Investigation of Measurement Condition for 3-Dimensional Spectroscopy by Scanning Transmission X-ray Microscopy

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Investigation of Measurement Condition for 3-Dimensional Spectroscopy by Scanning Transmission X-ray Microscopy

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Abstract. A sample cell for performing computed tomography (CT) was developed. The 3-dimensional (3D) structure of polystyrene spheres was observed and the fluctuation of reconstructed linear absorption coefficients (LAC) was 9.3%. To improve the quality of data in 3D spectro-microscopy, required measurement condition is discussed.

1. Introduction

Combination of X-ray microscopy and computed tomography (CT) is one of the advanced techniques to be realized in forth-coming diffraction limit synchrotron-based light sources, considering the high transmittance of X-rays and the high spatial resolution of the X-ray microscopy. This combination allows us to analyze 3-dimentional (3D) structures without any destructive process of samples. Among several CT techniques, the full-field microscopy [1-5] may be more suitable methods than the scanning one from the viewpoint of data acquisition time. The scanning transmission X-ray microscope (STXM) in the soft X-ray region has been established as a powerful tool for the 2D chemical analysis based on near edge X-ray absorption fine structure (NEXAFS) and also been applied to 3D chemical state mappings [6-8]. In the soft X-ray region, the thickness of typical samples is around one micron; therefore, the spatial resolution of microscopy, a few tens nm, is suitable for performing CT. In most cases, tomographic data by using scanning type microscopy have been acquired under the condition of restricted angle projection due to the limitations of the measurement time and the shape and mounting of samples. In order to realize more quantitative analysis, the full projection (180° or 360°) measurement is highly required [9]. In this report, an experimental result of CT measurements is shown. The feasibility and the measurement condition to perform 3D spectroscopic measurements in STXM are also discussed.

2. Development of a sample cell for the CT measurement

A sample cell for the CT measurement is shown in Fig.1. This cell consists of a base plate, a small two phase stepper motor (AM-1020, Faulhaber) with a spur gearhead (12/5) and a sample clamp. Samples can be attached on a tip of a needle or in a glass capillary tube, and then be held by the sample clamp.

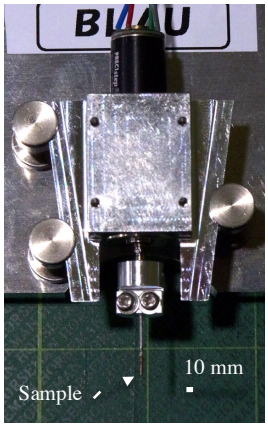

Fig. 1: The sample cell for CT

The stepper motor is driven by a programmable stepping driver (AD PM 00, Faulhaber) via a feedthrough. The controllable accuracy of the rotating angle is approximately 0.1°/full step. Design of the base plate is compatible with ALS-based STXM systems.

3. Experiments and results

3.1. STXM beamline, BL4U

The present measurement was performed at STXM beamline, BL4U, in UVSOR Synchrotron (Okazaki, Japan) [10]. In this experiment, a Fresnel zone plate (FZP) with a long focal length was used for the long working distance and focal depth. Parameters of the FZP is as follows; diameter of 240 μm, outermost zone width of 35 nm, diameter of a 0th order stop of 90 μm, pattern material of nickel on a silicon nitride membrane (100 nm thick) support. Diameter of an order select aperture (OSA) is 65 μm. In this experiment, the photon energy was 280 eV, below the carbon K-edge, and the focal length of the FZP is 1.90 mm. Size of a fixed exit slit used as a virtual source was set to 50×50 μm^2. In this geometry of the STXM, the working distance is ~400 μm, the spot size of the focused beam is 73×73 nm^2 and the calculated focal depth is 2.2 μm.

3.2. Sample and data acquisition

A cross sectional image of polystyrene spheres with diameter of 5 μm was obtained by CT. The spheres were fixed on a tip of tungsten needle with glue. In this experiment, the sample was rotated by 180° in total to get quantitative reconstructed values. 50 STXM images were totally acquired with rotating the sample by 3.6° each. The dwell time was 3 ms, the scanning step size was 150 nm, and the size of the image was 100×100 pixels. Due to wobbling of the sample position and refocusing, it took 2.5 hour to acquire all images. One of the STXM images is shown in Fig. 2. The rotational axis of the CT sample cell is along the vertical direction of the image.

