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]. 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), which is located 80 m from the source, and experimental hutch 2 (EH2), which 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 experiment 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 measurements combining two or more CT systems with different fields of view (FOVs) and spatial resolutions are available. Two types of system are in operation. One is a combination of nano-CT and micro-CT, which enables the 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 that realizes 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 selective depending on the sample size and requirement of experiments. As part of activities in this beamline, extraterrestrial samples obtained by Hayabusa2 spacecraft, which would include the amount of hydrous minerals, organic materials, and volatile elements, have been observed using the integrated CT system.

2. Application of integrated CT system and CT-analytical environment to Hayabusa2 returned samples that require high cleanliness

Ryugu samples obtained by Hayabusa2 spacecraft larger than 0.1 mm were observed using the integrated CT system at BL20XU prior to further destructive analysis, in order to reduce the contamination and damage given by terrestrial materials and the unnecessary consumption of samples [1,2].

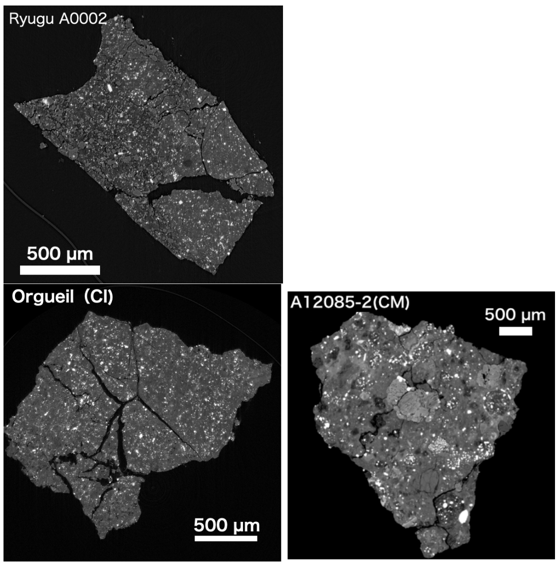

Fig. 1. Comparison of CT slice images of CM, CI chondrites, and Ryugu samples.

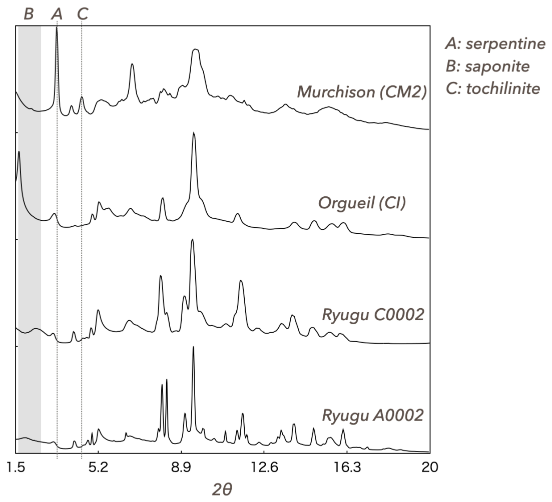

Fig. 2. XRD patterns of Ryugu particles and meteorites with 30 keV X-ray. Labeled peaks in the figure show characteristic peaks for CM and CI chondrites. Ryugu particles showed peaks of phyllosilicates (serpentine and saponite), similar to CI chondrites.

The integrated CT used in this measurement consisted of a wide-FOV CT, high-resolution CT, and XRD-CT, as described below. An X-ray detector with a 4K CMOS camera newly installed was applied in the wide-FOV CT. The pixel size and FOV of the detector are 0.85 μm and ~4 mm (4608 × 4608 pixels), respectively. Those for the high-resolution CT were 0.25 μm and 0.5 mm (2048 × 2048 pixels), respectively. For the XRD analysis, a wide-FOV detector with a pixel size of 19 μm and a FOV of 44 mm (2304 × 2304 pixels) was also newly installed and applied.

In addition to the integrated CT system, several types of equipment for the sample preparation, CT analysis, and sample processing developed in BL20XU, such as gloveboxes filled with circulated purified nitrogen, an atmosphere shielding holder, [3] and a wire saw, were also applied for the experiments. The wire size of the wire saw was 0.1 mm. The cutting procedure can be operated in the atmosphere-shielding environment and without any lubricants.

CT images and XRD patterns of Ryugu particles obtained using the integrated CT system showed features of phyllosilicates closely similar to those in CI chondrites, the most primitive class among meteorites (Figs. 1 and 2) [1,2]. XRD-CT images of Ryugu particles showed abundant dolomites, (Mg,Ca)-carbonates, similar to CI chondrites (Fig. 3).

