Small grains from Ryugu: handling and analysis pipeline for Infrared Synchrotron Microspectroscopy

Stefano Rubino[1], Zélia Dionnet[1], Alice Aléon-Toppani[1], Rosario Brunetto[1], Tomoki Nakamura[2], Donia Baklouti[1], Zahia Djouadi[1], Cateline Lantz[1], Obadias Mivumbi[1], Ferenc Borondics[3], Stephane Lefrançois[3], Christophe Sandt[3], Francesco Capitani[3], Eva Héripré[4], David Troadec[5], Megumi Matsumoto[2], Kana Amano[2], Tomoyo Morita[2], Hisayoshi Yurimoto[6], Takaaki Noguchi[7], Ryuji Okazaki[7], Hikaru Yabuta[8], Hiroshi Naraoka[7], Kanako Sakamoto[9], Shogo Tachibana[9-10], Sei-ichiro Watanabe[11], Yuichi Tsuda[9], and the Hayabusa2-initial-analysis Stone team

[1]*IAS, Université Paris-Saclay, CNRS, France,* [2]*Tohoku University, Japan,* [3]*SMIS-SOLEIL, France* [4]*CentraleSupélec, Université Paris-Saclay, CNRS, France,* [5]*IEMN, Université de Lille, France,* [6]*Hokkaido University, Japan,* [7]*Kyushu University, Japan,* [8]*Hiroshima University, Japan,* [9]*ISAS/JAXA, Japan,* [10]*The University of Tokyo, Japan,* [11]*Nagoya University, Japan*

Introduction: In December 2020, the sample return mission Hayabusa2 (JAXA) brought 5.4g of matter from the surface of C-type primitive asteroid (162173) Ryugu [1]. This extremely precious material is now the object of multiple studies with state-of-the-art techniques in laboratories all around the world (Initial Analysis phase). These studies will grant new insight on the origin and evolution of primitive hydrated planetesimals, and ultimately on the early stages of our solar system. Within the Hayabusa2-initial-analysis "Stone" team [1] led by T. Nakamura, we received several microscopic particles in July 2021, with the goal of performing IR hyper-spectral imaging and IR micro-tomography studies.

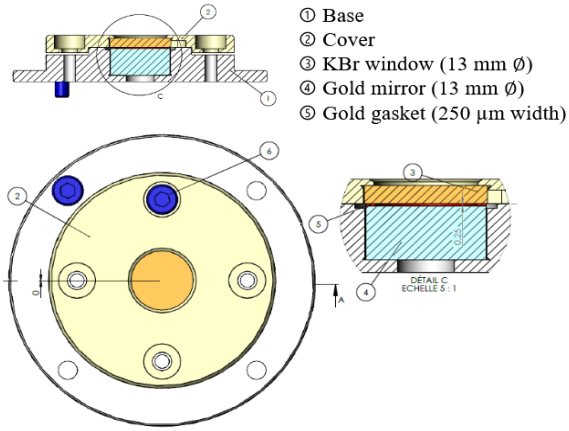

Figure 1. Sample-holder description

Samples transfer and handling: Ryugu samples are kept in a controlled N_2 environment at Sagamihara (JAXA) in order to avoid exposure to the terrestrial atmosphere. We took several steps in order to minimize the samples' exposure to air, before, during and after measurements. Prior to receiving the samples, we designed a custom sample-holder (Figure 1) for transferring particles from Japan to France and for their preliminary analysis. Particles from Ruygu were deposited on a gold mirror inside the sample-holder at Tohoku University (Japan). A KBr window installed in the cover of the sample-holder allowed us to acquire measurements of the particles while the sample-holder was still sealed. This custom-made sample-holder was kept as clean and sterile as possible (to avoid terrestrial contamination) and it had to fulfill two major requirements: fit on the microscope stages for our IR imaging and microspectroscopy studies and keep the samples in a dry N_2 environment (to avoid alteration from the atmosphere). The sample-holder was assembled inside a glovebox under N_2 atmosphere ($\Delta P \sim 6$ mBar, $H_2O = 1.0$ ppm, $O_2 = 1.2$ ppm). Multiple interfaces were used in order to prevent the mixing of the clean N_2 atmosphere and the ambient air (gold gasket, cleanroom static shielding bags), but also to avoid issues from shocks and vibrations (membrane boxes, foam-padded case). Once in Sendai, the empty sample holders were opened in a glovebox, and were loaded with 32 microscopic particles from Ryugu (distributed among four sample-holders: 14 particles from the first touchdown site in SH2 and SH3, and 18 particles from the second touchdown site in SH1 and SH4).
To monitor the atmosphere's changes in our sample-holders, these were accompanied by a "control"-holder, with grains of olivine and Fe dust (to monitor hydration and oxidation). A second "control"-holder, loaded with drierite (calcium sulfate) and some Fe dust, was kept in France, to monitor hydration and oxidation when not under dry N_2 conditions.

