Powder Diffraction at the Upgraded Materials Science Beamline

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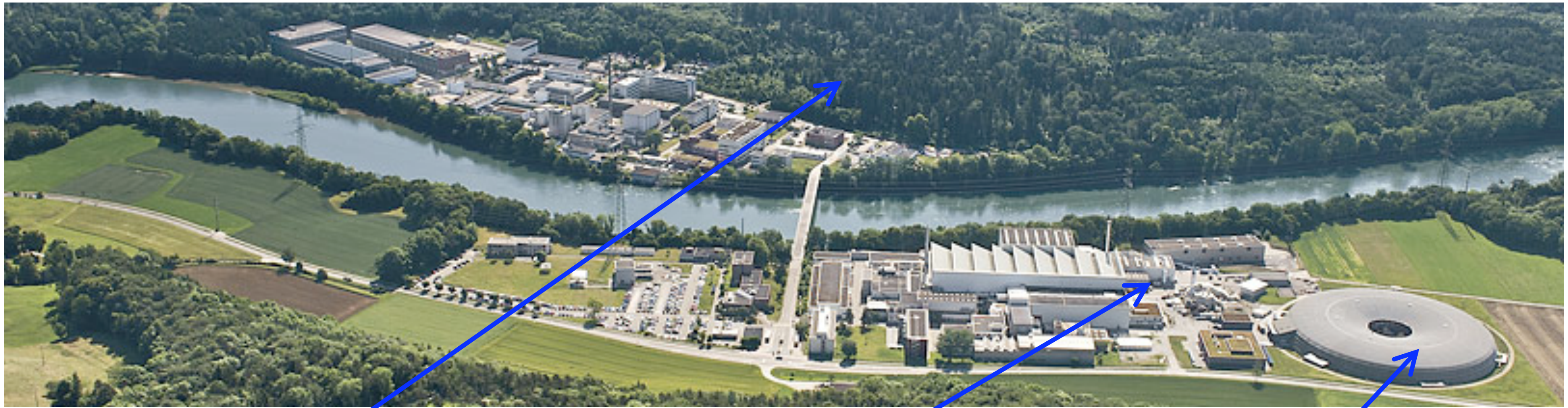


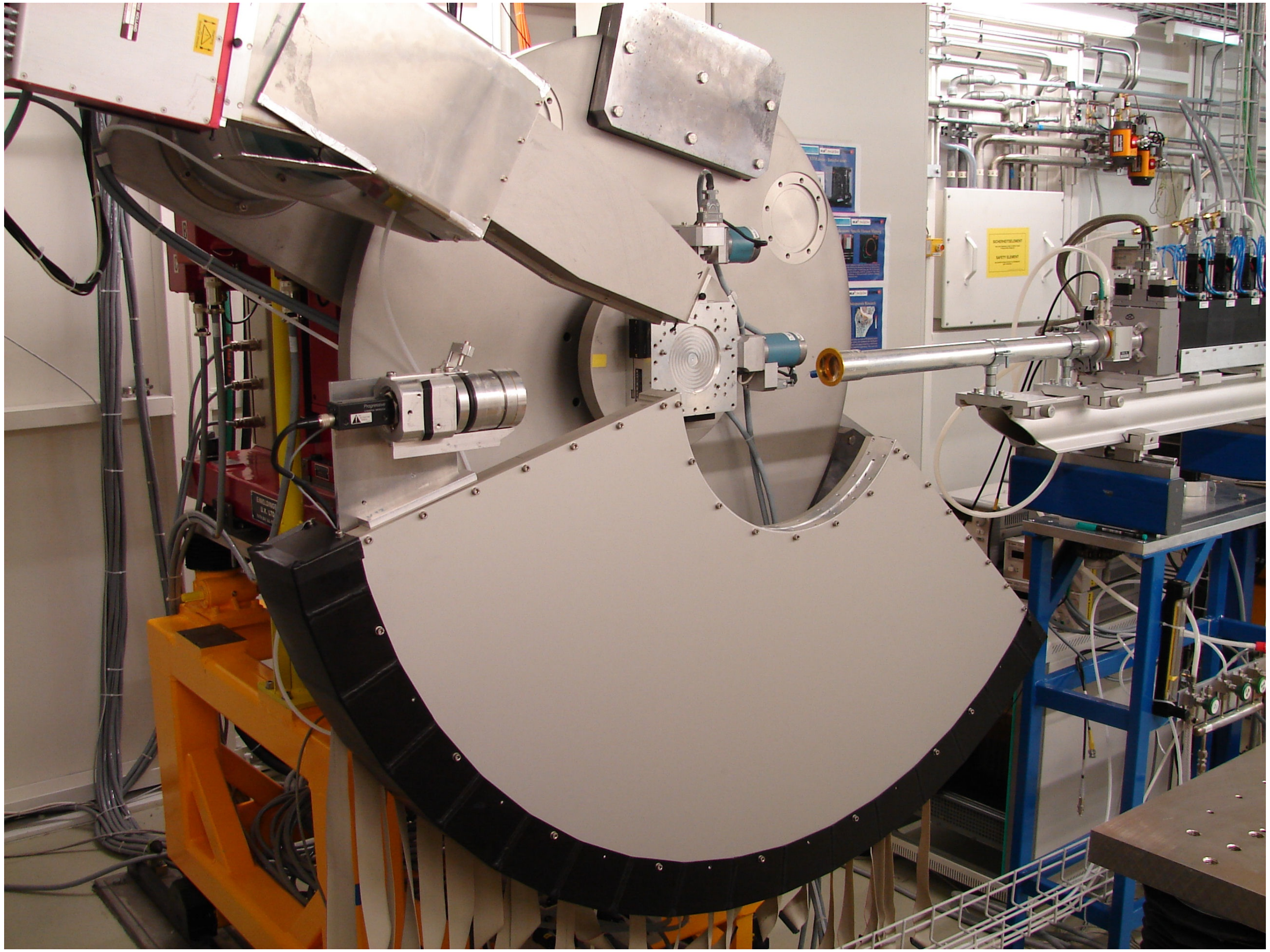
SwissFEL (future)

