The Art of Experiment – Symposium honoring David Nygren,
Lawrence Berkeley National Laboratory, 2-3 May 2014

Developments in Hard X-ray Optics – towards nm resolution

Björn Cederström

Physics department,
KTH Royal Institute of Technology,
Stockholm, Sweden

ROYAL INSTITUTE OF TECHNOLOGY

Topics and Themes

- Dave's connection to hard x-ray optics
- Why x-ray micro/nano beams
- The path to nm resolution
- Innovation and technological development
- Lensless imaging
- Extreme heat-resistant optics

ROYAL INSTITUTE OF TECHNOLOGY

Why x-ray micro/nano beams

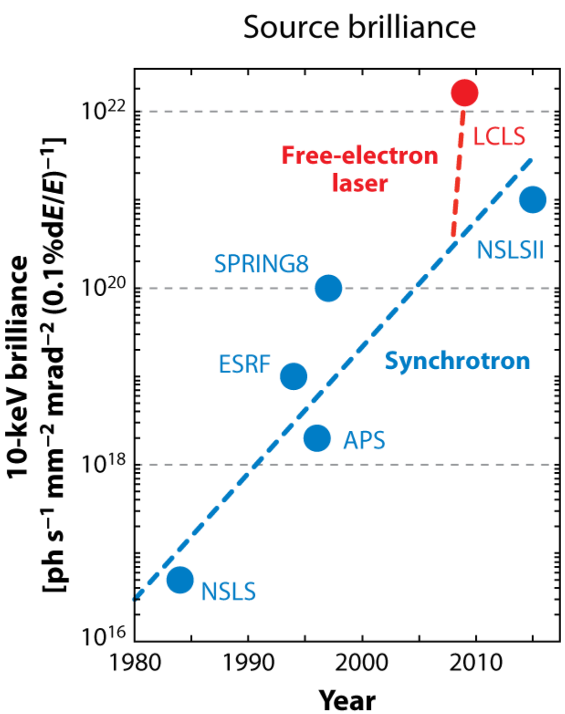
- **Short wavelength, 12.4 keV $\rightarrow$ 1 Å**

$$r = 1.22\frac{\lambda f}{D}, \quad \frac{f}{D} \approx 10^2 - 10^3 \Rightarrow r = 10 - 100\,\text{nm}$$

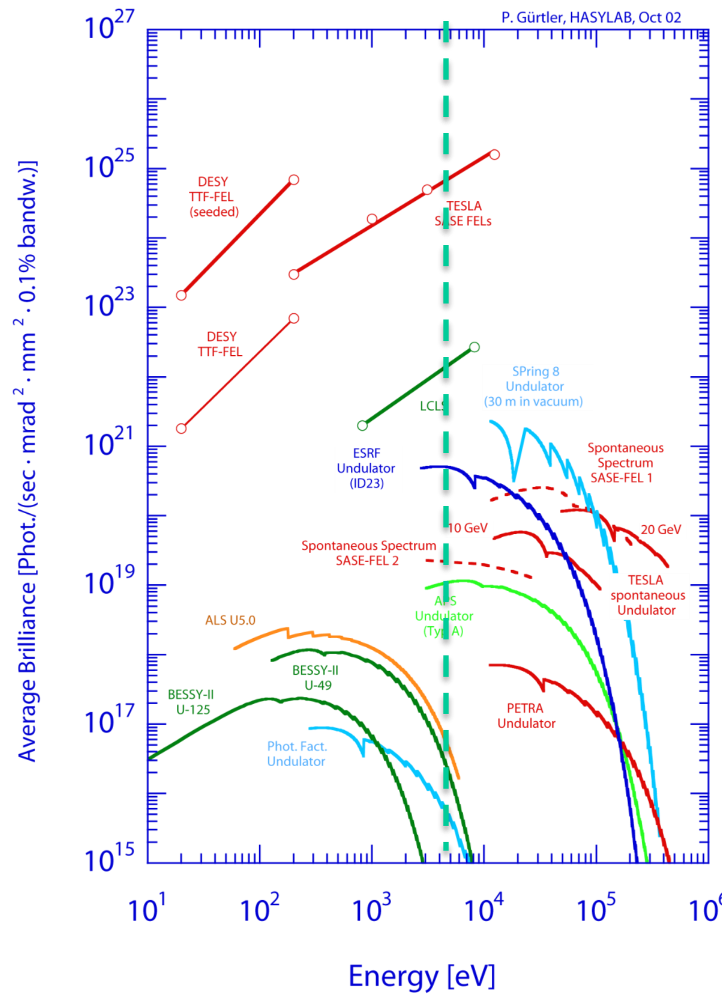
- **Weakly interacting probe**
- **Sensitive to**
  - **Structure**
  - **Elemental composition**
  - **Chemistry**
- **Minimal sample preparation**
- **In-situ measurements**

Sources

Lower beam emittance



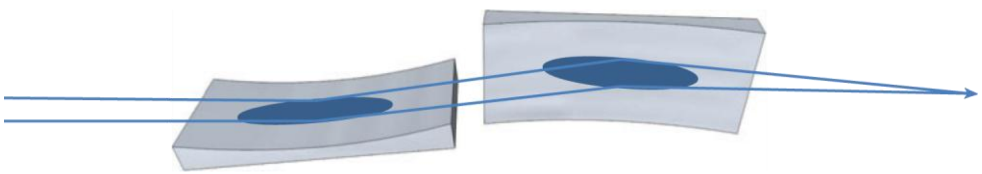
From Holt *et al.*, Annu. Rev. Mater. Res.. 2013, 43



Types of hard x-rays optics

Reflective

Kirkpatrick-Baez mirrors (1948)



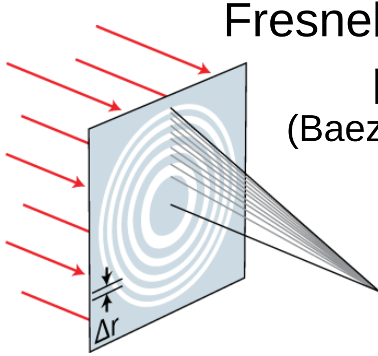
$$n = 1 - \delta + i\beta$$

$$\theta_{crit} = \sqrt{2\delta}$$

$$\delta \approx 10^{-5} \Rightarrow \theta_{crit} \approx 0.2^{o}$$

Diffractive

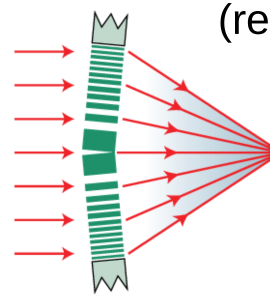
Fresnel zone plates (Baez, 1961)



Outer zone aspect ratio < ≈30:1

Multilayer Laue lens (recently at Argonne)

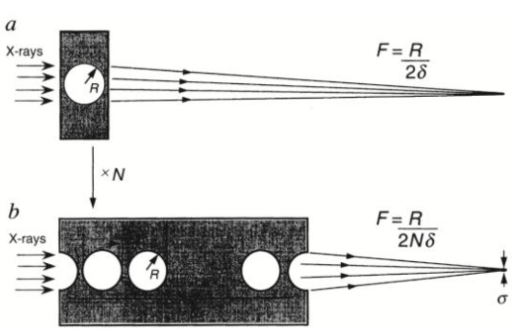
1D focusing



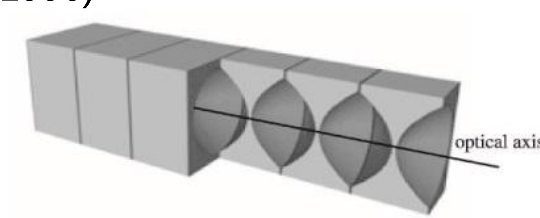
From Sakdinawat, Attwood, *Nat. Phot.* 4, 2010

Refractive

Compound refractive lens (Tomie/Snigirev 1996)

