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TECHNICAL REPORT

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Small grains from Ryugu: handling and analysis pipeline for infrared synchrotron microspectroscopy

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Abstract

Sample-return missions allow the study of materials collected directly from celestial bodies, unbiased by atmospheric entry effects and/or terrestrial alteration and contamination phenomena, using state-of-the-art techniques which are available only in a laboratory environment—but only if the collected material stays pristine. The scarcity of outer-space unaltered material recovered until now makes this material extremely precious for the potential scientific insight it can bring. To maximize the scientific output of current and future sample-return missions, the scientific community needs to plan for ways of storing, handling, and measuring this precious material while preserving their pristine state for as long as the 'invasiveness' of measurements allows. In July 2021, as part of the Hayabusa2 (JAXA) "Stone" preliminary examination team, we received several microscopic particles from the asteroid Ryugu, with the goal of performing IR hyper-spectral imaging and IR micro-tomography studies. Here, we describe the sample transfer, handling methods and analytical pipeline we implemented to study this very precious material while minimizing and surveilling their alteration history on Earth.

Keywords Sample-return mission, Hayabusa2, IR spectroscopy, Precious samples, Non-destructive analysis, Analytical pipeline

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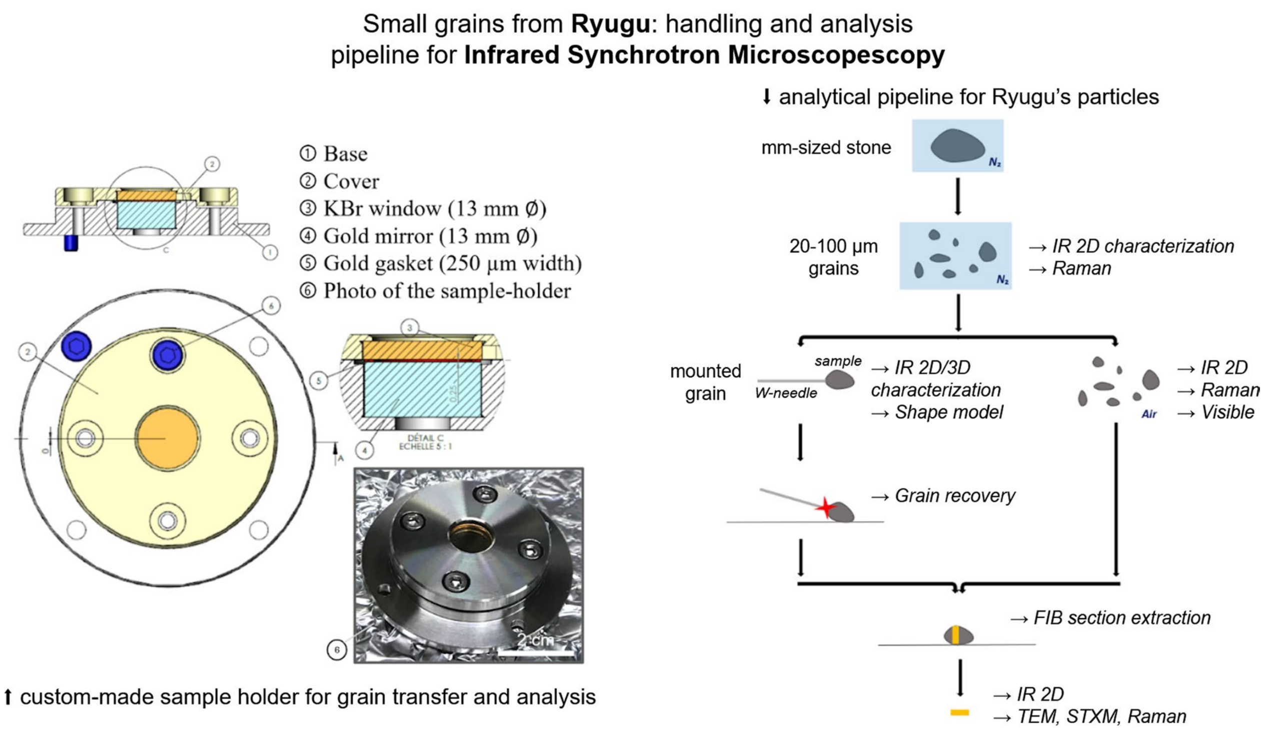
Graphical Abstract



Introduction

The study of materials from outer space is paramount in understanding how our solar system evolved from its beginning. In particular, the study of primitive bodies such as small asteroids, which didn't undergo differentiation processes and retained a composition similar to that of the original proto-planetary disk, is essential to investigate the youth of our solar system. Having access to the chemical composition and mineralogy of such bodies allows us to trace its evolution across time, from thermal alteration to space-weathering effects, and ultimately peer into its origins and how they relate to the early phases of our solar system. These studies can be performed by different means:

- from afar, using on-Earth telescopes or orbiting observatories (such as the recently launched James Webb Space Telescope) to study the surface composition of far-away bodies;
- using space-probes and sample-return mission, working with spacecrafts to map outer bodies and possibly collect materials from their surface;
- by proxy, conducting measurements while simulating space conditions on materials available on Earth, from terrestrial rocks—mineralogically homogenous but often contaminated by terrestrial organic matter—to samples more closely related to outer bodies, such as meteorites, micrometeorites, interplanetary dust particles (IDPs) or collected samples from sample-return missions.

