PREPARATION METHODS OF POLISHED SECTIONS OF RETURNED SAMPLES FROM ASTEROID RYUGU BY THE HAYABUSA2 SPACECRAFT. D. Nakashima[1], Y. Fujioka[1], K. Katayama[1], T. Nakamura[1], T. Morita[1], M. Kikuiri[1], K. Amano[1], E. Kagawa[1], H. Yurimoto[2], T. Noguchi[3], H. Yabuta[4], H. Naraoka[5], R. Okazaki[5], K. Sakamoto[6], S. Watanabe[7], Y. Tsuda[6], and S. Tachibana[8], [1]Tohoku University, Sendai 980-8578, Japan (dnaka@tohoku.ac.jp), [2]Hokkaido University, Sapporo 060-0810, Japan, [3]Kyoto University, Kyoto 606-8502, Japan, [4]Hiroshima University, Higashi-Hiroshima 739-8526, Japan, [5]Kyushu University, Fukuoka 819-0395, Japan, [6]ISAS/JAXA, Sagamihara 252-5210, Japan, [7]Nagoya University, Nagoya 464-8601, Japan, [8]The university of Tokyo, Tokyo 113-0033, Japan.

Introduction: The Hayabusa2 spacecraft returned samples of ~ 5 g from C-type asteroid Ryugu. The "stone" team received 18 stone samples from the ISAS curation facility and conducted analyses [1]. After a series of non-destructive analyses, polished sections of the Ryugu samples are prepared for electron microscopy. Polished flat surface with target mineral phases and/or clasts being exposed is essential for maximizing research achievements of the precious samples. 39 polished sections from the 11 Ryugu samples have been prepared so far. Here we describe preparation methods of polished sections of the Ryugu samples, which benefits international public researches of the Ryugu samples and ongoing and incoming sample return missions such as OSIRIS-REx and MMX.

Preparation procedures of polished sections: Preparation procedures of polished sections of the Ryugu samples are similar to those of [2-4]. The samples are mounted in epoxy disks; 5.6 mm in diameter with ~ 1.5 mm thickness and 7.8 mm in diameter with ~ 2 mm thickness. A Ryugu sample is mounted on a bunch of carbon fibers or a glass tube using a superglue (Fig. 1a). The sample is dipped in a mixture of Epofix and ethanol under vacuum and cured at room temperature for more than 24 hours (Fig. 1b). The epoxy-coated sample is cut off from carbon fibers (or a glass tube) and mounted on a mesa with a 1 x 1 mm square top of an epoxy rod with a diameter of 5.6 mm using a superglue (Fig. 1c). The orientation of the sample for mounting is determined using a birds-eye view created using X-ray CT images obtained at BL20XU of SPring-8 so that the maximum surface areas of target mineral phases and/or clasts are exposed. Then, the mesa is covered with Epofix and cured under vacuum at room temperature for more than 24 hours. The epoxy cylinder containing the sample is cut using a wire saw with a diamond wire of 130 μm in diameter (Fig. 1d). The wire saw is equipped with a binocular microscope and micrometer eyepiece, which enable fine adjustment of cutting positions. The cutting position is determined as 50 - 100 μm away from the targeted cross section, of which position is marked on the birds-eye view of the sample. Two to four thin disks were made from each millimeter-sized sample. The cut surface is coated with ethanol-mixed Epofix under vacuum and cured at room temperature for more than 24 hours. The sample surface is ground and polished by hand using trizact diamond lapping film under dry condition and/or with ethanol. Thus, polished sections of the Ryugu samples are prepared (Fig. 1e). After carbon coating, 5.6 mm epoxy disks containing the Ryugu samples with polished surface are loaded in 7-hole disks for electron microscopy (3-hole disks for 7.8 mm epoxy disks; [4]).

In summary, preparation of polished sections of the Ryugu samples requires multiple steps, which were established through various tests using CI chondrites as analogue materials of the Ryugu samples and preparation of polished sections of the real Ryugu samples. Here we describe three key points for successful preparation of polished sections of the Ryugu samples.

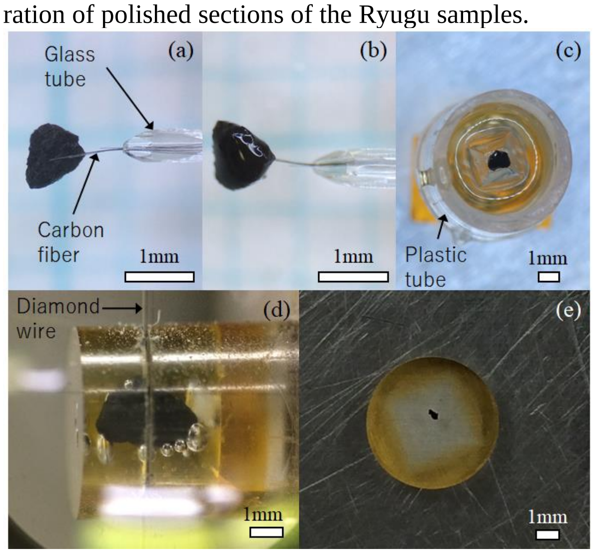

Fig. 1: Photomicrographs of an Orgueil meteorite sample before epoxy-coating (a) and after epoxy-coating (b), a photomicrograph of an Orgueil meteorite sample on an epoxy mesa (c), a photomicrograph of a Ryugu sample during cutting with a wire saw (d), and a photograph of a polished section of a Ryugu sample.

