Development of Scanning-Imaging X-Ray Microscope for Quantitative Three-Dimensional Phase Contrast Microimaging

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Abstract. A novel x-ray microscope system has been developed for the purpose of quantitative and sensitive three-dimensional (3D) phase-contrast x-ray microimaging. The optical system is a hybrid that consists of a scanning microscope optics with a one-dimensional (1D) focusing (line-focusing) device and an imaging microscope optics with a 1D objective. These two optics are orthogonally arranged regarding their common optical axis. Each is used for forming each dimension of two-dimensional (2D) image. The same data acquisition process as that of the scanning microscope system enables quantitative and sensitive x-ray imaging such as phase contrast and absorption contrast. Because a 2D image is measured with only 1D translation scan, much shorter measurement time than that of conventional scanning optics has been realized. By combining a computed tomography (CT) technique, some 3D CT application examples are demonstrated

1. Introduction

Since phase contrast (PC) imaging in hard x-ray region shows much higher sensitivity than absorption contrast (AC) imaging especially for low-Z materials, various types of PC x-ray microscope has been developed in the last decade. In particular, many approaches implemented in imaging (full-field) x-ray microscope optics have been proposed. On the other hand, scanning x-ray microscopy has also been widely studied for differential phase contrast (DPC) imaging. One of the advantages of the imaging microscope optics is high-throughput 2D imaging. It makes easy to apply to 3D measurement by introducing tomographic method. However, they are not still at a practical level in quantitative PC 3D imaging because it is difficult to satisfy all requirements for the PC 3D imaging such as sensitivity, linearity of image contrast, and system stability. On the other hand, scanning optics satisfies all requirements above mentioned. However, 3D imaging application has not been in practical use yet because data acquisition requires a lot of time

To satisfy advantages of the both systems; high sensitivity and high contrast linearity of scanning optics and high throughput of imaging optics, a novel x-ray microimaging system called as scanning-imaging x-ray microscope (SIXM) has been developed (Fig. 1) [1]. The system consists of a scanning microscope optics with a 1D focusing (line-focusing) device and an imaging microscope optics with a 1D objective. These two optical systems are set normal to each other regarding the optical axis for obtaining the scanning microscope data at each sample height. Therefore, a translation scan of the sample along the focusing direction is only required to obtain a 2D image. DPC image and AC image are arbitrary obtained by image processing after data acquisition. Some application examples on 3D CT experiments will be presented.

2. Optical system and experiment

The experiment has been carried out at the beamline 20XU of SPring-8. As shown in Fig. 1, the experimental setup of SIXM-CT system consisted of a 1D scanning CT system in the horizontal direction and a 1D imaging optics in the vertical direction. The system was illuminated with full-coherent beam of 8 keV x-ray. A pair of 1D Fresnel zone plates (1D-FZPs) with the same parameters was used as the 1D focusing device and as the 1D objective. The 1D-FZPs were fabricated at NTT-AT. Tantalum zone pattern with 1 μm thickness is deposited on a silicon carbide (SiC) membrane with 2 μm thickness. Outermost zone width is 100 nm, and total width of the 1D-FZP pattern is 155 μm. Focal distance at 8 keV is 100 mm. In the experiment, these 1D-FZPs were set inclined by 60 deg. in order to increase the effective zone thickness. Under this condition, measured value of diffraction efficiency was 0.279 (theoretical value is 0.316) [1]. An indirect-sensing x-ray camera consisted of a visible-light conversion unit called "Beam Monitor" (BM AA40P, Hamamatsu Photonics, K. K.) and a frame-transfer type charge coupled device (CCD) camera (C9100-02, Hamamatsu Photonics, K. K.) was used as an image detector. The Beam Monitor consists of a powder scintillator P43 (Gd_2O_2S: Tb, 10 μm thickness), and a couple of relay lenses. Typical parameters of the detector are as follows; number of the pixel is 1000 × 1000, maximum frame rate for full frame is 30 Hz, analog-digital conversion rate is 14 bit, and effective pixel size is 11.4 μm /pixel. Typical distance between the object plane and the image plane was roughly ~7 m. Therefore, typical magnification factor of the x-ray imaging microscope optics was ~70. In the raw image data obtained with the image detector, horizontal and vertical intensity distributions represent the far-field image of focusing 1D-FZP and 1D magnified image of the object, respectively. The raw image sequential dataset is recorded as translating the line probe across the sample. After the data acquisition, a 2D image is reconstructed from the raw image dataset. The reconstruction theory is just same as that of the conventional scanning optics with multi pixel detector.[3] In the case of the SIXM system, the reconstruction is processed only in the horizontal direction because the vertical position of the raw image is simply representing the magnified image of the object. Since the phase gradient can be measured only in the scanning direction, only the horizontal DPC image is obtainable (vertical DPC is not obtainable). A PC image representing phase shift distribution is reconstructed with linear integration of a horizontal DPC image. Effective pixel size in the vertical direction is determined by the magnification factor of the imaging microscope optics whereas that in the horizontal direction is the same as the scan pitch. Such asymmetric pixel ratio is numerically compensated after image reconstruction. Figure 2 shows x-ray images of a tantalum test chart (NTT-AT) obtained by the SIXM system. Spatial resolution of the system was measured to be 110 nm in the horizontal direction and approximately 400 nm in the vertical direction. More details of the SIXM system are described elsewhere. [1,2]