SLS synchrotron

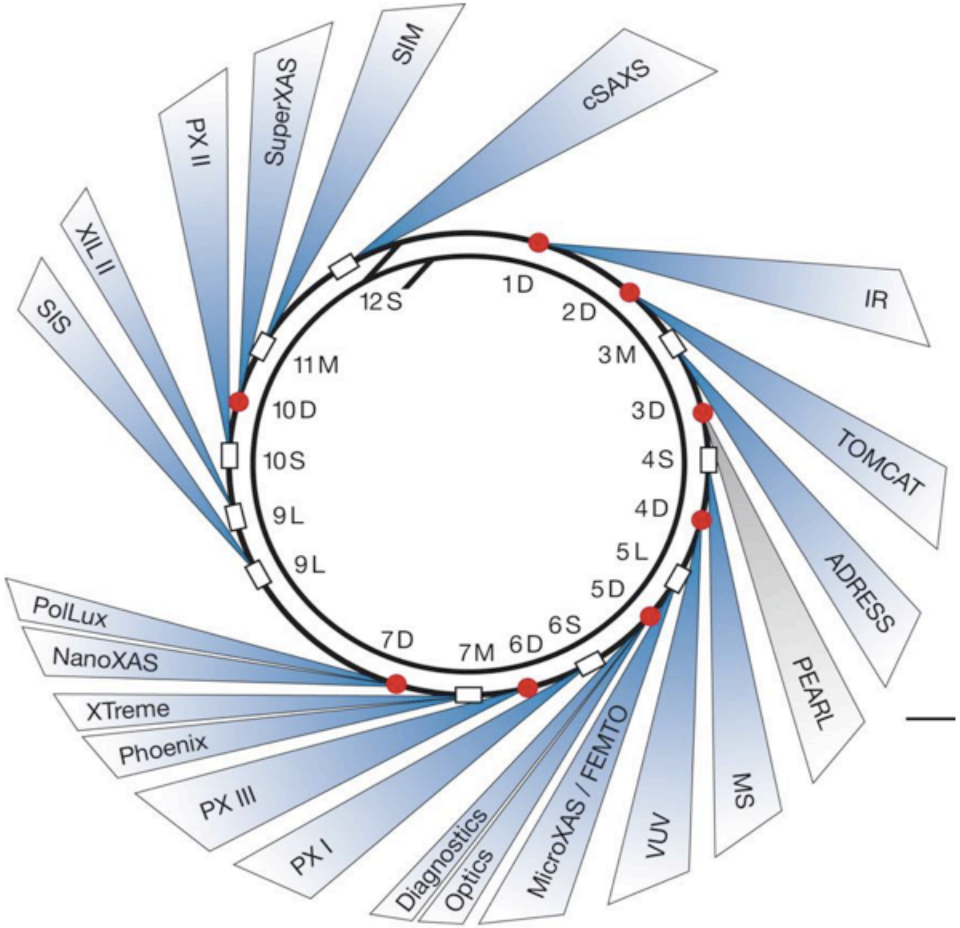
SINQ neutron source

μSR muon source

The Materials Science Beamline at SLS - Powder Station



Synchrotron: **e⁻ accelerated in a ring** (SLS: 2.6 GeV)
They pass through “Insertion Devices” (**IDs**) and produce **photons**
(between them, **X-rays**)



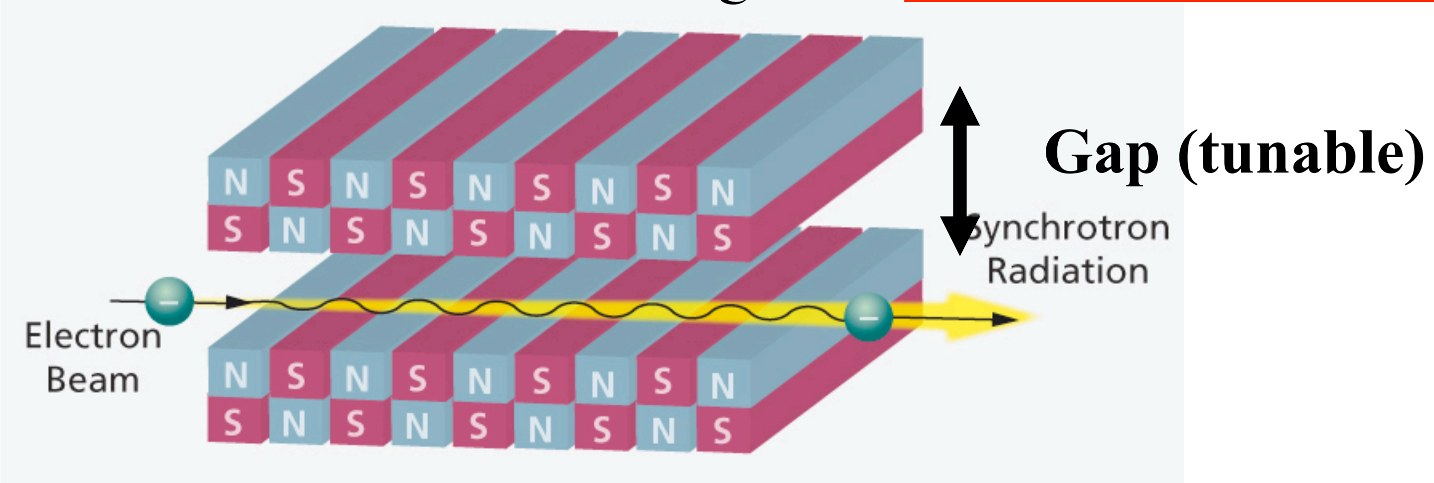
IDs : use a **magnetic field** to curve the electrons trajectory; then e^- emit photons by ***Bremsstrahlung***

