X-ray Reflecto-Interferometer Based on Refractive Optics

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Motivation

X-ray reflectometry (XRR) is a classical technique for thin-films and multilayers internal structure study. The appearance of new optics for X-ray laboratory and synchrotron sources increased opportunities for the development of X-ray investigation techniques, including reflectometry. In this work, we demonstrate a new X-ray reflectometry technique based on compound refractive lenses (CRL) for thin-film structures study.

The advantages of parabolic refractive lenses

- Large energy range 2 - 200 keV
- Beam conditioning optics: Condensers, Collimators, Monochromators, Harmonics rejecter
- Beam/emittance diagnostics
- Compatibility with crystal optics
- Coherence related techniques: micro (nano)-beam: diffraction, scattering & spectroscopy; full-field imaging and diffraction microscopy, dark-field microscopy, in-line interferometry

Calculations

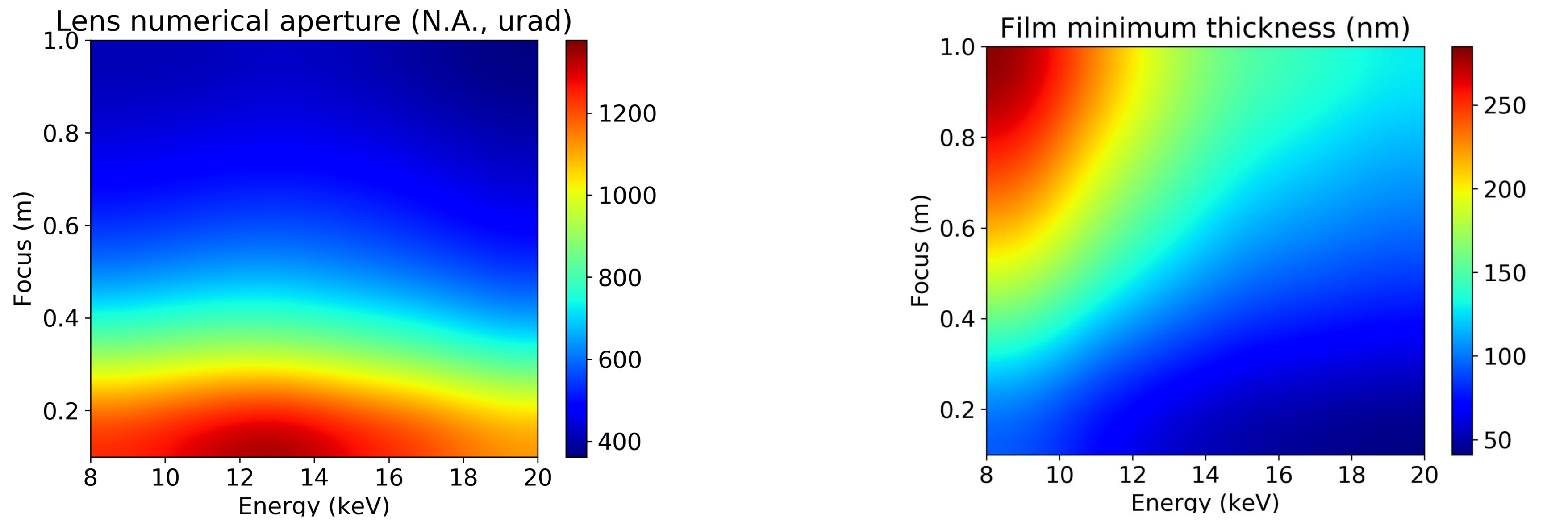
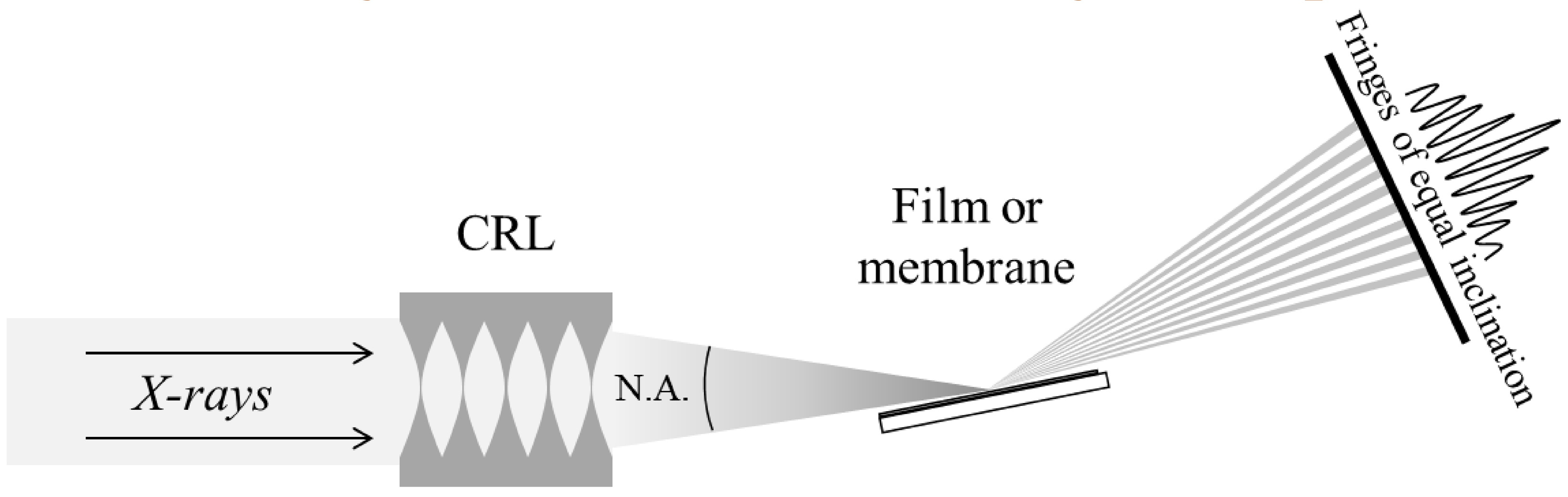


Figure 1. The results of the numerical calculation of the dependence of the numerical aperture and the minimum film thickness on the CRL focus and energy.

$$N.A. = \left(\frac{2\delta}{F\mu}\right)^{1/2} \qquad d_{min} = \frac{\lambda}{4}\cdot\left(\frac{2\delta}{F\mu}\right)^{-1/2}$$

N.A. - numerical aperture, F - lens focus distance, λ - wavelength, δ - decrement of refraction index, μ - absorption coefficient.

