WAVELENGTH DISPERSIVE SPECTROMETERS: LARGE-SCALE-FACILITY APPLICATIONS

http://www.xraylab.fi/

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WIDE RANGE OF APPLICATIONS

Compton spectroscopy — a few/several eV

hi-res x-ray emission spetroscopy — (sub-)eV

resonant inelastic x-ray scattering, crystal field and magnetic excitations — <100 meV

phonons in crystals, molecular vibrations — ~1 meV

vibrational dynamics in disordered systems — ~0.1 meV

Ultra-high-resolution spectrographs — <100 μeV

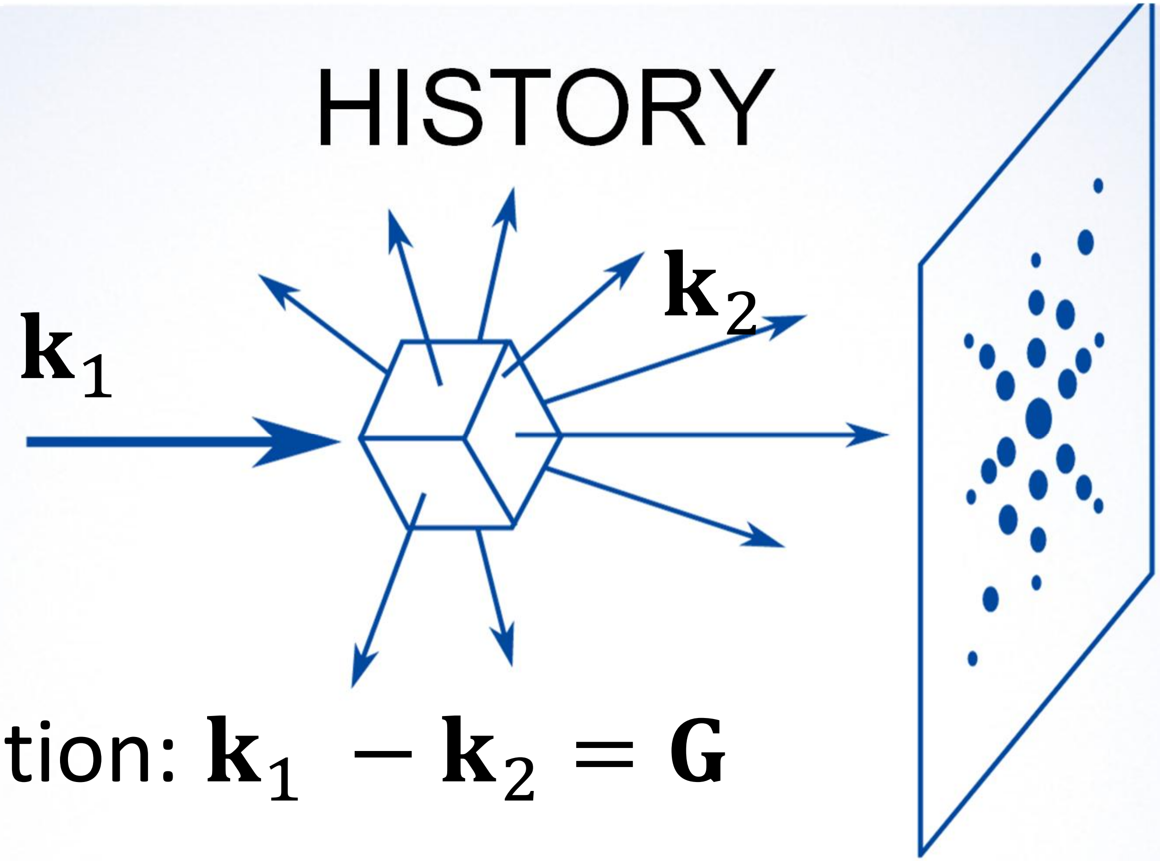
Yu. Shvyd'ko, S. Stoupin, K. Mundboth, and J-H. Kim, Phys. Rev. A 87 (2013) 043835

Need of a eV to sub-eV energy resolution for the emitted/scattered radiation

No x-ray detector capable of that

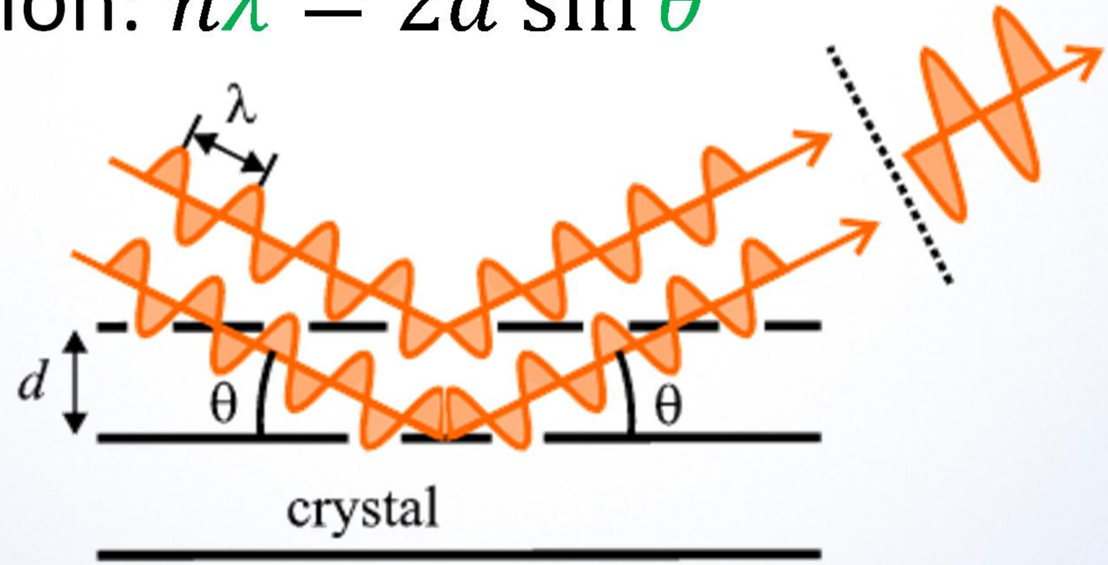
$\Rightarrow$ crystal x-ray optics

HISTORY



Laue equation: $\mathbf{k}_1 - \mathbf{k}_2 = \mathbf{G}$

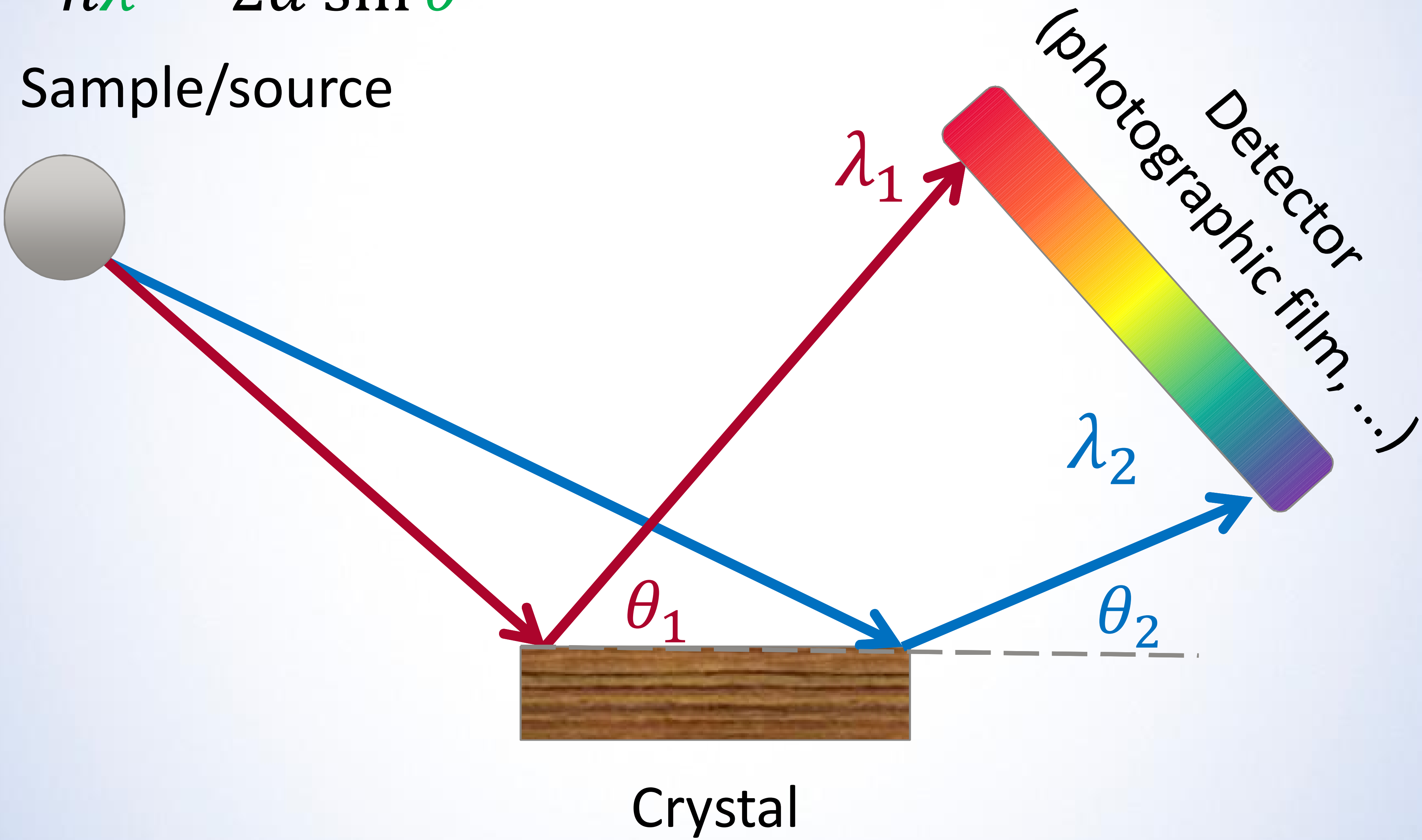
Bragg equation: $n\lambda = 2d \sin\theta$



EARLY SPECTROGRAPHS (1920-)

Bragg, Seeman, ...

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

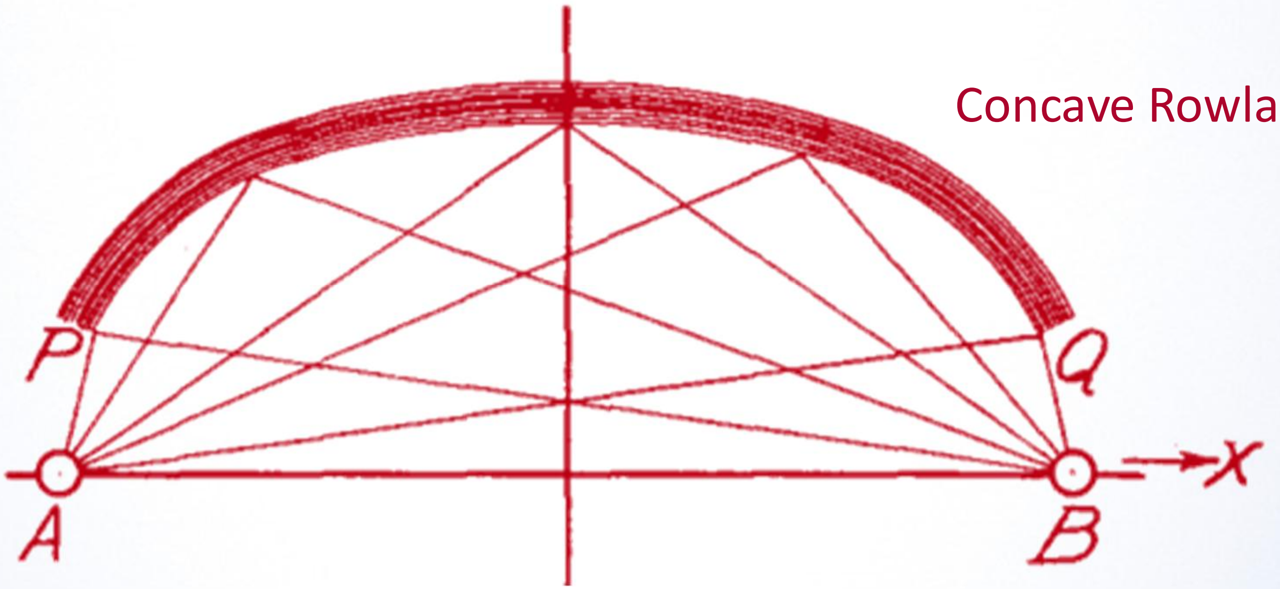


THE MULTIPLE CRYSTAL X-RAY SPECTROGRAPH

By Jesse W. M. DuMond and Harry A. Kirkpatrick

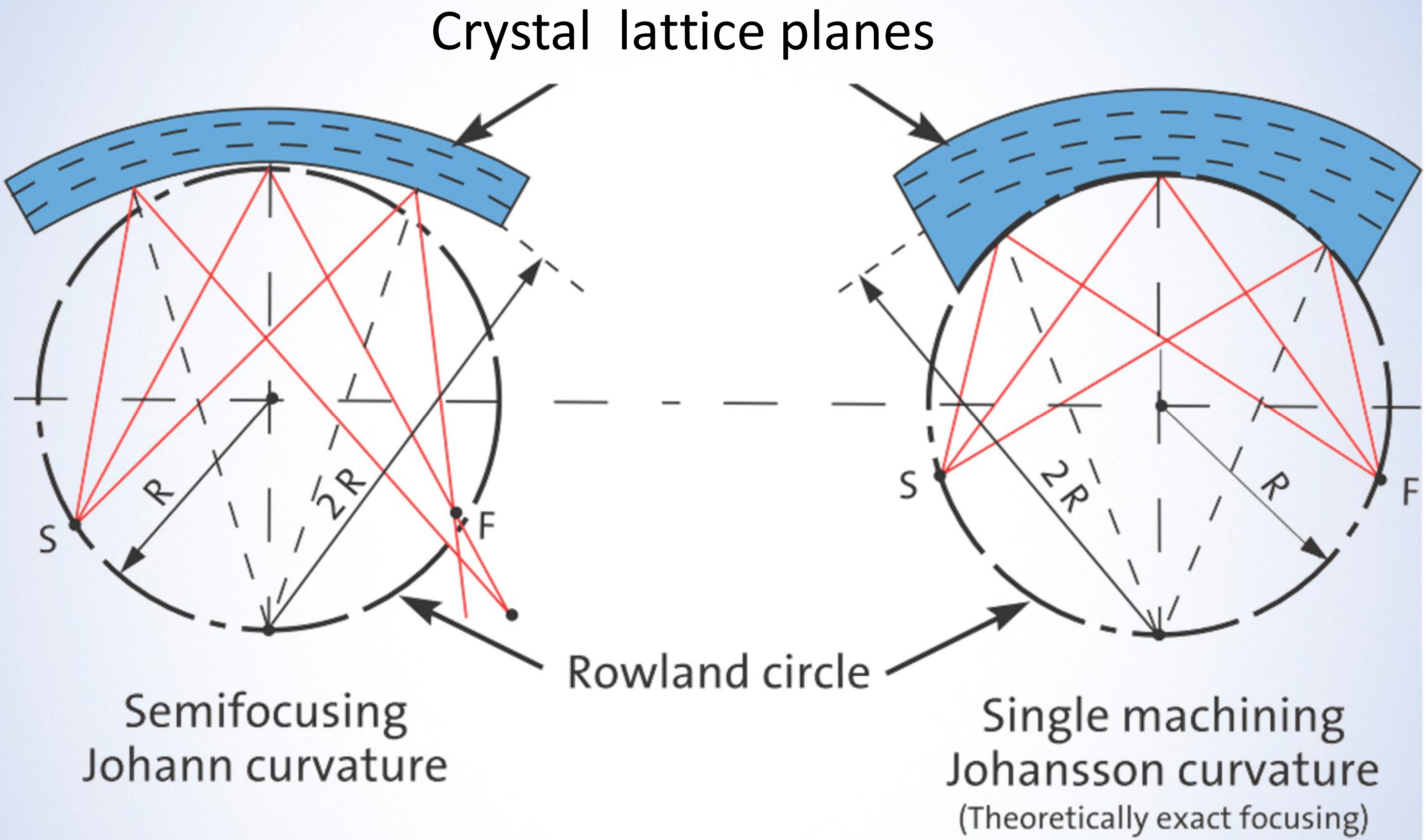
ABSTRACT

The need for improvements in scattered x-ray spectroscopic technique along the lines of increased intensity and contrast is discussed and a new instrument composed of fifty small units, each a Seeman Spectrograph in itself cooperating to form a single spectrogram, is described in detail. The technique of adjusting the instrument is also described.



