X-ray optics

from the macro- to the nano-world

Jürgen Härtwig

European Synchrotron Radiation Facility, BP 220, 38043 Grenoble Cedex, France

haertwig@esrf.fr

Outline

1. Introduction
2. Beam dimensions
3. Crystalline materials for X-ray optics
4. Monochromators
5. Collimation, focussing, magnification

Acknowledgements

Colleagues from the X-ray optics group:

Ray Barrett, Christian Morawe,

Anatoly Snigirev

Colleagues from other groups/beamlines:

Elodie Boller, Peter Cloetens, Jean Susini,

Paul Tafforeau, Lin Zhang, ...

SR Sources Worldwide
Courtesy of APS/ANL website

Worldwide more than 50 SR and XFEL facilities,

European Synchrotron Radiation Facility
Grenoble, France

only multi-national source

One of the three (older) high-energy SR sources in the world

ESRF - 6GeV, APS - 7GeV, SPring8 - 8GeV

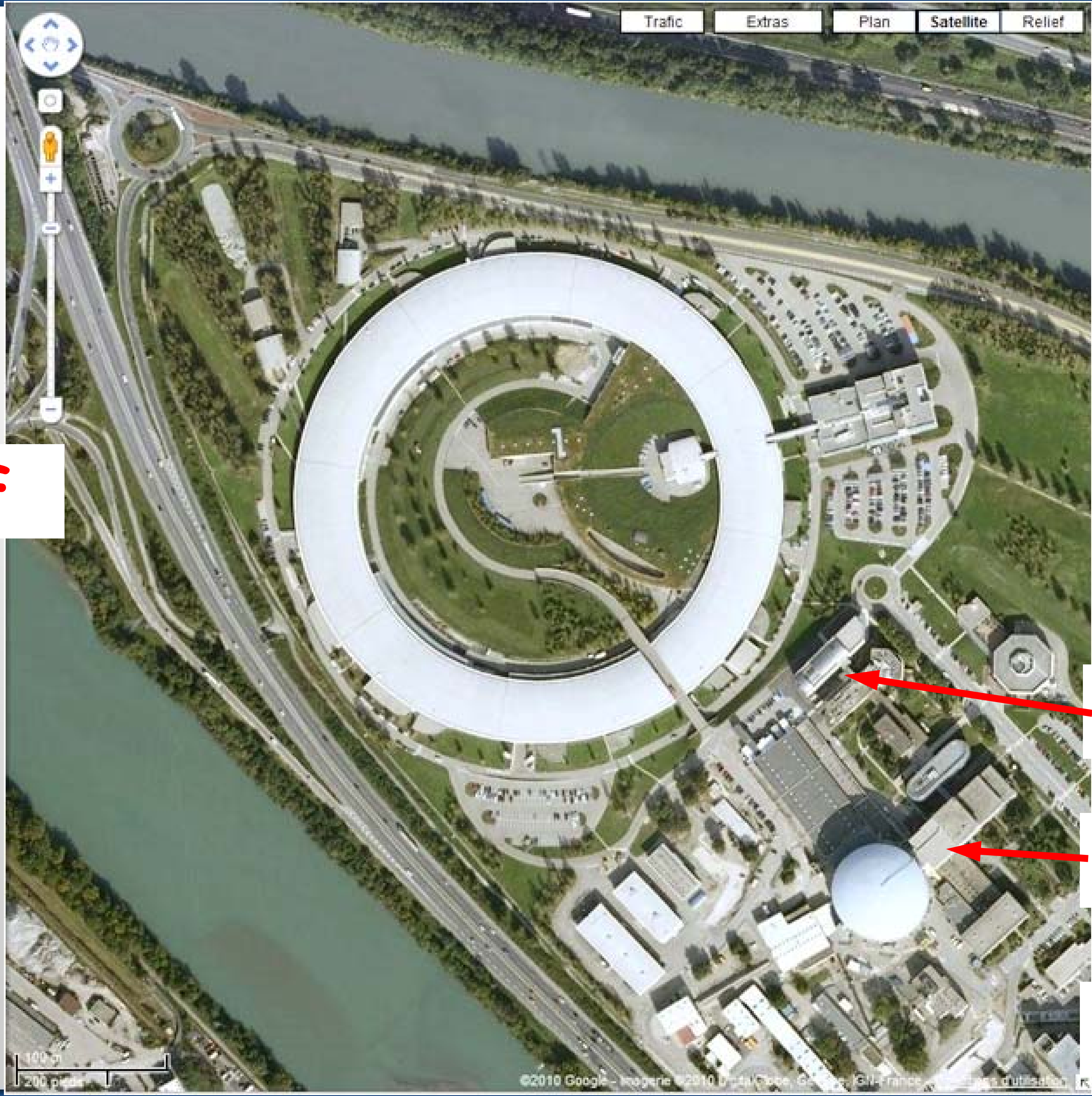
fourth (new) one – PETRA III - 6GeV, Hamburg

classical “medium” energy facilities
– 2.5GeV to 3.5GeV

Grenoble


Northampton
Ipswich
Gloucester
Luton
Chelmsford
Swansea
Bristol
Swindon
London
Southend-on-Sea
Cardiff
Reading
Woking
Gillingham
Dunkerque
Southampton
Brighton
Calais
Exeter
Bournemouth
Portsmouth
Eastbourne
Plymouth
Lille
Den Haag
Nederland
Netherlands
Rotterdam
Nijmegen
Eindhoven
Enschede
Bielefeld
Paderborn
Recklinghausen
Essen
Dortmund
Düsseldorf
Wuppertal
Kassel
Brussel
Bruxelles
Maastricht
Köln
Marburg
Aachen
Siegen
Bonn
België
Belgique
Belgium
Koblenz
Frankfurt am Main
Wiesbaden
Amiens
Saint-Quentin
Luxembourg
Darmstadt
Le Havre
Beauvais
Kaiserslautern
Mannheim
Guernsey
Jersey
Caen
Rouen
Saarbrücken
Cergy
Karlsruhe
Heilbronn
Évreux
Reims
Metz
Paris
Stuttgart
Saint-Malo
Nancy
Ulm
Brest
Strasbourg
Reutlingen
Rennes
Laval
Le Mans
Troyes
Colmar
Orléans
Villingen-Schwenn
Freiburg im Breisgau
Mulhouse
Blois
Konstanz
Angers
Dijon
Basel
Zürich
Tours
Besançon
Saint-Nazaire
Nantes
Liechtenstein
Schweiz
Suisse
Svizzera
Switzerland
Châteauroux
Bourges
France
Lausanne
La Roche-sur-Yon
Niort
Genève
La Rochelle
Limoges
Lyon
Annecy
Bergamo
Chambéry
Novara
Milano
Grenoble
Torino
Saint-Etienne
Brive-la-Gaillarde
Piacenza
Valence
Bordeaux
Alessandria
Gap
Genova
Montauban
La Spezia
Albi
Avignon
Montpellier
Monaco
Santander
Toulouse
Béziers
San Sebastián
Pau
Martigues
Aix-en-Provence
Antibes
Carcassonne
Bilbao
Marseille
Hyères
Pamplona
Vitoria-Gasteiz
Perpignan
Logroño
Andorra
Burgos
©2010 Google - Données cartographiques ©2010 Tele Atlas -

ESRF

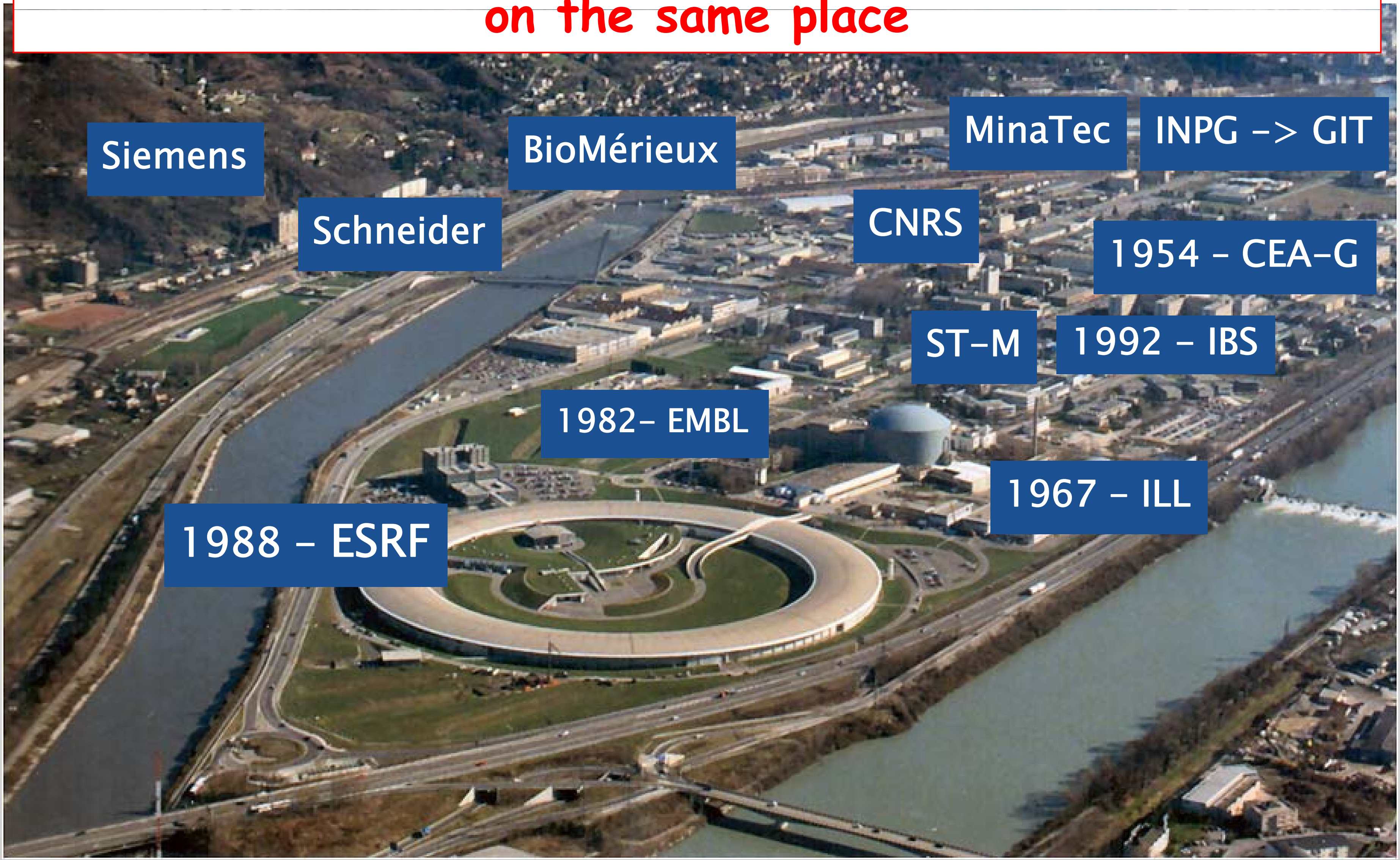


EMBL

ILL

(IBS)

Several scientific institutions and industrial enterprises on the same place



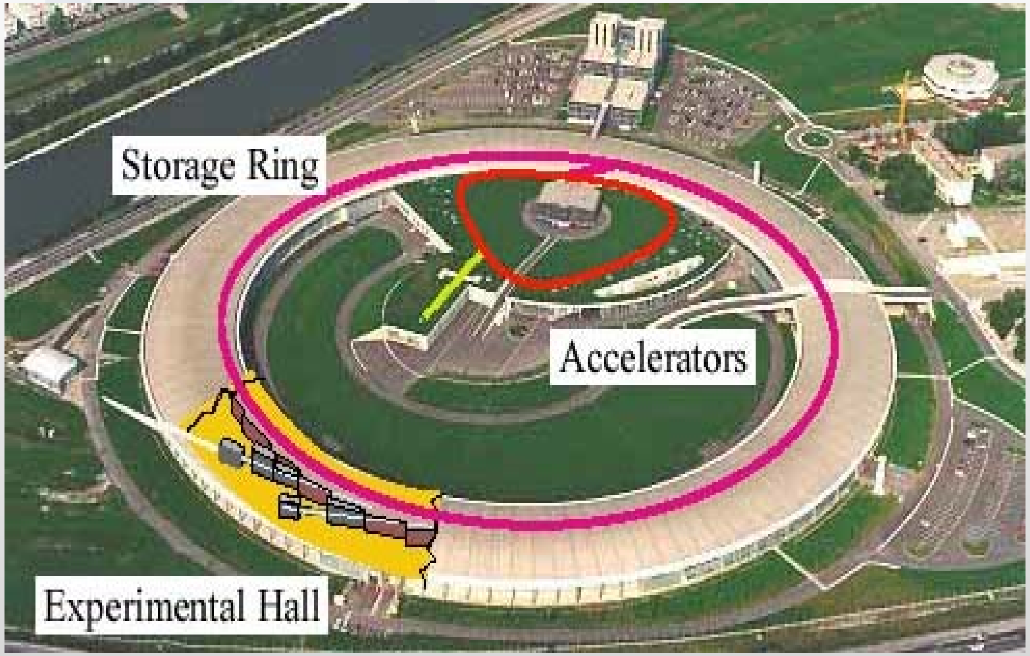
European Synchrotron Radiation Facility

- **1975** Project of a European third-generation synchrotron source

- **1988** Signature between the governments of 12 Member States.

- **1992** First electron beam in the storage ring. Commissioning phase.

- **1994** User operations with 15 beamlines



- **1998** 40 beamlines

- **2008** Upgrade Programme 2008-2017

Members and Scientific Associates

Contribution to ESRF budget (and share of beam time)

France	27.5%
Germany	25.5%
Italy	15%
UK	14%
Belgium/Netherlands	6%
Spain	4%
Switzerland	4%
Denmark/Norway/ Sweden/Finland	4%
	100%

Annual budget ~82 M€

Associated Countries	5.05%
Portugal	1%
Israel	1%
Austria	1%
Poland	1%
Centralsync	1.05%
(Hungary, Czech Republic, Slovakia)	

We are a user facility

In 2008

About:

2000 submitted proposals

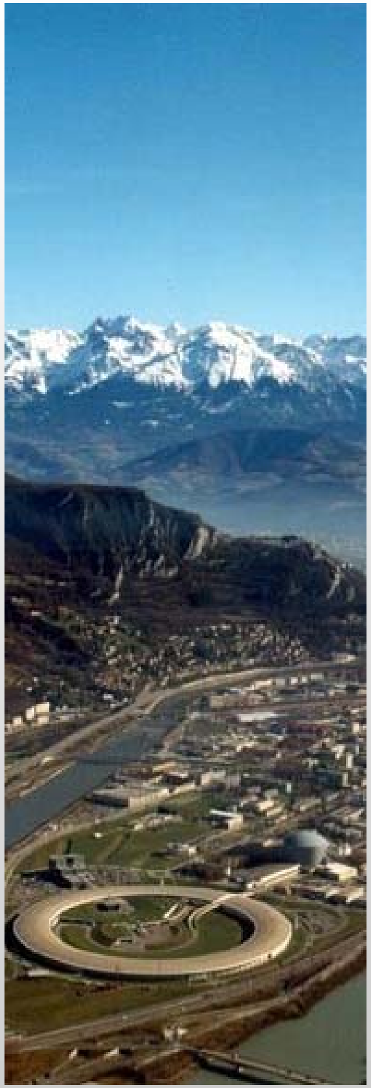
900 proposals allocated beamtime

31100 shifts (8h) of beamtime requested by applicants

13900 shifts allocated after peer review

17000 shifts scheduled for user experiments

6200 number of user visits

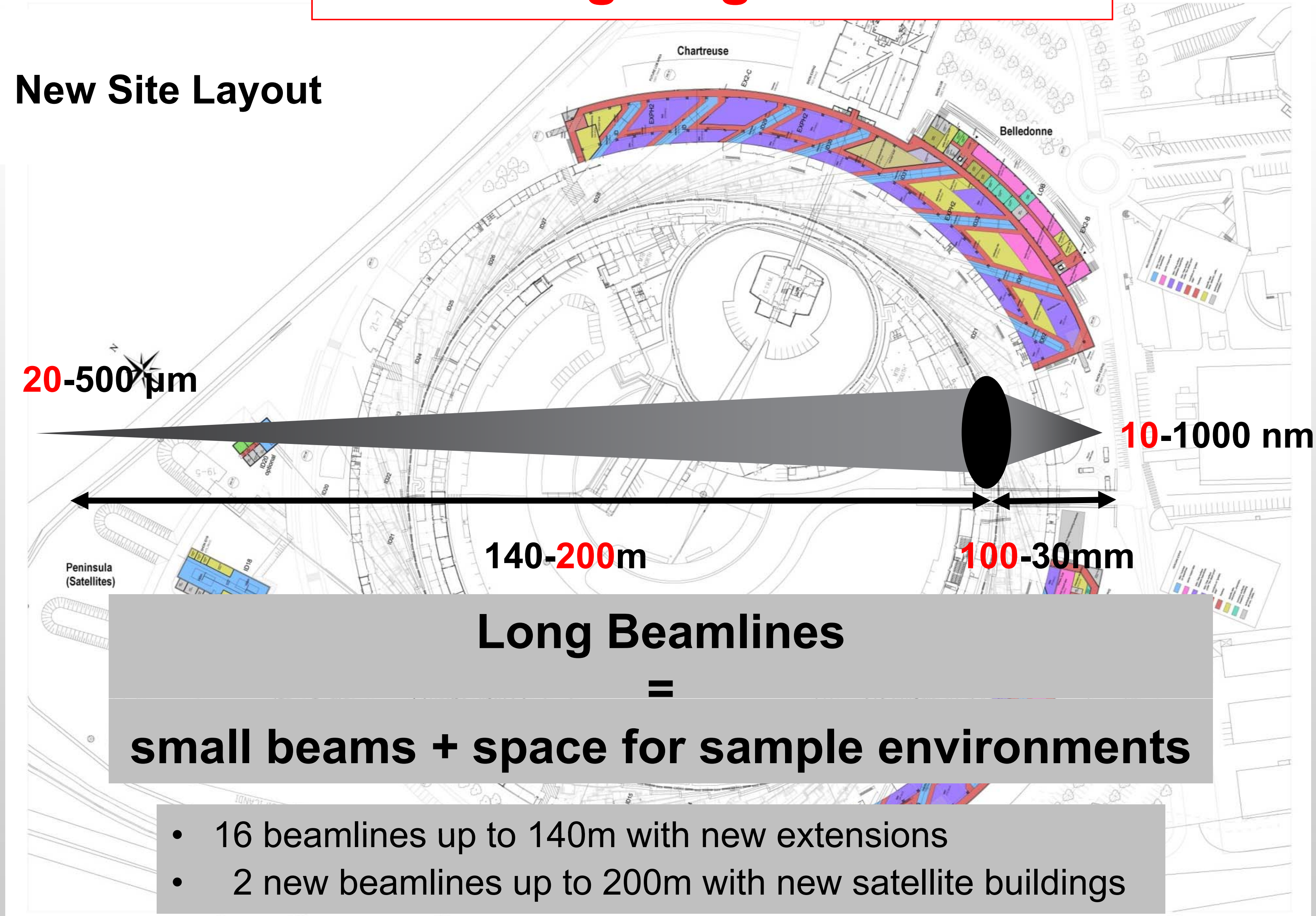


The ESRF Upgrade Programme

Five key components of the Upgrade

Enabling long beamlines

New Site Layout



Long Beamlines

=

small beams + space for sample environments

- 16 beamlines up to 140m with new extensions
- 2 new beamlines up to 200m with new satellite buildings

Optics Group

Group Head
Jürgen Härtwig

Crystal Laboratory	Mirrors and Metrology Laboratory	Multilayer Laboratory	Refractive Optics & MOTB
Responsible *Jürgen Härtwig*	Responsible *Ray Barrett*	Responsible *Christian Morawe*	Responsible *Anatoly Snigirev*
~ 1988	~ 1989	~ 1990	~ 2004

"Permanent" access to optics beamline BM05 as well as R&D and test beamline ID06

Why created?

Facilities for production, test and calibration were needed

X-ray optics

Tasks:

To transform the beam to obtain the best matching with the experiment – from source to sample to detector.

It acts on:

- shape
- wavelength/energy
- divergence
- polarisation

- slits, pinholes
- filters, windows
- mirrors
- monochromators (crystals, multilayers)
- beam splitters (crystal monochrom.)
- lenses (CRLs), Fresnel zone plates
- combined elements (ML gratings, Bragg-Fresnel lenses)
- phase plates (crystals)

Beam dimensions

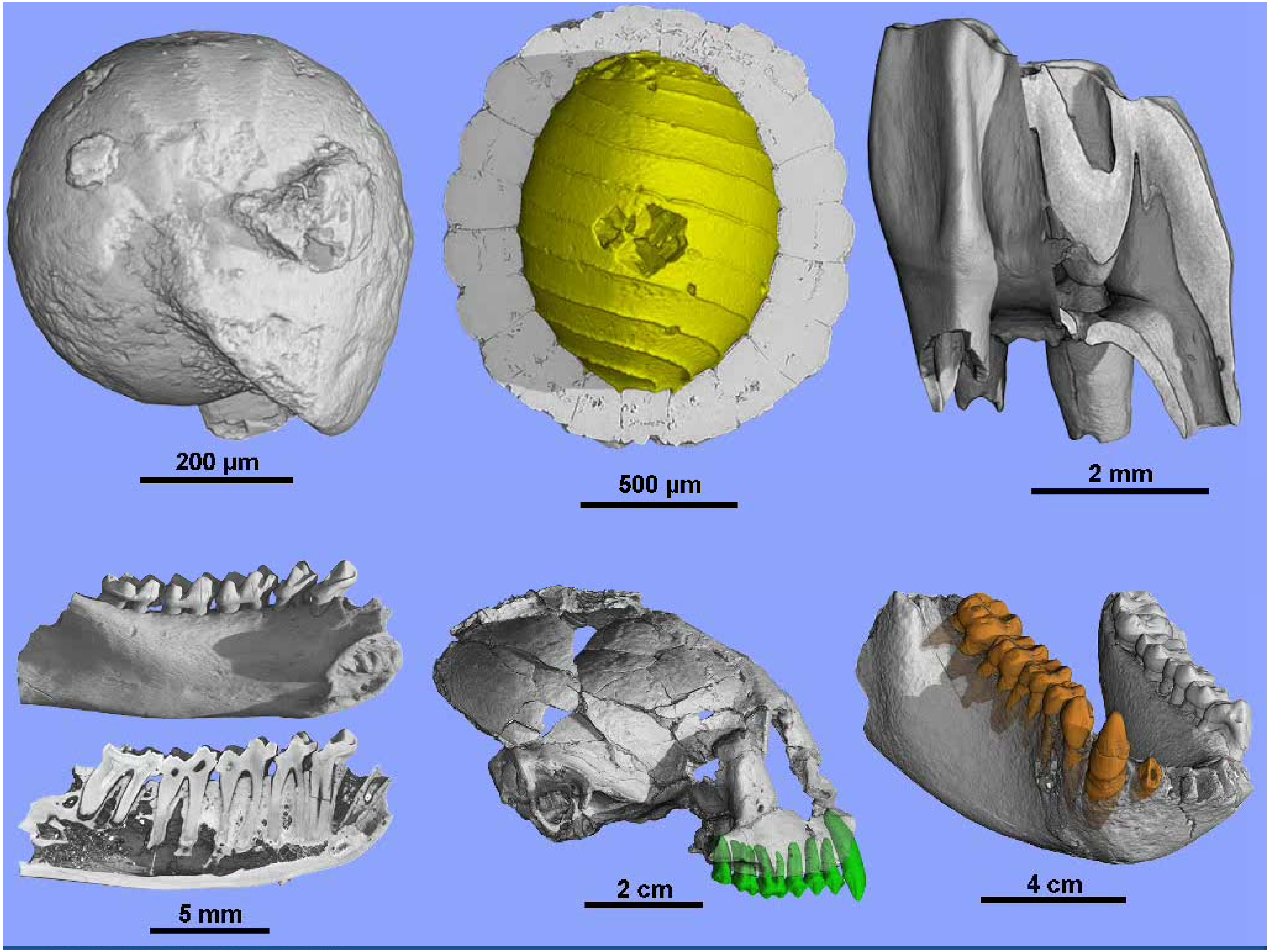
Few years ago – micro-beams were modern,

now – nano-beams are in vogue.

But - we need all kind of beams:

large beams (decimetre sized)
and small ones (nanometre sized),

"parallel", divergent and focussed beams.

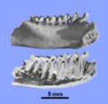

200 µm
500 µm
2 mm
5 mm
2 cm
4 cm

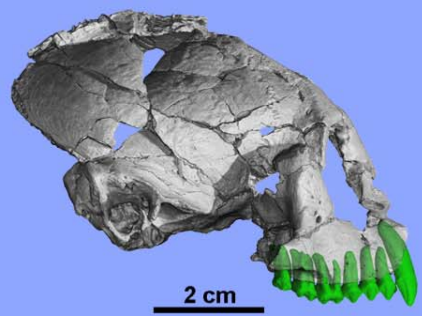
Nearly 4 orders of magnitude in dimension

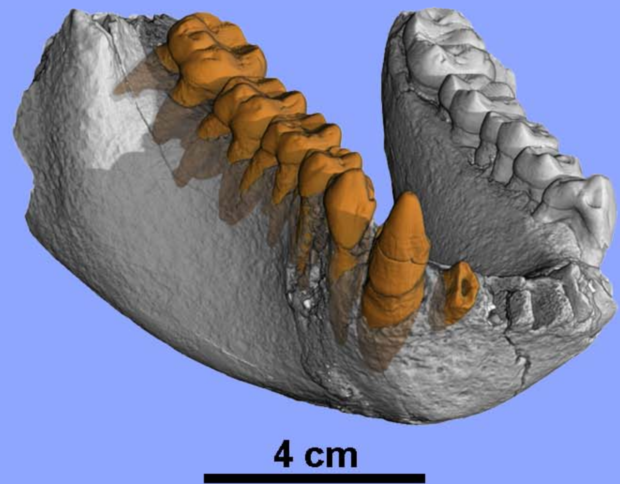
Without scanning

Without magnification

Multi scale experiments!







Examples from Paul Tafforeau

Example of effective pixel sizes of the FReLoN cameras used for parallel beam imaging in the X-ray imaging group

280nm to 30µm
2 orders of magnitude

FReLoN camera			Optics (µm)										Scan Size
			0.28	0.7	1.4	2.8	5.06	5.4	7.5	8.06	20	30	
			ID19	ID19	ID19	Pool	ID19	BM05	ID19	ID17	ID19	ID19	
2k14 14 µm	no binning	Pixel size (µm)	0.28	0.70	1.40	2.80	5.06	5.40	7.46	8.06	20.00	29.47	13 Go (1500 views)
		Field of view (mm)	0.57	1.43	2.87	5.73	10.36	11.06	15.28	16.51	40.00*	40.00*	
	binning	Pixel size (µm)	0.56	1.40	2.80	5.60	10.12	10.80	14.92	16.12	40.00	58.94	2 Go (900 views)
		Field of view (mm)	0.57	1.43	2.87	5.73	10.36	11.06	15.28	16.51	40.00*	40.00*	
			High resolution microtomograph				Medium Resolution microtomograph						

*Limited by ID19 beam size

Some properties of crystalline materials for X-ray optics

Material	Be	C*	Si	Ge
Atomic number, A	4	6	14	32
Integrated reflectivity 111-reflection, 8 keV	2.0	6.3	8.2	18.0
Absorption coefficient, μ, at 8keV (cm^{-1})	1.8	7.5	141	402
Thermal conductivity, κ, at 297K, ($Wcm^{-1}K^{-1}$)	1.9	I: 5-18 IIa: 23 Isotop. pure: 35	1.5	0.64
Thermal expansion coefficient, α, at 297K, ($10^{-6}K^{-1}$)	7.7	1.1	2.4	5.6
Figure-of-merit, $\kappa/\mu/\alpha$, at 297K, (kW)	140	2780	4.4	2.8

Silicon is the most used crystalline material

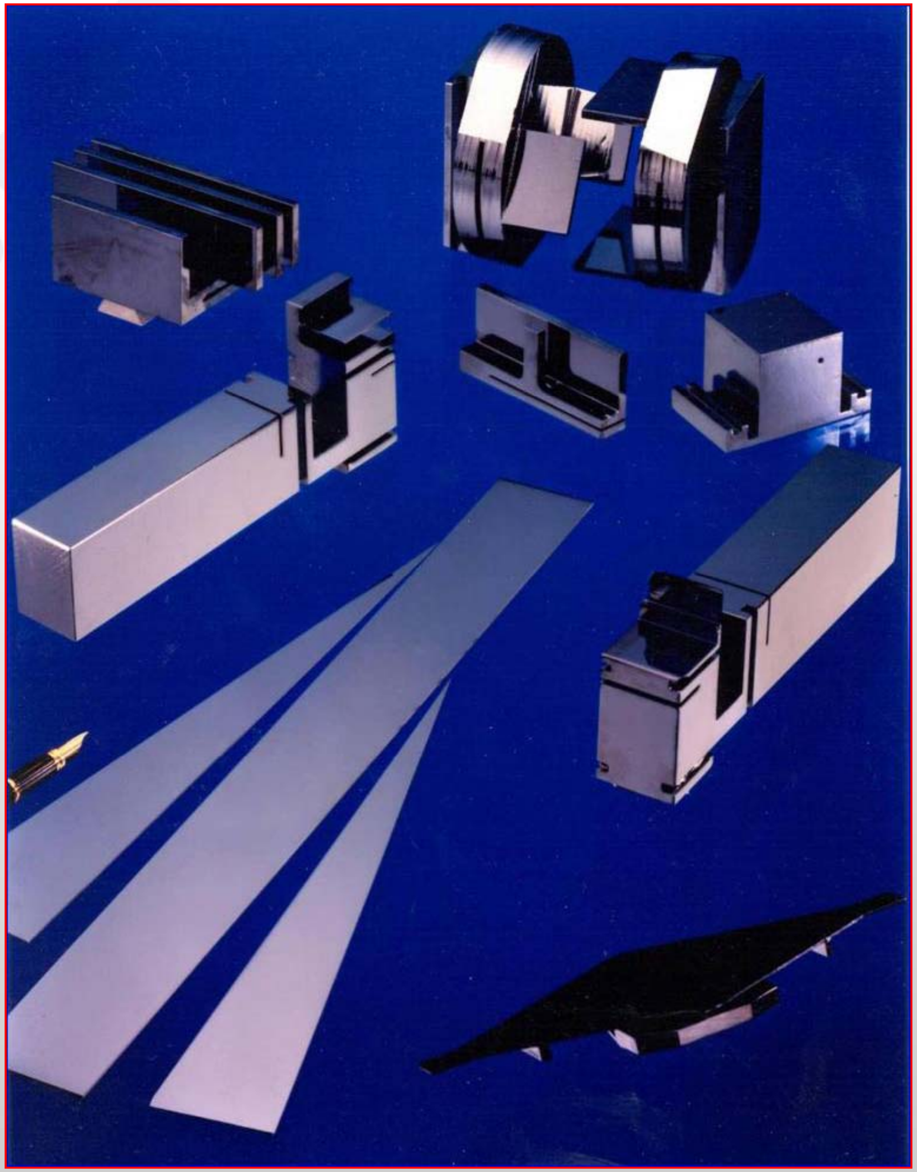
However, also diamond is used from the very beginning of the ESRF, but less

Crystal laboratory:

Manufacturing of nearly all **perfect crystal monochromators & analysers, etc** for all ESRF beam lines, CRG beamlines and external laboratories.

Most crystals are made from top purity **silicon float zone** ingots 100 mm in diameter (Wacker).