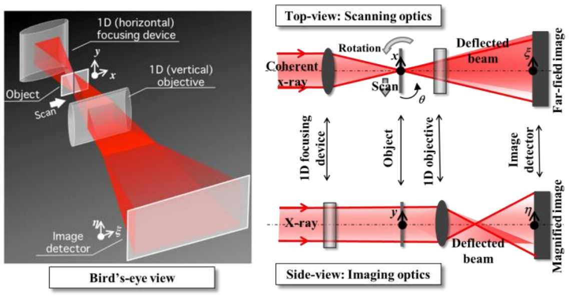

Figure 1. Conceptual drawing of SIXM optical system, left: bird's-eye view, upper-right: top-view, and lower-right: side-view.

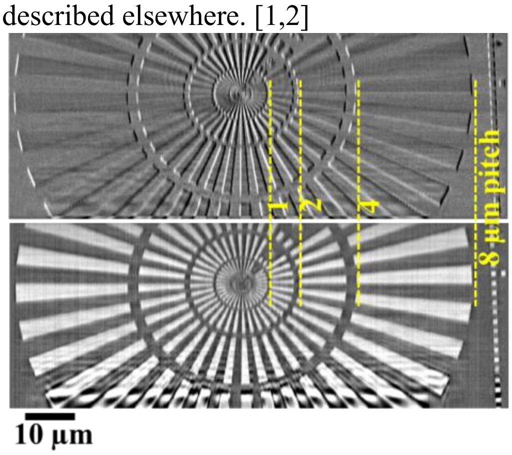

Figure 2. X-ray images of a tantalum test chart (NTT-AT), above: DPC image, and below: AC image. Pixel size 125 nm (H) x 162 nm (V), dwell time is 0.1 s, and measurement time is 80 sec.

3. 3D CT applications

3.1. Human hair

A human hair was observed as a feasibility test for the application of low-Z materials. Although internal structures of human hairs have been widely studied by using scanning electron microscope and so on, it has been impossible to observe the intact structure because samples must be cut out at the observed region and are required some preparation such as metal shadowing. Owing to use of the DPC scanning x-ray microscope, inner structures of intact human hairs was successfully observed,[3] and this method is now utilized to the developments for hair-care products. The scanning x-ray microscope, however, has never enabled to investigate 3D internal structure because of its low throughput as mentioned in chapter 1, although the 3D observation have been strongly required to understand the inner structures of human hairs completely. Although x-ray 3D observations by using several types of PC imaging microscope optics such as Zernike method, Talbot interferometer, and defocus were collaterally attempted, these attempts were hardly succeeded due to poor contrast linearity, low stability and low sensitivity of these systems.