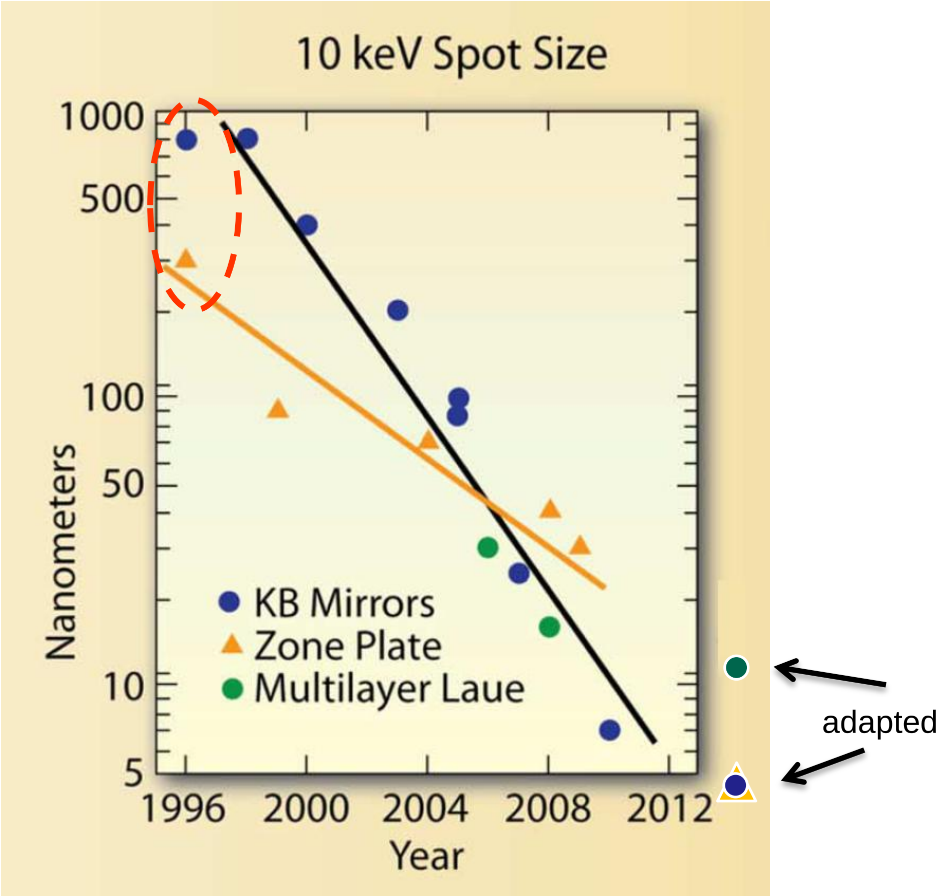


From Snigirev et al, Nature 384, 1996



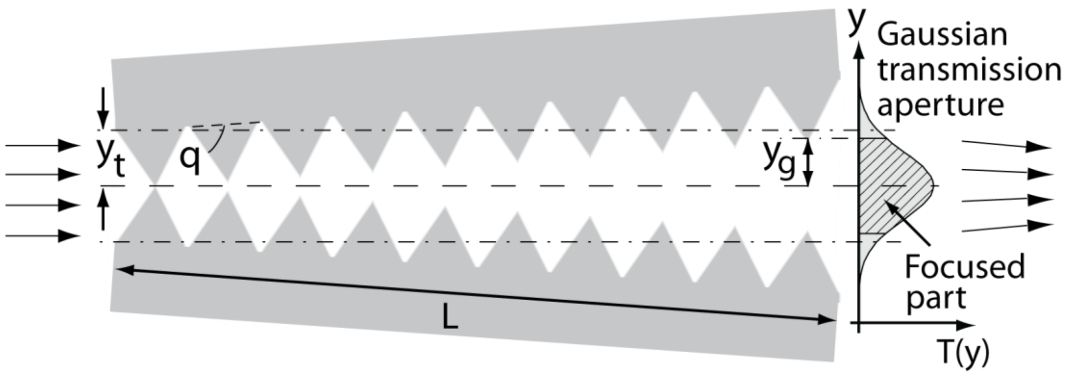
Parabolic (Lengeler, 1999)

Achievable spot size vs. time

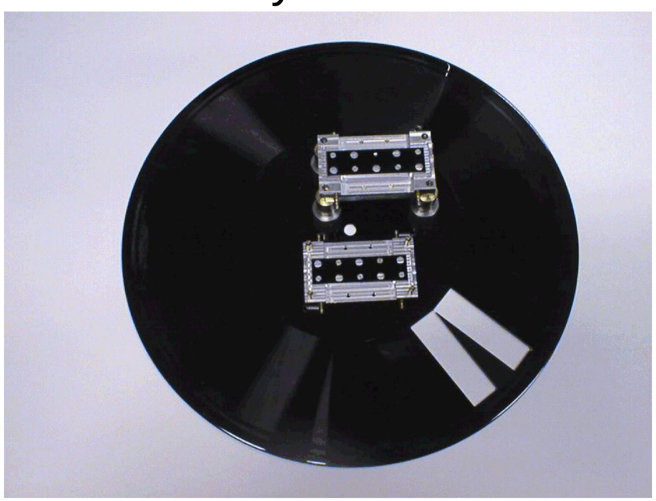


From Ice *et al*., Science 334, 2011

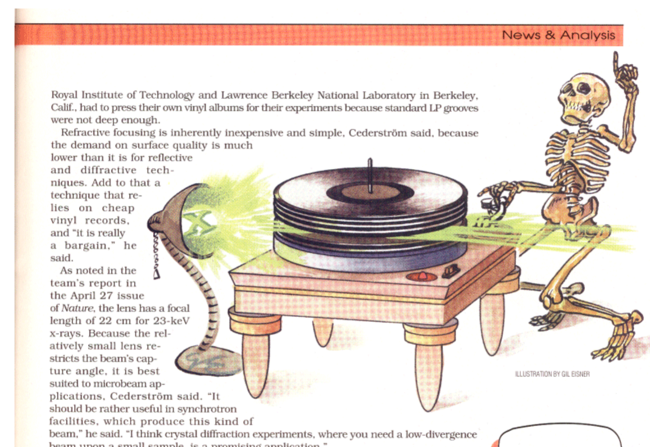
The Multi-Prism Lens



Vinyl LP lens



Cederström *et al.*, Nature 404, 2000



News & Analysis

Royal Institute of Technology and Lawrence Berkeley National Laboratory in Berkeley, Calif., had to press their own vinyl albums for their experiments because standard LP grooves were not deep enough.

Refractive focusing is inherently inexpensive and simple, Cederström said, because the demand on surface quality is much lower than it is for reflective and diffractive techniques. Add to that a technique that relies on cheap vinyl records, and "it is really a bargain," he said.

As noted in the team's report in the April 27 issue of *Nature*, the lens has a focal length of 22 cm for 23-keV x-rays. Because the relatively small lens restricts the beam's capture angle, it is best suited to microbeam applications, Cederström said. "It should be rather useful in synchrotron facilities, which produce this kind of beam," he said. "I think crystal diffraction experiments, where you need a low-divergence beam upon a small sample, is a promising application."

Old LPs Find New Use in X-Ray Optics

STOCKHOLM, Sweden — In trying to develop a cheap way to focus x-rays, perhaps it should be no surprise that researchers turned to German technopop group Kraftwerk for help. The scientists bought one of the band's albums to cut up and form into a sawtooth refractive lens.

