PAUL SCHERRER INSTITUT

PSI

Christian David :: Paul Scherrer Institut (PSI)
Photon Science Division (PSD)
Laboratory for X-ray Nanoscience and Technologies (LXN)

Nanofabrication of diffractive X-ray optics – opportunities for neutron imaging?

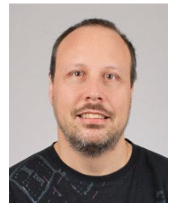
NFO workshop at PSI, March 2-3, 2023

PAUL SCHERRER INSTITUT PSI

X-ray Nano-Optics Group

present and (some) former members

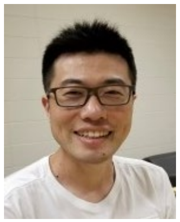
Christian David
Group head



Joan Vila-Comamala
Tenure track scientist

Adam Kubec
now XRnanotech AG

Florian Döring
now XRnanotech AG

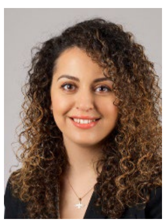


Peng Qi
PostDoc

Istvan Mohacsi
now PSI controls

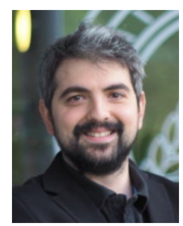
Mission:

To develop novel X-ray optical devices and instrumentation for Large Scale Facilities by applying advanced micro- and nanolithography



Nazanin Samadi
PostDoc

Talgat Mamyrbayev
PostDoc



Umut Sanli
now KNF Holding

Marie-Christine Zdora
now SLS Tomcat

Christian Grünzweig
now anaxam.ch

Mano Raj Dhanalakshmi Veeraraj
PhD student

Shuai Zhao
visiting PhD student

Diffractive X-ray optics developments at PSI

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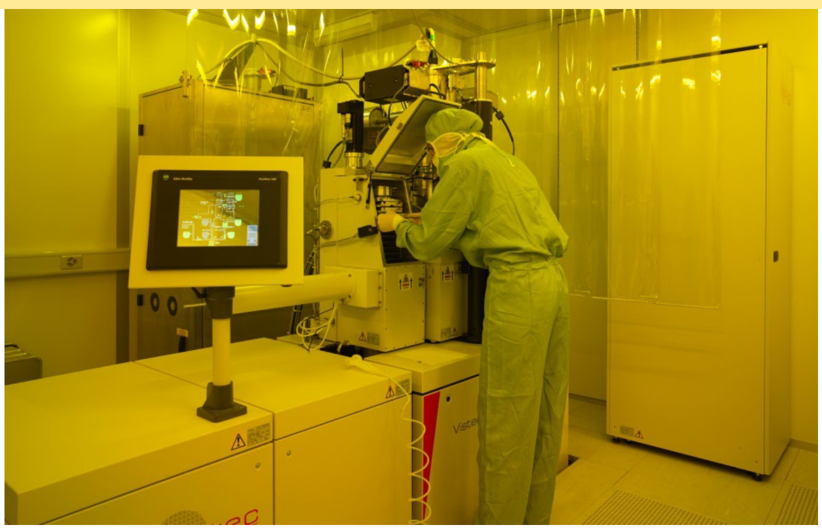


X-ray optics

X-ray refractive index:

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

with δ close to 0 and δ>1

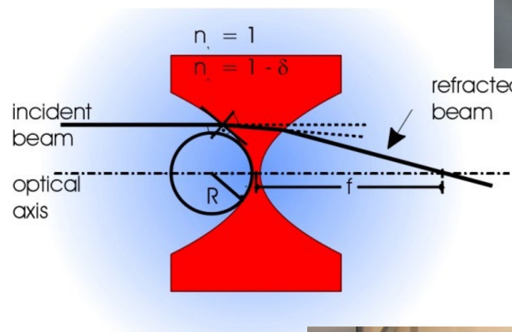
X-ray lenses:

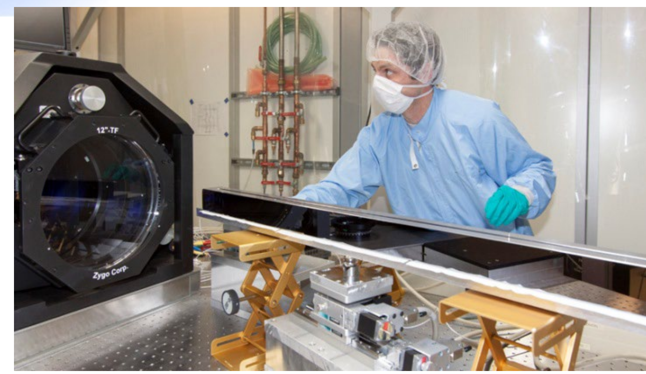
- Weak refracting power: $f = R/2\delta$
- Lossy, especially for soft x-rays
- Resolution limited by shape errors to typ. few 100 – 1000 nm
- **Very chromatic: $f \sim E^2$**

X-ray mirrors:

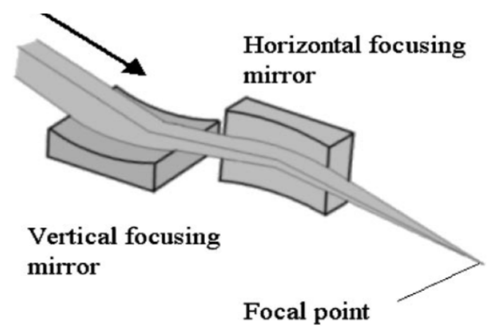
- Grazing incidence: $\alpha_{crit} \approx \sqrt{2\delta}$
- Can reach spot sizes < 100 nm, when shape errors are < few nm
- Long, bulky, complex, expensive
- **Achromatic**

Set of Beryllium lenses





1 meter long mirror at the XFEL.EU



Kirkpatrick-Baez mirror

Diffractive X-Ray Optics

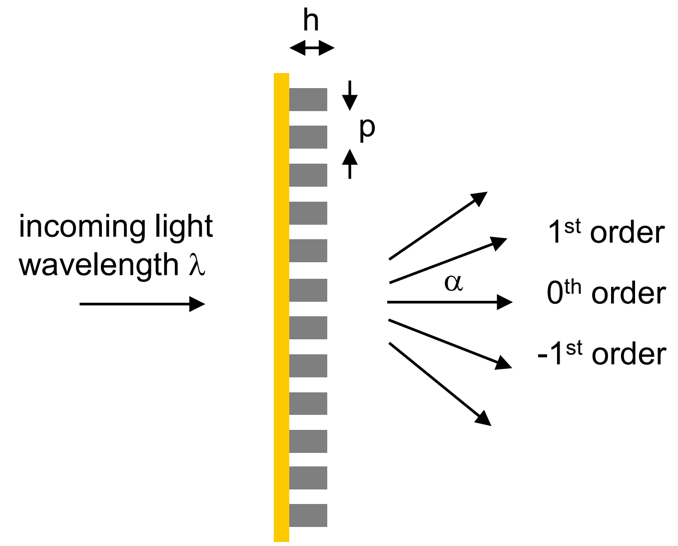
Diffractive optics: periodic structures on a transparent substrate

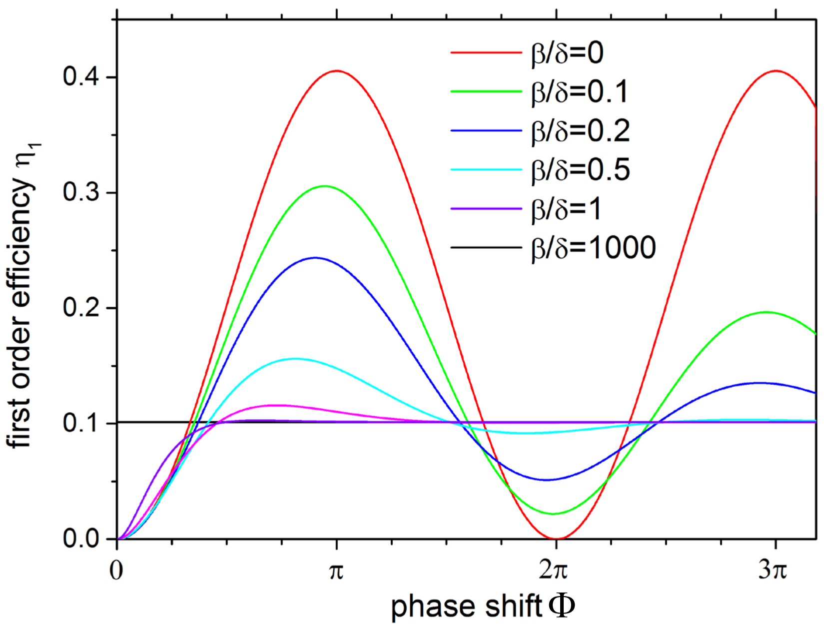
Chromatic: diffraction angle $\alpha \sim \lambda$

Several diffraction orders exist

=> efficiency is limited

$$\eta_1 = \frac{1}{\pi^2} \cdot \left(1 + e^{-2\beta/\delta \cdot \phi} - e^{-\beta/\delta \cdot \phi}\right) \cdot \cos\phi$$





Diffractive X-Ray Optics

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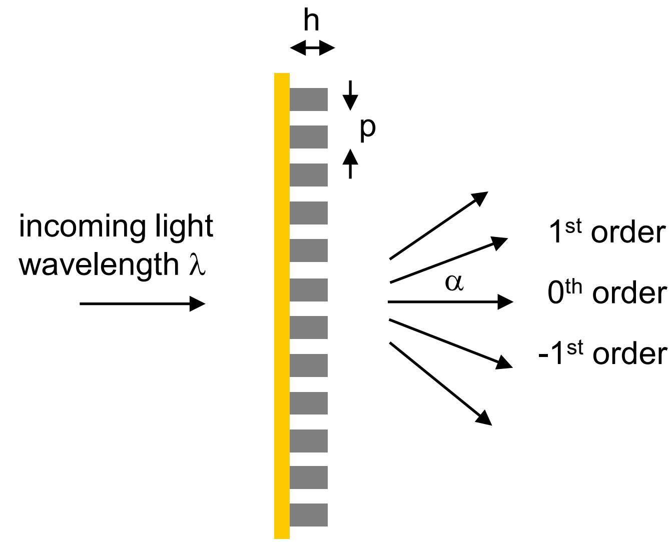
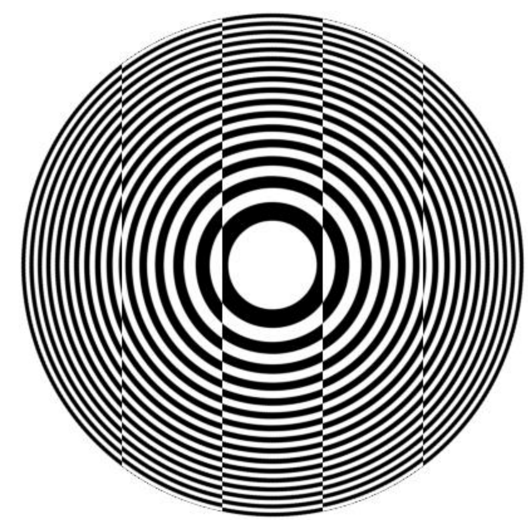


Wave front control:

