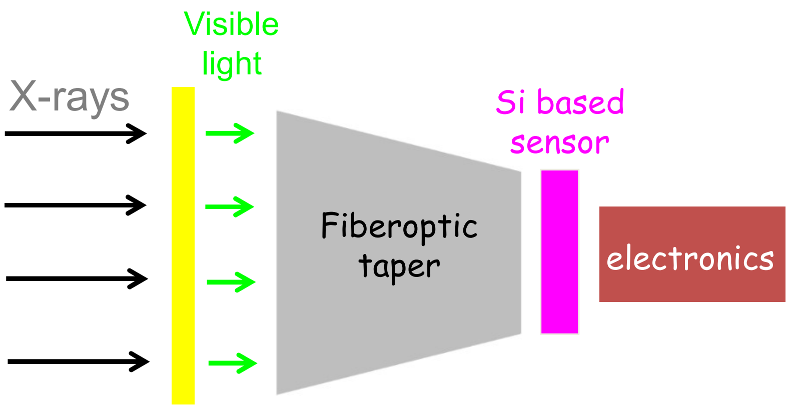
2D detectors

- Image plate
- Charge Coupled Device, CMOS
- (Hybrid) Pixels

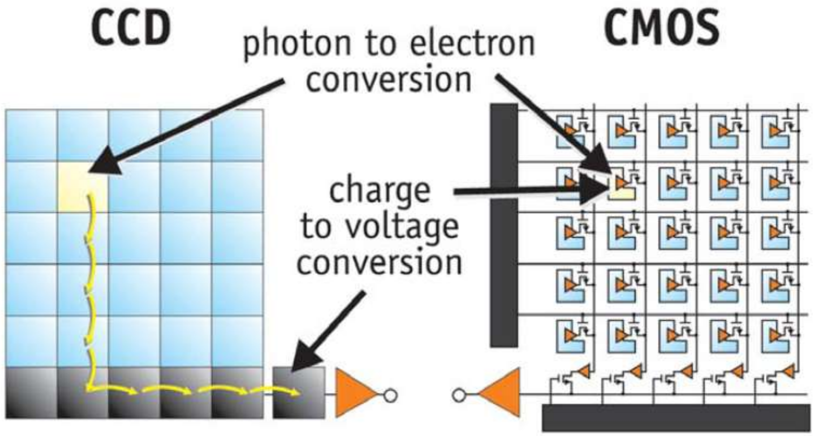
Important parameters :
- **Efficiency**
- **Dynamic**
- **Resolutions (spatial/energy)**

CCD/CMOS Detectors

Charge
Coupled
Device



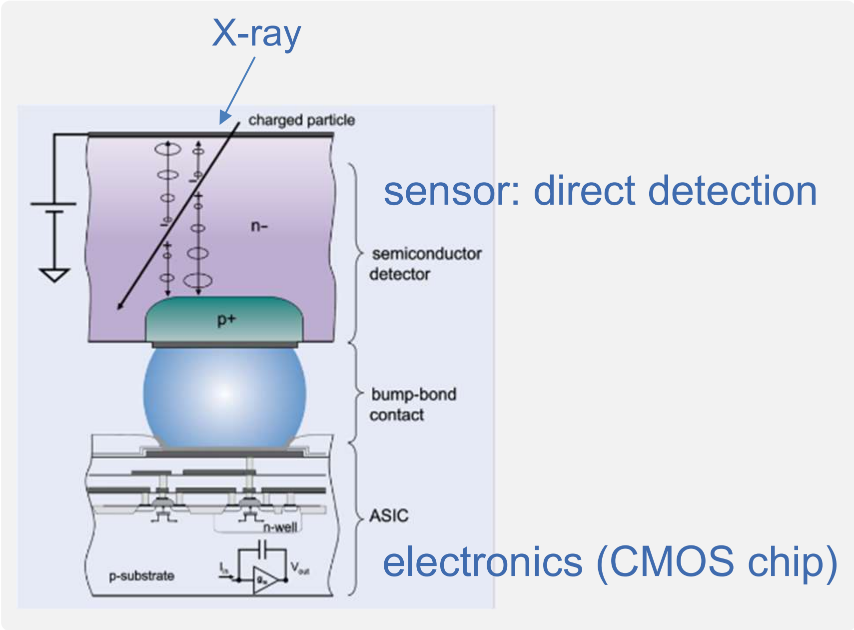
Complementary
Metal-Oxide
Semiconductor



Photon counting detectors

Xspectrum lambda2M GaAs

pixel: 55x55 µm^2



Integrating detectors

Adaptative Gain Integrating Pixel Detector (PSI - DESY) European XFEL

Dectris Eiger 2 X CdTe 9M

pixel: 75x75 µm^2

Detectors

	CCD	CMOS	Hybrid pixels
type	charge integrating	charge integrating	photon counting charge integrating
signal out of pixel	e- packet	Voltage	Voltage
signal out of chip	Voltage	Bits (digital)	Bits (digital)
signal out ouf camera	Bits (digital)	Bits (digital)	Bits (digital)
sensor complexity	+	-	--
pixel size	+ (~ 50 µm)	+	+ (~ 55 µm*)
dynamic range	-	-	++
uniformity (dark/illumination)	+/+	-/+	++/++
speed	-(5Hz)	+(100Hz)	++(a few kHz)
windowing (Region Of Interest)	--	++	++
antiblooming	-	++	++
continuous scans (shutter free)	-	+	+
dead zones	+	+	--

(Xspectrum detector)*

Experimental methods

Diffraction techniques

Angular dispersive Diffraction $2d \sin(\theta) = \lambda$

- Single Crystals (cf. lecture of E.-E. Bendeif on Wednesday)
    - Laue method (« white beam »)
    - Monochromatic beam
- Powders (cf. lecture of E. Elkaïm on Wednesday)
- High resolution powders

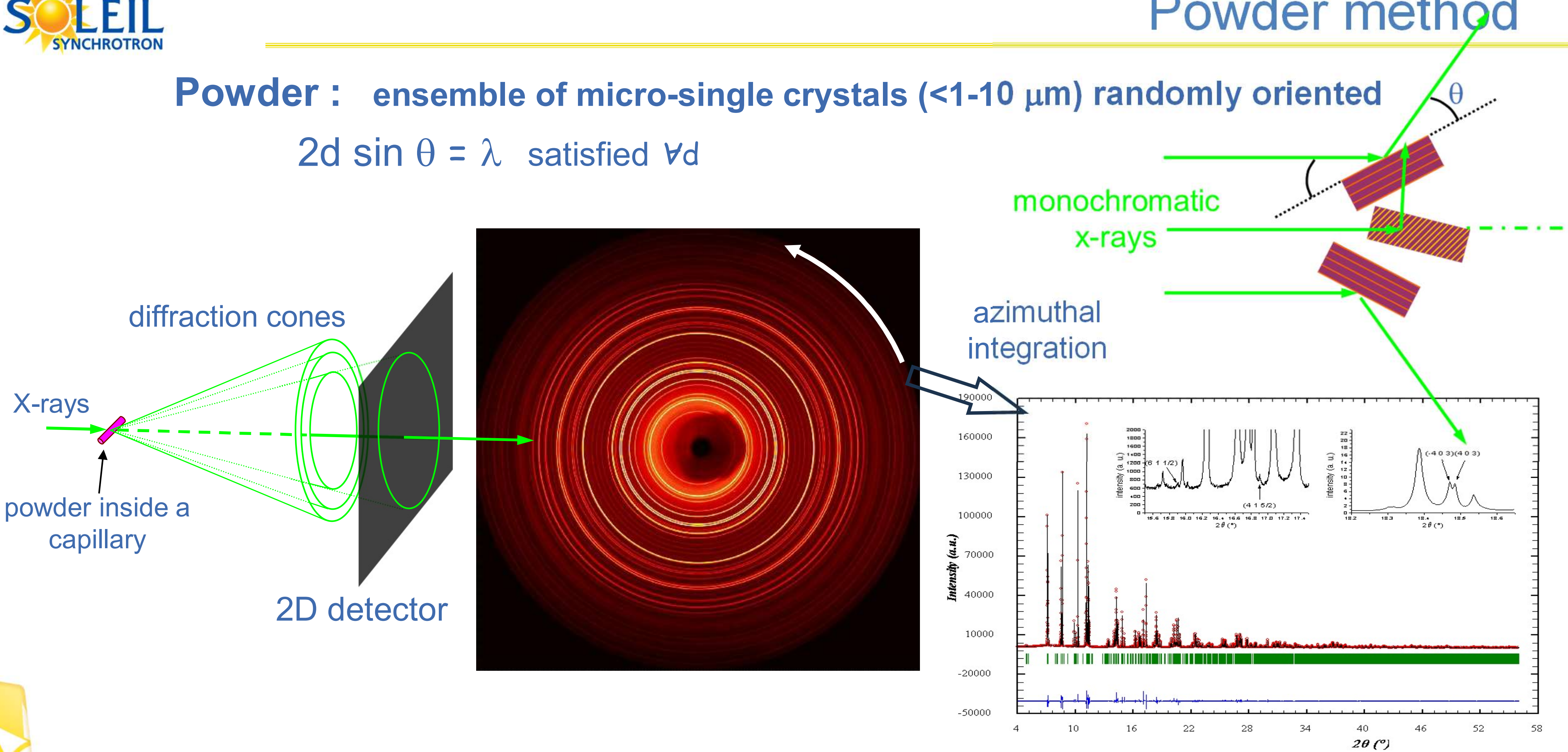
Energy dispersive Diffraction $d = 6.1999/E[keV] \sin(\theta_0)$