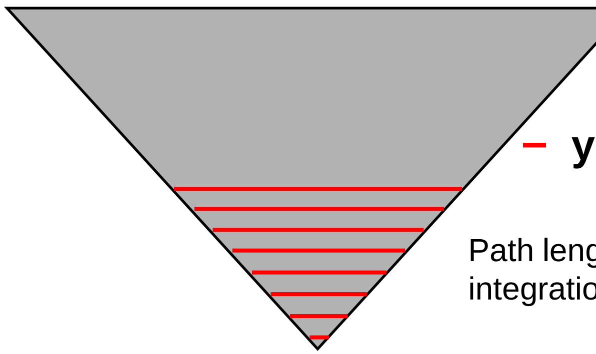
Besides its low cost, the Kraftwerk album was chosen because of its very long songs, enabling the researchers to cut long sections with uniform grooves and no interruptions, said Björn Cederström, the physicist who came up with the idea. Ultimately, however, he and his colleagues at Sweden's

AUGUST 2000

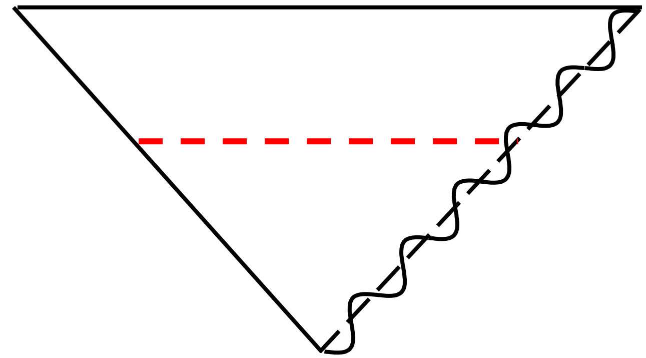
ROYAL INSTITUTE OF TECHNOLOGY

Tolerances

Insensitive to systematic figure errors



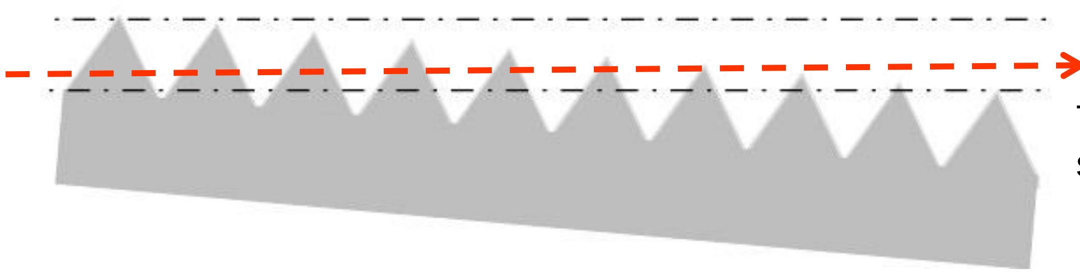
Path length at y is the integration of the area to y



Insensitive to random surface errors

"Random walk"

Phase error: $$\sigma_\phi = \frac{2\pi}{\lambda}\frac{\sigma_x}{\sin\beta}\sqrt{m}\delta$$

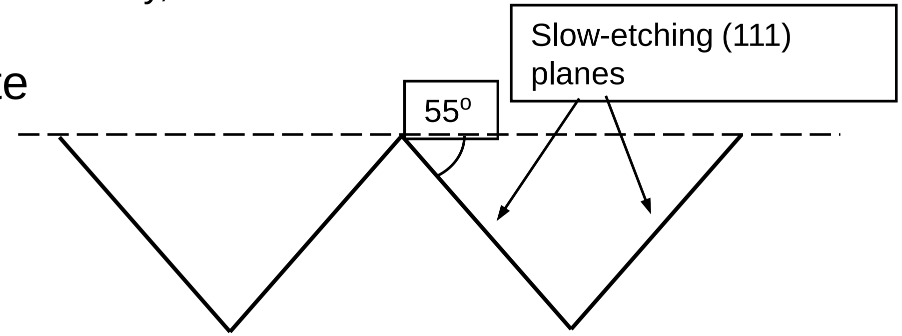


Traversing *m* surfaces

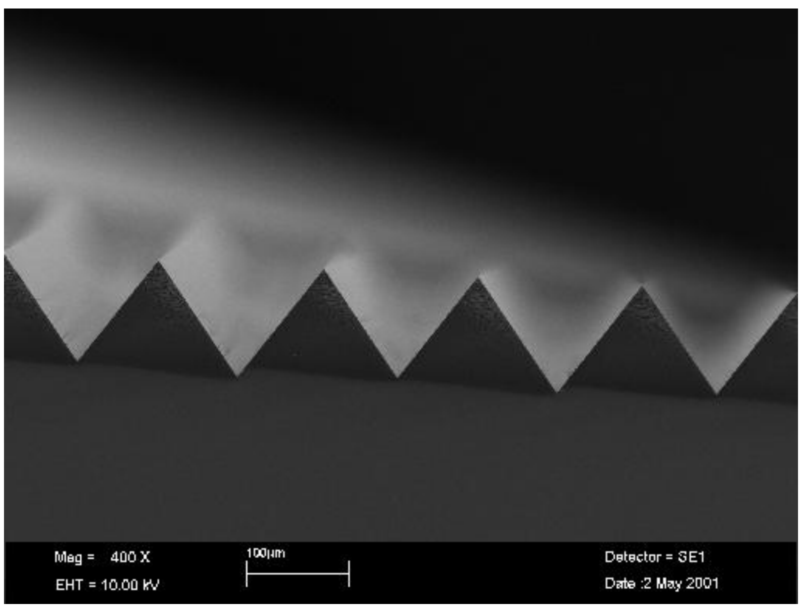
Anisotropic etching of Si lenses

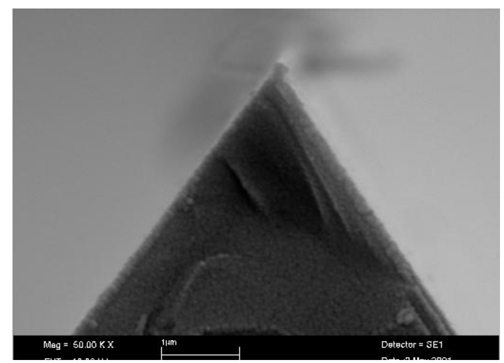
Proposed by Carolina Ribbing, Uppsala University, Sweden