The last item is of particular interest in the current context, since the scientific community all around the world has been preparing itself to welcome precious samples from C-type primitive asteroid (162173) Ryugu, collected by the Hayabusa2 spacecraft from JAXA (Watanabe et al. 2017). In December 2020, the Hayabusa2 mission brought back to Earth 5.4 g of matter from two different collect sites at the surface of Ryugu. In the near future, more samples collected from outer space bodies are expected to arrive on Earth, such as samples from another primitive asteroid, B-type (101955) Bennu, collected by the OSIRIS-REx mission from NASA (Lauretta et al. 2017). Studying these precious materials can be done with state-of-the-art techniques in laboratories on Earth. This can grant us unique opportunities to study materials which have not been affected by terrestrial alteration. These studies provide us with great

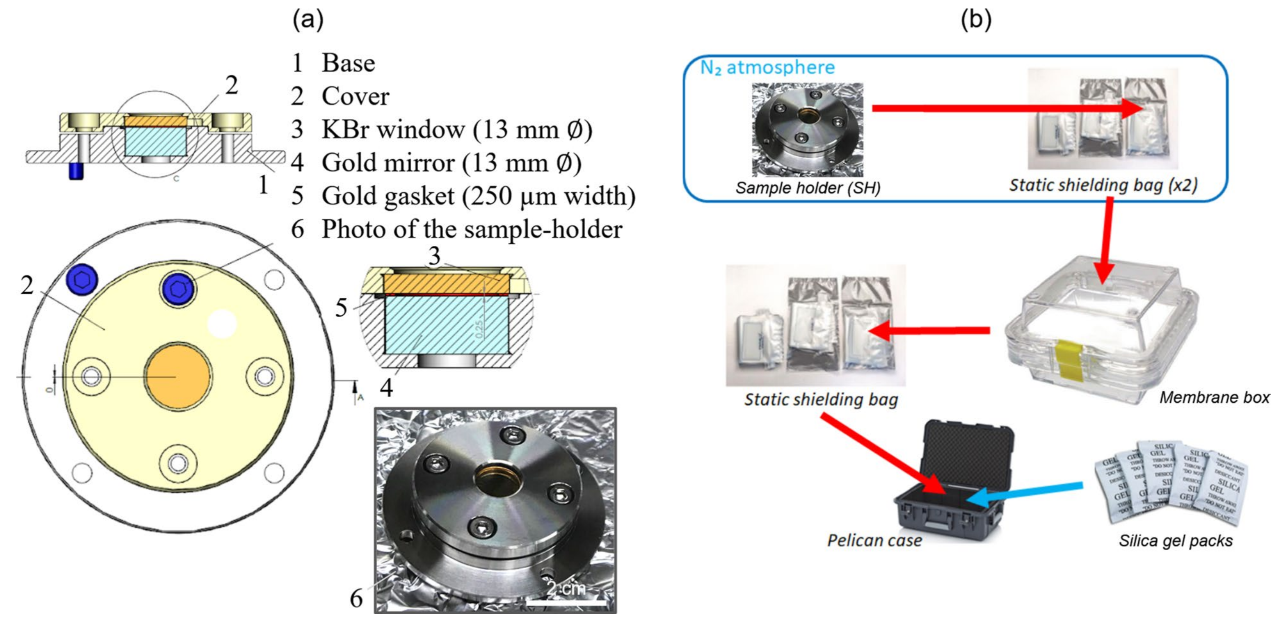


Fig. 1 **a** Schematics of our custom-made sample holder for the transfer of microscopic grains under N_2 atmosphere; **b** Sample transfer setup.

scientific insight on the early stages of our solar system that would not be obtainable otherwise (Vernazza et al. 2021). However, the scarcity of such materials calls for a certain degree of caution when transferring, handling and measuring these samples, to minimize sample loss and sample alteration post-arrival on Earth. Sample loss may occur due to manipulation, preparation or during destructive analyses. Exposure to the Earth atmosphere may also significantly alter the chemical composition of such delicate samples. Okazaki et al. (2017) show an example of terrestrial alteration processes on Itokawa particles from the Hayabusa mission, such as decomposition of amorphous materials. In the case of Ryugu, preliminary analysis of the returned samples has shown a lack of interstitial water (Yokoyama et al. 2022) and the presence of 23 amino-acid compounds (Nakamura et al. 2022), all interesting features which can be irreversibly altered by terrestrial alteration. It follows that there is a need to accurately plan for the arrival of such samples, to maximize the scientific output achievable before possible sample loss. Previous works have already contributed to the technical development of a systematic and coordinated analytical scheme for extraterrestrial materials (Aléon-Toppani et al. 2021; Ito et al. 2020; Shirai et al. 2020; Uesugi et al. 2020, 2014). In this work, we join this effort by describing the sample transfer methods and the analytical pipeline we applied to perform an extensive infrared characterization of several Ryugu particles in July 2021. Our analytical pipeline consisted in performing an extensive Infrared and Raman characterization of a number of isolated microscopic Ryugu particles prior to their exposure to the Earth's atmosphere, in an N_2 environment. Following this step, the N_2 seal was broken, and the samples underwent another extensive Infrared (in 2D and 3D) and Raman characterization in ambient air condition, before undergoing more destructive analysis, such as electron microscopy and FIB sectioning.

Sample preparation and sample transfer

On the 1st of June, four samples consisting of mm-sized stones—A0055, A0064, C0002 and C0046—arrived in Tohoku University at Sendai. All samples were stored in an N_2-filed glove box with oxygen concentration < ~5 ppm and dew points < − 50 °C. In this glove box, via a high-resolution optical microscope, approximately 30 particles were transferred by hand to our custom-made sample holders, using a very thin fiber and electrostatic force (no adhesive bond). Size range varied from 20 to 90 µm, with a particularly large particle of 150 µm. These samples had to travel from Tohoku University (Sendai, Japan), where the allocated particles were handled to the SOLEIL synchrotron in France while:

- under N_2 atmosphere, to avoid irreversible chemical alteration (example: Na sulfates growth on the particle surface (Nakamura et al. 2022, Okazaki et al. 2017);
- retaining their 3D structure during the trip for us to perform 3D IR characterization techniques;