Concave Rowland grating

DuMond & Kirkpatrick, Review of Scientific Instruments 1, 88 (1930), doi: 10.1063/1.1748677



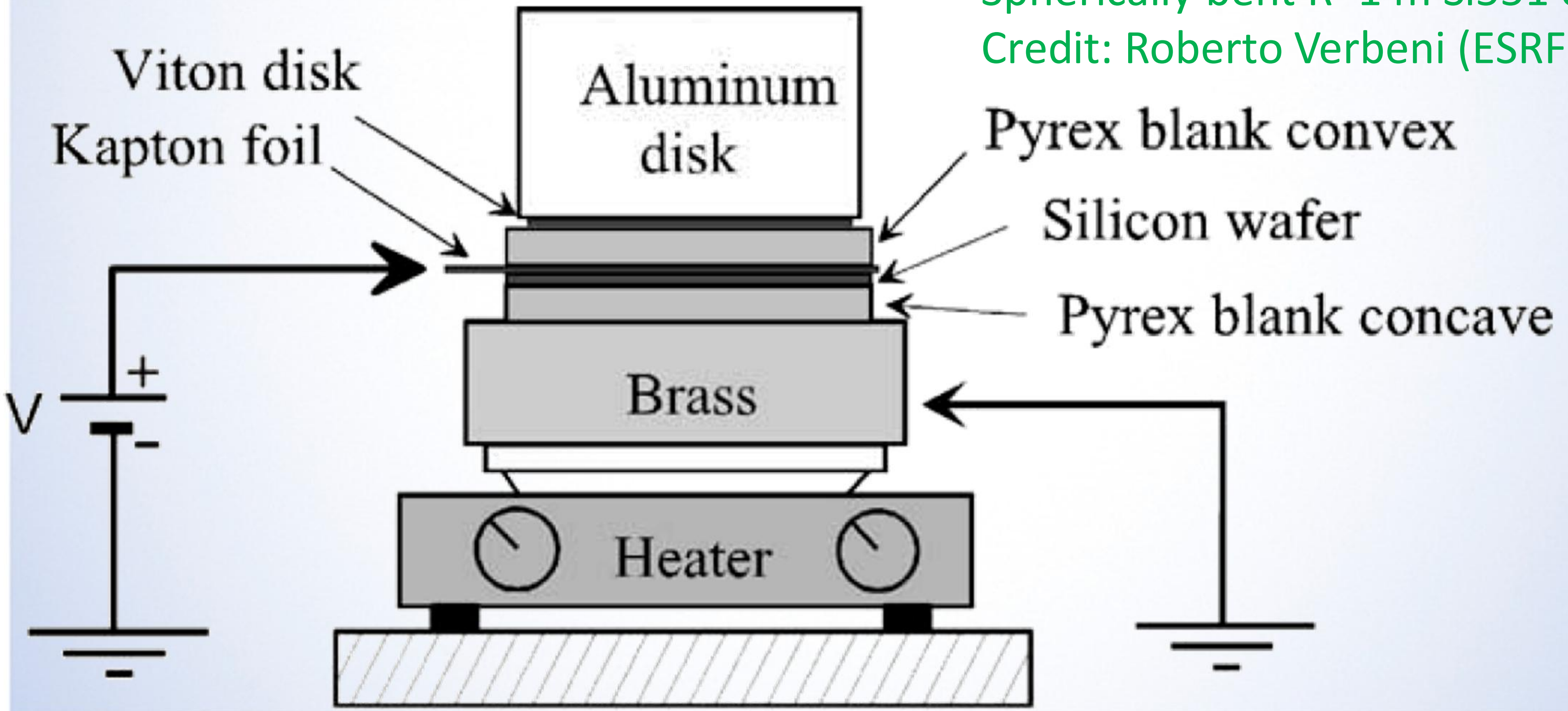
POINT-BY-POINT SCANNING OF SPECTRA

Figure courtesy of Saint-Gobain crystals

ANODIC BONDING TO A CURVED GLASS SUBSTRATE

Verbeni et al. Journal of Physics and Chemistry of Solids 66 (2005) 2299–2305

Spherically bent R=1 m Si551 crystal
Credit: Roberto Verbeni (ESRF)



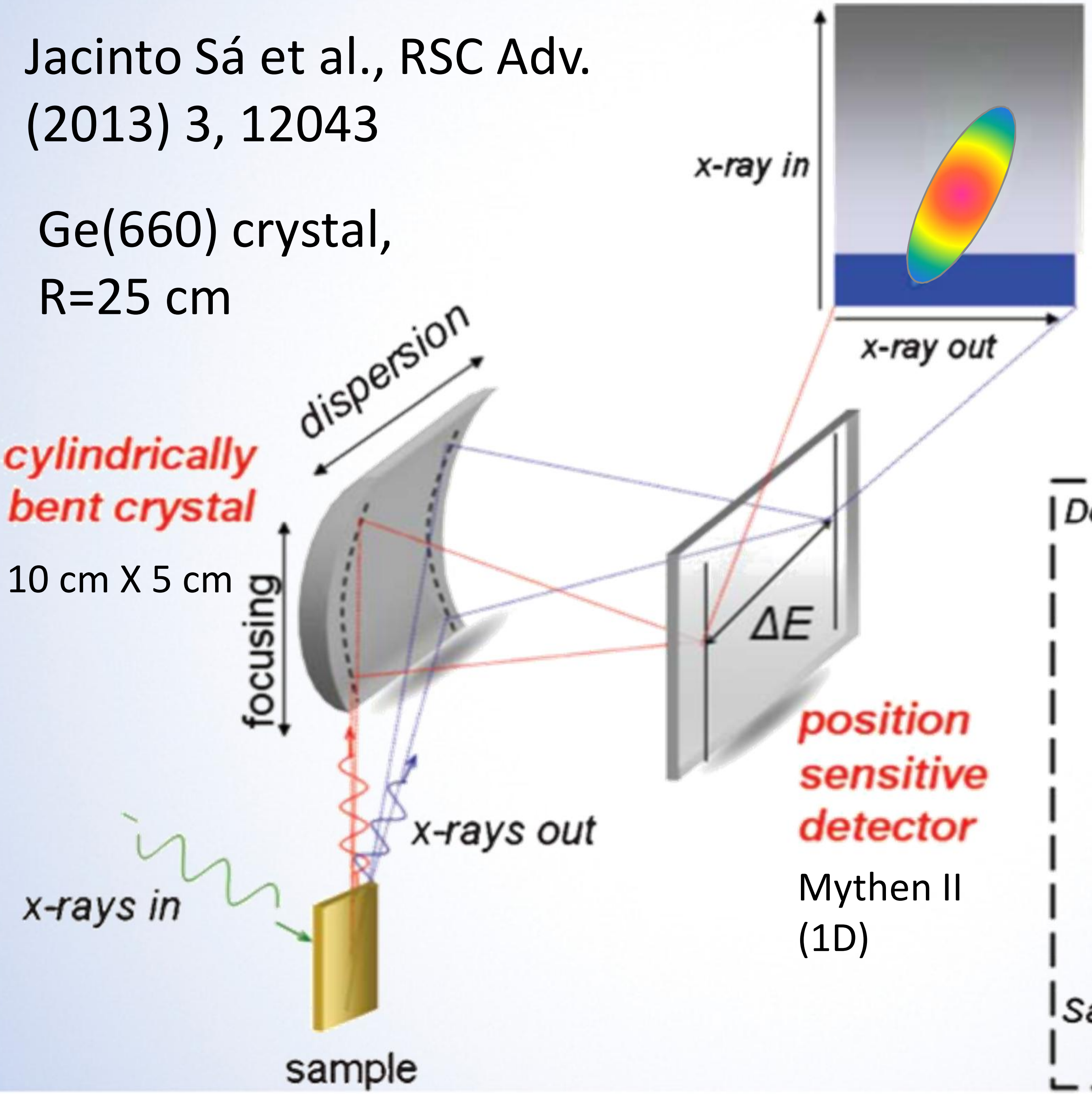
VON HAMOS GEOMETRY

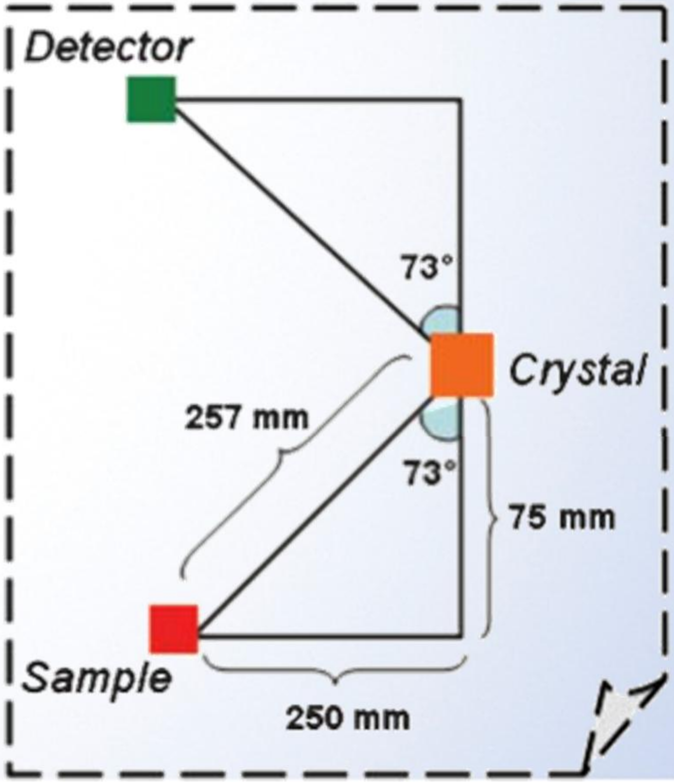
Jacinto Sá et al., RSC Adv. (2013) 3, 12043

Ge(660) crystal,
R=25 cm

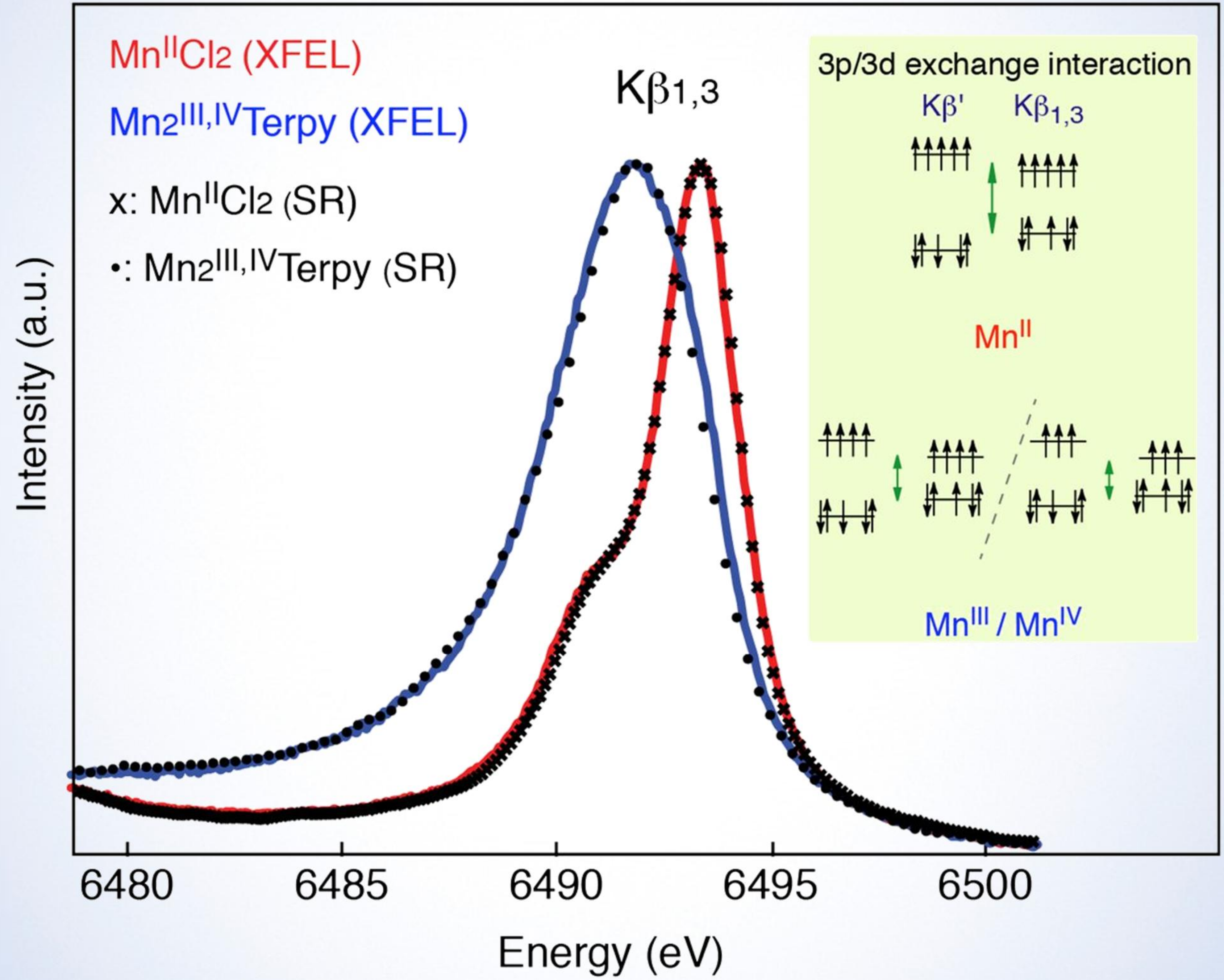
Example:
SuperXAS @ SLS

Energy resolution
~1 eV @ 10 keV





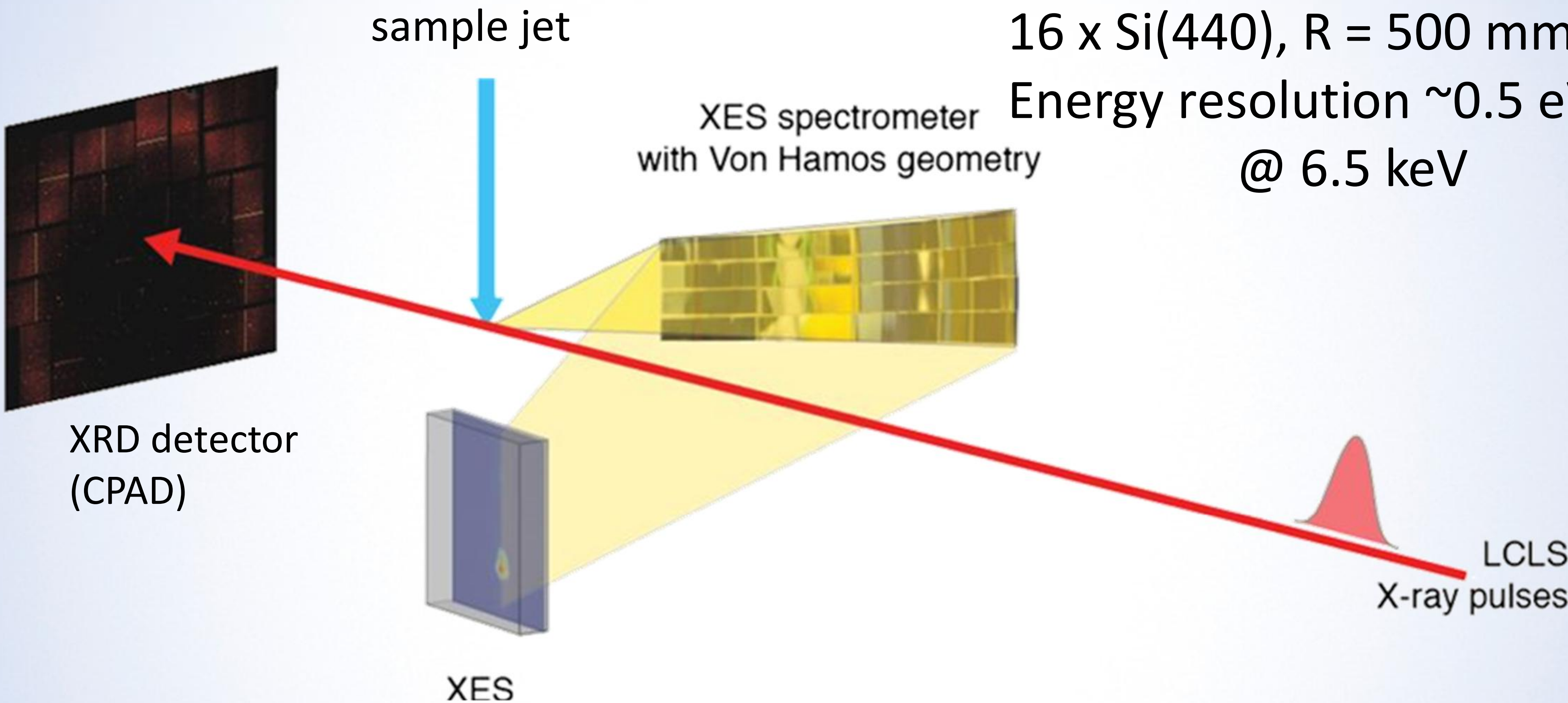
CXI @ LCLS   von Hamos



Roberto Alonso-Mori et al. PNAS 2012;109:47:19103-19107

VON HAMOS @ LCLS (CXI)

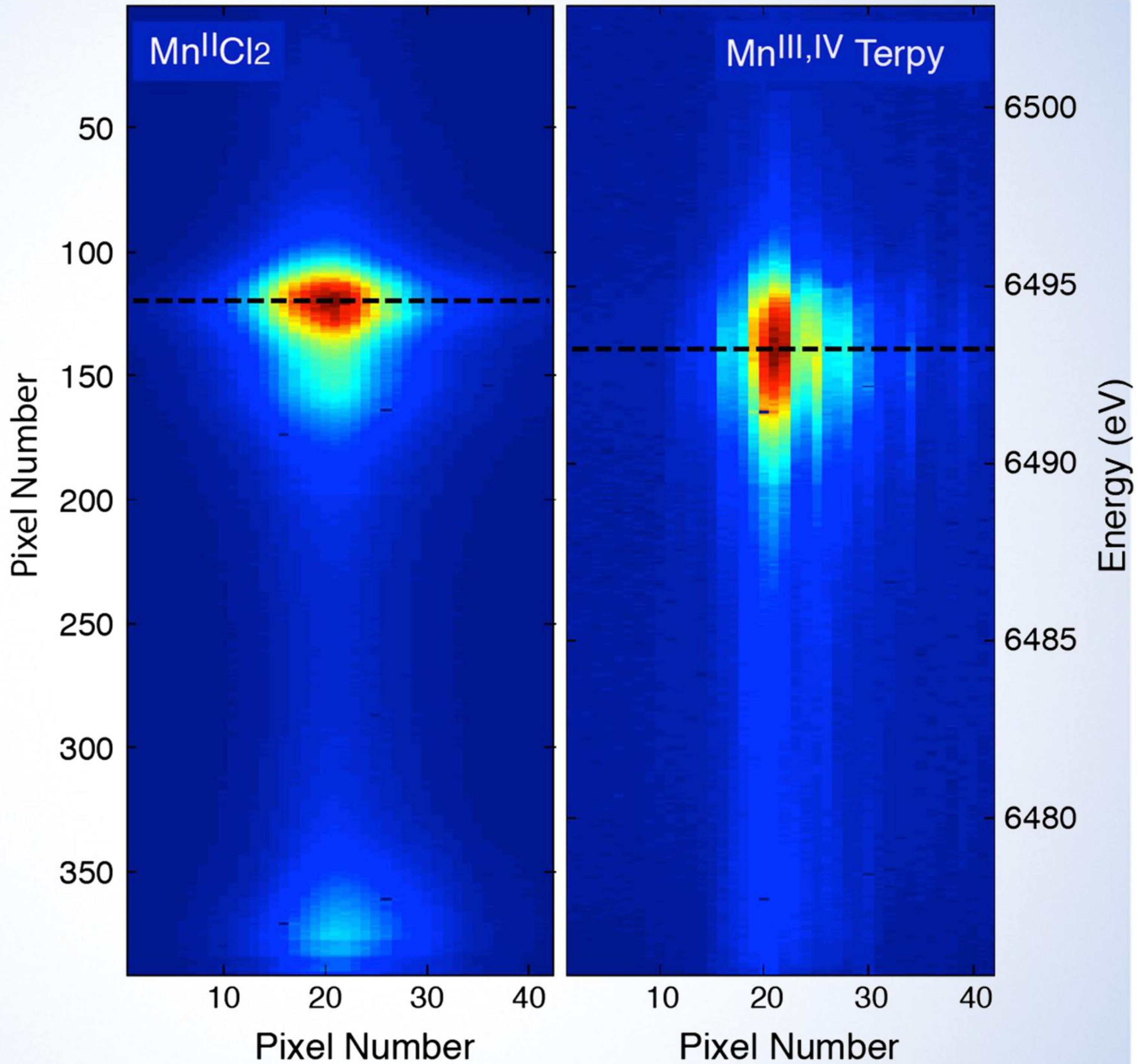
16 x Si(440), R = 500 mm
Energy resolution ~0.5 eV
@ 6.5 keV