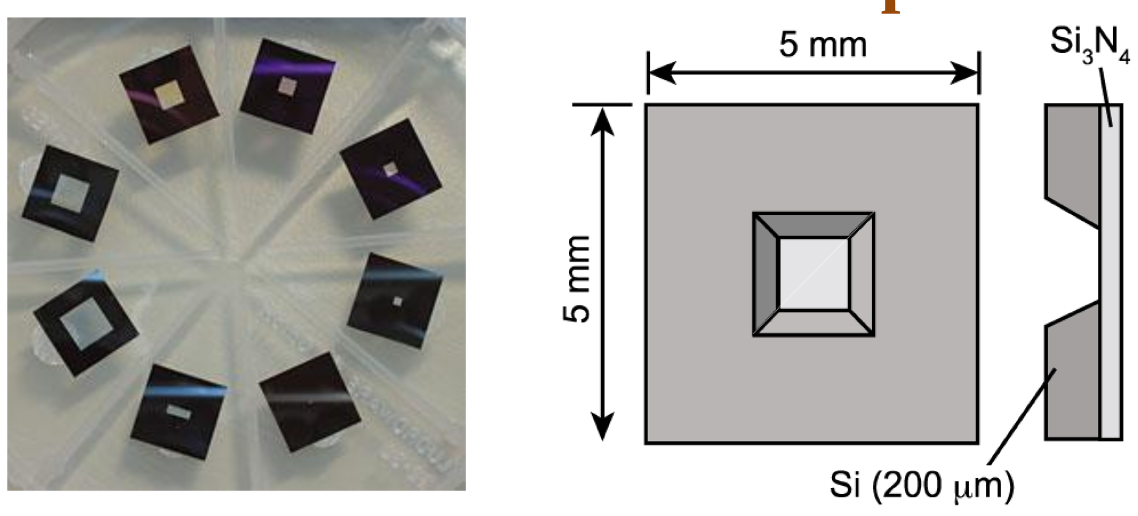
X-Ray Reflecto-Interferometry Concept



XRI advantages:

- high time resolution (one shot experiment & in-situ)
- high spatial resolution
- simple optics for large range of samples thicknesses
- lower requirements to quality and geometry of the sample surface

Samples



Film thickness:

- 200 nm
- 500 nm
- 1000 nm

*http://www.silson.com/

Experimental Results

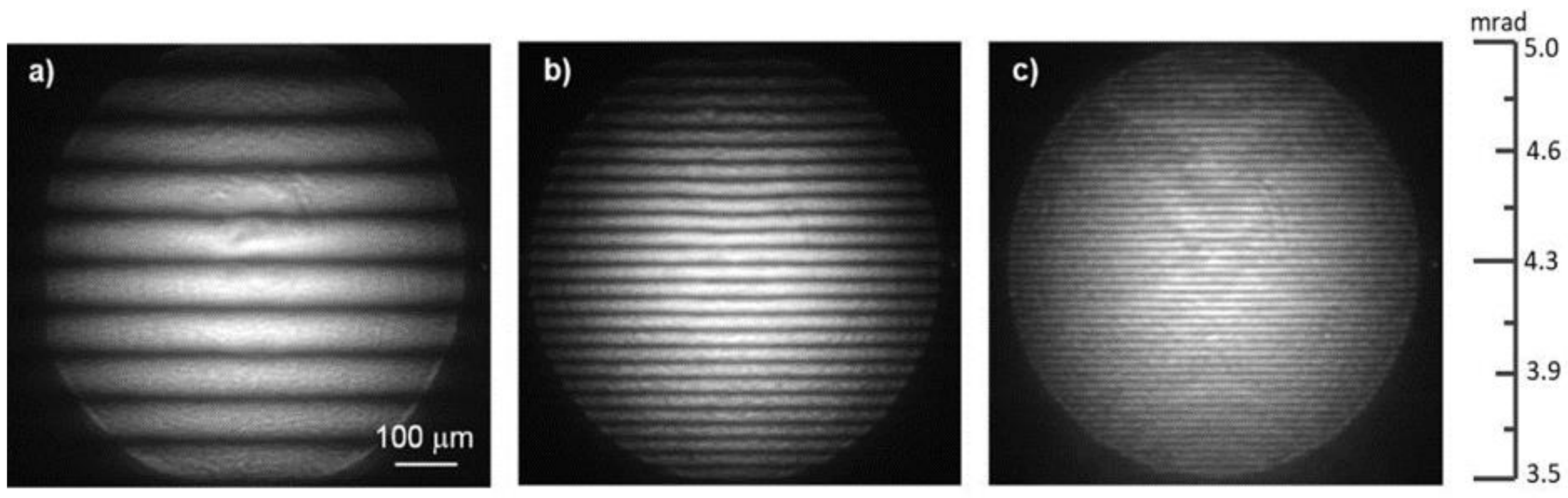


Figure 1. Reflecto-interferograms from Si_3N_4 membranes with thickness of 200 nm (a), 500 nm (b) and 1000 nm (c). The scale 100 mm corresponds to 224 mrad.