Energy dispersive + angular dispersive Diffraction

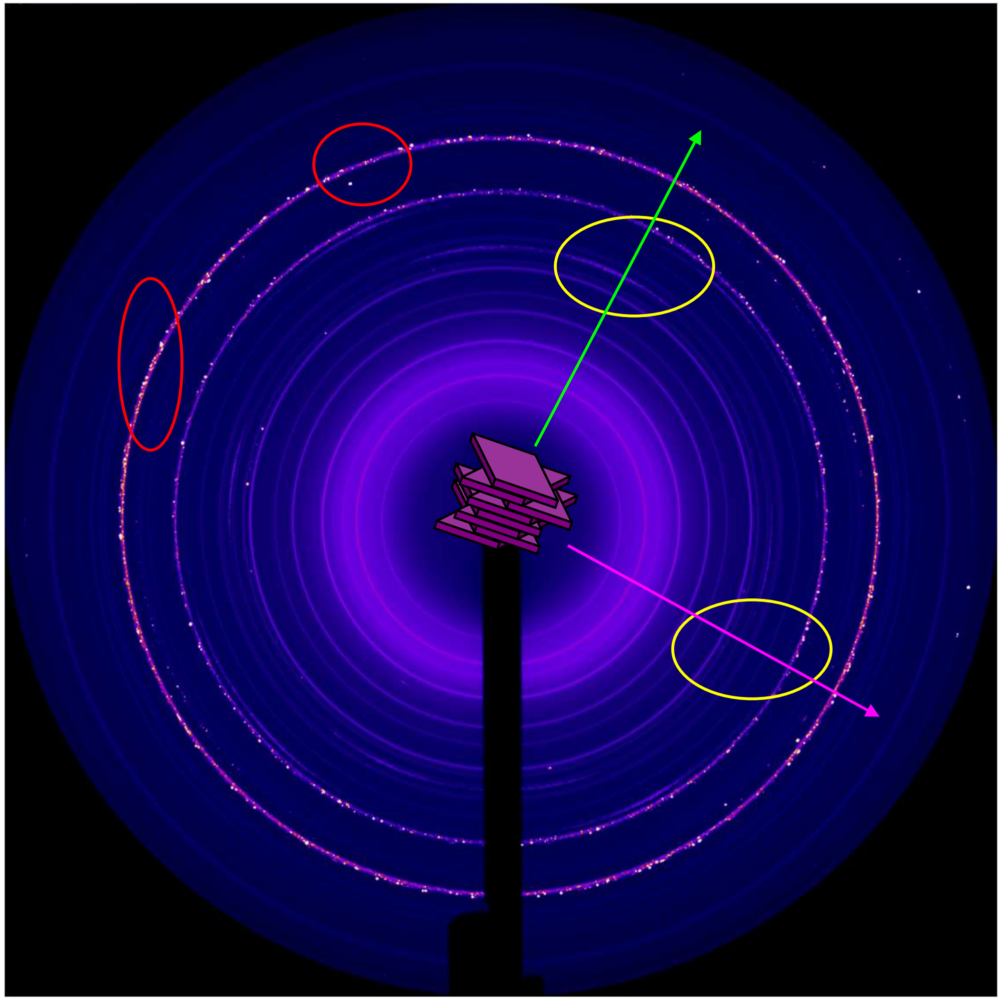
e.g. CAESAR setup (cf visit of the PSICHE beamline)

Powder : **ensemble of micro-single crystals (<1-10 μm) randomly oriented**

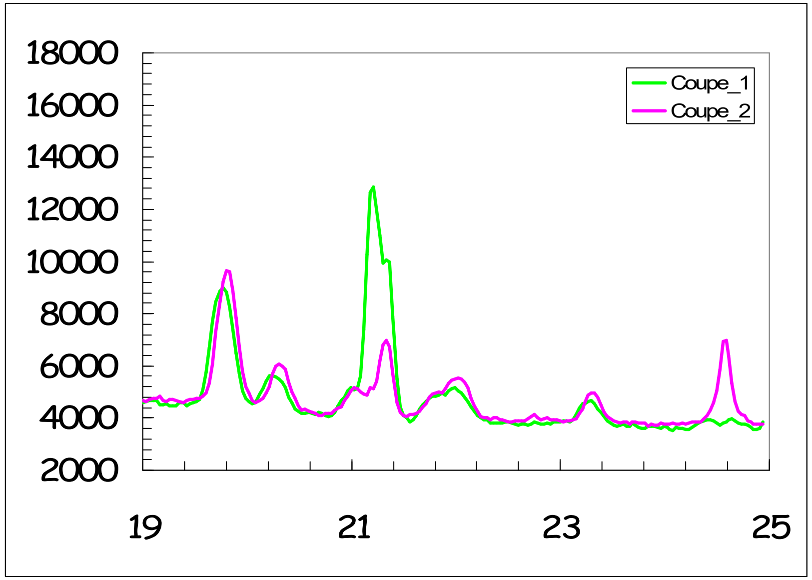
$2d \sin \theta = \lambda$ satisfied $\forall d$