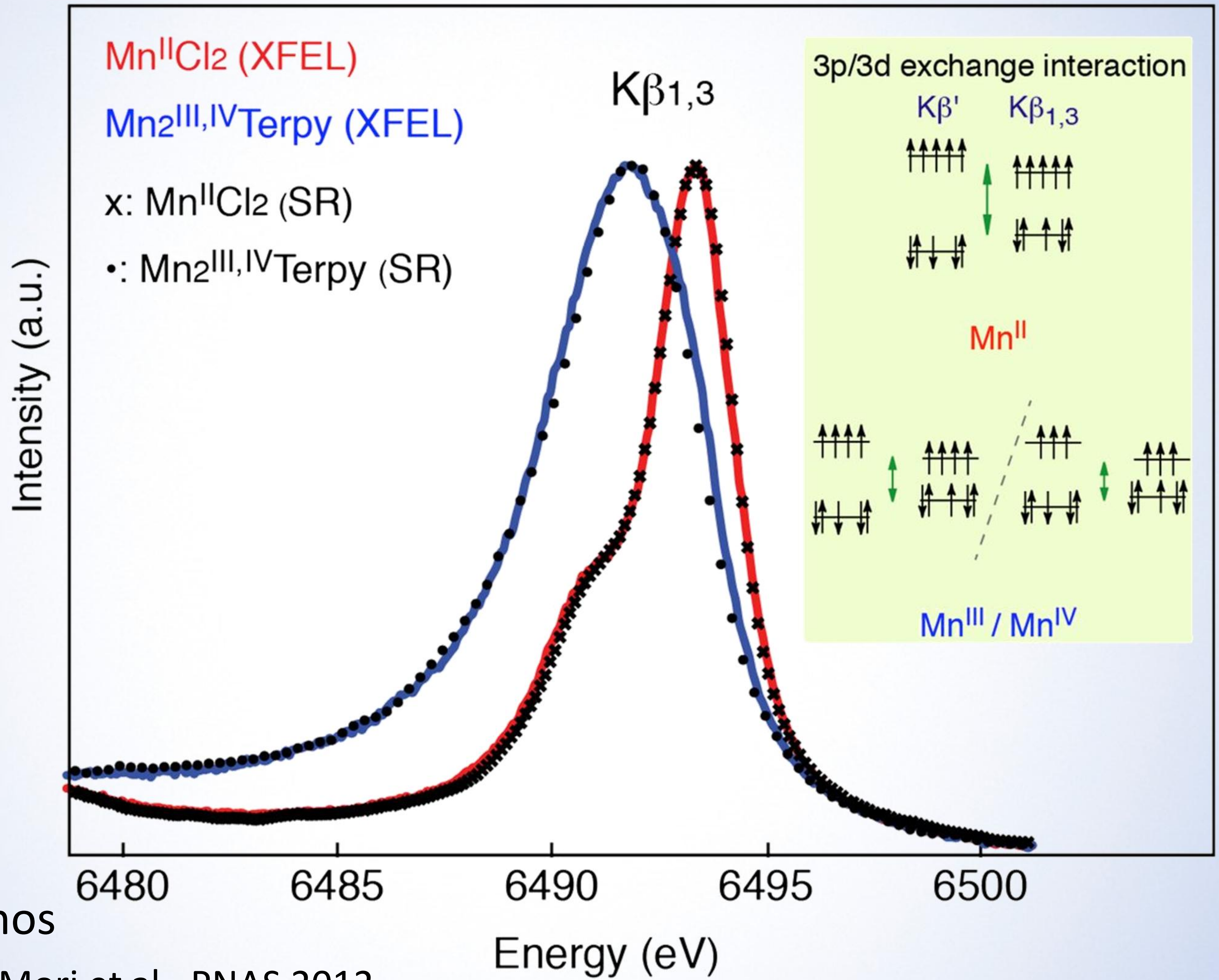
Cornell-SLAC Pixel Array Detector module composed of four 185 × 194-pixel CMOS ASICs (pixel size is 110 × 110 μm²) bump-bonded to high-resistivity silicon diodes

Roberto Alonso-Mori et al. PNAS 2012;109:47:19103-19107

Cornell-SLAC Pixel Array Detector module composed of four 185 × 194-pixel CMOS ASICs (pixel size is 110 × 110 μm^2) bump-bonded to high-resistivity silicon diodes



Roberto Alonso-Mori et al. PNAS 2012;109:47:19103-19107 Data from CXI @ LCLS



LCLS
von Hamos

R. Alonso Mori et al., PNAS 2012

VON HAMOS AT EUROPEAN XFEL

FXE instrument / Christian Bressler et al.

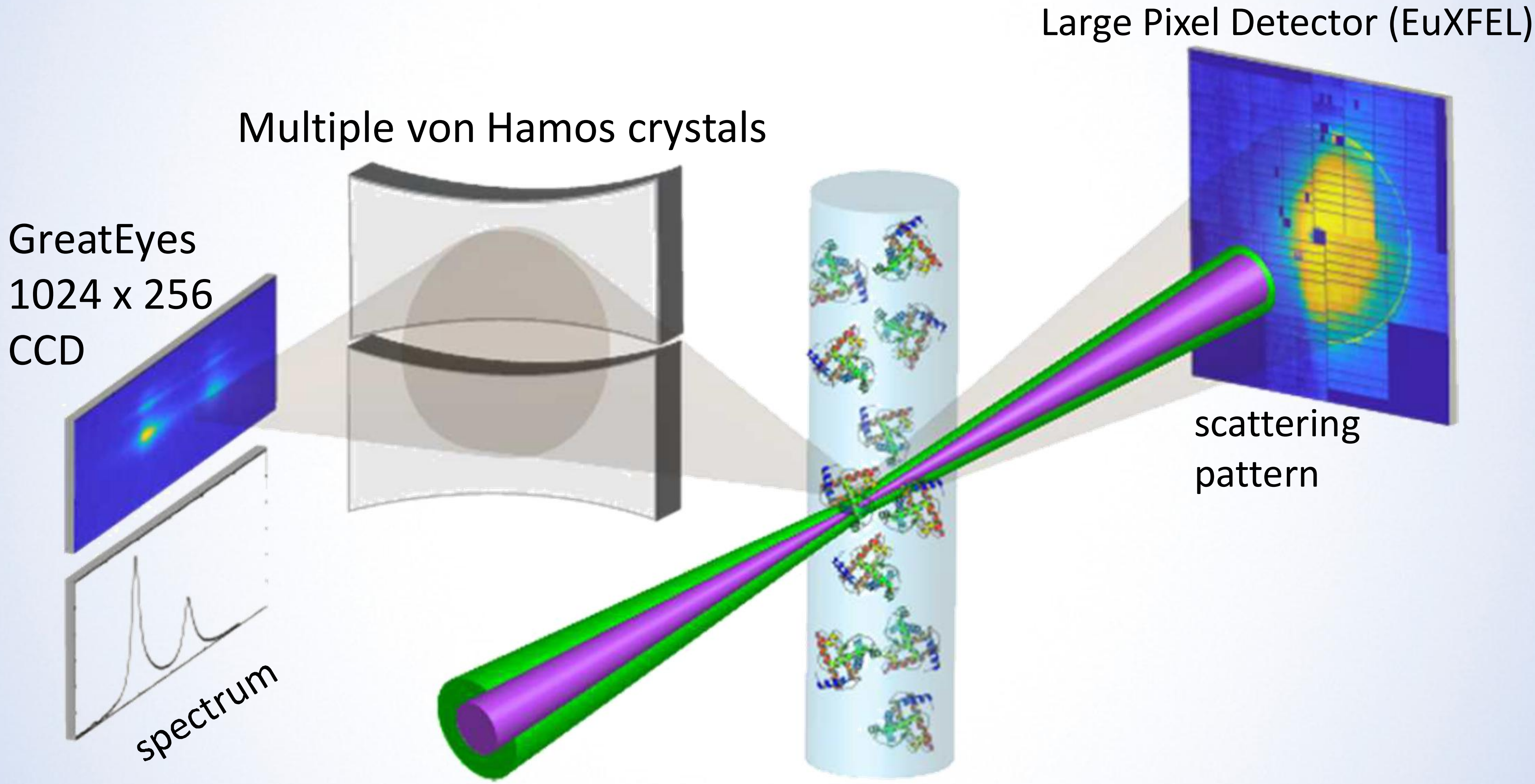
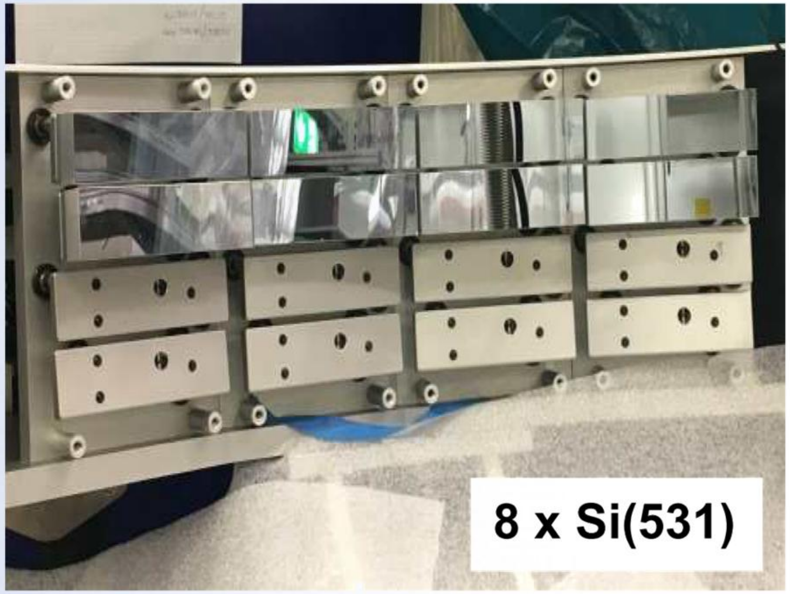


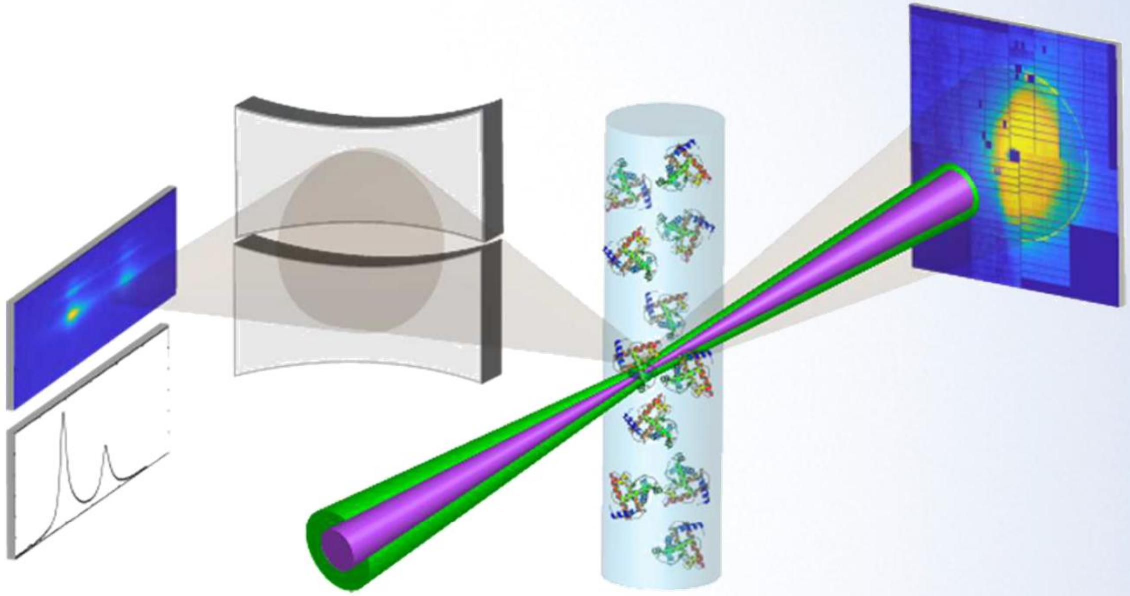
Figure courtesy of Christian Bressler, European XFEL, shared for this occasion

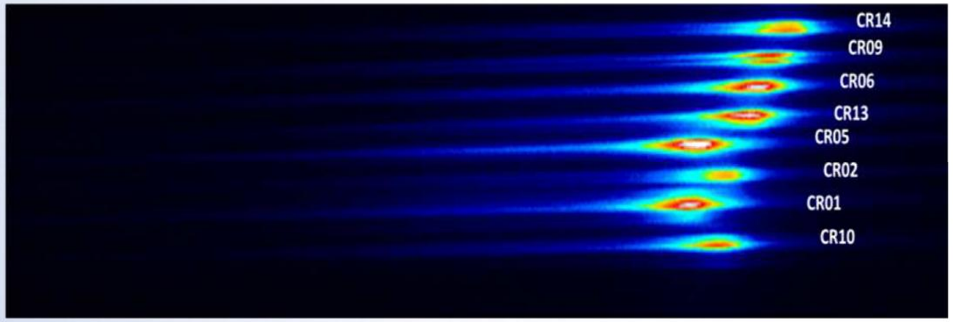
VON HAMOS AT EUROPEAN XFEL

FXE instrument / Christian Bressler et al.

30 bunches, 9.3 keV, ~100uJ/pulse,
focused to ~20 um



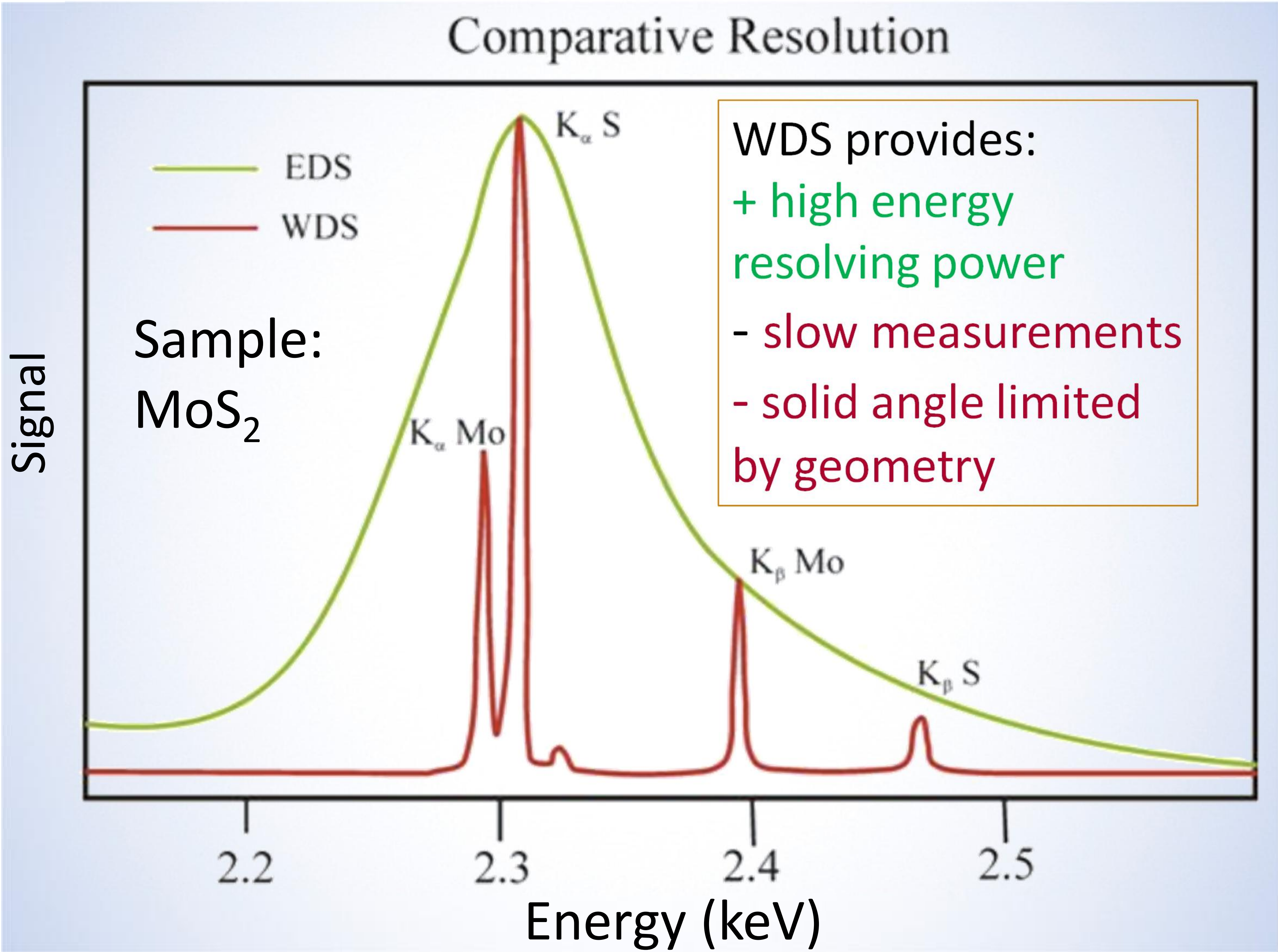




GreatEyes CCD detector

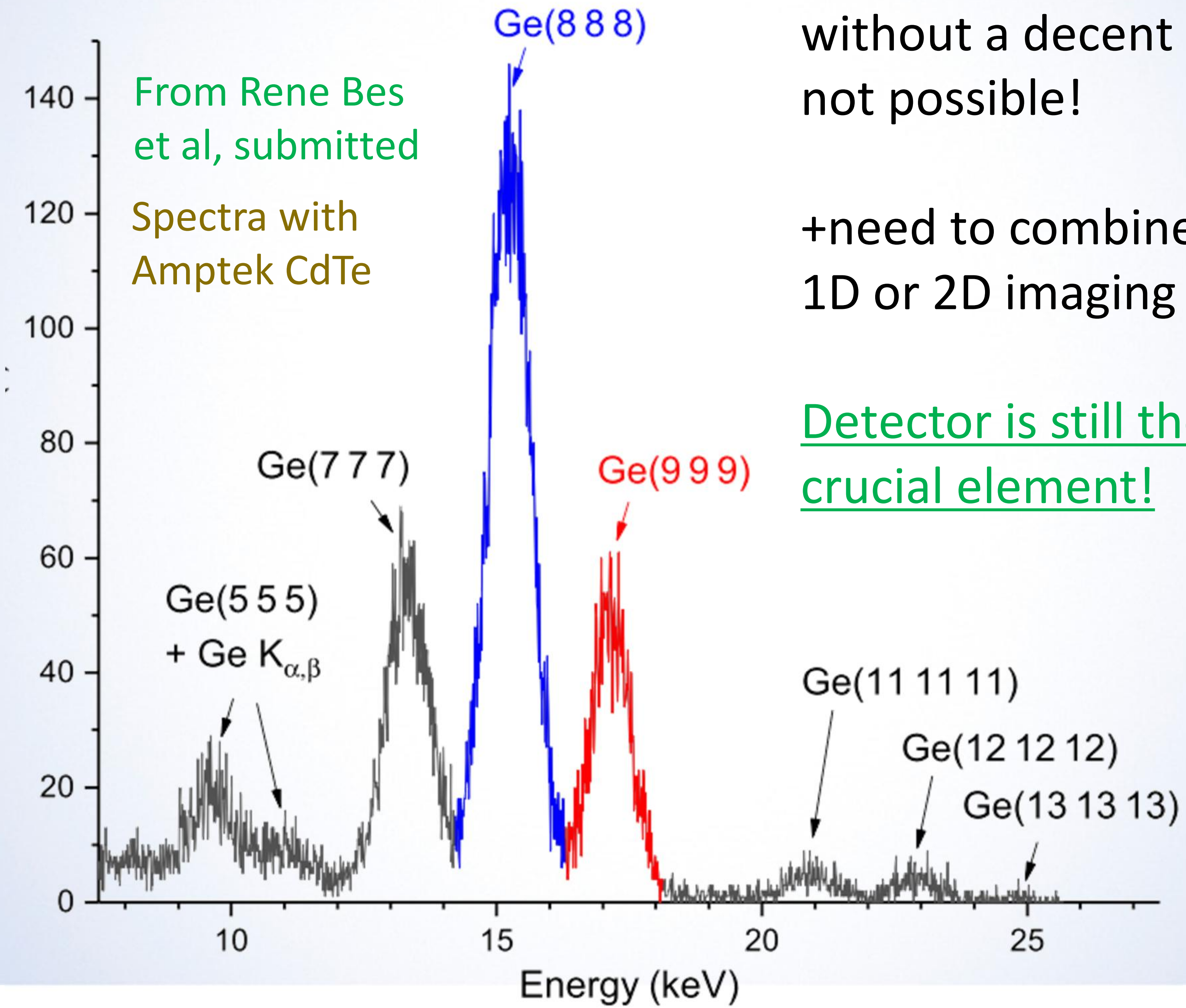
European XFEL

Slide courtesy of Christian Bressler, European XFEL, shared for this occasion



Comparative Resolution
EDS
WDS
Kα S
Kα Mo
Kβ Mo
Kβ S
Sample:
MoS2
WDS provides:
+ high energy resolving power
- slow measurements
- solid angle limited by geometry
Signal
2.2
2.3
2.4
2.5
Energy (keV)

High resolution, yes,
but how about harmonic rejection?