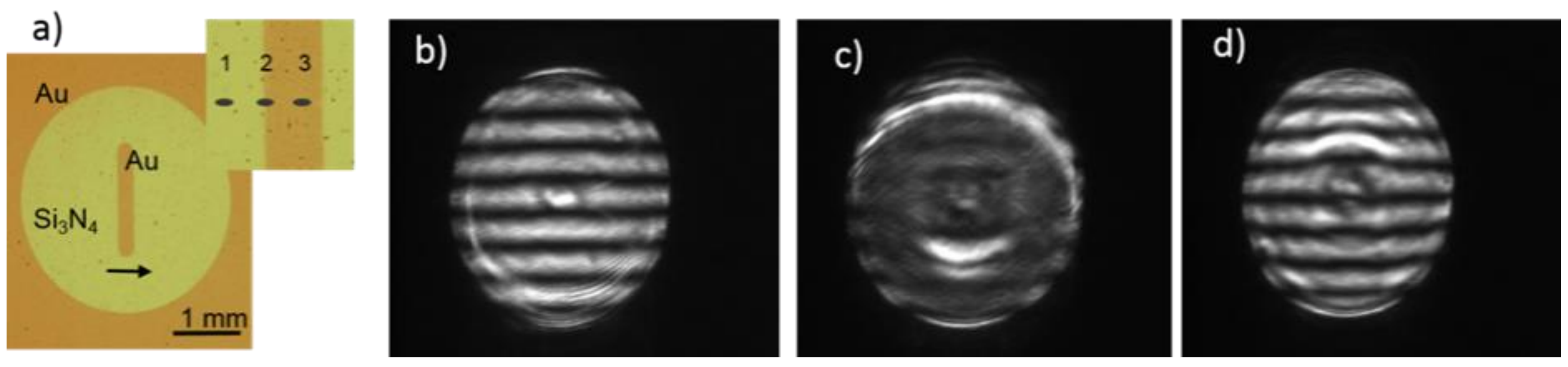


Figure 3. Image in the optical microscope of Au strip on the membrane. Insert shows locations where reflecto-interferograms were recorded crossing the Au edge. (b) Interferogram detected at the position 1 on the membrane/air interface. (c) Interferogram recorded at the position 2 at the boundary membrane/air and membrane/gold/air. (d) Interferogram registered at the position 3 on the membrane/gold/air interface at the centre of the gold strip. The intensity of interferograms is represented in logarithmic scale.

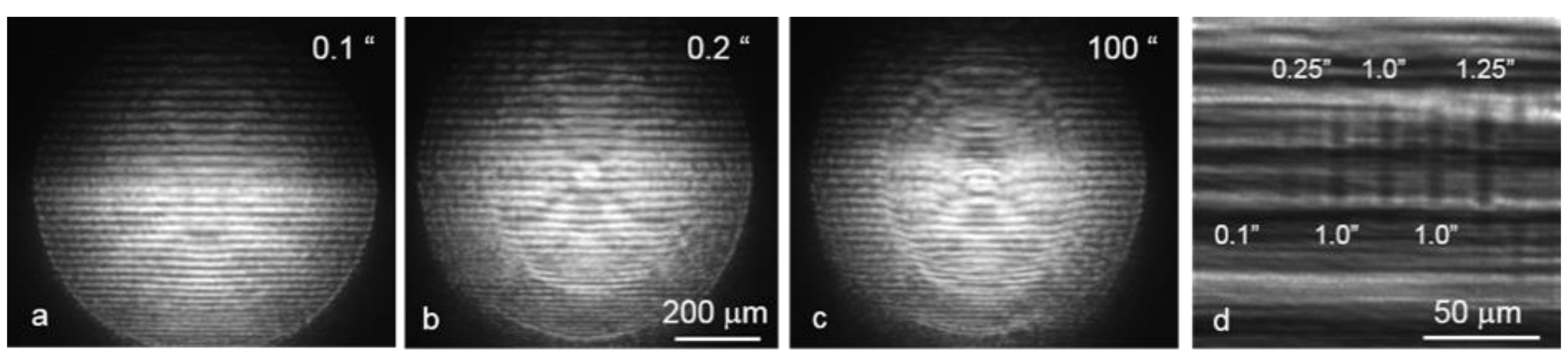


Figure 5. (a)–(c) Experimental reflecto-interferograms recorded at different exposures time. The intensity of interferograms is represented in logarithmic scale. (d) The X-ray topogram of the damaged structure.

