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Low-dose, high-resolution and high-efficiency ptychography at STXM beamline of SSRF

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Abstract. Ptychography is a diffraction-based X-ray microscopy method that can image extended samples quantitatively while remove the resolution limit imposed by image-forming optical elements. As a natural extension of scanning transmission X-ray microscopy (STXM) imaging method, we developed soft X-ray ptychographic coherent diffraction imaging (PCDI) method at the STXM endstation of BL08U beamline of Shanghai Synchrotron Radiation Facility. Compared to the traditional STXM imaging, the new PCDI method has resulted in significantly lower dose, higher resolution and higher efficiency imaging in our platform. In the demonstration experiments shown here, a spatial resolution of sub-10 nm was obtained for a gold nanowires sample, which is much better than the limit resolution 30 nm of the STXM method, while the radiation dose is only 1/12 of STXM.

1. Introduction

Since its first demonstration in 1999 [1], the coherent diffraction imaging (CDI) method has drawn extensive research interest within X-ray community [2, 3]. As a scanning version of CDI, Ptychographic CDI (PCDI, also called "ptychography") has the great potential to deliver diffraction-limited resolution imaging for extended samples[4, 5]. Similar to conventional CDI methods, the spatial resolution of ptychography is only limited by X-ray wavelength and the maximal scattering angle detectable. In this approach, a localized coherent illumination (also called "probe") scans across an extended sample by a small step size, ensuring that each scan position sufficiently overlaps with the adjacent ones. Therefore, structures in each illuminated area of the sample are expressed in multiple diffraction patterns. This redundancy makes it possible to retrieve both the probe and sample information simultaneously from the same dataset [5, 6]. Up to now, PCDI has been successfully applied in the study of a variety of samples in material science and biology with visible light [7], soft [8] and hard X-rays [5, 9], and electron beams [10].

The radiation damage is the key factor limiting the achievable resolution for biological samples in CDI. A PCDI platform with relatively low radiation dose, high resolution and high efficiency is developed at the scanning transmission X-ray microscopy (STXM) endstation of Shanghai Synchrotron Radiation Facility (SSRF). Compared to other FZP-based PCDI setups, we used a much larger illumination spot with 3-5 μm diameter by moving samples downstream from ZP focus, and a larger step size of 500-1000 nm in our platform, which results in fewer scanning positions and lower radiation dose when imaging the same sample area, though the larger light spot divergent from ZP focus will lead to a more difficult reconstruction due to the highly curved wavefront of the probe. By

combining the multimode reconstruction method [11] and up-sampling method [12] with the position-correction algorithm [13] in our home-developed ptychography reconstruction software, we can obtain high-quality images from our PCDI platform with much lower radiation dose and less data acquisition time. Several demonstration experiment results are shown below, in which a spatial resolution as high as 8.5 nm has been obtained for a gold nanowires sample, which is much better than that of the STXM imaging (30 nm at best), while the radiation dose is only 1/12 of STXM, and the data collection time is only about 1/3 of STXM. These results indicate a great application potential of our PCDI platform especially in life science.

2. Experimental setup

The soft X-ray PCDI platform was developed based on the STXM endstation of BL08U1A beamline at SSRF. This beamline has a photon energy ranging from 200 to 2000 eV produced by an undulator. The experimental setup of soft X-ray ptychography [8, 14] shares a number of similarities with STXM [15]. Therefore, by replacing the photomultiplier tube detector with an X-ray CCD detector and keeping the rest unchanged, the STXM setup can be easily adapted for ptychography.