Picking up Ge(999) reflection from this mess without a decent EDS... not possible!

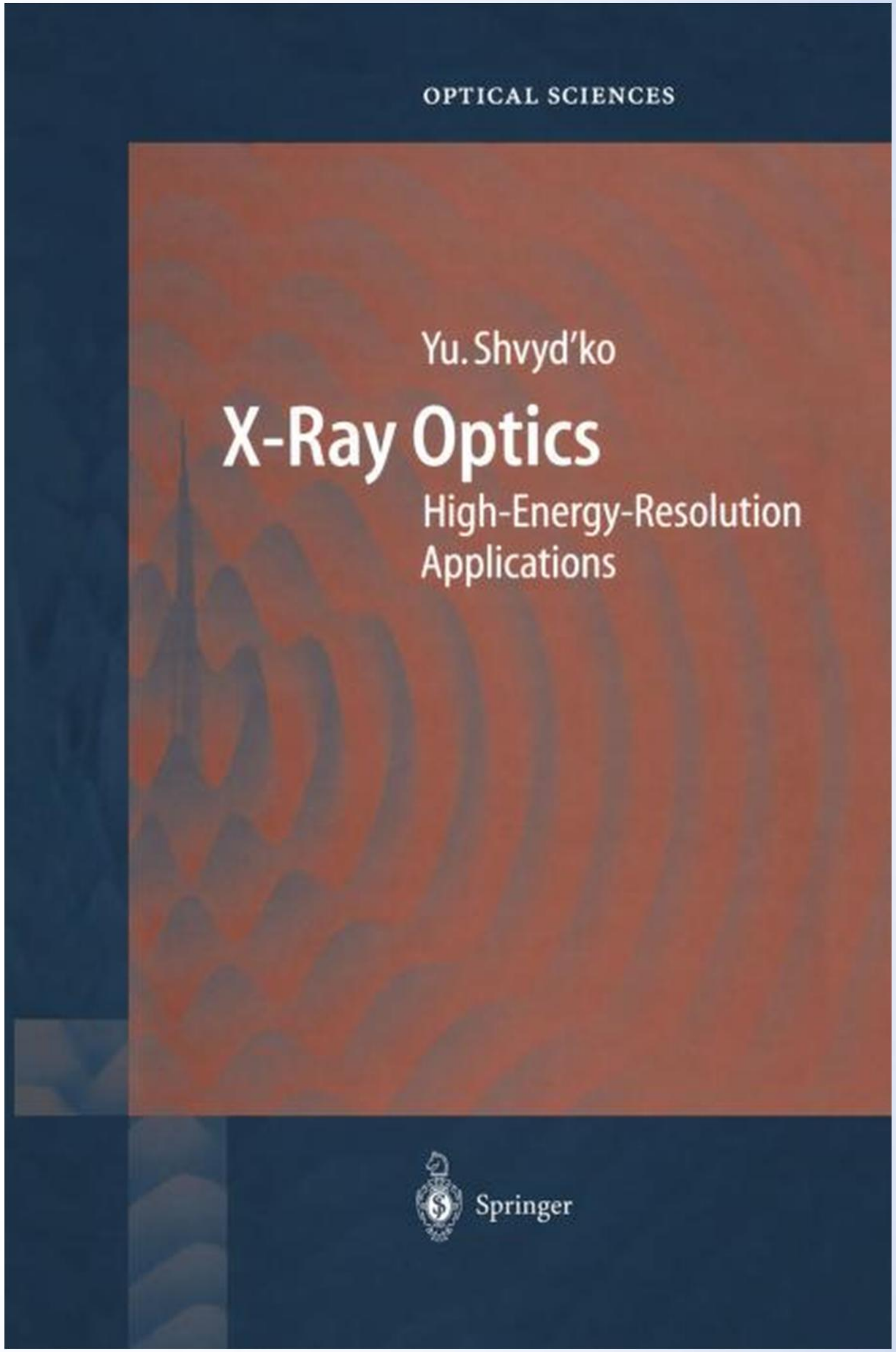
+need to combine with 1D or 2D imaging capability

<u>Detector is still the most crucial element!</u>

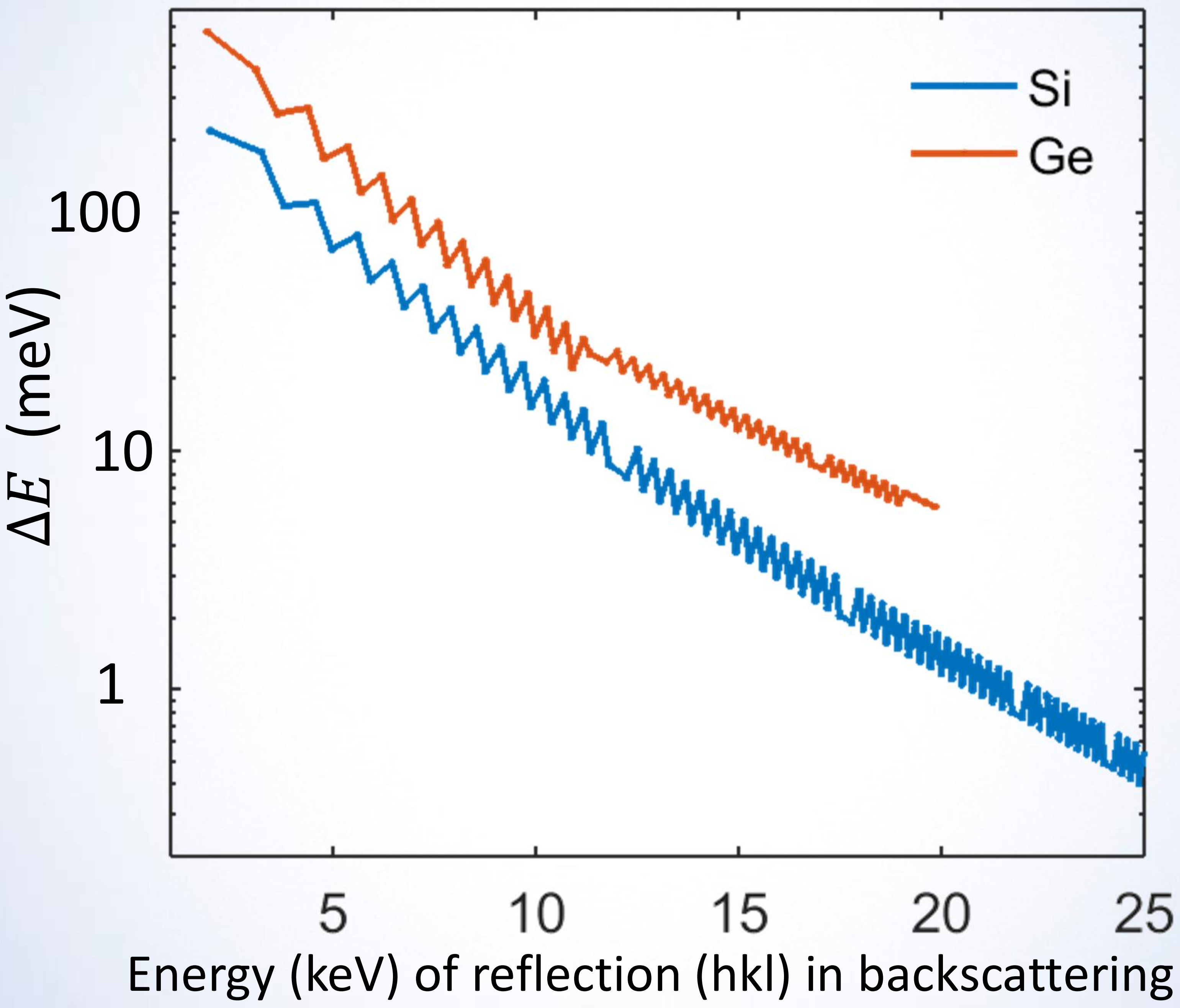
What determines the energy resolution?

- finite source size x
  $\Delta E / E = x / R \cot \theta$

- crystal properties
  - Darwin width for flat crystals
  - Bending causes elastic stresses, $\Delta E \propto d / R$

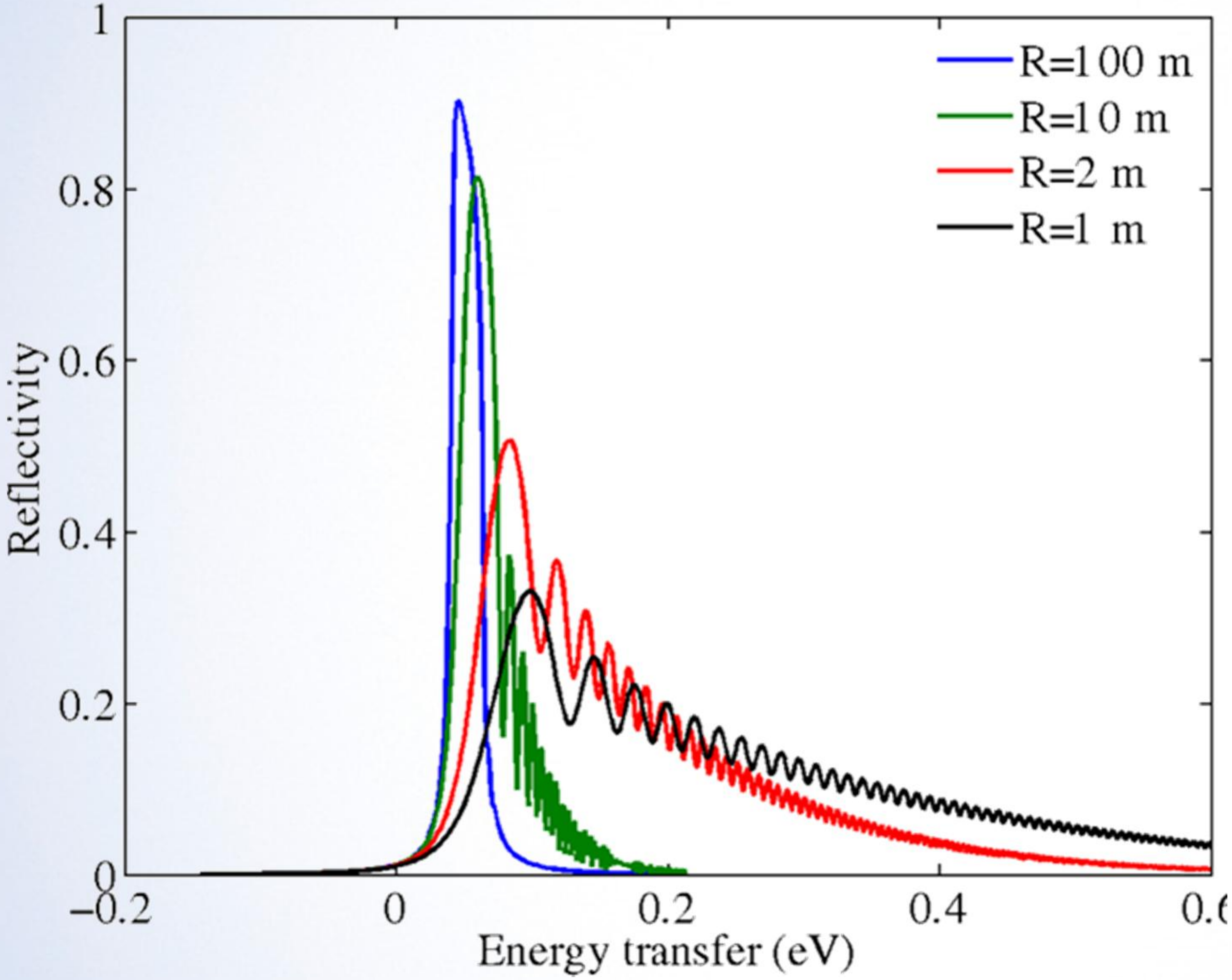


ULTIMATE ENERGY RESOLUTION / PERFECT CRYSTAL



Elastic bending ⇒ strain

Si(660) reflectivity curves near backscattering, $\theta \approx 88°$



$$\frac{\Delta E}{E} \approx \frac{d}{R}\left(\cot^2\theta - \nu\right)$$

ν = Poisson ratio

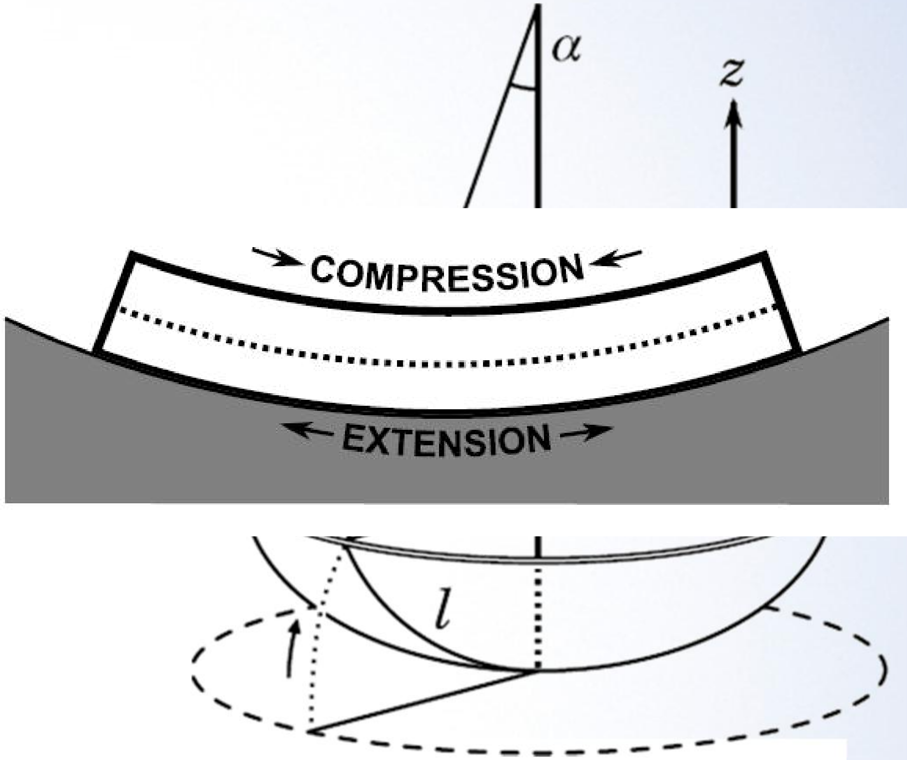
R=1 m Si spherically bent crystal, R. Verbeni (ESRF)

Taupin (1964), Takagi (1962,1969)

Implementation in XOP

- Diffraction curve for a macroscopic elastically bent crystal – both with finite area and finite thickness

- Spherical bending causes angular compression → contribution to strain proportional to $(r/R)^2$



$$\epsilon_{zz} = \epsilon^{v}_{zz}(z) + \epsilon^{h}_{zz}(x,y)$$

strain field

$$\epsilon_{zz}(r,\phi,z) = \frac{C_{31}+C_{32}}{C_{33}}\frac{z}{R} - \frac{r^2}{6R^2}\frac{S_{31}(\phi)+3S_{32}(\phi)}{\langle S_{21}(\phi)\rangle + 3\langle S_{22}(\phi)\rangle}$$

Ari-Pekka Honkanen et al.,
J. Synchrotron Radiat. 11, 762 (2014)
J. Appl. Cryst. 49, 1284 (2016)

C_{ij}=stiffness tensor
S_{ij}=compliance tensor

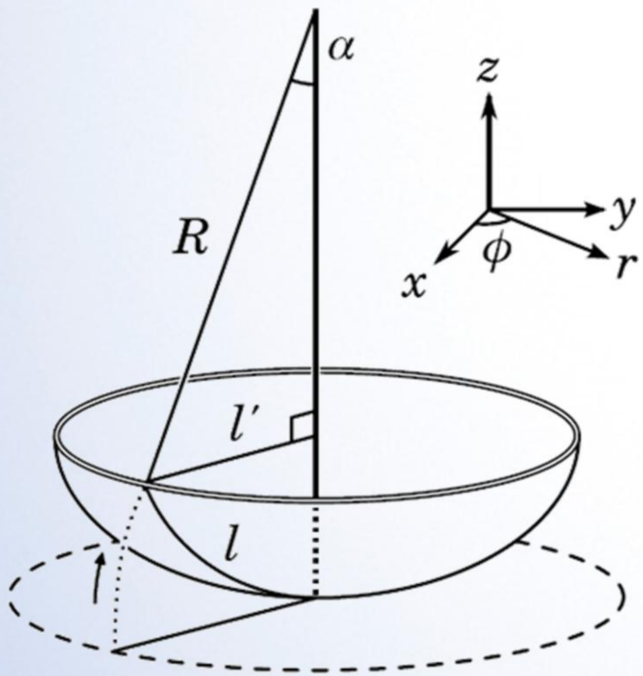
Study on the reflectivity properties of spherically bent analyser crystals