Powder method

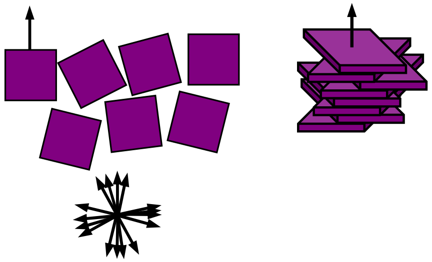


+/- continuous rings

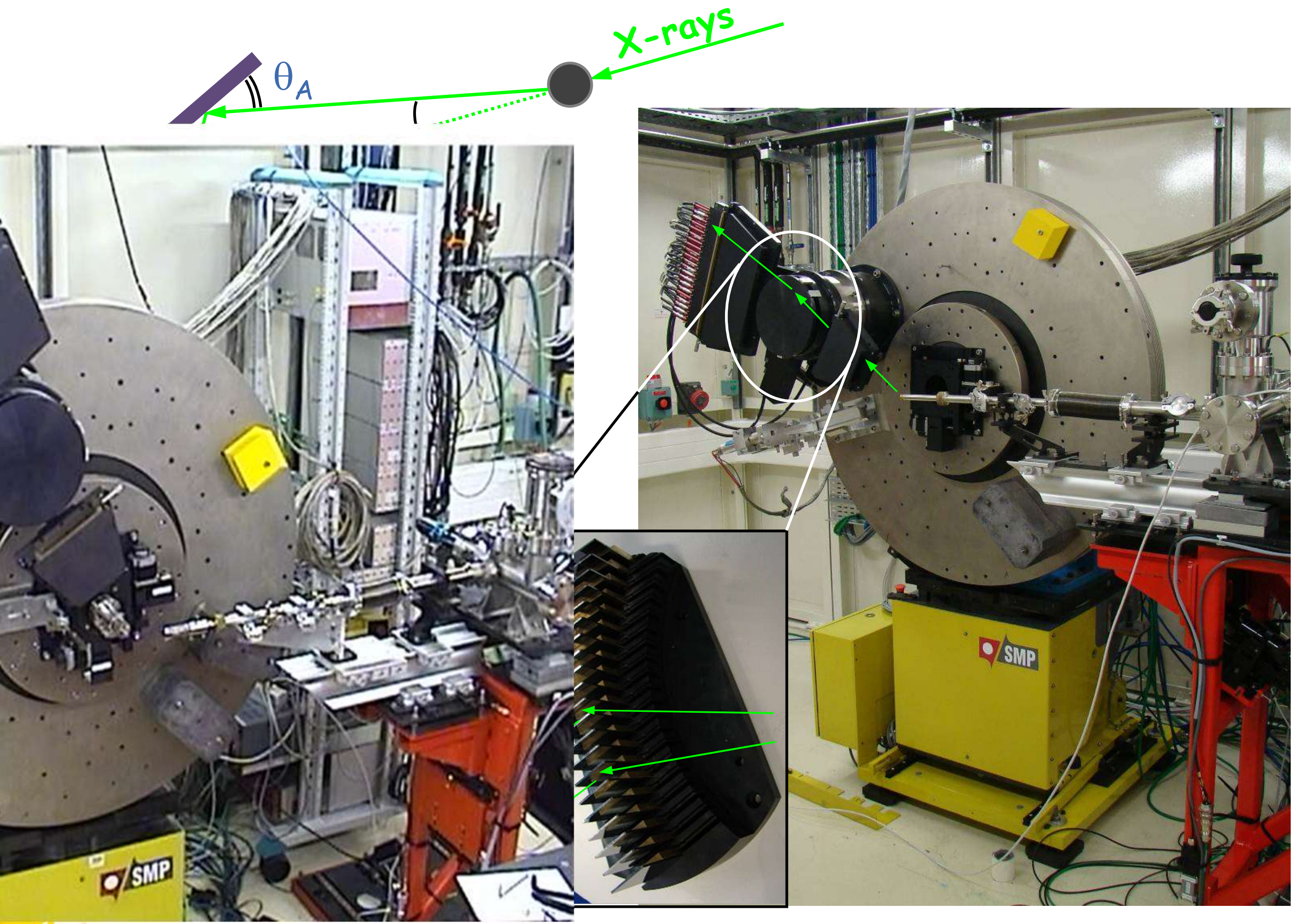


grain number effect

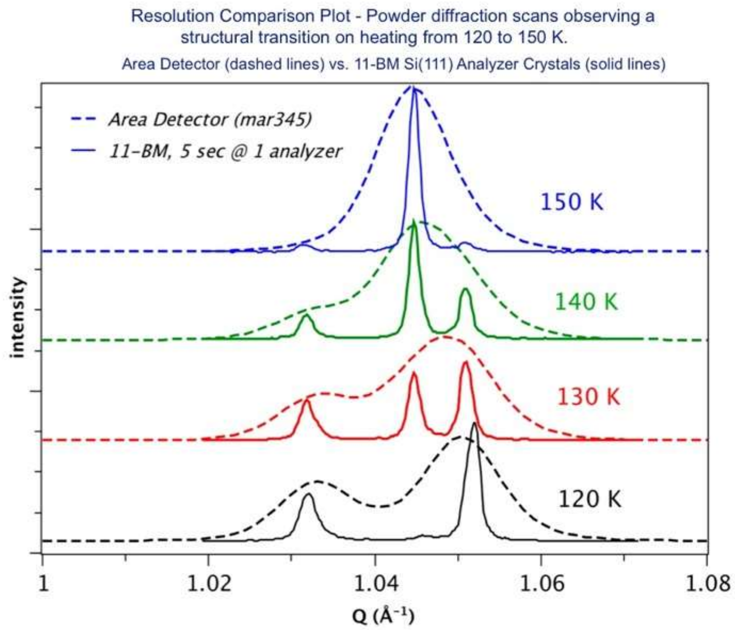
textured sample



High resolution powder diffraction

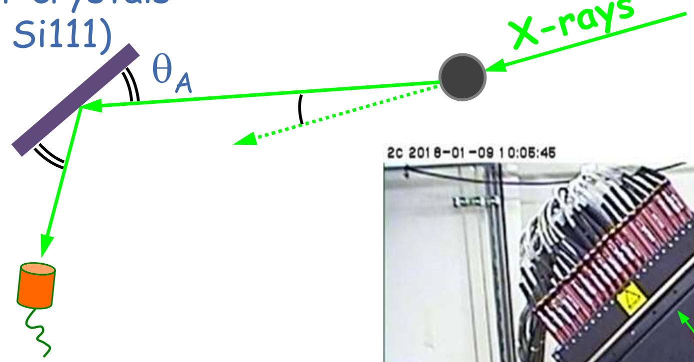


ex.: structural phase transition

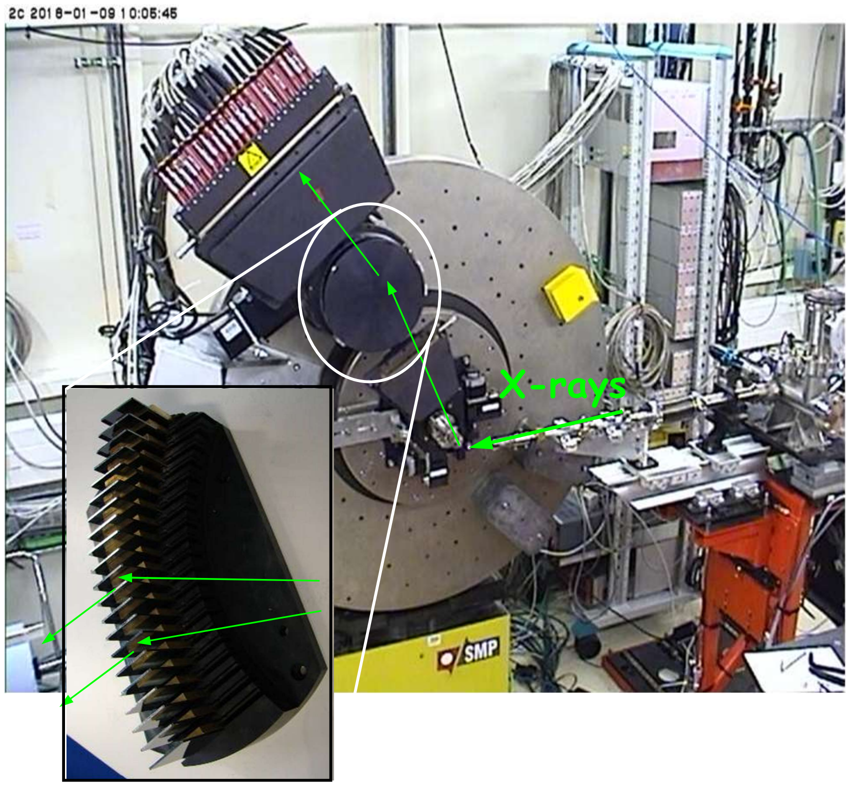


2-circles diffractometer @ CRISTAL

High resolution powder diffraction

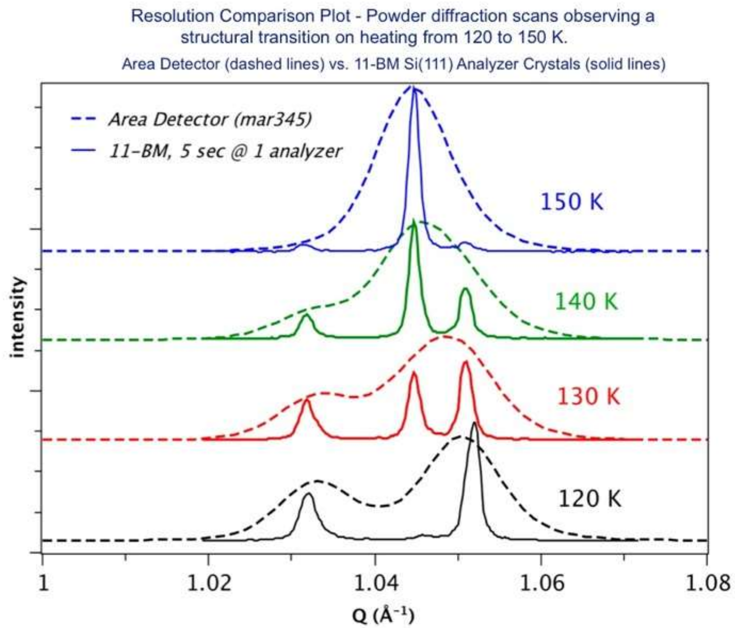


- signal/noise
  I_{max} / noise ~ 1000
- peak width
  $FWHM_{RS}$ ~ 0.004°
  ($FHWM_{lab}$ ~ 0.04°)
  ($FWHM_{Mythen}$ ~ 0.01°)