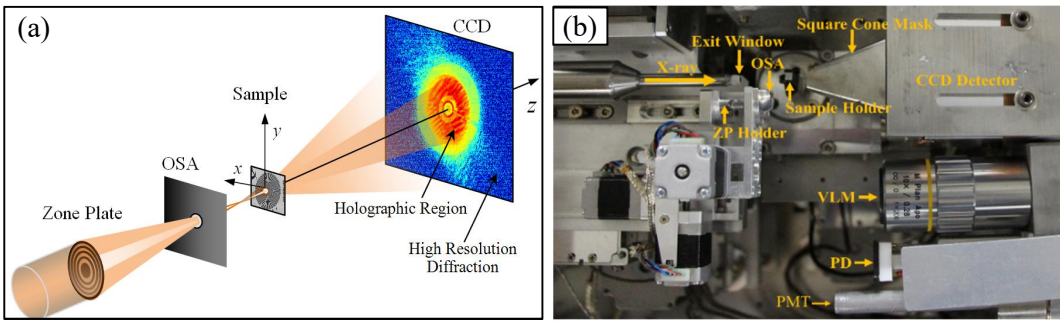

Figure 1. (a) A schematic diagram of the PCDI experiment setup based on STXM. The sample is moved downstream from the ZP focus, which increases the illumination spot to 3-5 um in diameter. (b) A photograph of the PCDI setup in the STXM chamber of SSRF. A square cone mask covers the CCD camera to reduce the noise on the diffraction signals.

As Figure 1 shows, A 200 μm diameter Fresnel zone plate (FZP) with 80 μm central stop and 30 nm outermost zone width is used to focus X-ray, and a 70 μm order sorting aperture is placed downstream of FZP to allow only the first order focused X-ray through. The sample (mounted on a 2D piezo stage) is placed 51 μm downstream of the ZP focus, resulting in an illumination spot of about 3 μm diameter on the sample for 700-710 eV energy (used for the experiments described below). The diffraction patterns are recorded by a 16-bit PI-MTE:2048B high vacuum compatible x-ray CCD detector, placed 70 mm downstream of the sample. A square cone mask covers the CCD front to prevent stray light from the laser interferometer. The CCD chip has a 2048×2048 imaging array with 13.5×13.5 μm^2 pixel size. A read-out time of 4.5 s/frame is needed in data acquisition.

3. Experimental results

We have imaged various samples using the PCDI method developed at the STXM beamline. To demonstrate the performance of the PCDI platform, we show two representative examples in this section: gold nanowires, and a bamboo tissue slice.

3.1. Gold nanowires

Gold nanowires with about 20 nm width placed on a Si_3N_4 window were imaged by a 3um illuminating spot with 704 eV photon energy. The sample was scanned with a step size of about 400 nm in a concentric circle grid of total 94 grid points. Each position was exposed for 200 ms. The dataset was reconstructed using 3 probe modes mixed-state reconstruction method, and the results are

shown in figure 2. An STXM image of the same sample with 20 nm scanning steps is also shown for comparison. According to a power spectral density (PSD) analysis, the image resolution reaches 8.5 nm, much higher than the STXM resolution of 32 nm.

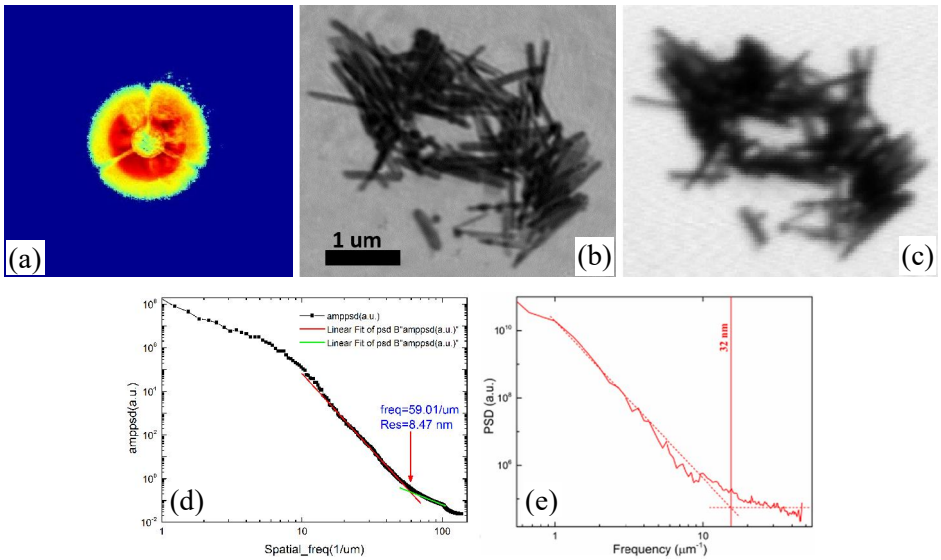

Figure 2. Ptychography imaging of Gold nanowires and its comparison with the STXM result. (a) is a typical diffraction pattern, the background noise of which was removed by subtraction of a dark CCD image and a thresholding process. (b) is the reconstructed amplitude image of the sample, (c) is the STXM image with 20 nm scanning steps. (d, e) are the PSD analyses of images (b, c), respectively, indicating a resolution of 8.5 nm for PCDI image and 32 nm for STXM image. The spatial resolution is determined by the critical frequency where the PSD curve significantly deviates from a power law decay into a random fluctuation regime.

