BL20XU Medical and Imaging II

1. Introduction

BL20XU is a medium-length (250 m) beamline with an undulator source in SPring-8 dedicated to applications of various imaging techniques. A liquid nitrogen-cooled Si double-crystal monochromator (DCM) is used to choose the X-ray energy (7.67– 37.7 keV with Si(111), and 12.4-61.5 keV with Si(220) reflection). To transport a clean and coherent X-ray beam, no X-ray optical devices except the DCM and X-ray windows are installed. There are two experimental hutches. Experimental hutch 1 (EH1) and experimental hutch 2 (EH2) are located 80 m and 245 m from the source, respectively. Various kinds of X-ray projection imaging, such as X-ray micro computed tomography (µ-CT), high-speed X-ray imaging, and coherent X-ray imaging, are available. Using both EH1 and EH2, two experiments unique to BL20XU, which require a long sample-to-camera distance (165 m), are possible. The first is an ultrasmall-



Fig. 1. Schematic diagram of multiscale-CT at BL20XU for (a) high-energy X-ray nano-CT mode and (b) μ-CT mode.

angle X-ray scattering (USAXS). The second is a high-energy X-ray nano-tomography (nano-CT). A multiscale-CT, which combines the μ -CT and nano-CT (Fig. 1), and an integrated CT consisting of the multiscale-CT and XRD-CT were developed. As a part of activities at this beamline, improvements of the measurement apparatus and techniques for highenergy X-ray nano-CT were conducted in FY2019.

2. Upgrading of high-energy X-ray nano-CT by installing beam-shaping condenser zone plate

High-energy X-ray nano-CT at BL20XU is based on a full-field X-ray microscope optics using a Fresnel zone plate (FZP) as an objective and a condenser zone plate (CZP) as an illuminating optics. It realizes 150 nm spatial resolution threedimensional imaging at an X-ray energy range above 20 keV. This system, which is often used as a part of the multiscale-CT system that is combined with the μ -CT system (Fig. 1), is widely used in various fields such as astronomy, mineral, material, device, battery, and industrial use.

A typical scan time is approximately 60–120 min with 1800 projection at 20 keV. However, the scanning time is too long for time-resolved measurement such as *in situ* observations. The lack of intensity is particularly severe in the higherenergy region above 30 keV, as the efficiencies of the FZP and CZP decrease. Therefore, a higher intensity of illumination is required for shorter scan time measurements. A newly developed CZP, which is designed to increase the illumination intensity of illumination, was installed. A critical illumination is available using an FZP as a condenser (Fig. 2(a)). However, the field of view in this case is not sufficiently large because the focused beam size is too small with a highly collimated beam from the synchrotron radiation (SR) light source.

To overcome this, a hollow-cone illumination (Fig. 2(b)) is commonly employed in the SR fullfield X-ray microscope system. Such an illumination is realized by employing a CZP with multiple diffraction gratings with an equally spaced pitch (Fig. 2(b), left). Since diffracted beam from each segment overlaps at the object plane, the intensity of the illuminating beam at the object plane is proportional to the number of the segments of the CZP (in the case of incoherent illumination). Conventional CZP (octagonal CZP, O-CZP) consists of 8 segments (Fig. 2(b))^[1], whereas the newly developed CZP (beam-shaping CZP, BS-CZP) has 44 segments (Fig. 3(b)^[2]. Therefore, the newly developed CZP should have a 5.5 times higher illumination intensity than the conventional CZP.



Fig. 2. Conceptual drawing of illumination in full-field microscopy. (a) Critical illumination using FZP as the condenser, (b) hollow-cone illumination using O-CZP, and (c) illuminating with BS-CZP.



Fig. 3. Zernike phase-contrast X-ray image of the tantalum test chart (left) and its magnified view (right). X-ray energy, effective pixel size, and exposure time are 37.7 keV, 38.4 nm, and 6 s, respectively.

The performance test using the BS-CZP for twodimensional imaging was conducted using a tantalum test chart (XRESO-50HC; NTT Advanced Technology, Japan) with a fine structure of up to 50nm lines and 50-nm spaces with a 500-nm thickness. Figure 3 shows the Zernike phase-contrast X-ray image for an X-ray energy of 37.7 keV, where the left side shows an effective field of view of approximately 60 µm and the right side represents a magnified view showing that this optical system has a resolution as fine as 100-nm line width. The intensity of the image is approximately eight times larger than before employing the BS-CZP. The reason why the obtained gain is larger than estimated is due to the inner structure of the BS-CZP because the fabricated pattern depth is larger than that of the O-CZP. This results in a higher diffraction efficiency. Figure 4 depicts multiscale CT images of the Murchison meteorite, which is a type of carbonaceous chondrite. Figure 4(a) shows the µ-CT image. Nano-CT precisely measured the region of interest, which corresponded to the central region of the chondrule (Fig. 4(b)). The sample size was around 3 mm and the X-ray energy was 37.7 keV. A fibrous-like fine-grained crystalline structure

characteristic of a hydrous mineral was observed inside the meteorite.

Multiscale-CT measurements realize nondestructive three-dimensional imaging of bulky and mm-sized samples with nm-scale resolution. A higher X-ray energy is required as the sample size increases (Fig. 4). The BS-CZP increases the intensity by approximately an order of magnitude, which greatly improves the utility of nano-CTs, especially in the high energy X-ray region above 30 keV.



Fig. 4. Murchison meteorite imaged via multiscale-CT.
(a) Virtual cross-sectional image measured with the μ-CT mode. (b) Interior CT image of the circled ROI in (a) measured with the nano-CT mode. In the μ-CT mode, the X-ray energy, pixel size, and exposure time are 37.7 keV, 0.52 μm, and 50 ms, respectively, to acquire 1800 images in a 180° rotation. In the nano-CT mode, the X-ray energy, effective pixel size, and exposure time are 37.7 keV, 38.9 nm, and 2 s, respectively, to acquire 1800 images in a 180° rotation.

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References:

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