Ethanol-mixed epoxy: It was suggested from the remote-sensing data taken by the Hayabusa2 spacecraft that the surface material of asteroid Ryugu is similar to

CI chondrites [5]. Therefore, it is considered that the Ryugu samples are highly porous and fragile. Low viscosity epoxy resin is required for embedding the Ryugu samples, which permeates cracks on the sample surface and avoids break-up during cut and polish.

For reducing viscosity of epoxy resin, ethanol is mixed with Epofix with volume ratio of 1:1. Epofix is colorless and transparent and cures under vacuum at room temperature. After preparation of Epofix resin based on the supplier's manual, ethanol is added and stirred for 2 min. Choice of epoxy resin, the mixing ratio of ethanol and Epofix, stirring time, and cure time and were drawn from various tests, which will be reported in a separated paper.

Owing to the low viscosity, thin epoxy coating forms on the surface of the Ryugu samples, which enables to identify the shapes of the samples and easy handling with tweezers and needles (Fig. 1b).

Handling under dry condition: In general, water and/or ethanol are used as antifriction and coolant for cutting and polishing of the meteorite samples. However, use of liquids including acetone leads break-up of the Ryugu samples, which may be due to expansion of the Ryugu matrix with highly porous nature by absorption of liquids. Such a problem has never been observed for CI chondrite samples. Thus, it is important to handle the Ryugu samples under dry condition during cutting, polishing, and cleaning. Air duster and/or paint brush are used for cleaning.

Even if handling the Ryugu samples under dry condition, they absorb atmospheric moisture. For example, the polished surface of one Ryugu sample was broken when polishing with 0.5 μm trizact diamond lapping film (finishing polish) after leaving the polished section for ~ 4 hours in the room with ~ 70% humidity. Thereafter, the polished sections were dried at ~ 60 °C under vacuum for more than 12 hours and then repolished.

Use of X-ray CT images: The stone team observed the Ryugu samples with synchrotron radiation X-ray CT at BL20XU of SPring-8 [1], from which internal structures and birds-eye views are reproduced. These data are essential for exposing the maximum surface areas of target mineral phases and/or clasts. As described above, the birds-eye views are important for preparation of polished sections. The CT slice images are used as a distance indicator to the targeted cross sections.

Artificial zoning on the polished surface: Fig. 2a shows an optical microscope image of the polished section A0106-001a. The interior regions of the sample are dark and have scratches compared with the periphery and regions along the cracks. The dark/light structure reflects the difference in surface roughness, which is caused by difference in hardness. Since the dark/light feature on the polished surface was kept being observed during polishing for several μm in depth, there may be a difference in hardness between the sample surface and interior three-dimensionally.

The BSE image of the polished section with no carbon coating show the inverse zoning to the optical observation; the interior is lighter than the periphery and regions along the cracks (Fig. 2b). As shown in the EDS spectra (Figs. 2c-d), the periphery is more enriched in carbon than the interior, and therefore the zoning reflects the difference in carbon concentrations. Thus, the sample surface (and regions along the cracks) is harder and more enriched in carbon than the interior. In conjunction with the fact that zoning is not observed in the X-ray CT images taken before making the polished section, the zoning is artifact, and it is considered that ethanol-mixed epoxy permeated only into the periphery and regions along the cracks and reinforced them. Similar artificial zoning is observed in other polished sections of the Ryugu samples but not in those of CI chondrites. The unique artificial zoning reflects highly porous nature of the Ryugu samples.

References: [1] Nakamura T. et al. (2022) Science submitted. [2] Nakamura T. et al. (2008) Science 321,1664–1667. [3] Nakamura T. et al. (2011) Science 333, 1113-1116. [4] Nakashima D. et al. (2011) M&PS 46, 857–874. [5] Kitazato K. et al. (2019) Science 364, 272-275.

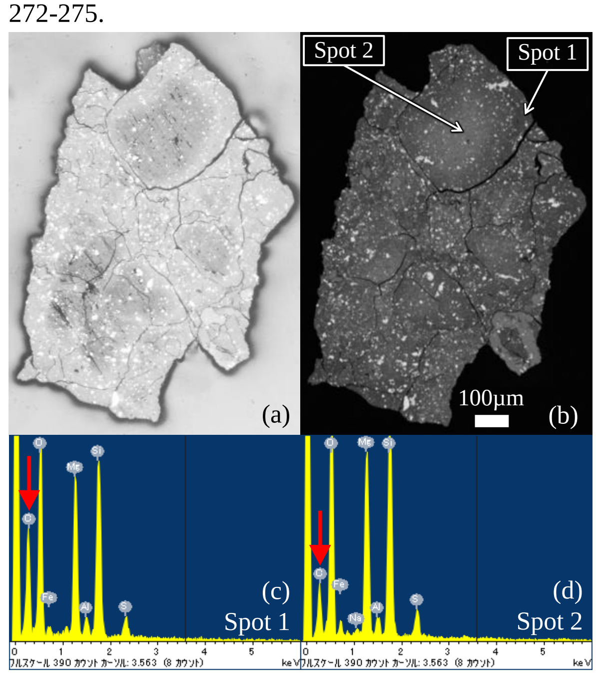

Fig. 2: An optical microscope image of A0106-001a (a), a BSE image of A0106-001a (b), EDS spectrum at the spot 1 (c), and EDS spectrum at the spot 2 (d). Carbon peaks in the EDS spectra are indicated by red arrows.