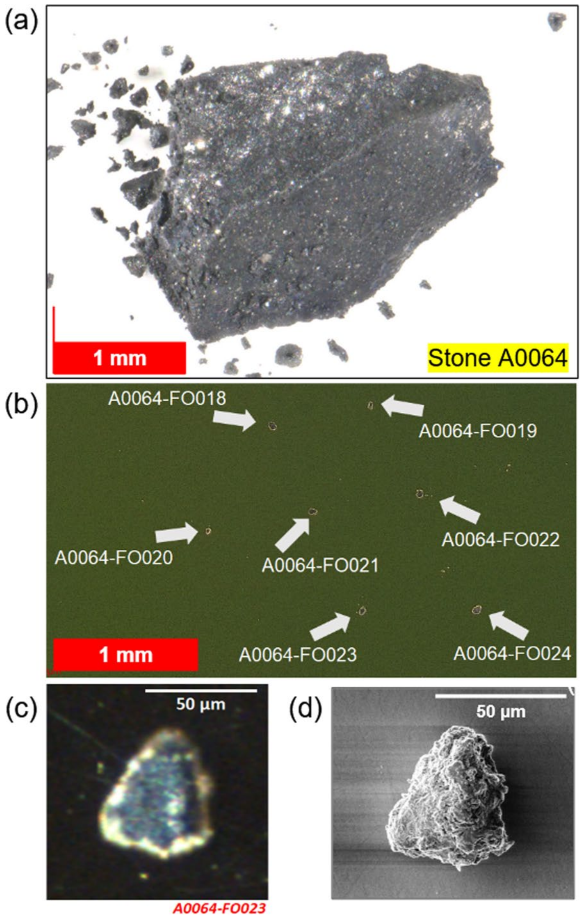


Fig. 2 **a** Ryugu mm-size grain A0064, **b** seven microscopic grains picked from A0064 stone, resting on the gold mirror of sample holder #3, **c** visible image of one of the picked grains, A0064-FO023, **d** SEM-SE image of A0064-FO023 (HV 2 kV—50 pA—ICE detector)

- remain measurable by IR reflectance spectroscopy while sealed inside the sample holder, under N_2 atmosphere.

These constraints led to the use of an appropriate sample holder, which would not only be used for transportation, but also for performing our preliminary infrared characterization while keeping the samples under N_2 atmosphere. No pre-made commercial solution was found adequate. Instead, we designed and assembled a custom-made sample holder.

The sample holder is made of five parts (see Fig. 1a): a base, a cover, a gold mirror, a gold gasket and a KBr window. The base and the cover are made from non-magnetic stainless steel. The gold mirror is inserted into the base and fixed using a lateral screw. Particles from Ruygu are deposited on this gold mirror for their transfer and preliminary analysis. The KBr window is glued to the cover using an epoxy glue (3 M™ Scotch-Weld™ *Epoxy* Adhesive *2216*, used for ultra-vacuum applications). The KBr window allows us to acquire measurements through the cover, keeping the sample holder seal intact. The gold gasket sits on the mirror and keeps the particles from being crushed. To some degree, it also acts as a seal, isolating the samples.To avoid contamination, the base and the cover underwent a heavy cleaning process: the pieces were first degreased, then were put in a bath of fluorocitric acid, followed by heated ultrasonic cleaning (80 °C max at 27 kHz) with an alkaline cleaning agent and finally a heated ultrasonic cleaning (80 °C max at 27 kHz) with deionized water. After the cleaning procedure, all the pieces were put inside cleanroom-ready static-shielding bags. The whole system was then assembled inside a glovebox under N_2 atmosphere ($P \sim 6$ mBar, $H_2O = 1.0$ ppm, $O_2 = 1.2$ ppm). Each sample holder was then put inside two cleanroom static-shielding bags, while still being inside the glovebox. This double protection would be able to keep the dry N_2 atmosphere sealed inside the bags. The bags holding the sample holders were then removed from the glovebox and each one was put inside a membrane box, to reduce the damage from shocks and vibration during sample transfer. These membrane boxes were also put inside larger cleanroom static-shielding bags, which were heat-sealed, to further isolate the sample holders. Finally, all sample holders traveled from France to Japan in a pelican case padded with foam and silica gel packs (Fig. 1b).

Once arrived in Sendai, the empty sample holders were opened in another glovebox, and were loaded with microscopical particles from Ryugu. Following a similar protocol to that of the outward journey, 32 particles were sent to France under a dry N_2 atmosphere, distributed among four sample holders (14 particles from the 1st touchdown-site (particles labeled starting with A) in SH2 and SH3, and 18 particles from the 2nd touchdown-site (particles labeled starting with C) in SH1 and SH4). To monitor the atmosphere's changes surrounding our sample holders, a few contingency measures were taken. During the whole trip, the sample holders were accompanied by a "control"-holder: this one would carry a few control samples that would easily react to changes in the atmosphere, such as iron dust (Iron powder, − 200 mesh, 99 + % (metals basis), Thermo Scientific™ Alfa Aesar 000737.A1, typical size of a few microns) to monitor oxidation processes. This "control"-holder was not exposed to the same environment as the Ryugu particles, to avoid contamination. A second "control"-holder, loaded with

