Development of diffraction enhanced imaging at beamline BL07 at the SAGA Light Source and its application

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Abstract. We have developed a diffraction enhanced imaging (DEI) system at beamline BL07 at the SAGA Light Source. BL07 is a beamline for using high energy X-rays up to 35 keV generated from a superconducting wiggler placed in the straight section of the storage ring. The DEI measurement system is composed of a Si(220) asymmetric crystal for expanding the beam, a Si(220) crystal analyser, and a high-resolution CCD camera. We demonstrated the observation of a rope recovered from the ruins of the Mietsu Navy in Japan.

1. Introduction

Traditional X-ray imaging is a powerful tool to observe the internal structure of materials, but is not sufficiently sensitive for materials that consist of light elements because their absorption effects are excessively weak and do not produce a clear contrast. Phase imaging, which uses the phase shift of X-rays in materials to produce an image contrast, is an innovative technique used to solve this problem. The cross section of a phase shift is about 1000 times larger than an absorption cross section, which enables us to observe the structure of soft materials such as bio-materials or polymers. Compared to several other phase imaging methods that use a crystal interferometer [1-3], propagation-based phase imaging[4], and X-ray Talbot interferometry [5, 6], diffraction enhanced imaging (DEI) has advantages such as a wide dynamic range of the material density and a simple experimental arrangement [7-9]. In the DEI method, refraction angles of X-rays penetrating materials are analyzed by a crystal analyzer.

The SAGA Light Source has started operation in 2006 with three beamlines. In the initial stage of our development, we had developed the DEI system at BL15, which uses a bending magnet as a light source. We used X-rays at energy of 14 keV, however, this X-ray energy was not sufficiently high to enable the application of DEI to dense or thick specimens. In the recent years, we have constructed an additional beamline, called BL07, with a superconducting wiggler as a light source. It can produce X-rays with the energies of up to 35 keV, making it suitable for use with the DEI method. Hence, we developed a DEI measurement system for BL07 to perform the structural investigations of soft materials.

2. Equipments

2.1. High-energy X-ray beamline BL07
BL07 uses high-energy X-rays from a newly developed superconducting wiggler [10, 11]. A schematic diagram of the beamline is shown in Fig. 1. The wiggler is installed in the straight section of the storage ring. The white beam from the wiggler is monochromatized by a double-crystal monochromator. X-rays with the energies from 5 to 35 keV are available in the beamline. The beamline is also equipped with a bend-cylindrical total reflection mirror for eliminating higher-order harmonics and focusing the X-ray beam. The focal point is selected as an arbitrary position in the experimental station by changing the mirror angle and bending.

2.2. DEI measurement system at BL07
A schematic diagram of the DEI measurement system developed at the rear hutch in BL07 is shown in Fig. 2. For DEI and other imaging experiments, the beamline focusing mirror is removed from the beam axis to illuminate a wide area of the sample. The photon flux is about $1.1 \times 10^8$ photons/sec/mm$^2$ at 20 keV, and $2.9 \times 10^7$ photons/sec/mm$^2$ at 30 keV. The X-ray beam is first diffracted by an asymmetric Si(220) crystal to expand the beam size in the vertical direction and reduce the angular divergence. A series of three Si(220) crystals with offset angles of 7.62 °, 6.08 °, and 5.06 ° are prepared for expanding crystals, which are designed to expand the vertical beam size typically by a factor of 10 for 20, 25, 30 keV, respectively. The appropriate crystal is selected for an X-ray energy between 20 and 30 keV. An area of 60 mm in the horizontal direction and 20 mm in the vertical direction can be illuminated. The sample is mounted on a rotating stage for computed tomography (CT) measurements. The X-ray refracted angle is analyzed by a Si(220) analyzer, which is placed next to the sample. The images are obtained by a CCD camera with a pixel size of 7.4 μm. An area of 36 mm × 24 mm (4872 × 3248 pixels) can be observed in a single snap shot.