More than **1.5 tons of silicon** single crystal material has been processed in about **twenty years.**



Example manufacturing crystal monochromators and analysers

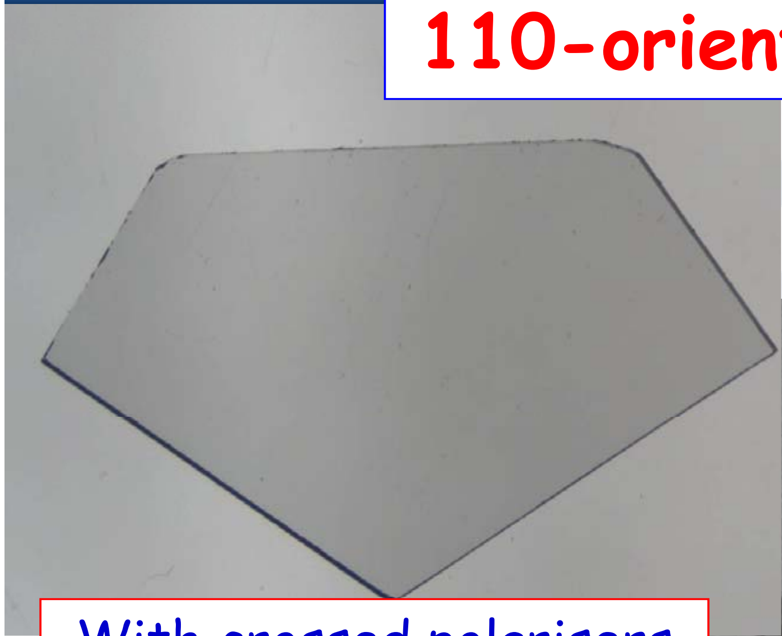
What is now available and used for which applications

p-CVD diamond	filters, windows
nc-diamond	(coming up)
sc-CVD diamond	detectors, maybe phase plates, not (yet) for monochromators
HPHT, type Ib	used for, but problematic as beam splitters, monochromators; lower quality phase plates
HPHT - type IIa	highest quality!, monochromators, beam splitters, high-quality phase plates; substrates for better CVD growth to have better layers for detector applications

HPHT IIa

Paul Balog/Element Six

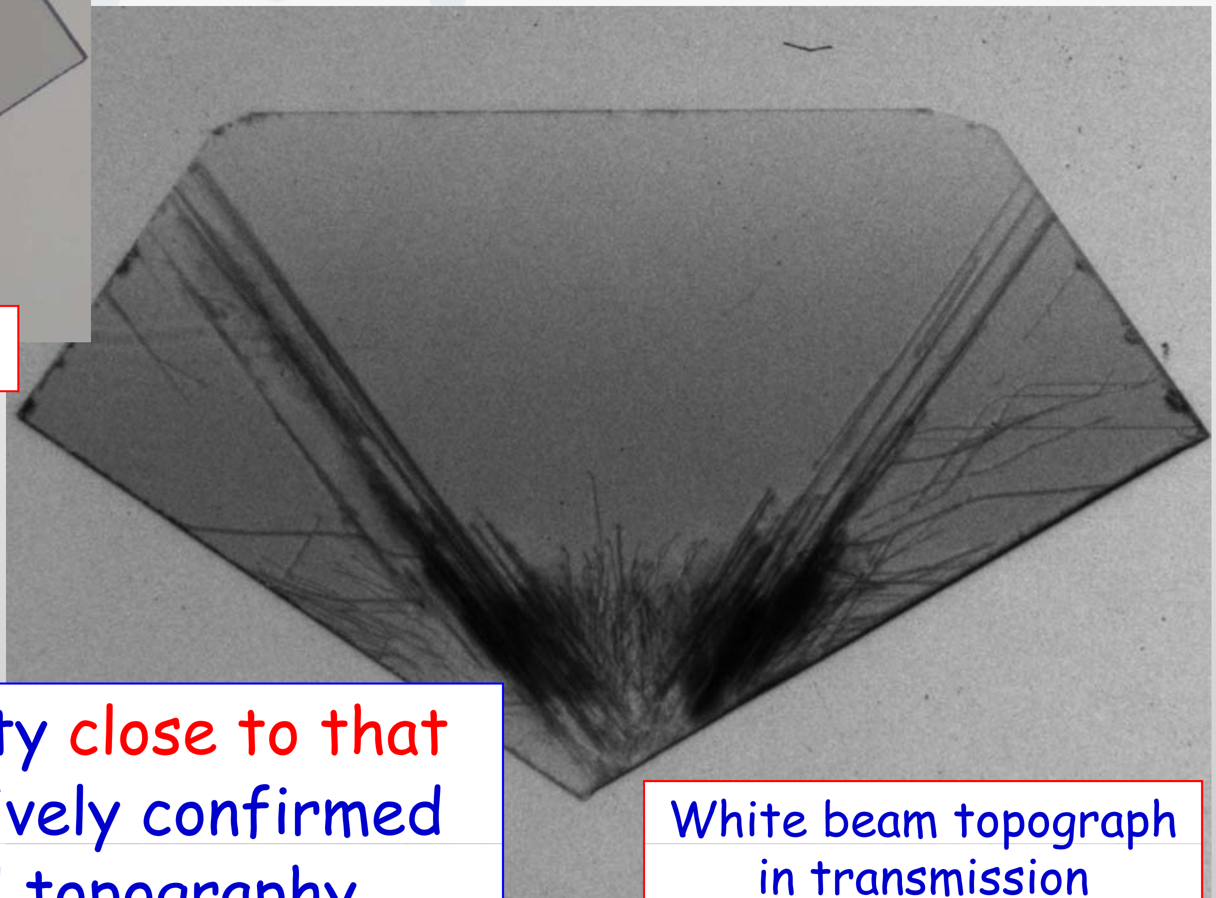
110-oriented plate



With crossed polarisers

Dislocation free areas of 6x4mm^2 and more!!!

11.3x6.5x0.70



White beam topograph in transmission

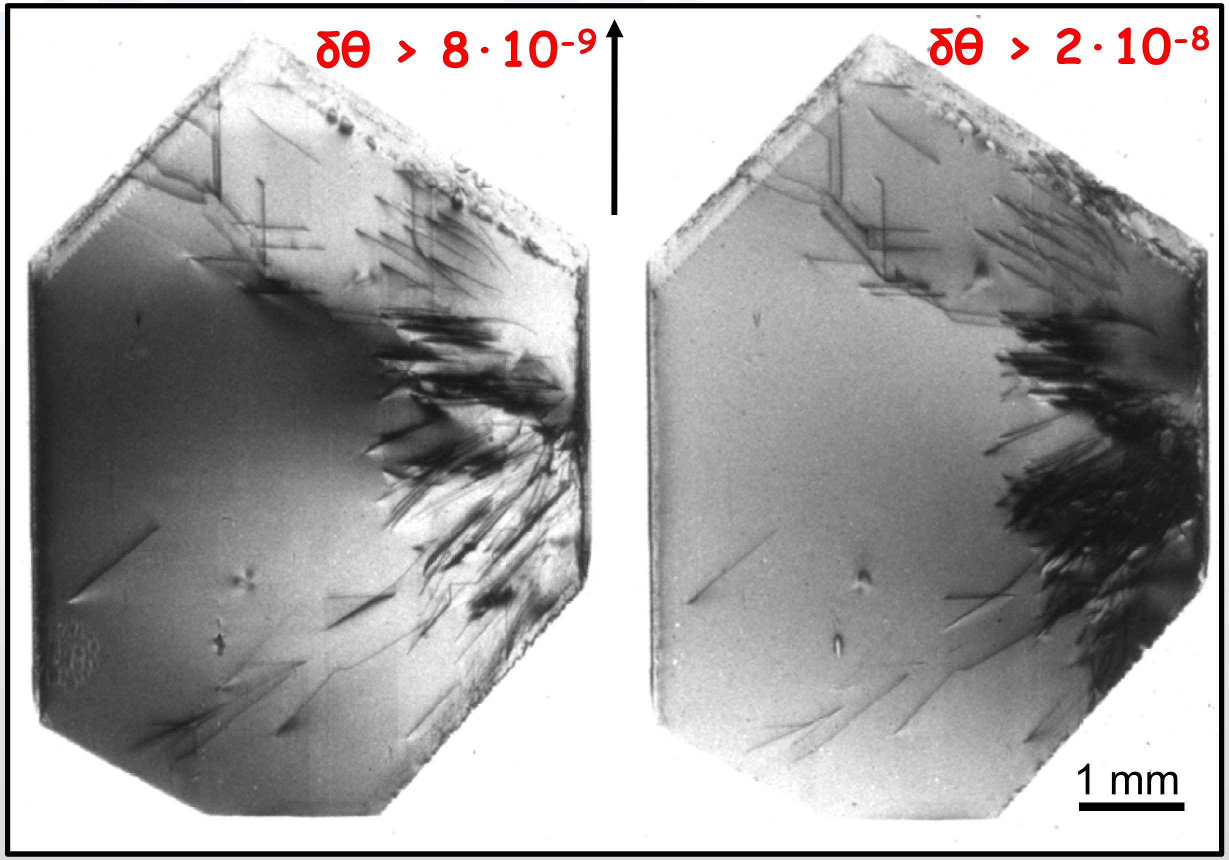
Locally crystal quality close to that of silicon, quantitatively confirmed by double crystal topography

Increasing strain sensitivity

110-oriented crystal plate

Element Six recent delivery

bent silicon 880, diamond 660 reflections 20 keV.



X-ray topographs taken with a non-dispersive setup collected with the bendable monochromator. Bragg case.
Left: working point on the **steeper low angle side** of the rocking curve.
Right: working point on the **less steep high angle side**.

Quantitative analysis basing on one topograph

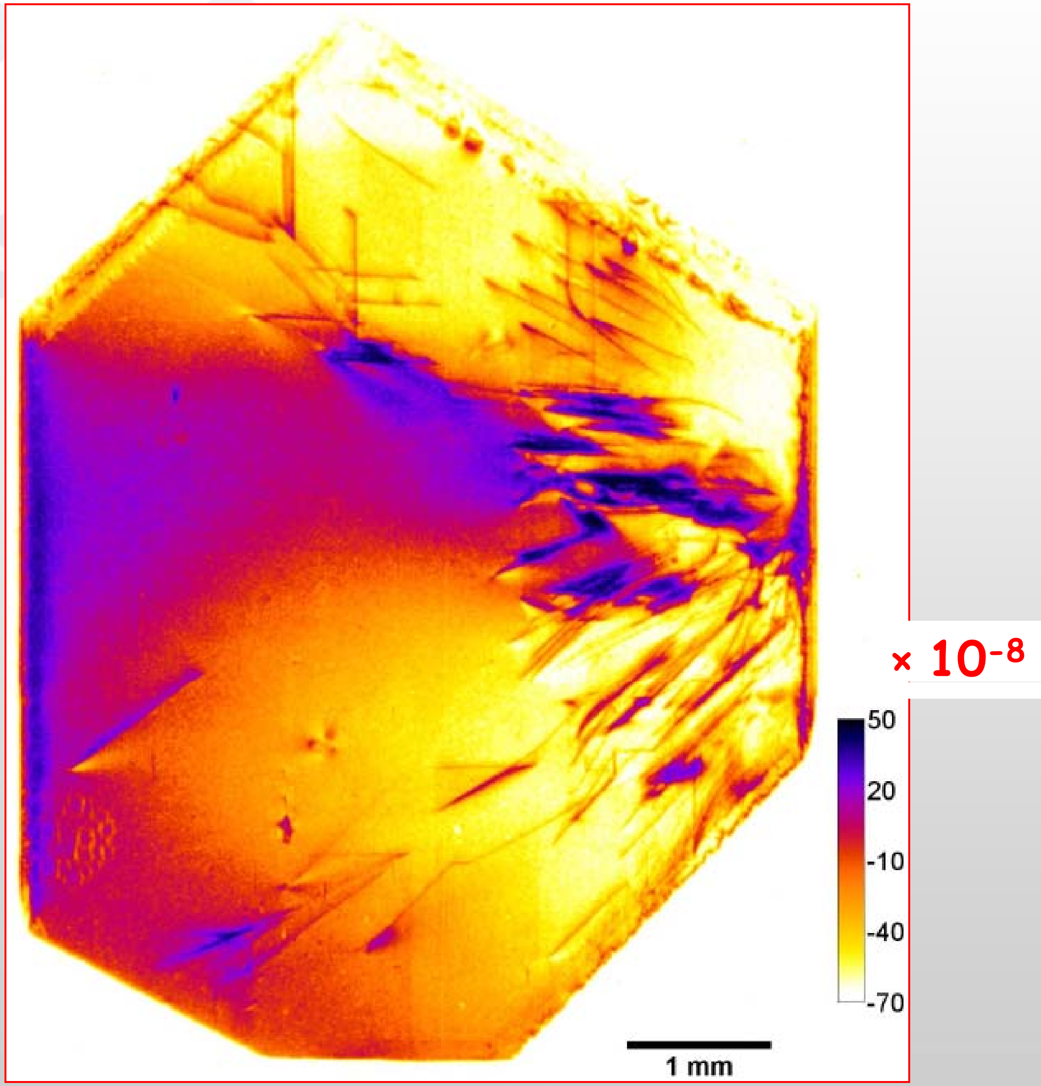
110-oriented crystal plate

effective misorientation map based on one topograph

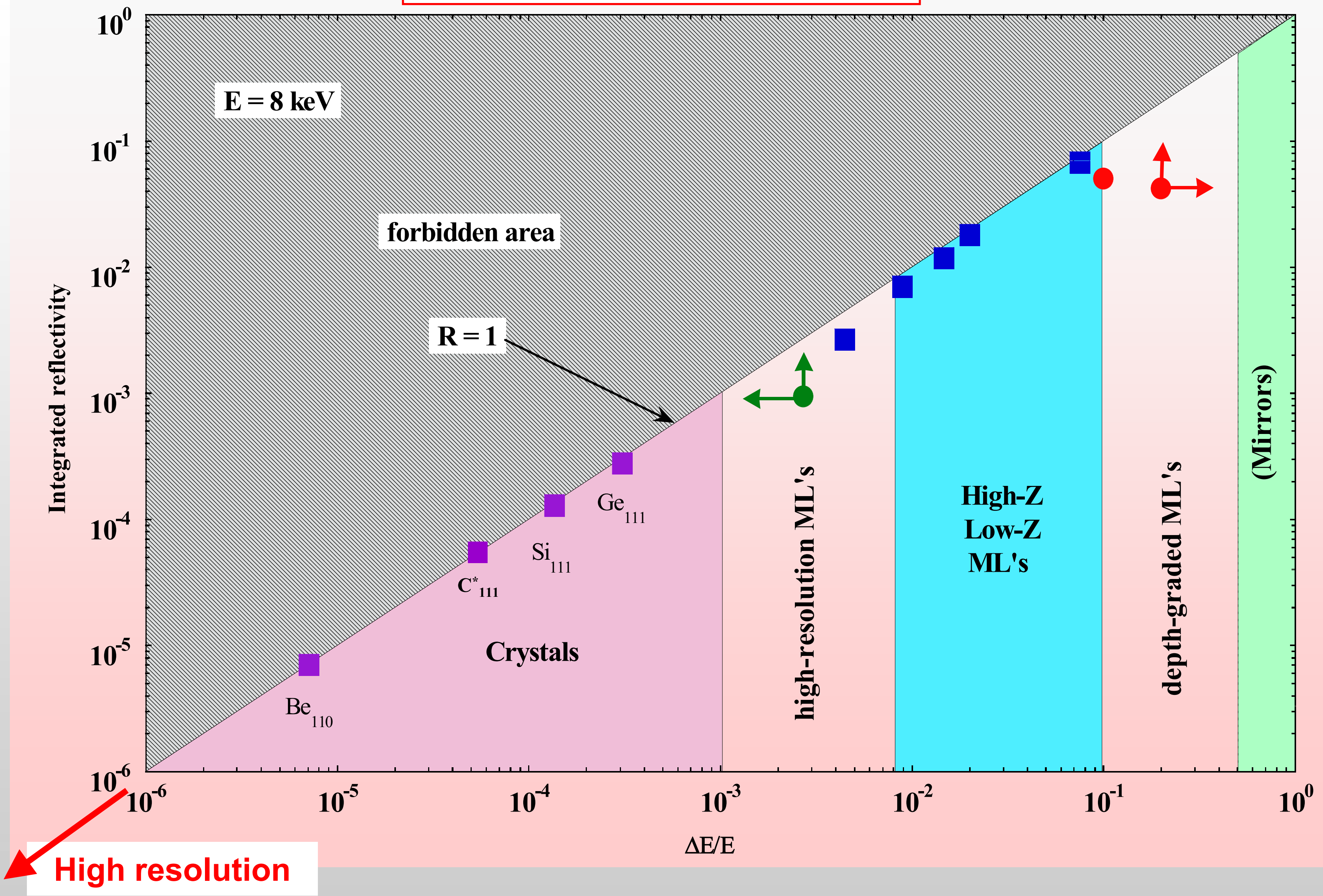
20keV, Si [880] C* [660],
$\delta\theta > 8 \cdot 10^{-9}$

The effective misorientation is of the order of 4×10^{-8} for a region of interest of 0.5×0.5 mm^2 and 1×10^{-7} in a region of 1×1 mm^2

Similar like in 100-plate
Sample is slightly bent!