- Bending magnet (one curve)
- Wiggler (many curves)

-**Undulator** : most modern, very many small curves: **interference**

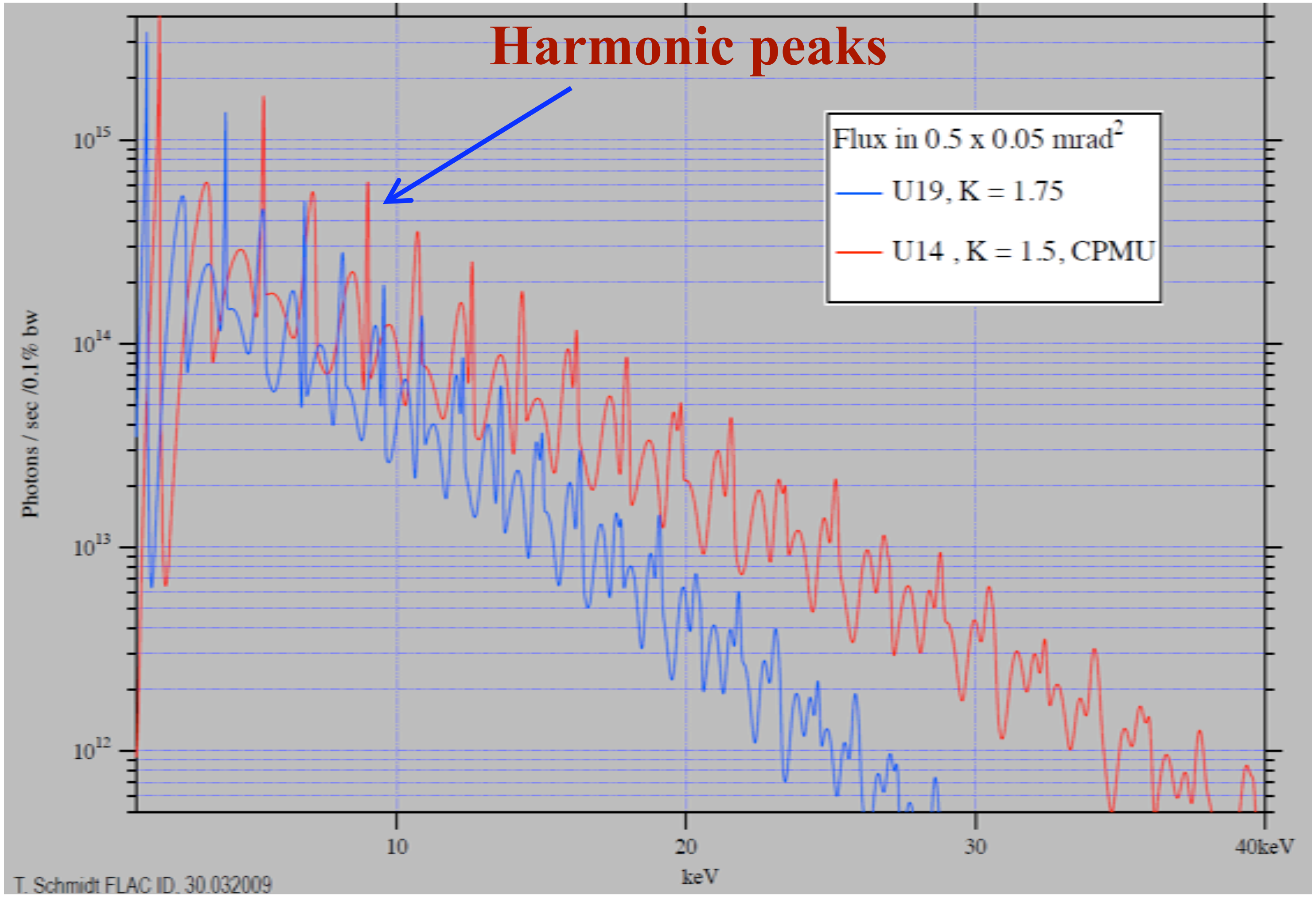
because the X-rays are emitted in ~ the same direction

U14: High magnetic field (cryocooled), very short period *1* of ***4 mm*** between magnets : **installed 2010 at MS-Powder**