2-circles diffractometer @ CRISTAL

ex.: structural phase transition



Powder method advantages

- no (« big ») single crystals
- multi scale approach (pair distribution function cf. lecture of P. Bordet on Thursday)
- identification de phase
- (very) fast
- …

BUT 3D info lost!!!

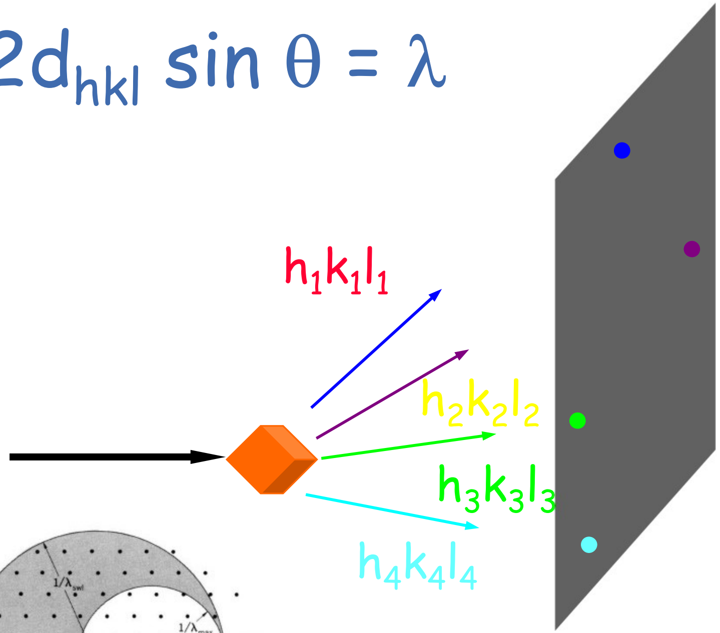
- symmetry equivalent Bragg peak superimposed
- overlaps of peaks with close d_{hkl} …

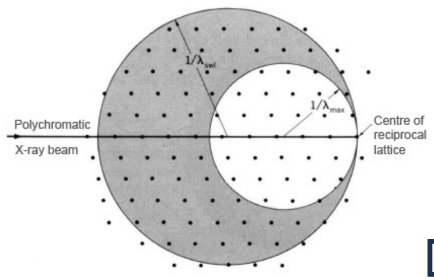
Energy dispersive methods

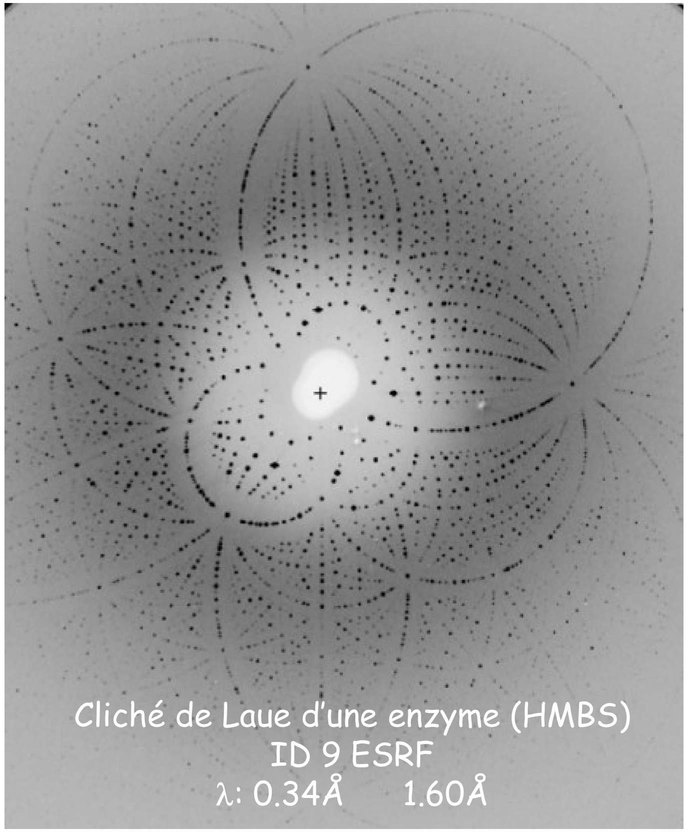
- constraining sample environment (e.g. studies under Pressure)
- energy resolved detector

Laue Method: white beam, catch all θ

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



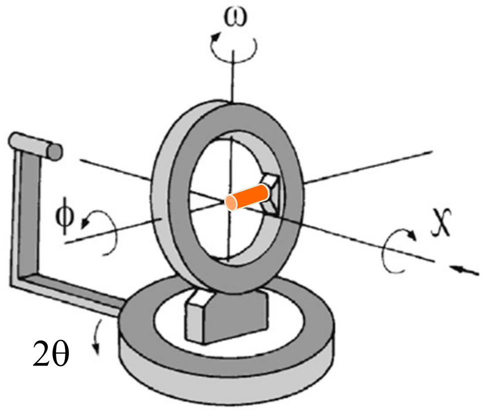


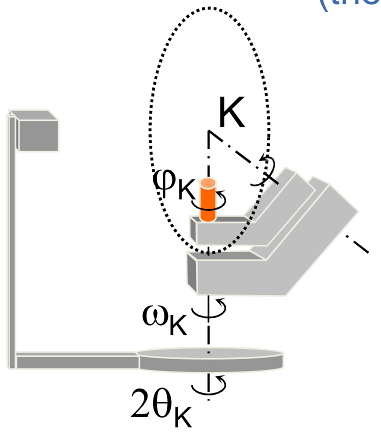


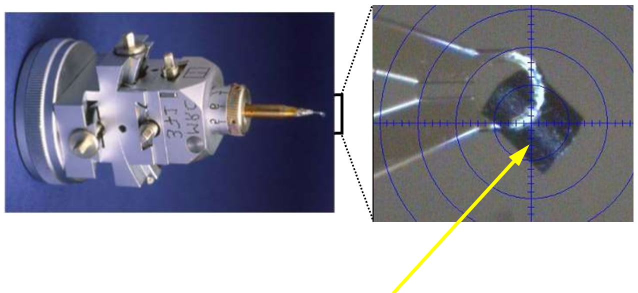
⇨ simultaneous recording of a large number of Bragg peaks

4-circles diffractometers: monochromatic beam
orient the crystal in any directions
measure Bragg peak intensities

