BL20XU
Medical and Imaging II

1. Introduction

BL20XU, which is the only medium-length (250 m) beamline with an undulator source in SPring-8, is designed for application to 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) reflection and 12.4–61.5 keV with Si(220) reflection]. In order 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) is located 80 m from the source, and experimental hutch 2 (EH2) is located 245 m from the source. Various types of X-ray projection imaging, such as X-ray microcomputed tomography (μ-CT), X-ray high-speed imaging, and coherent X-ray imaging, are available. By using both EH1 and EH2, two types of experiments unique to BL20XU, both of which require a long sample-to-camera distance (165 m) are available; one is ultrasmall-angle X-ray scattering (USAXS), and the other is high-energy X-ray nano-tomography (nano-CT). Multiscale CT measurement combining two or more CT systems with different fields of view (FOV) and spatial resolutions is available. Two types of systems are in operation. One is a combination of nano-CT and micro-CT, which enables observation of a sample of around 1 mm diameter with a spatial resolution of 200 nm. The other is based on using a large beam size at EH2 to realize a FOV of up to 6 mm and a spatial resolution of 1 μm. A combination of multiscale CT and X-ray diffraction CT (XRD-CT), called integrated CT, is also available. They are selected in accordance with the sample size and requirements of experiments. By using the system, it is possible to search important portions (region of interest, ROI) of a sample nondestructively.

Such portions are generally small in size relative to the original samples. Multiscale tomography enables us to observe such small objects in large samples without breaking them. However, in order to obtain information other than structure, such as elemental composition, it is still necessary to extract the ROI by processing samples and analyzing them on dedicated beamlines or instruments.

As part of the activities in this beamline, a system for high-precision sample processing in an atmosphere-shielded environment has been developed.

2. Development of sample processing methods in atmosphere shielded environment.

After determining the ROI using micro-CT, nano-CT, and XRD-CT, the following steps are required to expose the ROI: 1. rough cutting, 2. checking for location of cutting surface, 3. high-precision grinding, and 4. polishing of surface.

In the beginning of sample processing, a sample holder having a hole of the sample shape was prepared using a 3D printer with 3D shape data obtained from a CT scan in advance (Fig. 1). The created holder can hold irregularly shaped and fragile samples during cutting, and the cutting surface can be clarified in a microscopy image. As shown in the figure, the sample holder has slits that show the cut surface. The position of the slits was set manually during the operation of software for data preparation of the sample holder, installed in the data server of BL20XU.

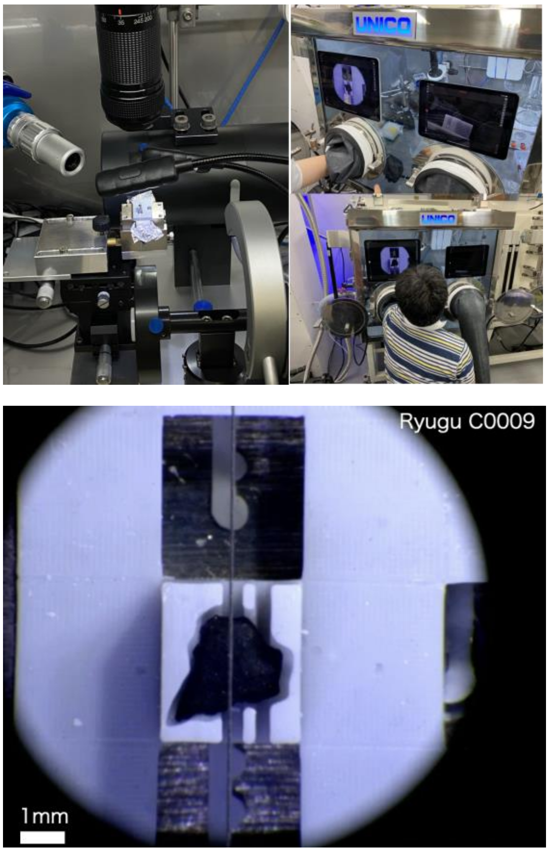

Fig. 1. Photographs of the wire saw in glovebox equipped at BL20XU. Cutting was carried out while viewing images from two cameras above and at the side of the sample, on a tablet screen. The sample was fixed to the stage without any adhesive materials by the holder with slits for cutting formed by a 3D printer.