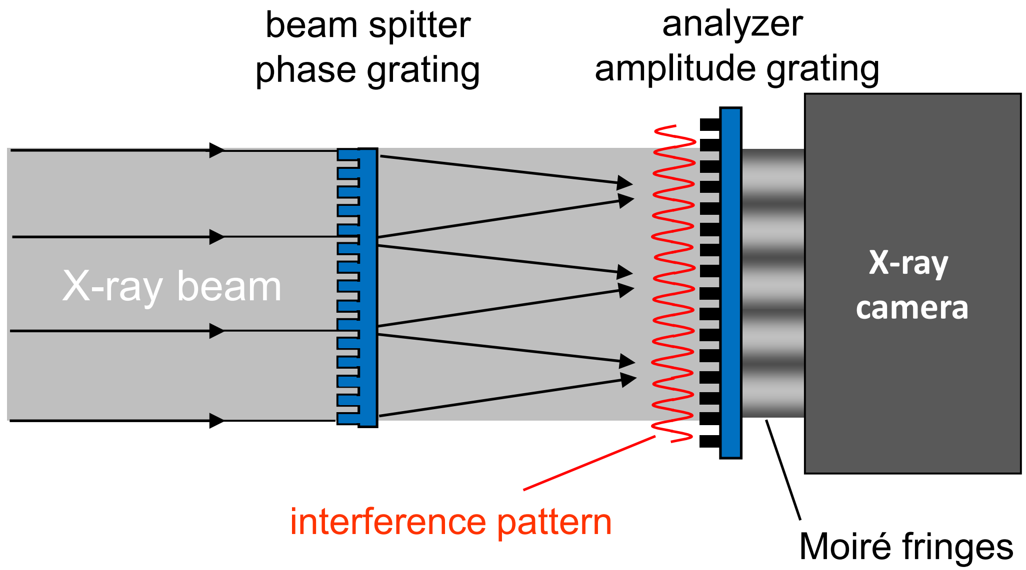
Pattern distortion of p/2

Wave front distortion of $\lambda/2$



Fresnel zone plate with two foci

Grating interferometry

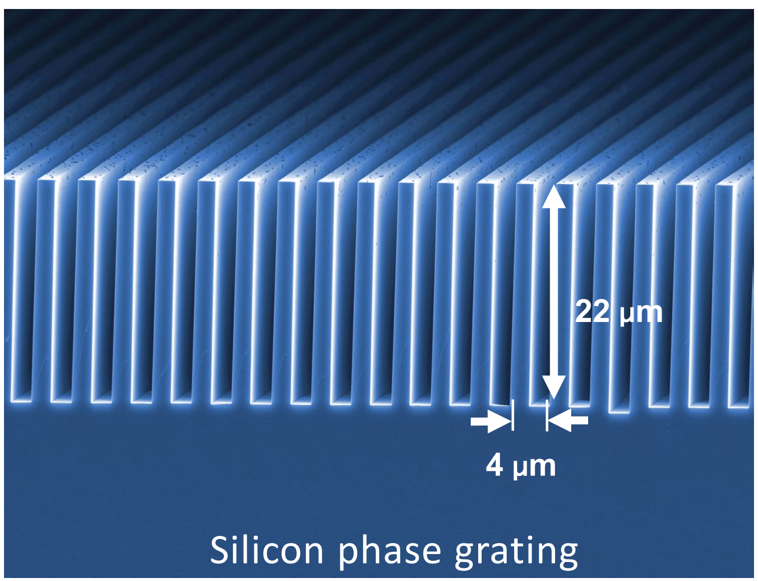


Distorted incident wave front

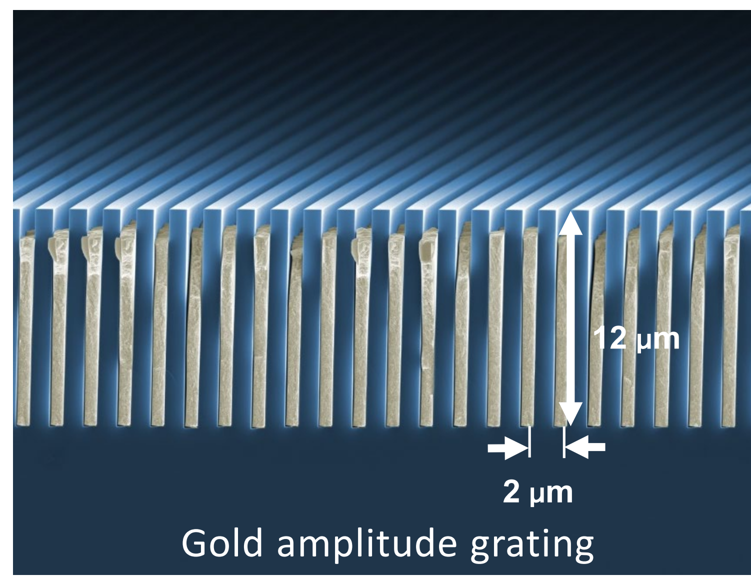
Distorted Moiré fringes

Accuracy of grating lines: p/100

Wave front sensitivity: λ/100

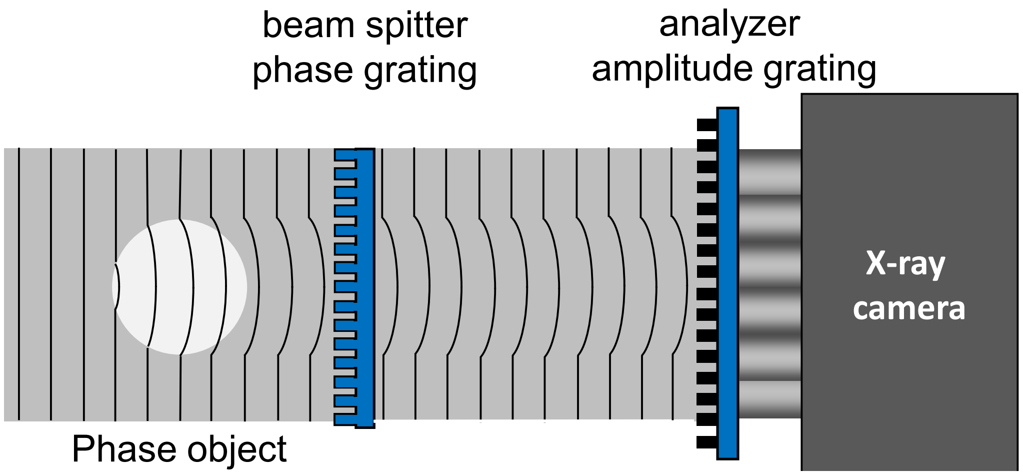


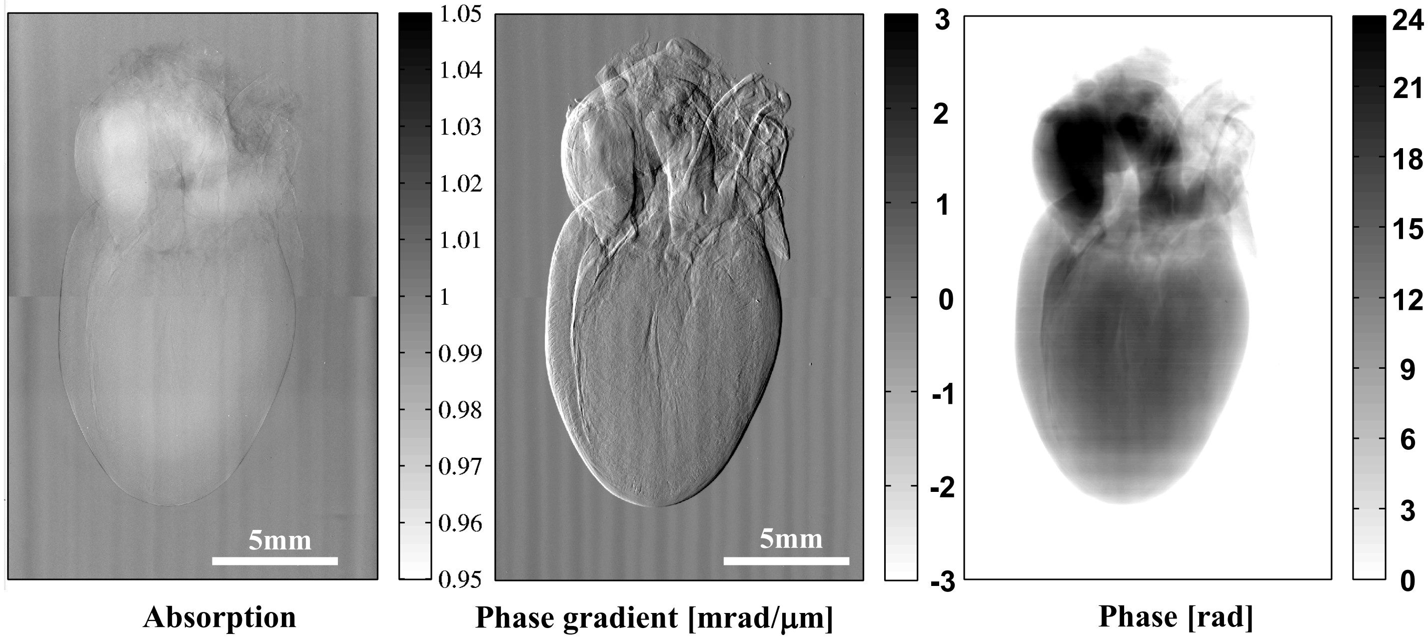
Silicon phase grating



Gold amplitude grating

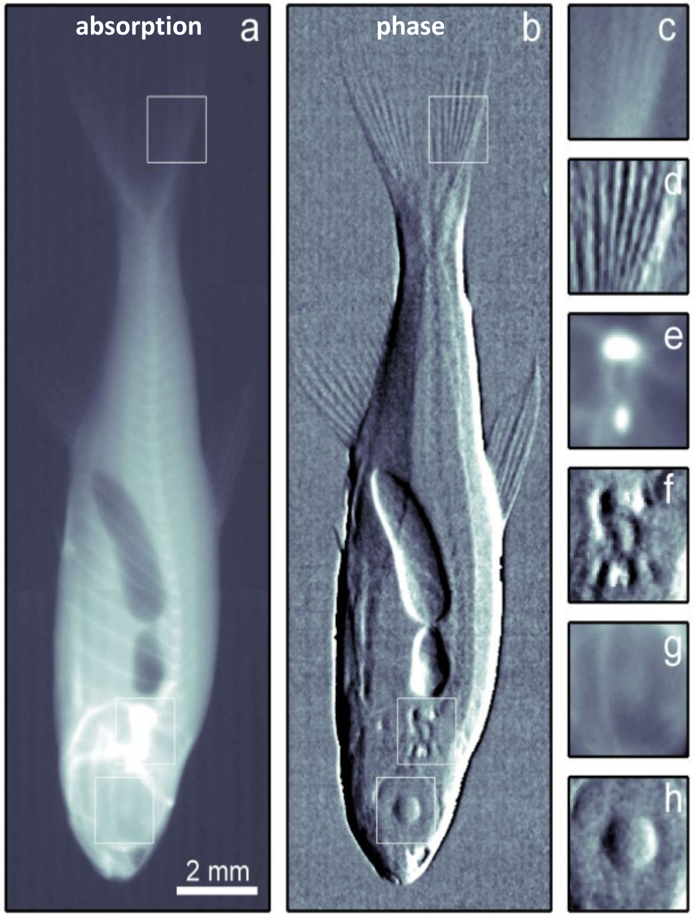
X-Ray phase contrast imaging



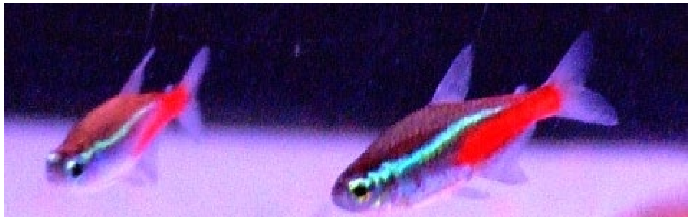


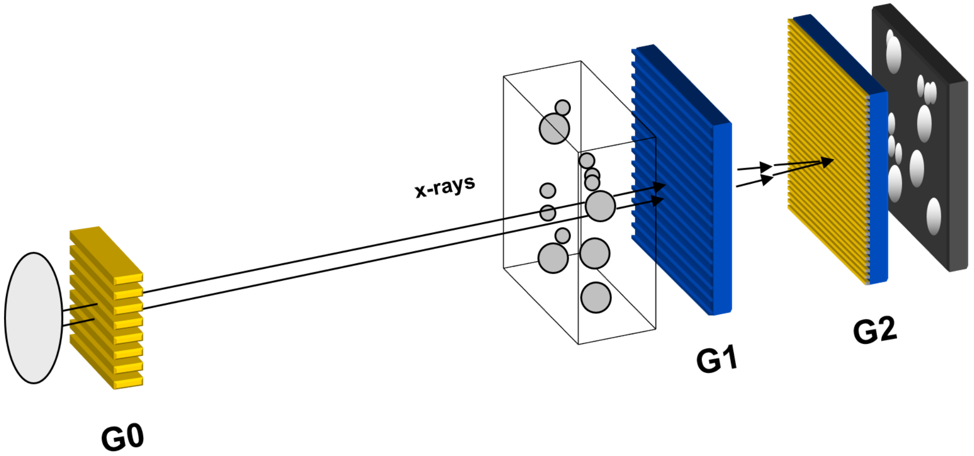
Rat heart imaged at 17.5 keV

X-ray phase contrast imaging at x-ray tubes



Radiography of a small fish (E=17.5 keV) 2006



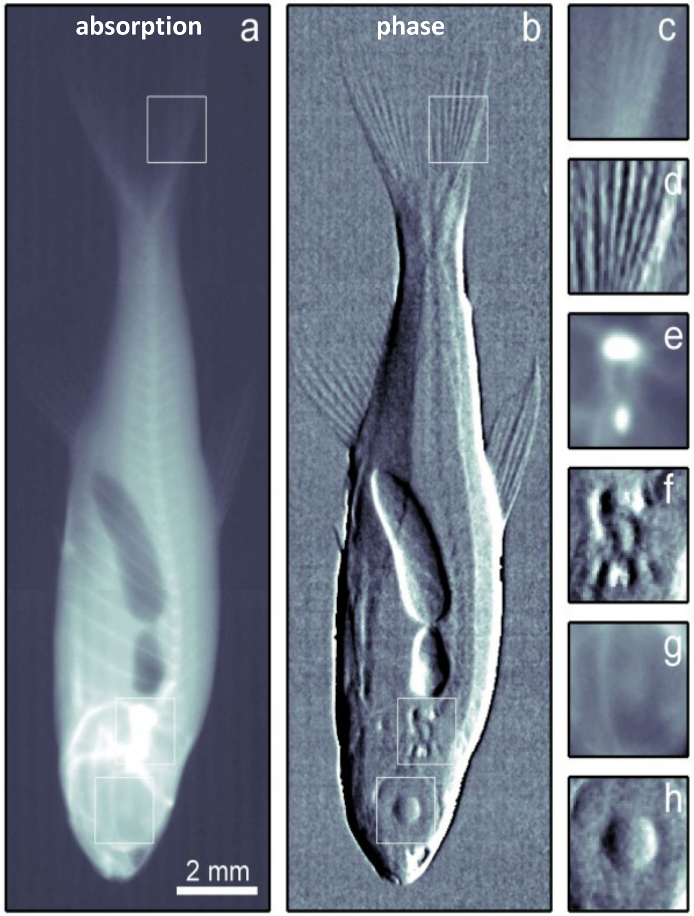


Grating interferometry using three gratings

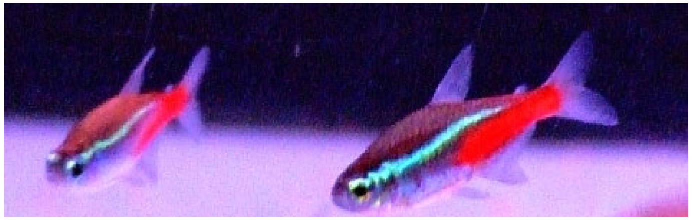
- **Method works with incoherent, broadband radiation**
- Standard x-ray tubes can be used for phase imaging
- Applications in medical imaging, non-destructive testing, homeland security,…
- Several collaborations with industry ongoing

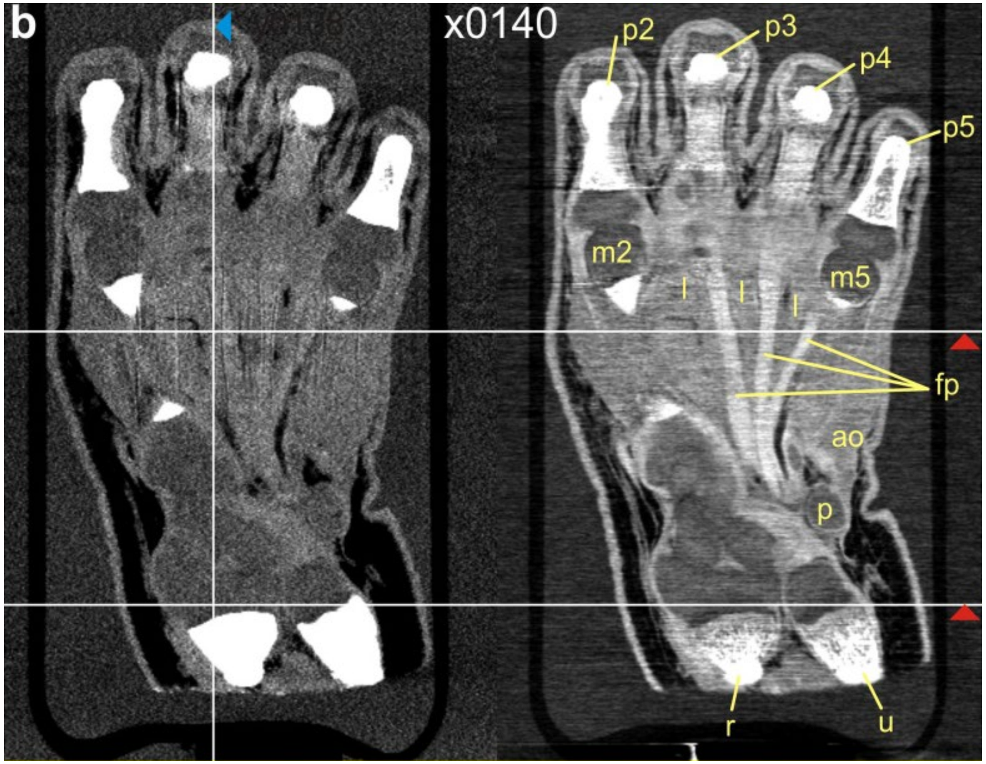
F. Pfeiffer, T. Weitkamp, O. Bunk, C. David, *Nature Physics* **2** (2006) 258; T. Donath et al. *Investigative Radiology* **45** (2010) 445