(the highest number the best, the most accurate the best)







sample: dim_{max} < 50 mm

Eulerian geometry

« kappa » geometry

χ = combinaison of ω, κ and φ rotations

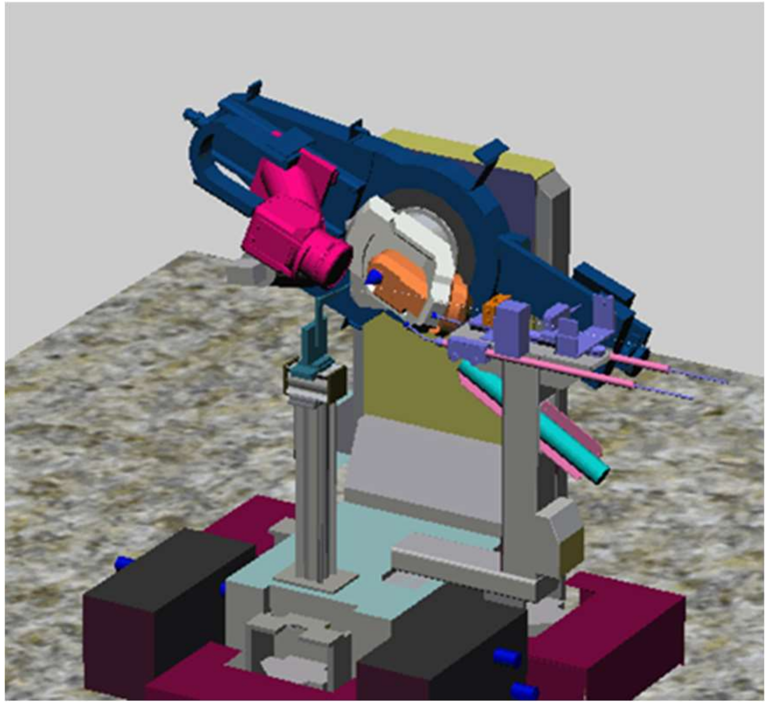
shadowing effects reduced

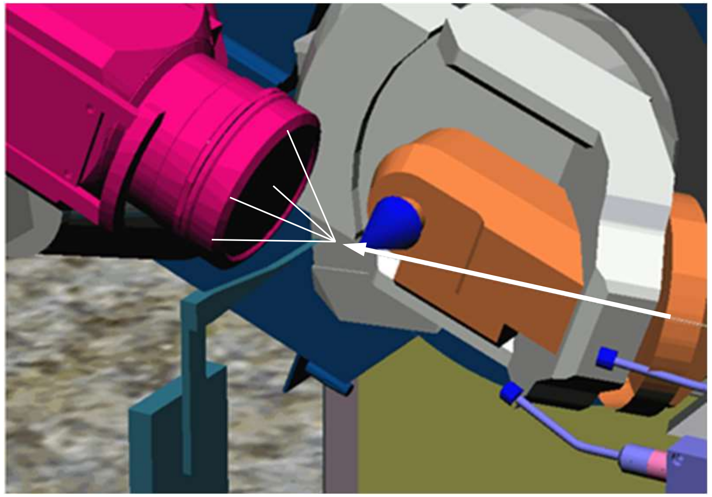
+++ sample environnments

+++ 2D detector

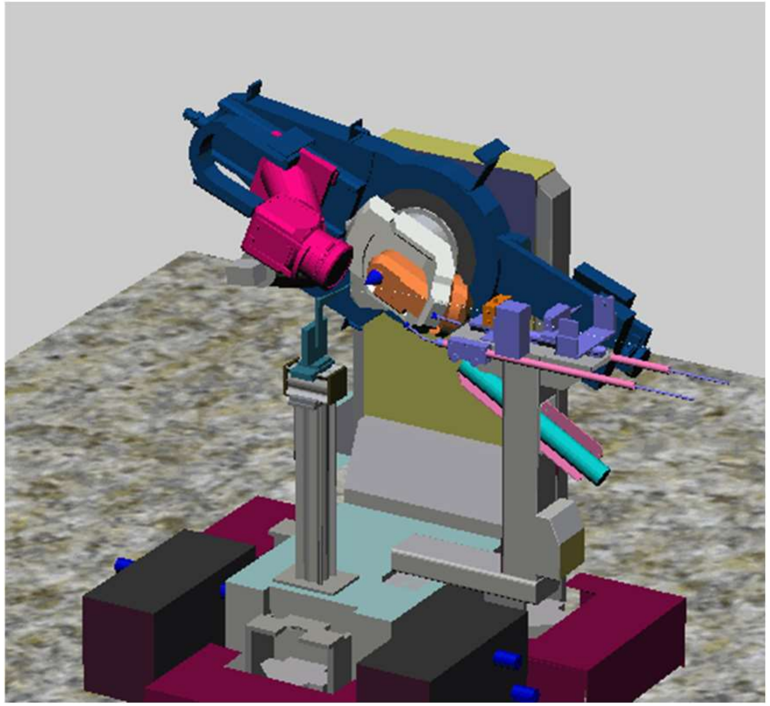
Single Crystals: rotating method

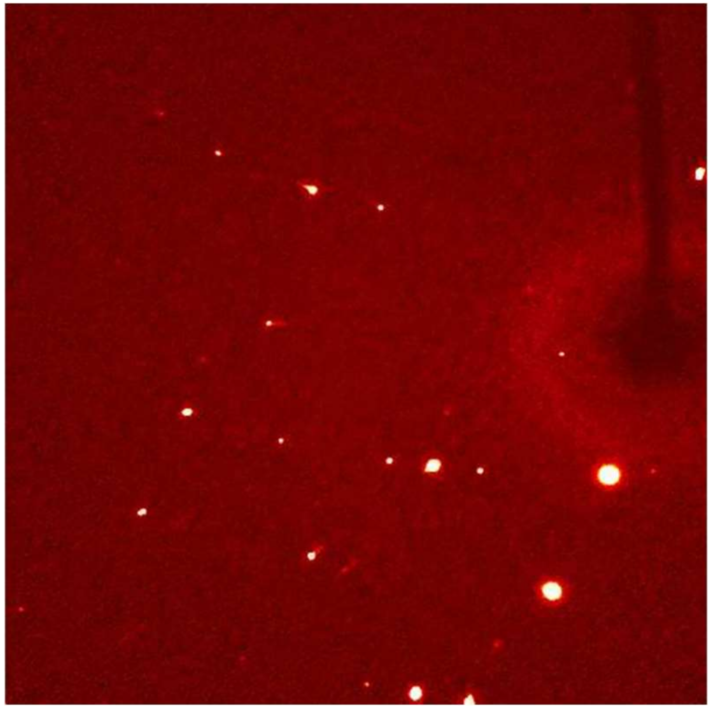
- (continuous) rotation of the crystal (shutter-less method)
- simultaneous intensity measurements typically during 0.05-0.1°

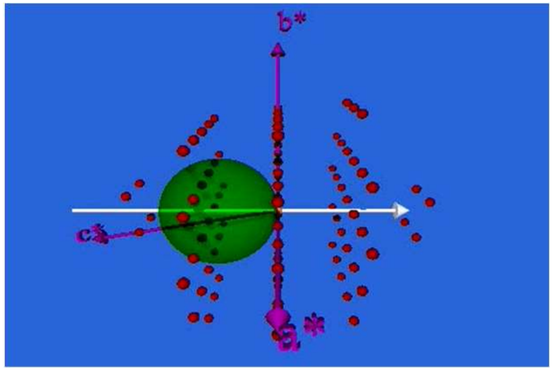




Single Crystals: rotating method

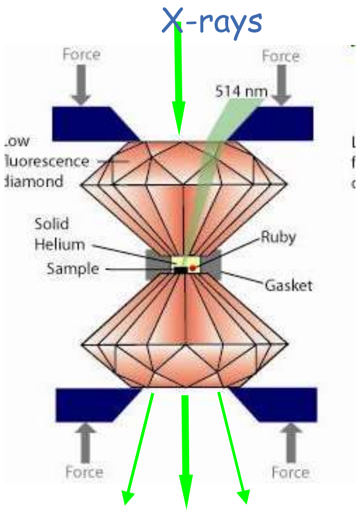


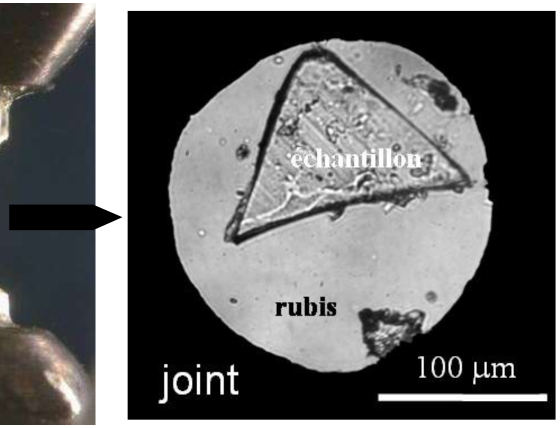




Non ambient conditions

Non ambient conditions: **pressure**





Diamond Anvil Cell

- transparent to X-rays/ visible light
- P_{max} ~ 5 - 300 Gpa*
- small footprint (can be mounted inside a cryostat)
- limited angular aperture