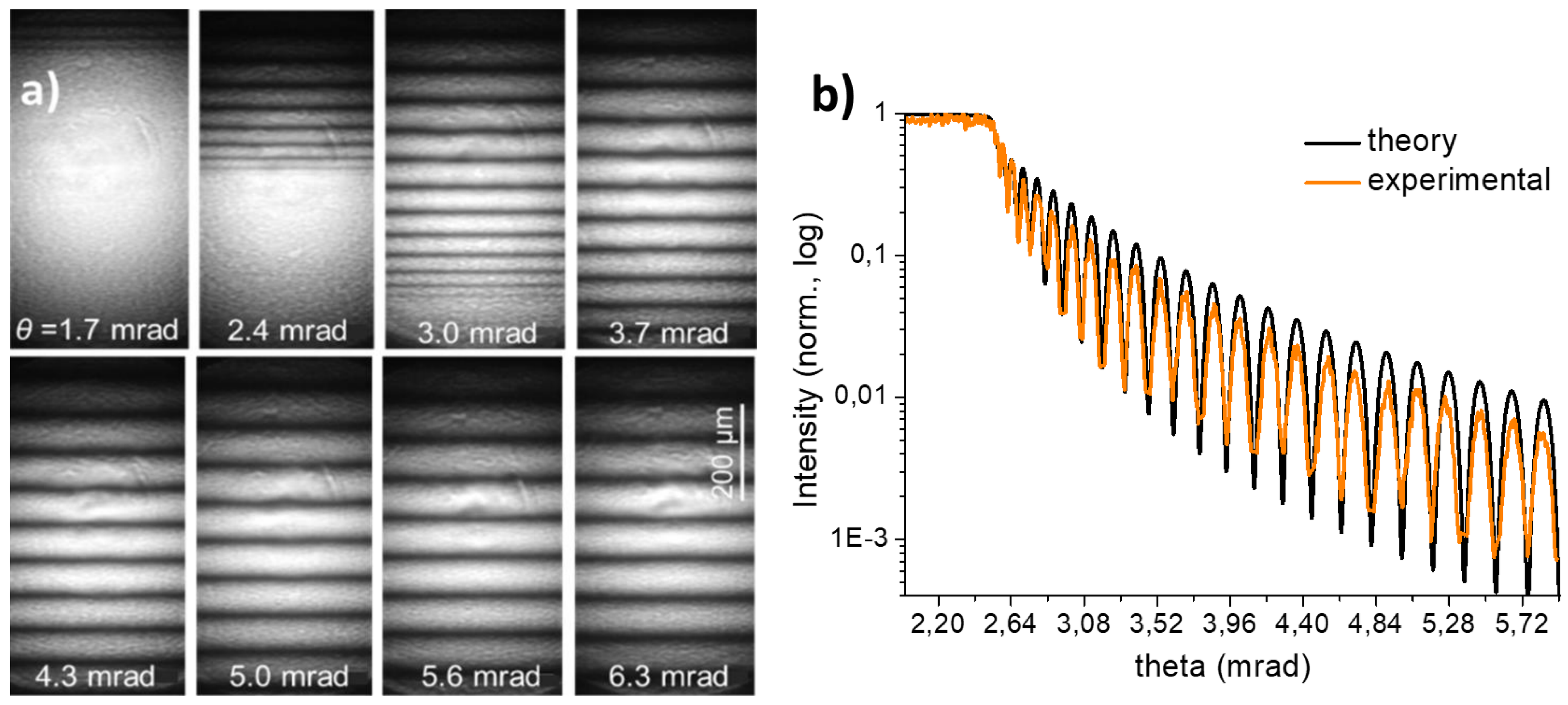


Figure 2. Interference patterns for a 200 nm membrane at different inclination angles with 14.4keV X-rays (a). The scale 200 mm corresponds to 486 mrad. The intensity is represented in logarithmic scale. The experimental curve obtained by combining interferograms recorded at membrane inclination angles from 2.4 to 5.6 mrad (b**).**

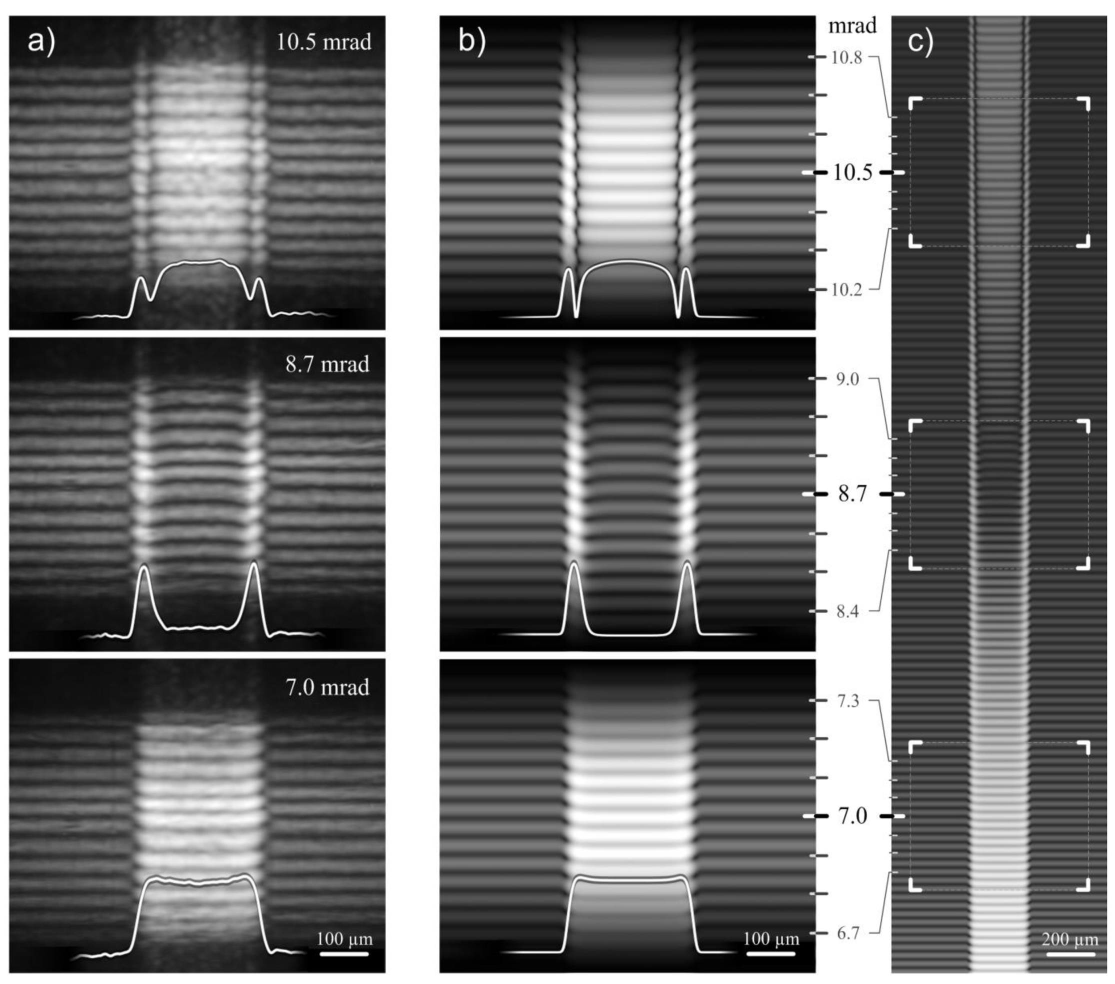


Figure 4. (a) Experimental reflecto-interferograms recorded at different angles 7.0, 8.7, 10.5 mrad. (b) Numerically simulated reflecto-interferograms at the inclination angles corresponded to experimental data. (c) Calculated interferogram in a wider range of inclination angles. The intensity is represented in logarithmic scale.

Main conclusion

The new reflecto-interferometry technique opens a wide horizons for both rapid thin-film and multilayer systems analysis, studying of the processes dynamics at the surface and in sample depth, and for complex structured and biological samples studying. The recording of the interference pattern in a single shot allows a express analysis of films, which is especially important for radiation sensitive materials, including organic and biological films. The spatial resolution property is very significant possibility to study samples with small lateral size, and is also necessary for the local analysis of the film.

Reference: S. Lyatun, D. Zverev, P. Ershov, I. Lyatun, O. Konovalov, I. Snigireva & A. Snigirev, *"X-ray reflecto-interferometer based on refractive optics",* J. Synchrotron Rad. **26**, https://doi.org/10.1107/S1600577519007896, 2019.