Samples reception and characterization: Upon arrival at synchrotron SOLEIL (France) in early July, the sample holders were inspected with a binocular through the KBr windows. We were able to identify at least 24 out of 32 of the original Ryugu particles, either based on their morphological correspondence with the grains prepared at Tohoku, and/or thanks to their clear IR "Ryugu-like" spectral signatures (mainly the 2.7 µm feature [2]). The particle size range was from 20 to 80 µm, with a particularly large particle in SH4 larger than 100 µm.
The analytical pipeline started with a full spectral characterization using the IR synchrotron beam while keeping the sample-holder closed, in order to minimize exposure to air. During the first three weeks of July, we analyzed all the identified Ryugu grains in their sample holders using three different FTIR microscopes: (1) a mid-IR large wavelength range MCT/B-equipped and synchrotron-radiation-fed microscope, (2) a far-IR bolometer-equipped microscope, (3) an FPA-equipped imaging microscope. Multiple detectors were used to access different wavelength ranges, covering from near-IR to far-IR (1 µm to 35 µm). This large spectral coverage allowed us to detect carbonates, organics, and phyllosilicates with different band positions [3]. Most of the IR spectra showed interesting features. Hyperspectral maps in the mid-IR of the individual particles were also acquired, in order to assess the compositional heterogeneity at the 5-10 µm scale. A few Raman spectra and maps were also acquired on isolated fragments, to assess the presence and structure of endemic organics in Ryugu's particles and to get complementary information on the mineralogy.

Thanks to a Raman measurement on a µm-spot on the gold substrate, we managed to detect remotely molecular oxygen inside SH2, which indicates the holders at some point lost their air-shut condition, probably in flight from Japan to France. We estimate that the grains were exposed to air for about 72-96 hours, before we received them and we put them again into a dry N_2 environment. However, we did not observe any modification of the KBr (a control KBr window we exposed to air for 24h showed clear modifications), and the anhydrous control samples we sent along the Ryugu sample holders showed no evidence of water adsorption nor hydration. So we inferred that Ryugu grains remained in a relatively dry environment, although in presence of O_2, probably thanks to the presence of desiccants that prevented an increase in humidity.
In mid- and late-July, we opened sample holders SH2 and SH3, and we mounted 6 Ryugu grains on W or Al needles using two different FIB-SEM microscopes in Saclay and in Lille [4]. These grains were then measured in both transmission and reflectance mode, by Infrared Computed Tomography [5] (IR-CT) and Infrared Surface Imaging [6] (IR-SI) respectively. IR-CT allows us to assess the compositional heterogeneity of small particles in a 3D space, 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. Data processing and analysis of this large set of data is still ongoing. Our primary method of analysis, IR spectroscopy, is totally non-destructive, which implies that after our measurements a few particles can be sent to other analytical teams in the scope of combining different studies on the same sample. Some of these particles are now being analyzed by X-ray computed tomography at SPring-8 (Japan) [7].
Overall, the outcome of the last months has shown that the agreed handling, transfer and analysis pipelines are valid and may be applied for future sample-return missions.

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