Toward Hard X-ray Transmission Microscopy at the ANATOMIX Beamline of Synchrotron SOLEIL

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Among the techniques that are being implemented on the imaging beamline ANATOMIX at Synchrotron SOLEIL [1,2], hard X-ray full-field microscopy is of paramount importance. The transmission X-ray microscope (TXM) on ANATOMIX, based on diffractive optics, is designed for photon energies around three working values of 6.6, 10 and 18 keV. It aims at a spatial resolution down to 100 nm or less, corresponding to pixel sizes down to 30 nm.

The diffractive optics for the TXM—beam shapers (condensers), objective zone plates and phase masks for Zernike phase contrast—are manufactured at the Laboratory for Micro- and Nanotechnology of the Paul Scherrer Institut in Villigen, Switzerland. A first set of beam shapers and objectives has been produced, and tests in absorption contrast were recently conducted, using a temporary mechanical setup.

These test measurements were carried out with a set of optics designed for 10 keV: a beam shaper [3] illuminating a field of view of $40 \times 40 \,\mu\text{m}^2$, with a physical aperture of 2.5 mm diameter and smallest zones of 50 nm width, and objective zone plates of the same outermost zone width and a diameter of 100 µm, resulting in a focal length of 40 mm at 10 keV. The diffractive X-ray optics were made of iridium using the frequency-doubling method [4], with structure heights of about 1 µm for the beam shaper and 1.6 µm for the objective zone plates, which were patterned on both sides of the membrane [5]. Other elements included a central stop placed a few cm upstream of the beam shaper, an orderselecting aperture (OSA, pinhole with 0.3 mm diameter) 70 mm from the sample and a diffuser (sheets of paper) placed upstream of the OSA. Two indirect, lens-coupled detectors were used: one for alignment, placed just downstream of the objective zone plate and consisting of a 22-µm-thick lutetium aluminum garnet (LuAG) scintillator, a 10× magnifying microscope optics and a CMOS-based digital camera with 6.5-µm pixels (Hamamatsu Orca Flash 4 V2), resulting in an effective pixel size of 0.65 µm. The other detector, used to record the actual TXM micrographs, was placed 4.5 m downstream of the objective zone plate and composed of a 100-µm-thick scintillator, photo-camera optics with an overall magnification of 3.4 and a CCD camera (PCO 4000, 9-µm pixels) using 2×2 pixel binning, resulting in an effective detector pixel size of 5.3 µm. Including the X-ray magnification factor of 110, this resulted in a pixel size of 49 nm at sample level. The alignment detector and sample stage were mounted on standard translation stages (Huber Diffraktionstechnik, Rimsting, Germany), the TXM optics on piezo-based translation stages (SmarAct, Oldenburg, Germany).

Figure 1 shows a TXM micrograph obtained with this setup on a resolution test chart (model XRESO-50, NTT-AT, Japan; tantalum structures of 500 nm height). The smallest structures in the center of this reference sample of "Siemens-star" type, resolved in the image (see inset of Figure 1), have a period of 100 nm and are thus just at the Nyquist limit of resolution for the pixel size of 49 nm.

Using the same setup, a tomography test was then performed. A rock shale sample with pyrite inclusions served as a test object. The sample was placed on a high-precision rotation stage (model RT150v3, LAB Motion Systems, Heverlee, Belgium) whose runout had previously been characterized at SOLEIL to be on the order of 20 nm. Figure 2 shows a vertical reconstructed slice (i.e., a slice parallel to the tomography rotation axis) of a tomography volume of $900\times900\times900$ voxels (field of view $45\times45~\mu\text{m}^2$) obtained in 4 hours (600 projection angles over 180° , 5 s exposure time per projection micrograph, superposition of 5 scans of this type). The resolution in the tomogram is estimated to around 250 nm, limited mostly by mechanical drift due to temperature variations during the scan. The support structure of the preliminary test setup was far from optimized in this respect.

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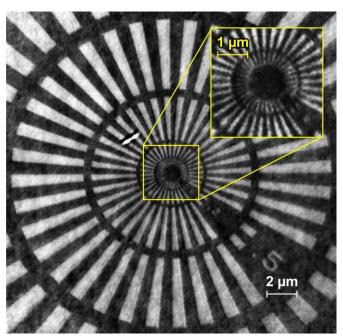


Figure 1. Detail of a TXM micrograph showing a reference chart with smallest widths of lines and spaces of 50 nm (center of inset).

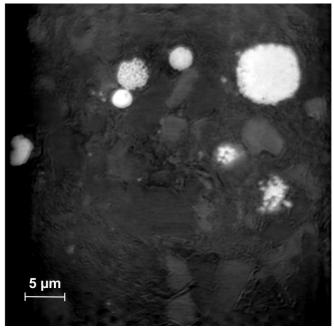


Figure 2. Sagittal slice of local tomography on a shale-rock sample with pyrite inclusions (light gray). The sample had a diameter of 160 μm.

References:

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