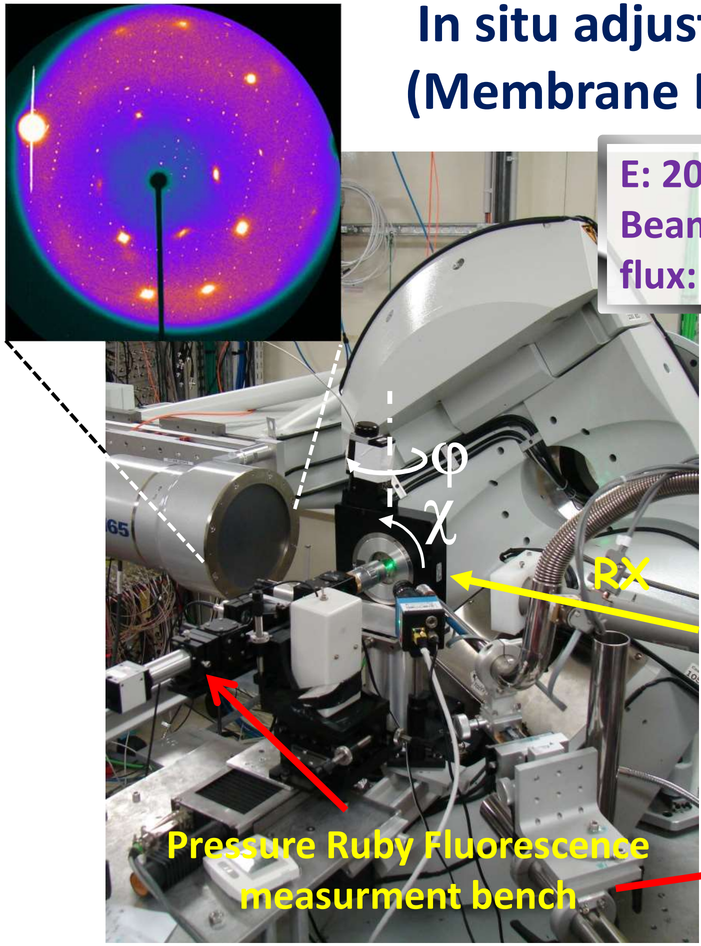
*(*1 GPa = 10 kbars)*

Non ambient conditions: **pressure**

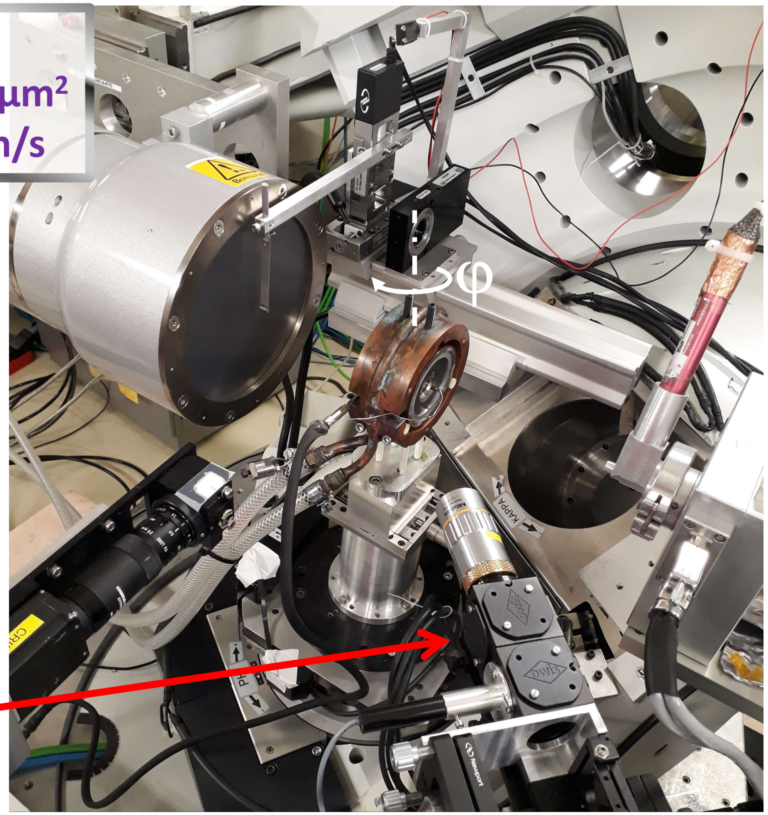
In situ adjustement of Pressure (Membrane Diamond Anvil Cell)

P: amb → 70 GPa

E: 20 – 30 keV
Beam size: 25 x 25 µm^2
flux: ~ 10^{10} - 10^{11} ph/s



Ambiant Temperature
(config. mesure P)



High Temperature setup
→ 700 K

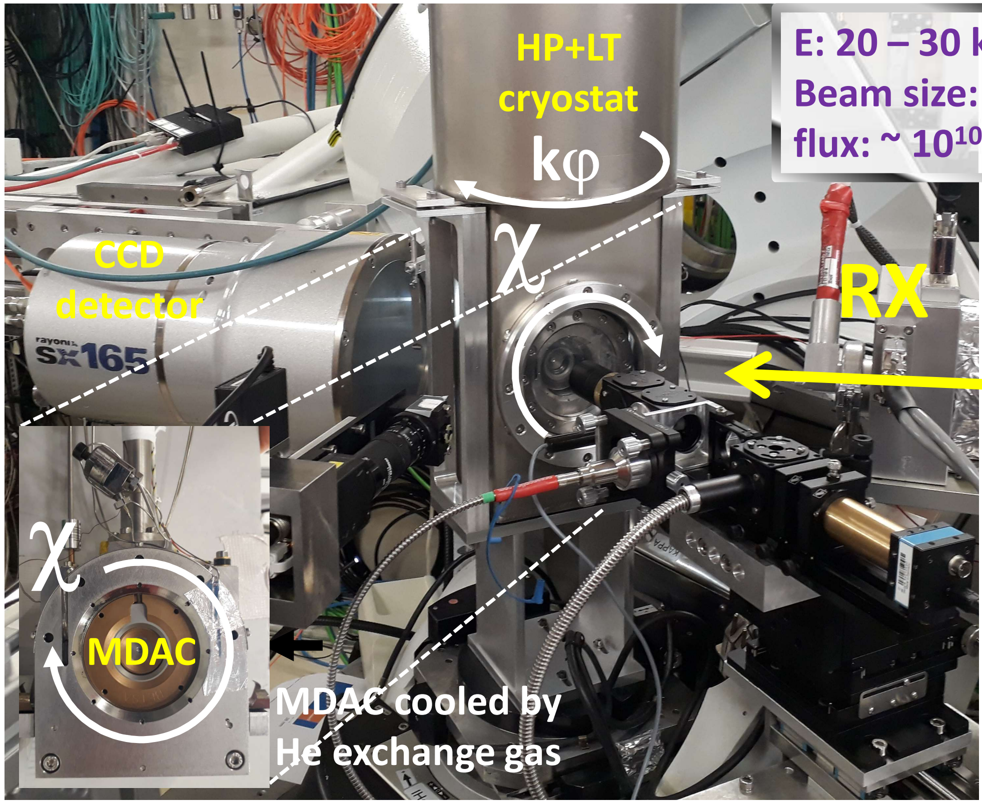
Non ambient conditions: **pressure**

In situ adjustement of Pressure (Membrane Diamond Anvil Cell)