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X-Ray Coherent Optics Laboratory

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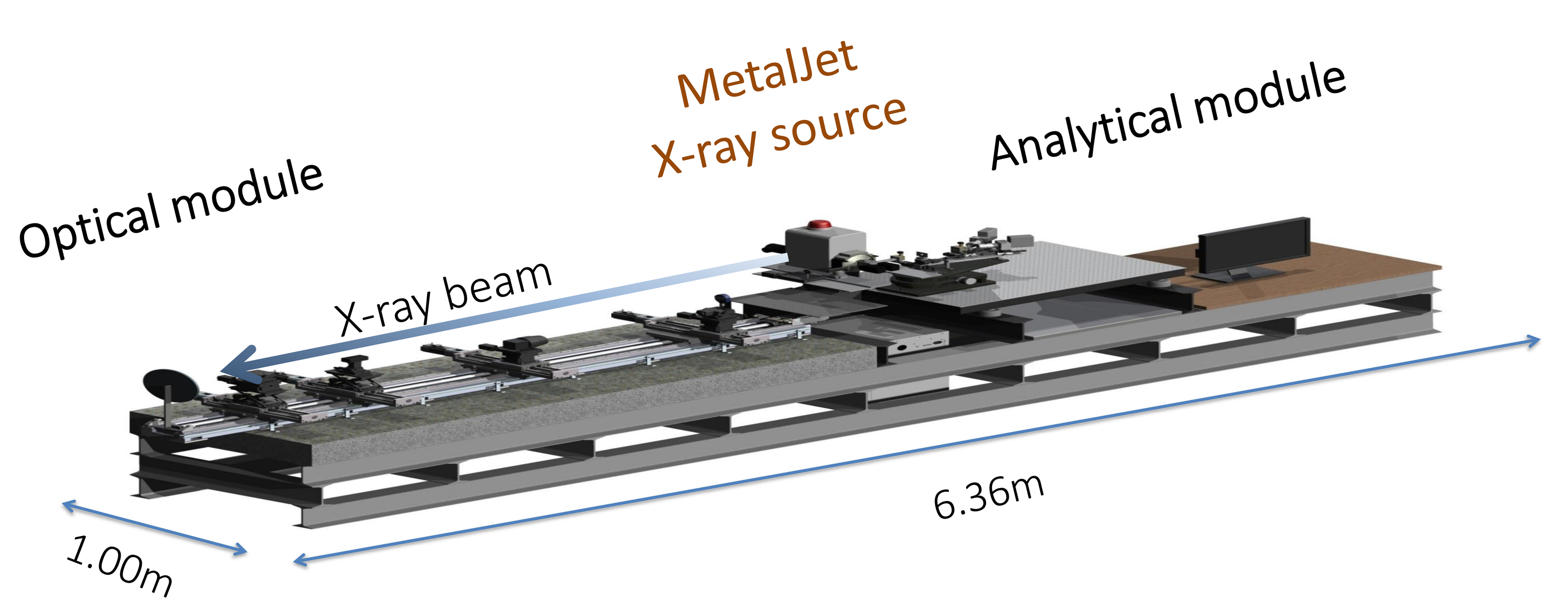
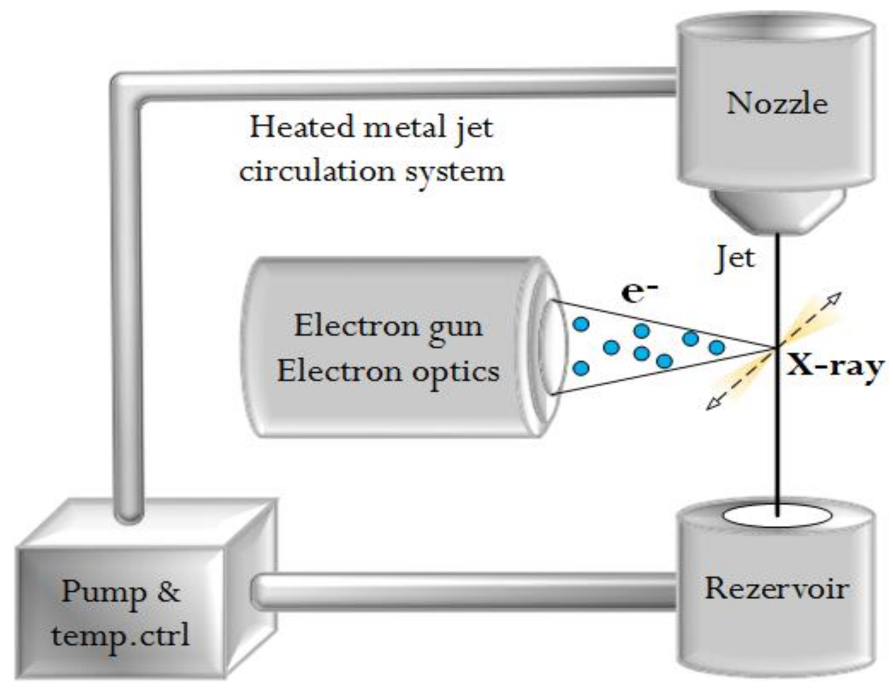
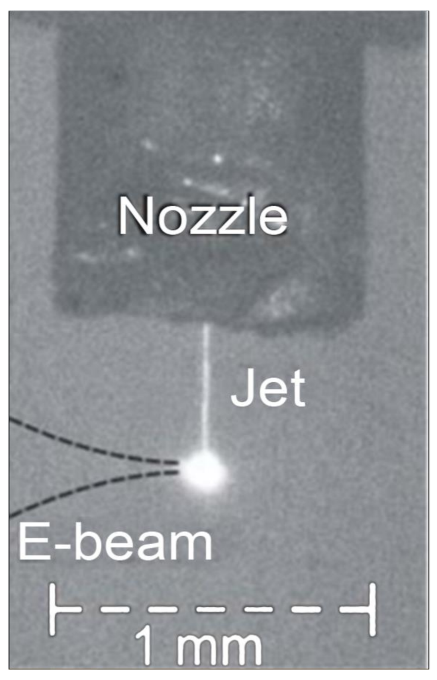
Laboratory complex «SynchrotronLIKE»







Basic MetalJet specifications

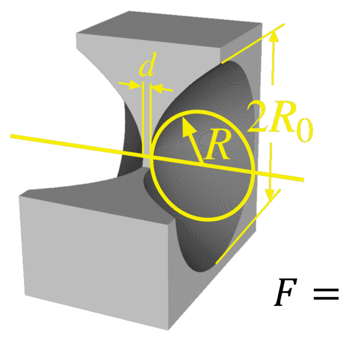
- Min. focal spot size ~ 5 um
- X-ray energy Ga k-alpha (9.25 keV)
- Power – 300 W
- Accelerate voltage up to 70 kV
- Brightness – $1.5 \cdot 10^{11}$ [Ph/(s·mm²·mrad²·keV)]
- Emission stability – less 1%
- Minimum focus-object distance – 18 mm
- Beam angle – 13°