Monochromators



Ch. Morawe

Pink beam setups for absorbing samples

Simplest "monochromator" – no monochromator

but using only: filters, source spectrum, scintillator screen response spectrum

BM05 data

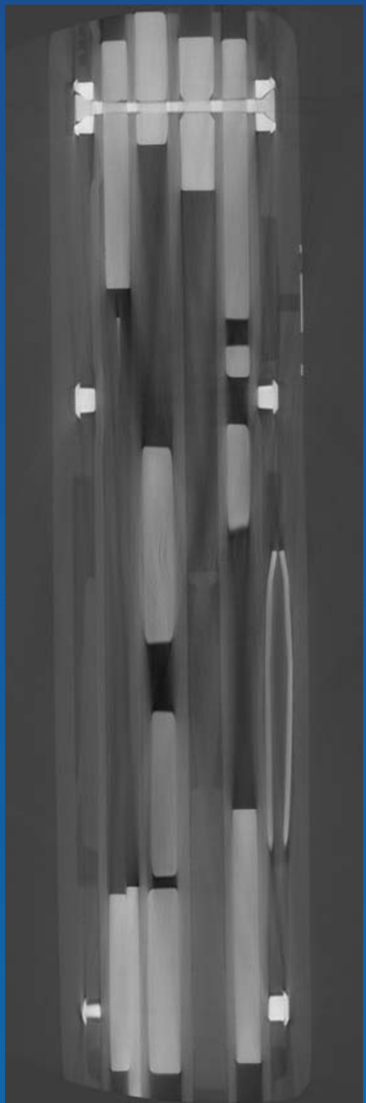
Pink beam setups for absorbing samples

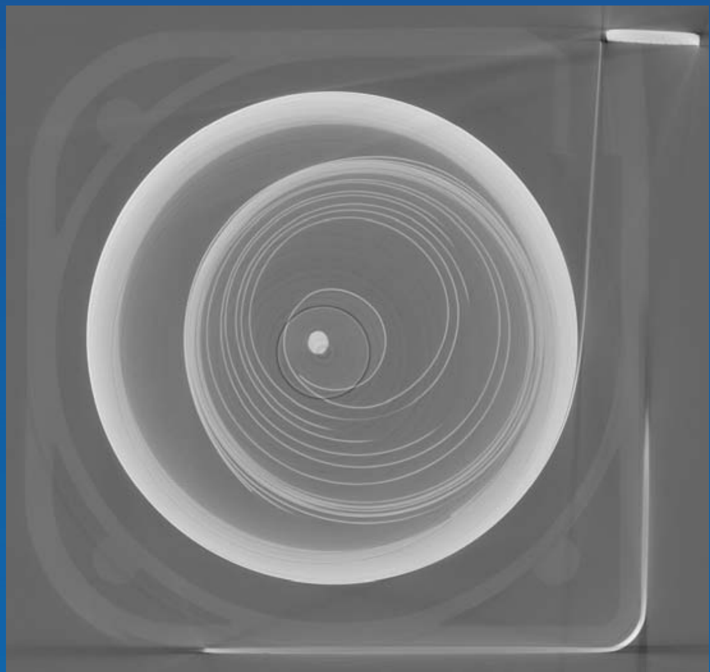
BM05 with scans performed in 3-4 minutes in high energy pink beam. For the phone and the meter, the energy was around 70 keV and for the knife, it was around 140 keV.

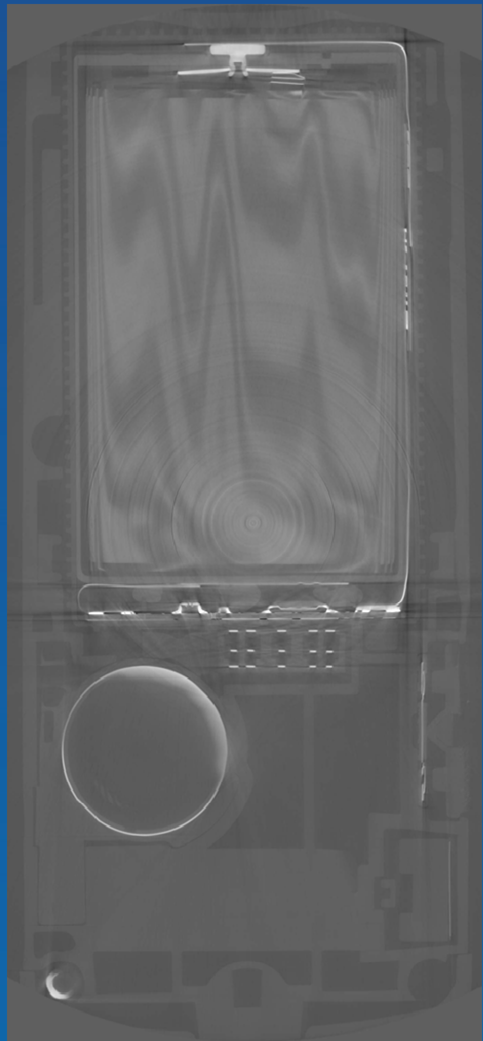
BM05 data

Pink beam setups for absorbing samples

BM05 with scans performed in 3-4 minutes in high energy pink beam. For the phone and the meter, the energy was around 70 keV and for the knife, it was around 140 keV.







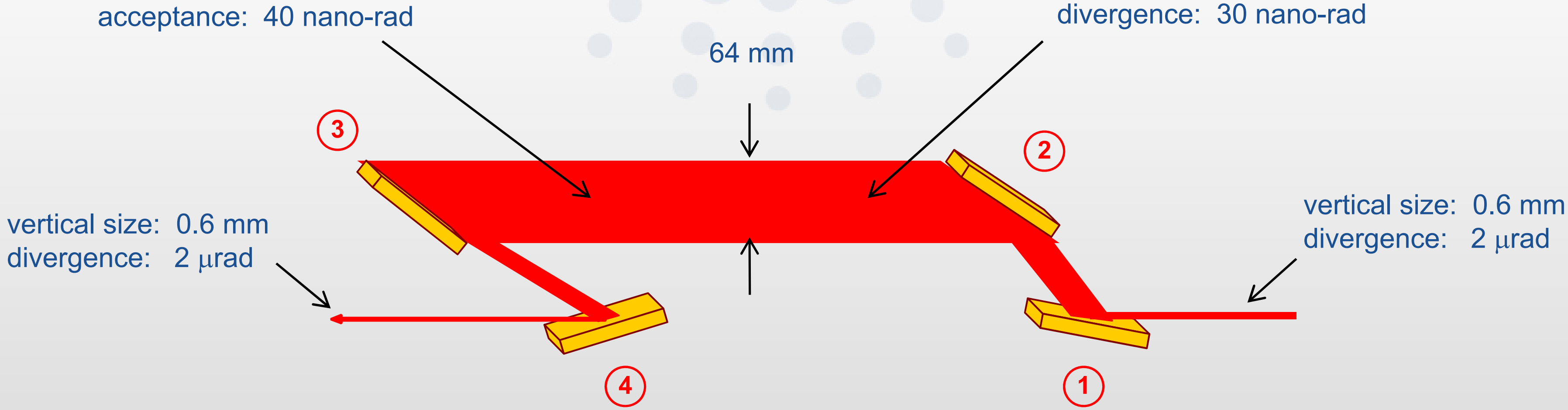
BM05 data

High-resolution optics for Nuclear Resonance Scattering

"0.2 meV" monochromator:

(theoretically expected performance)

Alexander Chumakov
ID18/ESRF



1st crystal:
Si(400)
$\Theta_B = 18.469^o$
asymmetry: $|b| = 0.13$
$\Theta_{in} = 3.9^o$
$\Theta_{out} = 33^o$
angular acceptances:
$\Delta\Omega_{in} = 23\ \mu rad$
$\Delta\Omega_{out} = 3\ \mu rad$
footprint: 9 mm

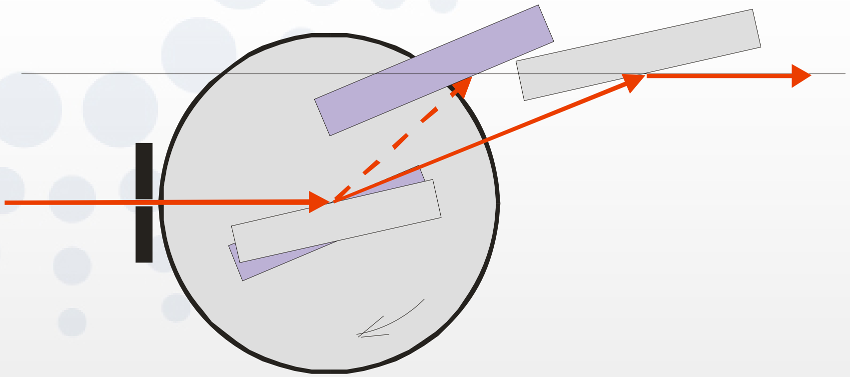
2nd crystal:
Si(400)
$\Theta_B = 18.469^o$
asymmetry: $|b| = 0.13$
$\Theta_{in} = 3.9^o$
$\Theta_{out} = 33^o$
angular acceptances:
$\Delta\Omega_{in} = 23\ \mu rad$
$\Delta\Omega_{out} = 3\ \mu rad$
footprint: 71 mm

3rd crystal:
Si(12 2 2)
$\Theta_B = 77.533^o$
asymmetry: $|b| = 25$
$\Theta_{in} = 25.9^o$
$\Theta_{out} = 1.0^o$ ($\Theta_c = 0.125^o$)
angular acceptances:
$\Delta\Omega_{in} = 0.45\ \mu rad$
$\Delta\Omega_{out} = 11\ \mu rad$
footprint: 88 mm

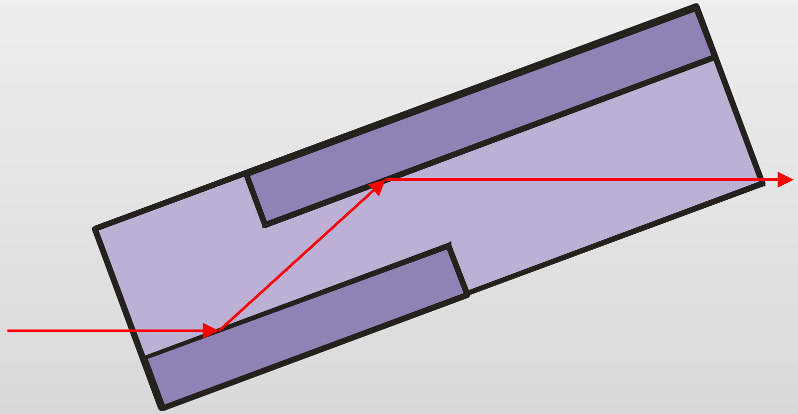
4th crystal:
Si(12 2 2)
$\Theta_B = 77.533^o$
asymmetry: $|b| = 5$
$\Theta_{in} = 30.8^o$
$\Theta_{out} = 5.9^o$
angular acceptances:
$\Delta\Omega_{in} = 1\ \mu rad$
$\Delta\Omega_{out} = 5\ \mu rad$
footprint: 3 mm

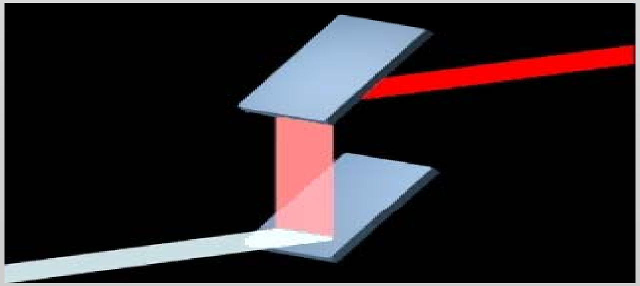
Crystal monochromators

- Fixed exit double crystal monochromator
- - more then one movement necessary



- Channel-cut monochromator
- - NOT fixed exit
- - naturally aligned
- - weak link plus piezo movement for detuning etc.



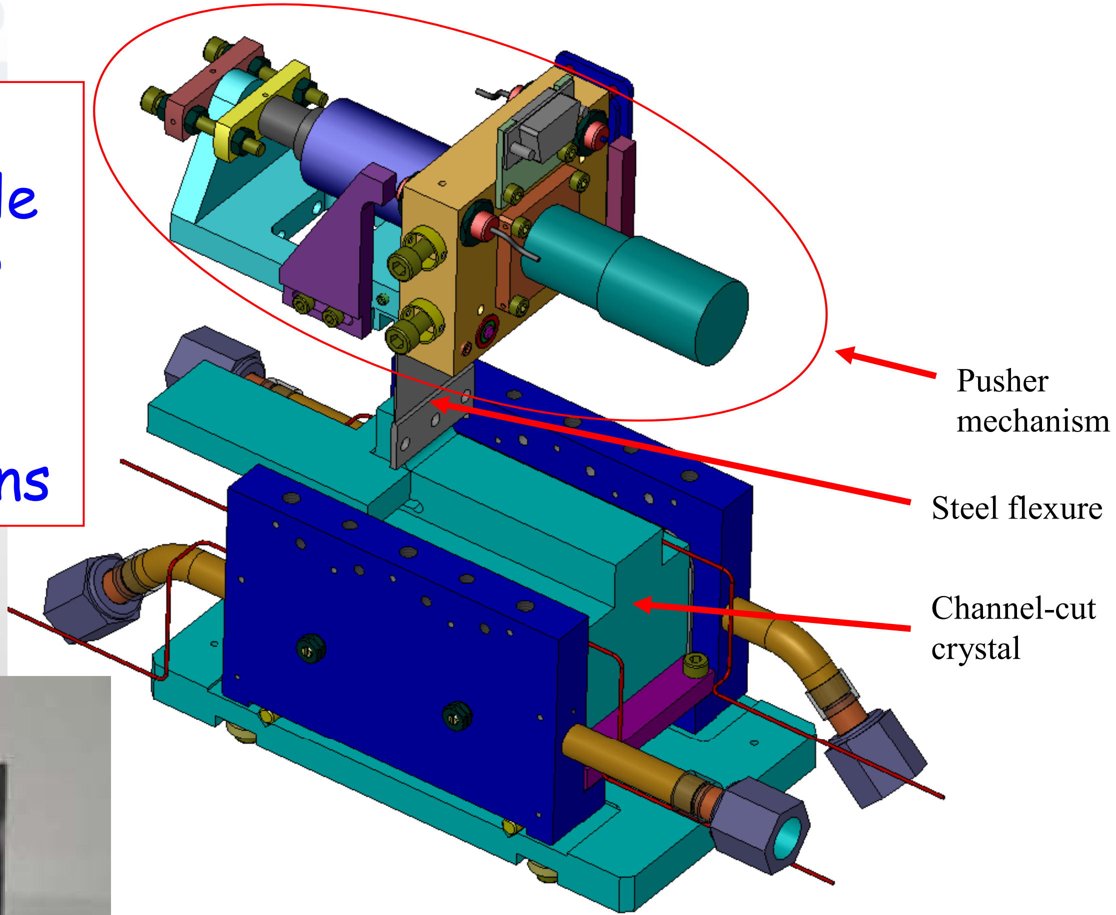


Crystal monochromators

"Generic" cryogenically cooled channel-cut double crystal monochromator

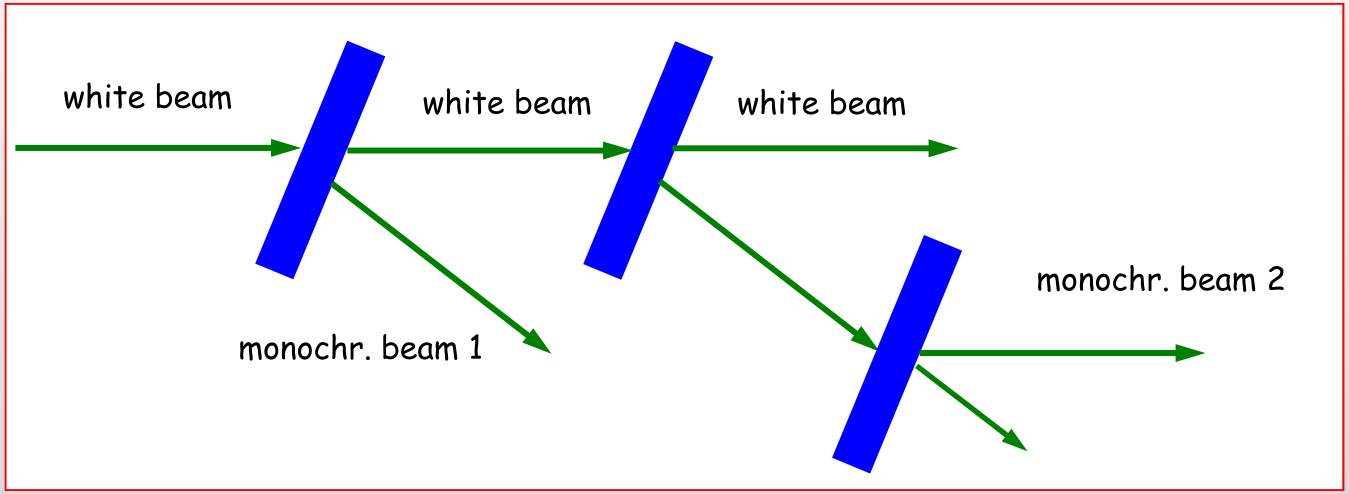
no fixed exit

high heat-load applications



Crystal assembly (simplified for visibility)