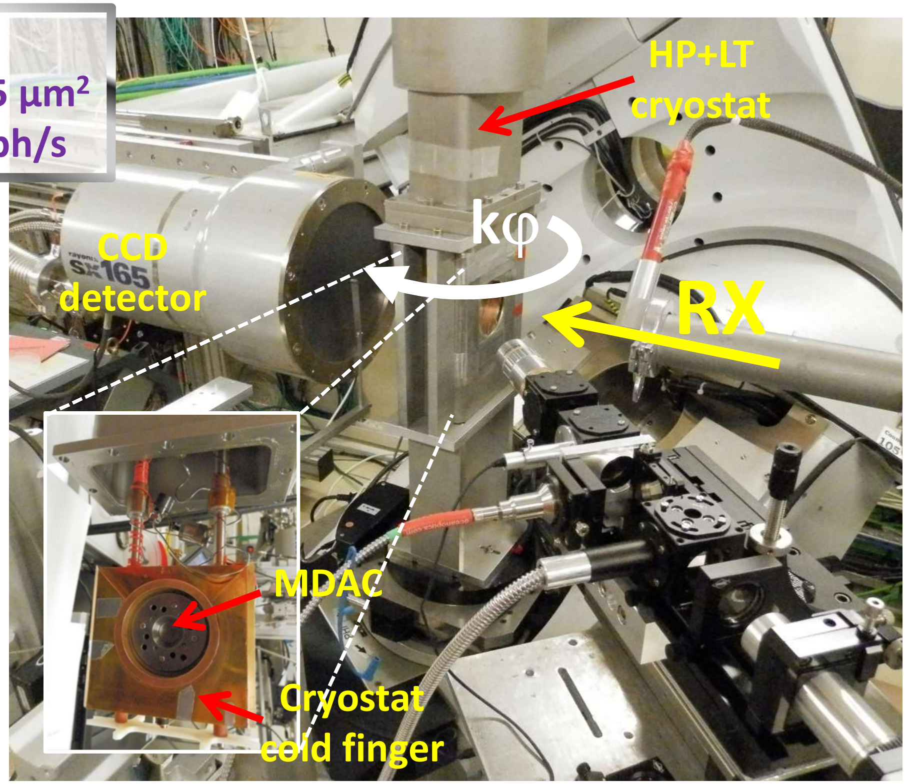
P: amb → 70 GPa

T_{min} : 6K

E: 20 – 30 keV
Beam size: 25 x 25 µm²
flux: ~ 10^{10} - 10^{11} ph/s



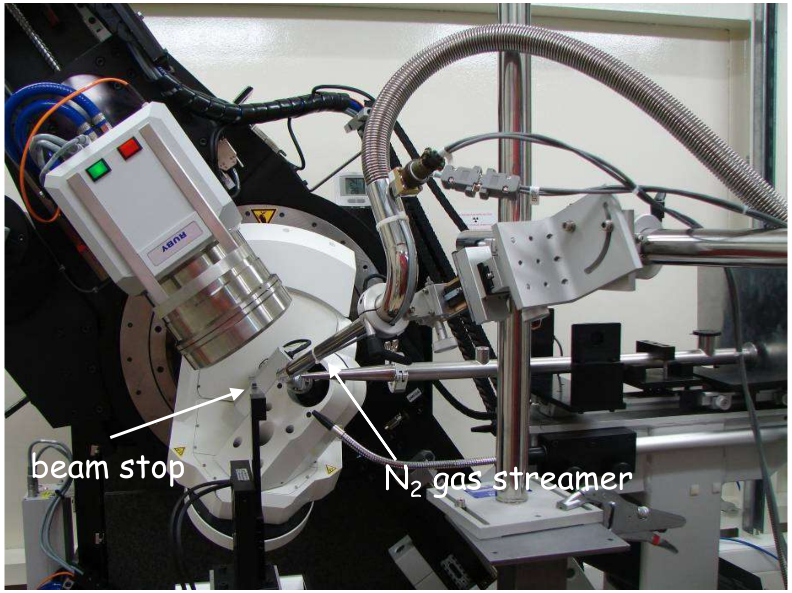
Single Crystal XRD Low Temperature setup (config. P measurement)