X-Ray source technology

X-Ray tubes are conventional microfocus tubes with the solid-metal anode replaced by a liquid-metal jet. The metal jet supports higher electron beam power and can generate higher x-ray flux. **>1000 kW/mm² e-beam power density!**
(rotating anode ~ 1000 kW/mm²)

X-Ray refractive optics

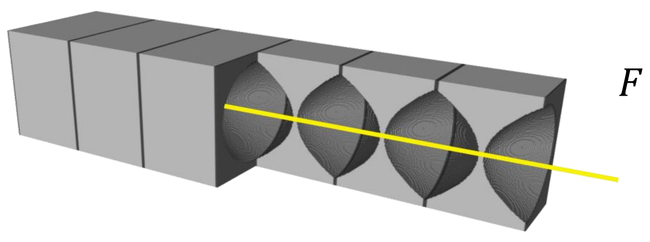
Single lens



$R = 0.05 \div 1.5$ mm
$2R_0 = 0.3 \div 1.5$ mm
$d \approx 0.1$ mm

$F = \dfrac{R}{2\delta}$

Compound lenses

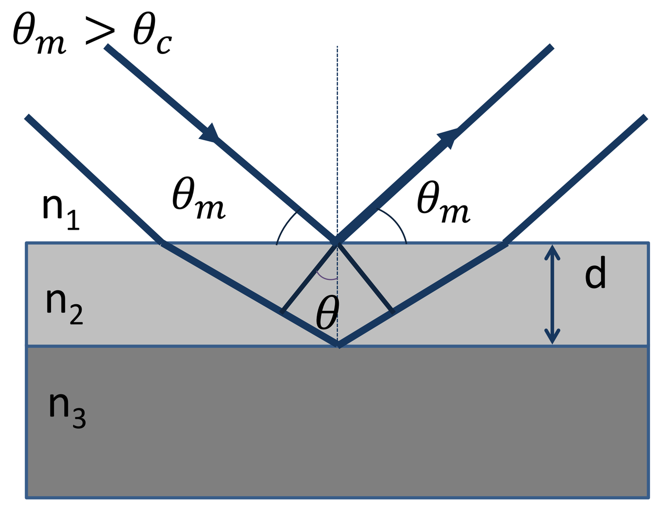


$F = \dfrac{R}{2\delta N}$

The advantages of parabolic refractive lenses:

- Large energy range 2 - 200 keV
- Beam conditioning optics: Condensers, Collimators, Monochromators, Harmonics rejecter
- Beam/emittance diagnostics
- Compatibility with crystal optics
- Coherence related techniques: micro (nano)-beam: diffraction, scattering & spectroscopy; full-field imaging and diffraction microscopy, dark-field microscopy, in-line interferometry

X-Ray reflectivity



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

$n_2 \cos(\theta) = n_1 \cos(\theta_m)$

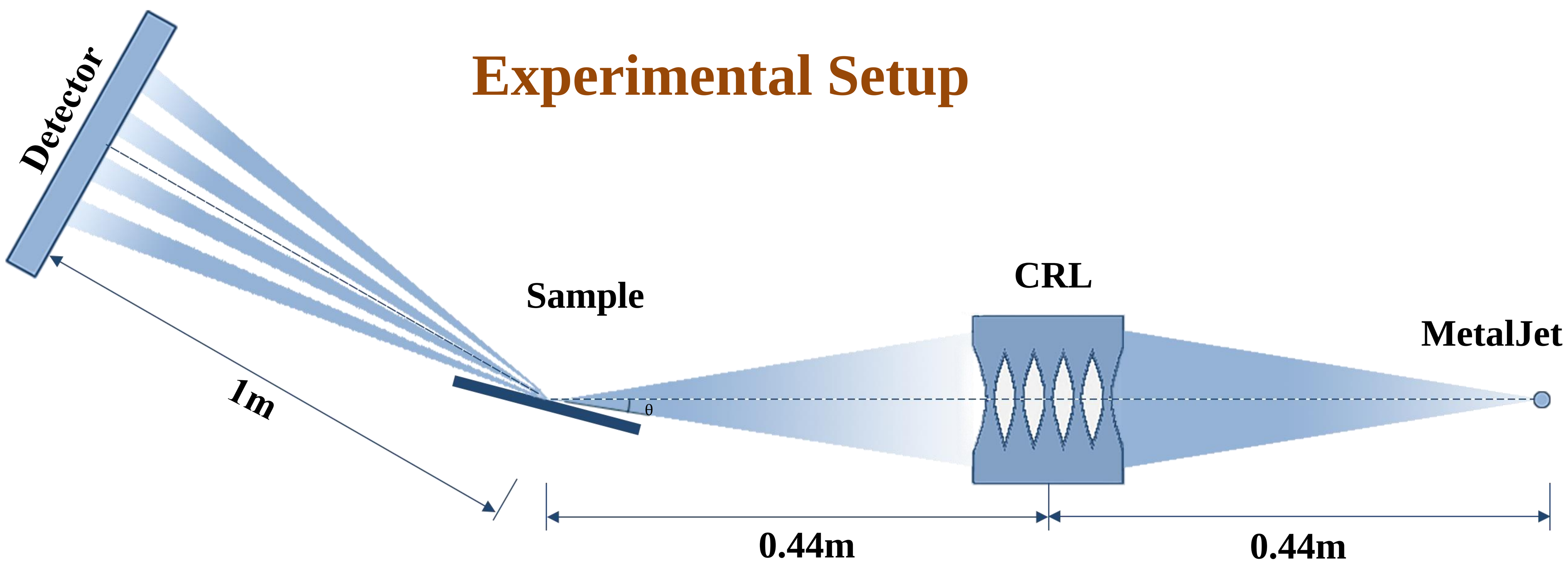
$n = \cos(\theta_c) \approx 1 - \frac{\theta_c^2}{2}$