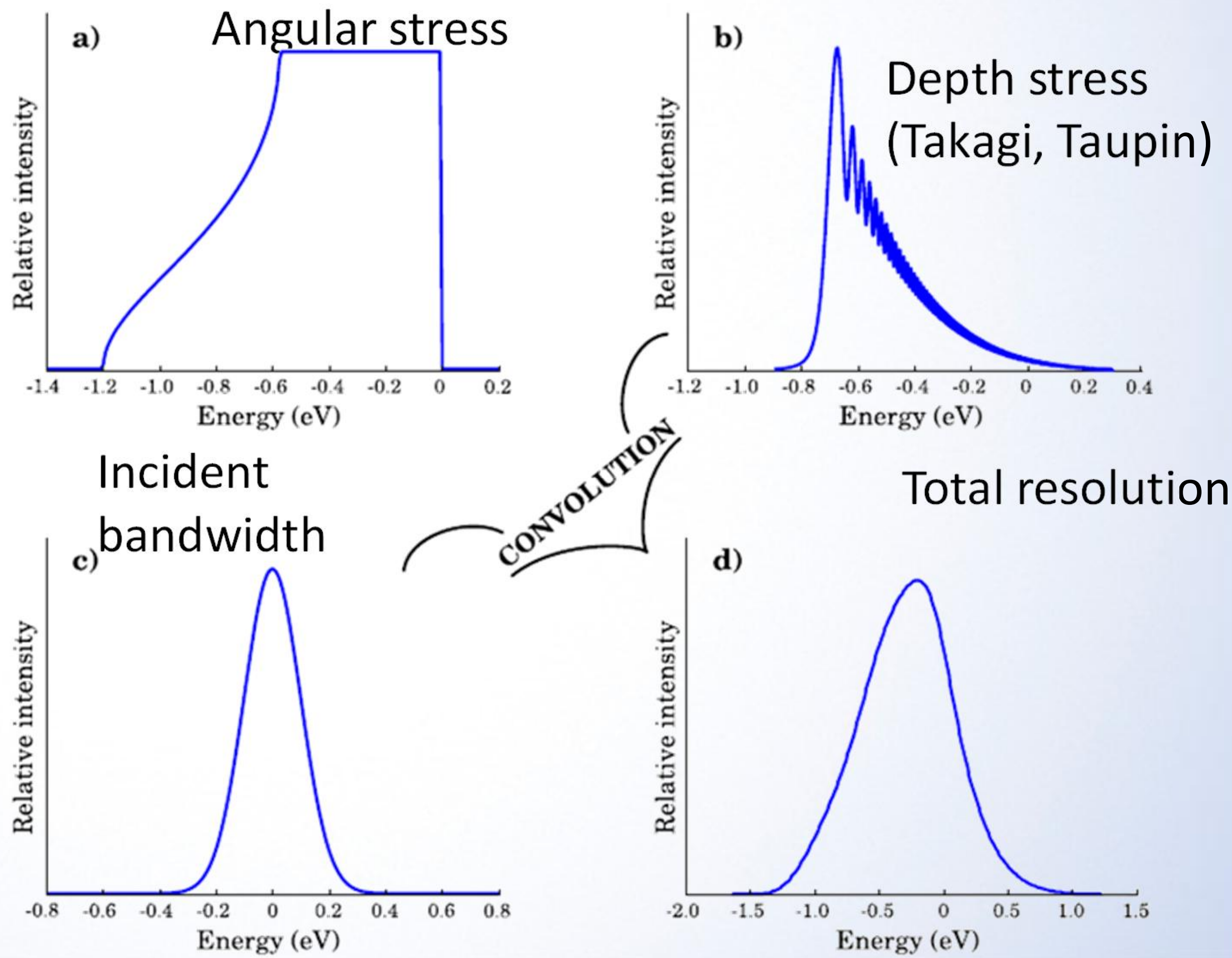
J. Synchr. Rad. 21, 762 (2014)

Ari-Pekka Honkanen,[a] Roberto Verbeni,[b] Laura Simonelli,[b] Marco Moretti Sala,[b] Giulio Monaco[b] and Simo Huotari[a]

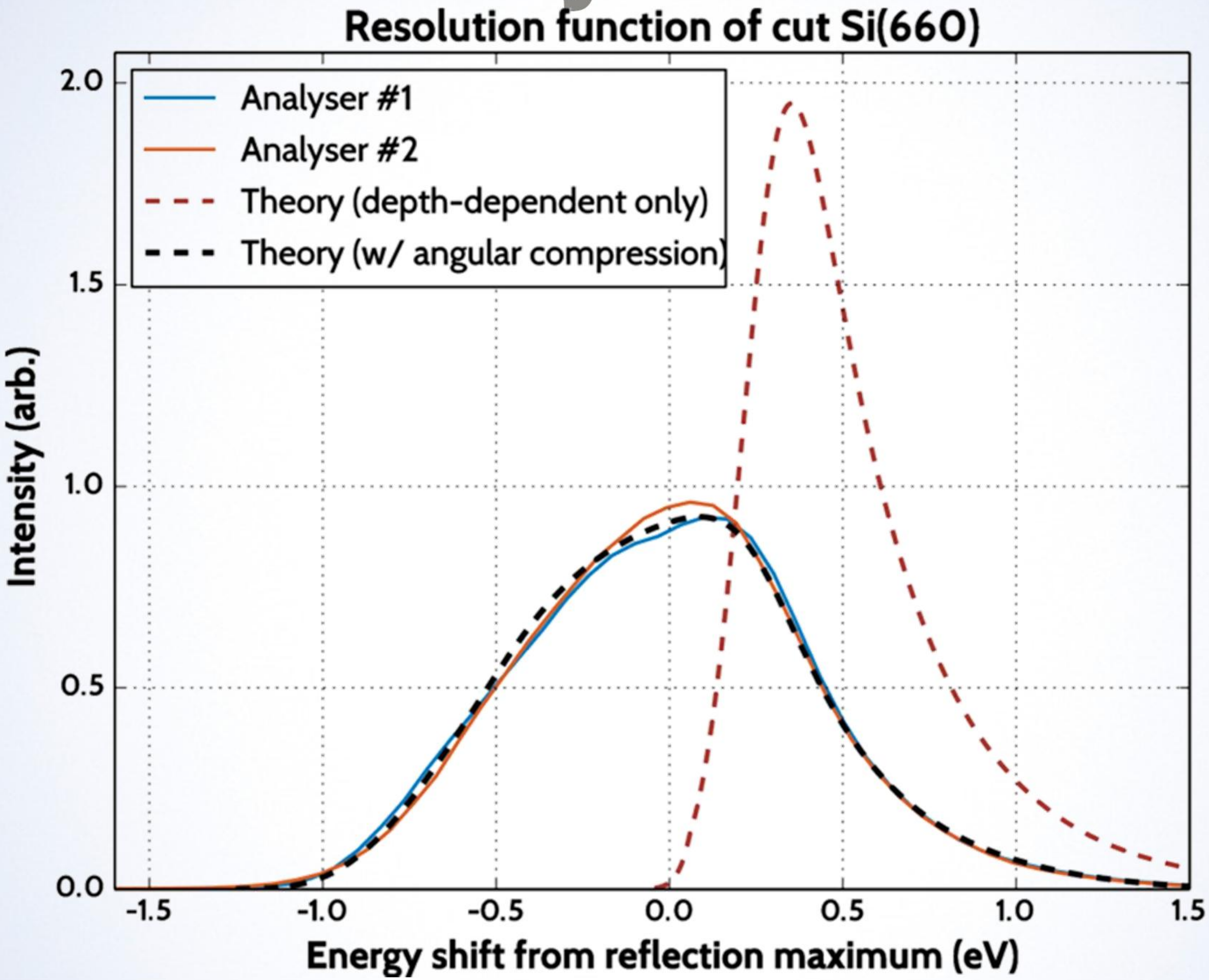
Department of Physics, P. O. Box 64, FI-00014 Helsinki, Finland, and [b]European Synchrotron Radiation Facility, B. P. 220, F-38043 Grenoble cedex, France

Simulation for 9.7 keV, Si(660), R=1 m 4-inch wafer (100 mm)





Bent crystal diffraction

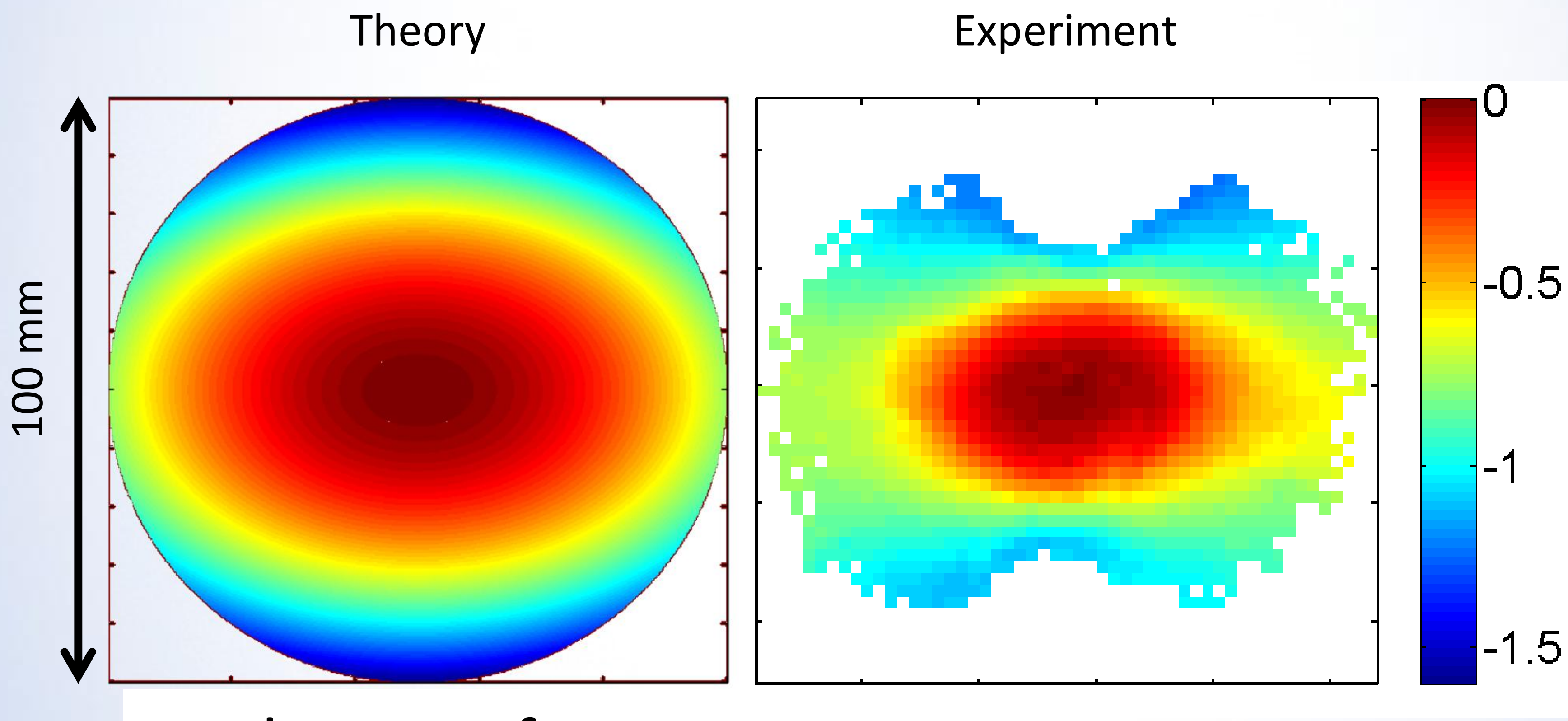


$R = 1$ m,
$E = 9.7$ keV

Ari-Pekka Honkanen et al.,
J. Synchrotron Radiat. 11, 762 (2014)
J. Appl. Cryst. 49, 1284 (2016)

Quantifying angular strain

Distribution of energy shifts caused by angular strain
Si(660) in near backscattering, E=9.7 keV, R=1 m

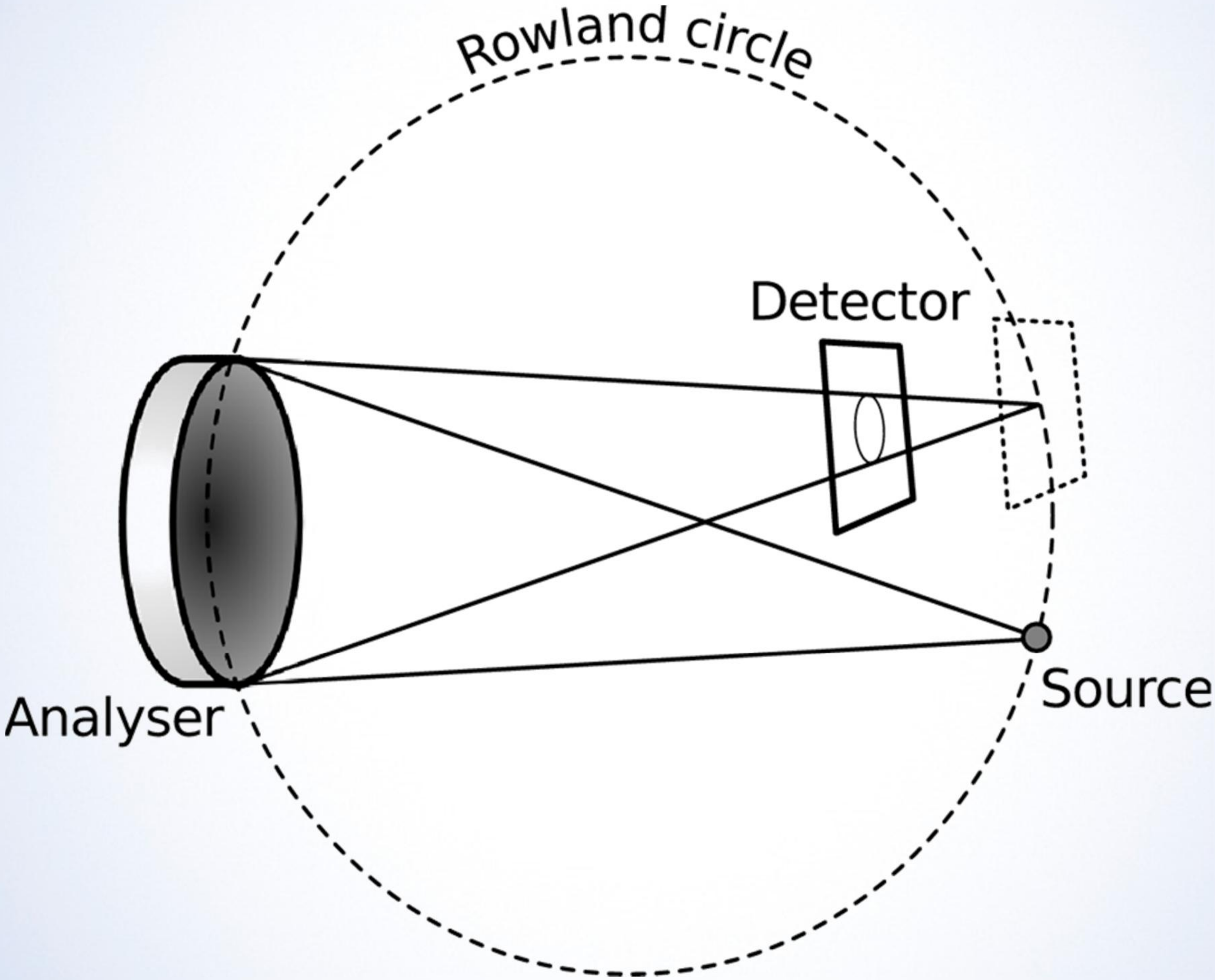


Analyzer surface

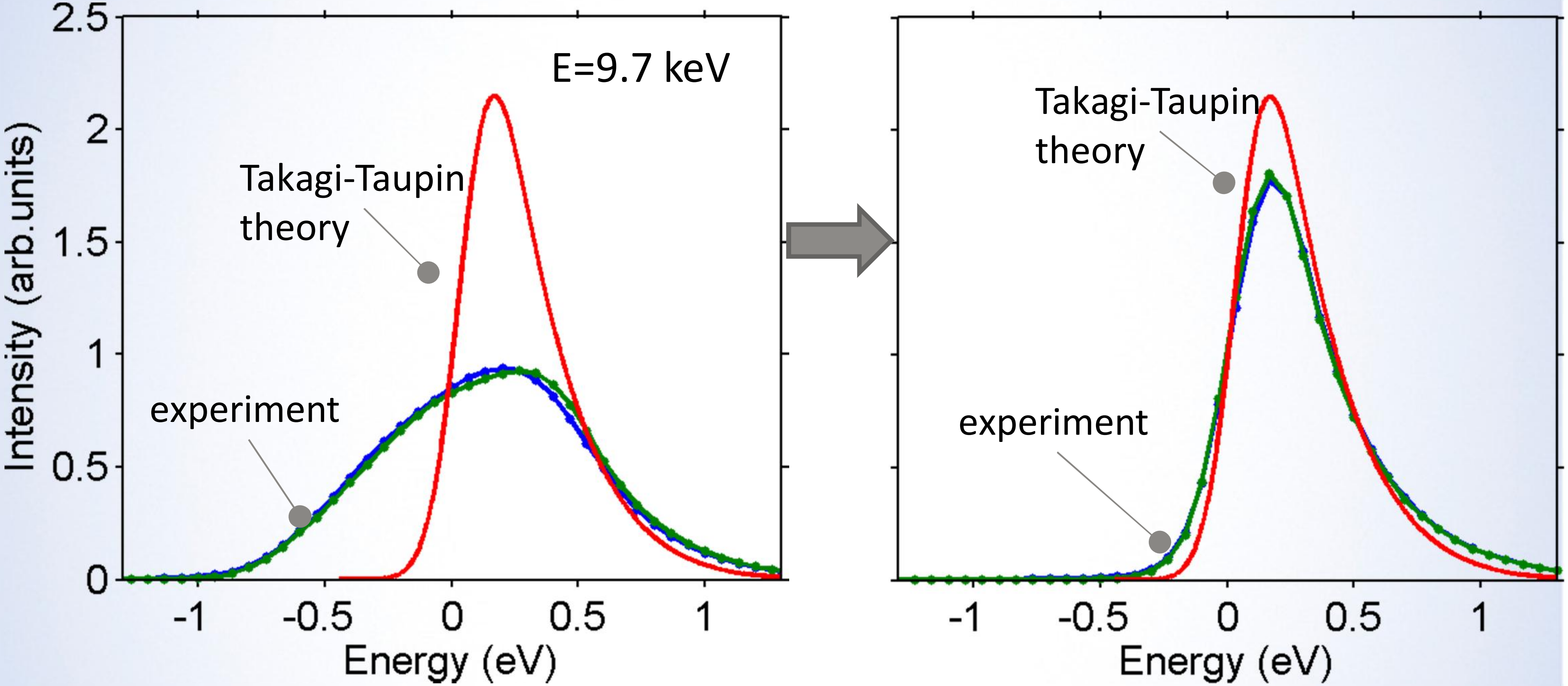
ID20, ESRF

Ari-Pekka Honkanen et al.,. J. Synchrotron Radiat. 11, 762 (2014)
J. Appl. Cryst. 49, 1284 (2016)