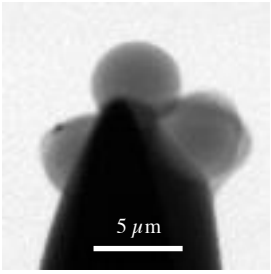

Fig. 2: STXM image of a sample

3.3. CT image reconstruction

The alignment of a series of 50 STXM images was done manually to extract sinograms. Cross sectional images were reconstructed by a home-made filtered-back projection algorithm with assuming the incident X-rays are parallel beams. One of the reconstructed cross sectional images (CT image) and a 3D volume image are shown in Fig. 3 (a) and (b), respectively. A profile along the radial direction of Fig. 3(a) is shown in Fig. 3(c). Judging from 10-90% of the edge of the profile, the spatial resolution is estimated as >300 nm. In Fig. 3(d), a histogram of the CT image is shown and a peak of the linear attenuation coefficient (LAC) at 0.173 μm^{-1} implies polystyrene (*cf.* 0.185 μm^{-1} at 280 eV [11]). An FWHM of that peak is 0.016 μm^{-1}, 9.3% of the peak LAC.

4. Discussion

In this experiment, structure of the sample is homogeneous so that the fluctuation of reconstructed LACs (i.e. the FWHM of the peak) will become an error in spectroscopy by CT. The fluctuation of LACs was 9.3% in this experiment and should be suppressed. There are several reasons for this fluctuation; (a) resolving power of the monochromator, (b) beam hardening, (c) photon flux, (d) degradation of the spatial resolution and (e) error by reconstruction algorithm, where (a), (b) and (c) can be negligible. The resolving power of the monochromator at BL4U is >3,000 in this configuration [10] so that the fluctuation of the LAC is less than 0.01%. The effect of beam hardening due to the higher order incident X-rays, such as bright edges on the CT image, is not clearly seen in Fig. 3(a) and 3(c) [12]. The photon flux onto one pixel was 3.5×10^4 [photons/dwell] and the fluctuation is estimated as 0.5%.

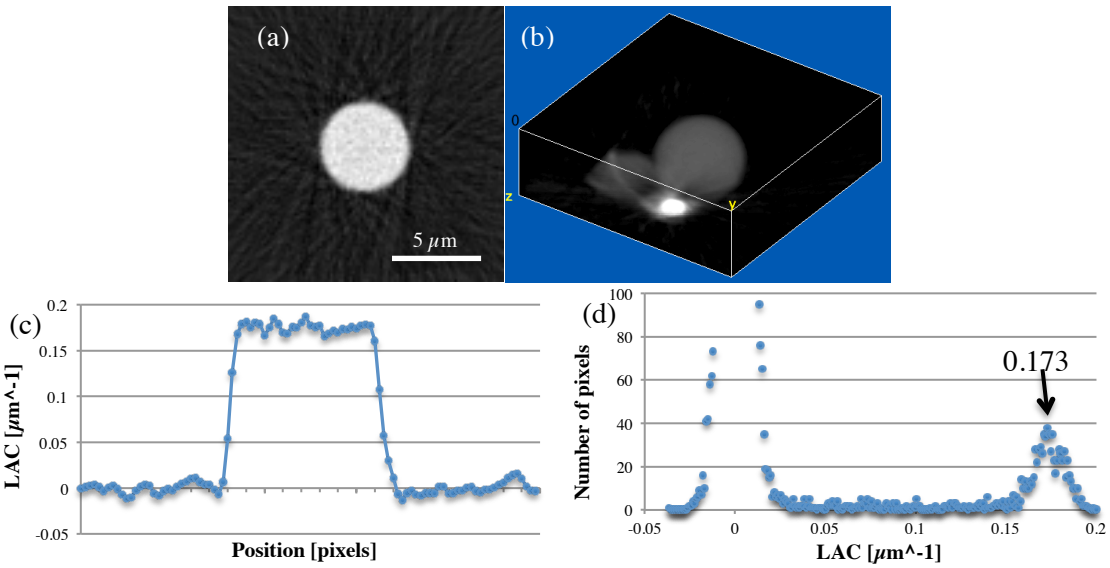

Fig. 3: Reconstructed images of the polystyrene spheres; (a) a cross sectional image and (b) 3-dimensional volume image. (c) A profile along radial direction and (d) a histogram of (a).

To estimate (d) error by the reconstruction algorithm and the required number of rotations, the reconstruction of a phantom with the same size as in the experiment, 100×100 pixels, is simulated. The distributions of LACs of the phantom is shown in Fig. 4(a). Sinograms are extracted from the phantom with 25, 50, 100, 150, 200 and 250 rotations per 180°, respectively. The phantoms are reconstructed and their FWHMs at the peak at 0.185 μm^{-1} are plotted in 4(c). In this plot, the FWHM saturates at 0.0021 μm^{-1} above 100 rotations and the fluctuation is 1.1%. Therefore, we can improve the fluctuation caused by reconstruction algorithm from 2.2% to 1.1% by increasing the number of rotation from 50 to 100.