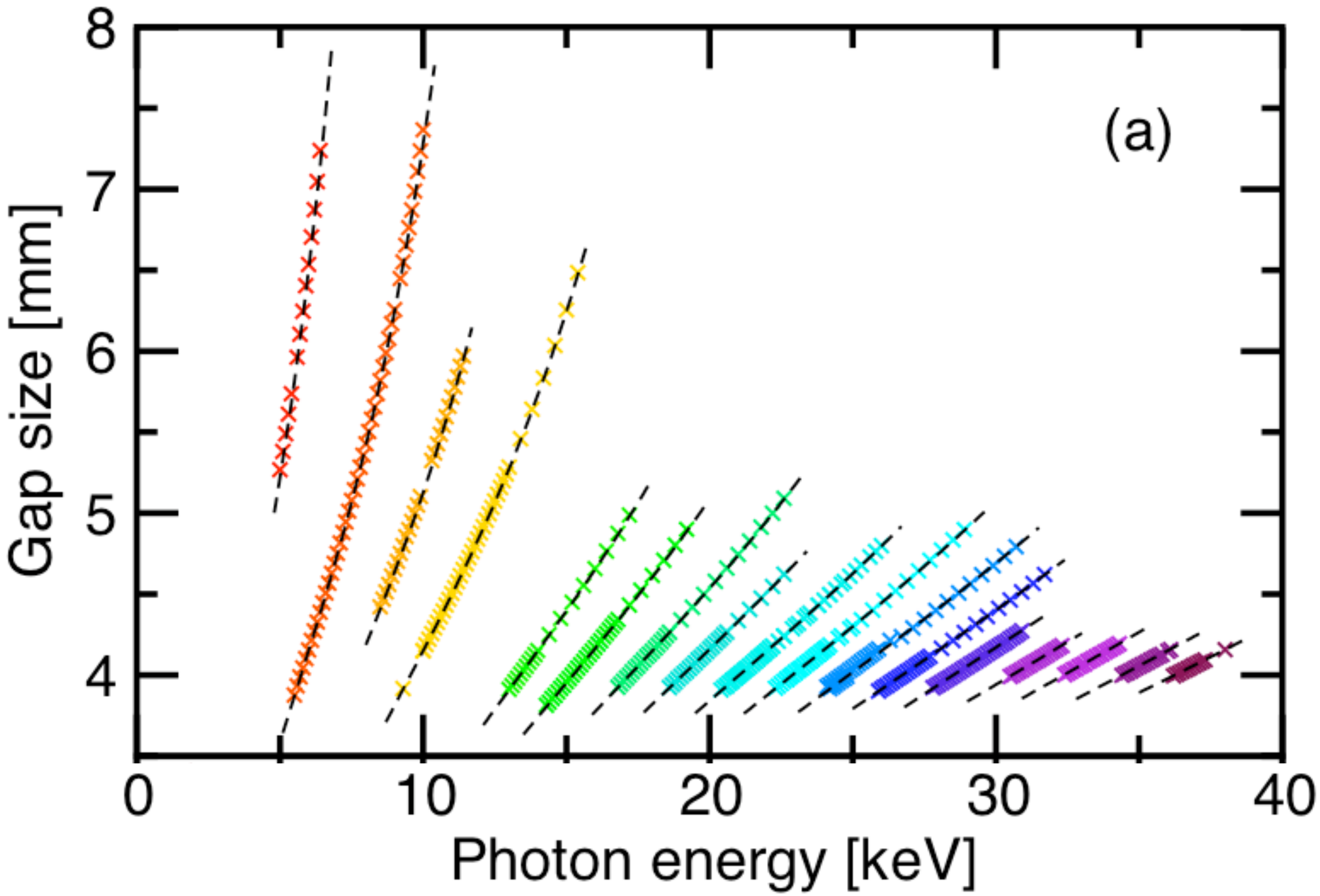
U14 undulator @ MS 2010:

Advantage on older undulators: more flux at high E



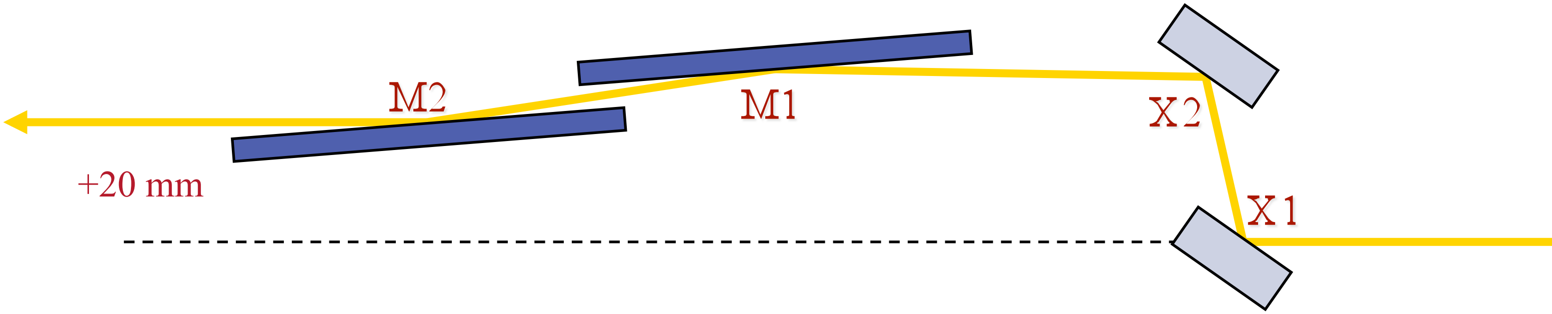
Advantage on former wiggler: especially more brilliance!