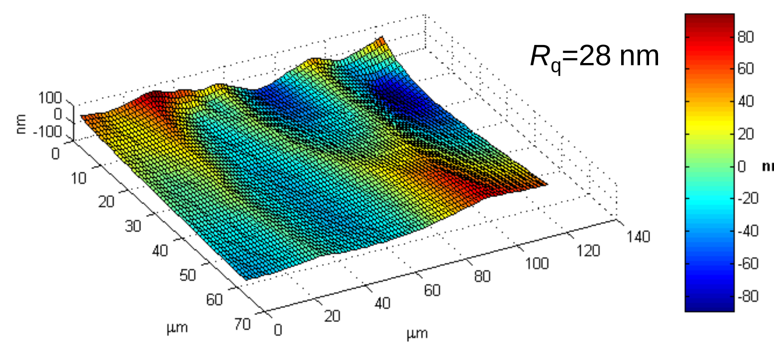
Silicon wafers (100) as substrate



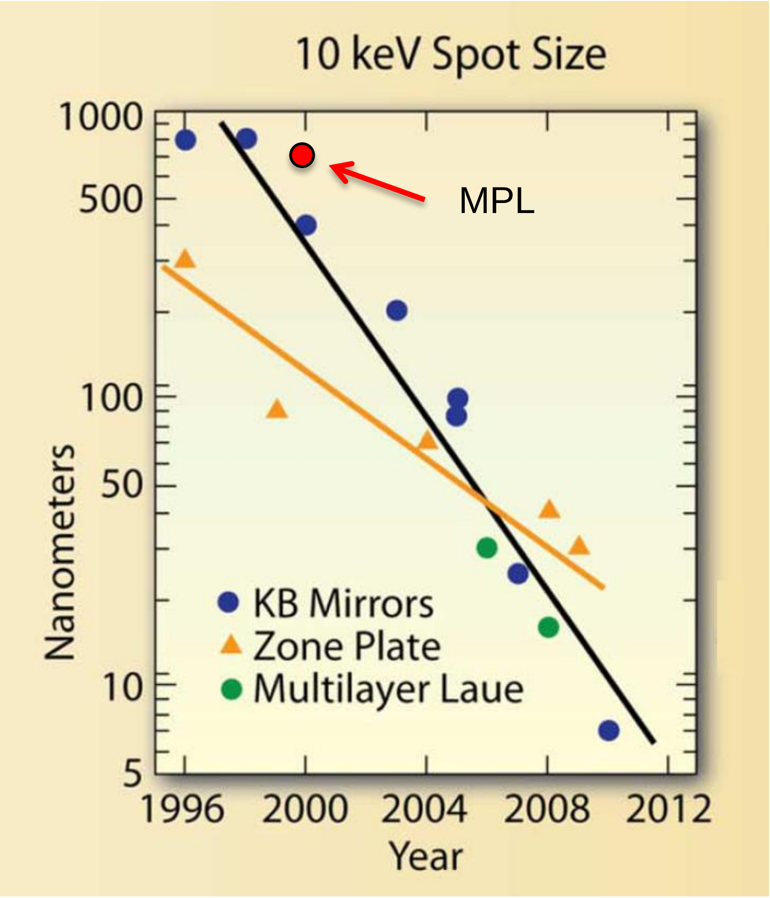
SEM images





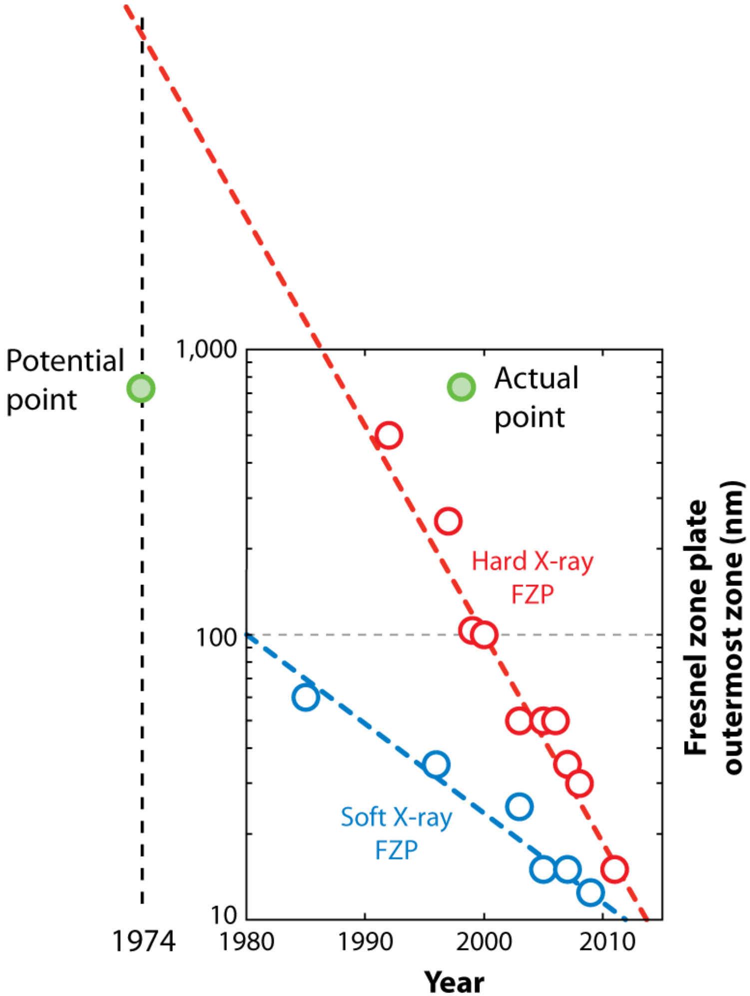


Achievable spot size vs. time



From Ice *et al*., Science 334, 2011

Alternative historic scenario



Adapted from Holt *et al.*, Annu. Rev. Mater. Res.. 2013, 43

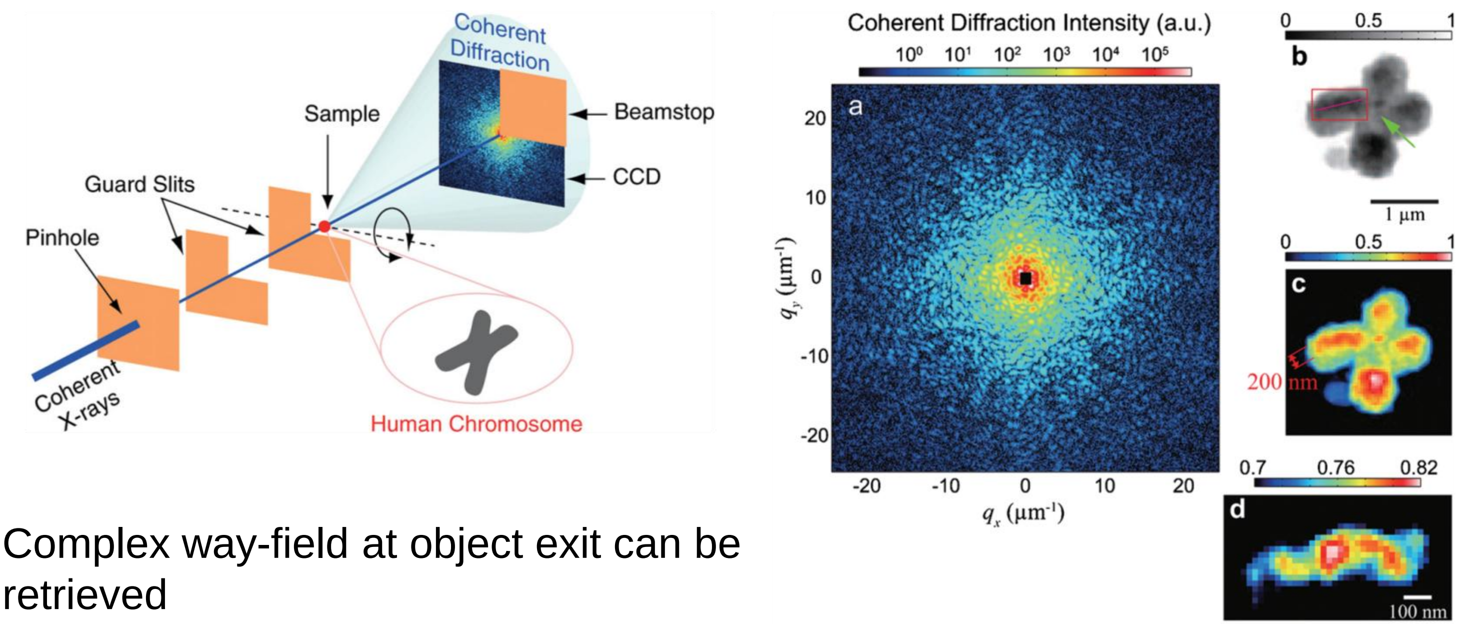
ROYAL INSTITUTE OF TECHNOLOGY

Lenless coherent x-ray imaging

Idea from Sayre, 1952

Nishino *et al.* at Spring-8

PRL 102, 018101 (2009) PHYSICAL REVIEW LETTERS week ending 9 JANUARY 2009



Complex way-field at object exit can be retrieved

5-keV x-rays