X-ray phase contrast imaging at x-ray tubes



Radiography of a small fish (E=17.5 keV) 2006



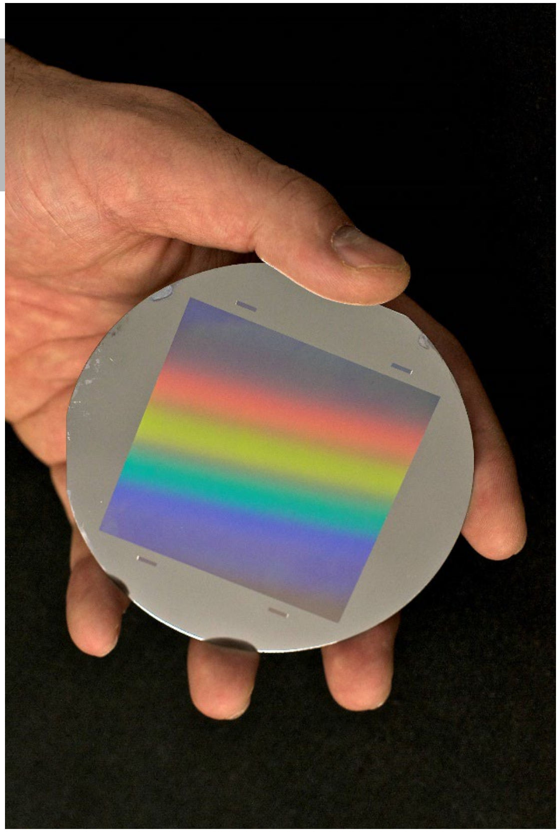


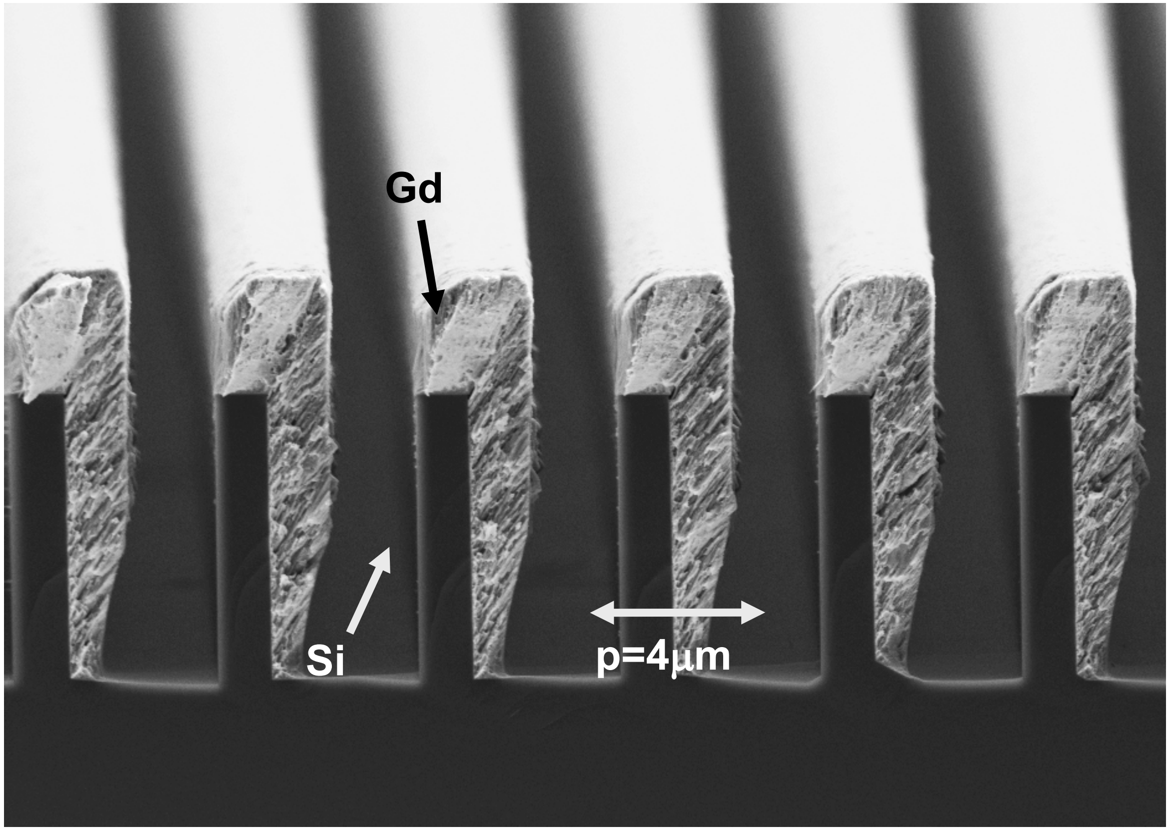
Slice through tomographic data set of a hand (E=28 keV) 2010

- **Method works with incoherent, broadband radiation**
- Standard x-ray tubes can be used for phase imaging
- Applications in medical imaging, non-destructive testing, homeland security,…
- Several collaborations with industry ongoing

F. Pfeiffer, T. Weitkamp, O. Bunk, C. David, *Nature Physics* **2** (2006) 258; T. Donath et al. *Investigative Radiology* **45** (2010) 445

Grating interferometry with neutrons

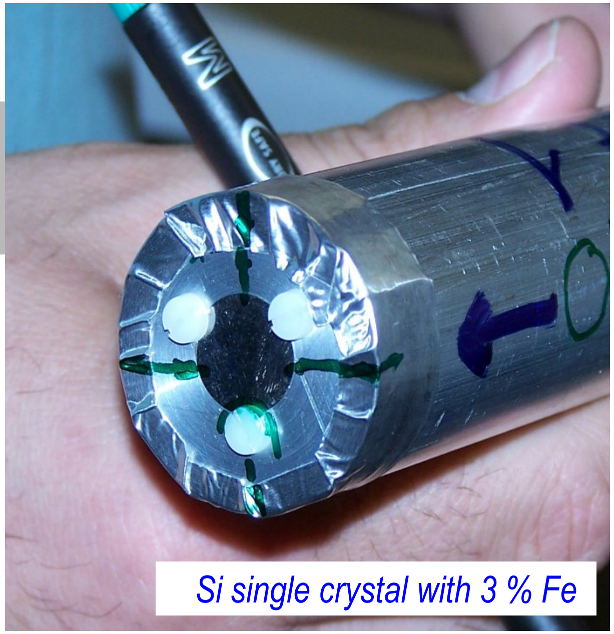


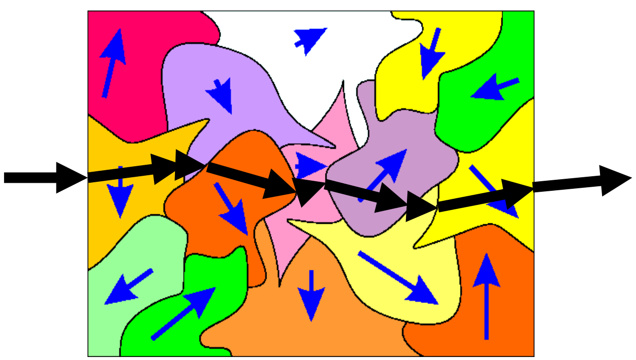


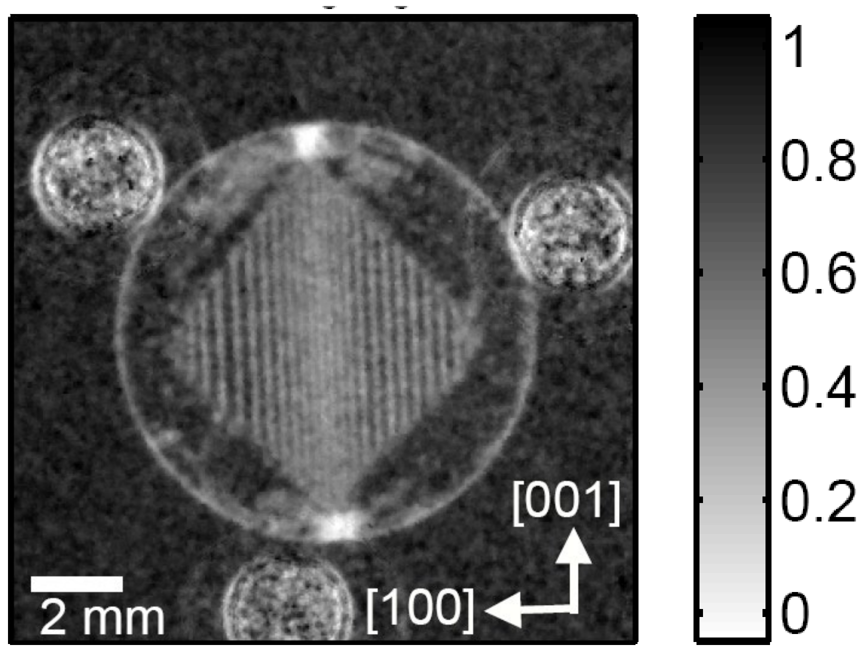
C. Grünzweig, F. Pfeiffer, O. Bunk, T. Donath, G. Kühne, G. Frei, and C. David, *Design, fabrication, and characterization of diffraction gratings for neutron phase contrast imaging,* Review of Scientific Instruments **79** (2008) p. 053703–6

Grating interferometry with neutrons

collaboration with F. Pfeiffer, C. Grünzweig, E. Lehmann (PSI) & R. Schäfer (*TU Dresden)*



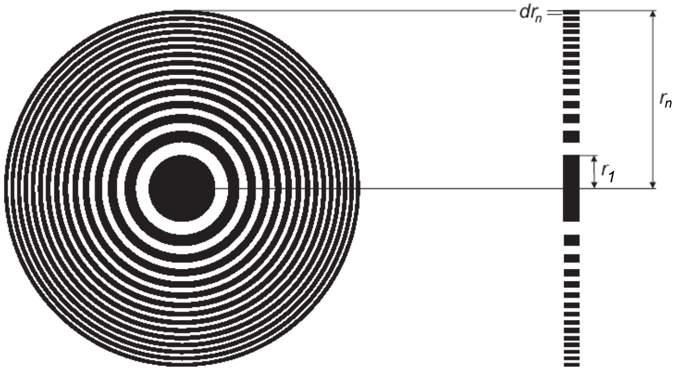


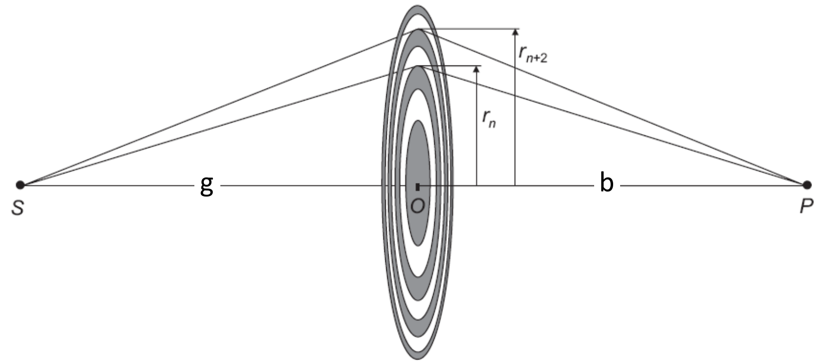


Neutron dark field contrast

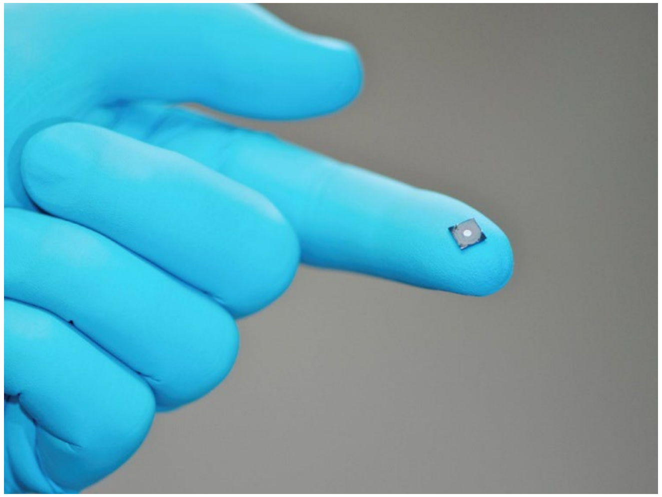
*C. Grünzweig, C. David, F. Pfeiffer et al. Physical Review Letters **101** (2008) p. 025504*
*M. Strobl, C. David, F. Pfeiffer, et al. Physical Review Letters **101**, (2008) p. 123902*
*C. Grünzweig, C. David, F. Pfeiffer et al., Applied Physics Letters **93** (2008) p. 112504*

Diffractive X-ray lenses - Fresnel zone plates

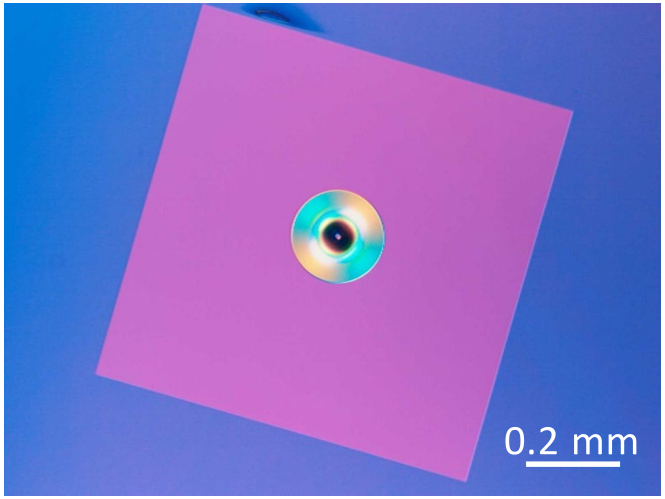




$$r_n{}^2 = \frac{gb(g+b)n\lambda + \frac{1}{4}\left[(g+b)^2 + gb\right](n\lambda)^2 + \frac{1}{8}(g+b)(n\lambda)^3 + \frac{1}{64}(n\lambda)^4}{(g+b+n\frac{\lambda}{2})^2} \approx n\lambda f\,, \quad f\text{: focal length}$$