Quantifying angular stress



RESOLUTION FUNCTION CORRECTION

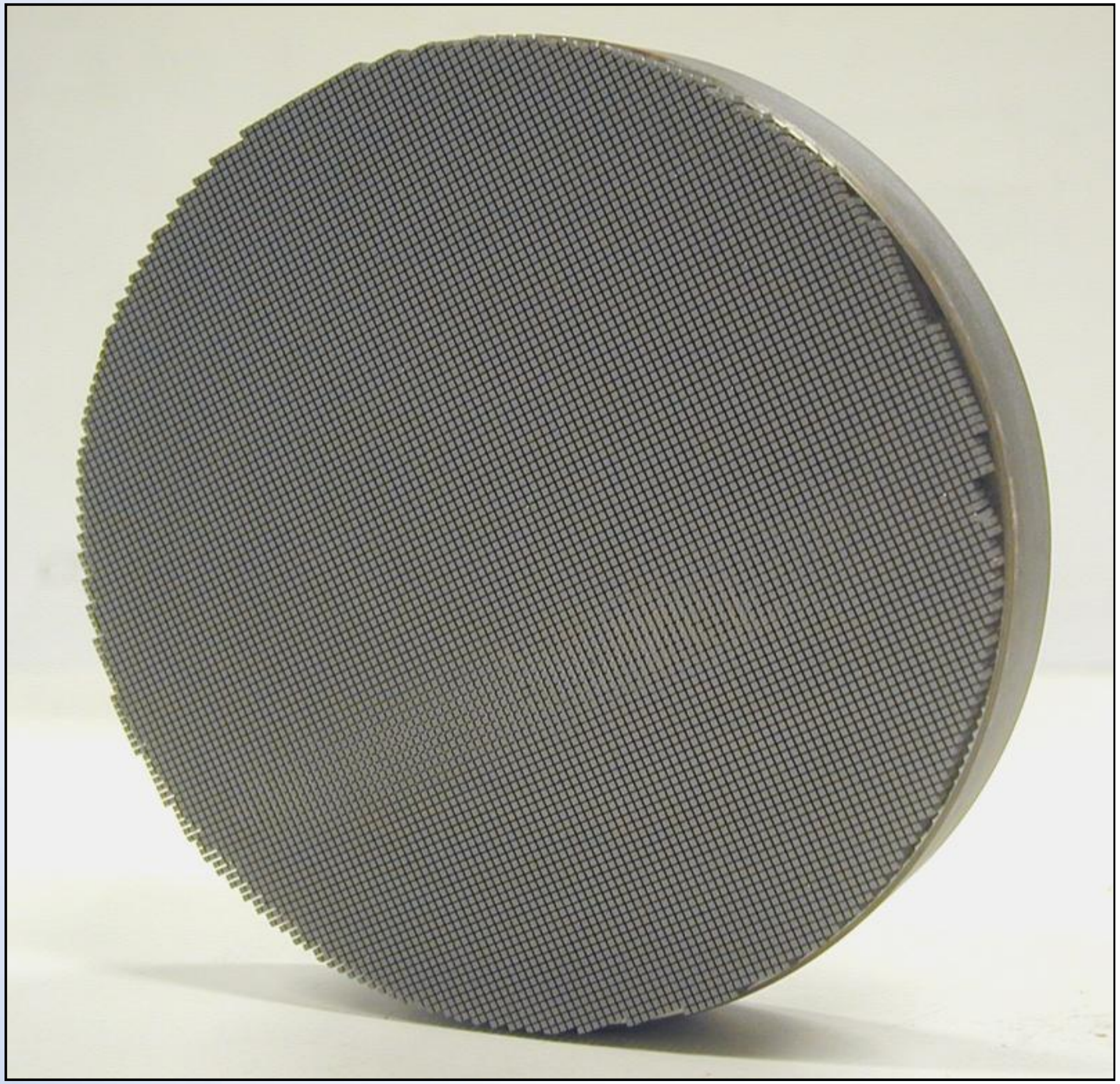


(Honkanen, pixelwise compensation method)

J. Synchr. Rad. 21, 762 (2014)

DICED CRYSTALS

Not bent, instead: several small flat crystallites (each ~mm^2) to approximate the curved surface



In Rowland geometry
Energy resolution set by Darwin width
FWHM ~1 meV @20 keV)

Blasdell and Macrander, Rev. Sci. Instrum. 66, 2075 (1995)

Masciovecchio et al. Nucl. Instrum. Meth. B 111, 181 (1996); Nucl. Instrum. Meth. B 117, 339 (1996)

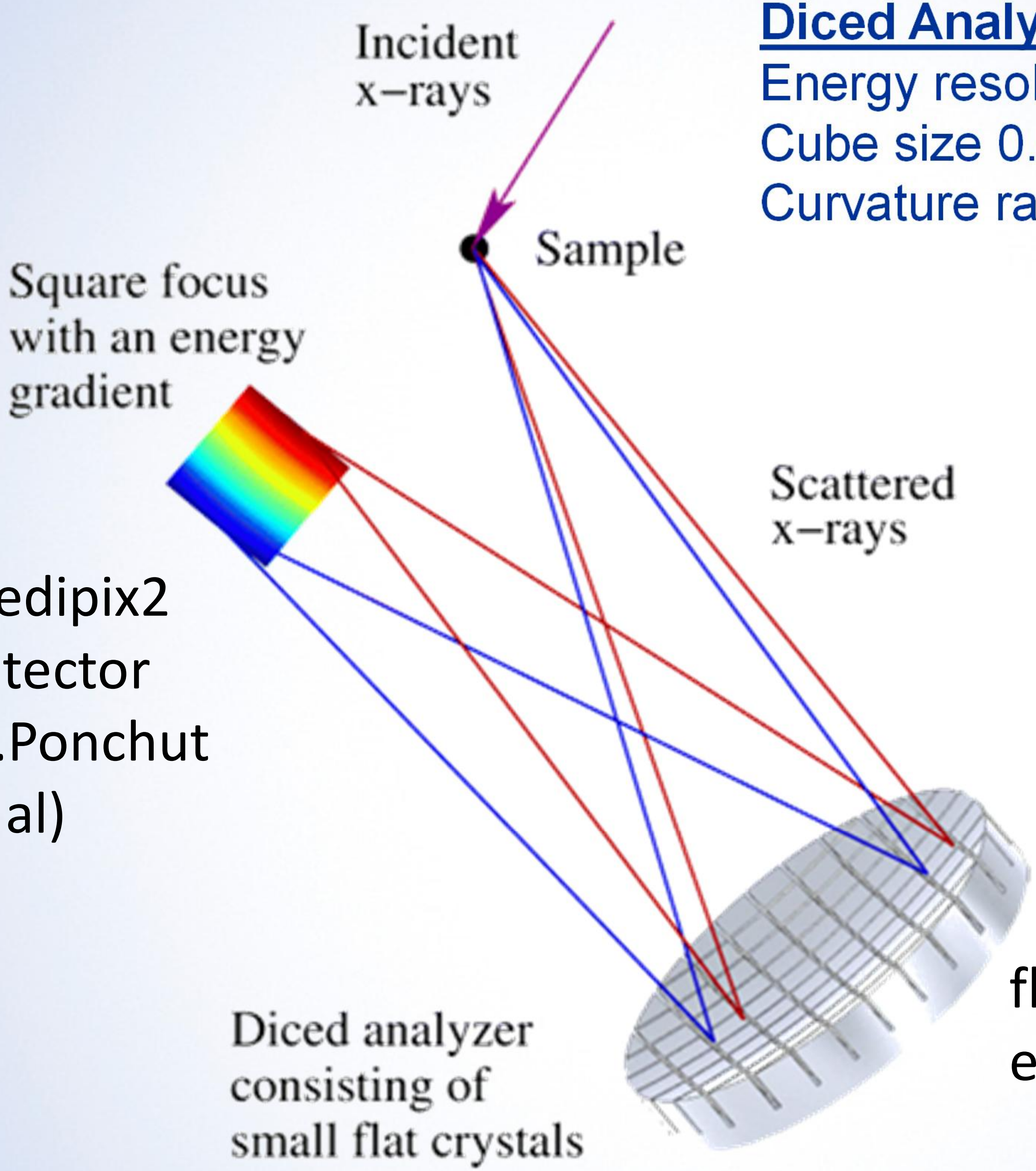
Figure: ESRF crystal analyzer lab; Roberto Verbeni et al.

Diced Analyzers

Energy resolution ~ 1 meV @ 20 keV

Cube size 0.8 mm x 0.8 mm x 3 mm

Curvature radius 1 m to >10 m

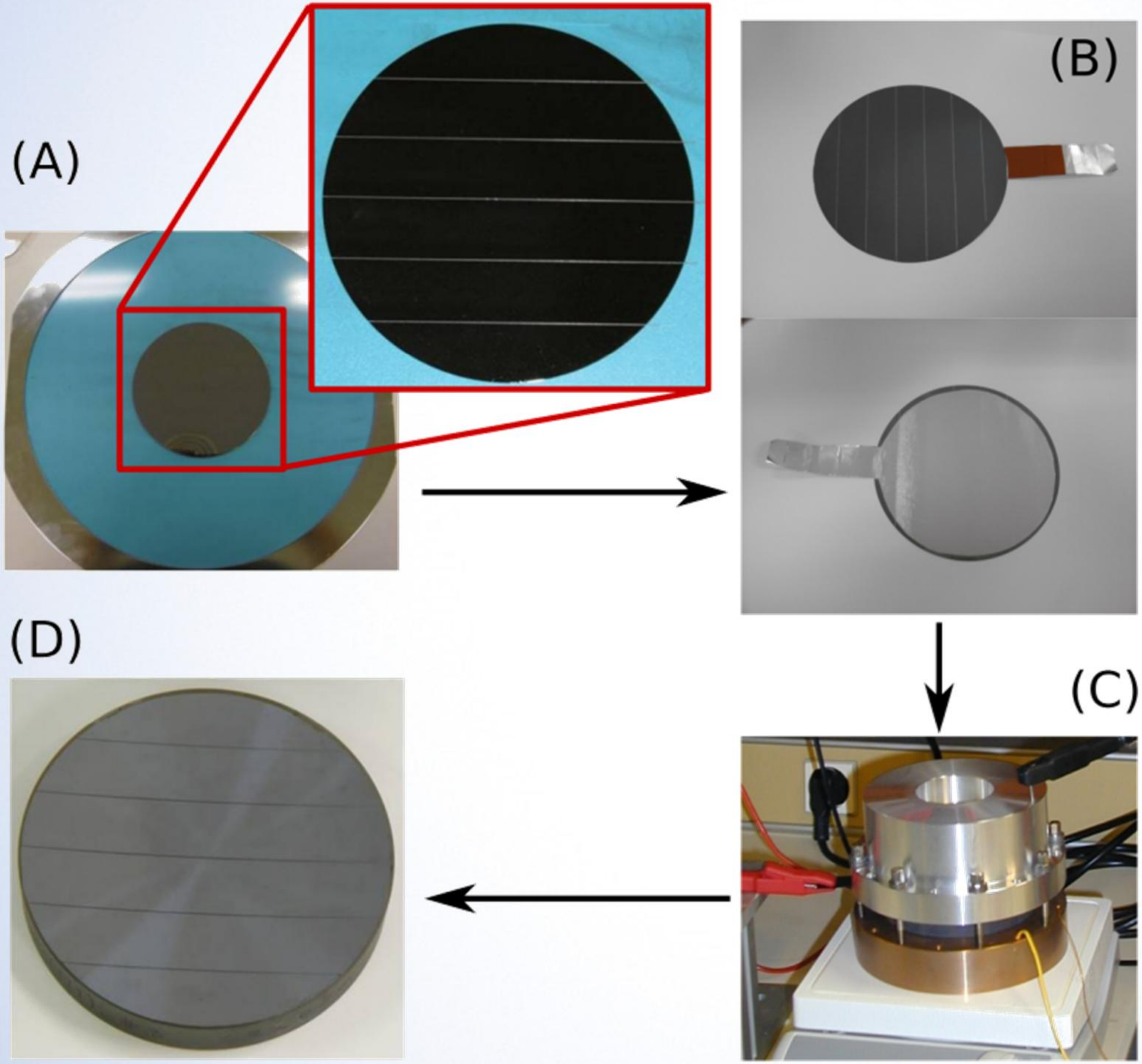


Medipix2 detector (C.Ponchut et al)

flat crystallite size exaggerated for clarity

from S. Huotari et al. JSR 2005

STRESS-RELIEVED CRYSTALS



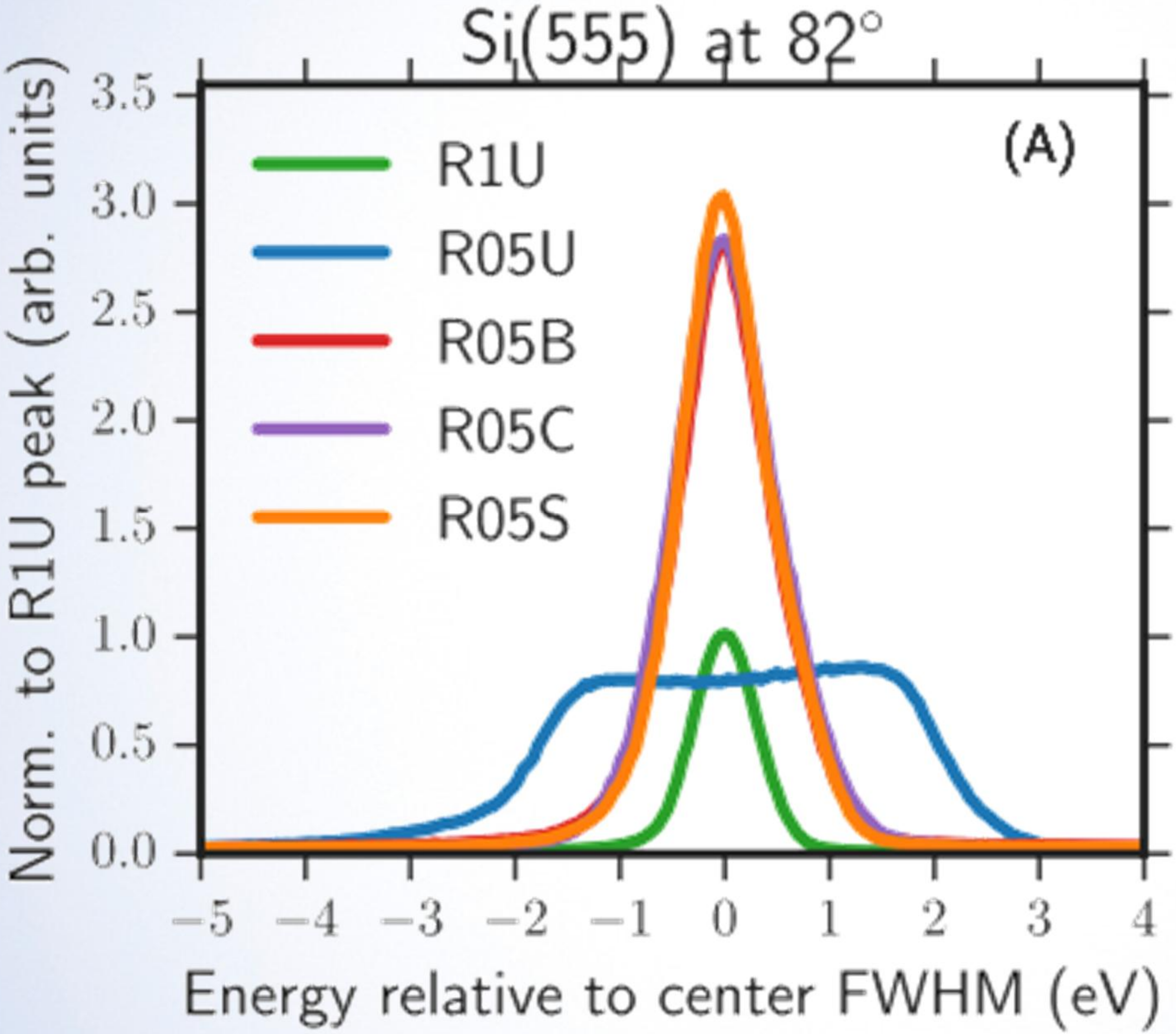
ESRF Crystal Lab

R=0.5 m
(cf. standard
R=1 m up to now)

150 μm thick wafer
cut into 15 mm slices

Rovezzi et al.,
Review of
Scientific
Instruments **88**,
013108 (2017)

STRESS-RELIEVED CRYSTALS



Rovezzi et al., Review of Scientific Instruments **88**, 013108 (2017)

R=0.5 m crystals compared to R=1.0 m crystal

Modern instruments

Cover page of Journal of Synchr. Radiat. March 2017 :

Huotari et al., JSR 2017

Spectrometer located at **European Synchrotron Radiation Facility** (ESRF, Grenoble, France)

Beamline ID20:

Energy range 4-20 keV

inelastic x-ray scattering (resonant and non-resonant)