Continuous spectrum: we **adjust the gap** and select the right **harmonic**



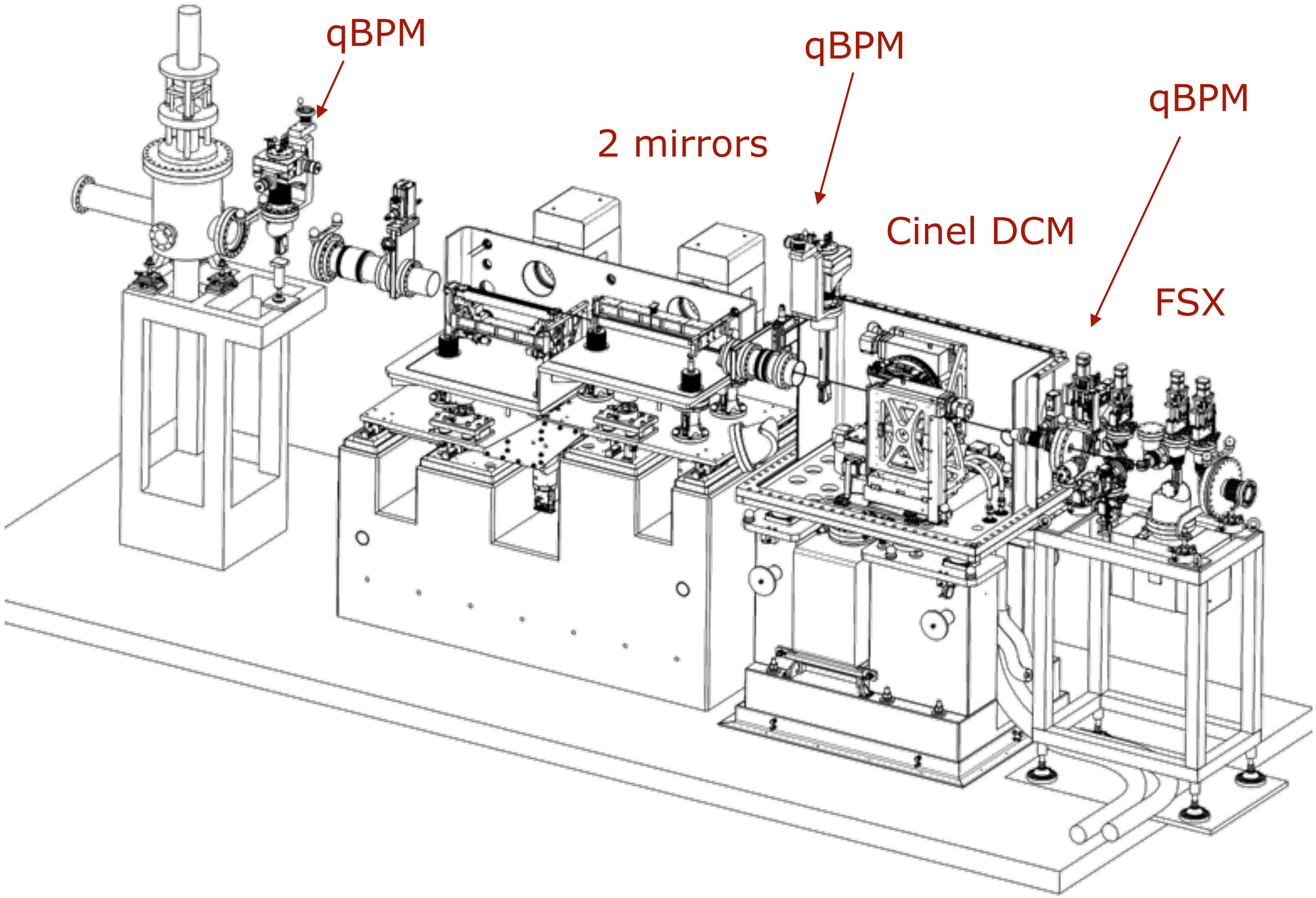
Plot of measured peak position vs. E and gap size for several harmonics

Undulator optics @ MS

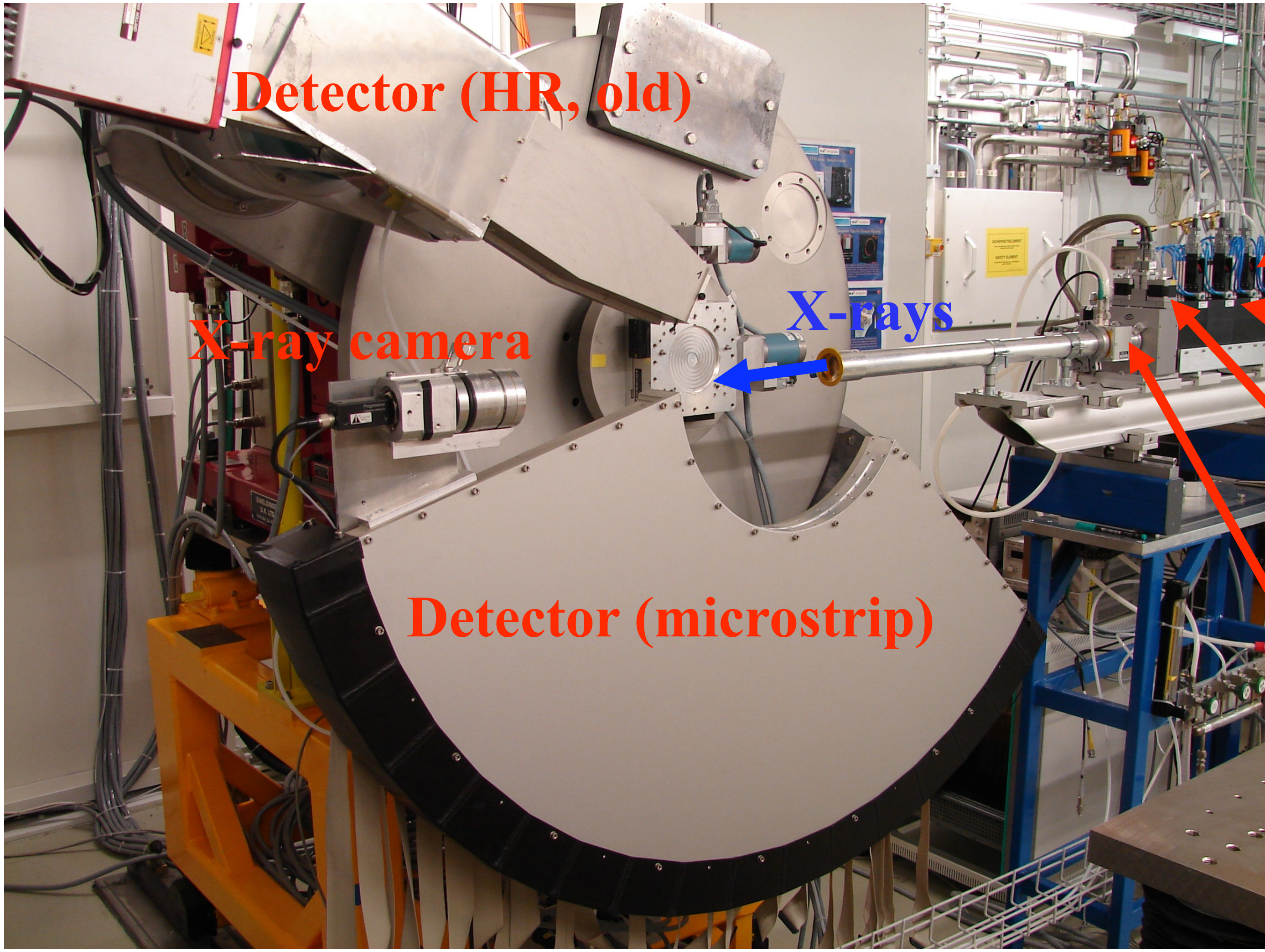


- DCM-mirror-mirror, **fixed exit** (+20 mm)
- Mirrors @ 85% α_{crit}; Si-, Rh-, Pt-coatings
  - 400 mm long, < 0.5 μrad slope error
- X1 Liquid nitrogen-cooled
  - Rotates only, fixed position
- X2 Translates horizontally and vertically with energy
  - Sagittal focussing