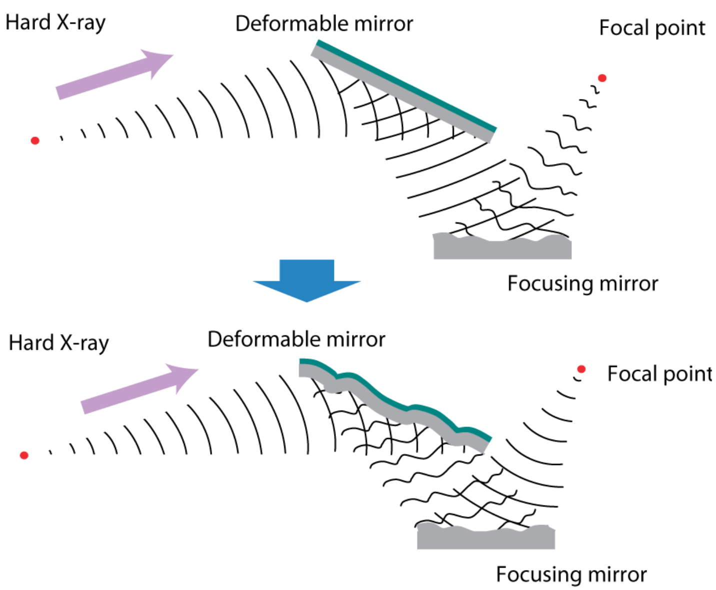
Phase-contrast

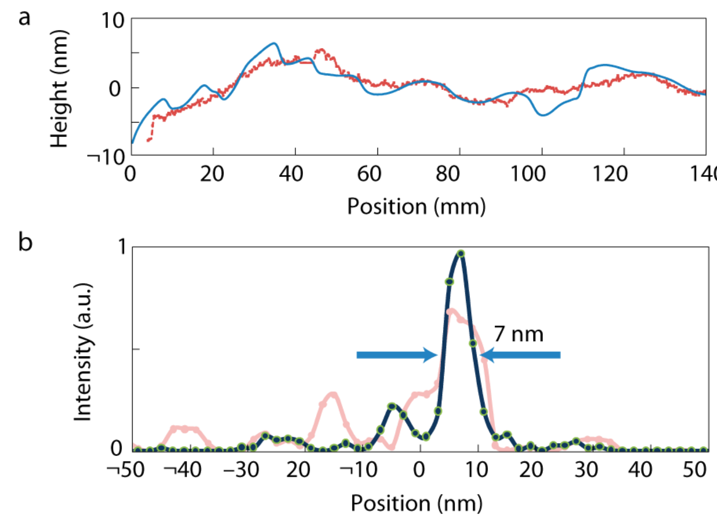
Dose = 2×10^{10} Gy for 3D acquisition

Recent progress in KB mirrors

Mimura *et al*., Nature Phys. 6, 2009

In situ wavefromt correction



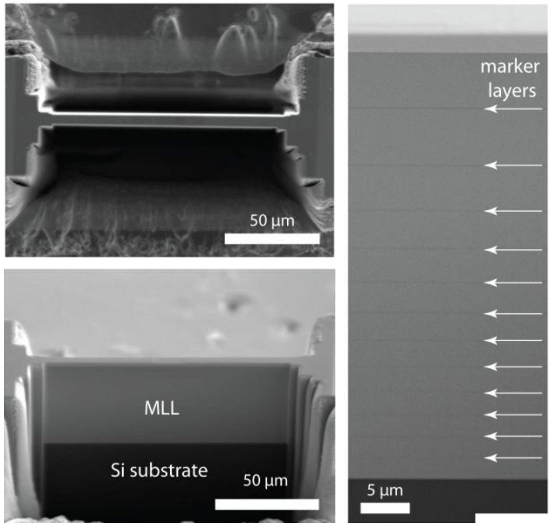


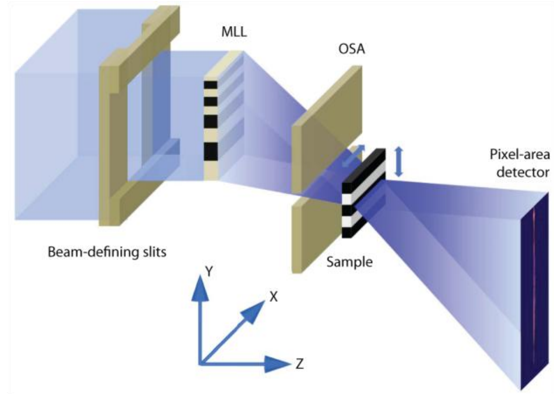
Mirror substrate shaped by elastic emission maching (EEM)
(Mori, Yamauchi, Endo (1987)

Interferometry

Recent progress in MLL lenses

Huang *et al.*, Scientific Reports 3, 2013



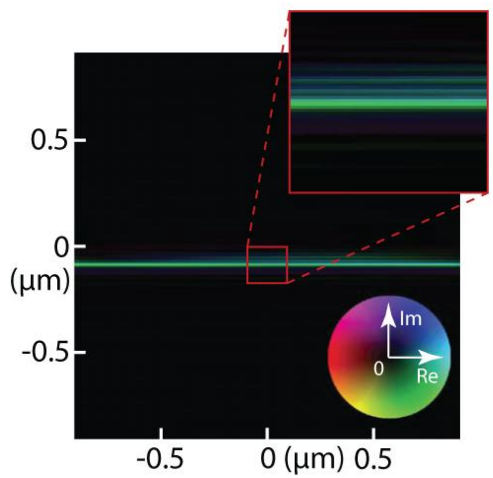


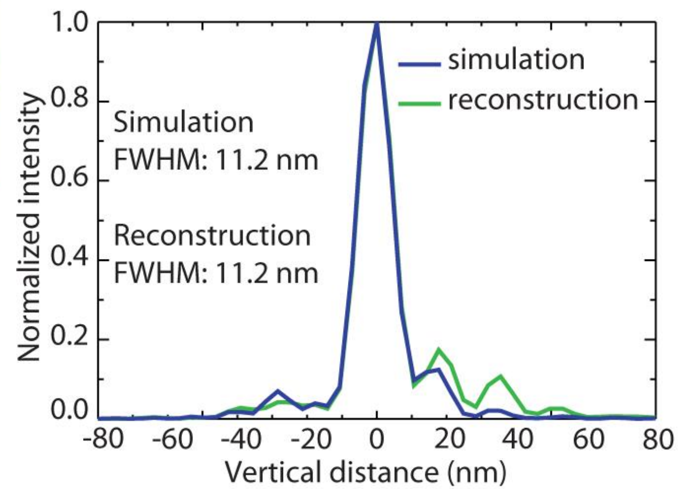
43 µm aperture
4 nm outermost zone width
6 µm thick
12 keV