$\theta^2 = \theta_m^2 - \theta_c^2$

$\theta_m^2 = \frac{m^2\lambda^2}{4d^2} + \theta_c^2$

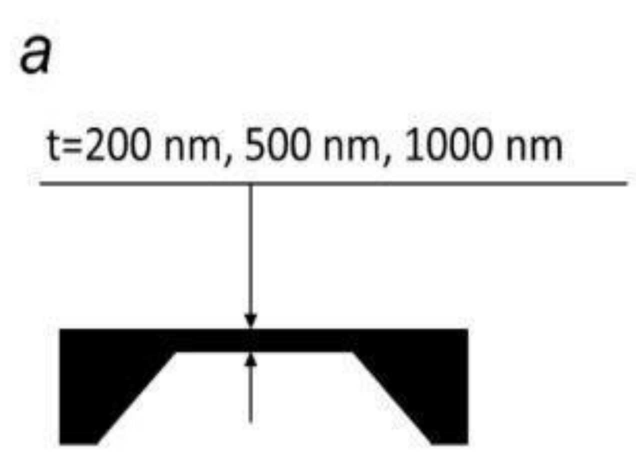
$$d = \frac{\lambda}{2}\frac{1}{\sqrt{\theta_{m+1}^2 - \theta_c^2} - \sqrt{\theta_m^2 - \theta_c^2}} \approx \frac{\lambda}{2}\frac{1}{\theta_{m+1} - \theta_m}$$

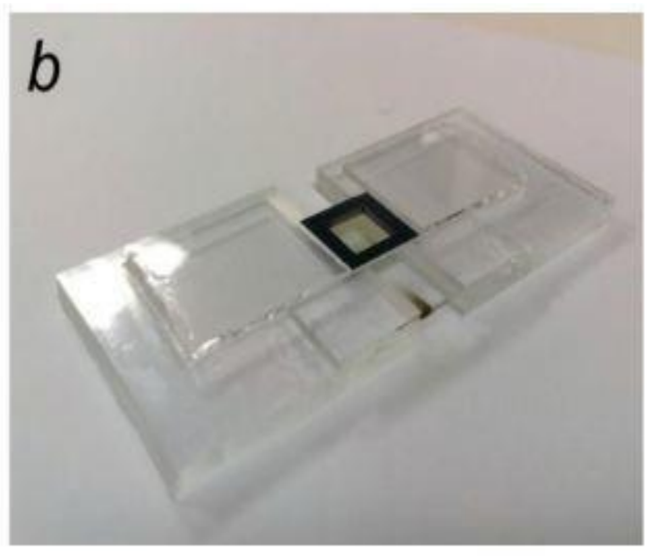
Experimental Setup



Detector Photonic Science – pixel size 6,5um
Compound refractive lens Be, N=29, R=50um

Samples

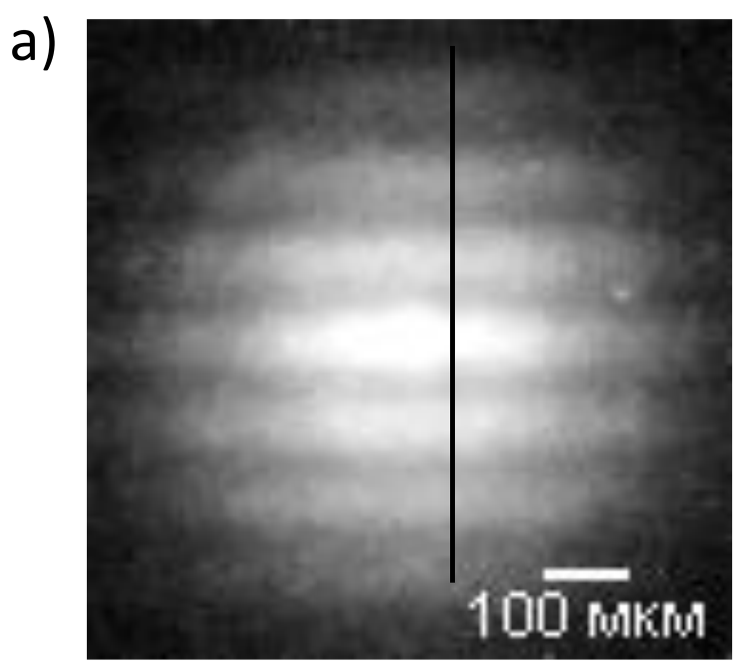


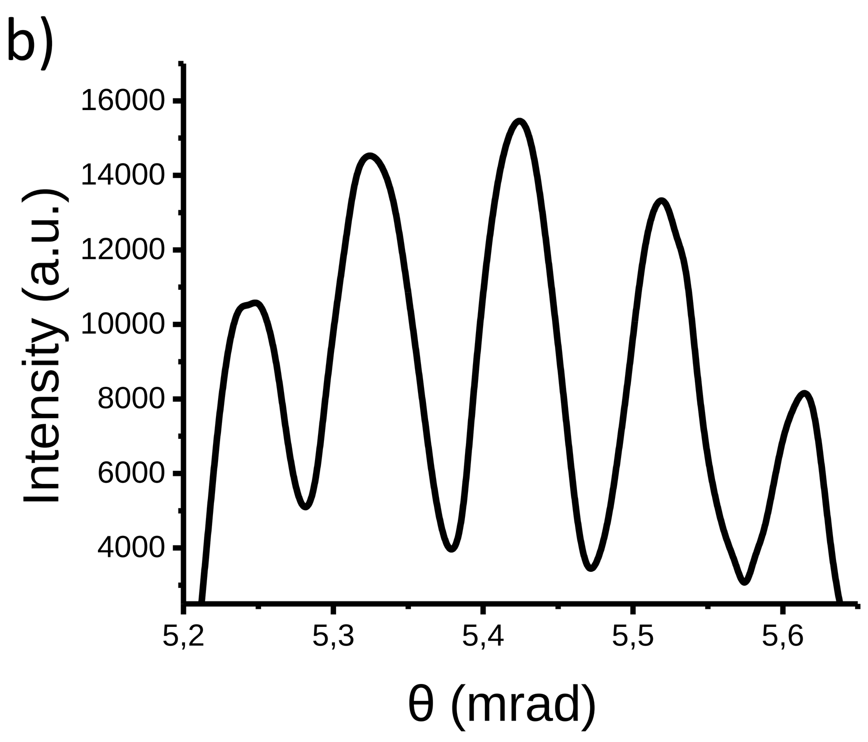


The Si_3N_4 membranes drawing (a). Specially designed glass holder for membranes with burnt beam print (b)