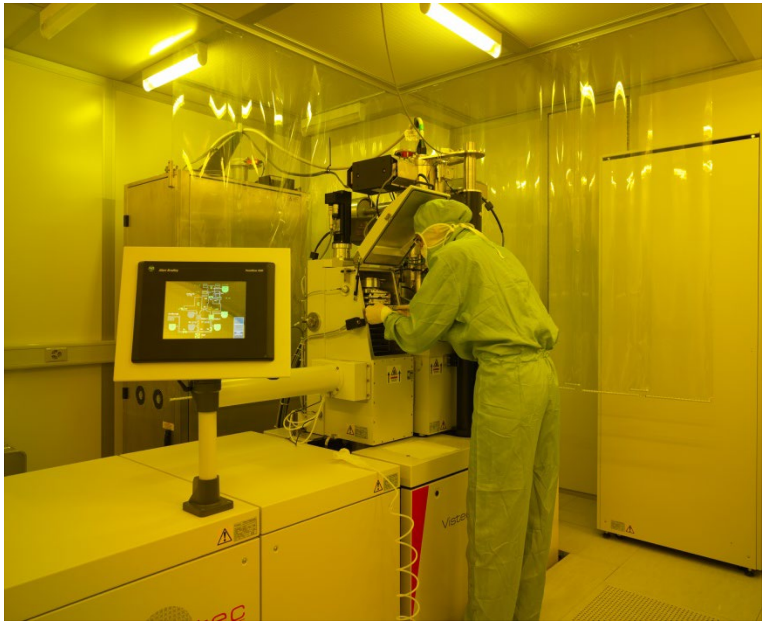


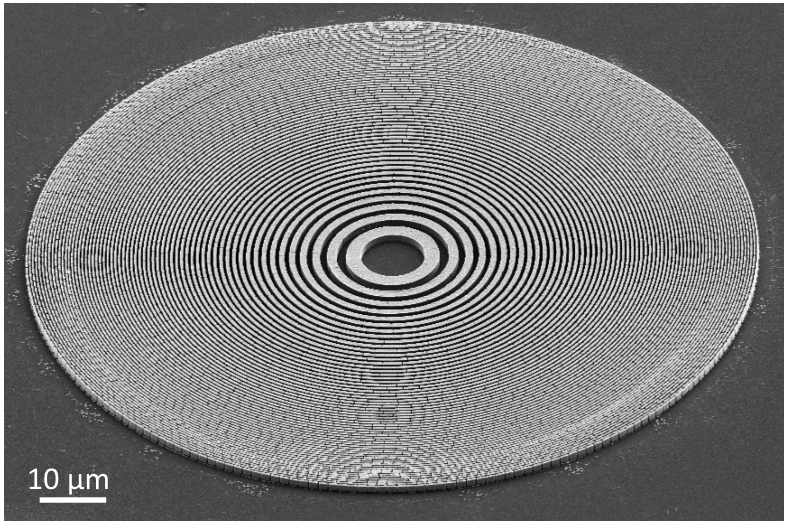
Fresnel zone plate chip

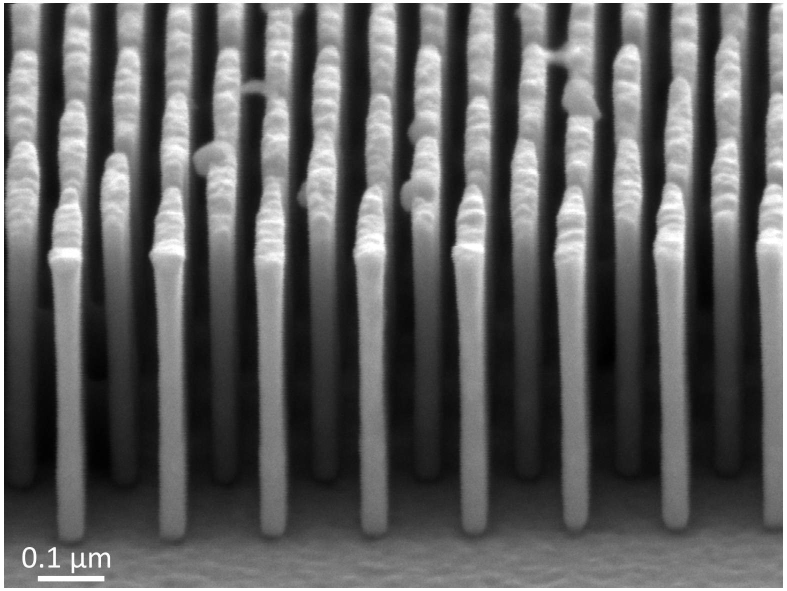


Fresnel zone plate on Si_3N_4 support membrane

Zone plate fabrication







50 nm Au zone plate structures, 500 nm high

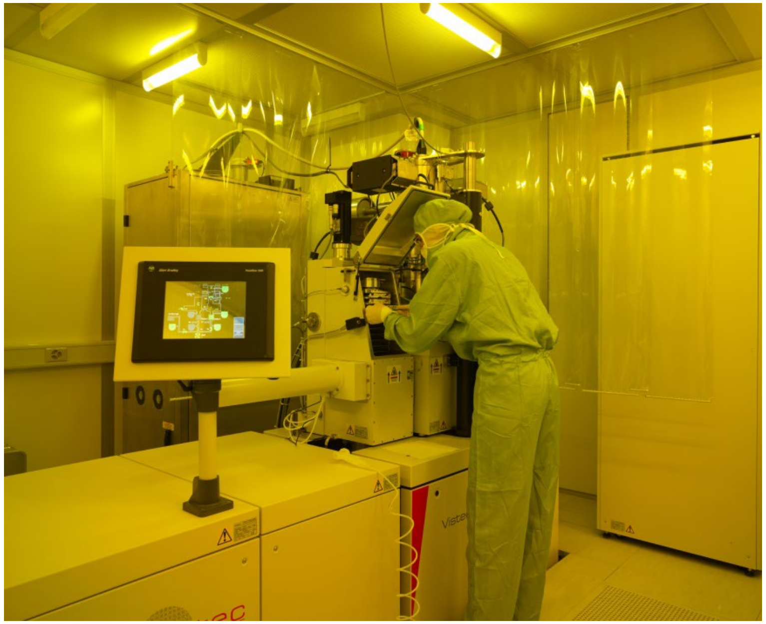
- Resolution is determined by smallest zone width
- Optimum efficiency requires π phase-shift

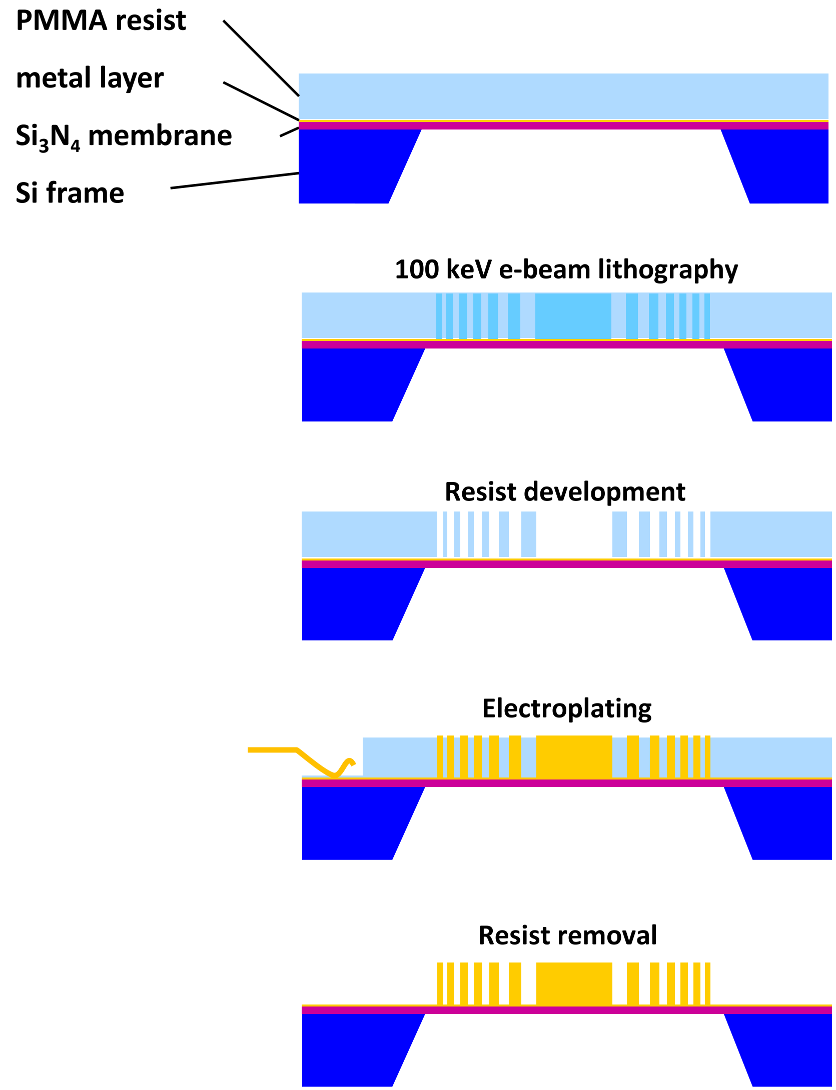
=> Nanostructuring with high aspect ratios

- High voltage e-beam writers are the ideal tools

S. Gorelick, et al., *Nanotechnology* **21** (2010) 295303

Zone plate fabrication





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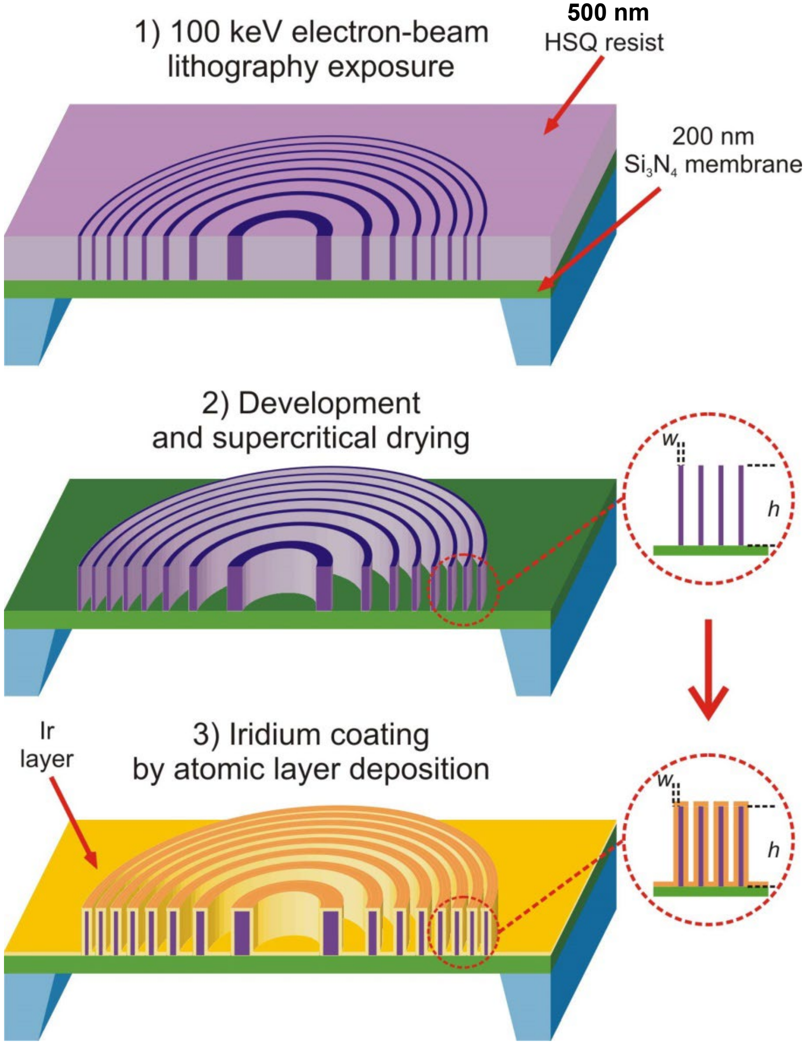
S. Gorelick, et al., *Nanotechnology* **21** (2010) 295303

Line doubled Fresnel zone plates

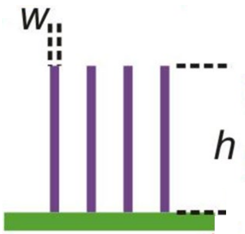
- Resolution of e-beam lithography for dense line patterns is limited by secondary electron range
- Solution: exposure of sparse pattern in low density material
- Coating with high density material results in doubling of zones

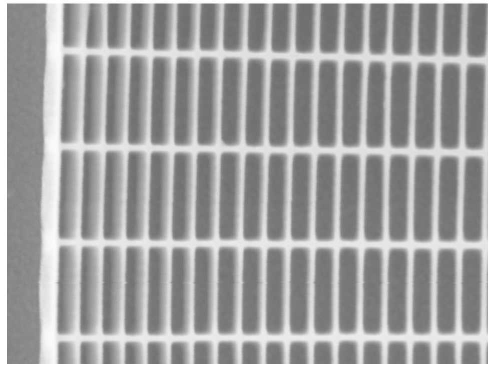
=> 2x higher resolution

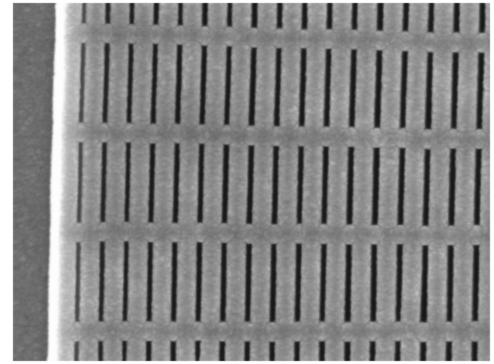


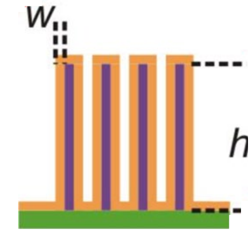
K. Jefimovs et al. Phys. Rev. Lett. **99** (2007) 264801; J. Vila-Comamala et al., Ultramicroscopy **109** (2009) p. 1360
J. Vila-Comamala et al., Nanotechnology **21** (2010) 285305; J. Vila-Comamala et al., Optics Express 19 (2011) 175