New optics @ MS



The Materials Science Beamline at SLS : Powder Station



Slits I

Filters

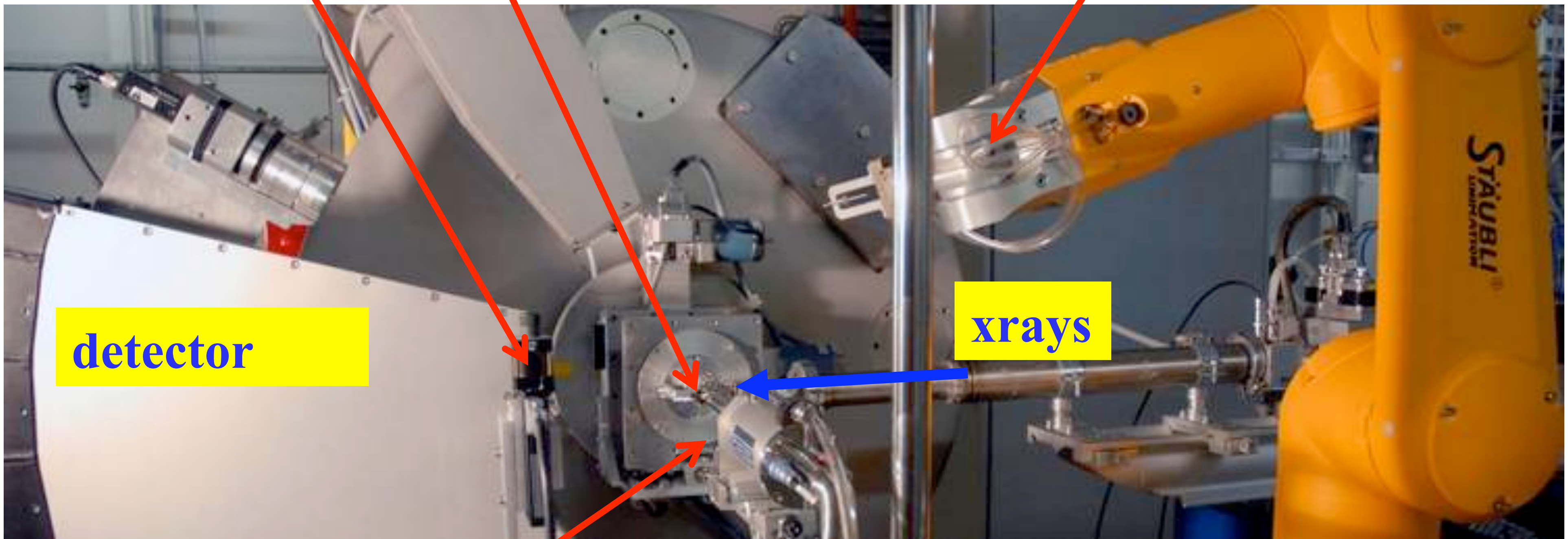
Slits II

Monitor

Sample

Sample changer arm

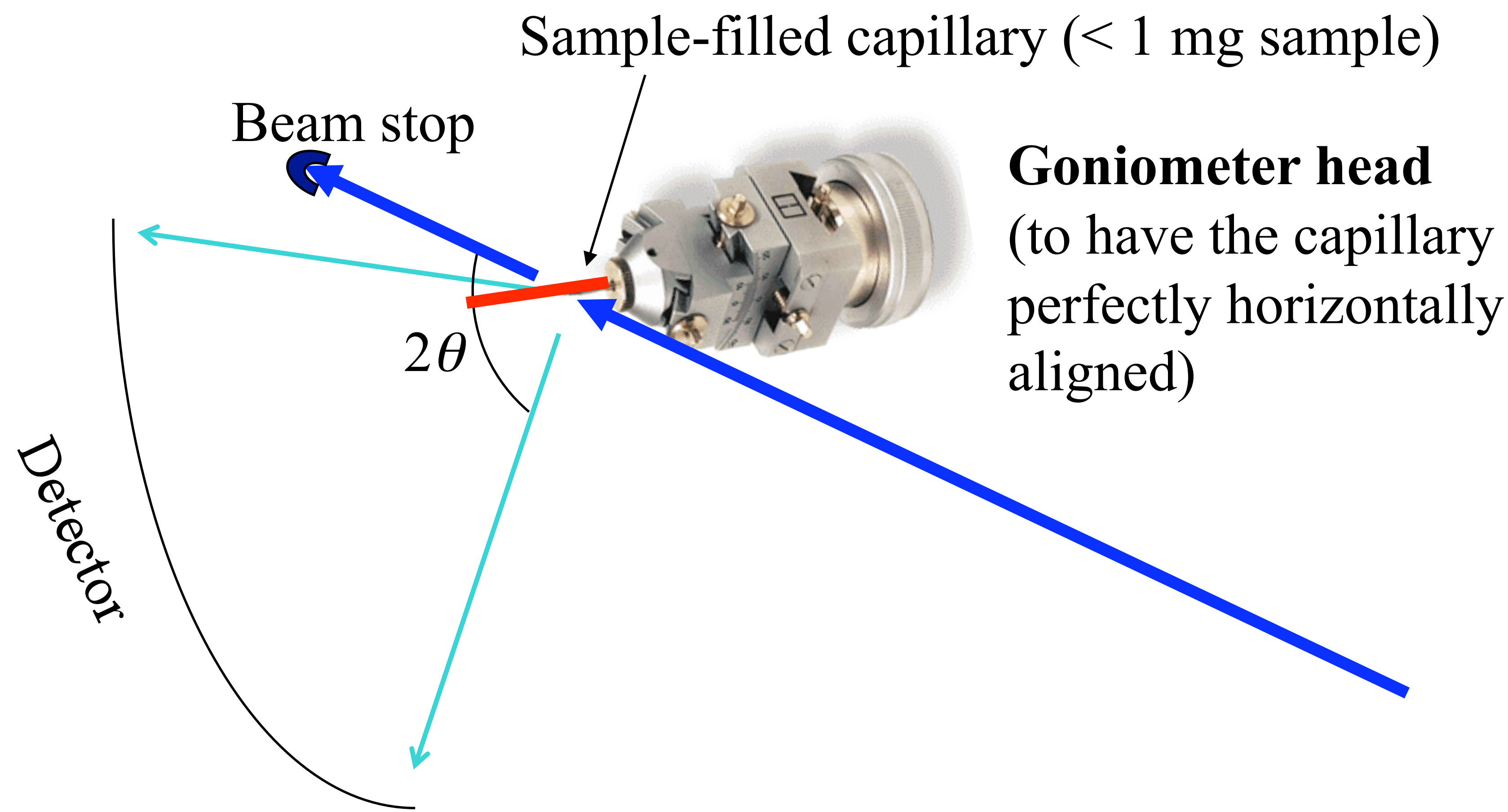
Beam stop



Sample environment (here: N_2 jet, 90 K … 500 K)

Sample : within glass capillary (0.1 … 1 mm diam.)

We measure the scattered xrays as a function of the angle 2θ



Xray beam **at the station**:

-parallel

residual divergence (mono, Si 111 Darwin width) : FWHM ~ 35 μrad = 0.0018 deg at 10 keV, ~ 18 μrad = 0.0010 deg at 30 keV

Source: 34 μrad = 0.0018 deg

Measured: **less…** [mirrors act as slits, sharp U14 resonances]

-monochromatic, continuously **tunable*** between 5 keV and 40 keV ± 2 eV; wavelength: **λ** [Å] **= 12.398 / E**[keV] = 0.31 … 2.48 Å

-brilliant … up to ~ 10 mW/mm^2 = 10^{13} ph/mm^2/s [unfocused]