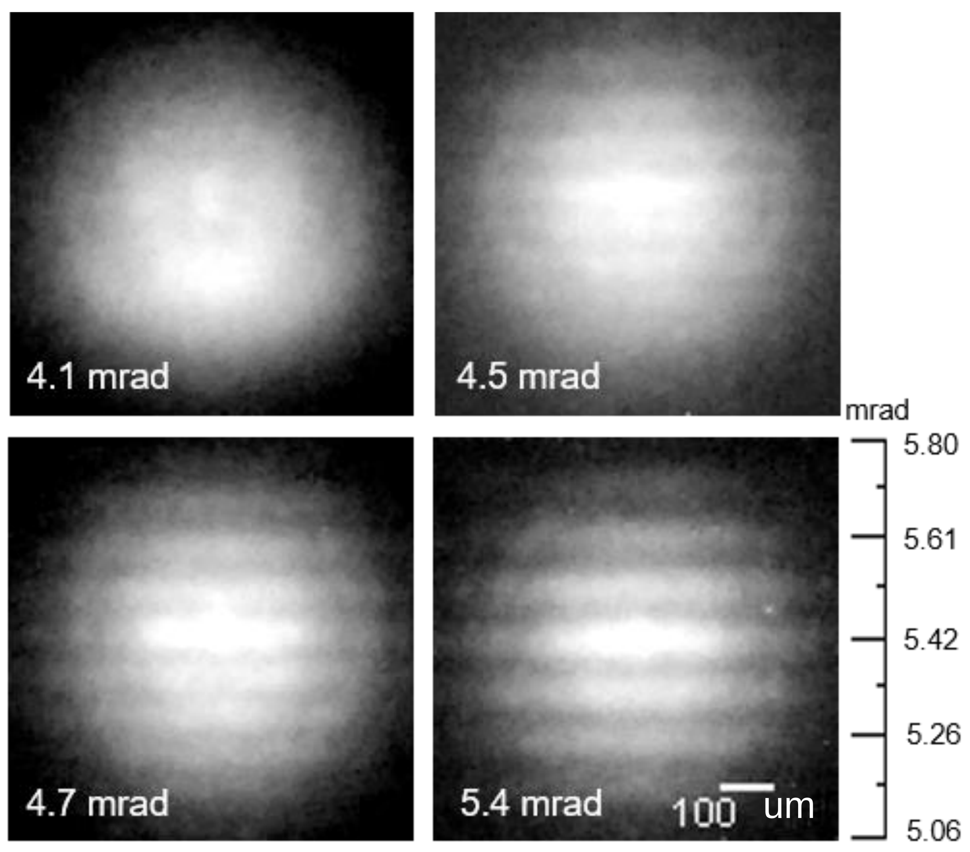
*http://www.silson.com/

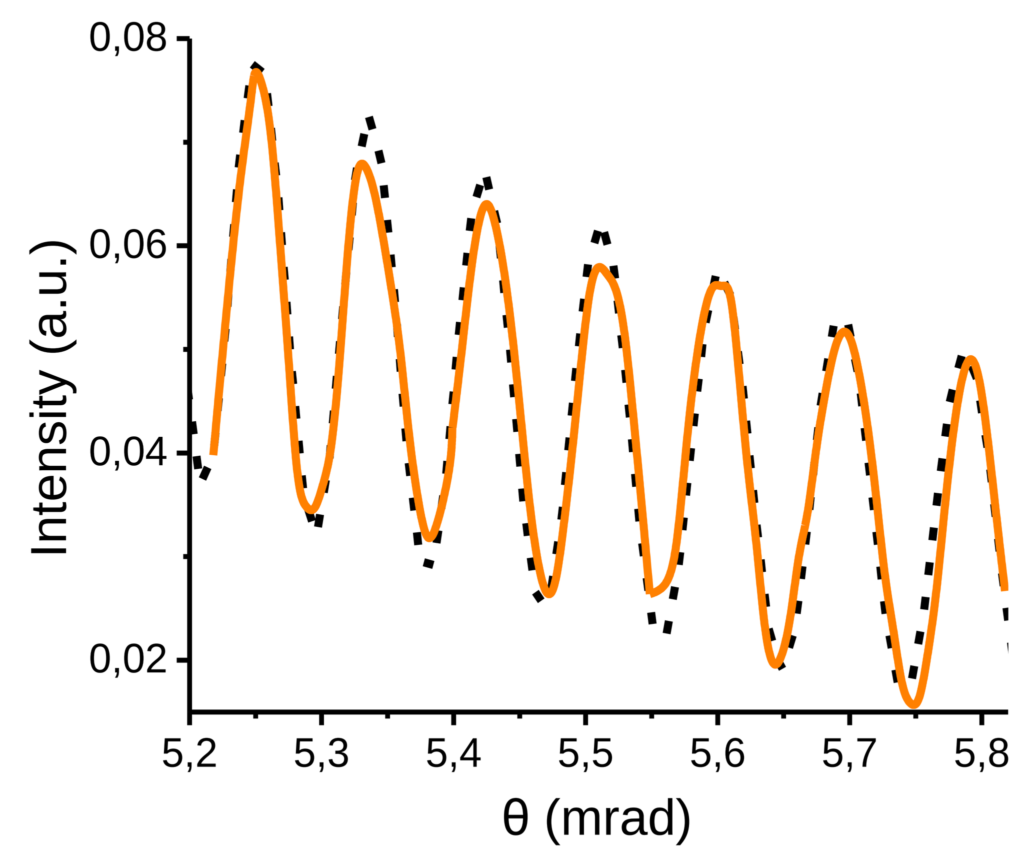
Results





Interference patterns of 500 nm membrane at inclination angle is 5.4 mrad with 9.25keV X-rays (a). The experimental curve (b).





Interference patterns for a 500 nm membrane at different inclination angles with 9.25 keV X-rays. The experimental reflectivity curve obtained by combining interferograms recorded at membrane from 5.2 to 5.8 mrad inclination angles.

Main advantages

- **high time resolution (one shot experiment & in-situ)**
- **high spatial resolution**
- **simple optics for large range of samples thicknesses**
- **lower requirements to quality and geometry of the sample surface**

Reference

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