X-Ray Optics in Stockholm

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&
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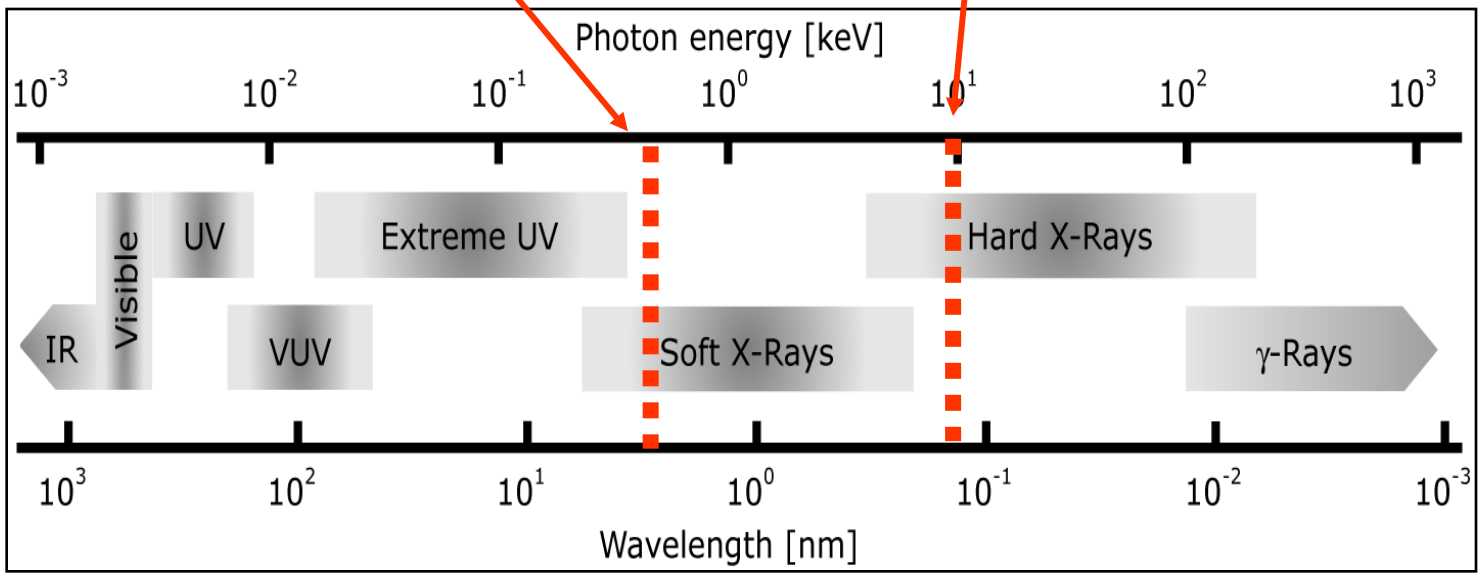
Applied Physics/KTH
Stockholm

Today

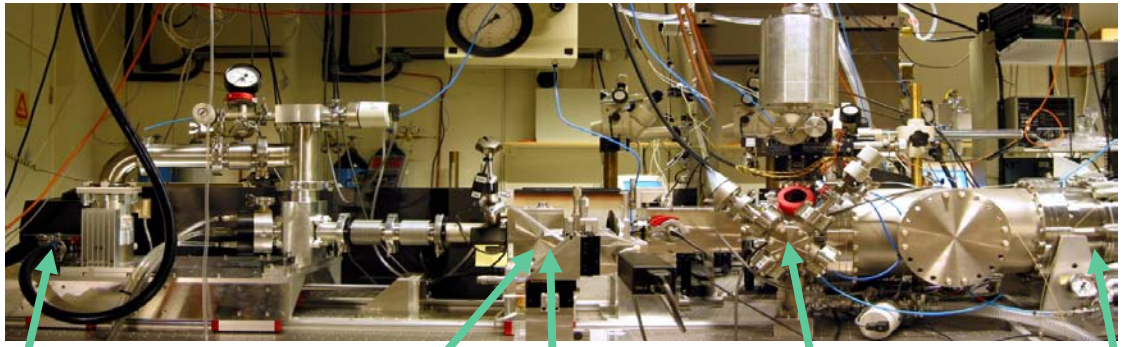
Soft x-rays :
Diffractive x-ray optics
for laboratory x-ray microscopy

Hard x-rays :
Diffractive x-ray optics
for MAX IV, XFEL, LCLS

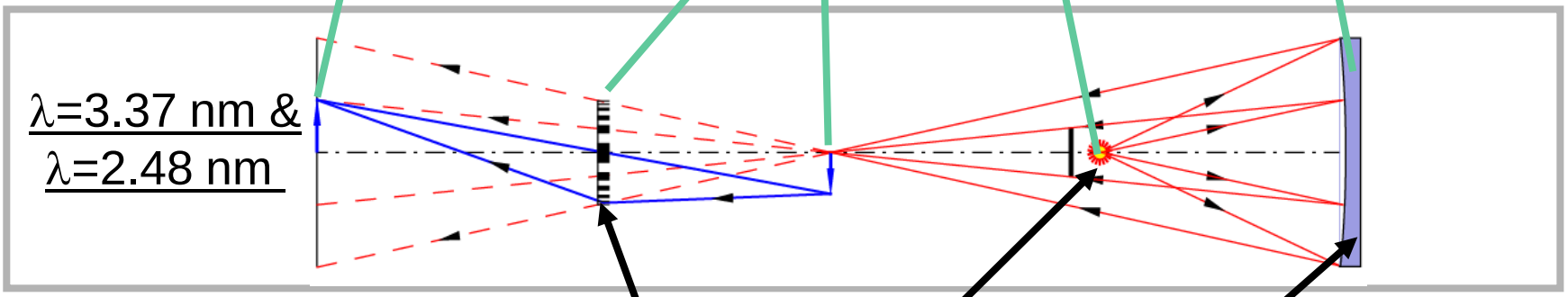


Soft x-rays:

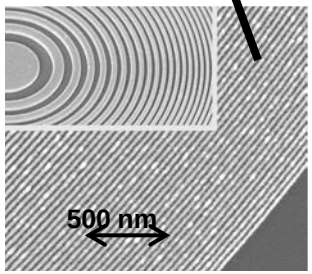
Laboratory water-window x-ray microscopy

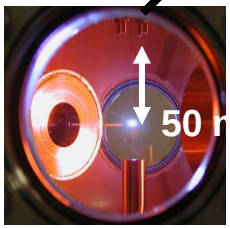


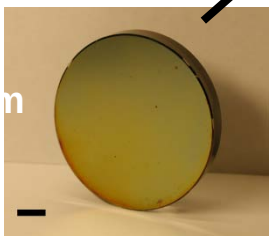
λ=3.37 nm &
λ=2.48 nm



Micro zone plates for high-resolution imaging







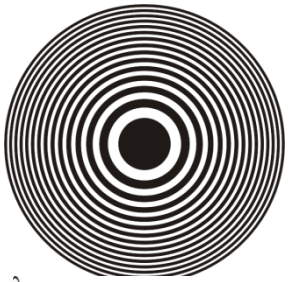
Normal-incidence multilayer mirrors as condensors

Berglund et al, J. Microsc. (2000), Johansson et al, RSI (2002) Takman et al, J. Microsc. (2007)

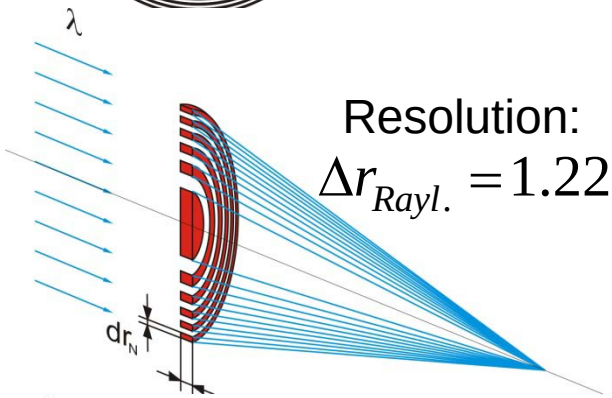
Soft x-ray optics:

Zone plates

Basics

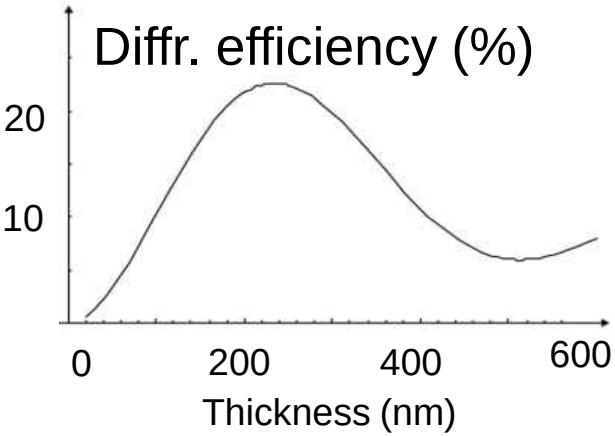


Circular diffraction gratings



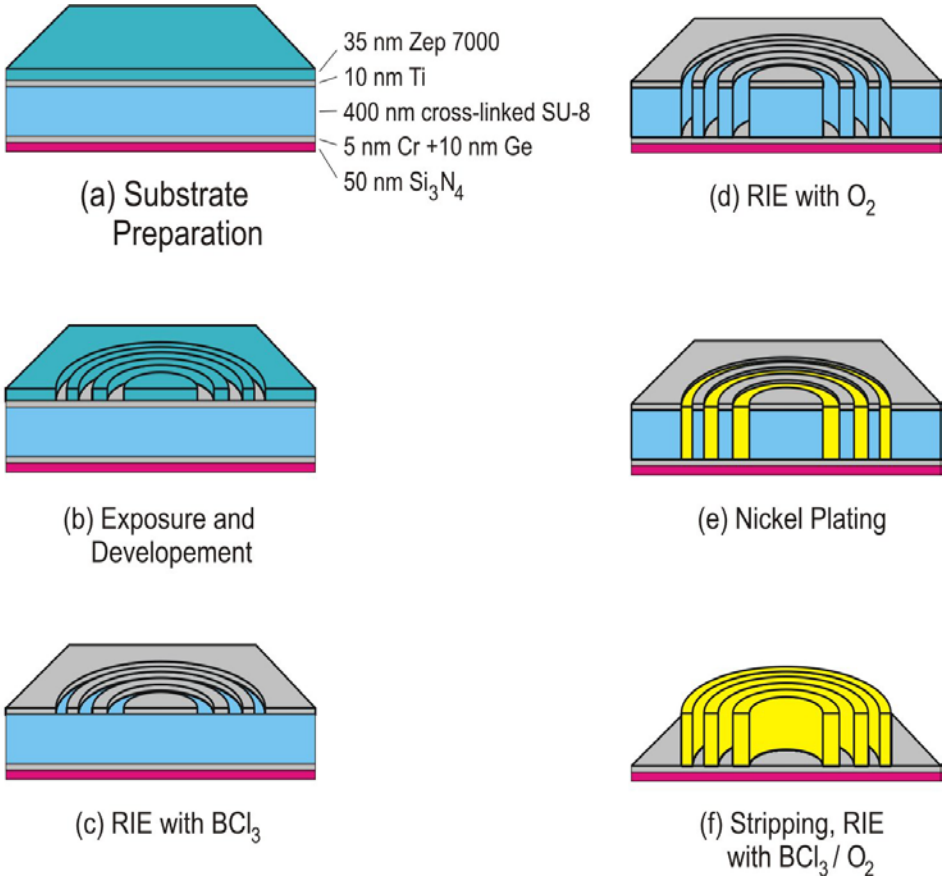
Resolution:

$$\Delta r_{Rayl.} = 1.22 dr_N$$



Nano-fab process for Ni zone plates

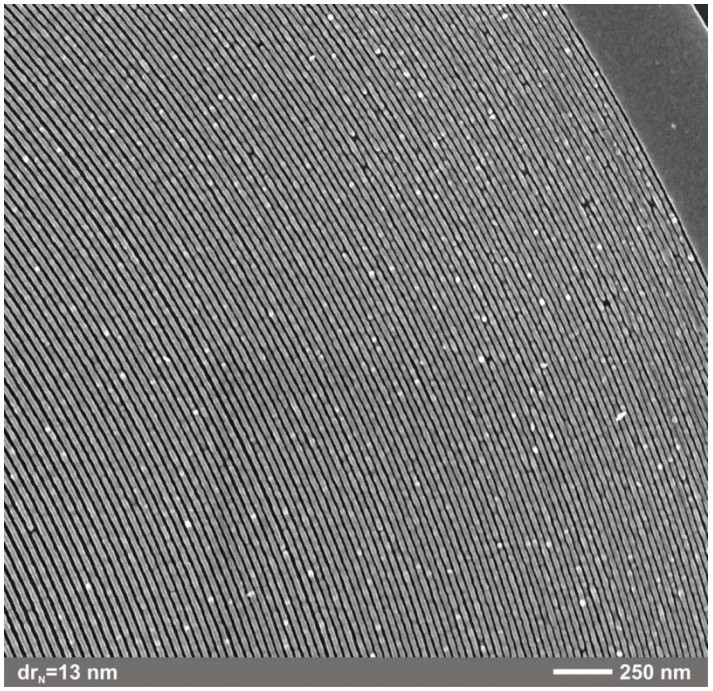
@ Albanova Nanofab Lab



Holmberg et al, MNE (2003),

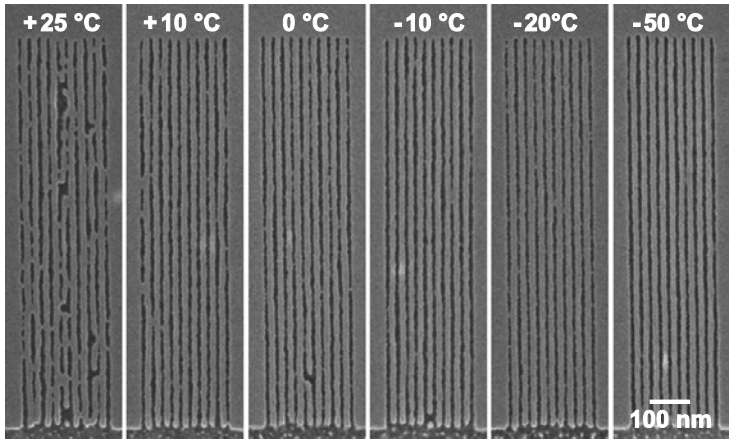
dr_N=13 nm Ni zone plates

Single-write Ni ZP

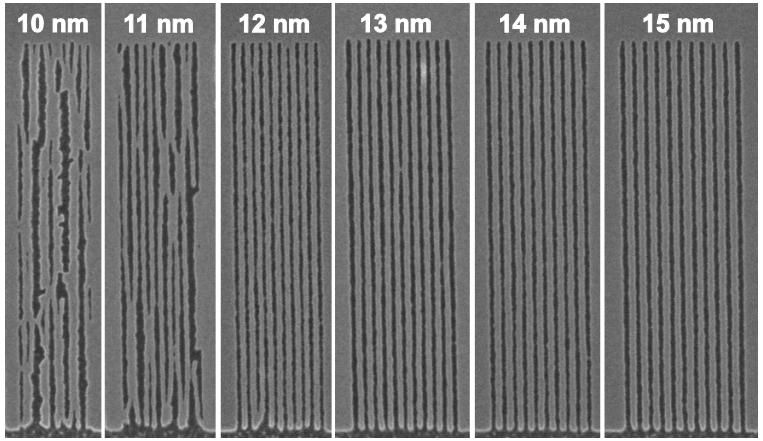


Ø=19 µm
dr_N=13 nm; h=35 nm
f=100 µm @ λ=2.48 nm
Excellent uniformity
Efficiency: 2.7% (15 nm/h=55 nm zp)

Cold development: ZEP in hex acet



Mold stability

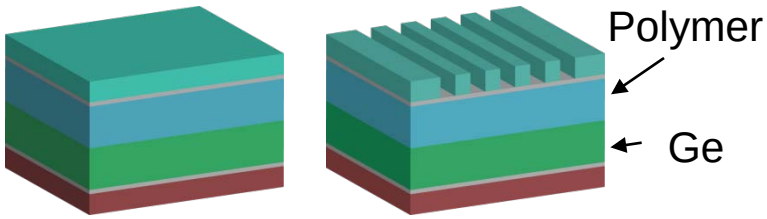


Reinspach et al, JVST B (2009)

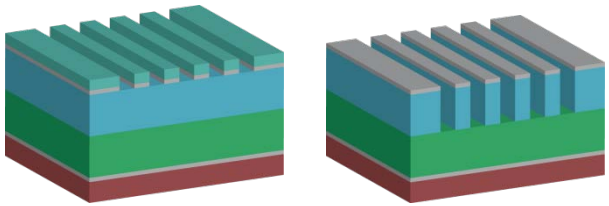
Multi-material zone plates:

Ni-Ge efficiency enhancement

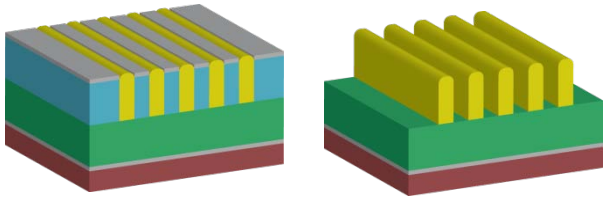
Our standard nickel process PLUS one extra step



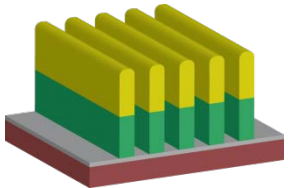
1. Substr. prep.
2. E-beam litho.



