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Automatic Calibrations of Sample Misalignment for Nanotomography at SSRF

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Soft X-ray scanning transmission X-ray microscopy (STXM) at Shanghai Synchrotron Radiation Facility (SSRF) is capable of achieving high spatial resolution of 30 nm[1]. STXM has the ability to observe the two-dimensional nanoscale patterns, it cannot image the three-dimensional (3D) structure of the object, especially the cell samples. Combining the advantages of STXM and CT technology, nanotomography was proposed[2]. Three-dimensional fine structure of the sample with high spatial resolution is very important in many areas, e.g. materials and biology[3-4]. Nano-CT system based on STXM endstation has been developed. Once the turntable rotates, the sample will be misaligned seriously, which renders the sample deviating from focus. Only if relocating and refocusing the sample, the experiment can be continued, which will deteriorate experimental efficiency and waste precious experimental time, e.g. it takes about 15 hours to accomplish a nano-CT experiment with manual calibration of rotation axis. A new method has been developed to calibrate the sample misalignment automatically and accurately, which improves the experimental efficiency. With the method, there is no manual alignment and refocusing work for each angle, saving half of the whole experiment time.

The glass capillary is used as the testing sample, stretched to 5 micron diameter by a microelectrode puller. The new proposal includes recording the trajectory of the sample with the visible light microscope (VLM) in the first step, then analyzing and calculating the misalignment of the sample relative to the turntable, finally moving the new stage above the turntable along the x and z directions to make sure the sample is in the axis, accordingly, calibrating the position automatically.

The calibration requires testing the initial position of the sample relative to the turntable axis R and tangent angle α (Fig. 1a), then moving the sample to the axis. The process is as follows: firstly, moving the sample to the focus and recording the position (x_1,z_1), then rotating the sample 180°, and the sample needs to refocus due to large wobble, recording the position (x_2,z_2) again (Fig. 2a). The parameters R and α can be calculated by the following equation:

$$(2R)^2 = (x_2 - x_1)^2 + (z_2 - z_1)^2 , \quad (1)$$

$$\alpha = \arctan\left(\frac{x_2 - x_1}{z_2 - z_1}\right) . \quad (2)$$

Then, as shown in Fig. 1b, moving the turntable along the x axis of -(x_2-x_1)/2 to make sure the turntable's axis in the optical axis, rotating the sample with an angle α and the coordinates of the sample

are (x_3,z_3), shown in Fig. 1c. Finally, moving the new stage to $R(1-\cos\alpha)$ along the z direction, as can be seen in Fig. 1d, the sample is in focus and it is ready for nanotomography experiment.

In the experiment, calculating the values of automatic calibration, where R=356.34 μm, α=-0.9053 rad and the corresponding x and z offset were 280 μm and 136.32 μm. θ=0°, 20° and 40° were used as exemplary description in Fig. 2. The results of the images without automatic calibration using VLM are shown in Fig. 2 (the left column images). Without correction, the misalignment was very serious, e.g. θ=20°, the misalignment was 40 μm and 110 μm was misaligned while θ=40°, so the nano-CT experiment couldn't be continued efficiently. In comparison, automatic calibration images are shown in Fig. 2 (the right column images). Basically, sample alignment was eliminated and controlled within 1 μm, which fulfilled the experimental requirement of accuracy. The nano-CT experiment has been conducted with calibration, together with image pre-processing and tomography reconstruction, to produce the 3D structure image of the porous silicon ball is shown in Fig. 3.

By means of pre-measurement position calibration using optical microscopy, sample wobble during automatic nanotomography measurements can be minimized to 1 μm, which can enhance the experimental efficiency dramatically.

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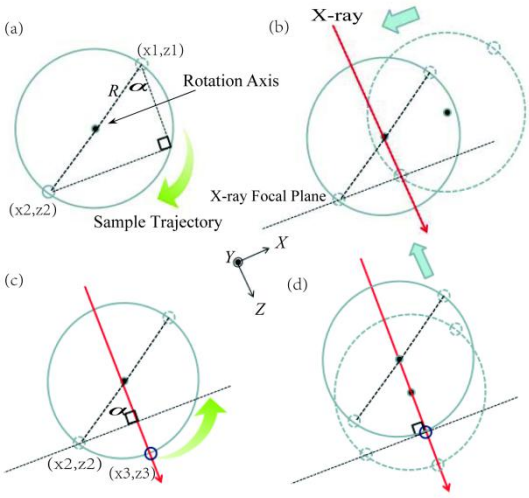

Fig. 1. Geometry for optical microscopy calibration of sample positions prior to nanotomography . (The clockwise direction is positive.)

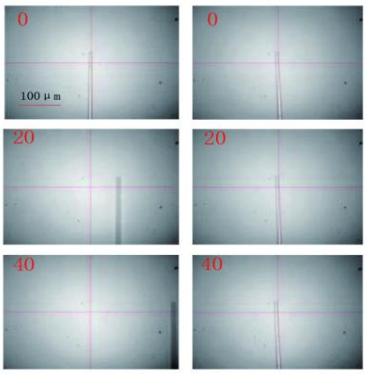

Fig. 2. With the sample rotating, VLM images for the CT scanning.

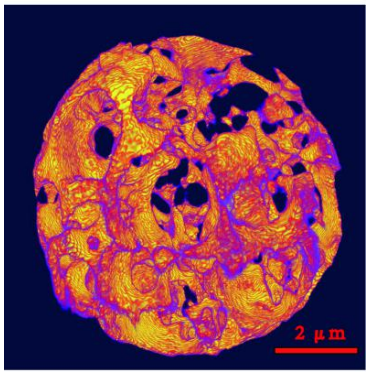

Fig. 3. The 3D structure of the porous silicon ball.