beam splitter monochromators



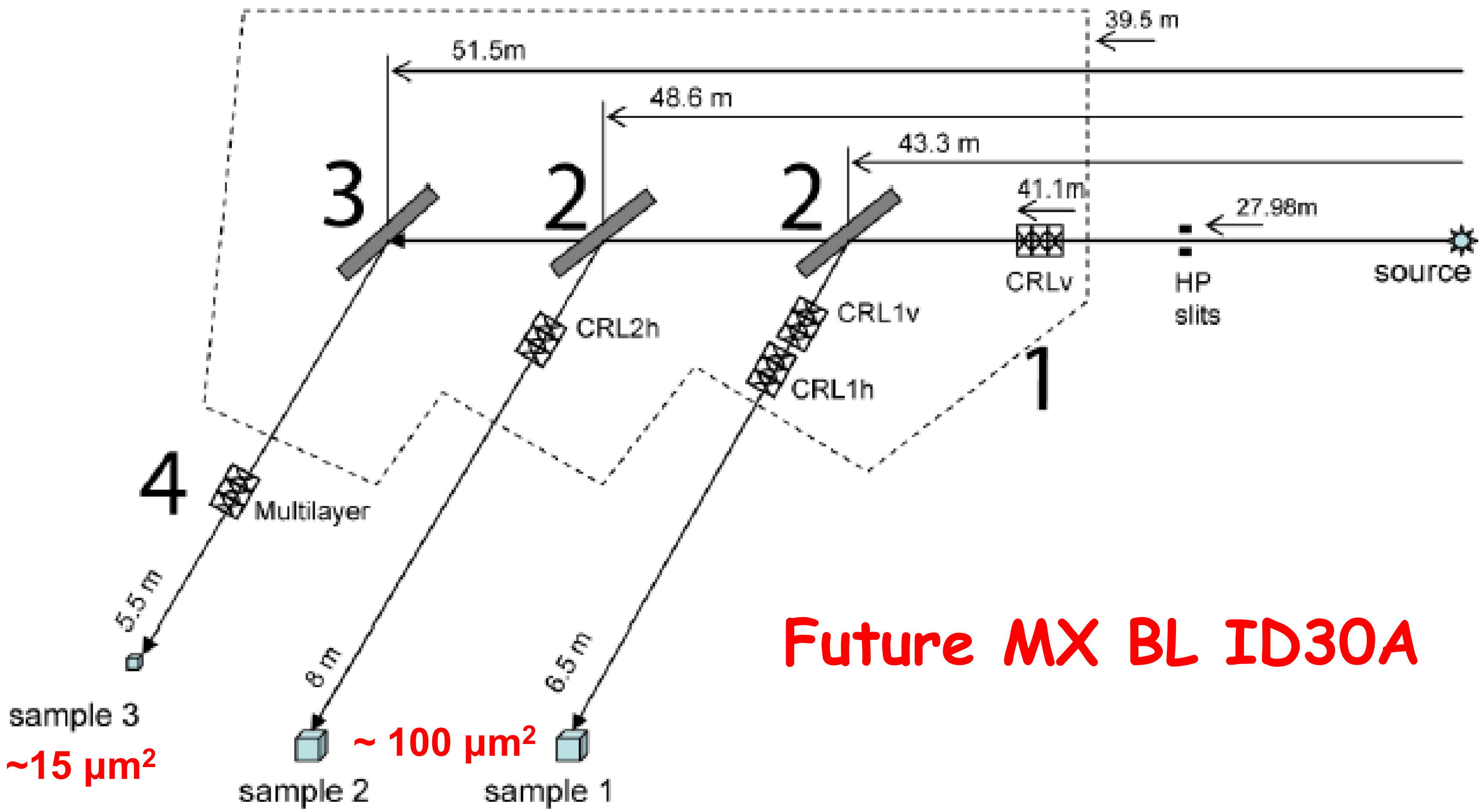


Figure 2.7. Layout of the optical elements of ID30A in OH2. (1) represents a vertically focusing white beam Compound refractive lens (CRL), (2) (111)-cut diamond monochromators, (3) a Si(111) monochromator and (4) horizontal focusing multilayer. The high power primary slits are located in the first optical hutch (OH1).

Two-dimensional versus

two times one-dimensional focussing

Source often very asymmetric:

horizontal dimension >> vertical dimension

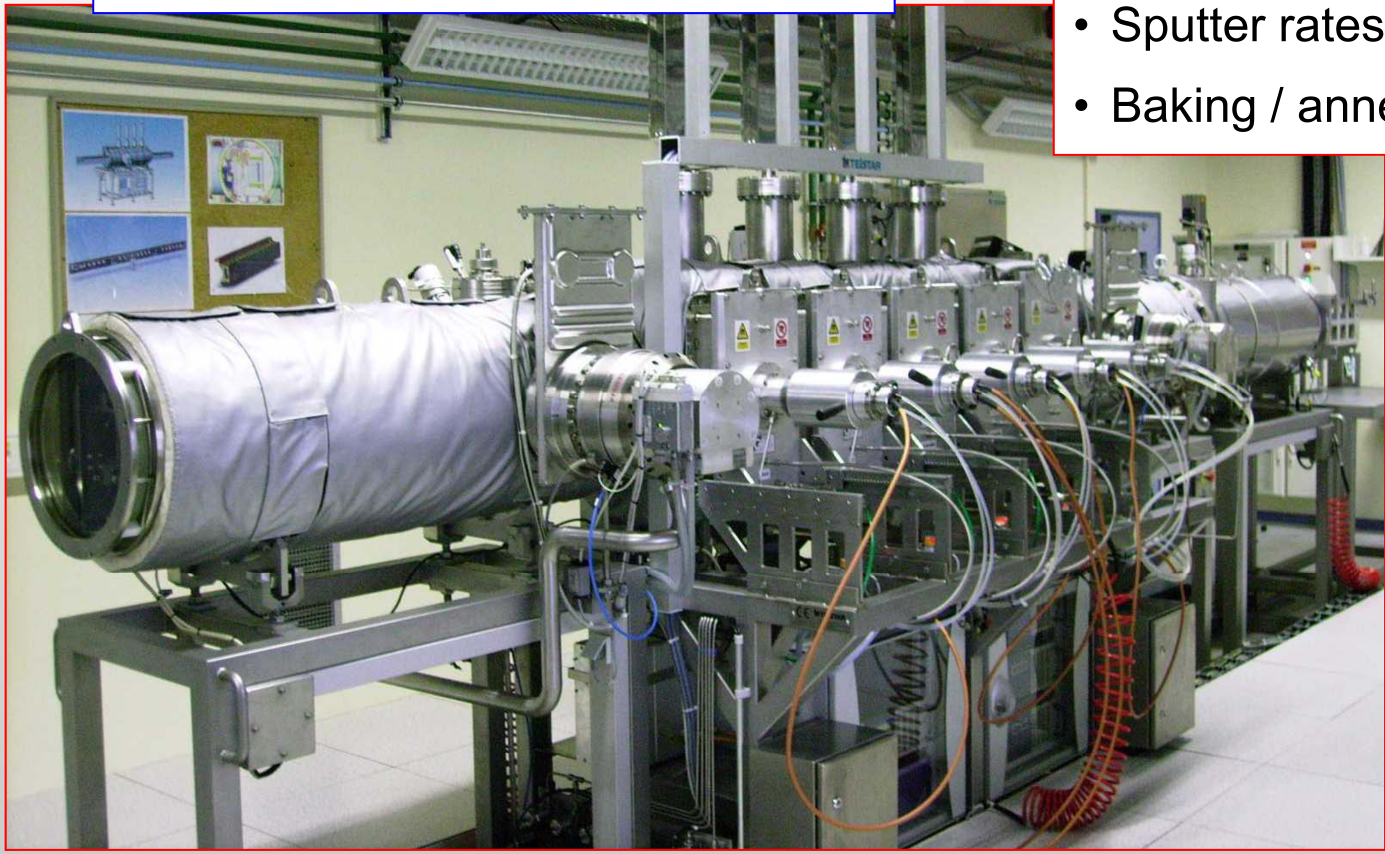
Multilayer laboratory:

Manufacturing:

single layer mirrors,
multilayer monochromators,
focusing optics

New machine since 2008

- Magnetron sputtering
- 4 fixed sources
- Moving substrates
- Max size: 1000x150x100 mm^3
- Sputter rates: 0.1…0.5 nm/s
- Baking / annealing furnace

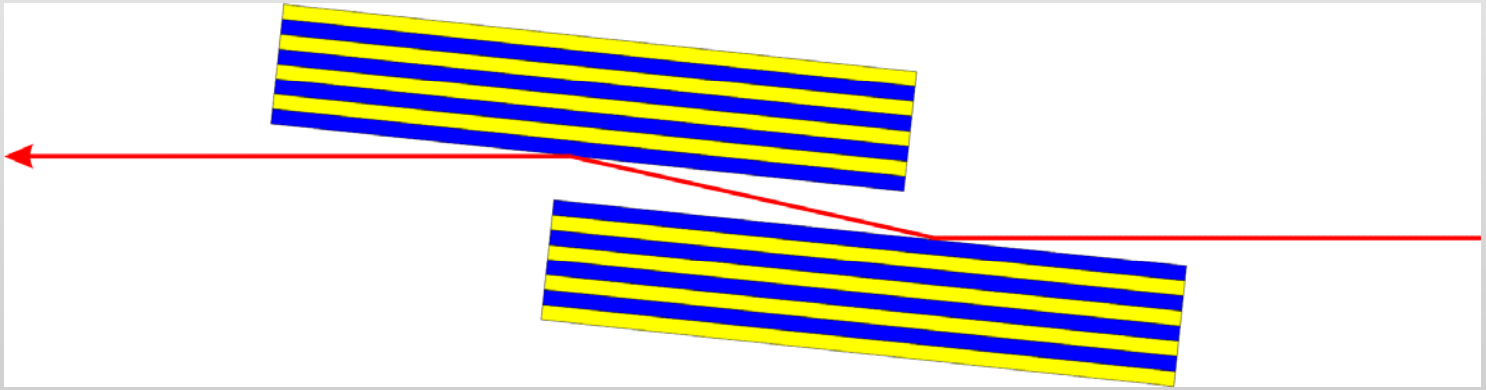


Ch. Morawe

ML monochromators

Multilayer high flux double monochromators (white beam)

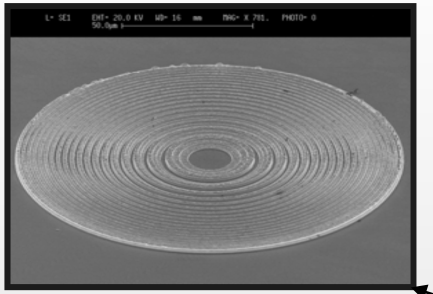
- Two bounce optics
- 100x larger bandwidth compared with Si(111) → ~100x more flux
- Radiation and heat load issues !



Collimation, focussing, magnification

X-ray focussing - several ways/devices

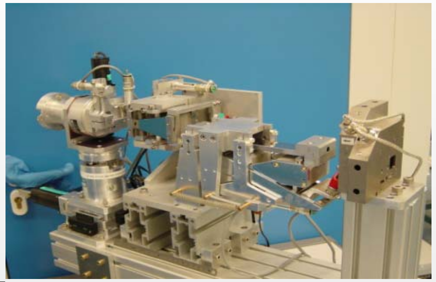
Diffractive "lenses"

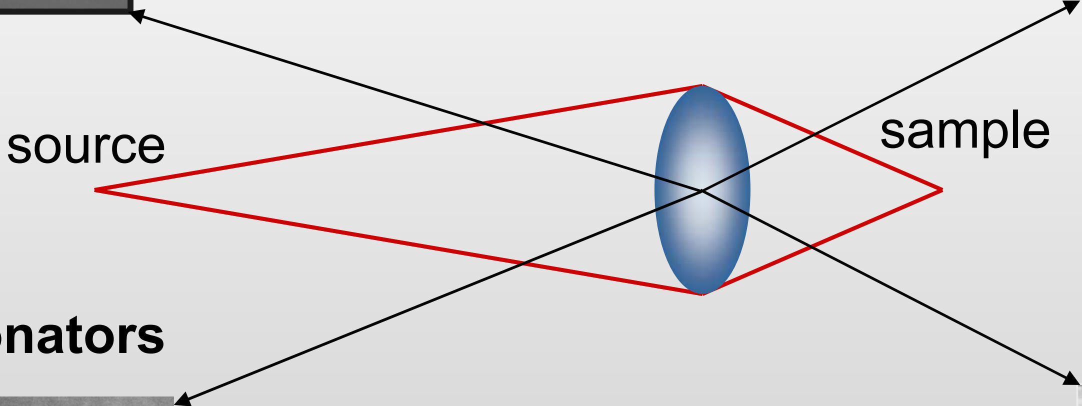


- *Fresnel zone-plate*
- *Bragg-Fresnel*
- *Crystals*
- *Multilayers*

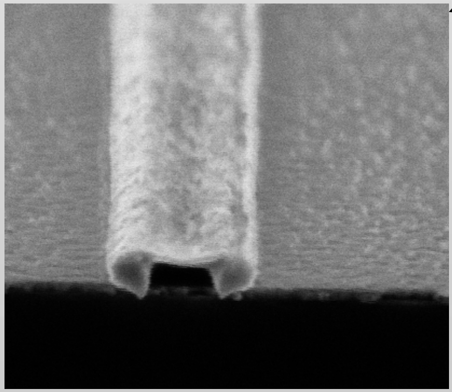
X-ray Reflectors

- *Kirkpatrick-Baez*
- *Wolter mirror*
- *Ellipsoidal mirror*
- *Micro-channel*
- *(Poly)capillaries*





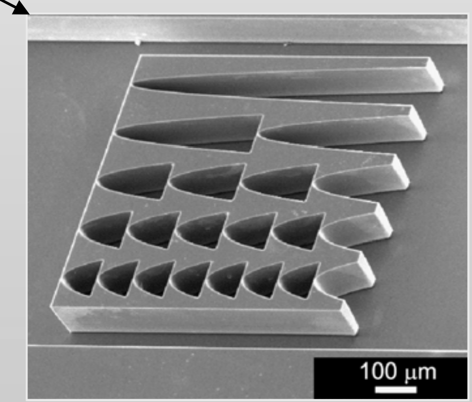
X-ray resonators



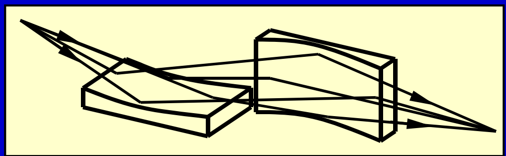
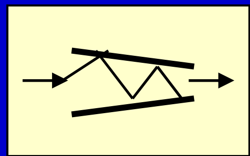
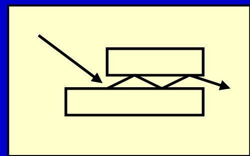
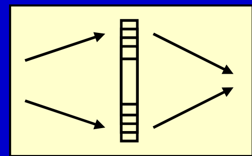
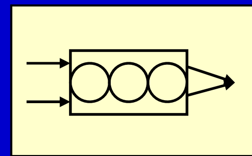
- *Waveguides*

Refractive lenses

- *Compound refractive lenses*



Focusing Optics for Hard X-rays (E > 6 keV)

	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
						
Energy	< 30 keV	< 80keV	< 20keV	< 20keV	< 30 keV (80)	<1 MeV
Bandwidth $\Delta E/E$	w. b.	10^{-2}	w.b.	10^{-3}	10^{-3} - 10^{-4}	10^{-3}
resolution	**25 nm** @20keV **Mimura 2009** **7 nm**	**$41x45nm^2$** @24keV Hignette 2006	**50 nm** Bilderback 1994	**$40x25\ nm^2$** Salditt 2004	30 nm @20 keV **Kang, 2006** **17 nm, 2007**	**50 nm**@20keV Schroer, 2004 **150nm**@50keV Snigirev,2006