3. Ti etch
4. Polymer etch

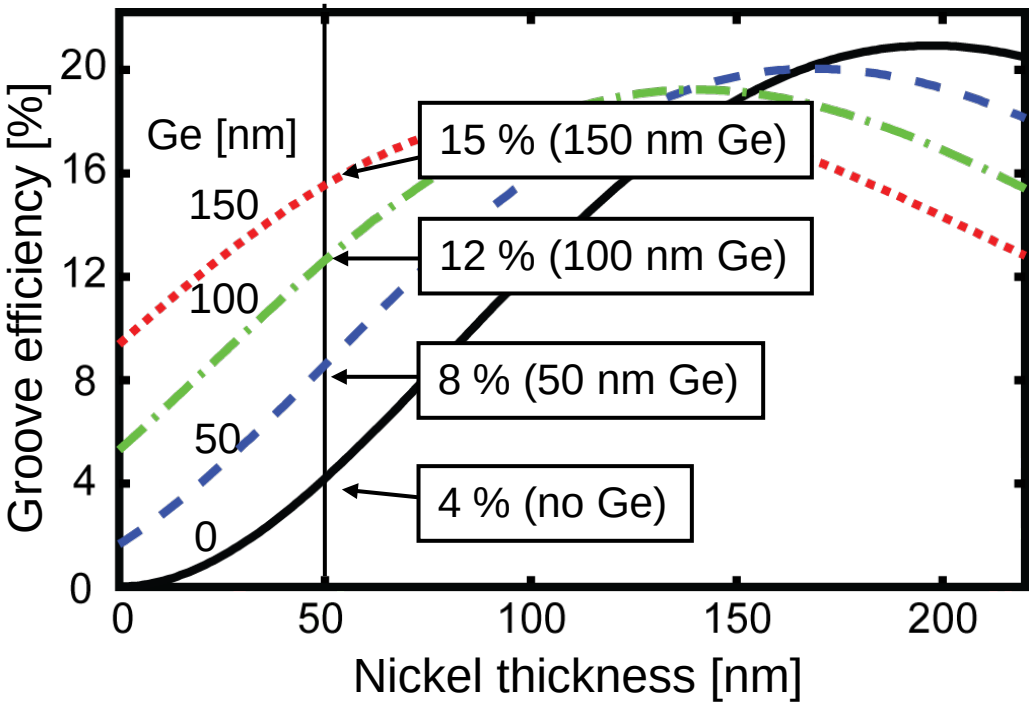


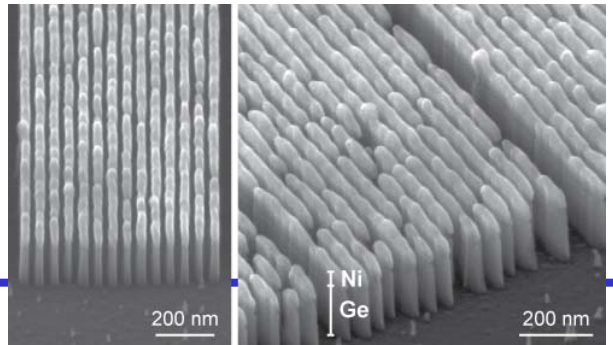
5. Ni plating
6. Mold strip



7. Ge etch, CHF_3

Multiply the efficiency of narrow-line Ni zone plates





30 nm zp made

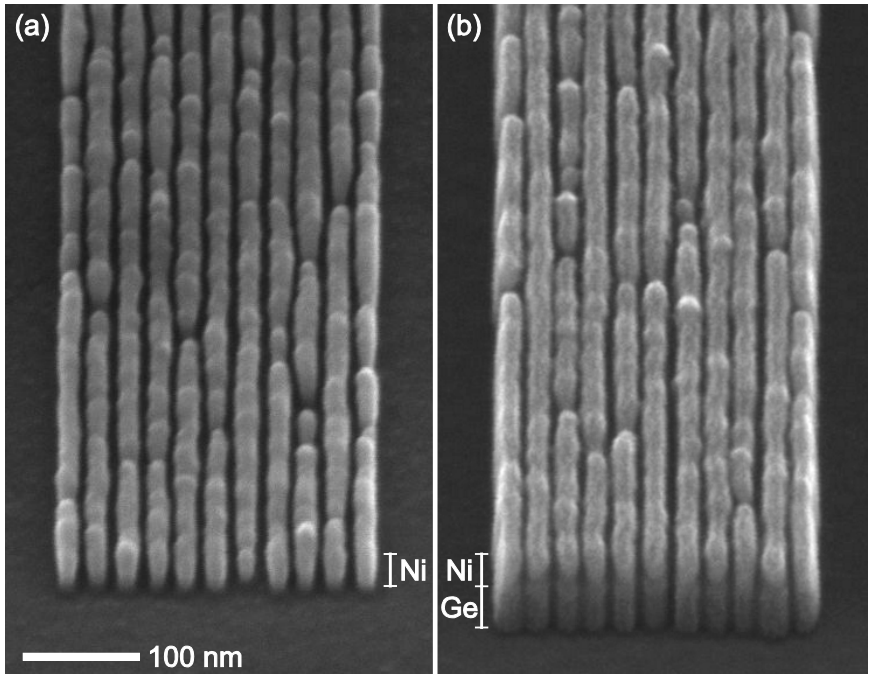
Lindblom et al, JVST (2009)

Recent results:

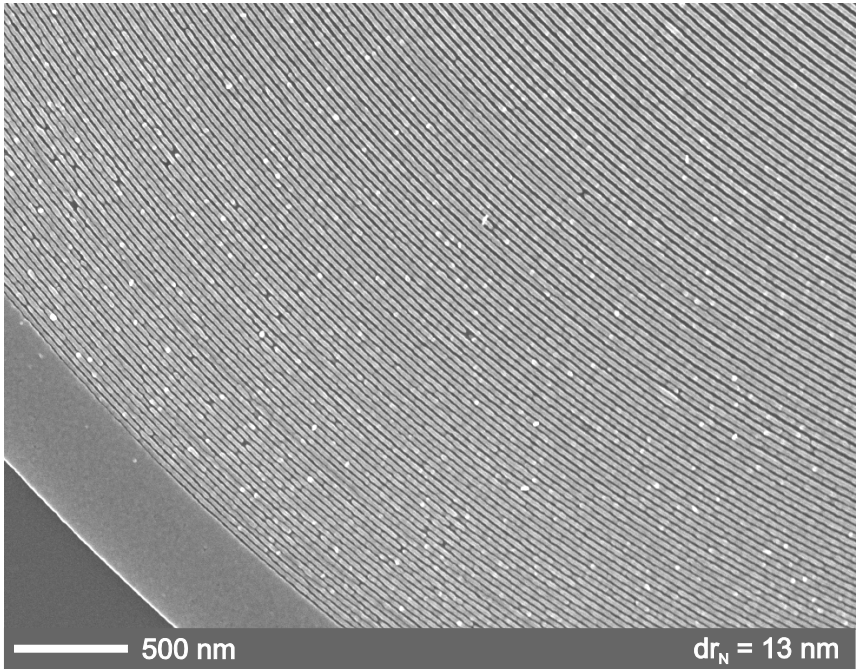
13 nm Ni-Ge zone plates

13 nm Ni-Ge gratings

w/ 35 nm Ni and 45 nm Ge



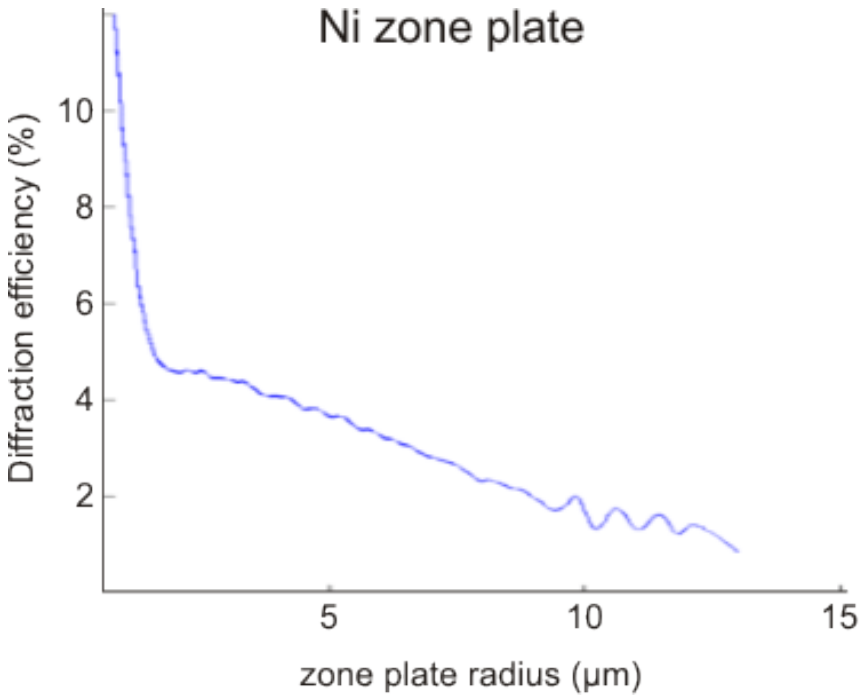
13 nm Ni-Ge zone plate

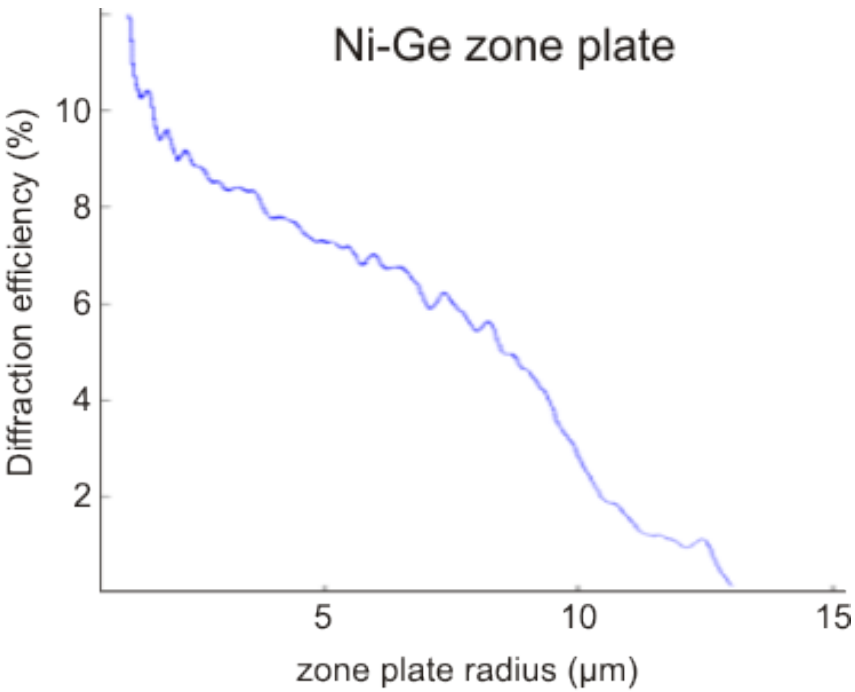


Diam: 19 µm
Focal length: 100 µm (@2.48 nm)
dr_N: 13 nm:
Thickness: 35 nm Ni + 45 nm Ge

Reinspach et al, JVST (2011)

15 nm Ni-Ge zone plate efficiency



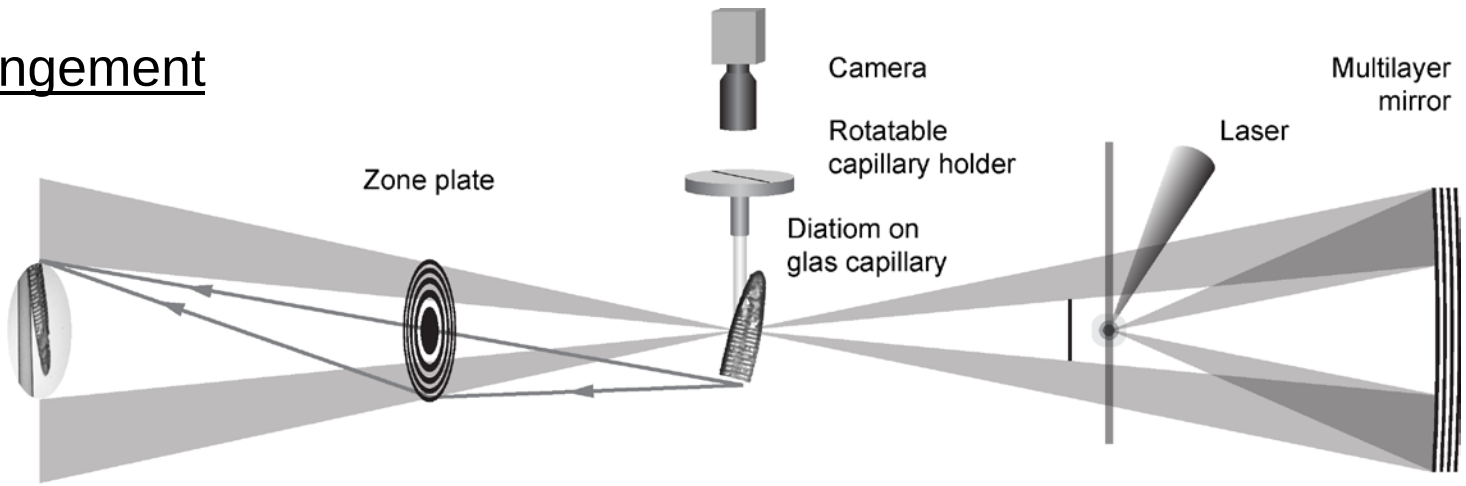


Average: 2.4%

Average: 4.3%

Cryo micro-tomography w/ lab. water-window XRM

Arrangement



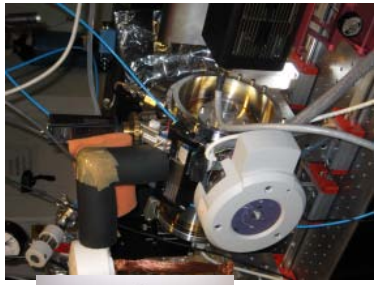
λ= 3.37 or
λ= 2.48 nm

Diatom reconstruction

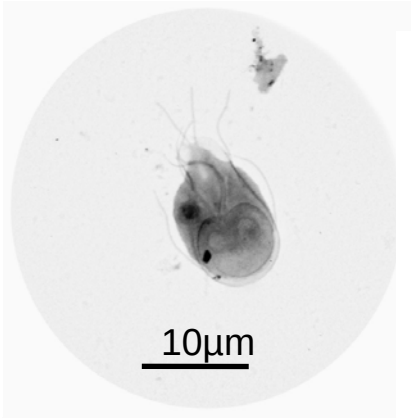
λ=3.37 nm
Filtered. back. proj.
53 projections
140 nm resol.

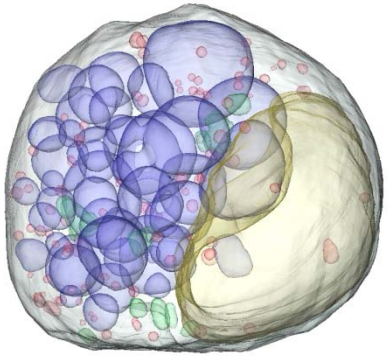
Bertilsson et al, Opt Expr (2009)

First lab cryo tomo: parasites and human kidney cell



λ=2.48 nm
Filtered. back. proj.
200 projections



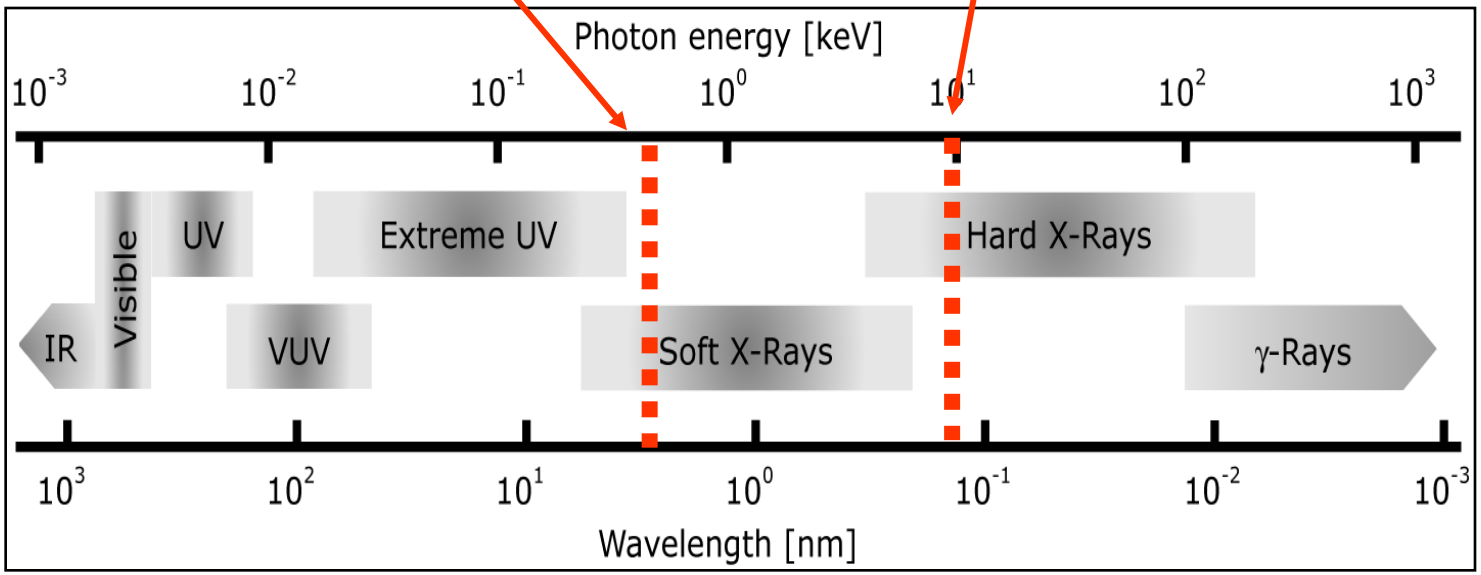


Bertilsson et al, Opt Lett (2011); Hertz et al J. Struct Biol (2012)