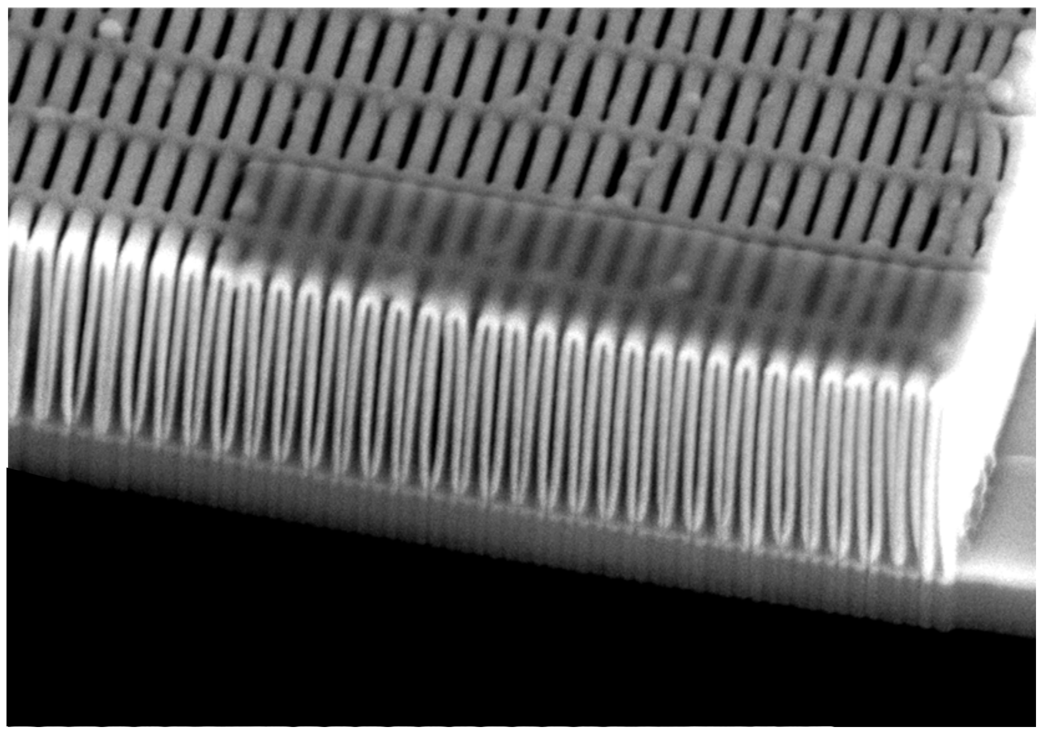
Line doubled Fresnel zone plates







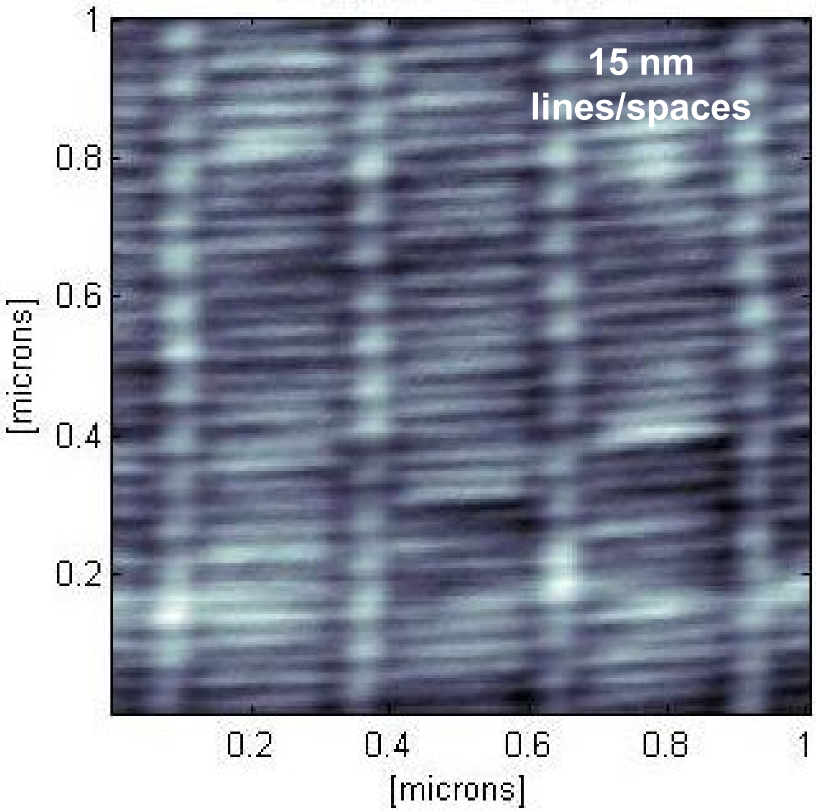




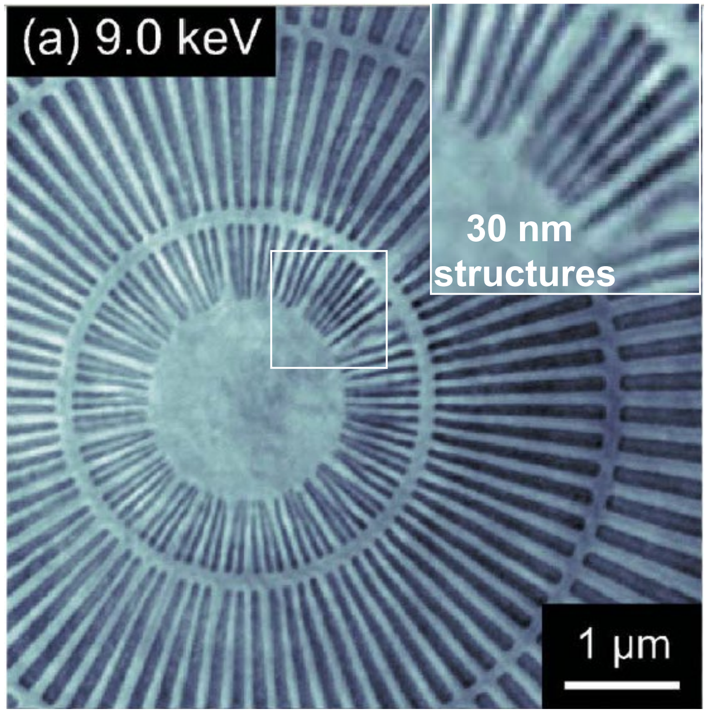
FIB cross-section of 25 nm wide, 550 nm high Ir zone plate

Line doubled Fresnel zone plates

with A. Menzel, A. Diaz (SLS-cSAXS), J. Raabe, B. Watts (SLS-PolLux)



Test structures imaged in scanning mode at SLS-cSAXS. Photon energy: 6.2 keV

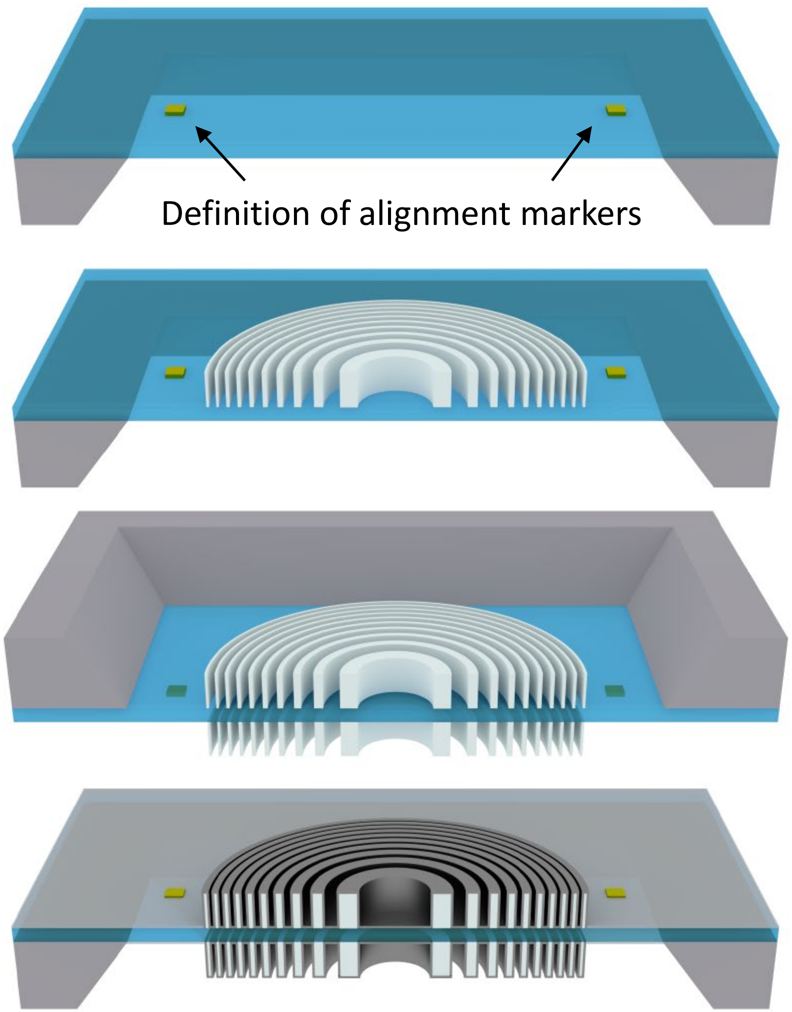


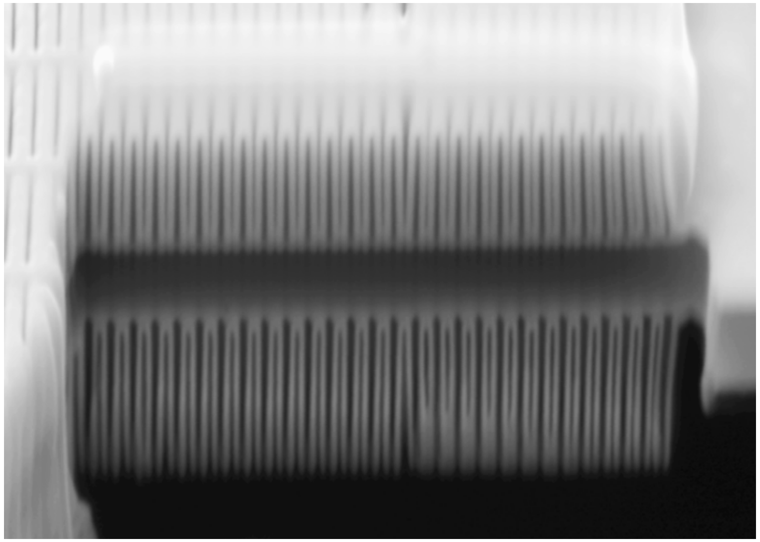
Full-field microscope images taken at APS 32-ID. Spatial resolution: <20 nm. Photon energy: 9 keV

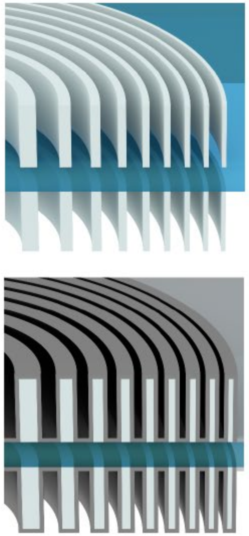
K. Jefimovs et al. *Phys. Rev. Lett.* **99** (2007) 264801; J. Vila-Comamala et al., *Ultramicroscopy* **109** (2009) p. 1360
J. Vila-Comamala et al., *Optics Express* **19** (2011) 175; J. Vila-Comamala et al. *J. Synchrotron Rad.* **19** (2012) 705

Double-sided, line-doubled Fresnel zone plates

with A. Menzel, A. Diaz (SLS-cSAXS), A. Somogyi, C.M. Kewish (Synchrotron Soleil)





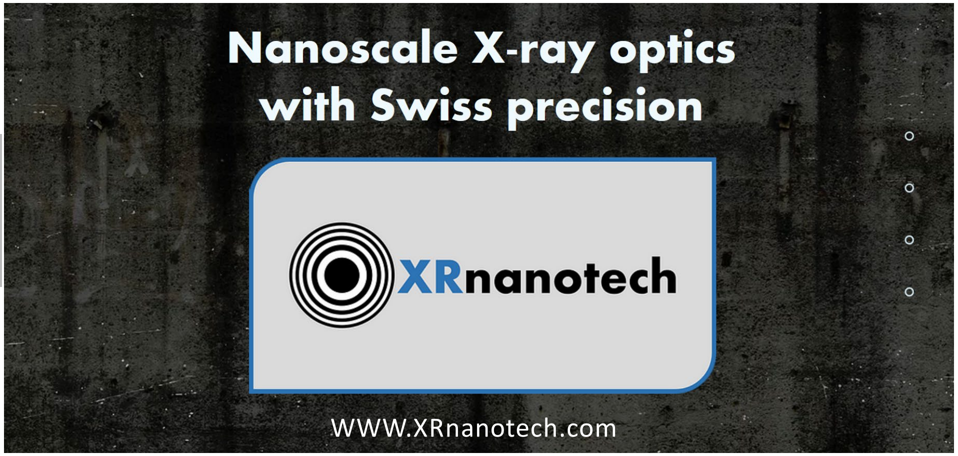


- 30 nm wide zones, 1200 nm height, aspect ratio ≈40
- 10% efficiency at 9 keV (~2.5x improvement)