Samples were separated with the wire saw along the slit placed at a designated position close to the ROI. Figures 1 and 2 show images for examples of sample cutting. In this case, samples from the asteroid Ryugu, obtained by the Hayabusa2 spacecraft, were set in sample holders with a cutting slit, and cut by a wire saw in a glovebox filled with purified nitrogen. The distance between the target surface and the actual cut surface was around 100 µm (Fig. 2).

The relative position of the target surface to the cut surface was determined by comparing the Backscattered Electron (BSE) image of the cut surface obtained by scanning electron microscopy (SEM), which was performed in the sample preparation room of BL20XU, with a CT image of the slice. Then, the piece that includes the ROI was fixed to the sample holder with glue and ground to expose the ROI, without lubricants. In the case of sample processing of Ryugu particles, the wire saw was also used for grinding.

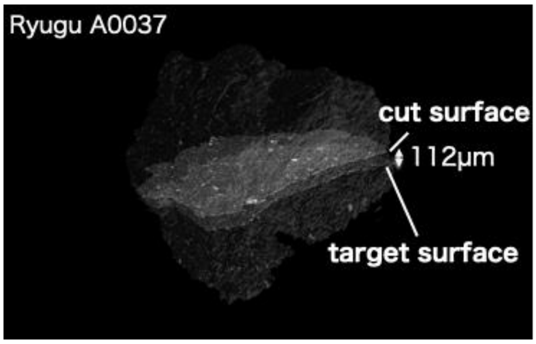

Fig. 2. Geometry of the cut surface and objective surface of Ryugu particle A0037.

Finally, the surface was polished with a cross-section polisher to obtain a mirror surface (Fig. 3). The BSE image shows that the polished surface was clear enough to see the texture. The polishing procedure was not yet atmosphere-shielded. However, it can be processed without any lubricants, as well as automatically without difficulty compared with polishing samples manually in a glovebox. The device will be improved into an atmosphere-shielded system in future work.

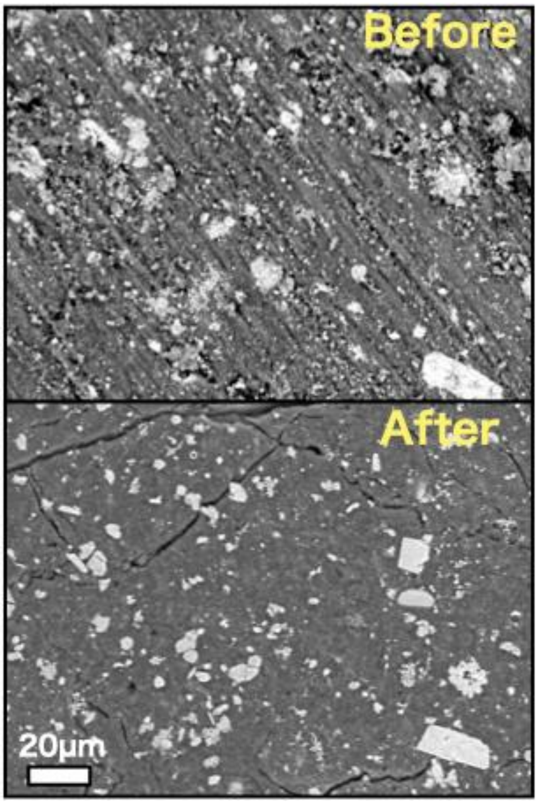

Fig. 3.BSE images for a surface of Ryugu particle A0002 before (upper) and after (lower) polishing with a cross-section polisher. The surface before polishing shows a number of scratches formed by the wire saw, making it difficult to observe its texture.

The processed sections with the surface of the ROI was provided to downstream analyses, such as field emission electron probe microanalysis (FE-EPMA), (nanoscale) secondary ion mass spectrometry (SIMS), nano-tomography with dual-energy tomography (DET) at BL47XU, and scanning fluorescence X-ray microscopy (SFXM) at BL37XU.

The equipment and methods developed in this work were applied in the analysis of Ryugu samples [1–4]. They can also be applied to the analysis of other anaerobic samples in other scientific fields, such as charged batteries and deliquescent materials.

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