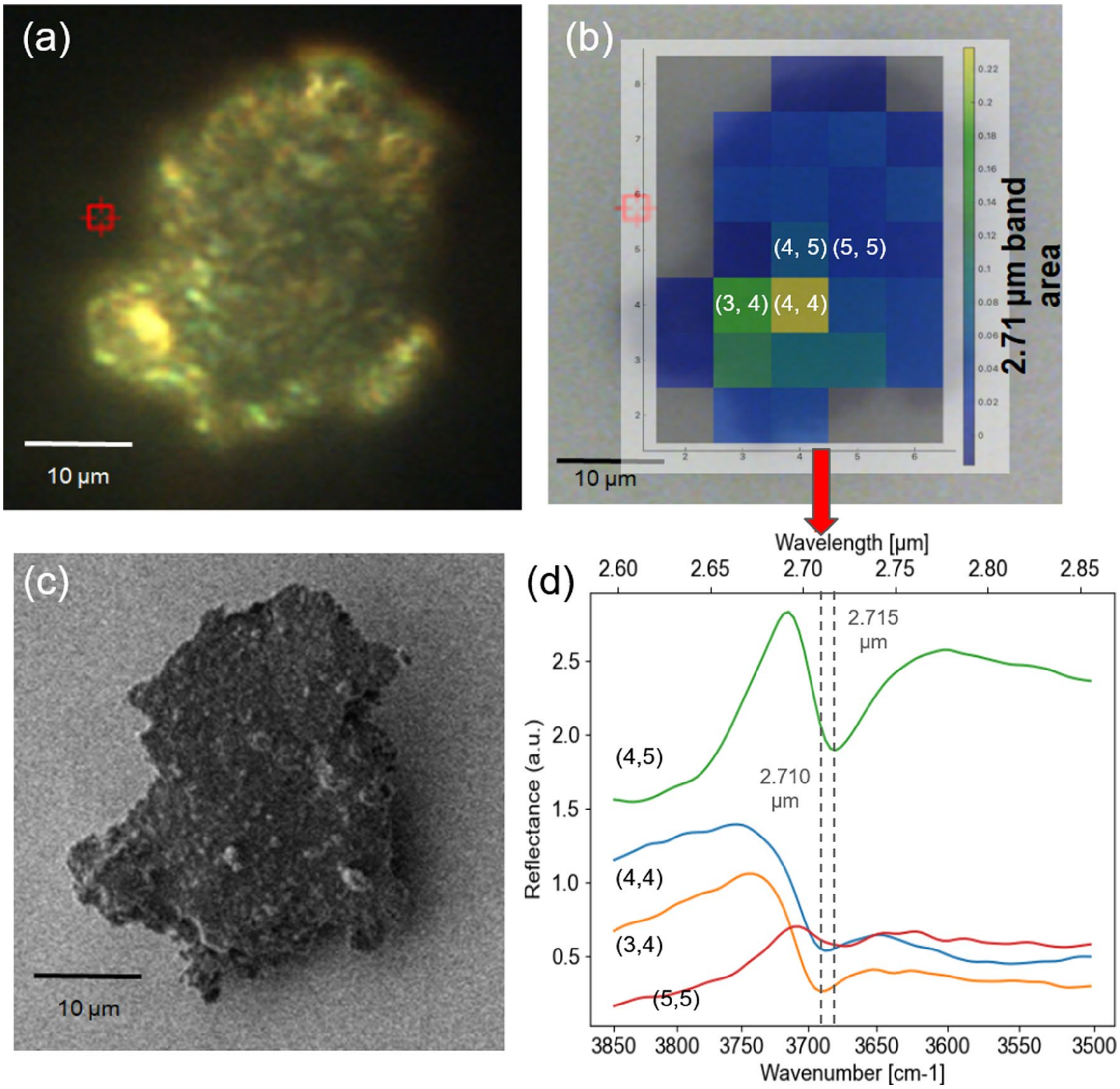


Fig. 3 SH3/A0064-FO020 in closed sample holder (through KBr window) **a** Optical image, **b** 2.7 µm feature (hydration feature) band area integrated on hyperspectral image from MCT/A detector with synchrotron source, overlaid with an optical image of the grain for co-localization purposes, **c** SEM-SE image of the grain taken after having opened the sample holder, **d** spectra centered on the 2.7 µm hydration feature of the selected grain, acquired through the KBr window with a Continuum microscope with a FTIR spectrometer equipped with an MCT/B detector, synchrotron-radiation-fed

drierite (>98% $CaSO_4$ and <2% of $CoCl_2$, from Fisher Scientific,) and some Fe dust, was kept in France, to monitor hydration and oxidation when not under dry N_2 conditions.

Sample reception and characterization

Measurements inside the sample holder (N_2 atmosphere)

The samples arrived at SOLEIL (France) in early July. The sample holders were first inspected using an optical microscope through the KBr window (Fig. 2).

We successfully identified 28 out of 32 of the original Ryugu particles by their morphological correspondence with the grains prepared at Tohoku University and their characteristic spectral feature at 2.7 µm, typical of Ryugu (see Fig. 3). The remaining 4 samples moved during transportation and were not found on the gold mirror.

Once the samples were identified, the analytical pipeline began with a full spectral characterization using IR synchrotron beam, from 1 to 20 µm, while keeping the sample holder sealed to minimize exposure to air. The goal of this first step in our analytical pipeline was threefold:

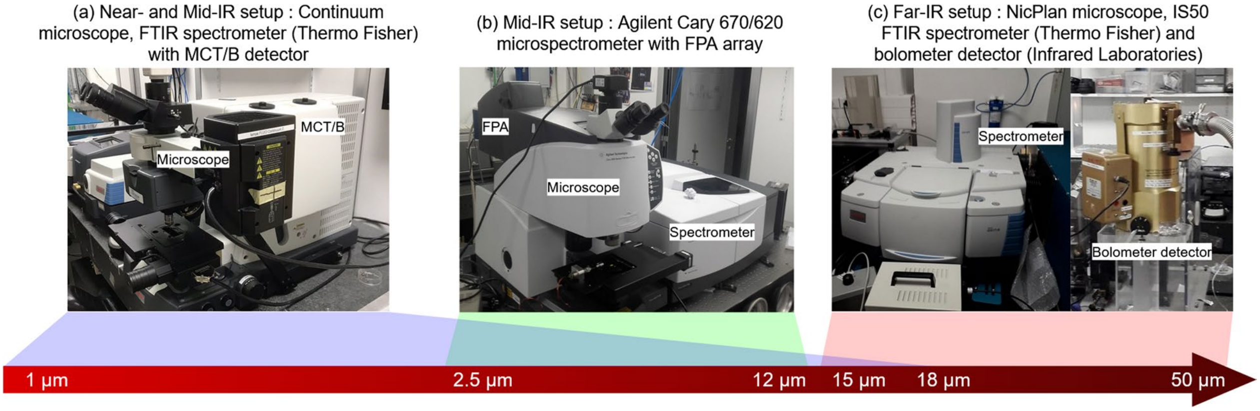


Fig. 4 The different systems used and their respective spectral ranges

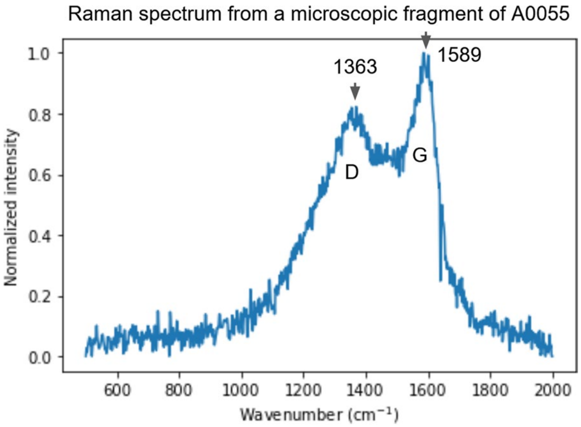


Fig. 5 Raman spectrum of a fragment detached from a microscopic grain originating from stone A0055

- Identify spectral features of interest in each Ryugu grain and derive their spectral parameters (for instance, position and depth of the (M)-OH stretching feature around 2.7 µm, position of the Si–O stretching silicate feature around 10 µm, presence/absence of the carbonate feature around 7 µm).
- Select grains to be mounted on needles for 3D IR characterization.
- Have a track record of the spectral signature of the grains prior to the opening of the sample holder, to follow possible grain alteration by terrestrial processes.

Our sample holder design allowed us to easily acquire measurements using all the available IR microscopes available at the SMIS-beamline (SOLEIL Synchrotron)

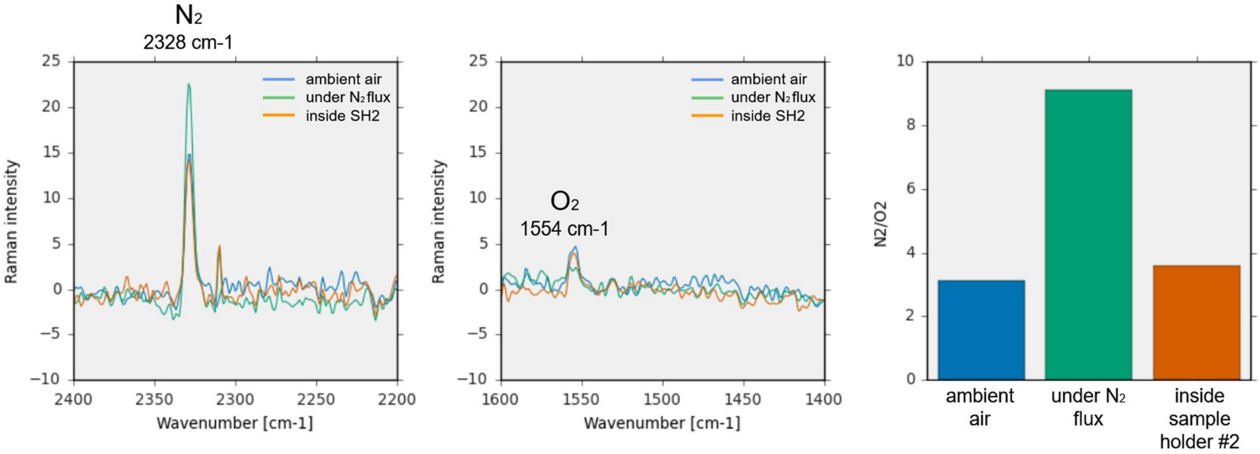


Fig. 6 Raman spectra on SH2, detecting presence of both N_2 and O_2 inside, with an N_2/O_2 ratio similar to that of ambient atmosphere

