Recent developments in hard X-ray tomography

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Abstract

A new technique for magnified hard X-ray tomography using compound refractive lenses (CRLs) has been tested. A full-field X-ray microscope was included into a conventional microtomography setup at a synchrotron undulator beamline. Experiments were carried out at 19.7 keV with a monochromatic beam as well as with the so-called “pink” beam using a larger energy bandwidth. During this pilot experiment a resolution of about 1 \(\mu\)m was already achieved, which corresponds to the best resolution obtained with phase-contrast enhanced microtomography. The technique has the potential to increase the spatial resolution of hard-X-ray microtomography to a scale of several hundred nanometers. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

In the field of hard X-ray tomography one objective is the improvement of spatial resolution. Several ways can be followed to achieve this goal. One possibility is the use of high-resolution X-ray films or detector screens coupled with an optical microscope system and a CCD camera [1], pushing the resolution to new limits.

Increased resolution can also be achieved by modifying the geometry of the X-ray beam, for example using scanning techniques with focused X-ray beams or imaging of stretched projections in fan beam geometry or with asymmetrically cut crystals [2,3].

Recently developed optical elements for hard X-rays, such as Fresnel zone-plates (FZPs) [4], Bragg–Fresnel lenses (BFLs) [5,6], and compound refractive lenses (CRLs) [7–10], opened a large field of new possibilities for this purpose and are comparable to lenses for visible light [8,10]. These devices are used in combination with a coherent light source (third generation synchrotron) for resolution enhanced phase contrast imaging and holographic techniques [11–13].

For magnified tomography, already shown with soft X-rays [14,15], we have built a full-field microscope for the much more penetrating hard X-rays using a parabolic compound refractive lens (CRL) [9,10]. The CRL consists of a chosen number of single parabolic aluminum lenses [9,10,16], so that the geometry of the X-ray microscope and the X-ray energy are independent. This device can be used at high incident intensities...
and at high photon energies and has a large depth of field.

The aim of the study was to show an alternative possibility to the existing methods in order to improve the resolution in magnified hard X-ray tomography with later on optimized parameters. Given the strong chromatic aberration of the lenses, the question that arose was, how far the use of X-ray light with a large energy bandwidth ("pink beam": $\Delta E/E = 10^{-2}$) permits tomography with reasonable resolution. In this case the high photon flux would considerably reduce the acquisition time.

2. Experimental setup

The scheme of the experiment carried out at the European Synchrotron Radiation Facility (ESRF) at the micro-Fluorescence Imaging and Diffraction beamline ID22 is shown in Fig. 1. The polychromatic beam from the undulator was deflected by a palladium-coated plane silicon mirror at a deflection angle of 0.3° to cut off the higher undulator harmonics. As an alternative to the setup with a conventional double-crystal fixed-exit Si-111 monochromator ($\Delta E/E = 10^{-4}$ at 20 keV) a molybdenum foil of 250 µm thickness was introduced into the beam.

The latter setup referred to as “pink-beam” can be used to filter out one part of a single undulator harmonic ($\Delta E/E \sim 10^{-3}$), slightly below the absorption edge of the filter, with the advantage of a high transmitted flux ($I_\text{pink} = 100 \times I_\text{mono}$).

At a distance of 41 m from the 800-µm-wide and 30-µm-high (FWHM) undulator source, the sample was mounted on a tomography stage with horizontal rotation axis (see also Refs. [17,18]). The sample was a 100-µm-thick boron fiber with a 15-µm-thick tungsten core [13]. A magnified real image of the fiber was projected with a compound refractive lens (CRL) onto the detector.

For briefness only the experiment with 10-fold X-ray magnification in pink beam mode is described as a typical example for other experiments comparing different beam modes and magnifications (see Ref. [19]).

The object-to-detector distance was 24 and 2.1 m between object and lens for the experiment with 10-fold magnification. The photon energy was 19.7 keV and a CRL made of $N = 36$ single lenses was used. The theoretical diffraction limit is for this configuration 0.5 µm.

The images were detected with a 12 µm thick Europium-doped Lutetium Aluminum Garnet (LAG) scintillator and recorded by a microscope optic on a high-resolution CCD-based “FreLoN2000” (2048 × 2048-pixel; 14 bit dynamic range) camera system [18]. The effective pixel size of the whole detector system was 2.8 µm. After darkfield correction and flatfield normalization of the projections, the tomograms were reconstructed with the filtered backprojection technique [20].

3. Results

Experiments comparing pink-beam and monochromatic beam tomography at low X-ray magnification (3.4 ×), showed no significant difference in resolution for both modes (for detailed description see Ref. [19]).

Then a tomography in pink-beam mode with 10 × X-ray magnification at an energy of 19.7 keV was carried out (see Fig. 2). Using an exposure time of 8 s for each projection and 250 projections over an rotation angle of 180° a resolution of about 1 µm was obtained for the reconstructed slices. This result is comparable to the optimal resolution achieved with a conventional microtomography setup without magnifying X-ray optics. In combination with the experiments at
low X-ray magnification [19], it turned out that the X-ray microscope becomes the resolution limiting factor. The difference between the theoretical diffraction limit of the CRL (0.5 μm) and the resolution of the reconstructed tomogram slices (1 μm) can be partially explained by the quality of the lenses, which had been heavily used before and showed traces of surface degradation (oxidation). Surface roughness of the lenses further decreases the image quality and may become the dominant resolution-limiting factor [9,10,16].

These promising results indicate that the limits of resolution in hard X-ray microtomography can be brought to lower values as soon as a sufficient amount of single high-quality aluminum lenses will be available.

4. Conclusion

Magnified hard X-ray tomography with compound refractive lenses allows to achieve a spatial resolution of 1 μm in the reconstructed tomograms that exceeds the resolution of the detector system of several microns. The resolution of the test experiment corresponds already to the optimal resolution achievable with conventional microtomography.

Further improvements in resolution will be going along with the progress in lens technology. With a larger number of high-quality single lenses it will be possible to use higher energies. The use of lenses with low surface roughness and low absorbing materials (Be) will open the field to sub-micron hard X-ray tomography in future. The combination of low absorbing X-ray lenses with X-ray photon efficient low resolution scintillator screens will also reduce the acquisition time in general.

References