Today

<u>Soft x-rays :</u>
Diffractive X-ray optics
for Laboratory X-Ray Microscopy

<u>Hard x-rays :</u>
Diffractive X-Ray Optics
for MAX IV, XFEL, LCLS



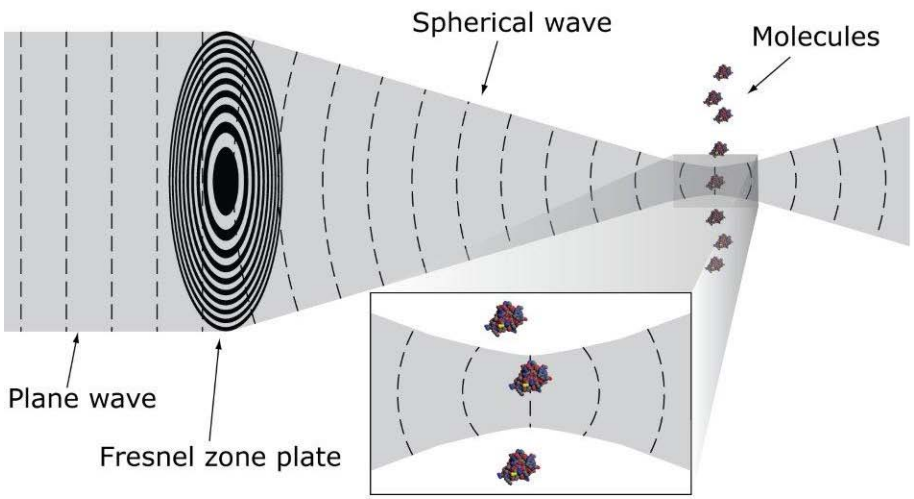
Hard X-Ray Diffractive Optics

(PI: Ulrich Vogt; Nanolab: Anders Holmberg)

XFEL @ Hamburg

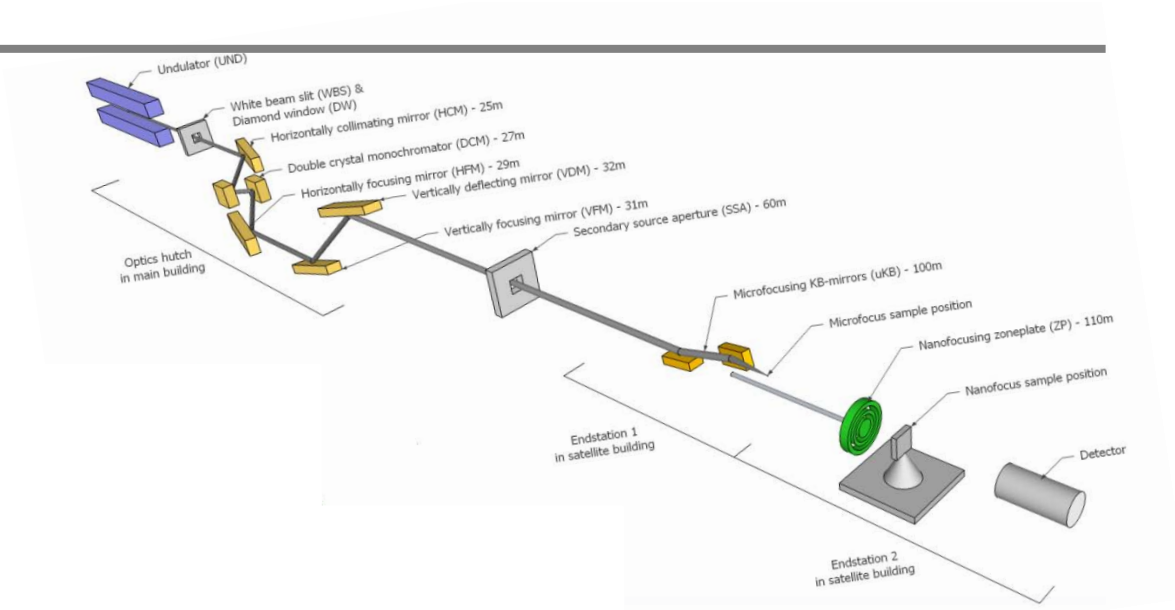
- New materials for substrate and optic
- Cooling of high heat load
- Large diameter (>1 mm)
- Diffraction-limited (low aberrations)
- Metrology for efficiency and wave front
- ”Mass production” of single-shot optics?

LCLS



High-brilliance sources

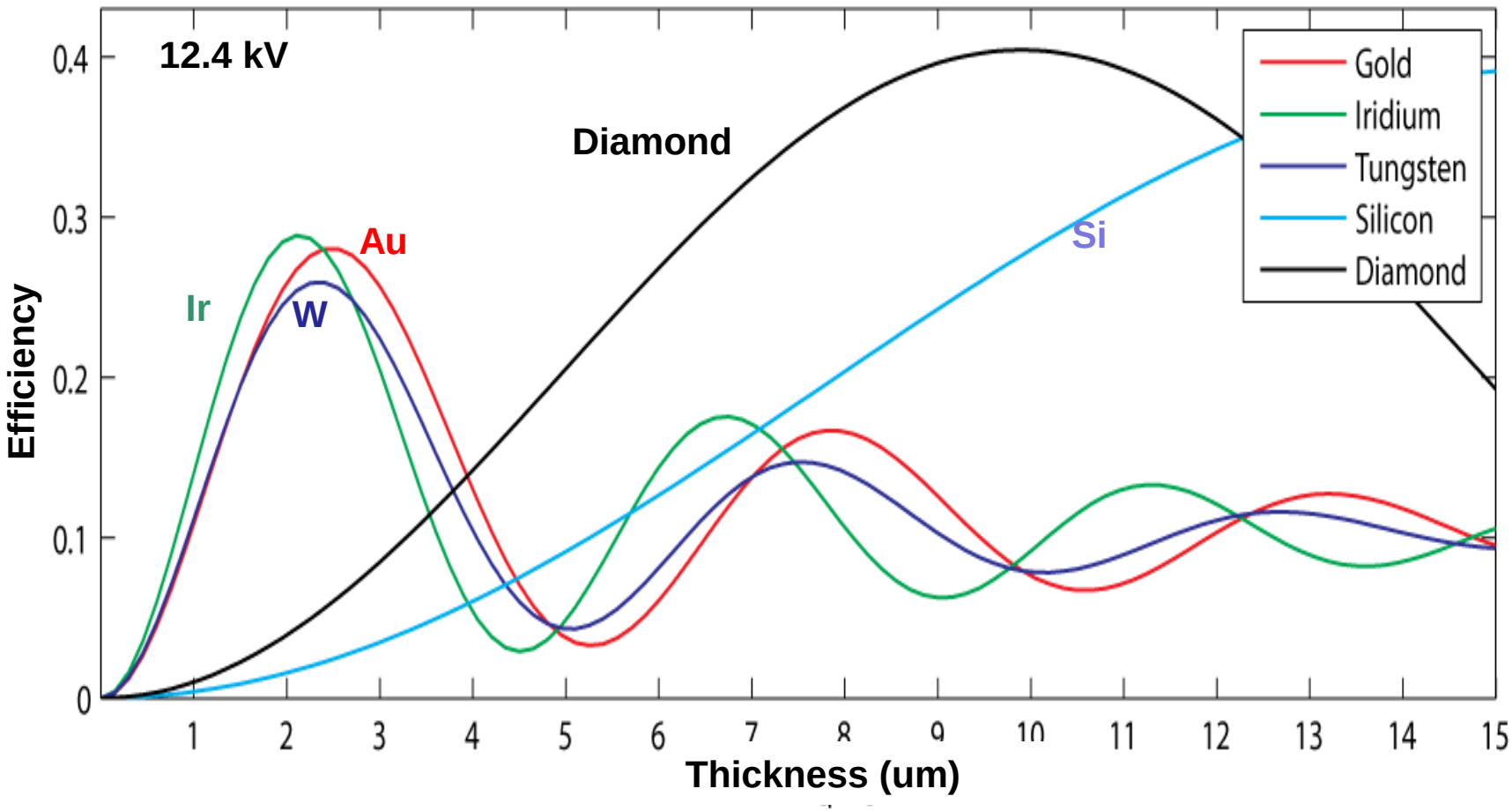
- MAX IV – NanoMAX BL



Lab sources

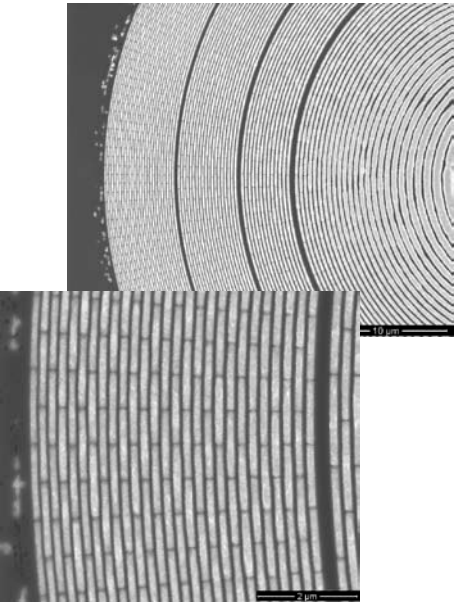
Hard x-ray zone plates:

Materials



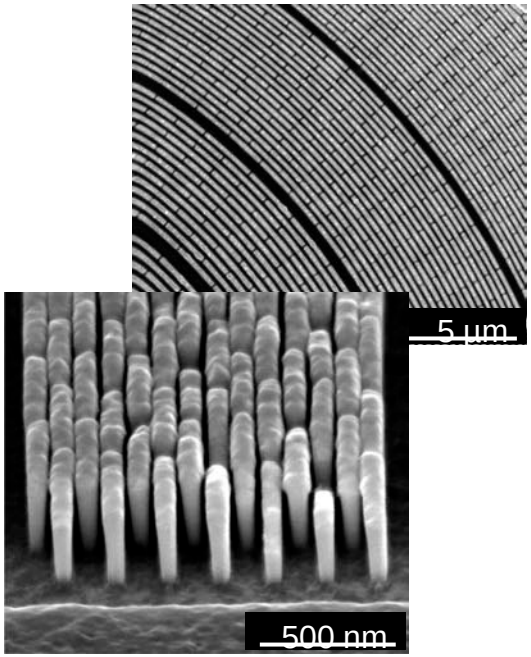
Hard X-Ray Metal Zone Plates:
Fabrication

Gold
Electroplated



100 nm half-period,
670 nm high,
8% eff @ 8 keV

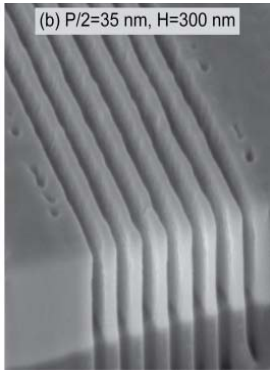
Platinum
Electroplated



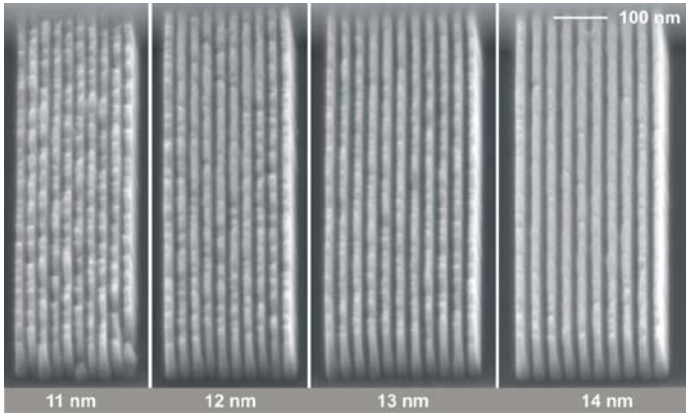
50 nm half-period,
400 nm high,
5.5-6.5 % eff @ 8 keV

Chubarova et al, Microel. Engin (2011).

Tungsten
SF_6 /O_2 dry etch



Half pitch: 35 nm
Height: 300 nm
Aspect ratio: 8.5



Half pitch: 11-14 nm
Height: 90 nm
Aspect ratio: 6-8

Reinspach et al, JVST B (2011)

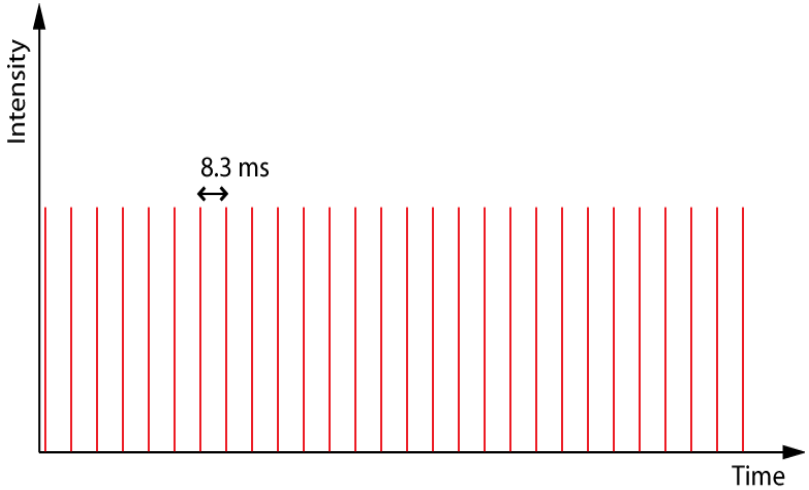
Thanks to R Barrett for eff. meas !

Free-electron lasers:
Source properties

	LCLS	European XFEL (SASE1)
Photon energy [Wavelength]	8 keV [0.15 nm]	12.4 keV [0.1 nm]
Pulse energy	2 mJ	2 mJ
Repetition rate	120 Hz	Trains of 2700 pulses in 0.6 ms
Beam size at lens position	750 μm (FWHM)	982 μm (FWHM)
E/ΔE	500	1000

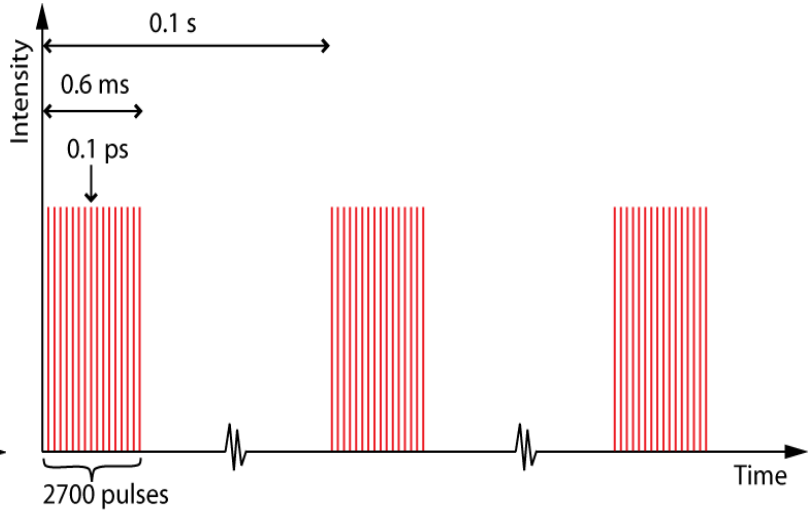
Single-pulse: ~200-300 mJ/cm²

LCLS



- 240 mW average power

European XFEL

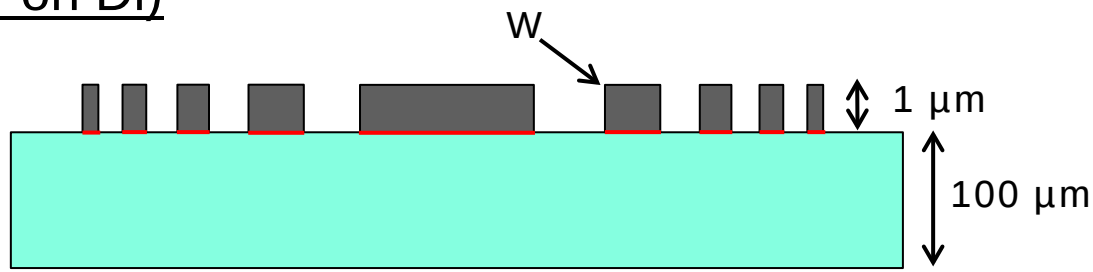


- 54 W average power
- 9000 W in pulse train

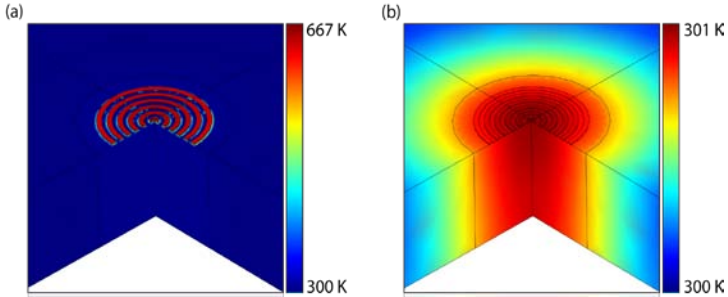
Hard x-ray metal zone plates:

Thermal-Load Simulations

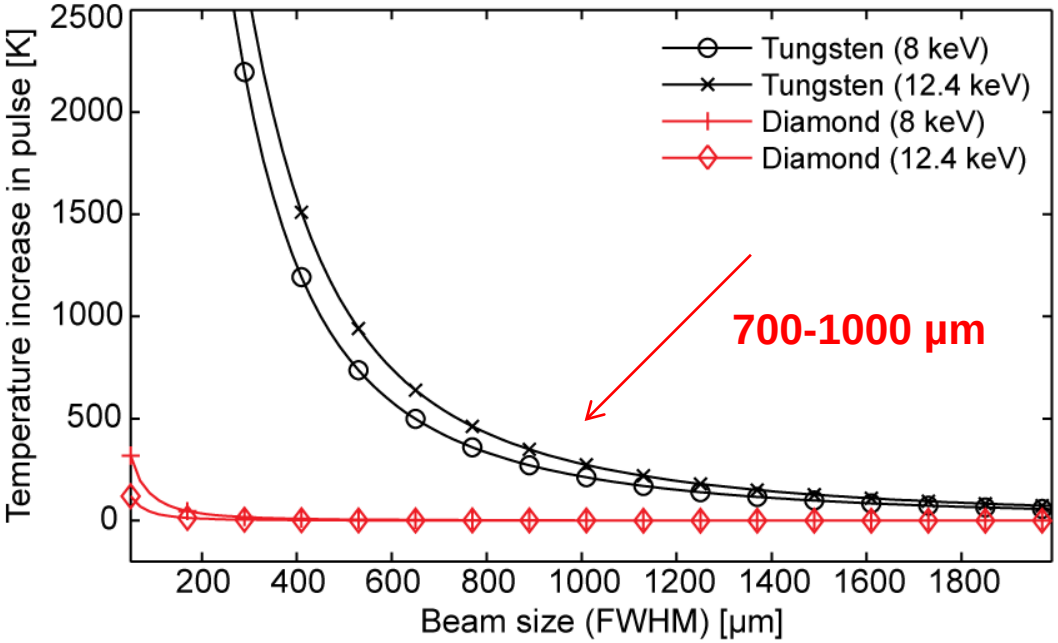
Tungsten on diamond (W on Di)



Single-pulse simulation



t=0 t=5 µs



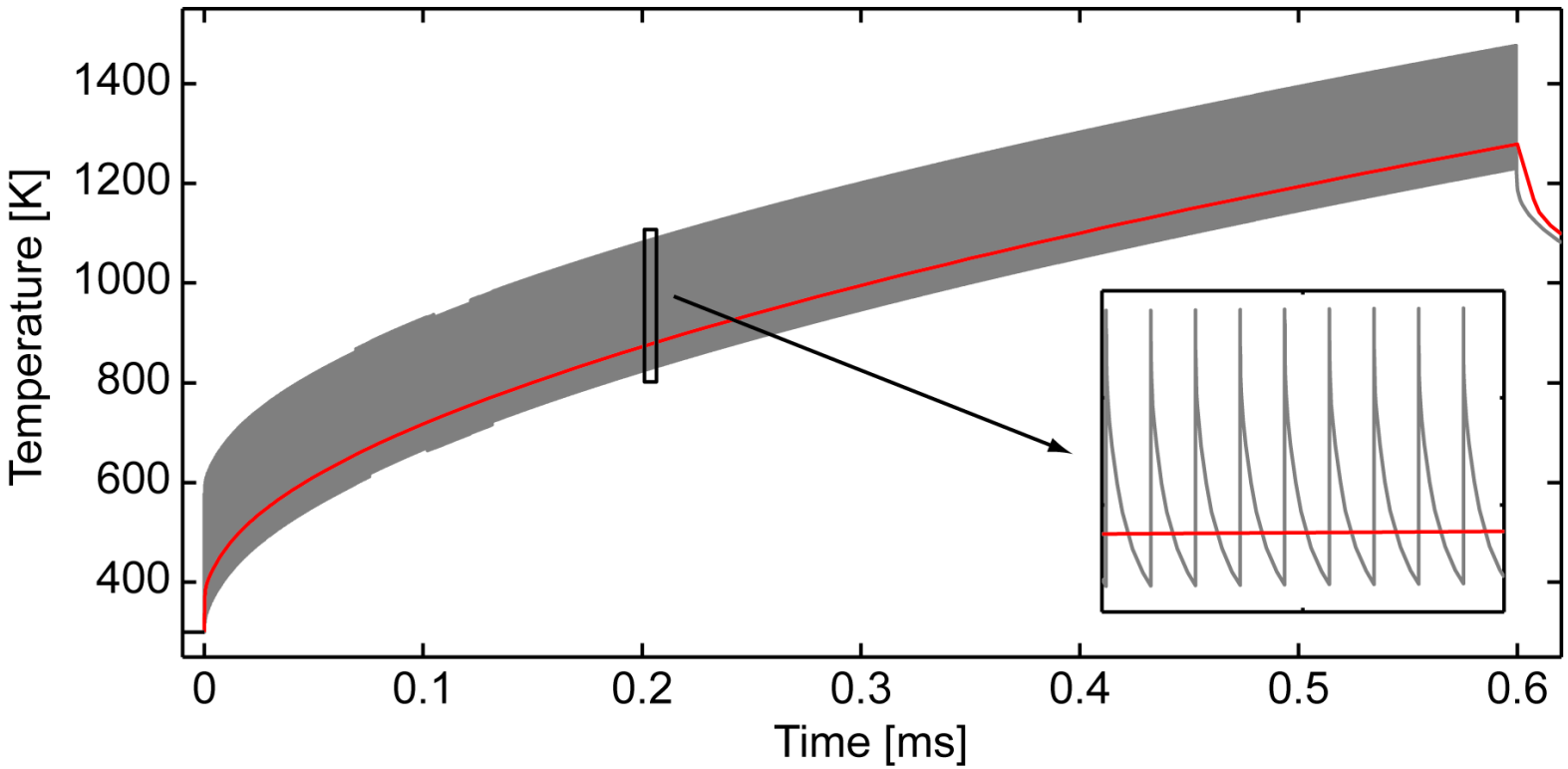
Nilsson et al, NIM A (2010), Nilsson et al, SPIE (2011)

W on Di zone plates:

Thermal-Load Simulations for XFEL

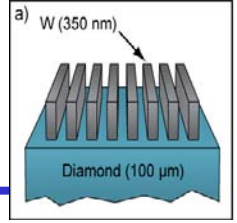
SASE1 @ XFEL:

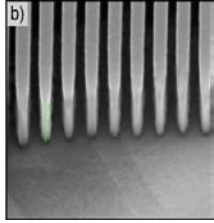
Temperature in center of zone plate (Full pulse train)