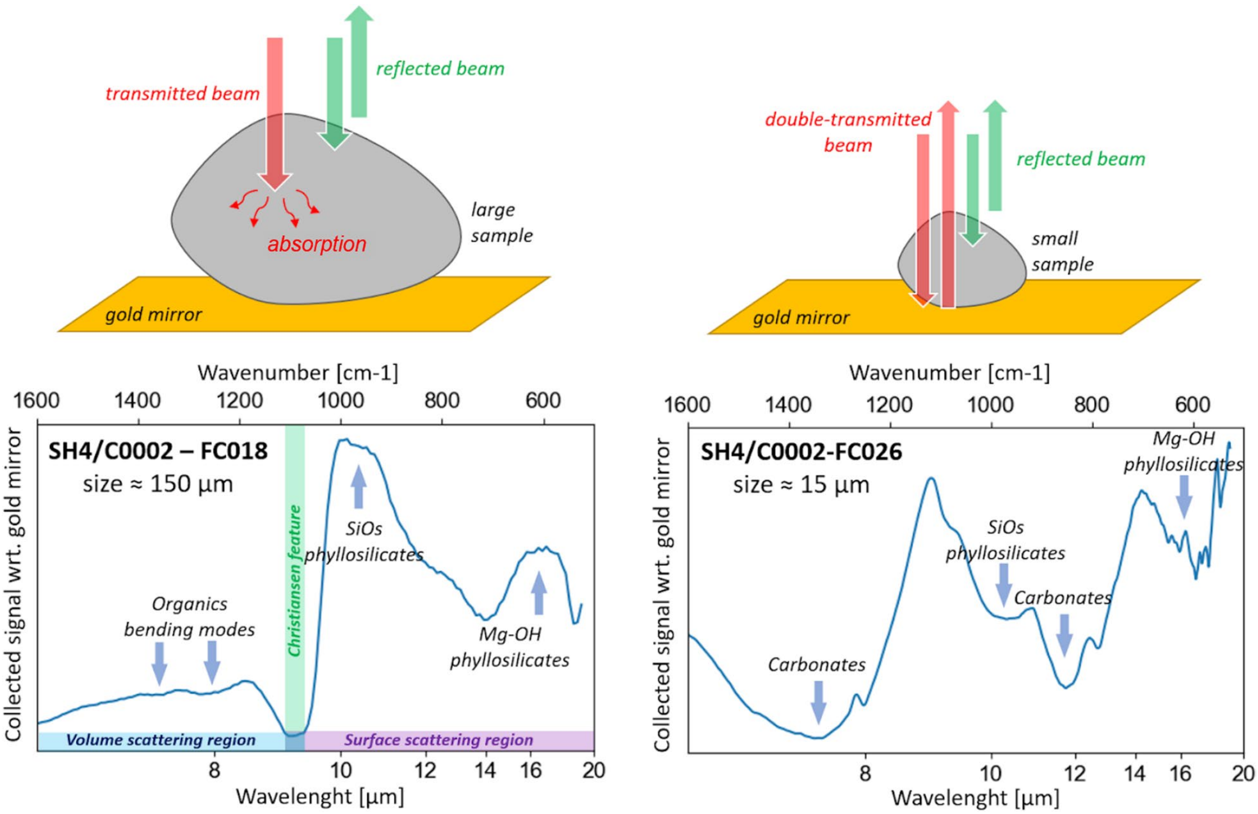


Fig. 7 [Left panel] cartoon modeling large grain behavior for infrared measurements on gold; in the bottom panel, the collected signal with respect to a gold background from C0002-FC018, the largest grain in our set (size of approximately 150 µm). The collected signal behaves as expected from a standard reflectance measurement (spectral bands point downward before the Christiansen feature and upward after). [right panel] cartoon modeling large grain behavior for infrared measurements on gold, highlighting the contribution of a double-transmitted beam going through the sample and shining back toward the detector; in the bottom panel, the collected signal with respect to a gold background from C0002-FC026, one the smaller grains in our set (size of approximately 15 µm). The collected signal behaves erratically, with all the bands pointing downwards. The collected signal behaves like a transmission spectrum, even though it has been acquired in a reflectance configuration.

without the risk of compromising the samples. To cover the above-mentioned spectral range, we used three different FTIR microscopes (see Fig. 4):

1. a Continuum microscope with a FTIR spectrometer equipped with an MCT/B detector, synchrotron-radiation-fed, allowing us to probe both the near and mid-IR spectral ranges (from 1 to 18 µm);
2. a NicPlan microscope with a IS50 FTIR spectrometer (Thermo Fisher), equipped with a bolometer detector (boron doped silicon, 4.2 K cooled, Infrared Laboratories) and a solid-state Si beamsplitter, allowing us to probe the far-IR range (from 15 to 50 µm);
3. an Agilent Cary 670/620 micro-spectrometer using the internal Globar source, equipped with a focal plane array (FPA) detector, allowing us to acquire spectral maps and hyperspectral images in the mid-IR range (from 2.5 to 12 µm).

The large spectral coverage obtained by coupling all these instruments allowed us to detect carbonates (around 7 µm), organics (around 3.4 and 6.2 µm), and phyllosilicates (around 2.7 µm for the metal-OH stretching vibration and 10 µm for the SiO stretching vibration). Hyperspectral imaging in the mid-IR allowed us to start probing the composition heterogeneity of individual grains.

Raman spectra and maps were also acquired to investigate the characteristics of the endemic aromatic organics in Ryugu's grain, as well as to complement mineral identification data. Raman data is acquired using a DXR Raman microspectrometer from Thermo Fisher with a 532 nm exciting laser radiation, using a low power—typically in the 0.5–0.5 mW range—to avoid heating of the samples. Nonetheless, due to the preciousness of the samples, these measurements were done on isolated small fragments detached from the main grains, to avoid alteration