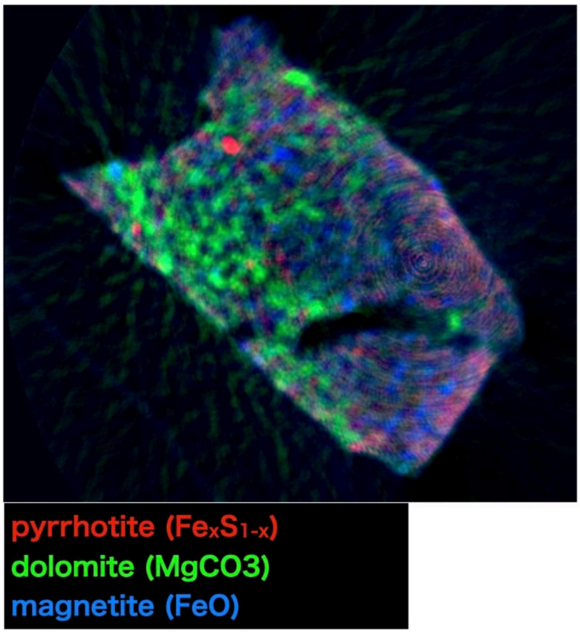

Fig. 3. XRD-CT image of A0002 Ryugu particle.

On the basis of the results of the CT analysis, regions of interest (ROIs) inside the samples were determined. Then, the samples were divided into pieces at near ROIs using the wire saw. To hold irregularly shaped and fragile samples during cutting, a sample holder for cutting was prepared using a 3D printer and CT data (Fig. 4). In this case, glues and adhesive materials could not be applied

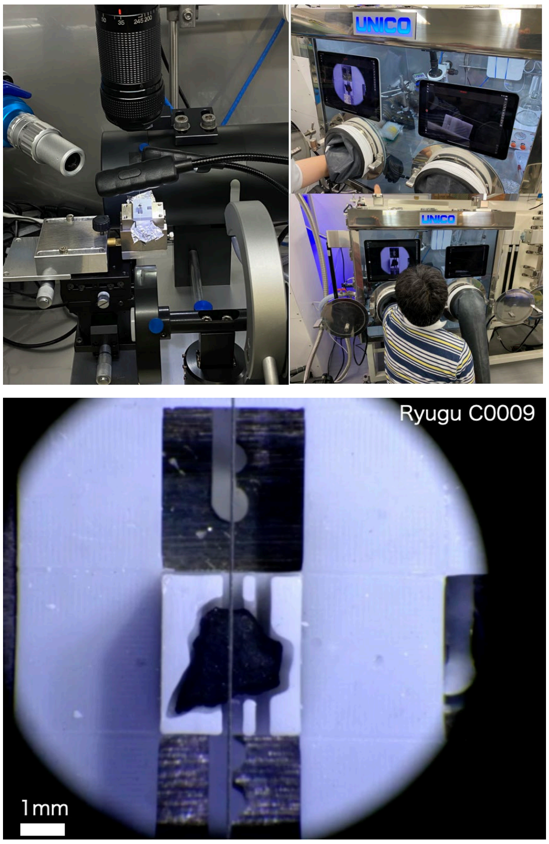

Fig. 4. Photographs of the wire saw in a glovebox equipped at BL20XU. Two cameras from the upper and the side portions of the sample with a tablet screen were installed. Without any adhesive materials, the sample was fixed to the stage by the holder with slits for cutting formed by a 3D printer.

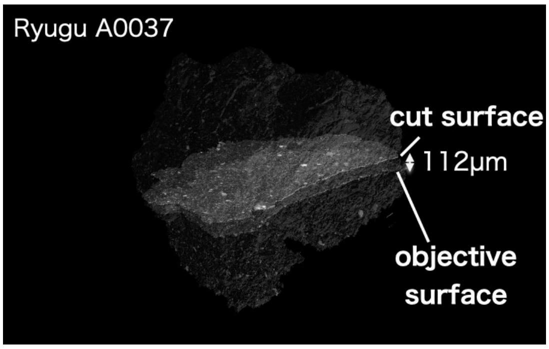

Fig. 5. Geometry of cut and objective surfaces for a Ryugu particle, A0037.

because they will cause serious contamination to the samples. The samples were divided with the wire saw along a slit in the holder, which was placed just above the ROI. The distance between the objective surface and the actual cut surface was around 100 μm (Fig. 5). After cutting, the piece that includes the ROI was provided for further detailed analysis using SEM, EPMA, SIMS, and TEM.

Other pieces were also provided for the analysis of bulk chemical composition using INAA and LaF mass spectrometry.

The equipment and methods developed for the analysis of Ryugu samples can be applied to the CT analysis of other anaerobic samples in other scientific fields, such as charged batteries and deliquescent materials.

Uesugi Masayuki and Takeuchi Akihisa
Japan Synchrotron Radiation Research Institute

References:

[1] Ito, M. et al. (2022). *Nature Astronomy*. **6**, 1163-1171.
[2] Nakamura, T. et al. (2022). *Science*. **379**, 787.
[3] Uesugi, M. et al. (2020). *Rev. Sci. Instrum*. **91,** 035107