Figure 3 shows CT images of human hair obtained with the SIXM-CT system. Measurement time was approximately 3.5 hours. Figures 3(a) and 3(b) show the virtual cross-sectional images in the transversal plane and in the sagittal plane of the sample, corresponding to the horizontal plane and the vertical plane of the system, respectively. Three-dimensional rendered image and its virtually cropped view are also shown in Figs. 3(c) and 3(d), respectively. In the transversally sliced CT image of Fig. 3(a), some defects and melanin indicated as small particles with lower and higher density than the neighbors, respectively, medulla at the center region of the hair shaft, and layered structure in the peripheral region of cuticles are clearly seen. In the sagittal cross section image of Fig. 3(b), shapes of defects and melanin are found to be extent along to the direction of hair shaft, while they are granular in the transversal direction. Therefore, the SIXM-CT system has distinctly revealed for the first time that their three-dimensional geometries are not globular shape, but prolate (rugby ball-like) shape whose long axes are along to the hair shaft.

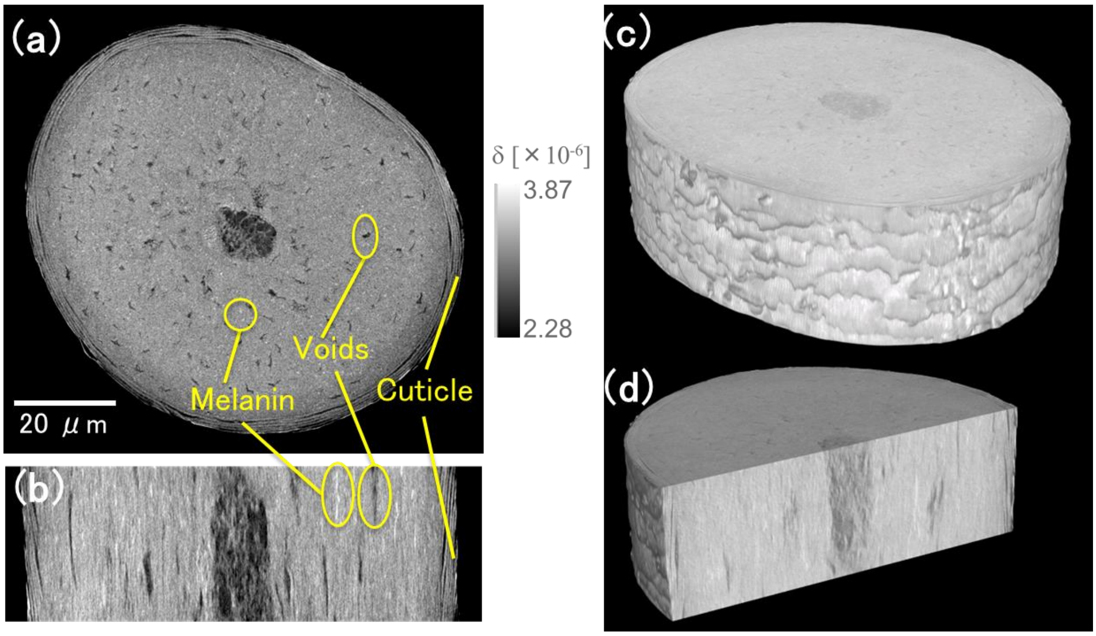

Figure 3. CT images of human hair; upper-left, horizontally-sliced CT image, lower-left: vertically-sliced CT image, and right: 3D rendered images. Scan pitch (H) is 125 nm, pixel size (V) is 323 nm, number of CT projection is 301 for 180 deg, dwell time is 20 ms, and measurement time is 210 min.