Magneton sputtering

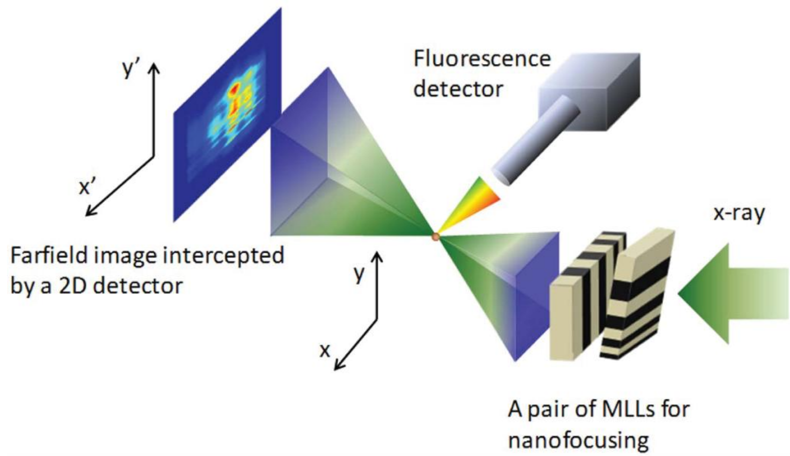
FIB milling

6510 layers of Si/WSi_2





Application of MLL lenses



APS + NSLS

Yan *et al.*, Scientiic reports 3, 2013

Cermet anode in fuel cell

<50 nm resolution

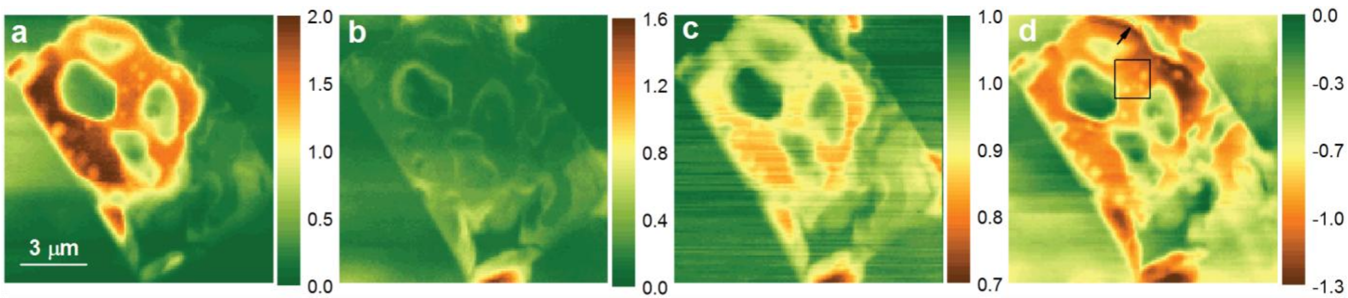
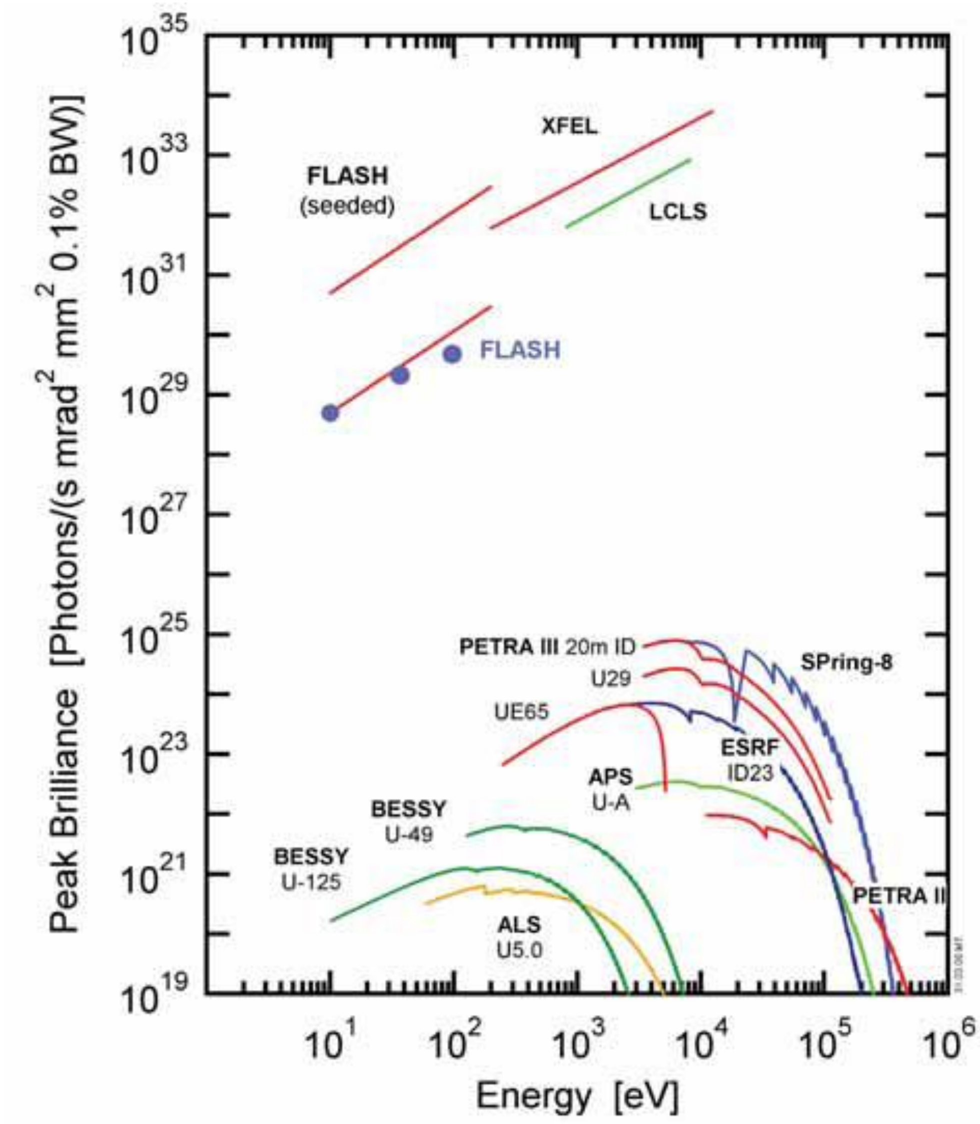


Figure 3 | Ni K a fluorescence (a), Pt La fluorescence (b), x-ray transmission (c) and reconstructed phase (d) images (units in radian) of the SOFC sample shown in Fig. 2a. The arrow in (d) points to a crack, which is barely seen in (a), (b) and (c). A zoom-in image of the rectangle area in (d) with a high resolution can be found in the supplementary material.

XFELs compared to 3rd gen. synchrotrons



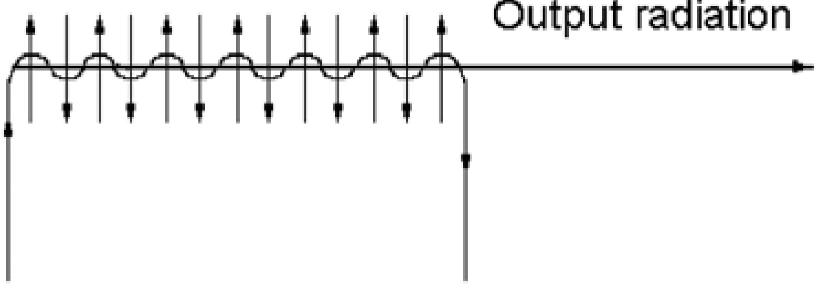
Peak brilliance:
x10^8 higher than
synchrotrons

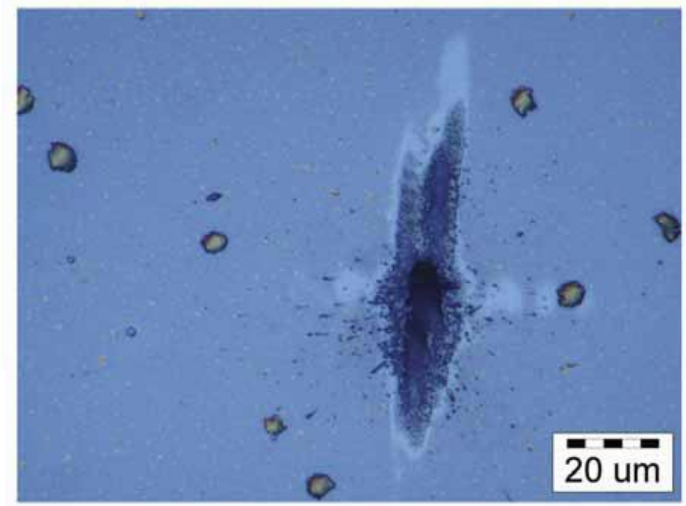
XFEL extreme power beams

Based on SASE principle (self-amplified spontaneous emission)
≈ 200 m long undulator

