Broadband X-ray full field microscopy at a superbend

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Abstract. Over the last decade, synchrotron-radiation based X-ray Tomographic Microscopy (SRXTM) has established itself as a fundamental tool for non-invasive, quantitative investigations of a broad variety of samples, with application ranging from space research and materials science to biology and medicine. The beamline for TOMographic Microscopy and Coherent Radiology experiments (TOMCAT) has been recently equipped with a full field, hard X-ray microscope with a theoretical pixel size down to 30 nm and a field of view of 50 microns. The nanoscope performs well at X-ray energies between 8 and 12 keV: here we illustrate the experimental setup and the performance of the instrument in both microscopy and tomography mode.

1. Introduction

The combination of the unique properties of X-ray microscopy (high-resolution, large penetration depth and chemical speciation) with advanced tomographic methods as well as the exceptional properties of third-generation synchrotron radiation sources allow to obtain volumetric information of a specimen with voxel size in the micrometer range.

Even rookie synchrotron-CT users require today flexible endstations, capable to cover field of views (FOV) varying up to three orders of magnitude with corresponding spatial resolution and/or acquisition mode. Frequently, such users also demand an easy and fast swap between antipodal configurations, for instance between a 10 mm FOV with 10 microns resolution in phase contrast and a 50 microns FOV with 100 nm resolution in pure absorption.

Reaching and trespassing the isotropic barrier of 100 nm remains a challenge and only few instruments, mostly in the soft X-ray range, i.e. for energies well below 10 keV have this capability [1]. The most common approach to achieve sub 100 nm (2D) resolution is full-field X-ray microscopy. The key optical elements are a condenser to provide illumination of the sample and an objective lens to produce a magnified image of the sample on the detector. The illumination should be as homogeneous and intense as possible and the numerical aperture should be matched to that of the objective lens in order to obtain optimum resolution [2]. Instead of the more conventional Fresnel zone plates, tapered capillaries or mirrors, we made use of a beam-shaper with complex optical functionality as condenser in the X-ray regime. The idea of the design is to divide a conventional Fresnel zone plate (FZP) into sectors, keeping the local spatial frequency within each sector constant (in our case square gratings). As a result, each sector will produce a flat-top illumination in the focal plane [3].
2. Experimental Setup
The nanoscope installed at the TOMCAT beamline is approximately 12 meters long. The beamshaper, is placed just after the exit window of the beamline, at 17 meters from the source. The X-ray beam at that position is tailored to a size of 2x2 mm$^2$ by the exit slits of the beamline. A motorized slits pair (JJ-Xray, Type 3 Slit Air Version) is used as an adjustable order sorting aperture (OSA). The sample is mounted on an air-bearing rotation stage with an xy-centering device. As an objective lens, a gold FZP with a 100 nm outermost zone and a diameter of 100 microns is used [4]. Immediately downstream the zone plate, the high-resolution microscope (normally used at TOMCAT for routine experiments in the micron range) can be inserted into the beampath. This allows a very quick alignment of beamshaper, beamstopper and zone plate. The focal length of the FZP used for this experiment is 64.5 mm and 80.6 mm at 8 keV and 10 keV respectively. By positioning the 2D detector at distances larger than 10 m (Photonic Science Hystar Camera, 2048x2048 pixels, true 14 bit dynamic range, 1:3 magnifying fiber optic taper and 1.5 mg/cm$^2$ Gadox scintillator resulting in a optical resolution of 4.7 microns) we can obtain X-ray magnifications up to 180x.

3. System performance
The radiographic imaging properties of the system have been characterized at 8 keV. We were targeting a resolution of 100 nm. For this purpose, as a test sample, we used a 20 microns diameter gold zoneplate with an outermost zone width of 100 nm. Figure 1 shows a resolution measurement for an X-ray projection. Figure 1b depicts the corresponding X-ray radiographic image (absorption image) of the test sample shown in figure 1a. A line profile over the central region of the zone plate (Fig. 1c) shows that intensity oscillations at the frequency of 5 lp/μm could be detected with sufficient contrast (Fig. 1d). The detector has been used in 2x binning mode, giving a theoretical pixel size of approximately 60 nm.

![Figure 1](image1.png)

**Figure 1.** (a) SEM of the reference sample: 20 microns diameter gold FZP with 100 nm outermost zone width. (b) Corresponding X-ray projection acquired at 8 keV. (c) Line profile and (d) zoom in into the profile, showing contrast at the target resolution of 100 nm.

We developed a test sample which is easy to align and ideally suited for tomographic investigations. It consists of a BaTiO$_3$ sphere of 22 microns diameter with calibration holes of 5, 2, 1, 0.5 and 0.25 microns diameter, milled with focus ion beam, see Figure 2a. The sphere has been coated by a thin gold layer to avoid charging during FIB milling. In the corresponding radiographic projection, see Fig. 2b,
the gold coating is visible on the right side, while in the center the typical conical shape of the FIB holes are recognizable. After tomographic data acquisition (501 projections over 180°), we generated an isosurface rendering which clearly shows the entrance point of the reference apertures, see Fig. 2c. Holes with diameters of 5, 2, 1 and 0.5 microns on the surface of the sphere are clearly visible. The position of the 0.25 microns hole can be grasped as well.

**Figure 2.** (a) SEM of the reference sample used for nanotomography: a BaTiO$_3$ sphere of 22 microns diameter with calibration holes of 5, 2, 1, 0.5 and 0.25 microns. (b) Corresponding X-ray projection acquired at 10 keV. (c) Surface rendering showing the entrance points of the reference holes. (d) Virtual cuts through the test sample, showing the typical cone shape of the FIB-milled holes. The FIB-milling in deep recesses resulted in holes of aspect ratio of approximately 5:1. Therefore the 5 microns hole was milled all the way through the sphere, while the 2 microns only reached the center of the sphere.

4. Outlook
In the next future we will further improve the stability and fine tune our registration algorithms. A new, larger beamshaper will collect a beam area of up to 3x3 mm$^2$ and tailor it down to 50x50 microns$^2$, therefore augmenting the flux at the sample position by a factor of 4. We also plan to update the present detector with a more efficient system which should result in a reduction of the exposure time by at least a factor of 2.

Finally, our efforts will be focused in the tentative of producing phase contrast images with the nanoscope.

**References**
[1] Meyer-Ilse W, Hamamoto D, Nair A, Lelievre SA, Denbeaux G, Johnson L, Pearson AL, Yager D, LeGros MA, Larabell, “High resolution protein localization using soft X-ray microscopy”, Journal of Microscopy **201**, 395-403, (2001)
[2] Born M & Wolf E, “Principles of Optics”, 7th ed., Cambridge University Press, 595 (1999)
[3] Jefimovs K, Vila-Comamala J, Stampononi M, Kaulich B, David C, “Beam-shaping condenser lenses for full-field transmission X-ray microscopy”, Journal of Synchrotron Radiation **15**, 106-108 (2008)
[4] Jefimovs K, Bunk O, Pfeiffer F, Grolimund D, van der Veen J F, David C, Fabrication of Fresnel zone plates for hard x-rays, Microelectronic Engineering **84**, 1467–1470 (2007)