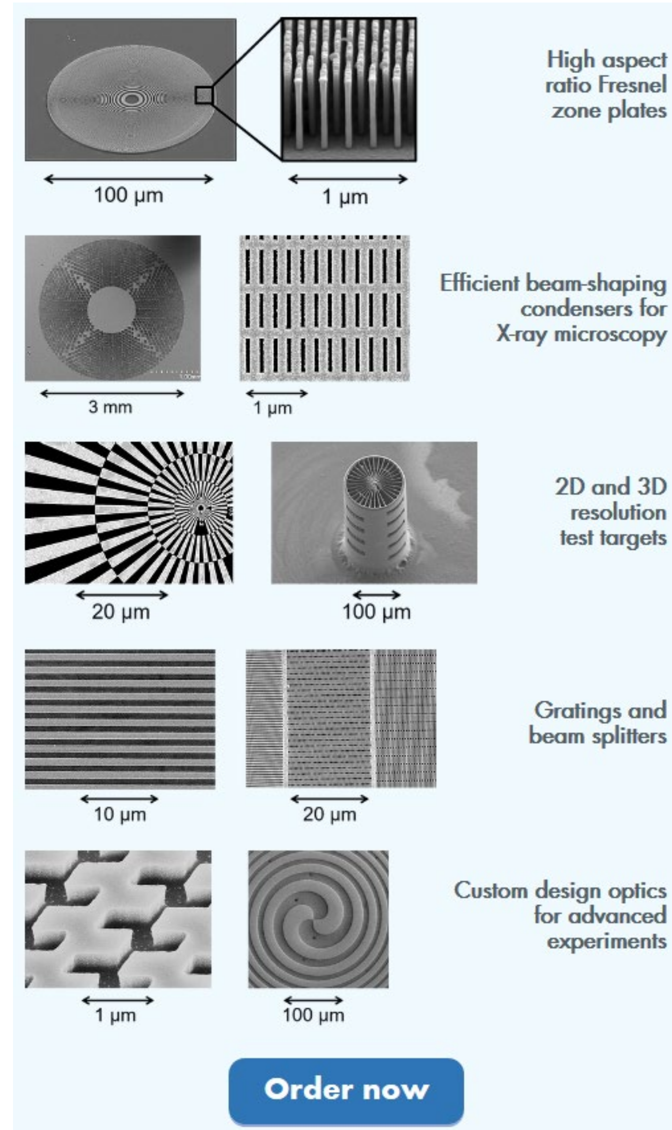
I. Mohacsi, et al., *Optics Express* **23** (2015) 776

PSI's zone plates are popular…

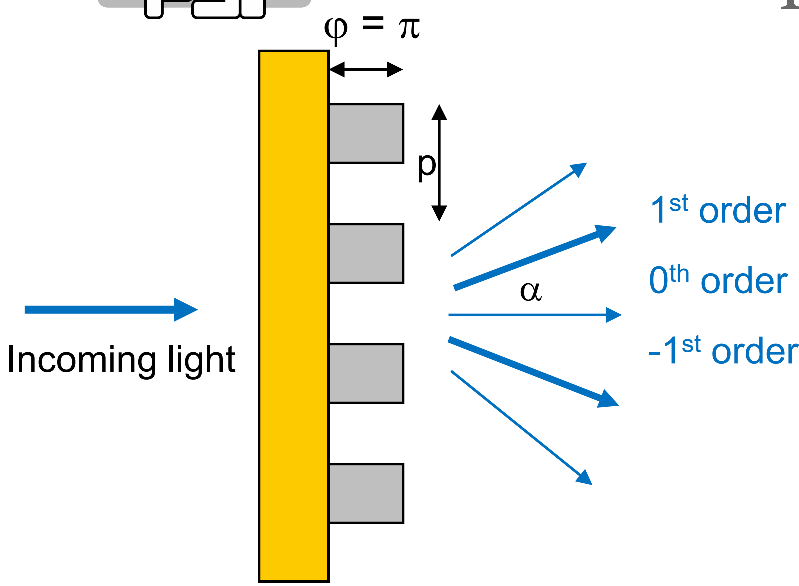
XRnanotech: a PSI startup to commercialize X-ray optics

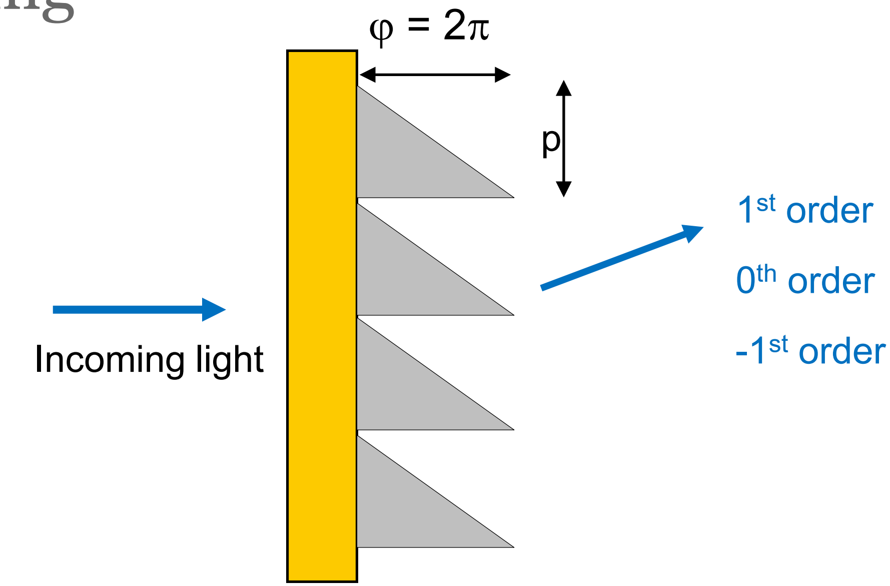


Autumn 2019: Florian Döring wins the PSI Founder Fellowship

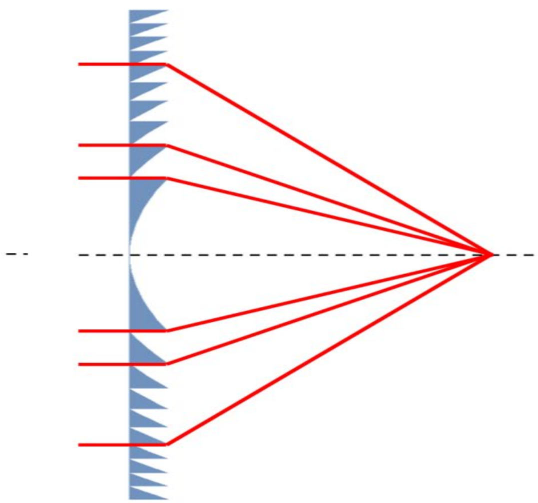


Blazing

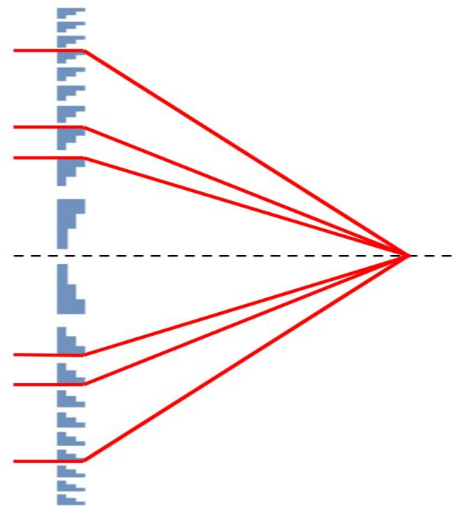




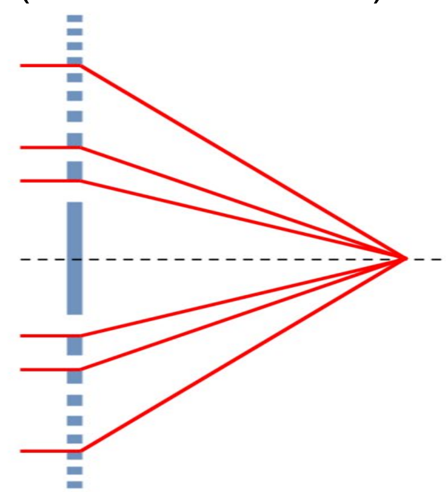
Blazed lens
(up to 100%)



Staircase zone plate
(up to 81% for 4 levels)

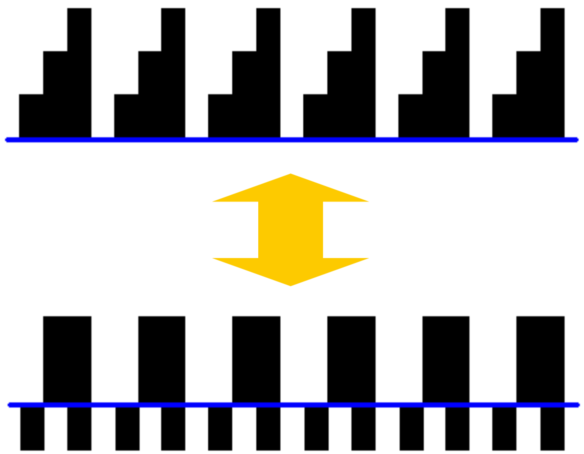


Binary zone plate
(limited to < 40.5%)

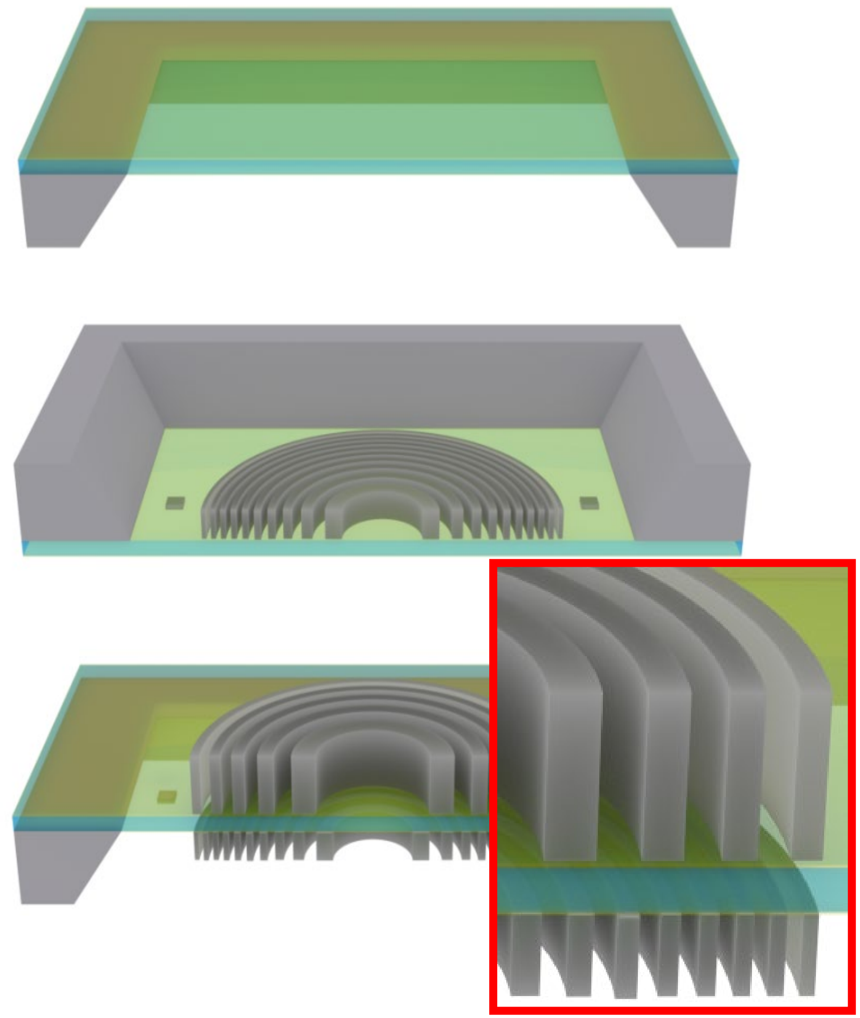


Blazed, double-sided Fresnel zone plates

I. Mohacsi, with A. Menzel, A. Diaz (SLS-cSAXS), A. Somogyi, C.M. Kewish (Synchrotron Soleil)



- Stacking of two zone plates can be used to achieve a staircase (blazed) profile
- Negative diffraction orders are suppressed, positive orders enhanced



I. Mohacsi, et al., *Optics Letters* ***41*** *(2016) p. 281-284, DOI: 10.1364/OL.41.000281*

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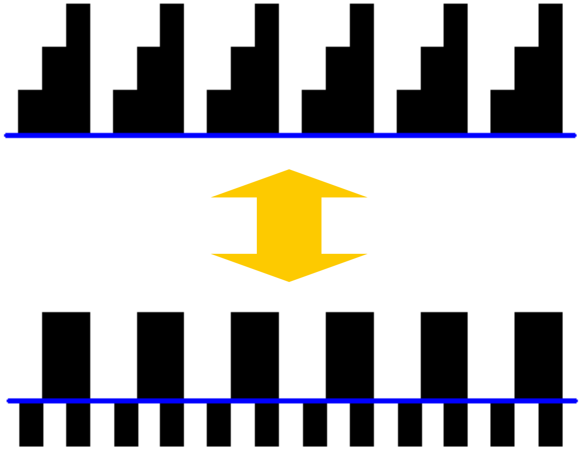
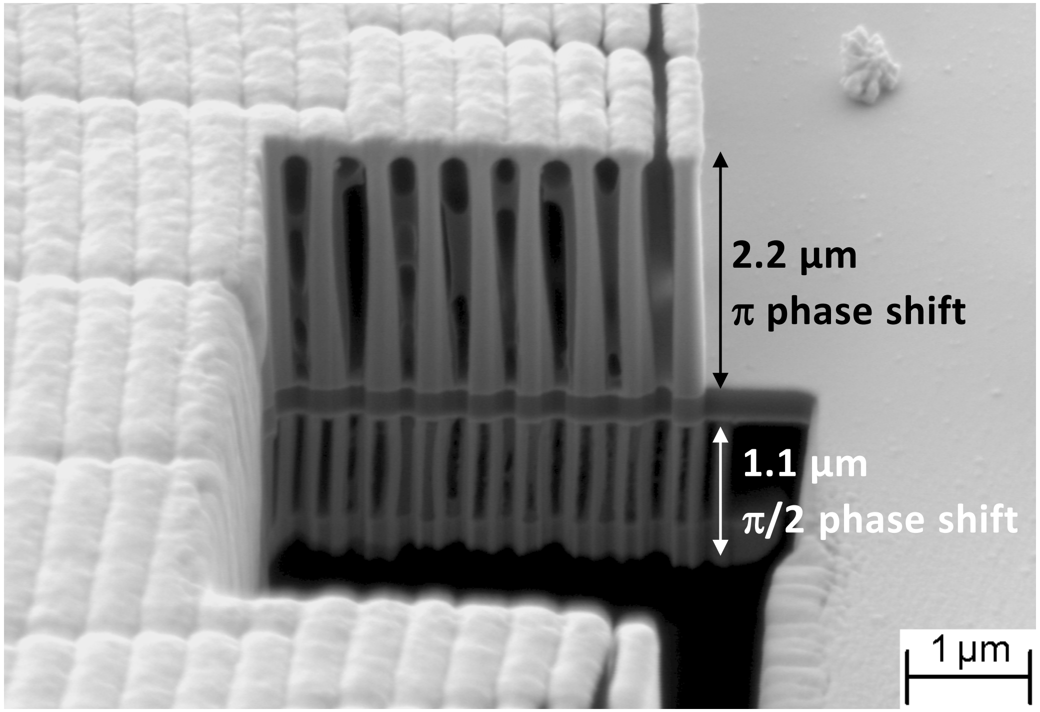


- Stacking of two zone plates can be used to achieve a staircase (blazed) profile
- Negative diffraction orders are suppressed, positive orders enhanced
- Measured **up to 55%** efficiency for Nickel zone plates at E = 6.2 keV (λ = 2 Å) with 200 nm zone width



FIB cross-section of a blazed Ni zone plate for 6.2 keV X-rays

I. Mohacsi, et al., *Optics Letters* ***41*** *(2016) p. 281-284, DOI: 10.1364/OL.41.000281*

PAUL SCHERRER INSTITUT

Can we use diffractive lenses for high-resolution neutron microscopy?