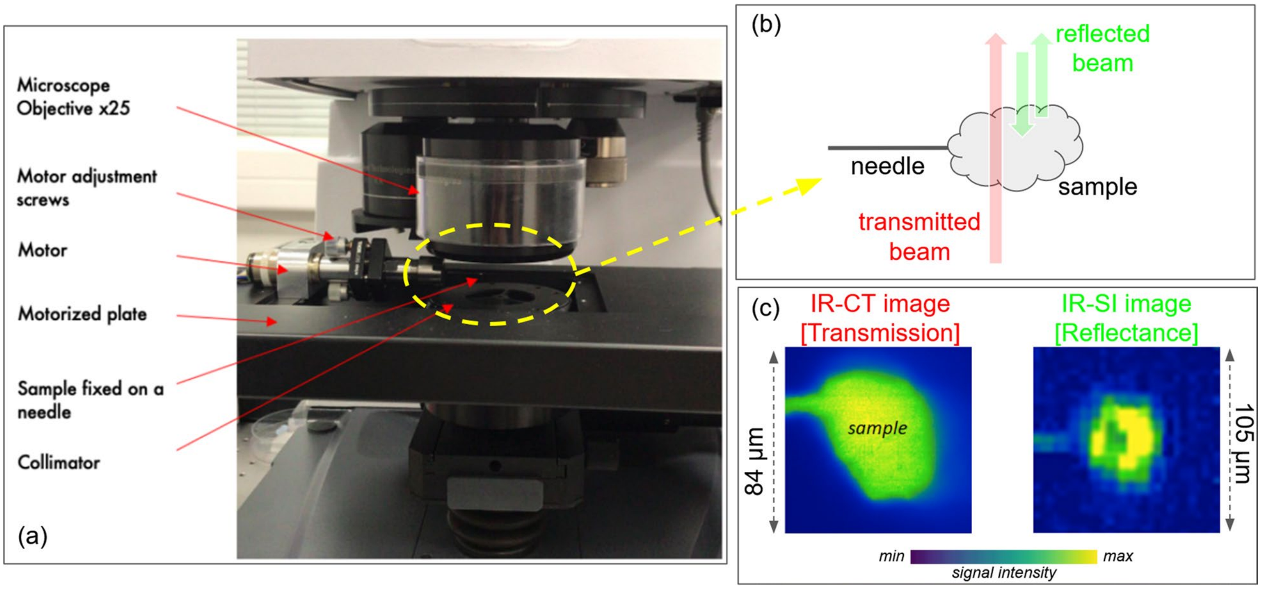


Fig. 8 IR-CT and IR-SI principle of measurement. **a** description of 3D measurements setup pieces, **b** schematization of beam path for IR-CT measurements (transmitted beam) and IR-SI (reflected beam), **c** example of 1 hyperspectral image from IR-CT and IR-SI imaging techniques. The images shown in panel **c** correspond to the continuum signal at 2.7 µm transmitted (for IR-CT) or reflected (for IR-SI) by the sample. For IR-CT, 90 images are acquired, one every 2°, rotating the sample from 0° to 180°: the final dataset consists of 128 × 128×90 spectels (a spectel is the equivalent of a pixel for hyper-spectral imaging: only one value can be associated to a pixel, while a whole spectra can be associated to a spectel). For IR-SI, 18 images are acquired, one every 20°, rotating the sample from 0° to 360°: the final dataset consists of 32 × 32×18 spectels

from the Raman laser. An example of a measured Raman spectrum is shown in the figure below (Fig. 5).

The D and G bands of the endemic organic matter are clearly visible and their position is compatible with CI-chondrites, supporting the results and discussion lead in Nakamura et al. (2022) (see science publication for a more in-depth scientific discussion).

Raman was also used to detect molecular oxygen inside one of the sample holders (SH2, see Fig. 6), indicating that the holders had lost their air-shut condition at some point, probably in flight from Japan to France.

Upon reception, we realized that the small static-shielding bags holding the sample holders were torn open, possibly due to the pressure difference between the inside and the outside of the bags during the flight. This had probably led to the air from the larger static-shielding bag entering the sample holders. The grains may have been exposed to air for about 72–96 h during transportation, before putting them again in a dry N_2 atmosphere. However, we did not observe any modification on the sample holder's KBr window (a control KBr window we exposed to air for 24 h showed clear modifications, such as opaqueness, creases and wavy patterns across the surface). We inferred that Ryugu grains remained in a relatively dry environment in spite of the presence of O_2, probably thanks to the presence of numerous desiccant packs in the traveling case, which prevented an increase of humidity.

To easily acquire FTIR spectroscopic measurements through the KBr window, the grains were arranged onto a gold mirror. This fact had unforeseen consequences on the spectroscopic measurements of all small particles (size smaller than 100 µm), with the collected spectra showing some peculiarities affecting the surface scattering spectral region (from approximately 9 µm and above). In standard conditions, the IR beam would shine onto the particle surface, be reflected by the grain's surface and be then collected for analysis. This is what we observed for the largest particle in our set of grains, which had a size of approximately 150 µm (Fig. 7, left panel). However, for smaller particles (size < 100 µm), the IR beam is able to go through the grain, similar to what happens in transmission measurements. The transmitted beam would then hit the gold mirror where the particle would rest, shining back inside and through the measured grain, to be collected for spectral analysis (Fig. 7, right panel). This means that the collected beam would be a mix of reflected signal and double-transmitted signal. Their respective contributions may be difficult to gage, but for small grains one would think that this double-transmitted signal would dominate. The consequences of this effect