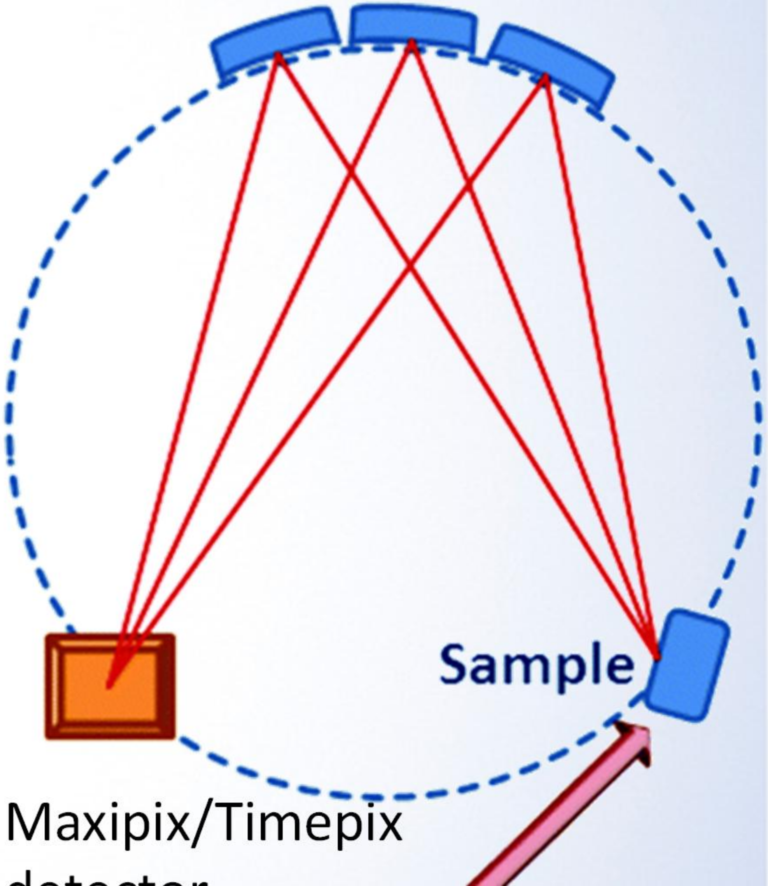
x-ray emission spectroscopy

high-energy-resolution fluorescence detected XAS

ID20@ESRF

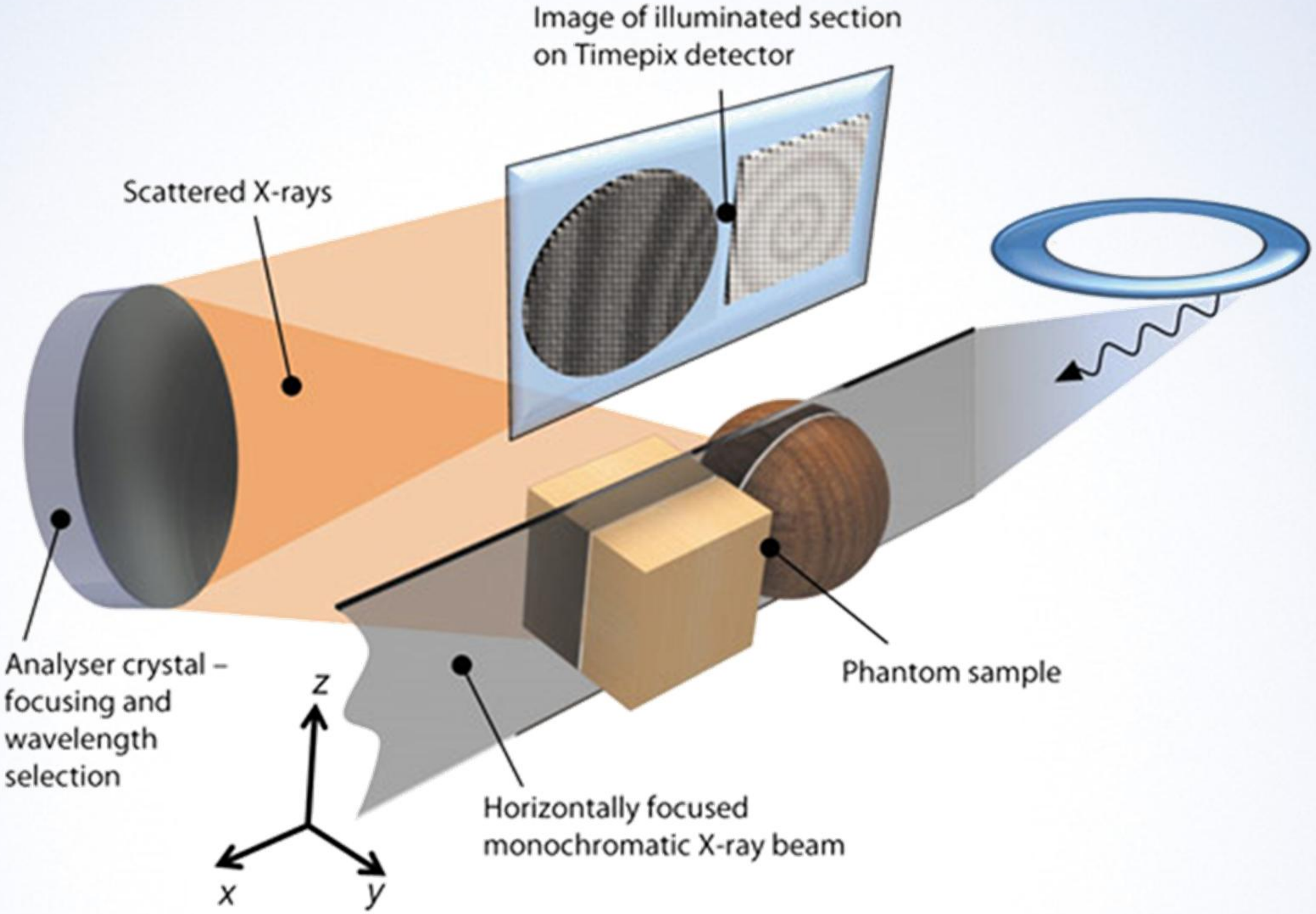
Roberto Verbeni and his 72 analyzers

3 x 4 Johann spherically bent analyzer crystals in a module



Maxipix/Timepix detector
256x256 pixels
55 x 55 μm^2 each

Credit: ID20 team (Laura Simonelli, M. Moretti Sala, Ali Al-Zein, R. Verbeni, M. Krisch, G. Monaco, et al.)



nature materials

LETTERS

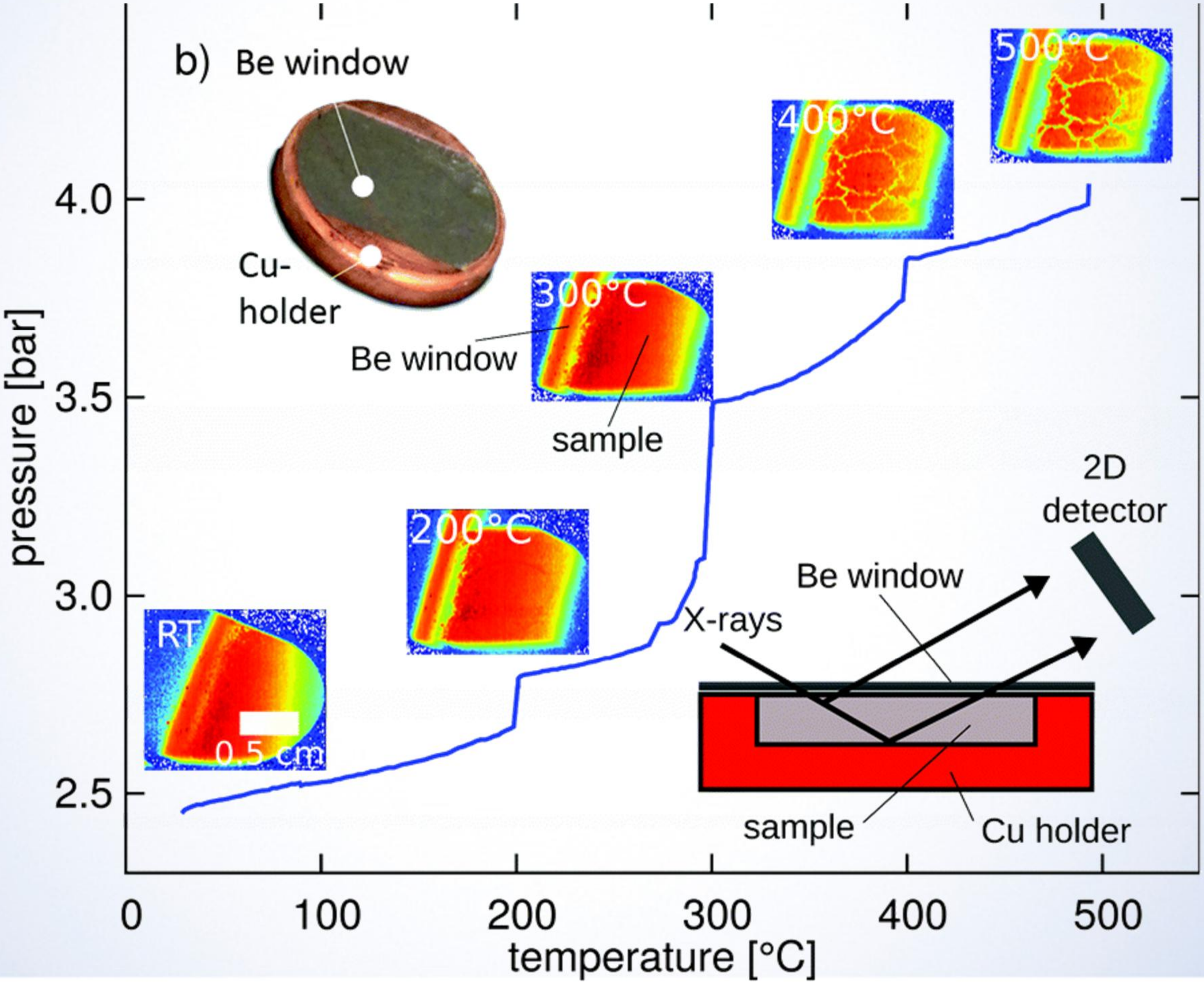
PUBLISHED ONLINE: 29 MAY 2011 | DOI: 10.1038/NMAT3031

Direct tomography with chemical-bond contrast

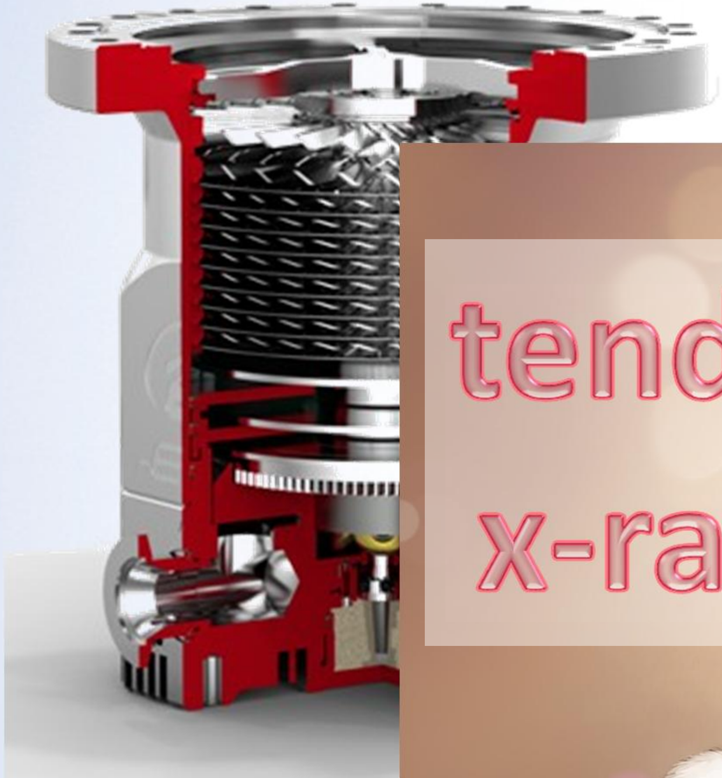
Simo Huotari[1,2]★, Tuomas Pylkkänen[1,2], Roberto Verbeni[1], Giulio Monaco[1] and Keijo Hämäläinen[2]

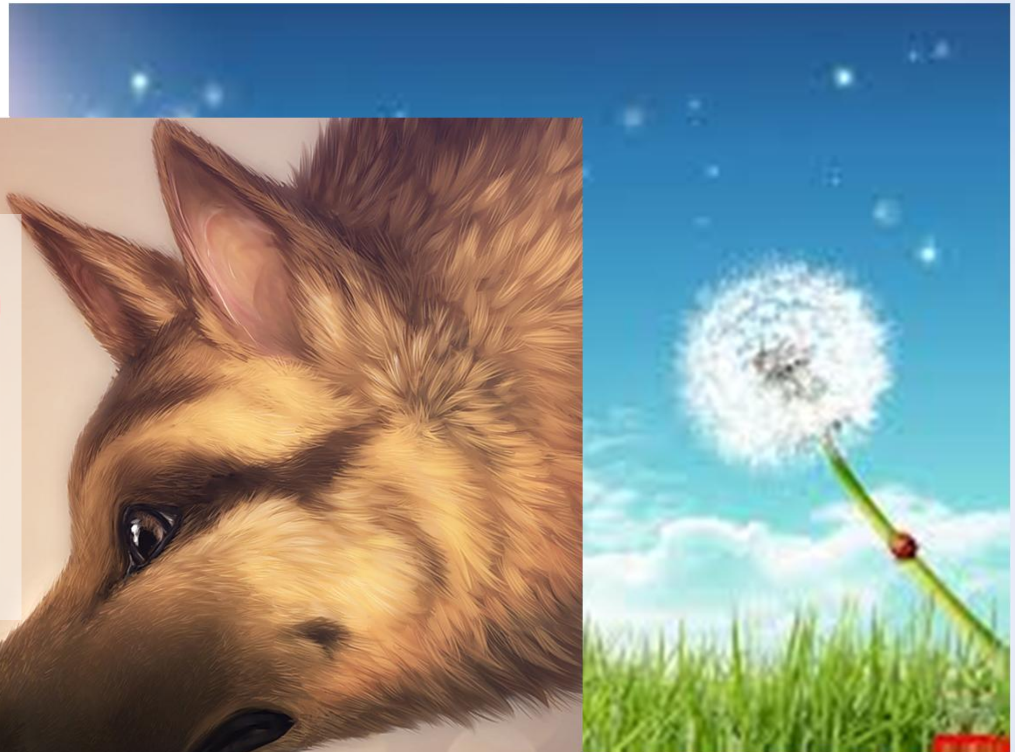
IMAGING/SPECTROSCOPY WITH 2D DETECTOR + CRYSTAL OPTICS

Ch. Sahle et al., PCCP 18, 5397 (2016)



SOFT – TENDER – HARD X-RAYS

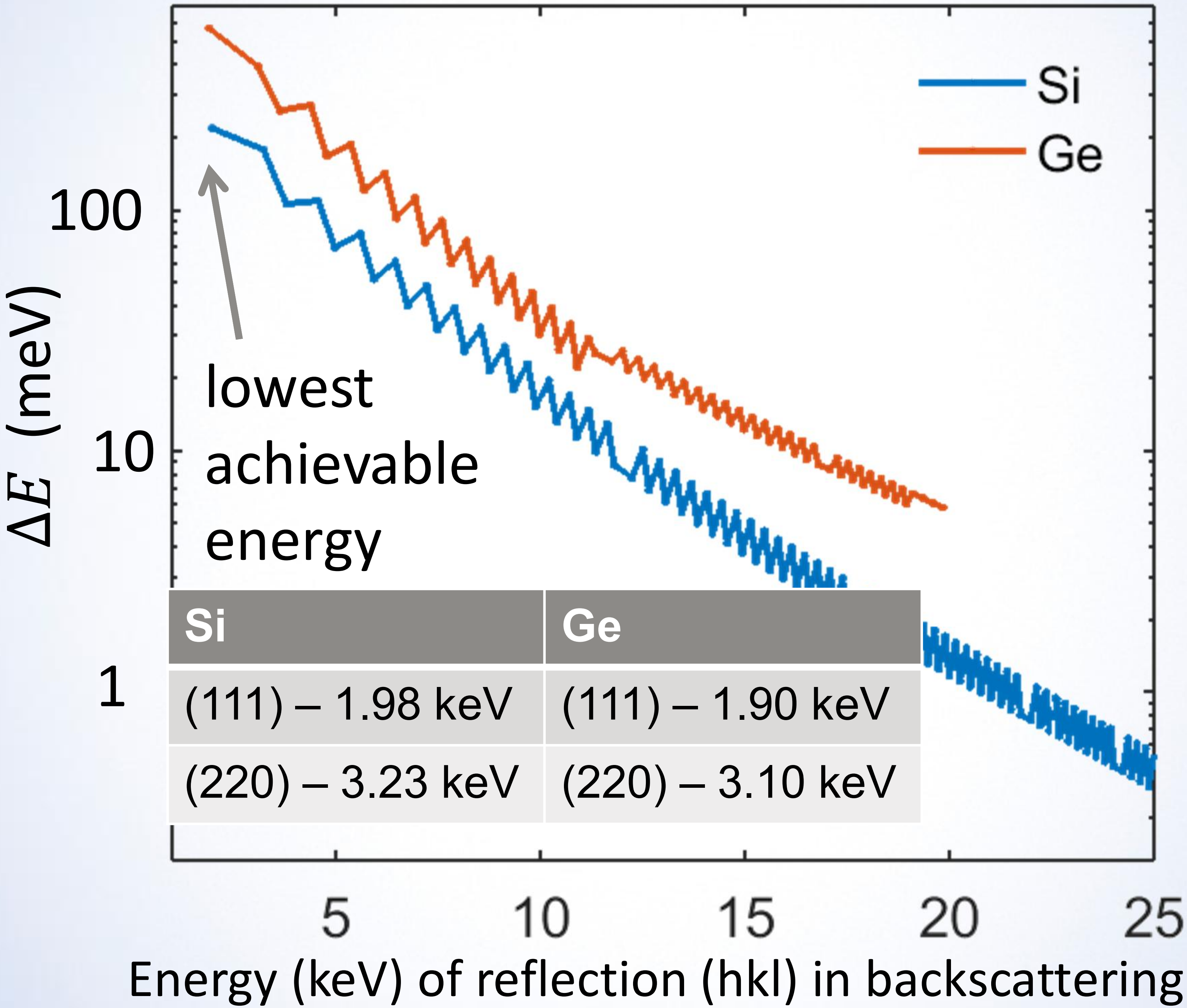




Soft x-rays re

work in air

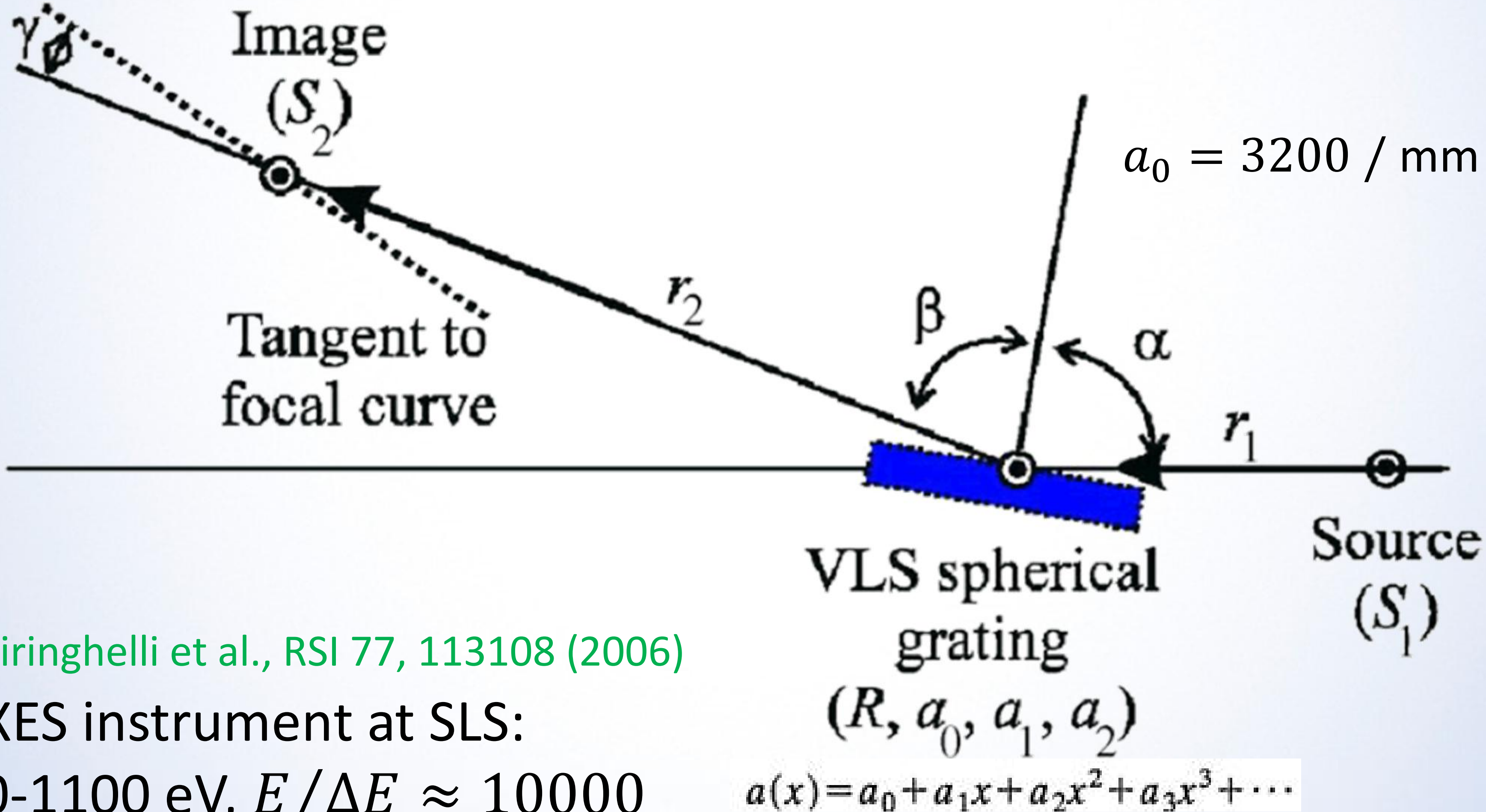
ULTIMATE ENERGY RESOLUTION BY DARWIN WIDTH