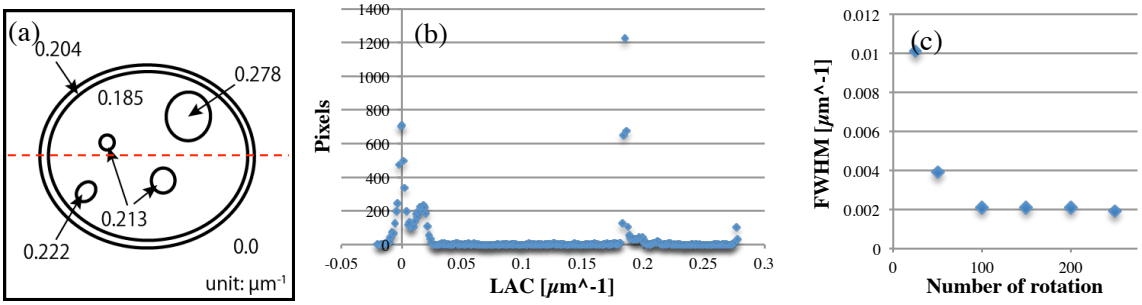

Fig. 4: (a) Distribution of LAC of the phantom and (c) a histogram of the reconstructed sinogram with 100 rotations. (c) The FWHMs of the peak at 0.185 μm^{-1} are plotted against the number of rotations.

In the experiment, the expected spatial resolution was around 150 nm, similar to the scanning step size, but >300 nm in fact. This degradation of the spatial resolution was caused by mechanical issues, such as insufficient focal depth of the FZP, instability of the sample and wobbling of a rotational axis of the stepper motor. Effect of the degraded resolution is simulated by reconstructing the phantom which is filtered by Gaussian with radius of 1 pixel. The histograms of the reconstructed normal and filtered phantoms are shown in Fig. 5(a) and the detail of the peaks around 0.185 μm^{-1} is shown in inset. The FWHM of the filtered phantom becomes slightly larger than the normal one, 0.0024 and 0.0020, respectively. Furthermore, the peak of the filtered phantom has a tail to the higher LAC and its peak shifts toward the higher LAC. Profiles of the reconstructed phantoms are shown in Fig. 5(b). The profiles are extracted from center of the phantom, shown in Fig. 4(a) as a dashed line. Even though

integrated intensities of normal and filtered phantoms are same, reconstructed LACs of the filtered one are slightly higher than normal one. This result is due to the smaller diameter of the profile of the filtered phantom in Fig. 5(b). Therefore, density of the reconstructed LACs of the filtered phantom becomes higher than the normal one.

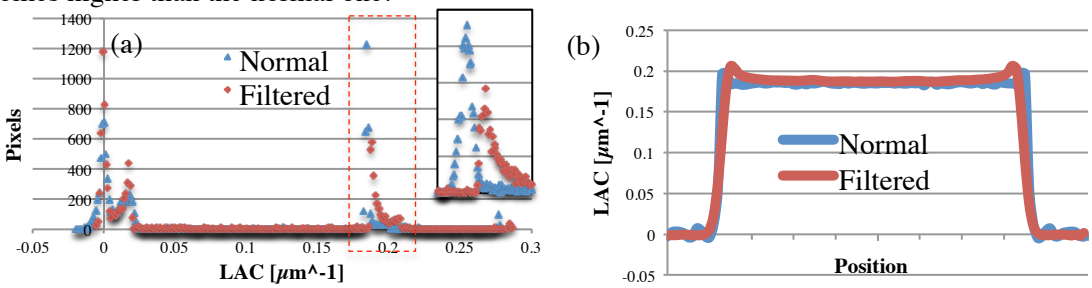

Fig. 5: (a) Histograms of the CT image of the normal and Gaussian filtered phantoms and inset shows detail of the peak around 0.185 μm^{-1}, (b) profiles of reconstructed images

5. Conclusion

The first trial of CT by STXM was performed and the fluctuation of the reconstructed LAC of 9.3% was obtained. This fluctuation should be suppressed to perform spectroscopic measurement by CT. Then, we investigated the measurement condition by simulation to obtain more reliable reconstructed LACs and 100 rotations per 180° is necessary for data acquisition. The spatial resolution of STXM is also effective to obtain reliable LACs so that the FZP with suitable parameters, spatial resolution and sufficient focal depth, has to be improved. For performing actual 3D spectro-microscopy, the new CT sample cell should be developed for high stability and low wobbling. That reduces the measurement time remarkably (e.g. 17.5 hour is estimated in current condition).

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