- Refractive indices and wavelengths of multi-keV X-rays and cold neutrons are quite similar
- Fresnel zone plate made of binary, 8 µm high nickel structures could have ~40% efficiency
- Blazing schemes could be applied for even higher efficiencies

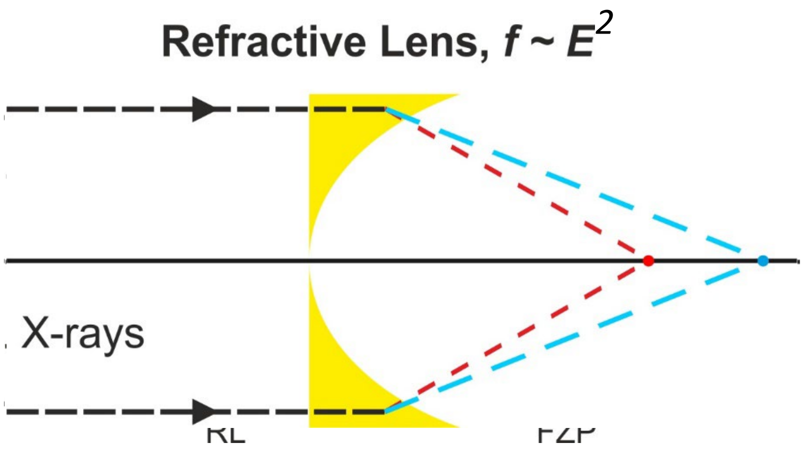
Problem: chromatic aberrations of FZPs (f ~ E)
require bandwidth $\delta\lambda/\lambda$ of typ. <1%.
Low brilliance of neutron sources is a problem!

- Suggestion to combine refractive and diffractive optics was discussed intensely with H.F. Poulsen, DTU Denmark, but never materialized into joint experiments…

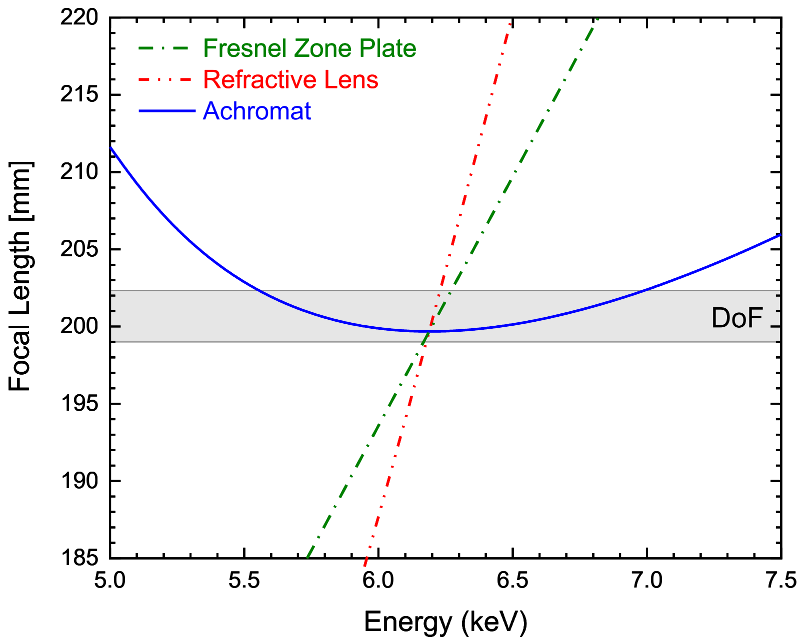
S.O. Poulsen, H.F. Poulsen, P.M. Bentley, *Refractive and diffractive neutron optics with reduced chromatic aberration*, NIMA **767** (2014) 415

An achromatic X-ray lens

with A. Kubec, J. Vila-Comamala, M.-C. Zdora, U. T. Sanli, P. Qi, and A. Diaz



Achromat condition: $f_{RL} = -2 f_{FZP}$



A. Kubec, M.-C. Zdora, U. Sanli, A. Diaz, J. Vila-Comamala, C. David, *Nature Communications* ***13*** (2022) p. 1305

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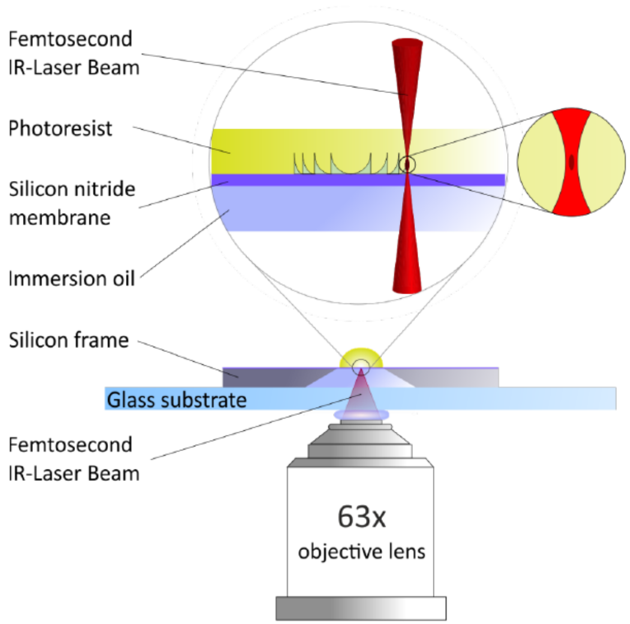
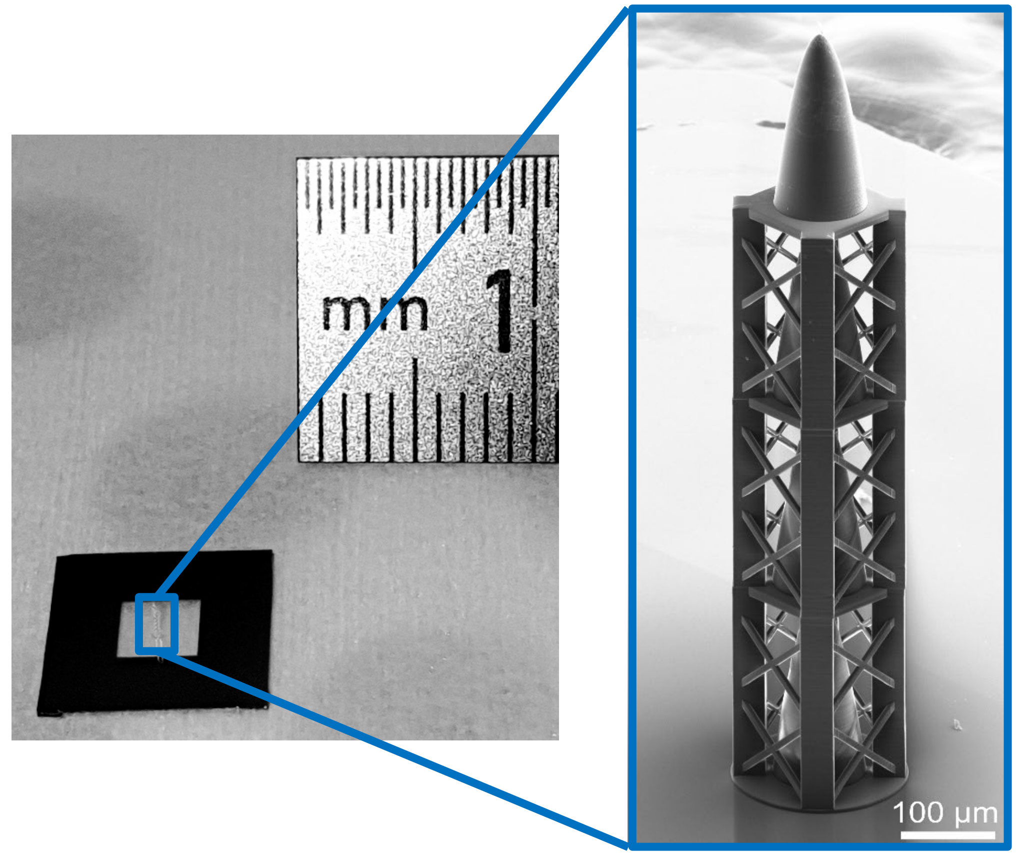


Two-photon 3D lithography tool "Nanoscribe" produces polymer structures sub-micron resolution



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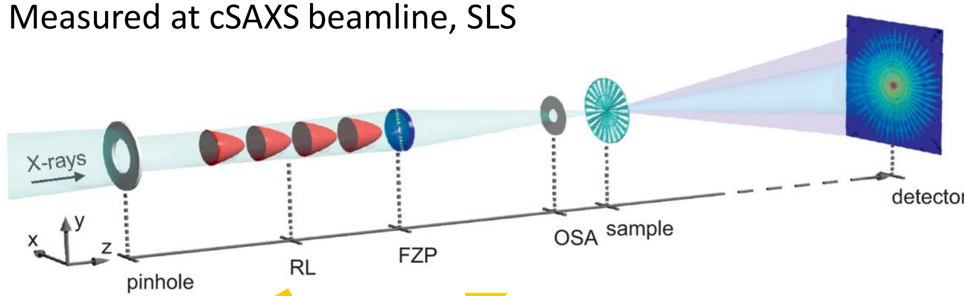
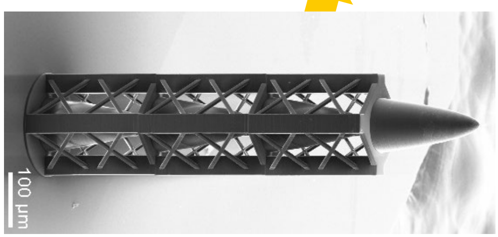
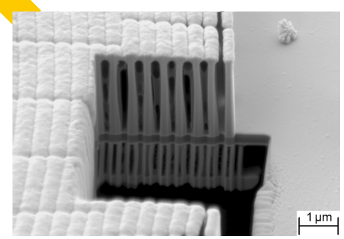
Measured at cSAXS beamline, SLS



Smaller spot?
Better quality?

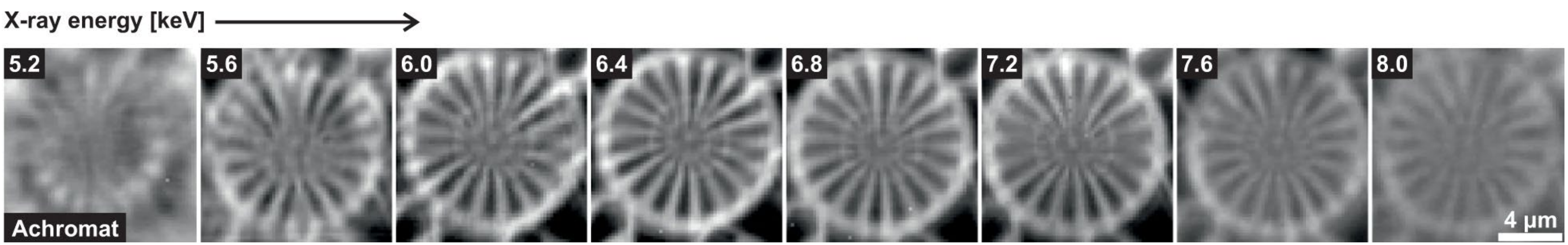


3D printed refractive lens



Blazed nickel Fresnel zone plate

- **500 nm focal spot**
- **Achromatic range: ~ 5.8 to 7.2 keV**



Scanning X-ray microscopy as function of X-ray energy without refocusing

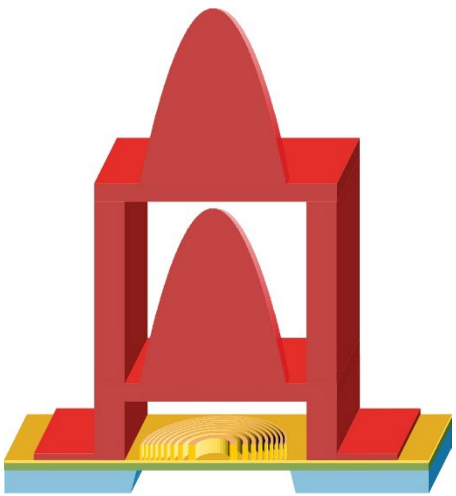
A. Kubec, M.-C. Zdora, U. Sanli, A. Diaz, J. Vila-Comamala, C. David, *Nature Communications* ***13*** (2022) p. 1305

Monolithic X-ray achromat

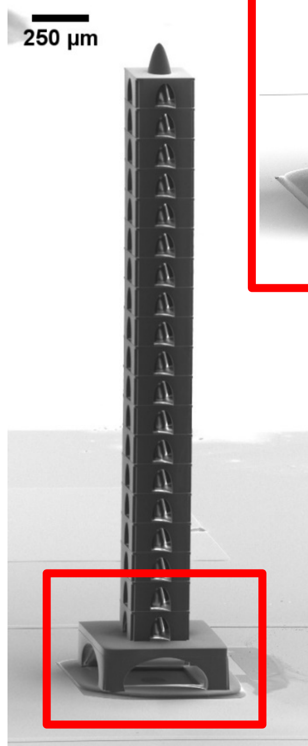
with J. Vila-Comamala, M.-C. Zdora, U. T. Sanli, P. Qi, and A. Diaz

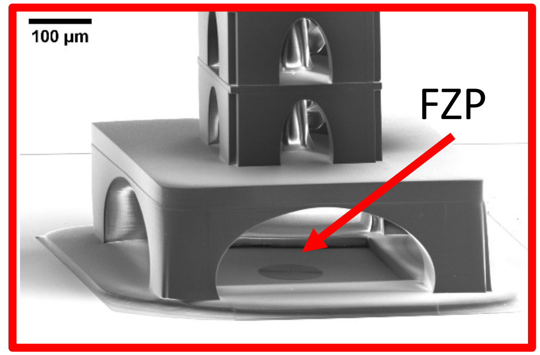
Smaller focal spot ⇨ taller refractive lens