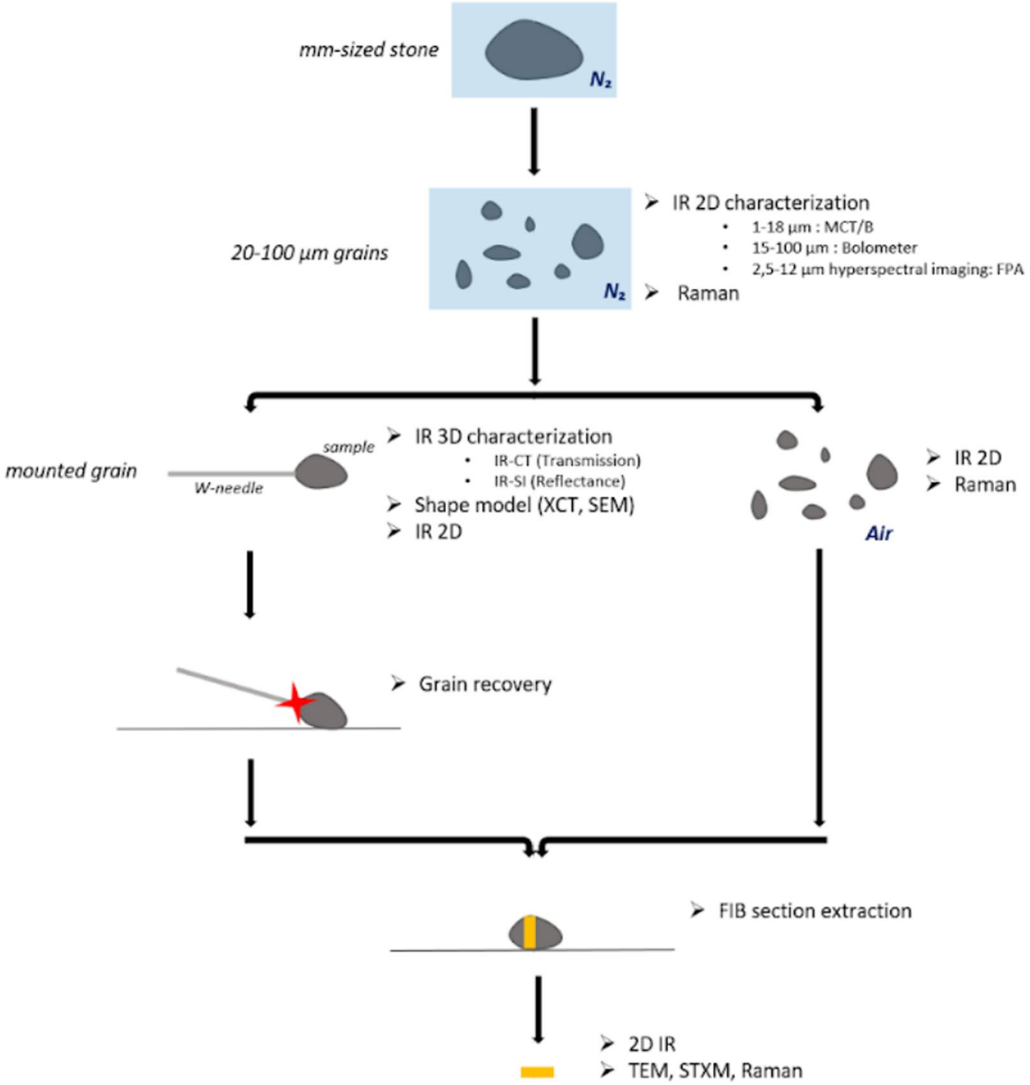


Fig. 9 Cartoon summarizing the analytical pipeline used for Ryugu's particles. Note that everything is stored under a dry N_2 atmosphere when not in this pipeline

on the spectra measured in these conditions are the following:

- Inversion of the spectral bands in the surface scattering region (above 9 µm);
- Interpretation of band intensity is not straightforward.

This quirk makes the interpretation of the collected signal from small grains more delicate (band intensity and band ratio not straightforward to discuss, the feature position is easier to apprehend), but does not invalidate the usefulness of the measured spectra. It is a well-known phenomenon that the reflectance may contain signals originating from the substrate material in addition to the signal originating from the sample under investigation, and one of the most common effects is the flipping of the IR spectrum. This has been observed in the studies involving thin meteorite sections or particularly small grain size meteorite powders (Skulteti et al. 2020).

Measurements out of the sample holder (ambient air)

Based on the spectral properties (clarity/heterogeneity of the hydration feature, interesting silicate features) obtained with the first step of our analytical pipeline, we selected 9 grains to be mounted on W and Al needle for 3D IR characterization, with sizes ranging approximately from 20 to 100 µm. The sample holders were opened, and the grains were mounted on W or Al needles using Pt-weld at two different FIB-SEM microscopes, a FEI Thermofischer Helios Nanolab 660 at MSSMAT in Saclay and a FEI Strata DB 235 SEM–FIB microscope at IEMN in Lille (Aléon-Toppani et al. 2021). These mounted grains underwent then 3D characterization in both transmission and reflectance, using Infrared Computed Tomography (IR-CT) and Infrared Surface Imaging (IR-SI) respectively (see Fig. 8). IR-CT allows us to assess the compositional heterogeneity of small particles in a 3D space (Dionnet et al. 2020; Yesiltas et al. 2017; Martin et al. 2013), while IR-SI allows us to assess the surface composition for larger particles, treating the grain as a planetary surface by projecting the 2D IR hyper-spectral maps on a 3D shape model (Dionnet et al. 2022). For IR-CT, a 25 × objective is used in combination with a high magnification system placed in front of the 128 × 128 pixel FPA detector: the projected pixel size is approximately 0.66 µm and the field-of-view is 84 µm. For IR-SI, only the 25 × objective is used to maximize signal-to-noise ratio, and the projected pixel size and FOV are 3.3 µm and 105 µm respectively (the detector size is reduced to 32 × 32).

From mounted grains, FIB sections are extracted to perform TEM analysis, following similar procedures to what are described in Aléon-Toppani et al. (2021).

The grains that have not yet been mounted remained in their respective sample holder. Some of these grains underwent complementary measurements, such as Raman micro-spectroscopy (Maupin et al. 2020). All grains are kept in a N_2 atmosphere when they are not being measured.

Conclusion

We received in July 2021 several particles from the asteroid Ryugu as part of the "STONE" preliminary analysis team. The samples traveled in a custom-made sample holder which allowed spectroscopic measurements while preserving the samples from weathering phenomena due to their exposure to the atmosphere. The sample holders lost their air-shut condition during the trip but were exposed only to dry air for less than 96 h, as the control samples which traveled alongside Ryugu's samples showed. Upon arrival, the N_2 atmosphere was restored and the samples were subjected to a precise analytical pipeline, presented in Fig. 9, with the goal of maximizing the scientific output achievable before possible sample loss.

Beside the loss of the air-shut condition of the sample holders due to the pressure difference between the first layer of static-shielding bags and the air inside the airplane cabin (issue which could be dealt with by replacing the first layer of static-shielding bags with a hard-shell vessel, such as a flange), the analytical pipeline summarized here allowed to fully characterize the spectral response of multiple grains in the IR range (from 1 to 20 µm), in both 2D and 3D, coupled with Raman spectroscopy, prior to subjecting the samples to smaller scale and more destructive electron microscopy measurements. The entire process granted us complementary results providing great insight on this very precious material.

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Author contributions
This work is part of the multi-analytical sequence of the Hayabusa2 "Stone" MIN-PET group, led by Tomoki Nakamura. Authors from Tohoku University selected and prepared the samples in the custom-made sample holders. The IAS/Soleil team handled the conception of these sample holders. Their transportation from Soleil to Tohoku University (and back) was possible thanks to a joint effort from both teams. The IAS/Soleil team also conducted the measurements in Soleil. Eva Héripré and David Troadec are responsible for mounting the microscopic samples on the metal needles for IR-CT and IR-SI. All authors read and approved the final manuscript.

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Availability of data and materials
The data that support the findings of this study are available from the corresponding author, Stefano Rubino (stefano.rubino@universite-paris-saclay.fr) upon reasonable request.

Declarations

Competing interests
The authors declare no competing interests.

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