⇒ Coherent x-ray pulses, <100 fs

$\lambda_{min} = 1$ Å ⇒ $E_{max} = 12.4$ keV

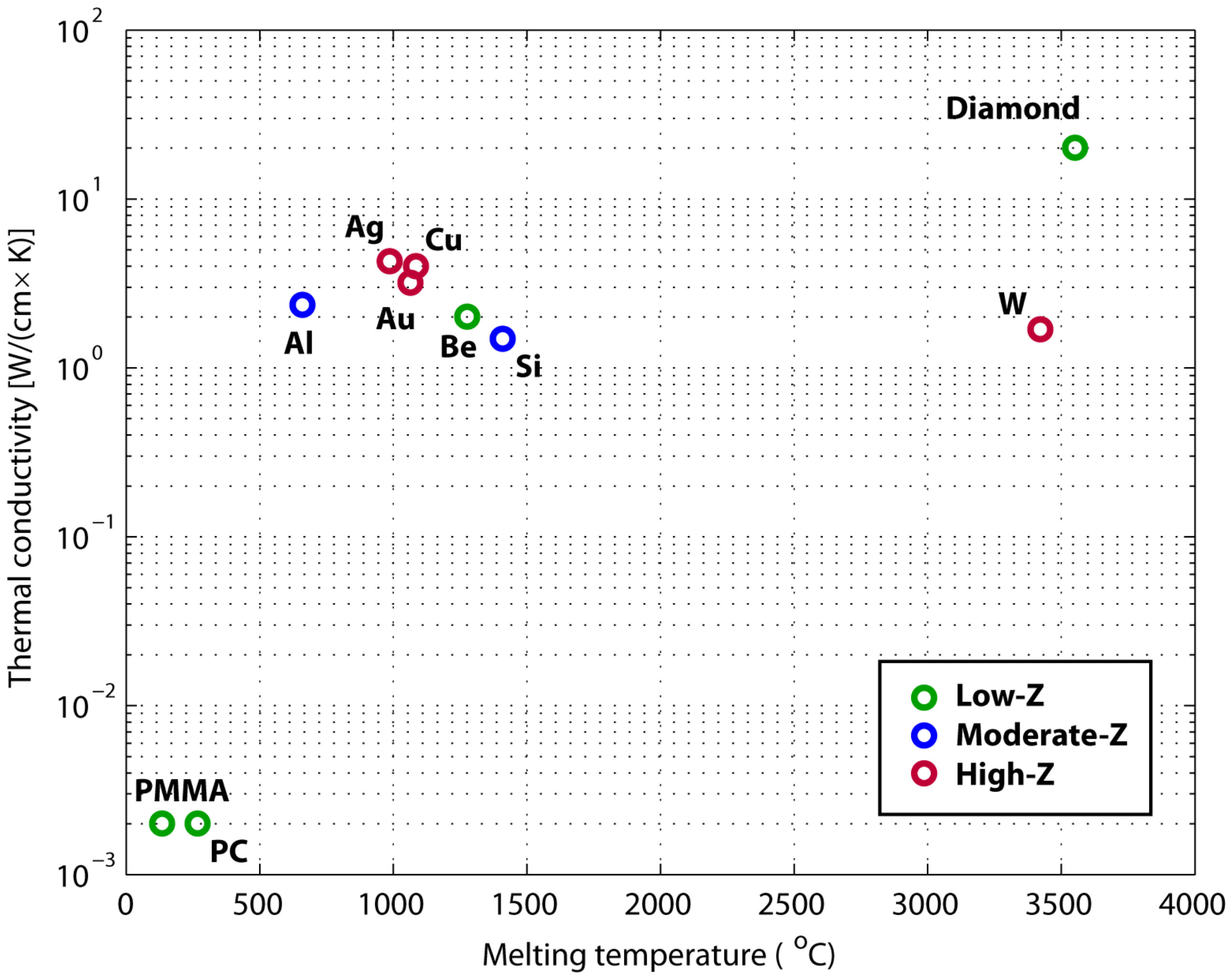




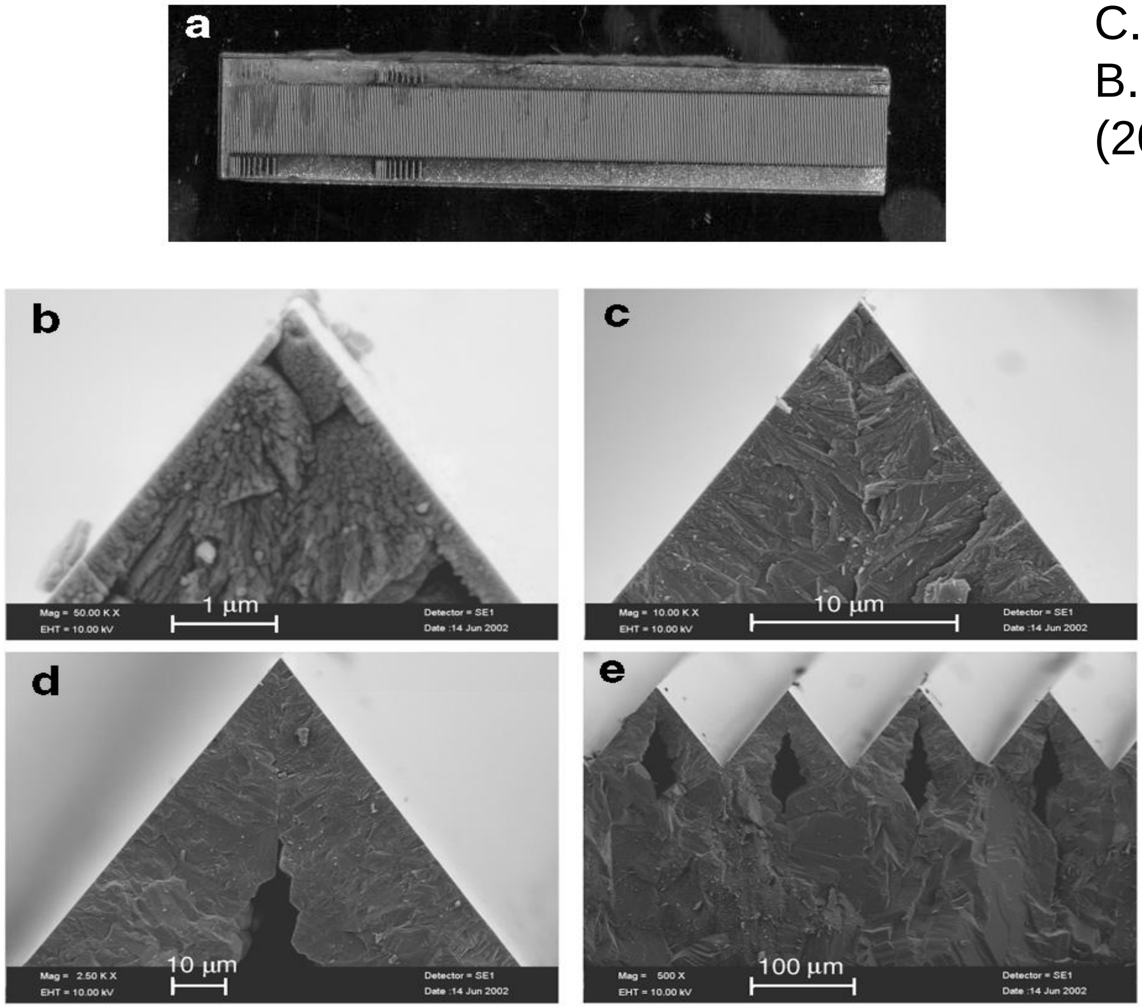
Ablation of gold target, VUV radiation
L. Juha *et al*, NIM A (2003)

	Peak	Average
X-ray beam power	24 GW	72 W
X-ray beam intensity	10^{14} W/cm^2	100 kW/cm^2

Thermal properties of materials



First diamond lens, CVDD on structured surface



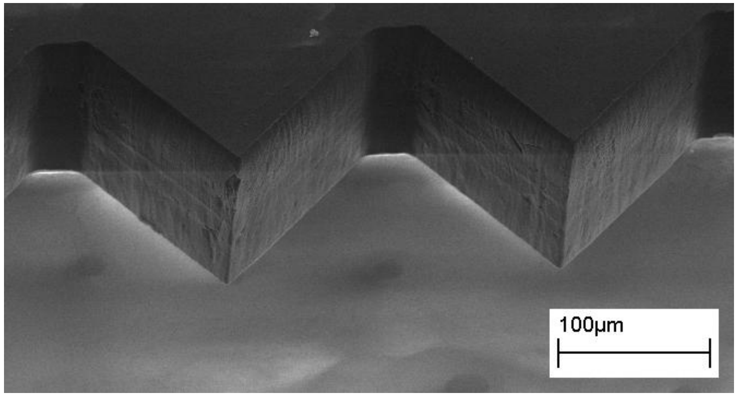
C. Ribbing,
B. Cederström
(2000)