-small … ~ 5 mm hor. x 0.7 mm vert. (FWHM)

-focussable … down to 200 μm x 30 μm (FWHM)

-fully polarized in the horizontal plane

Microstrip detector Mythen II

Photon-counting

Made of silicon (0.3mm to 1 mm thick)

Each X-ray ends up ionizing Si –> $Si^{+} + e^{-}$: cost 3.6 eV

n. of e^{-} produced = X-ray E / 3.6 eV

Charge pulse –> tension pulse –> digital counter

Thresholding (skip pulses below a certain voltage):

- rise halfwidth ~ 1.2 keV
- avoid crosstalk between pixels (thresholding > 50% beam energy)
- **eliminate fluorescence:** inelastic scattering can be cut off to <0.1% if it is at least **6 keV lower** in energy than the main beam; the elastic scattering (and Compton) are not attenuated.

Dead time ~ 200 ns, max rate ~ 3 MHz, dyn. range $1.6*10^{7}$ (24 bit)

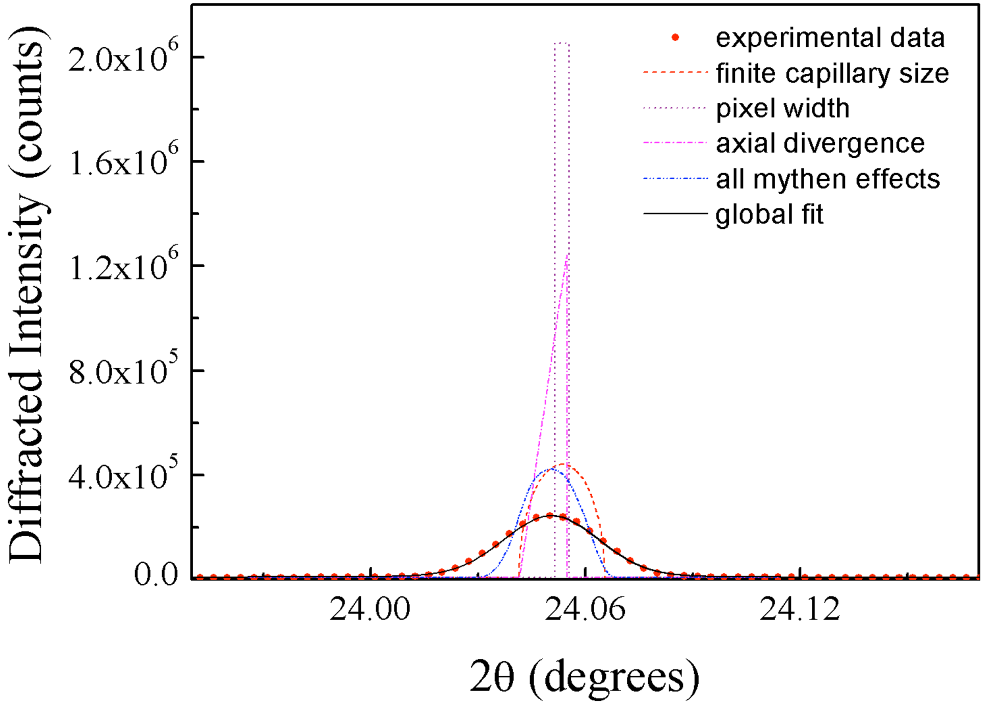
Min. acquisition time ~ 0.1 ms, lag time between acquisitions < 0.1 ms