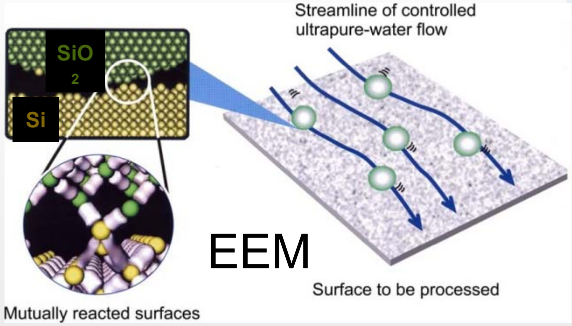
1-D!   2-D 50-80nm

17 nm ML Laue lense

A. Snigirev

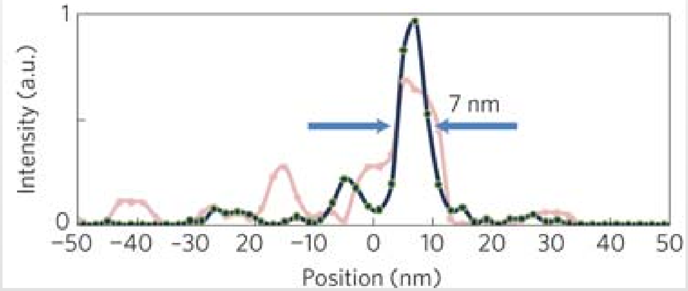
Current best focusing performance

Deterministic Polishing

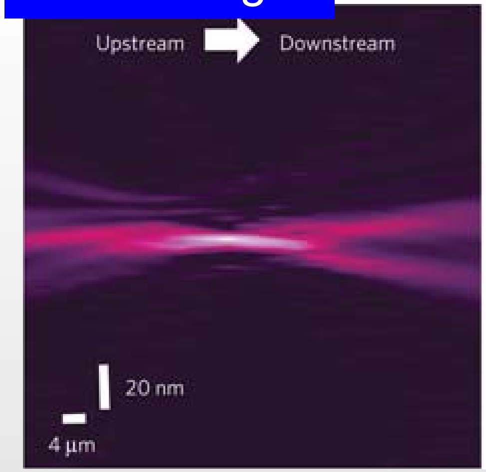


Advanced Metrology Techniques

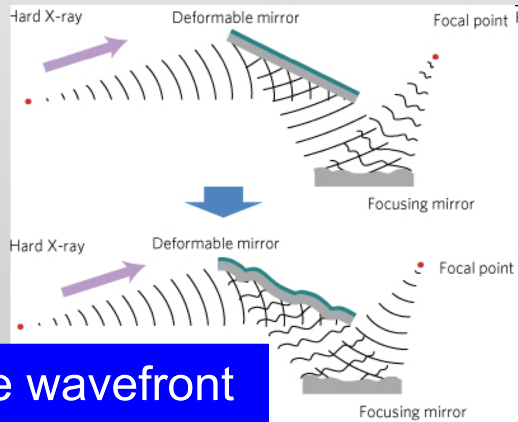
20keV: 7 nm measured focal spot



Wave-optical Modeling



Coherent illumination



Active wavefront correction

H. Mimura et al., "Breaking the 10 nm barrier in hard-X-ray focusing," *Nat Phys* **6**, (2010): 122-125

Present top and standard values

1-dim: mainly for demonstration

top value 7nm (1/2 KB)

2-dim: 2 to 3 times smaller

For real "routine" work 30-80nm

But not only spot size is important,

photon flux in the spot is crucial (scanning time)

$10^7 s^{-1}$ poor, $10^{12} s^{-1}$ fine

More or less all systems are used at ESRF beamlines

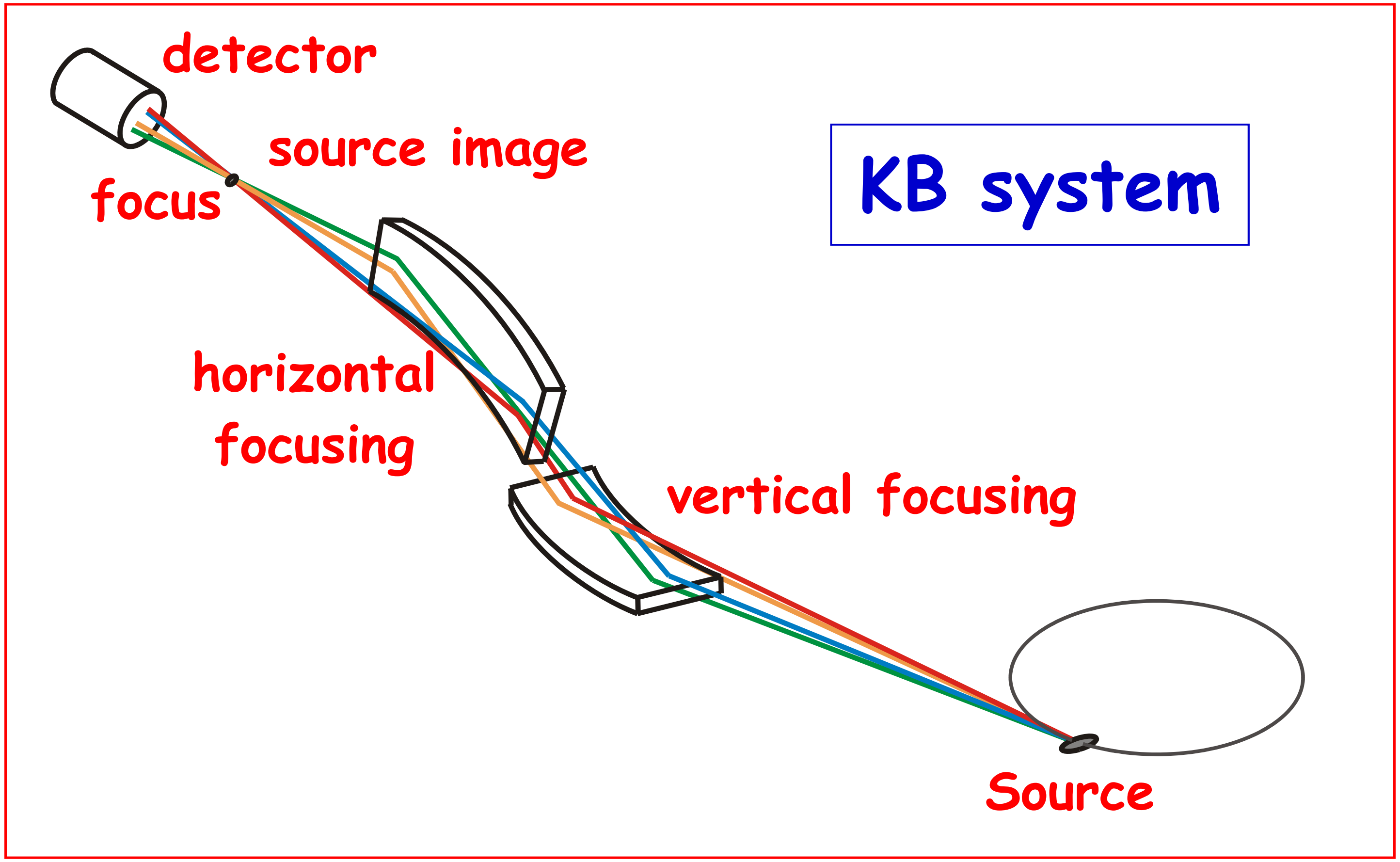
However two are most popular:

Refractive lenses

KB systems

Kirkpatrick-Baez (KB) focusing devices

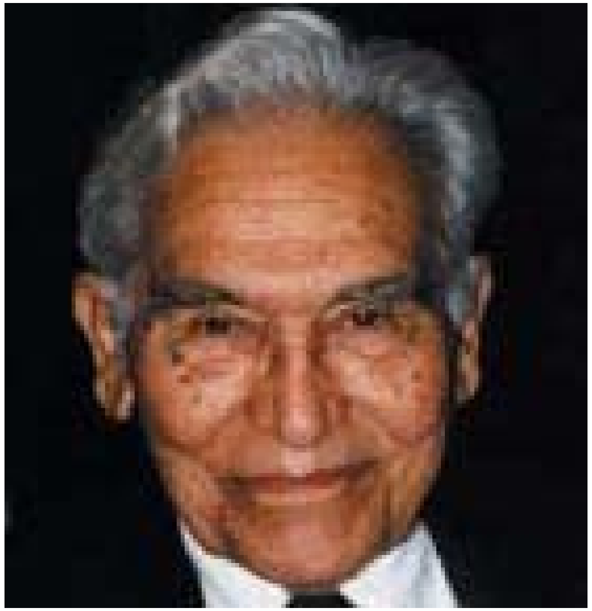
- Separate vertical and horizontal focusing (non-circular source)
- Technologically easier than single reflection ellipsoid
- Metal (single layer (mirror)) or graded ML coatings

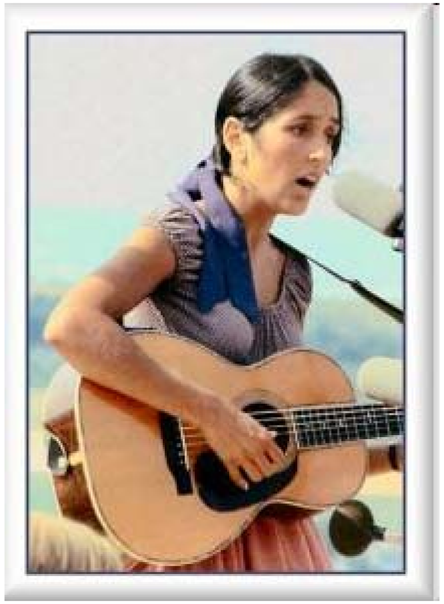


P. Kirkpatrick, A.V. Baez, J. Opt. Soc. Am. 38, 766 (1948)

Kirkpatrick-Baez (KB) focusing devices

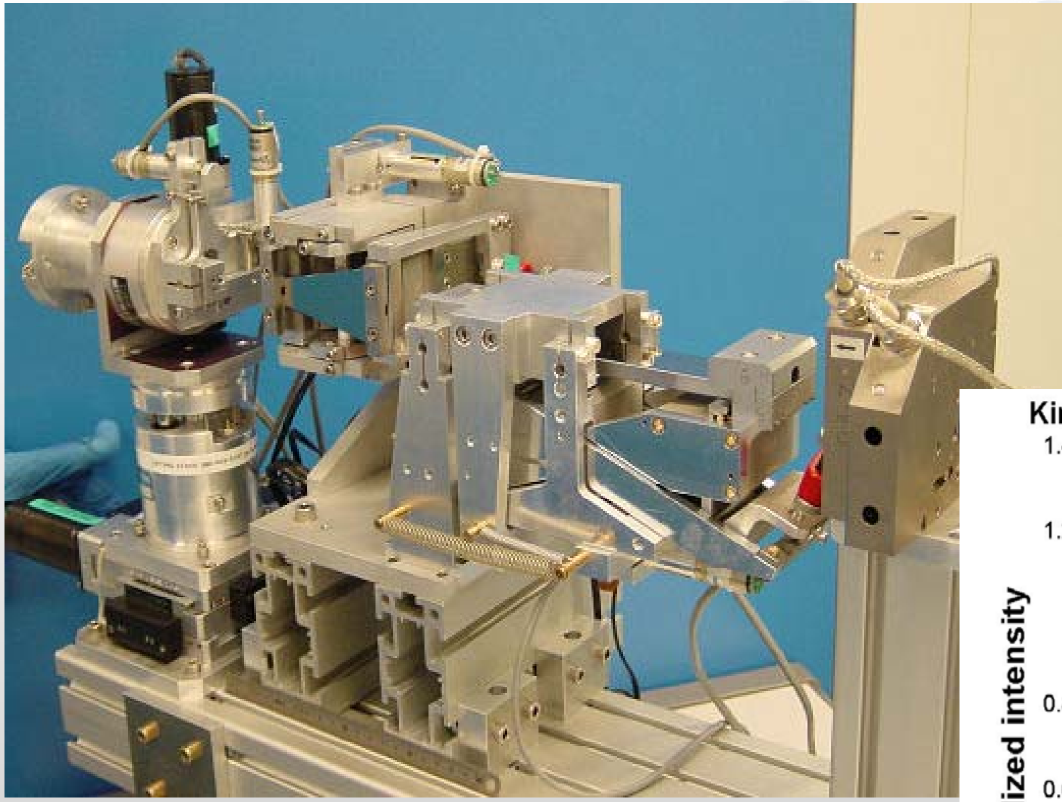
Albert V. Baez was the father of Joan Baez !





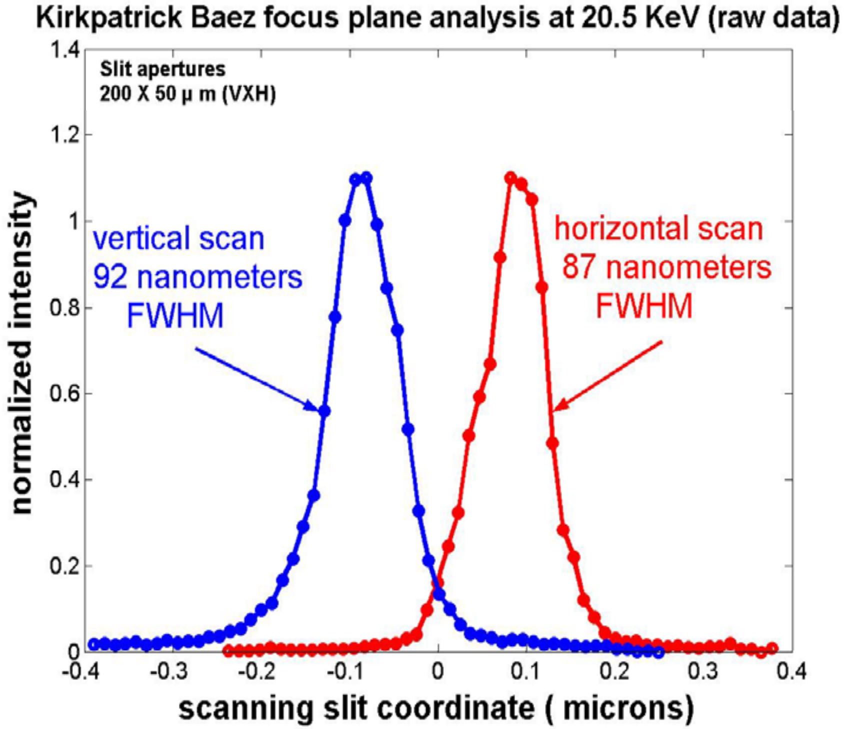
P. Kirkpatrick, A.V. Baez, J. Opt. Soc. Am. 38, 766 (1948)

Kirkpatrick-Baez system at the ESRF



microfocus on ID19 (20.5 keV)

Aperture V X H (µm)	Focus Size FWHM (nm)	Flux Ph/s @ 90mA
200 X 50	92 X 87	$5\ 10^{10}$
400 X 100	70 X 74	$2\ 10^{11}$
600 X 160	90 X 70	$4.5\ 10^{11}$

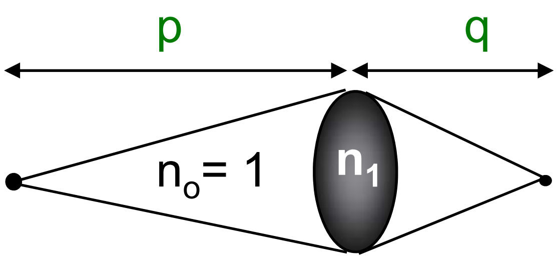


ESRF: O. Hignette *et al.*, 2007: 40nm

Spring 8: H. Mimura *et al.* 2007: 25nm

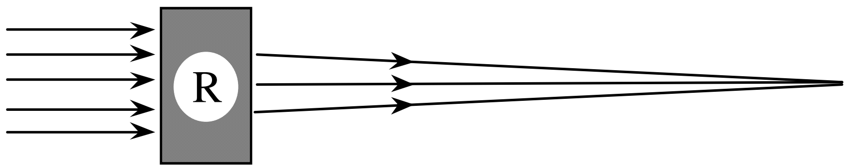
Spring 8: H. Mimura *et al.* 2009: 7nm

Compound refractive lenses



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

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



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

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

$|n_1| < 1$: concave lens

Example :

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

1 hole of 100 μm radius: F = 9 m

15 holes of 100 μm radius: F = 60 cm

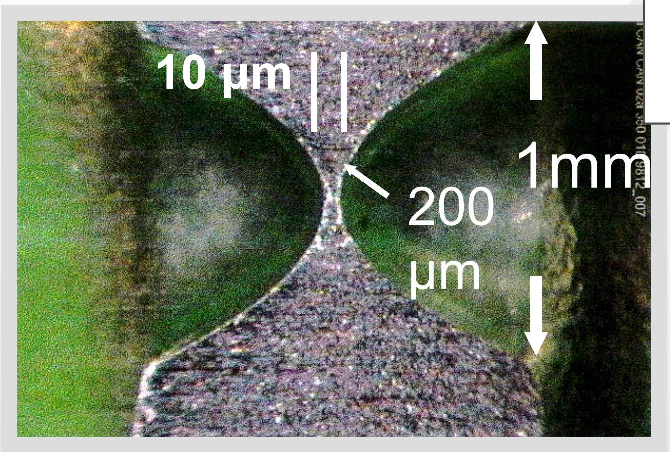
A. Snigirev et al. Nature, 384 (1996)