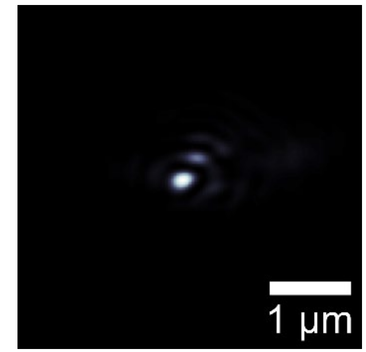
Reduce alignment complexity ⇨ FZP and RL on the same membrane chip



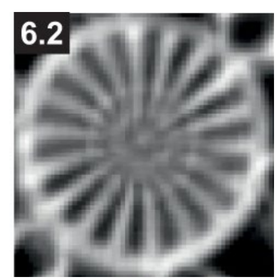
Fabrication steps:



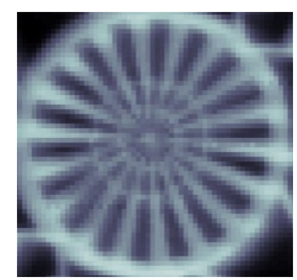




200 nm focal spot @ 7.1 keV photon energy



Two separate elements



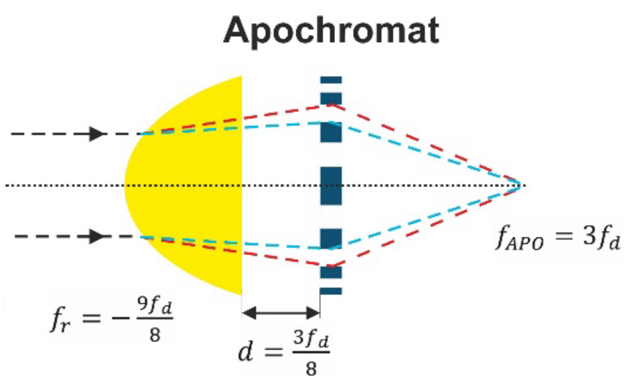
Monolithic achromat

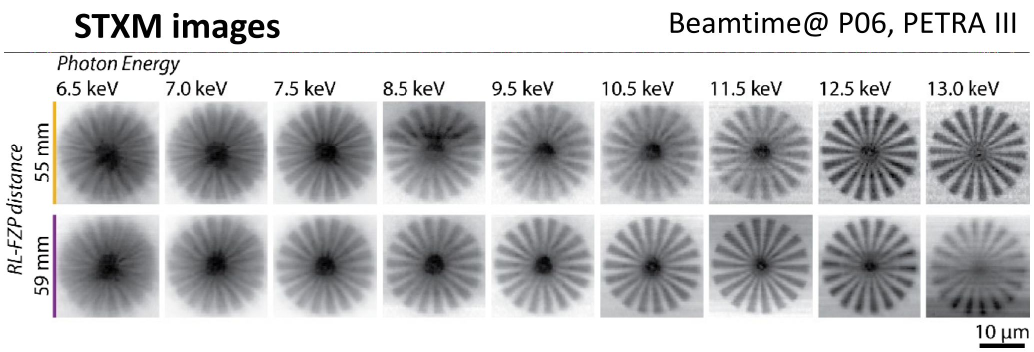
Scanning X-ray microscopy images

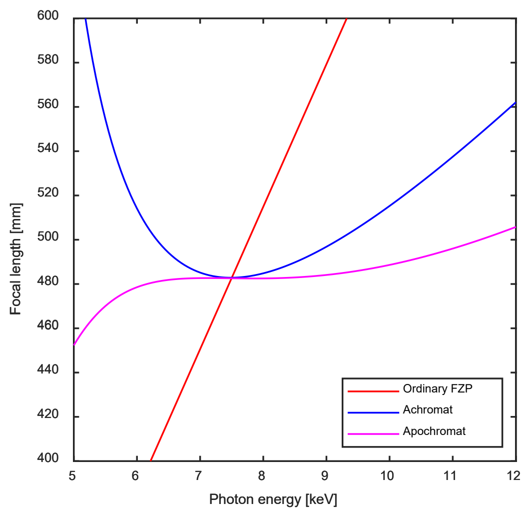
Apochromatic X-ray focusing

with J. Vila-Comamala, M.-C. Zdora, U. T. Sanli, P. Qi, A. Diaz & UniBasel collaborators

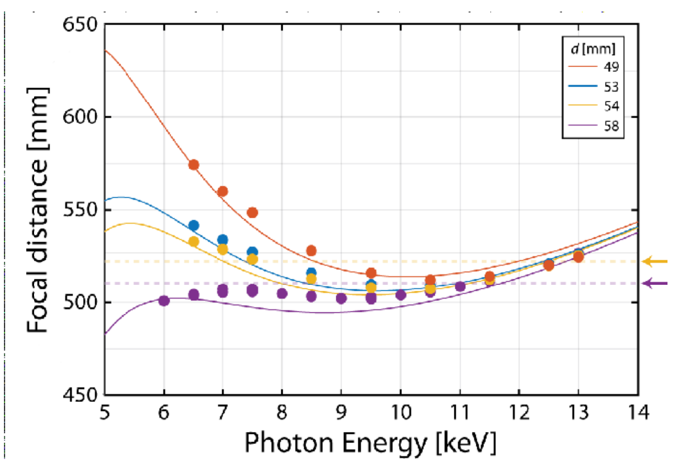
Increase the achromatic range by separating the two components





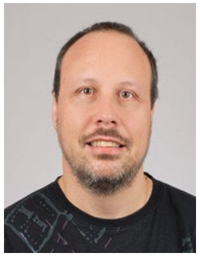


- 750 nm focal spot
- Apochromatic range: ~ 8.0 to 11.0 keV



Applications of achromats

with J. Vila-Comamala, M.R. Dhanalakshmi Veeraraj & UniBasel collaborators



Joan Vila-Comamala

Achromatic lenses allow **for efficient use of broadband, low billiance sources** that will not deliver sufficient signal when used with a monochromator!

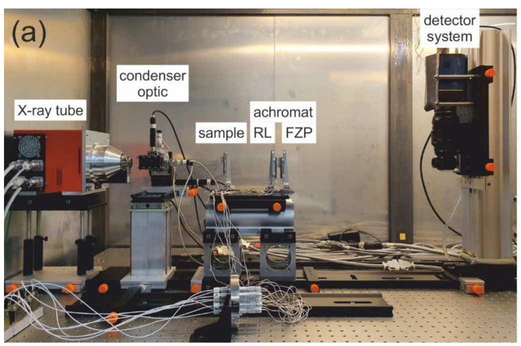
M.R. Dhanalakshmi Veeraraj

X-ray microscopy on a lab source

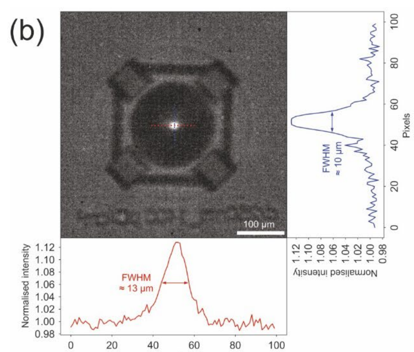
- **X-ray microscope** on an **X-ray tube**
- First result: achromatic 1:1 imaging of X-ray microfocus source

High-resolution neutron microscopy

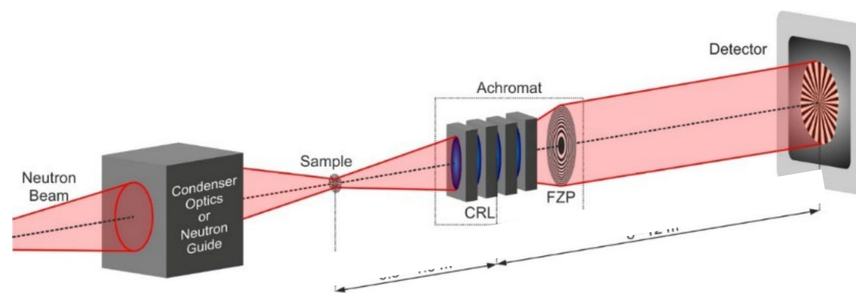
- **Neutron microscope** based on a **neutron achromat**
- Colaboration with Markus Strobl, SINQ



X-ray microscope setup

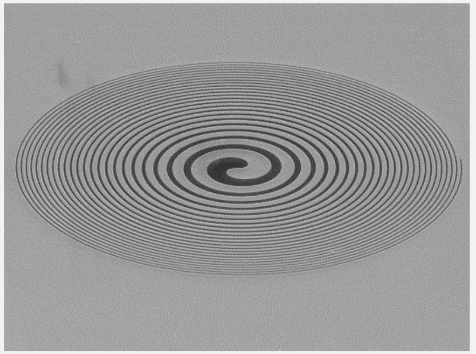


1:1 X-ray image of a 10 µm tube source with an achromat

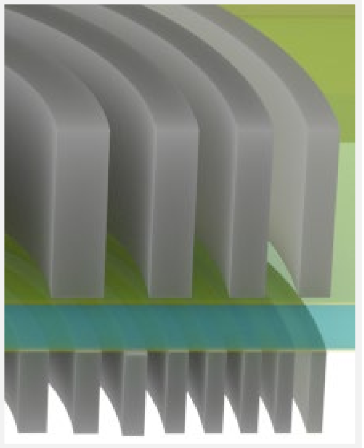


Planned set-up of a neutron microscope at SINQ-BOA

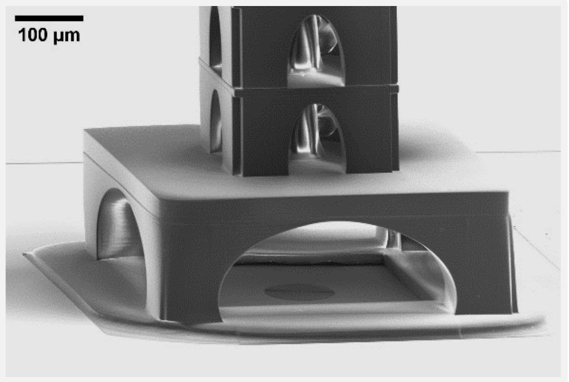
Conclusions

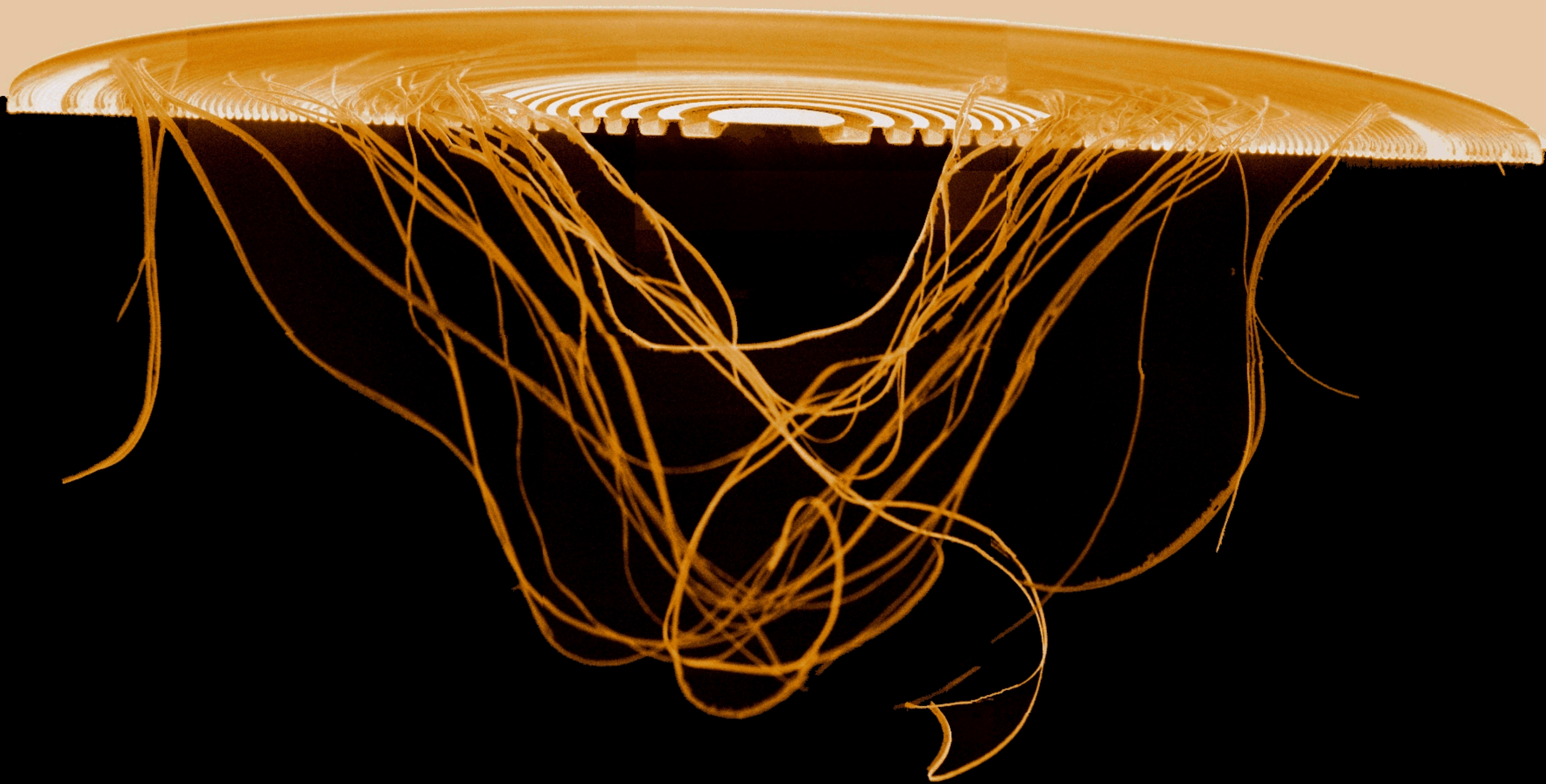


- *Diffractive x-ray optics have characteristic properties:*
    - Advanced nanolithography techniques can provide diffractive lenses with very high spatial resolution
    - Blazed optics can provide good diffraction efficiency
    - The devices are compact and easy to use
    - Complex optical functionalities can be implemented



- *Opportunities for neutron imaging:*
    - Low brilliance of neutron beams requires achromatic concepts
    - Grating interferometry was successfully transferred from X-rays to neutrons
    - Recent developments in achromatic lenses should enable high resolution neutron microscopy





Thank you for your attention!