Efficient, fast, simultaneous covering of 120 deg with 0.0037 deg resolution (> 30000 separate channels)

Accuracy (after flat-field & dead time correction) : ~ 0.01 %

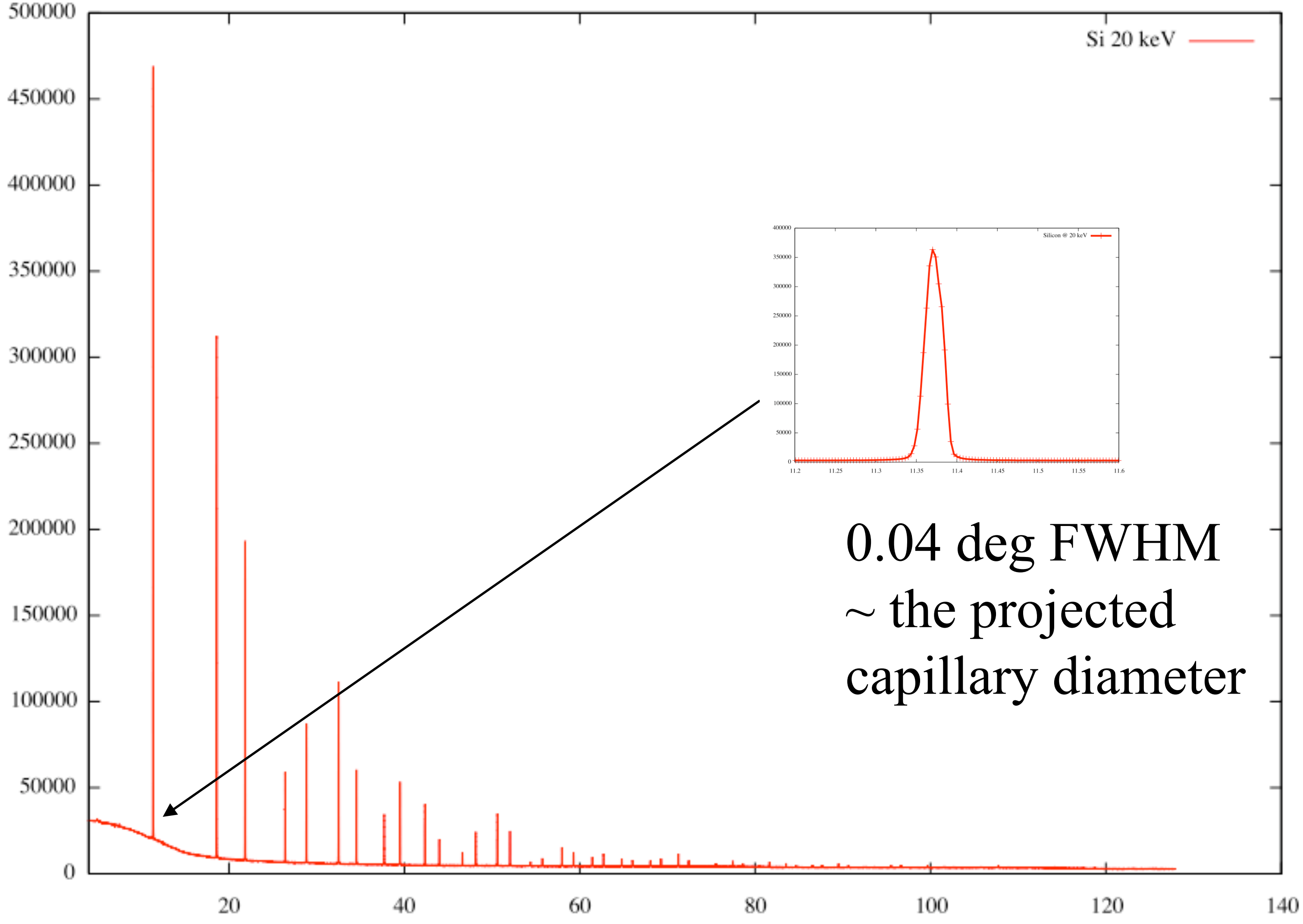
Instrumental Resolution Function

The Mythen detector has no analysis capability. The main contribution to the IRF becomes the projected cross section of the space region occupied by the sample! However, this is a well-known function. Other contributions have been parametrized as well.



J. Appl. Cryst. **39** (2006) 347-357
J. Synchrotron Rad. **17** (2010) 653-668
Z. Kristallogr. **225** (2010) 616-624

Peak profile with 0.5 mm capillary (NIST standard silicon powder, 111 peak, 20 keV)



Sample conditioning :

Temperature (several devices): we can set T from 4 K to 1800 K

Pressure: DAC, several Gpa

E field, B field, …

Sapphire capillary: pressure < 200 atm, T<1000 C, liquids/gases interacting… (soon: gas loading up to ~ 200 bar)

Tensile machine (for metal samples)

In situ experiments :

Fast acquisitions while **something** happens to the sample

High quality ~ 1 s, medium-low quality ~ 1 ms

Powder x-ray diffraction: gaining information

- Solving crystal structures
  - Polymorphism
  - Phase transitions
- Beyond crystal structure: **Total scattering**
  - Microstructure, size/shape distributions, QPA
  - Also non-crystalline matter
  - Interatomic vectors - first coordination shells

Silicon is one of the most abundant elements on earth

?

Silicon is the most abundant element in the MS beamline!

- monochromator crystals
- mirrors
- attenuators
- standard sample
- detector
- electronics and computer CPUs

Thanks to the MS upgrade crew: P. Willmott, S. Leake, N. Casati, F. Gozzo, M. Lange, D. Meister, W. Xiaoquiang, A. Bergamaschi, T. Schmidt, M. Calvi, J. Krempasky, …

Thanks for your attention!