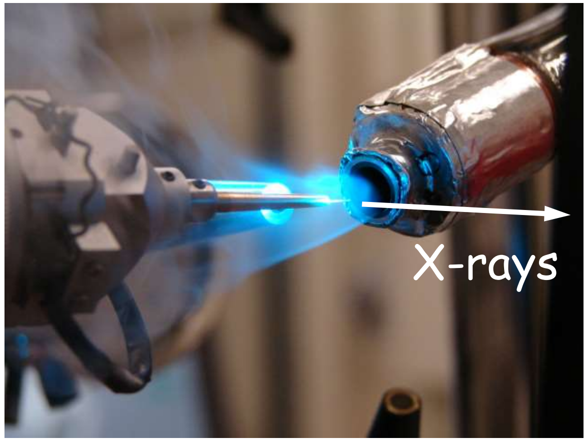


Powder XRD Low Temperature setup

Non ambient conditions: **low/high temperatures**

1) Blow a cold/hot gaz (N2, He, dry air) on the sample

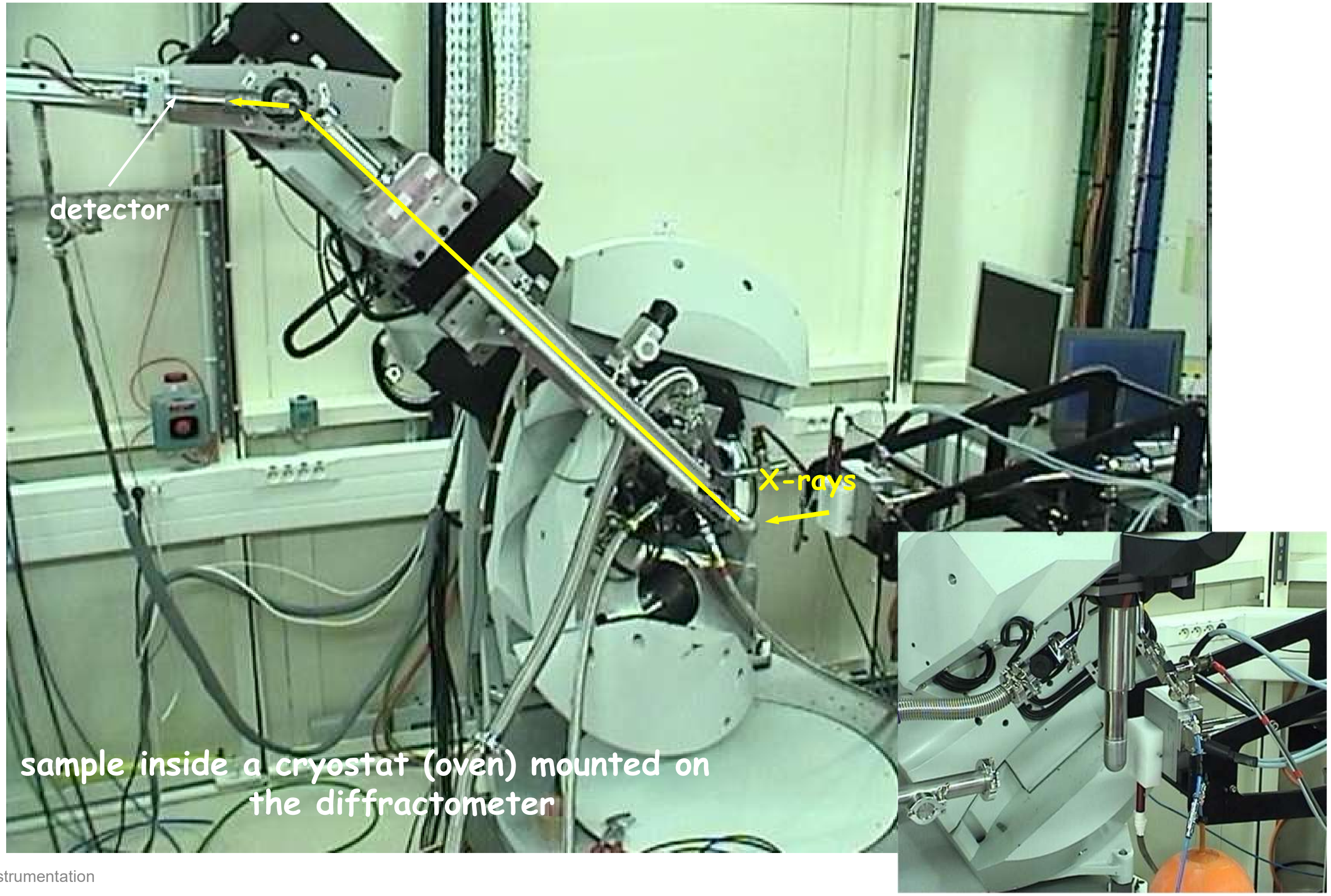




11K < T < 1000K

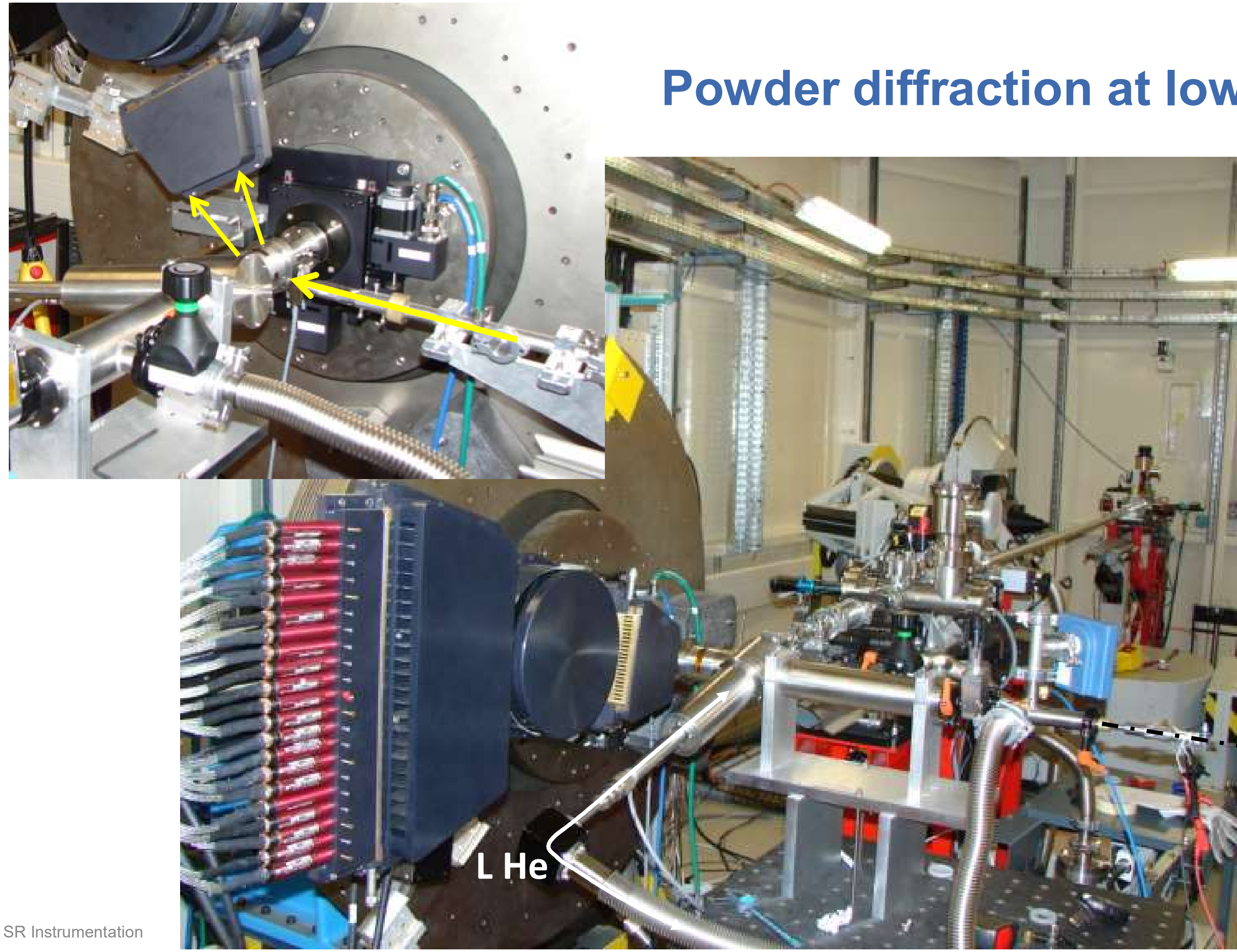
Non ambient conditions: **low/high temperatures**

2) Use a cryostat/oven



Non ambient conditions: **low/high temperatures**

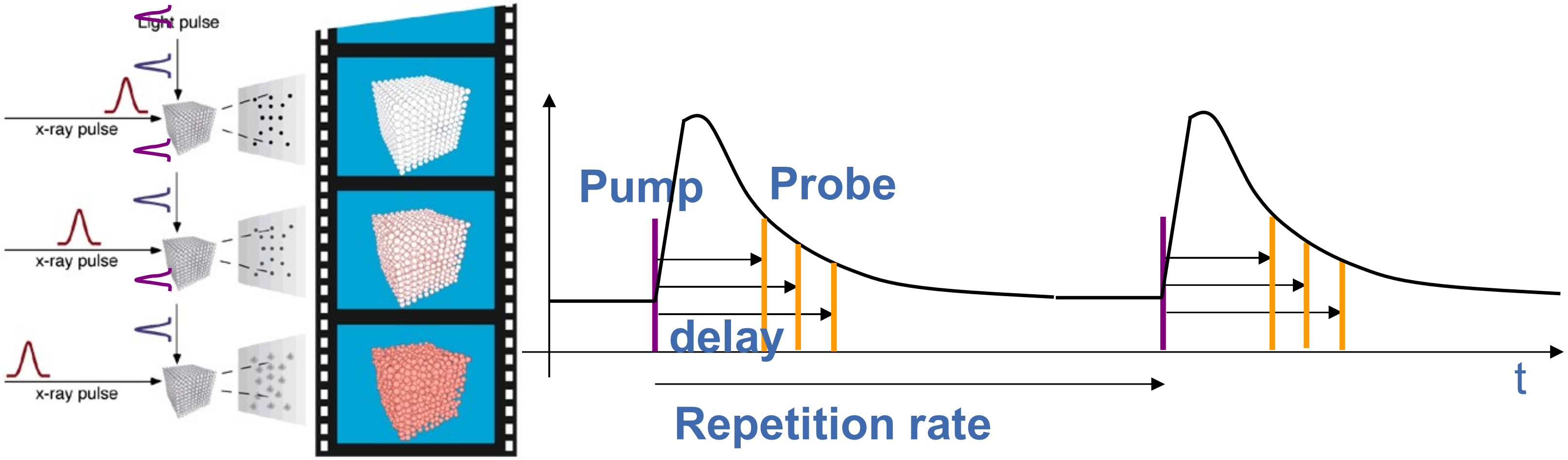
Powder diffraction at low temperatures



rotation a few t/s

pump-probe method (stroboscopic method)

reversible processus   time relaxation adapted with excitation frequency



Pump : laser pulse (~ 40 fs)
Probe : X-ray pulse (ESRF 100 ps, a few 10 fs@XFEL)

cf. lecture of C. Laulhé on Friday

Synchrotron Radiation

Brillance : weak intensities

Complexes structures
(very) small crystals (< 20 μm)
Charge density studies (beyong the sphericam model)
Time resolved experiments (operando)
Magnetic diffraction…

Low divergence : accuracy of rthe measurements

Phase identification/separation
Ab initio powder structure determination

Tunability

Optimal wavelength (absorption)
Resonant (anomalous) diffraction

Cohérence

Tunable polarisation (nature and/or direction)

Pulsed light

(ultrafast) time resolved experiment (a few 10 fs, ps)


ALBA (Spain)
SPRING 8 (Japan)
ELETTRA (Italy)
ANSTO (Australia)
SSRF (China)
NSRRC (Taiwan)
SLS (Swiss)
ALS (USA)
DIAMOND (England)
SIRIUS (Brazil)
European XFEL (Allemagne)
PETRA III (Germany)
ESRF-EBS (Grenoble)
BESSY (Germany)
MAX IV (Sweden)
CLS (Canada)
NSLS II (USA)
NSRRC (Taiwan)
SESAME (Jordania)