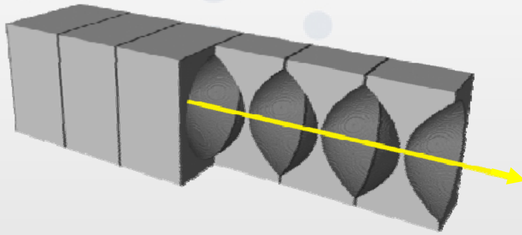
Advantages
- simplicity and low cost
- low sensitivity to heat load

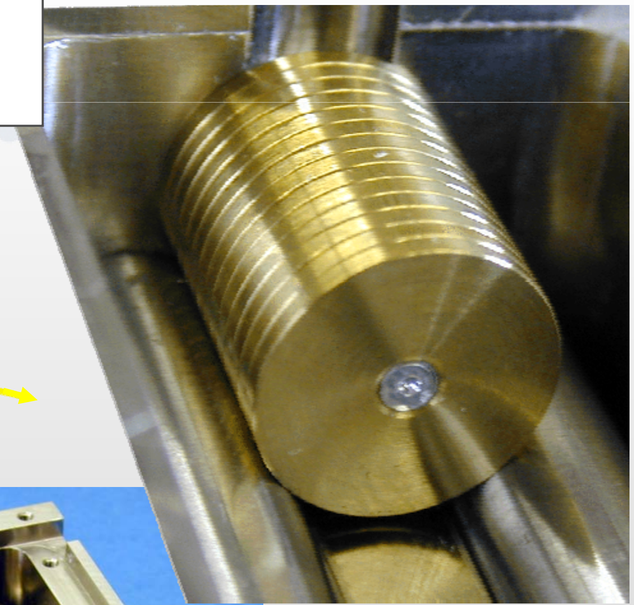
Disadvantages
- efficiency limited by absorption
- small aperture (limited resolution)
- strong chromatic aberrations (if not parabolic)

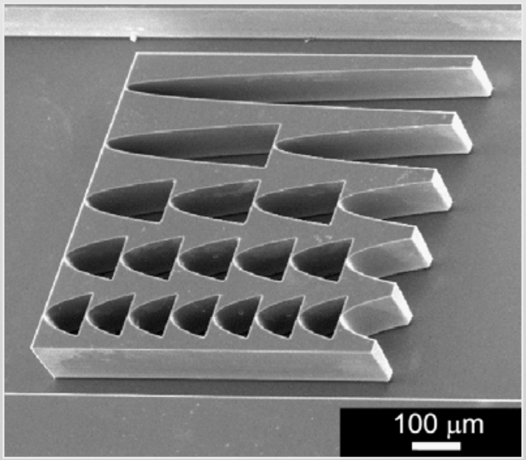
Refractive lenses with parabolic shape

Materials:
low Z, high density
Be, (C*), Al, Si, … Ni

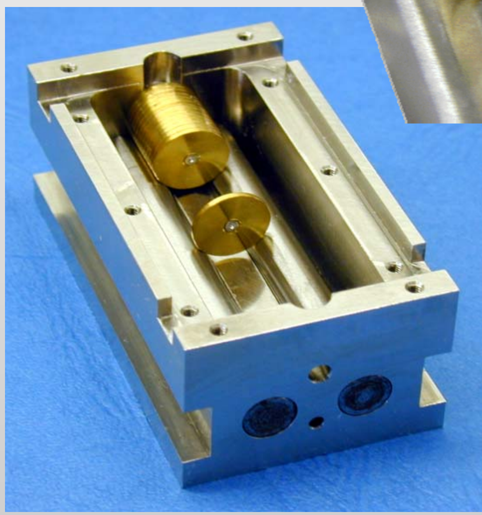






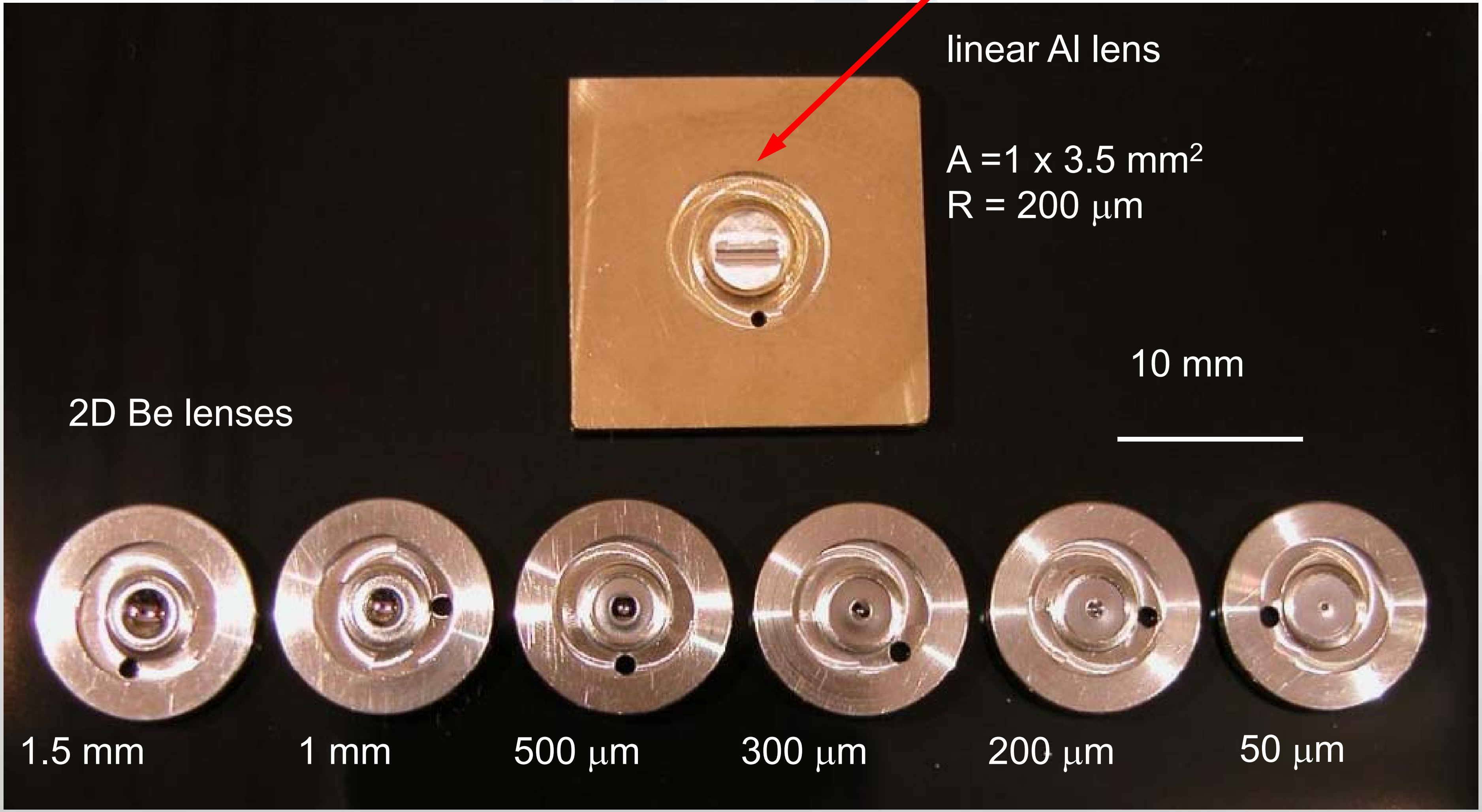


C. David et al.
PSI, Villigen, Switzerland



B. Lengeler, C. Schroer, M. Richwin,
RWTH, Aachen, Germany

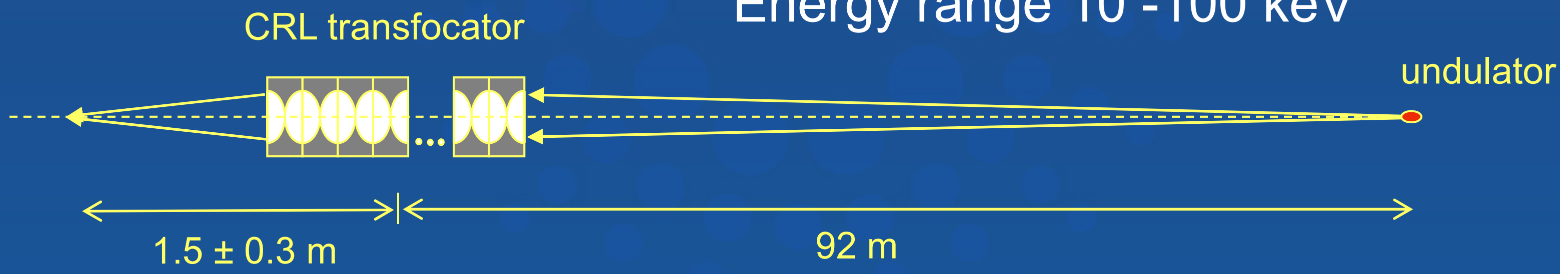
Newest development – linear lenses

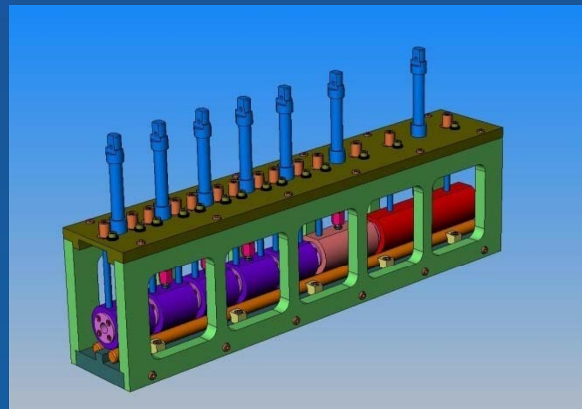


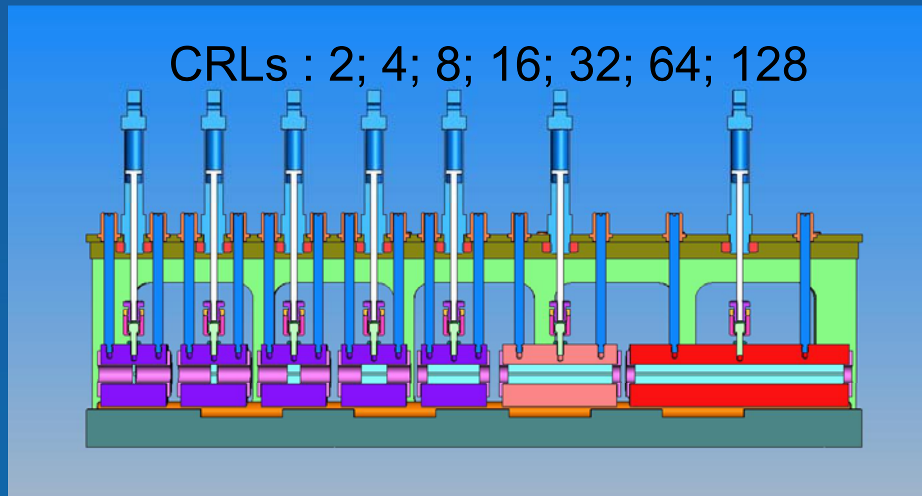
B. Lengeler et al., RWTH, Aachen, Germany

CRL transfocator

Energy range 10 -100 keV





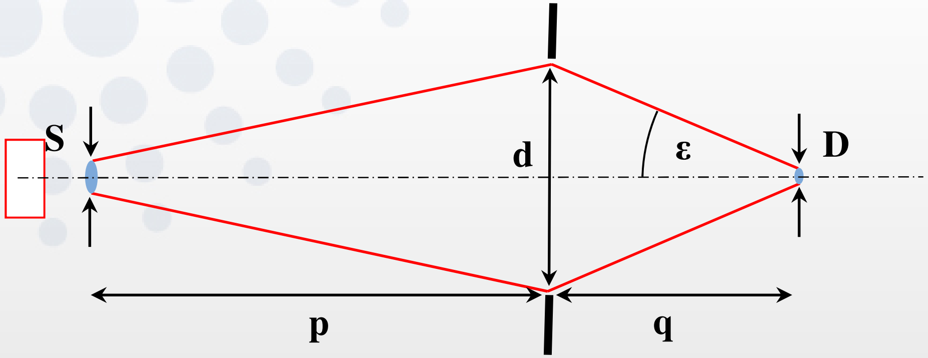


Limits in X-ray focusing

Diffraction limit $D_{FWHM} = C\frac{\lambda}{NA}$

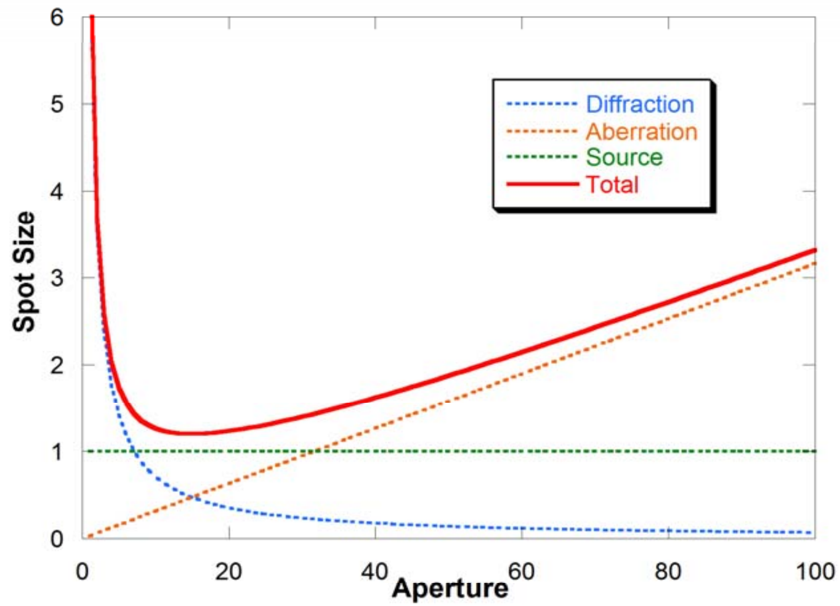
Numerical aperture $NA = n \cdot \sin\varepsilon$

Straight aperture $C = 0.44$



Source size limit $D = \frac{q}{p} \cdot S$

Small source, large distance!!!

