Hard x-ray nanobeam characterization by coherent diffraction microscopy

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We have carried out a ptychographic scanning coherent diffraction imaging experiment on a test object in order to characterize the hard x-ray nanobeam in a scanning x-ray microscope. In addition to a high resolution image of the test object, a detailed quantitative picture of the complex wave field in the nanofocus is obtained with high spatial resolution and dynamic range. Both are the result of high statistics due to the large number of diffraction patterns. The method yields a complete description of the focus, is robust against inaccuracies in sample positioning, and requires no particular shape or prior knowledge of the test object. © 2010 American Institute of Physics. [doi:10.1063/1.3332591]

The investigation of the structure of nanoscale objects has become increasingly important during the last decades, for example, in biology, chemistry (catalysis), physics, materials science, and nanotechnology. X-ray microscopy is ideally suited to determine structure and to describe physical processes occurring on the nanoscale, as it can access inner features of an object without destructive sample preparation and allows for in situ investigation.1 Different x-ray analytical methods can serve as the contrast mechanism, such as x-ray absorption spectroscopy, x-ray fluorescence analysis, and x-ray scattering, giving access to local chemical, elemental, and structural information of a specimen with high spatial resolution.2

During the past decade hard x-ray microscopy has made significant progress, in particular, as a result of the high brilliance of third generation synchrotron radiation sources, such as the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, the Advanced Photon Source (APS) in Chicago, and SPring-8 in Japan. X-ray microscopes based on focusing or full-field imaging have been developed, relying strongly on highly sophisticated x-ray optics. Today, spatial resolutions down to below 10 nm have been obtained.3,4 However, the smaller the beam the more difficult is its characterization, which is important to correctly interpret imaging results from these high-end scanning microscopes. So far, most characterization measures one-dimensional intensity profiles by knife-edge techniques. This characterization is tedious, requires the exact knowledge of the nanostructured knife-edge and highly accurate positioning stages, and yields only a few characteristics of the complex wave field of the nanobeam.

As the radiation in a diffraction-limited nanobeam is intrinsically highly coherent, diffraction imaging techniques with coherent illumination are well suited for their characterization.5 In particular, ptychographic coherent diffraction imaging6,7 based on the combination of scanning microscopy and coherent x-ray diffraction imaging offers a solution for full nanobeam characterization. In this technique, an object is scanned through a transversely modulated coherent beam, and at each position of the scan a far-field diffraction pattern of the object is recorded. From the diffraction patterns and knowledge of their positions in the scan, both the object’s complex transmission function and the complex illuminating wave field can be reconstructed.7–10

In this letter, we use this method to characterize the full complex wave field (Fig. 1) of the nanobeam in our hard x-ray scanning microscope, determining the intensities in the plane of the focus with high spatial resolution over a dynamic range of five orders of magnitude. The key to a faithful reconstruction is a large number of diffraction patterns (in this case more than 15 000) collected in the experiment, each of them encoding information about the illuminating wave field. For the beam characterization, no prior knowledge of the test object is needed, as long as it has a sufficiently high structural diversity10 and contrast. Due to the large overetermination of the illuminating wave field, its reconstruction proves to be very robust against unavoidable inaccuracies in the scanning positions. In addition, the test objects do not need to be perfectly in focus. At the same time, the object is reconstructed with a spatial resolution of well below 50 nm that is limited only by the highest diffraction angles covered by the detector.

![FIG. 1. (Color) 3D view of the focused x-ray beam. The amplitude of the wave field is encoded by brightness and the phase by hue (compare inset).](image-url)
The ptychographic imaging experiment was carried out at beamline ID13 of the ESRF to characterize the nanobeam of the hard x-ray scanning microscope based on nanofocusing refractive x-ray lenses. Each of these cylindrical lenses focuses the beam in one dimension. In the microscope, two of them are aligned behind each other in a crossed geometry to form a point focus about 13 mm behind the last lens. At an x-ray energy of $E=15.25$ keV, a highly coherent portion of the x-ray beam is collected and a full width at half maximum (FWHM) focus size of $67 \times 83$ nm$^2$ is expected in the horizontal and vertical directions, respectively.