Si	Ge
(111) – 1.98 keV	(111) – 1.90 keV
(220) – 3.23 keV	(220) – 3.10 keV

SOFT X-RAY SPECTROMETERS

- Natural crystals (Bragg) are not an option
- Grazing incidence concave diffraction gratings with variable line spacing (VLS)



$a_0 = 3200$ / mm

G. Ghiringhelli et al., RSI 77, 113108 (2006)

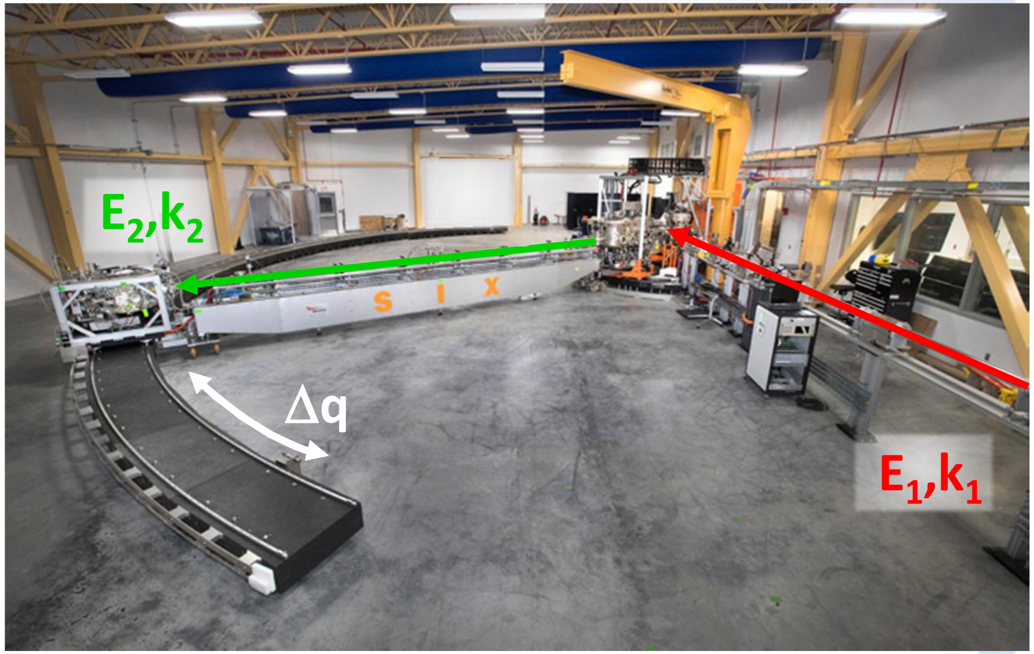
SAXES instrument at SLS:

400-1100 eV, $E/\Delta E \approx 10000$

SOFT INELASTIC X-RAY SCATTERING (SIX) AT NSLS-II

Resonant inelastic x-ray scattering at an unprecedented energy resolution for unrivaled sensitivity to low-energy collective excitations.

- 135-2300 eV energy range with 0.6 μm (V) x 6 μm (H) focus
- Resolving power up to 100,000
- Moving 15-m long arm for unbroken access to full range of momentum transfers
- Sample cooling to ~15 K



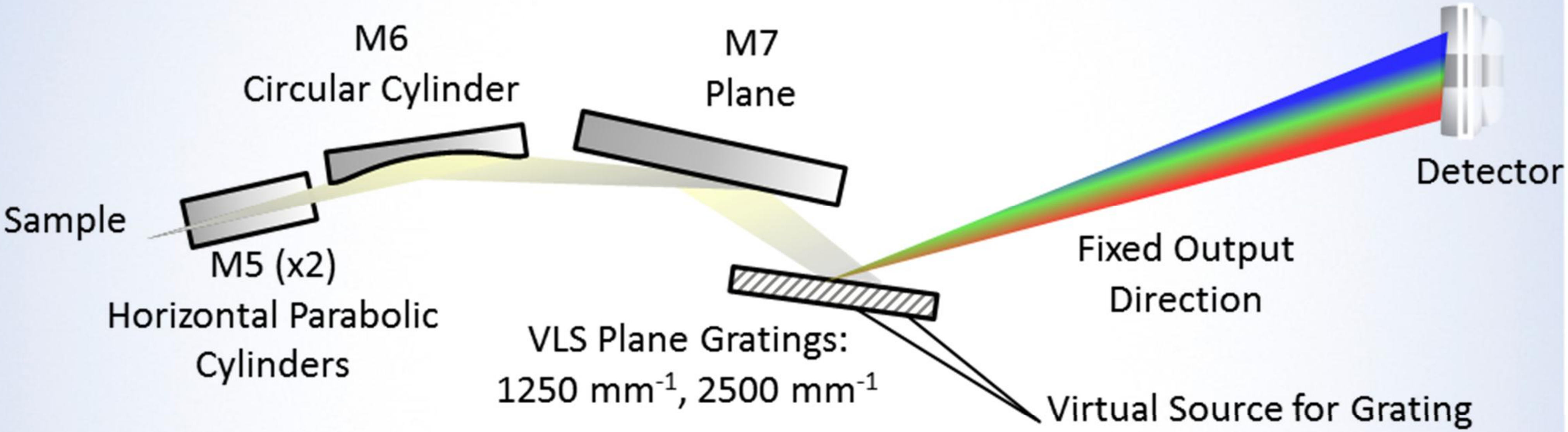
Goal: ΔE=15 meV at 1,000 eV

First light: Feb. 21st 2017

Slide courtesy from NSLS-II / Ignace Jarrige, Joe Dvorak et al. shared for this occasion

SIX OPTICAL DESIGN

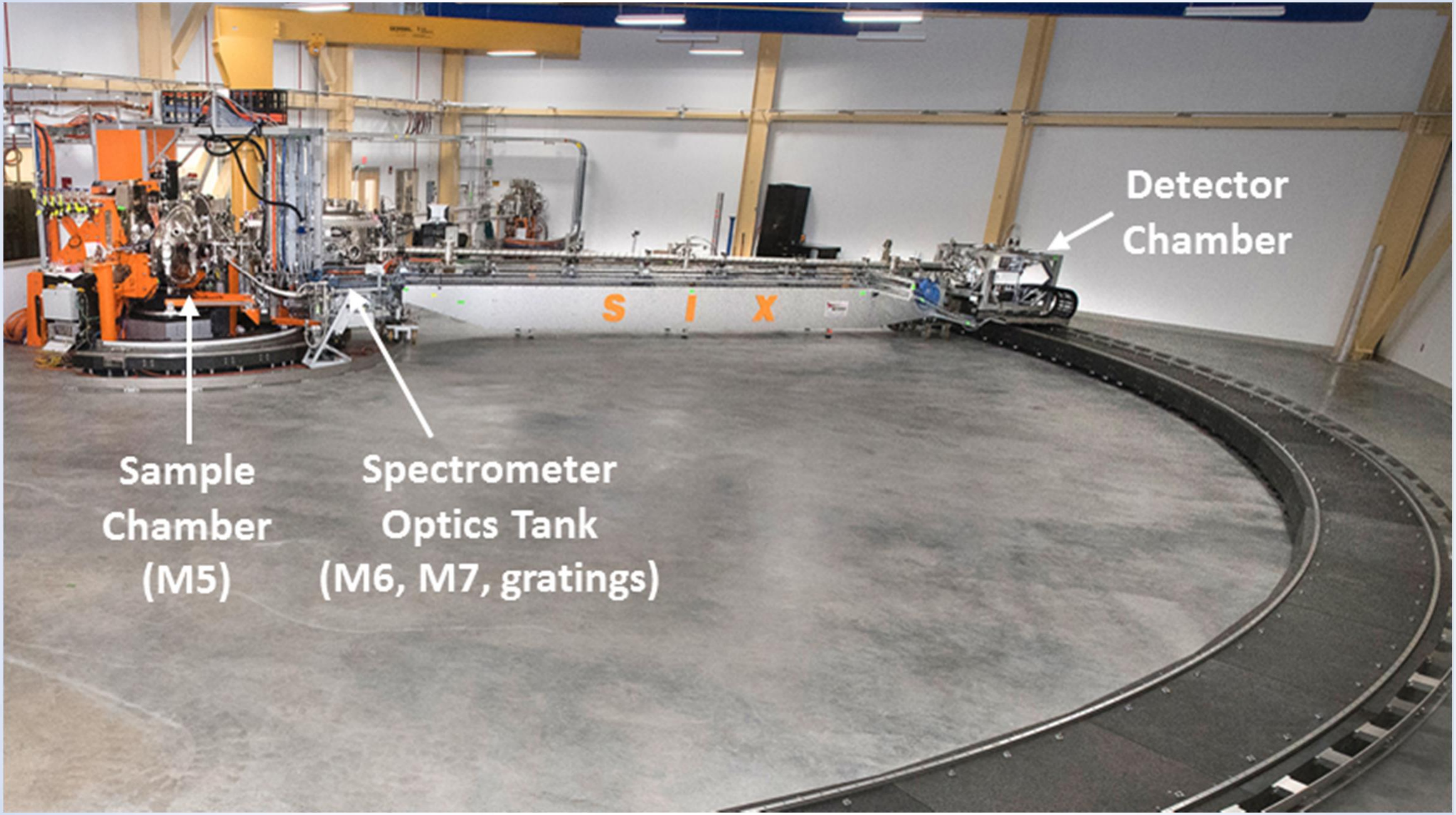
Slide courtesy from NSLS-II / Ignace Jarrige et al.



- **M5**: Horizontally collimating mirrors located close to sample for large solid angle
- **M6**: Vertical focusing mirror for virtual source for grating
- **M7**: Vertical deflecting mirror ⇒ beam outgoing angle constant

Slide courtesy from NSLS-II / Ignace Jarrige, Joe Dvorak et al. shared for this occasion

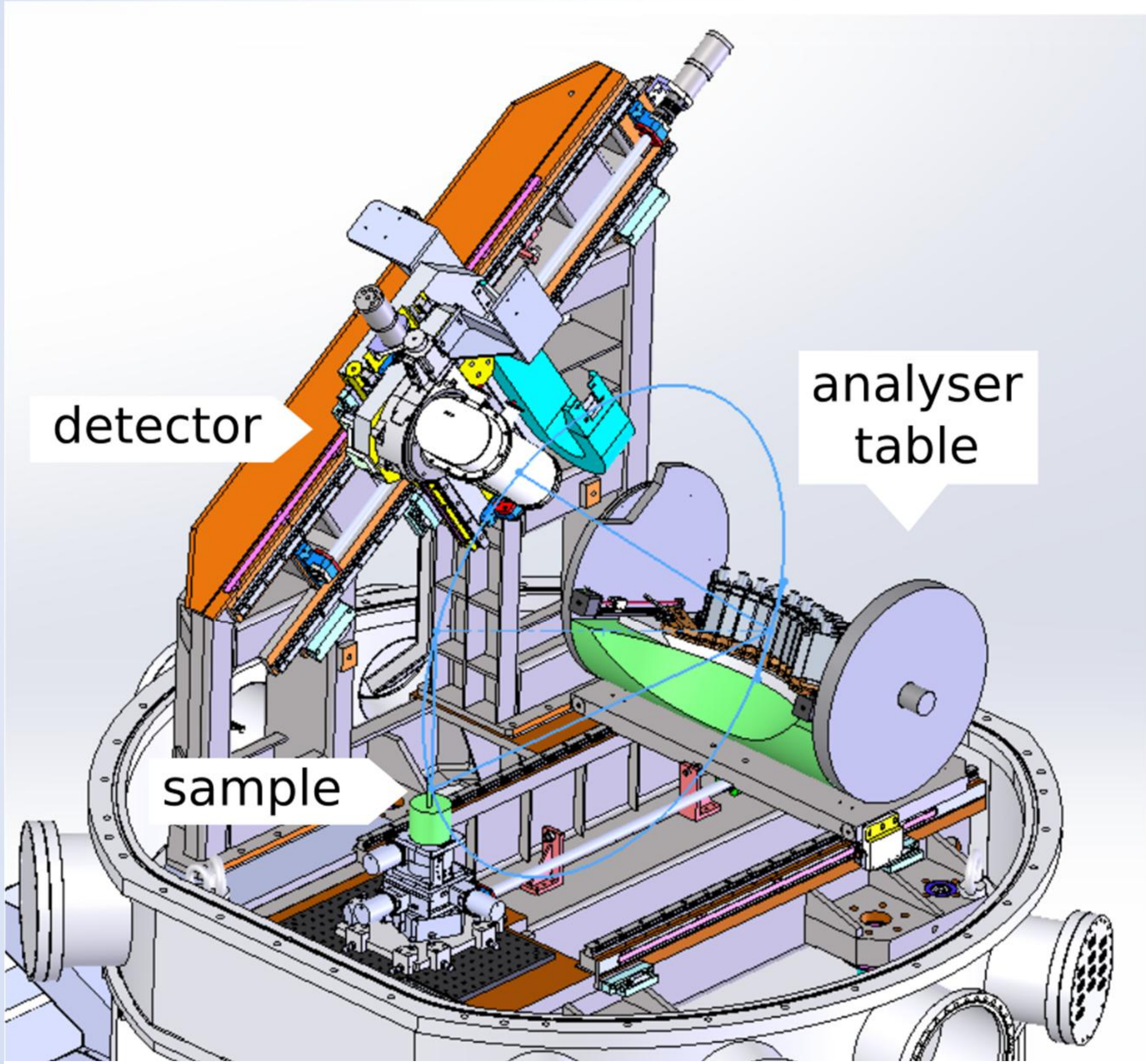
SIX SPECTROMETER @ NSLS-II



Slide courtesy from NSLS-II / Ignace Jarrige, Joe Dvorak et al. shared for this occasion

Large area in-vacuum detector for XES, ESRF ID26

M. Kocsis, M. Rovezzi, B. Detlefs, P. Glatzel, ESRF

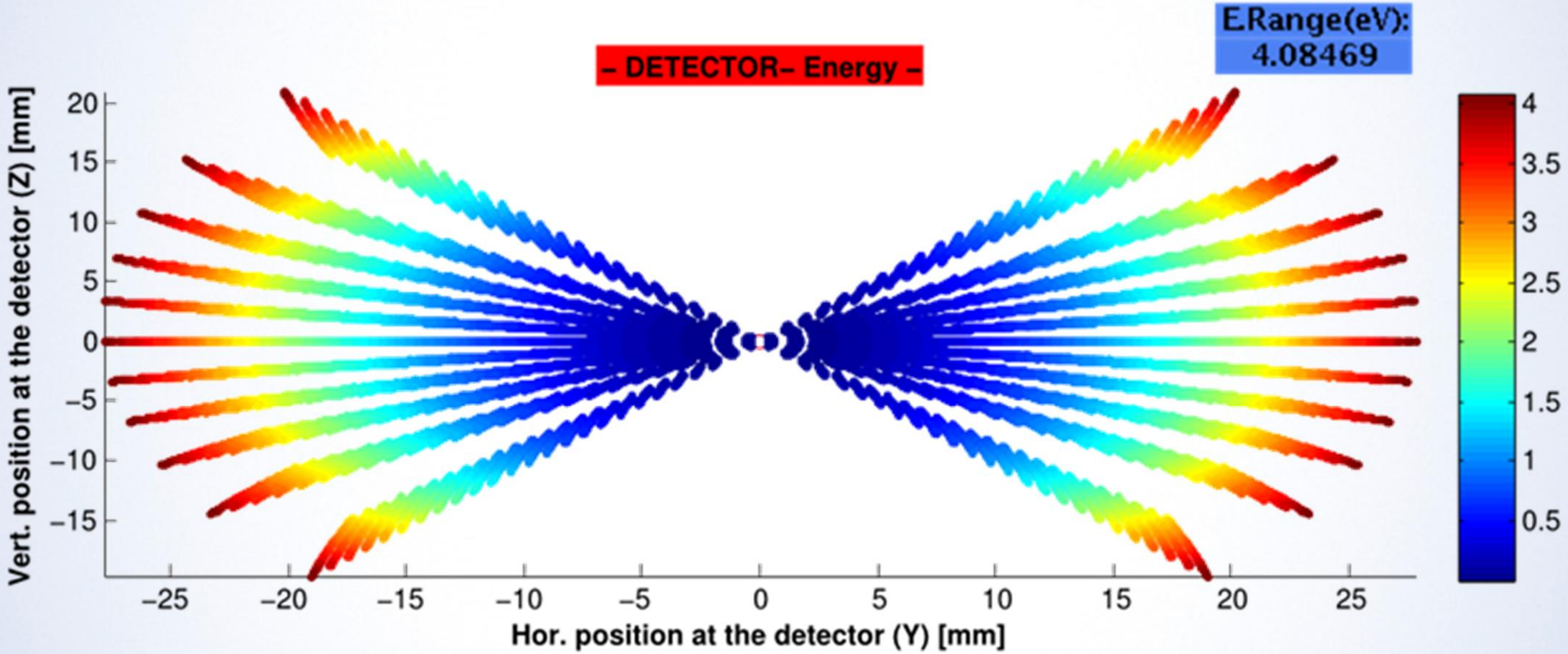


Slide courtesy from ESRF / Pieter Glatzel et al., shared for this occasion

- Energy 1.5—5.5 keV; very low electronic noise, high quantum efficiency (>80%)
- No suitable detector on the market → Inhouse
- 16 vertical wires gas flow proportional counter detector
- Gas: 15% CO_2 in Ar
- Vacuum compatibility: flexible hoses (inverse vacuum), thin polymer entrance window

M. Kocsis, M. Rovezzi, B. Detlefs, P. Glatzel, ESRF

Detector area at 35°/500 mm bending radius ≅ 50 x 40 mm^2



Slide courtesy from ESRF / Pieter Glatzel et al.,
shared for this occasion

THANK YOU!

Slides of spectrometers around the world:

NSLS-II (SIX): Ignace Jarrige, Joe Dvorak, SIX team

EuXFEL (FXE): Christian Bressler, XFEL team

ID26 in-vacuum spectrometer: Pieter Glatzel, M. Kocsis, B. Detlefs, M. Rovezzi, ID26/ESRF team

ID20: R. Verbeni, M. Moretti Sala, Ch. Sahle, L. Simonelli, C. Henriquet, M. Krisch, G. Monaco, ID20/ESRF

Team in Helsinki:

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