3.2. Carbonaceous chondrite (Allende meteorite)

Figure 4 shows CT images of a piece of Allende meteorite, which is classified into a carbonaceous chondrite, including organic materials, silicates and metals. PC-CT images and AC-CT images are shown in left and right half of Fig. 4, respectively. These two image contrast modes are derived from a single CT measurement data because image contrast modes can be arbitrary selected after data acquisition. Four times of CT scan were repeatedly performed with different vertical position of the sample because the field of view of the imaging microscope system (the vertical direction) was smaller than the vertical size of the sample. Measurement time for each CT scan was 2 hours. Shown images in Fig. 4 are the stitched ones of four CT image dataset in the vertical direction. Boundaries of stitched images are represented with arrowed lines in Fig. 4. Because the major component of the sample (silicates and metals) has a sufficiently high absorption for 8 keV x-ray, AC-CT image shows high image contrast as well as the PC-CT image. This method which enables 3D observation of PC and AC mode in one measurement shows feasibility of simultaneous investigation of high-Z such as metals and low-Z such as organic materials. Therefore, in the case of extraterrestrial sample, especially, the SIXM method will be one of the promising methods to search the beginning of life.

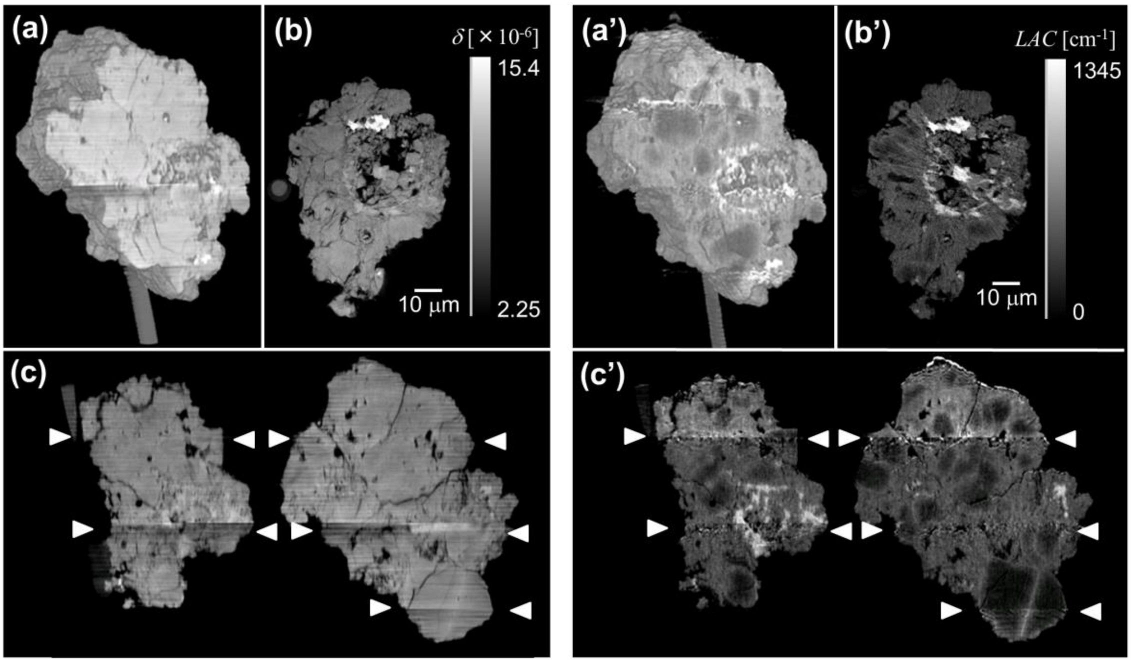

Figure 4. X-ray CT images of Allende meteorite, left: PC mode, and right: AC mode. (a) and (a') 3D rendering views, (b) and (b') virtual sectional images perpendicular to the rotation axis, (c) and (c') virtual sectional images parallel to the rotation axis. Horizontal scan pitch is 175 nm, vertical pixel size is 172 nm, number of CT projection is 501 for 180 deg, dwell time is 20 ms and measurement time is 120 min x 4.

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