In order to characterize the nanobeam, a sub-area of a two-dimensional resolution test chart $^{11}$ manufactured by NTT-AT was scanned in the focal plane with $125 \times 125$ steps and a step size of 40 nm, covering an area of about $5 \times 5$ $\mu$m$^2$. At each position of the scan, a far-field diffraction pattern (without beam stop) was recorded by a single photon counting and noise-free MAXIPIX detector having 256 $\times$ 256 pixels with an area of $55 \times 55$ $\mu$m$^2$, each. The detector was positioned at a distance of 1926 mm from the sample. A tube filled with helium was introduced between the sample and the detector in order to reduce background scattering from air. Due to the high x-ray optical contrast of the sample, an exposure time of 0.1 s per scan point was sufficient to collect diffraction data in the full field of view of the detector. The integrated number of photons in a single diffraction pattern was about $2 \times 10^5$. In parallel with the diffraction patterns, the fluorescence from the sample was recorded with an energy dispersive detector at each position of the scan.

The 15 876 diffraction patterns were used to reconstruct both the object and the illuminating wave field using ptychographic reconstruction schemes. The phase retrieval algorithm employed in this work is based on contributions of Rodenburg et al. The reconstruction of the wave field in the focus is implemented as proposed by Maiden and Rodenburg. The reconstruction was started with an initial Gaussian wave field (FWHM: 100 $\times$ 100 $\mu$m$^2$) and a flat object of unit transmission.

Figure 2(a) shows the reconstructed phase of the transmission function of the object with a pixel size of 17.8 nm. The scanned area is marked by a white dashed line. The comparison between the x-ray fluorescence map of tantalum [Fig. 2(c)] and the ptychogram [Fig. 2(a)] shows an increase in spatial resolution by at least a factor of 4 for ptychographic imaging over conventional scanning microscopy. In the ptychographic reconstruction the 50 nm lines and spaces are sharply resolved as depicted in Fig. 2(b). As the sample is slightly rotated around the vertical axis to allow for the detection of the fluorescence radiation from the side, structures with a vertical component have a more complicated contrast and appear slightly smeared.

Figure 3 shows the reconstructed wave field in the focus. In Fig. 3(b) the same wave field is shown and scaled such as to highlight the detailed shape of the wave field at low intensity. The side maxima are due to the limited aperture of the x-ray lenses of about 40 $\mu$m. They act in a way similar to a pair of slits. The slight tilt of the cross-shaped illumination function is related to the fact that also the lenses were slightly tilted as a whole, cf. Fig. 3(b). The central peak of the focus can be described by a Gaussian with a width of approximately $78 \times 86$ (vertical) nm$^2$ (FWHM), which is very close to the theoretical limits of $67 \times 83$ (vertical) nm$^2$. Figure 4 shows the intensity profiles through the focus along the vertical and horizontal directions, respectively. The reconstructed wave field fully characterizes the x-ray nanoprobe, giving a detailed picture of the full caustic of the beam. Figure 3(c) depicts a horizontal slice through the complex wave field rendered in three dimensions in Fig. 1.

Since the whole set of diffraction patterns contributes to the reconstruction of one common illuminating wave field, the photon statistics for recovering the illumination is significantly improved as compared to that for the reconstruction of the object function. This averaging effect reduces the presence of noise in the reconstructed wave field and is the reason that the intensity distribution can be resolved with a dynamic range extending over five orders of magnitude. Us-
ing a detector that covers a larger q-range, the improved statistics for the illuminating wave field should also result in a spatial resolution that is higher than that of the reconstructed object. The intensity of the side maxima is two or more orders of magnitude lower than that of the central peak [Fig. 4(b)]. Nevertheless, these low intensities are sufficient to accurately image areas of the object that lie outside the region scanned by the main focus, cf. Fig. 2(a).

Besides systematic drifts, we observed stochastic deviations in position mainly in horizontal direction that are visible in the fluorescence map [Fig. 2(c)] and that introduce slight artifacts in the reconstruction of the object [Fig. 2(a)]. Nevertheless, we did not observe that the positioning errors influence the reconstructed wave field. The convergence of the algorithm in view of the reconstructed wave field is very fast. The algorithm finds a quantitatively unique solution for the wave field, independently of the reconstructed region of the test object. This observation was verified by introducing random positioning errors in a numerical simulation.

While the wave field reconstruction is rather robust, that of the object function is much more sensitive to mechanical inaccuracies. In pursuit of high-resolution ptychographic microscopy mechanical stability will have to be improved in our future x-ray microscope. Smaller positioning errors might also be corrected computationally.  

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The test pattern consists of a 500 nm-thick nanostructured tantalum layer on a SiC-membrane (model ATN/XRESO-50HC by NTT-AT) and contains finest lines and spaces of 50 nm.

In this context, the difference map algorithm (Refs. 7 and 8) proves to be less robust compared to the sequential scheme (Ref. 9).

In this case, the reason that it does not appear as a square is related to linear drifts of the sample.