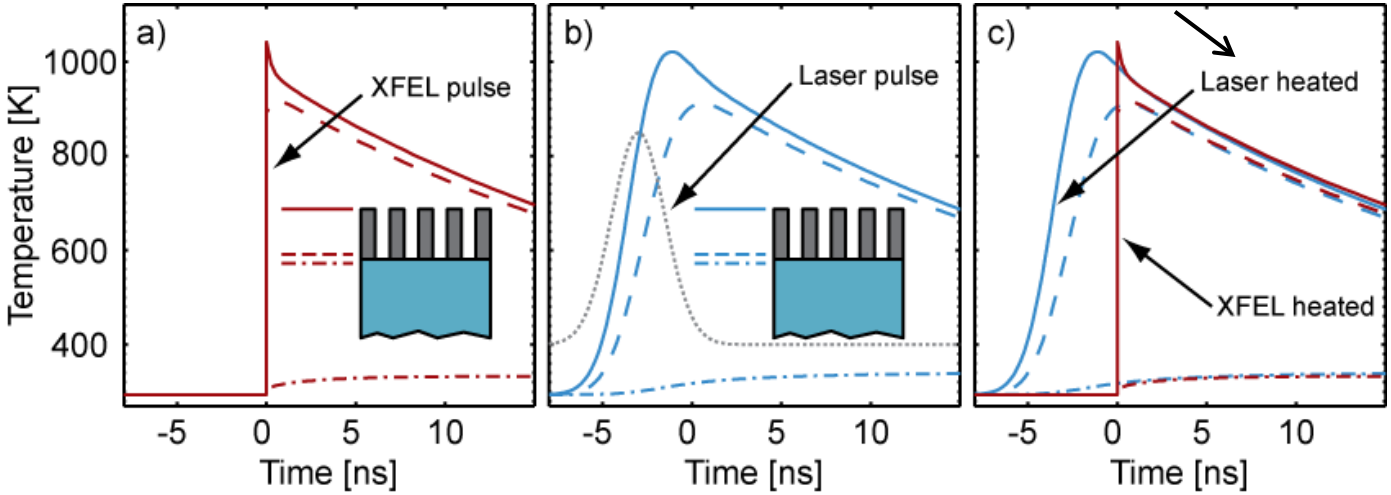
Nilsson et al, NIM A (2010),

Experiments vs Theory:
YAG laser heating & first LCLS exp





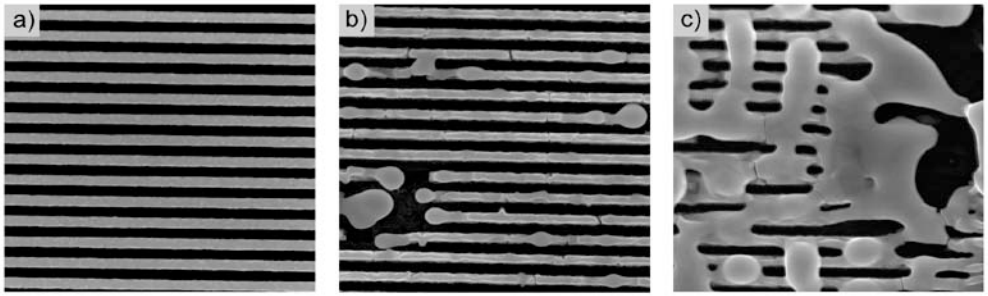
Single-pulse YAG-XFEL calibration



XFEL:
1mJ/6kV/350 mJ/cm^2

Corresponds to
532 nm YAG:
3.5 ns/57 mJ/cm^2

Low-rep-rate (20 Hz) YAG- laser operation:



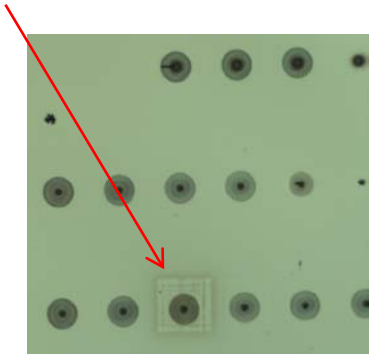
100 mJ/cm^2
1.7×10^6 pulses

180 mJ/cm^2
7×10^4 pulses

360 mJ/cm^2
1.2×10^3 pulses

100 mJ/cm^2 YAG ⇔590 mJ/cm^2 XFEL

First LCLS exp



6 kV, 120 Hz
1 mJ/500 µm

350 mJ/cm^2
10^5 pulses

LCLS exp: Thanks to Ch. David, PSI

Nilsson et al, submitted (2012)

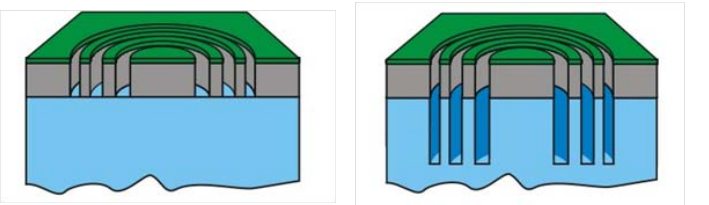
Multi-material zone plates:

Tungsten-Diamond (W-Di)

Why W-Di?

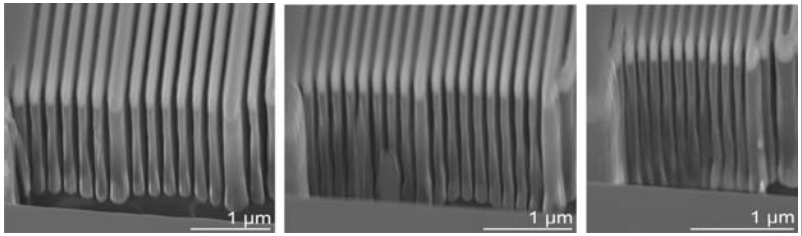
W ⇒ Small dr_n
High diffr efficiency
Di ⇒ High thermal conduct
Contributes to effic.

W-Di process



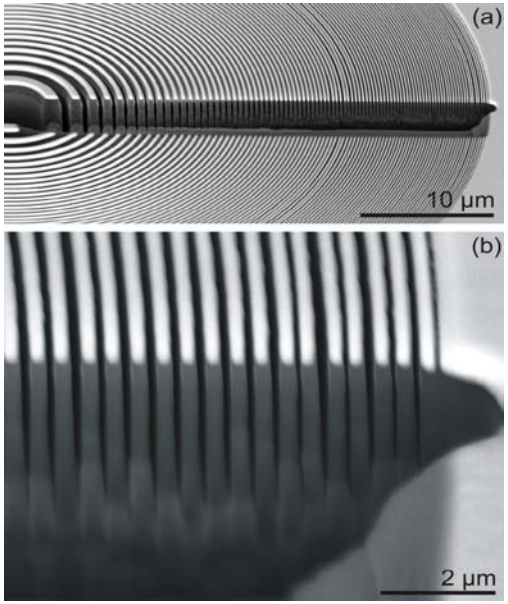
(a) Tungsten zone plate (b) Tungsten - diamond zone plate

SF_6/O_2 O_2



100 80 60
L/S (nm)

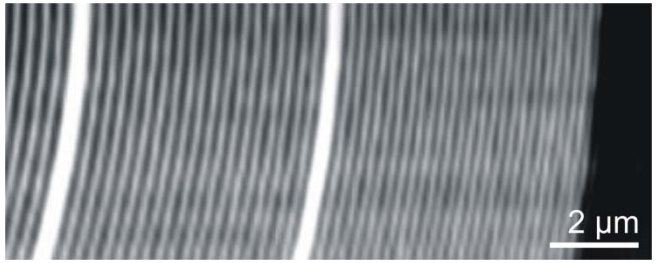
Results



W-Di zone plate
~200 nm W + ~2 µm Di
dr_n: 100 nm
Diam: 75 µm

Effic @ ESRF
dr_n=100 nm: 14%
dr_n= 50 nm: 7%

Imaging



100 nm W-Di zp images
100 nm L/S (Au)
@ ESRF

Uhlén et al, JVST (2011)

Summary & Future

- <u>Soft X-Ray Diffractive Optics:</u>
  - Improved efficiency with Ni-Ge multimaterial zone plates
  - Approaching 10 nm zone plates
  - Application: Laboratory x-ray microscopy
    - Approaches synchrotron quality
    - Resolution: <25 nm features
    - Contrast: improved via phase optics and system optimisation
    - Cryo 3D imaging
  - Next:
    - Increase efficiency
    - Shorter exp times w/ new laser; improved optics and system design
    - Applications: soils, colloids, cells, carbon content
- <u>Hard X-Ray Diffractive Optics:</u>
  - W on Di
    - Thermal properties appear OK for both LCLS and XFEL
    - Fabrication approaches 1:10 aspect ratio
    - W-Di increases efficiency
  - Next
    - Wavefront control
    - Prove small (<50 nm?) focus at LCLS
    - Increase efficiency.
    - NanoMAX BL @ MAXIV (U. Vogt et al)

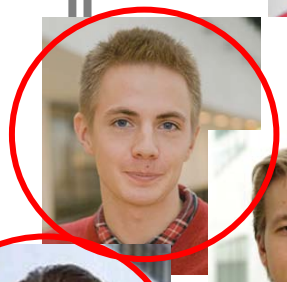
Biomedical & X-Ray Physics group

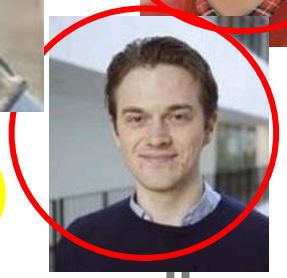
Thanks!










E. H.

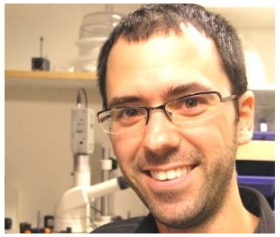
T. Z.

Soft x-rays:

Sources, optics, and microscopy

Hard x-rays:

Sources, optics & imaging

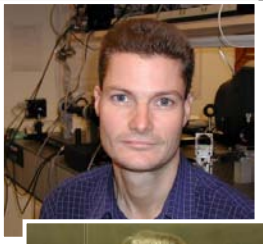
Ultrasonics & μ-fluidics:

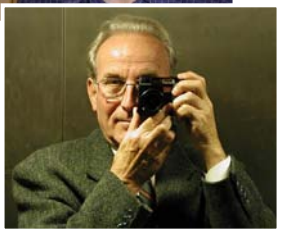
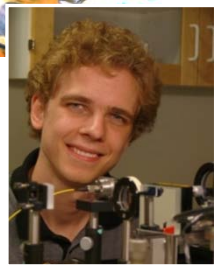
Bio-analytics and cell biol.





Teaching & technical



Optics:

Peripheral vision