Terrestrial Alteration and Contamination on Previously Allocated Ryugu Samples

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Introduction

The samples returned from C-type asteroid 162173 Ryugu are handled in a pure nitrogen environment within the clean chamber (CC) at the Extraterrestrial Sample Curation Center in Sagamihara (ESCuC) to prevent alteration and contamination by Earth's atmosphere and materials [1]. The Ryugu particles are then allocated for initial analysis [e.g., 2, 3], Phase II curation [e.g., 4, 5], and Announcement of Opportunity (AO) research. During these studies, Ryugu samples are processed into various forms for analyses, such as polished sections (PTS), FIB sections, tiny particles attached to needles etc. These samples processing often involves handling with exposure to the atmosphere. Therefore, these Ryugu samples may have experienced alterations from their pristine state as that were stored in the CC. In this study, we investigated the previously allocated samples returned from research analysis to assess their terrestrial alteration and contamination. The previously allocated samples have been subject to reallocation to the AO research. Therefore, this study aimed to provide information that supports AO applicants in planning and executing their research projects, as well as to investigate methods for improving sample storage and preservation.

Samples and Methods

Previously allocated samples of eight particles (A0002-01, C0009-00, A0218-05, A0218-21, C0229-23, C0087-05, C0087-02, C0087-04) and two polished sections (C0014-02_PS, and C0087-06_PS) were selected for this investigation. After returned to the ESCuC, these samples are stored in a dedicated nitrogen-purged glove box (GB6). Exceptionally, the samples used in terrestrial weathering experiments [5] were stored in ambient condition in a clean room. In this study, the samples were classified into three types based on the handling history to assess the effects of atmospheric exposure: (1) non-atmospheric exposure, (2) short-term (less than a month), and (3) long-term (more than two years) atmospheric exposure. To investigate the effects of alteration in samples with different atmospheric exposure durations, we performed Fourier Transform Infrared Spectroscopic analyses using the IRT-5000 + FT/IR6100 (JASCO). Additionally, sample observations were conducted using a field-emission scanning electron microscope (FE-SEM, SU6600: Hitachi-High Tech.) equipped with energy-dispersive X-ray spectroscopy (EDS, Oxford AZtec Energy) to verify contamination caused by microparticles. All analyses were performed in a clean room. For the samples returned under non-atmospheric exposure conditions, all processes were completed without atmospheric exposure by using an airtight container during sample transfer. These results were compared with data obtained from pristine Ryugu particles (i.e., C0054 and A0308: outreach samples).

Results and discussion

Terrestrial Alteration of Previously allocated Ryugu particles

The μ-FTIR analysis was conducted on the several particle samples (Fig.1). This analysis revealed that all the previously allocated samples exhibit an asymmetric sharp absorption band centered at 2.7 μm with broad absorption of 2.8-3.2 μm, while pristine Ryugu particles (C0054 and A0308) have only a sharp absorption centered at 2.71 μm. Non-atmospheric exposure and short-term exposure samples show the same state in depth of the absorption band at 2.8 to 3.2 μm, and deeper absorption in long-term exposure samples. Generally, absorption around 3.0 μm is associated with water (i.e., H_2O) absorption from ambient air. This could be attributed to the increased adsorption of water on the sample due to prolonged atmospheric exposure. This study also presented that even the non-atmospheric exposure samples were affected by terrestrial atmosphere during sample handling, analysis, and/or transportation. In the observation by SEM-EDS, we confirmed the

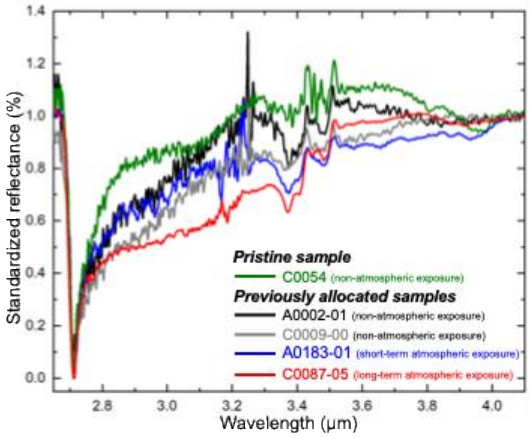

Fig. 1. Standardized FTIR spectra of selected Ryugu particles. All previously allocated Ryugu particles has broader absorption band than pristine particle.

precipitation of gypsum in long-term exposure samples (both particle and PTS) which were already reported in the same sample by [5] and this is considered as the result of terrestrial alteration by Earth's atmosphere (Fig. 2). No growth of gypsum was observed in these samples since the last observation in January 2023 [5]. On the other hand, gypsum was not observed in non-exposure and short-term exposure samples.