3. Experiments and results
In this study, we demonstrated DEI observations of the internal structure of a rope recovered from the ruins of the Mietsu Navy in Sage, Japan (Fig. 3(a)). In mid 19th century, Kyushu and Yamaguchi regions had lead the achievements of the modernization and industrialization of Japan by their own techniques and knowledges. Mietsu was one of the most important cites in this region because steamships were produced and maintained in the shipyard. The Mietsu Navy is nominated as a
candidate of world heritage site with other cites in Kyushu and Yamaguchi by the World Heritage Committee (UNESCO). The rope is considered to be used to tie up a ship in the shipyard. The internal structure of the rope attracts great interest because it may give us information for deducing the industrial technology at that time. In this study, we performed DEI measurements to estimate the potential to investigate the structure of the rope.

The measurements were performed at an X-ray energy of 24.7 keV. For each DEI image, 11 snapshots were recorded by rotating the analyzer crystal in 2.9-μrad steps to determine the refracted angles that were proportional to the phase gradient. The pixel size of the CCD camera was tuned to be 14.8 × 14.8 μm by 2 × 2 hardware binning with an exposure of 100 sec. The rope was mounted on the stage supported by a plastic tube, as shown in Fig. 3(b). To prevent the rope from drying, the rope was kept in a polyethylene bag filled with water during all the measurements.

Phase gradient images of a part of rope are shown in Fig. 4. The fine structure of the rope is described in the images by the enhancement effect at the edge of the structure. In Fig. 4(a) and (b), bubbles, wrinkle of the polyethylene bag and surface adherent matters are clearly observed in the image. According to the shape of the bubbles, round-shaped and odd-shaped bubbles are seen. In addition to these artifacts, the fine structure of fibers in the rope is seen in Fig. 5. The edges of the fibers are less clear than other artifacts such as the bubbles because the density difference between the fiber and the water is smaller than that between the water and the air.

Figure 3. Pictures of the sample and the experimental setup, (a) the shape and the size of the rope, (b) the experimental arrangement. The white square in (b) roughly indicates the position of the illuminated area of the sample.

Figure 4. Projection DEI image of the rope. The sample rotation angle around the vertical axis was (a) 0 ° and (b) 90 °. The arrows indicate the round-shaped and odd-shaped bubbles. An expanded image in the square in figure (a) is shown in Fig. 5. The rope lies between the broken lines in each image.
For more information about the positions of the artifacts, we observed a series of images by rotating the sample around the vertical axis. Compared with Fig. 4(a) and (b), the round-shaped bubble was at a distance from the axis of rotation, which means that it was outside of the rope in the water in the polyethylene bag. Meanwhile, the odd-shaped bubble was located close to the axis inside the rope. This bubble is considered to be formed when the water infiltrated into the rope. The images of fibers are also distributed from the surface to the inside of the rope. Even then, it is extremely complex to analyze how the fiber is twisted from a single projection image.

Additionally, we attempted to reconstruct three-dimensional images of the rope by DEI CT. However, it is difficult to reconstruct CT images because the position of the artifacts in the images gradually moved during the image process because the rope is extremely soft and hence could not maintain its shape.

4. Summary
We have developed a DEI measurement system at the high-energy X-ray beamline BL07 at the SAGA Light Source. X-ray energies of up to 35 keV can be achieved with a superconducting wiggler placed in the straight section of the storage ring. The imaging system consists of a Si(220) asymmetric crystal and an analyzer crystal. With this equipment, we observed the internal structure of a rope discovered in the ruins of the Mietasu Navy at 19th century, in Saga, Japan. The structure of the fibers, which is hardly visible in the traditional transmission image, was clearly observable in the DEI image. To analyze the fiber structure in detail, a three-dimensional CT image is necessary. Further, we need to develop a sample holder to hold the rope in order that it remains steady and do not move during the measurements.

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