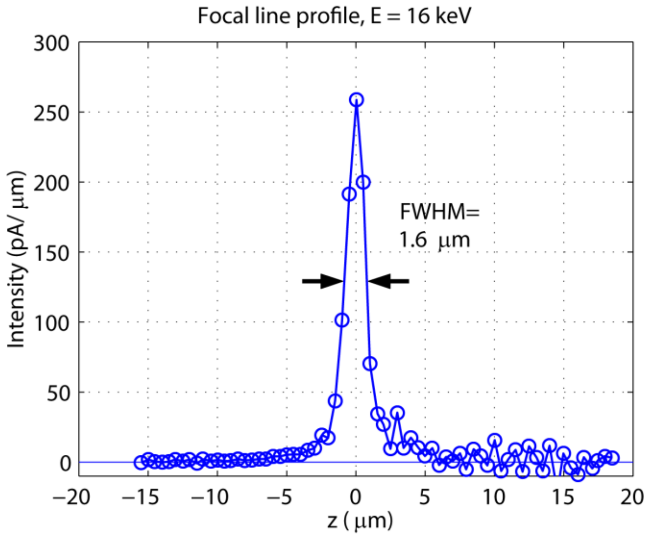
Laser-cut diamond MPL

100 μm CVD diamond wafer

Nd:YAG laser, 10 μm cutting width

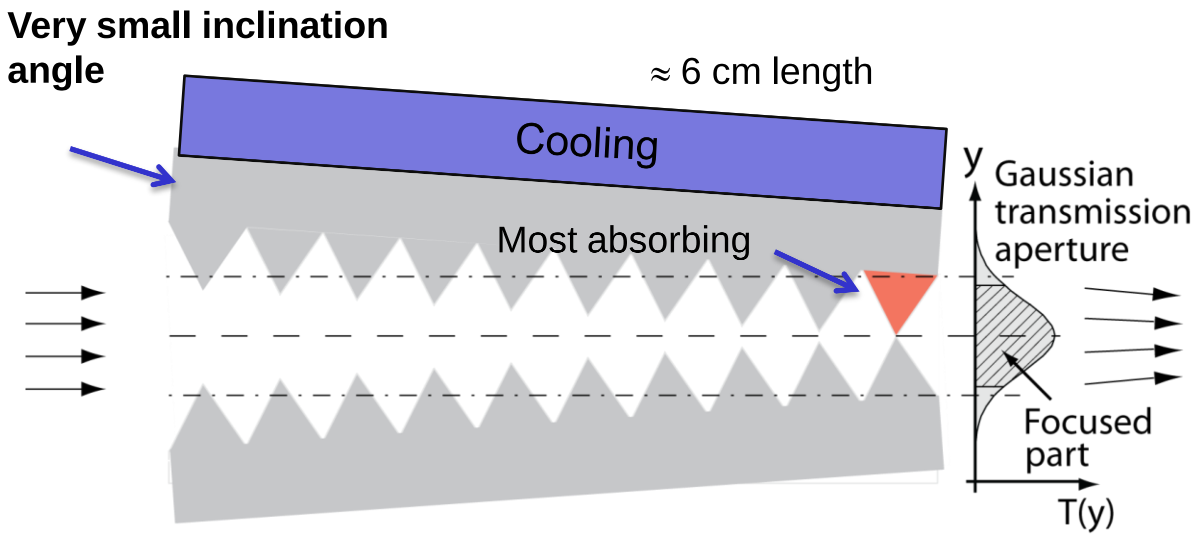
B. Cederström, C. Ribbing (2001)
in collaboration with Christoph Wild,
Fraunhofer Inst.

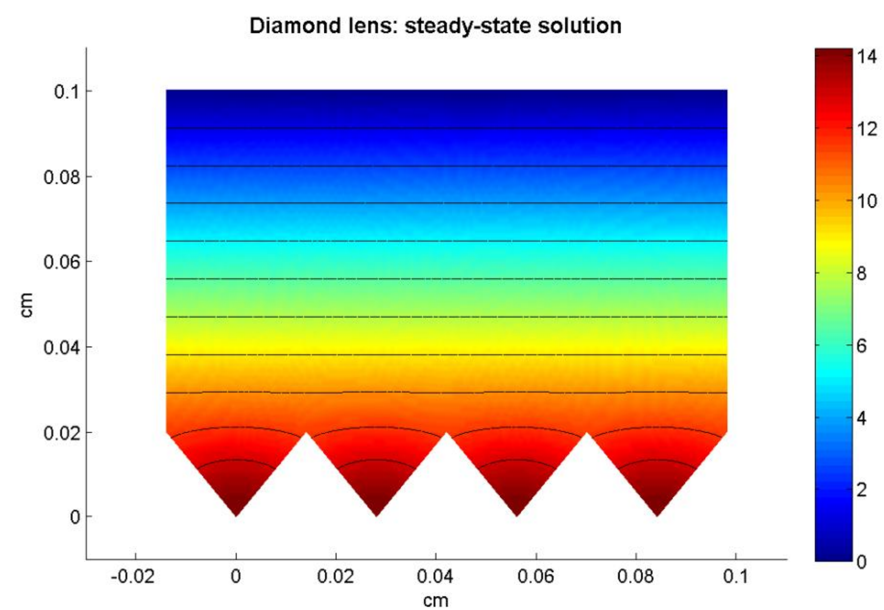




0.7 μm expected from theory

Heat absorption





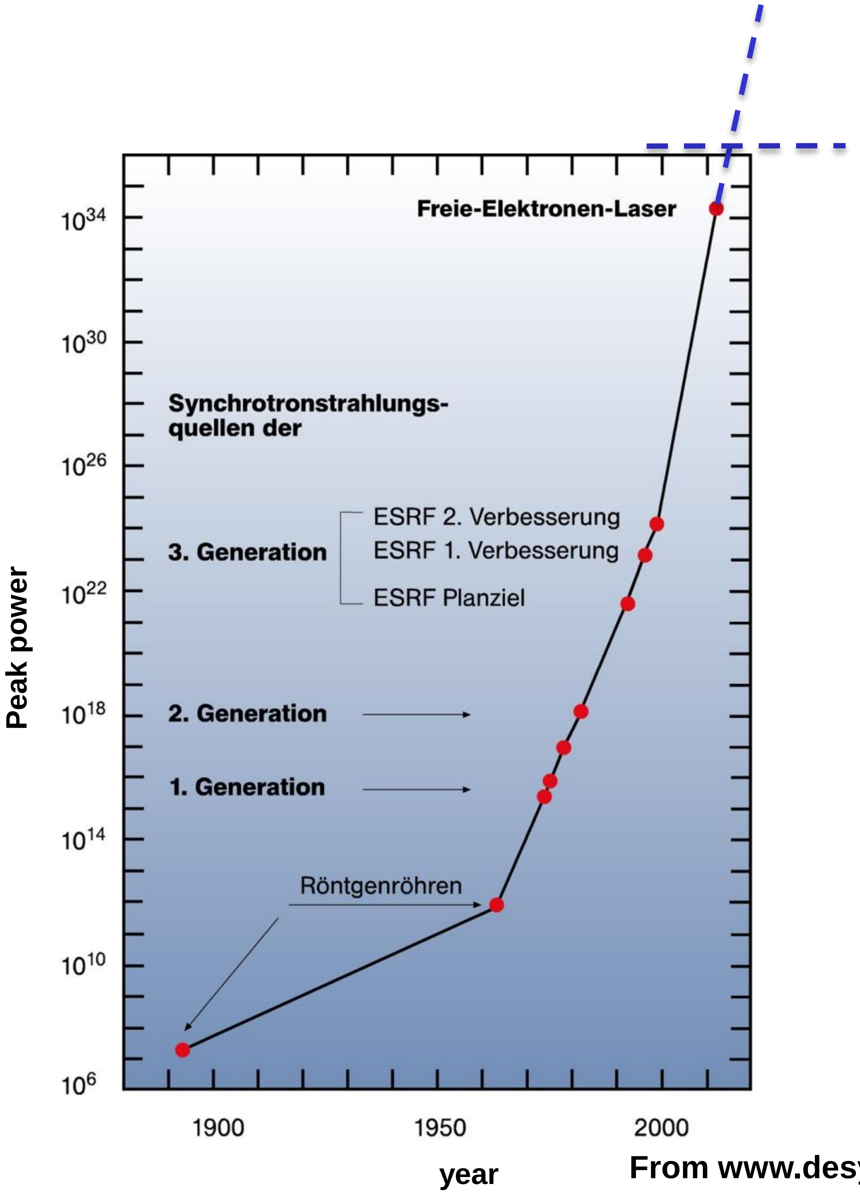
Extrame heal-load optics - conclusions

First laser-ablated diamond lenses showed promising focusing performance

For refractive optics in XFELs, Be and Diamond only feasible materials

For diamond ≈ 100x margin

Diamond-based FZPs
(C. David, PSI; group of H. Hertz, Stockholm)



From www.desy.de

Summar and Conclusions

- X-ray microscopy/imaging offers unique possibilities
- Hard x-ray resolution approaching the 1-nm limit
- Enabled by
  - development in sources and progress in micro-/nano-fabrication
  - new methods for wave-field retrieval
- Extreme flux and heat load on optics and specimens with new XFEO sources is a new challenge
- The future for hard x-ray microscopic imaging is bright