Figure 2 shows a significant improvement of PCDI image over STXM image. This can be seen from both the image comparison of (b, c) and the PSD resolution analyses of (d, e). An overall performance comparison between PCDI and STXM is shown in table 1 based on this experiment. Table 1 shows that PCDI has great advantages over STXM not only in spatial resolution, but also in radiation dose and data collection efficiency. The radiation dose is proportional to the exposure time of the sample when the incident beam is stable. Therefore, we compared the radiation doses of PCDI and STXM by comparing the total exposure times in the two imaging processes, and found that PCDI only needs 1/12 exposure time (dose) of STXM to reach a resolution of about 10 nm which is 3-3.5 times better than that of STXM. In addition, due to much fewer scanning grid points in PCDI than in STXM, the experiment efficiency of PCDI is also higher than that of STXM. In the current configuration of our PCDI platform, the speed of PCDI data acquisition could be about 3 times that of STXM.

Table 1. The performance comparison between STXM and PCDI at STXM beamline of SSRF.

Performance	STXM	PCDI	STXM : PCDI
Exposure time (dose)	226 s	18.8 s	12:1
Data acquisition time	30 min	11 min	~3:1
Spatial resolution	>30 nm	8.5 nm	~3.5:1

3.2. Bamboo tissue slice

A frozen-dried bamboo tissue slice attached on a Si_3N_4 window was imaged using a 710 eV ZP-focused X-ray. The illuminating spot size on the sample was 3um in diameter, which scanned across

the sample with a step size of 500 nm in a concentric circle grid of total 121 points. The sample-CCD distance was about 70 mm. The exposure time was 800 ms for each position. The dataset was reconstructed using 3 modes mixed-state algorithm, and the results are shown in figure 3. According to a PSD analysis, the image resolution reaches 28.6 nm, which is significantly higher the usual STXM resolution for biological samples (no better than 50 nm in our beamline). In addition, the phase image shows a better contrast than the amplitude image, which is usually the case for biological samples with weak X-ray absorption.

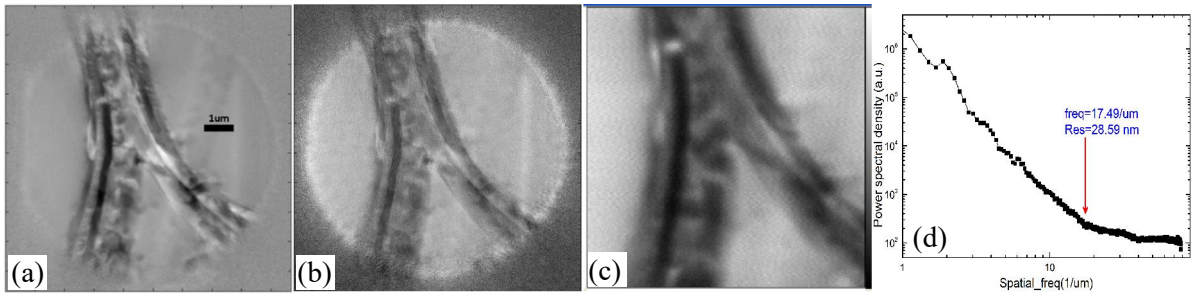

Figure 3. Ptychography imaging of Bamboo slice. (a) is the reconstructed phase image of the sample, (b) is the reconstructed amplitude, (c) is the STXM image with 30 nm scan steps. (d) is the PSD analysis of image (b), which indicates a resolution of 28.6 nm.

4. Summary and conclusion

We implemented a low-dose, high resolution and high efficiency ptychography platform at the STXM beamline of SSRF. A sub-10 nm resolution was obtained for material specimen by using relatively large illumination spot and large scanning steps and employing mixed-state reconstruction method combined with up-sampling and position-correction algorithms. Significant quality improvement was also observed for biological samples, which are especially sensitive to the radiation dose. A data acquisition rate of 3 times better than that of STXM was achieved, which can be further speeded up by using a faster CCD detector because at present over 90 percent of time is consumed by data read-out of CCD.

Acknowledgments

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