Contaminant microparticles on Previously allocated Ryugu samples

We often find C-rich microparticles in all particles and polished sections in the investigated previously allocated samples by SEM-EDS observation. Many of these microparticles are rich in carbon and nitrogen, which are considered to be organic matter. Organic molecules have been found in Ryugu particles, but the observed microparticles are attached on the sample surface and we have not observed them in that shape in pristine particles. Therefore, we interpret these C-rich microparticles were contaminated during the sample processing and/or analysis. Another type of C-rich microparticles were found in a particle after extraction of FIB sections. They are spherical shaped and rich in carbon and magnesium, and are likely the mixture of deposition liquid and sample material melted by irradiation of ion beam in the FIB sectioning process.

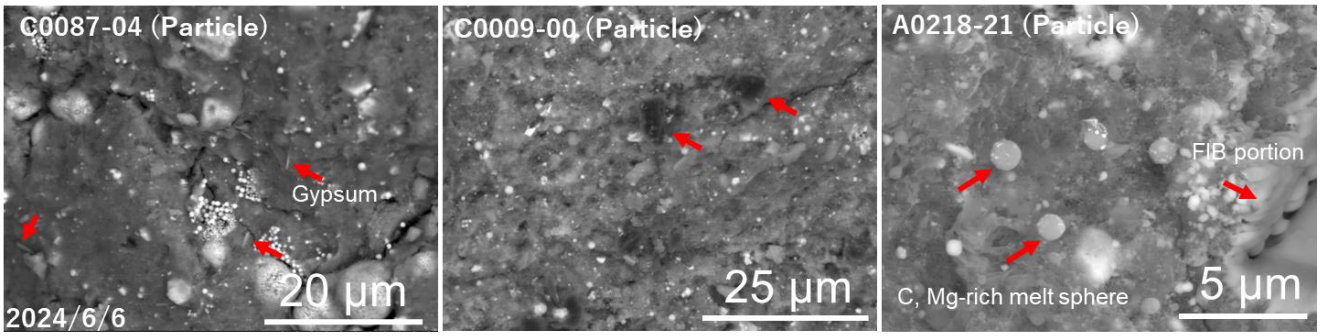

Fig. 2. BSE images of selected samples studied here. C0087 that used terrestrial weathering experiments contains lathy gypsum. The C-rich particles are present on the surface of the previously allocated samples (both particles and PTS). Additionally, C, Mg-rich particles, likely formed during the FIB process, are frequently observed near the FIB portion.

Summary

This study briefly evaluated the effects of terrestrial atmosphere on Ryugu particles using the previously allocated samples of different exposure time. We also found C-rich microparticles on the surface of the investigated samples as possible contaminants. As the previously allocated samples are available for reallocation to AO research, the results of this study are expected to be useful for researchers when selecting the adequate samples according to their research purpose. Especially, caution is required for considering spectroscopy and organic geochemistry for these samples. Further study is needed to understand the detailed effect of terrestrial atmosphere on samples returned in sample return missions, and more information is needed to assess the potential of contamination during the sample processing and analysis. These would contribute to improve the knowledge and techniques of astromaterials sample curation in ESCuC.

Acknowledgment

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References: [1] Yada. T. et al. Preliminary analysis of the Hayabusa2 samples returned from C-type asteroid Ryugu. *Nat. Astron.* **6**, 214-220 (2021) [2] Nakato. A. et al. Variations of the surface characteristics of Ryugu returned samples. *EPS.* **75**, 45 (2022) [3] Nakamura. T. et al. Formation and evolution of carbonaceous asteroid Ryugu: Direct evidence from returned samples. *Science.* **379**, 6634 (2022) [4] Ito. M. et al. A pristine record of outer Solar System materials from asteroid Ryugu's returned sample. *Nature.* **6**, 1163-1171 (2022) [5] Imae. N. et al., Mineralogical approach on laboratory weathering of uncontaminated Ryugu particles: Comparison with Orgueil and perspective for storage and analysis. *MAPS.* **59**, 7, 1705-1722 (2024).