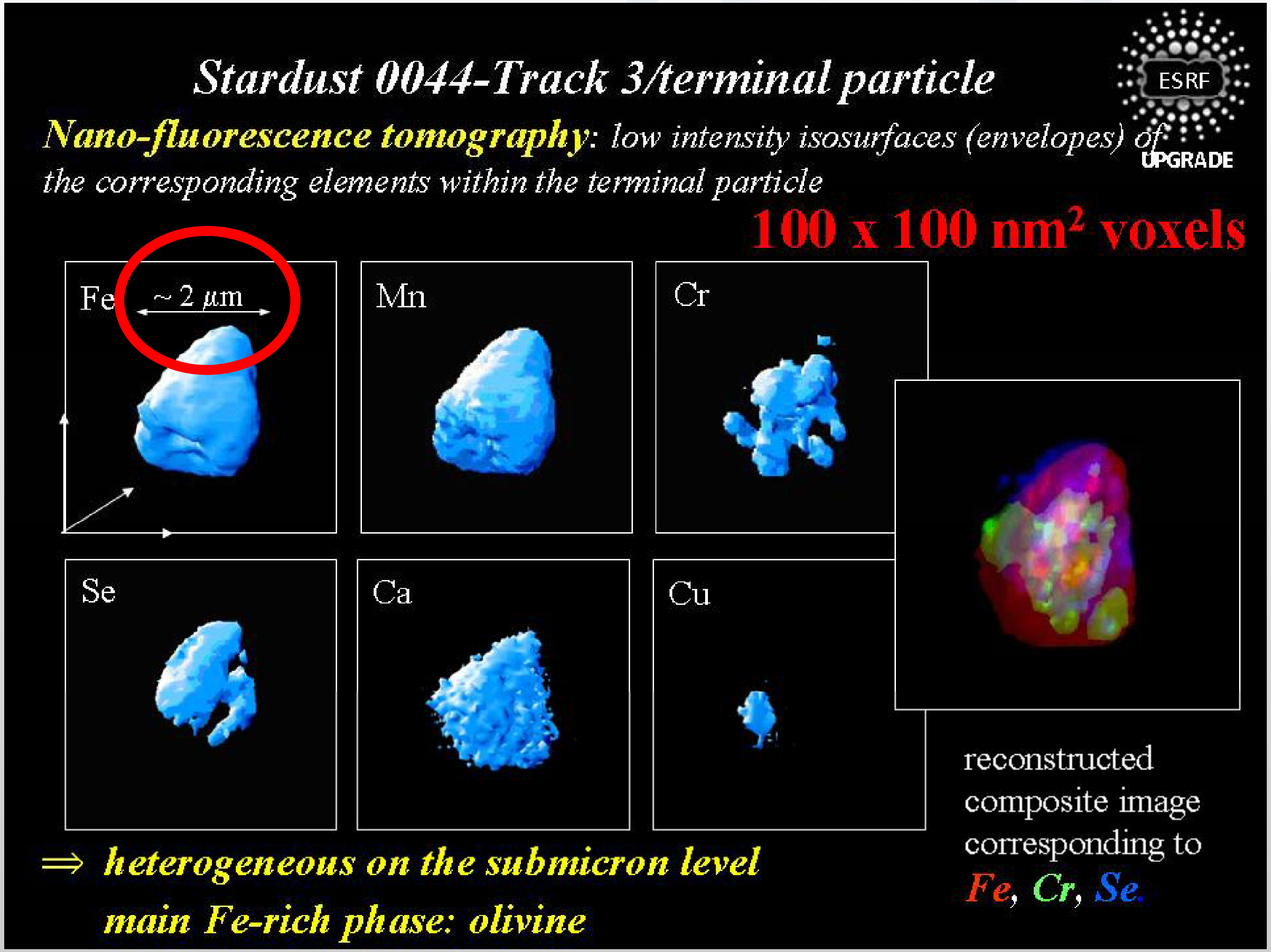


Further limitations

- **Volume diffraction**
- **Scattering**
- **Non-trivial design**
- **Fabrication accuracy**
- **Alignment**

Nanometer scale is of interest

Chemical Composition of a Comet Tail particle

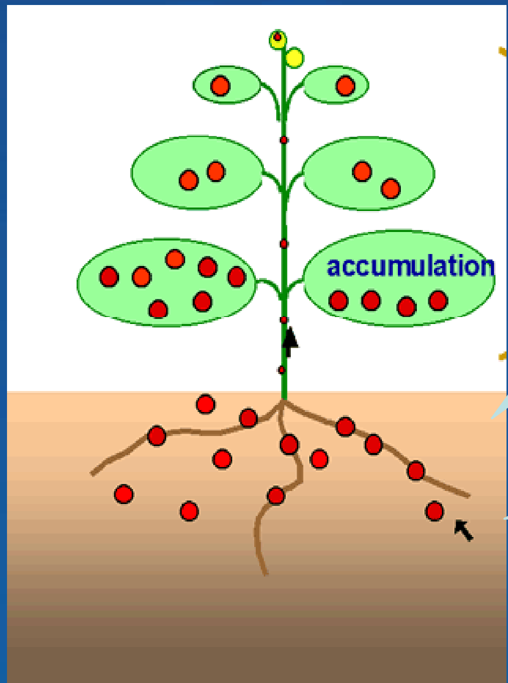


Silversmit G, Vekemans B, Brenker FE, Schmitz S, Burghammer M, Riekel C, Vincze L.
Anal Chem. 2009, 81, 6107-6112

(at ID13 of ESRF)

Phytoextraction in hyper accumulator plants

Arabidopsis Thaliana



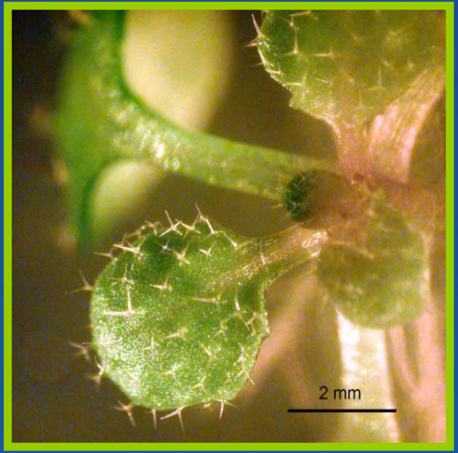
Anthropogenic activities

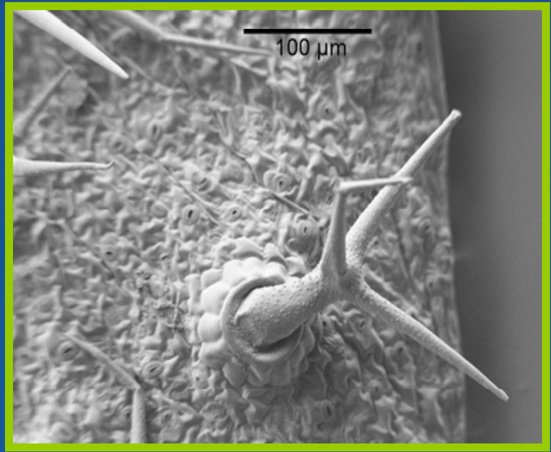
Cesium (Cs)
Nuclear activities

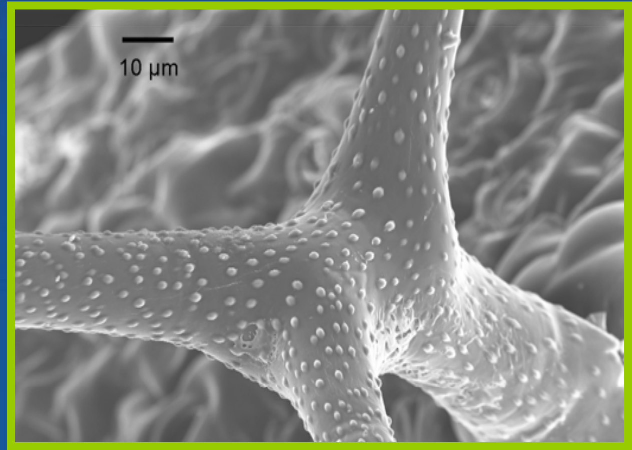
Cadmium (Cd)
Industrial (mining), agricultural activities (fertilizers)

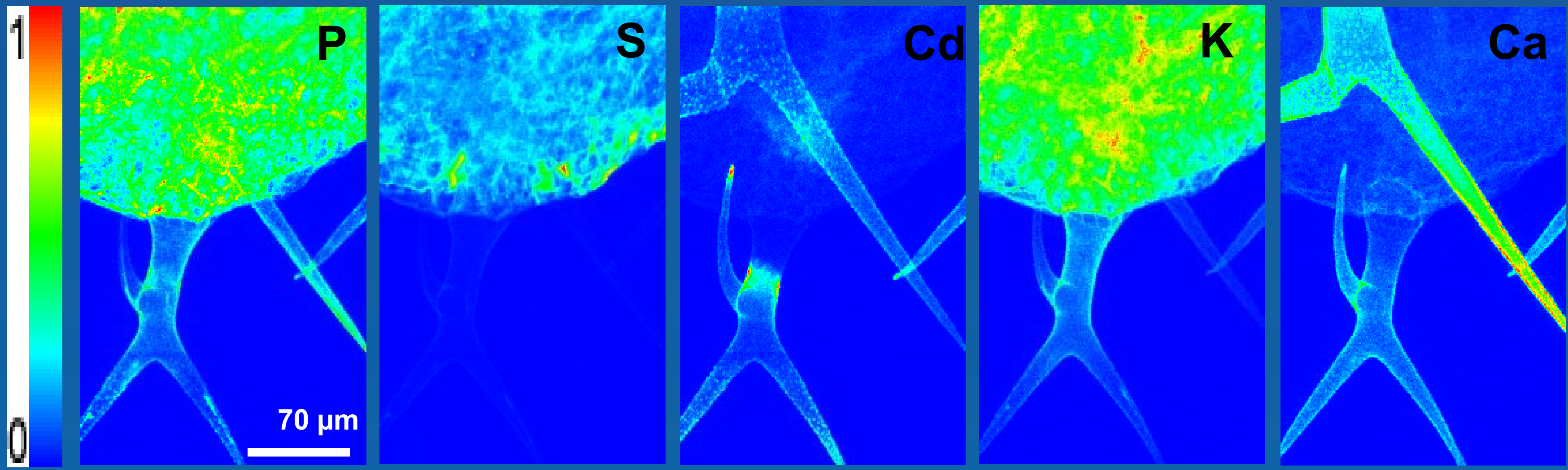
- **Green and low cost strategy for soil cleaning**
- **Mechanisms of metal accumulation ?**

µ-XRF in Trichomes of Arabidopsis Thaliana





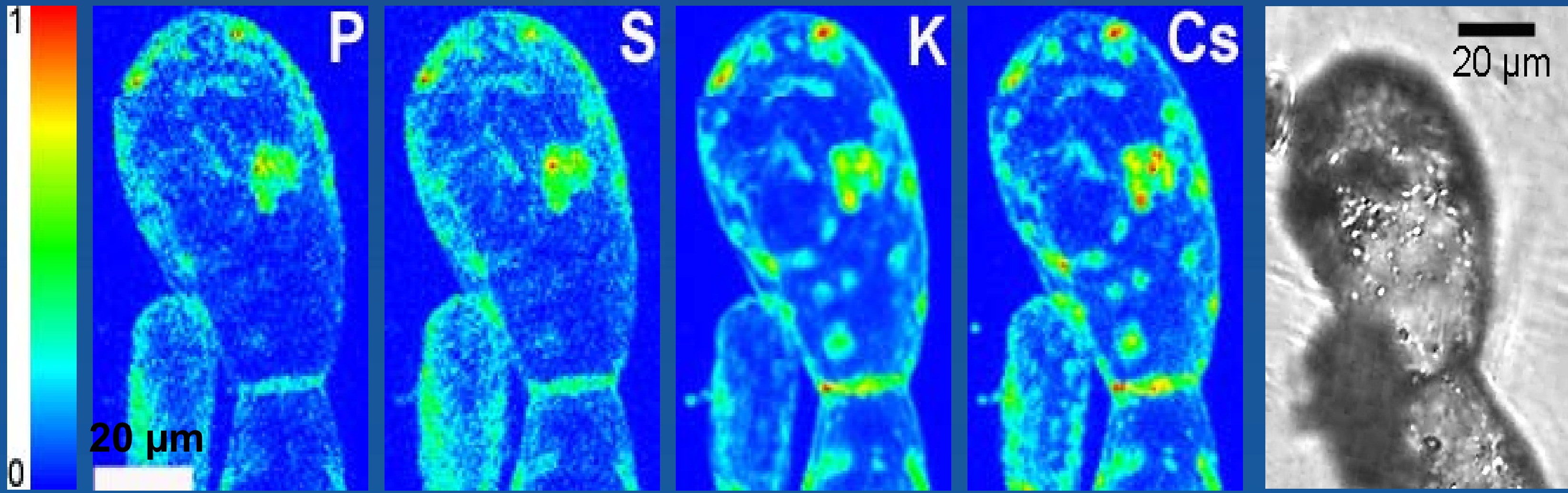


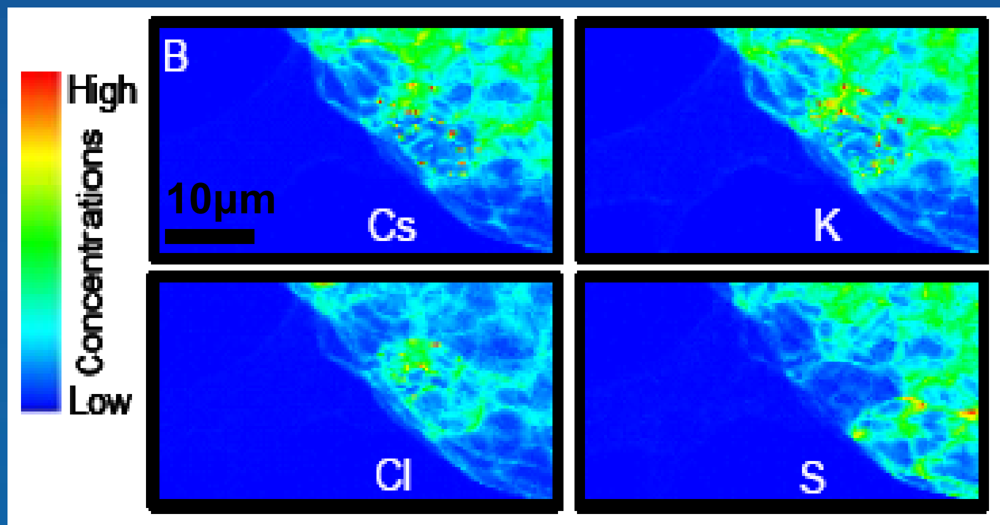


E_{ex}: 5.8 keV, probe size: 0.3x0.2µm^2, dwell time: 800 ms/pixel.

M.P. Isaure *et al*., *Biochimie* 88 (2006)

Cs accumulation in Arabidopsis Thaliana

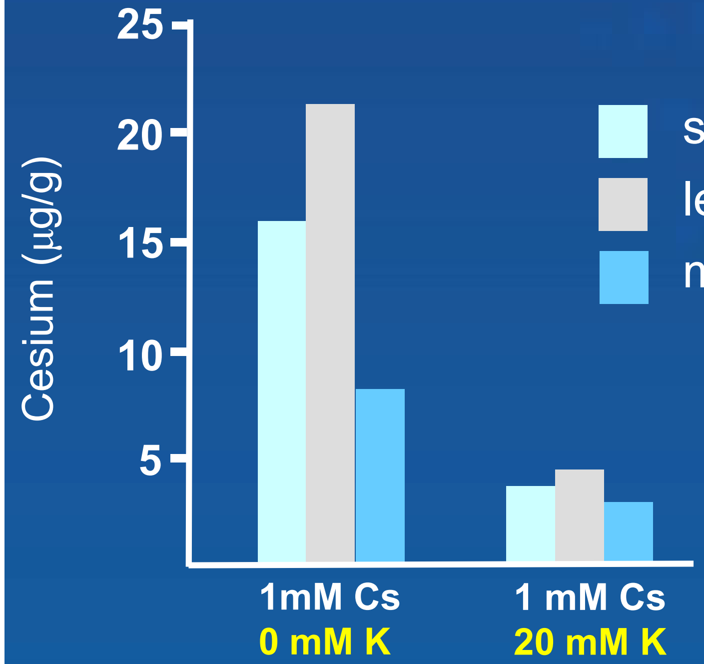


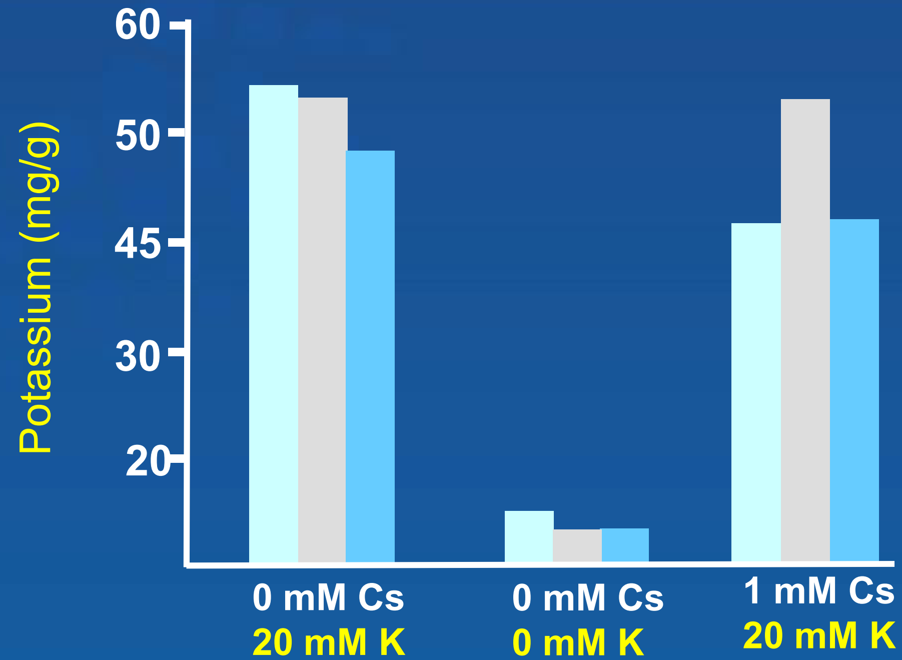


E_{ex}: 5.8 keV, probe size: 0.2x0.2µm^2, dwell time: 800 ms/pixel

M.P. Isaure *et al.*, *Biochimie* 88 (2006)

After 4 day-exposure to various concentrations of Cs and K





- **Potassium deficiency increases Cesium uptake**
- **Cs toxicity → interactions with intracellular K-binding protein sites**
- **A monitored application of Potassium**
  - ✓**alleviate the Cs toxicity (phytotoxicity)**
  - ✓**increase the Cs accumulation (phytoextraction)**

M.P. Isaure *et al.*, *Biochimie* 88 (2006)

The Future of the ESRF Looks Bright


Thank you for your attention!