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Contents

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ABSTRACT

Sample containers for storage and transportation of the returned asteroid samples recovered in the Hayabusa, Hayabusa2, and OSIRIS-REx missions have been developed in JAXA curation. Small particles of Hayabusa samples were first dropped on the quartz disks, and aluminum disks were transferred and stored in the multi-well concavity slides, and they have recently been moved to the φ10 mm individual sample holders. To check if the samples are derived from the Itokawa or other contaminants by SEM-EDS, the dedicated SEM holder is used to transfer samples between the chamber and the instrument. The samples are transferred to the other concavity slide and packed in the clamp-sealed transfer container to deliver samples to the other research institutes. Hayabusa2 samples consisting of fine particles to mm size grains are first stored in the bulk sample containers. Individual grains and aggregate samples are extracted from the bulk sample containers and moved to the φ15 mm or φ10 mm sapphire dish with a stainless steel outer holder. Facility-to-facility transfer container (FFTC), the O-ring sealed container, is used to deliver samples to the other institutes. A portion of the OSIRIS-REx samples received at NASA Johnson Space Center (JSC) is separated and transported to JAXA curation. Newly designed bulk sample containers are used to store the separated samples. The transfer containers designed by JSC curation are used to containerize the bulk sample containers and to deliver them to JAXA curation. Accumulative knowledge and experiences of extraterrestrial sample curation in the sample return missions, including the development of sample containers, have contributed to the successful science outcomes in the Hayabusa and Hayabusa2 missions. Development and improvement of sample containers continue for upcoming MMX and future sample return missions.

Keywords: Return Sample,Hayabusa,Hayabusa2,Curation,Sample Container,ample Storage,Clean Chamber

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1 INTRODUCTION

The Extraterrestrial Sample Curation Center of JAXA (ESCuC) was established to receive extraterrestrial materials returned from asteroids in the JAXA sample return missions, such as Hayabusa and Hayabusa2 missions [1] [2] [3]. In addition, ESCuC will receive a portion of samples returned from the asteroid Bennu in the NASA OSIRIS-REx mission [4]. These returned samples are stored and handled inside clean chambers kept with an extremely purified nitrogen atmosphere in ISO6 (class 1000) clean rooms in the ESCuC (Figs. 1, 2) to prevent the samples from being exposed to the Earth's atmosphere. Not only the atmospheric condition but also materials used in clean chambers play an essential role in controlling the contamination. Thus, ESCuC severely restricts the materials used in the clean chambers principally to stainless steel (e.g., SUS304), aluminum (e.g., A6061), Teflon, and glass materials (synthetic quartz and sapphire). All the products and tools used in clean chambers are made with these materials and cleaned with the specific cleaning process [5] [6].

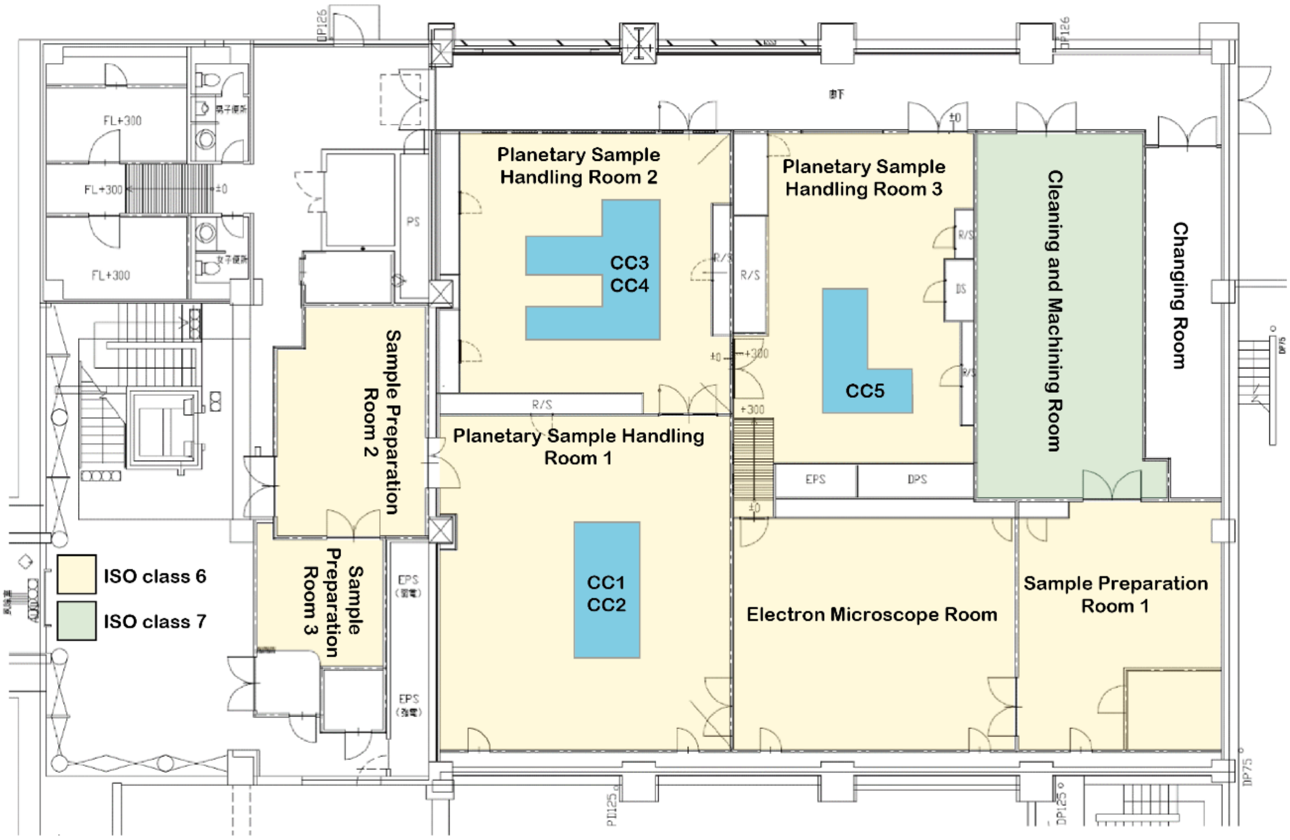

Fig.1 Layout of clean rooms in the ESCuC. Clean chambers CC1 and 2 for Hayabusa samples, CC3 and 4 for Hayabusa2 samples, and CC5 for OSIRIS-REx samples are equipped in ISO class6 clean rooms of Planetary Sample Handling Room 1, 2, and 3, respectively.

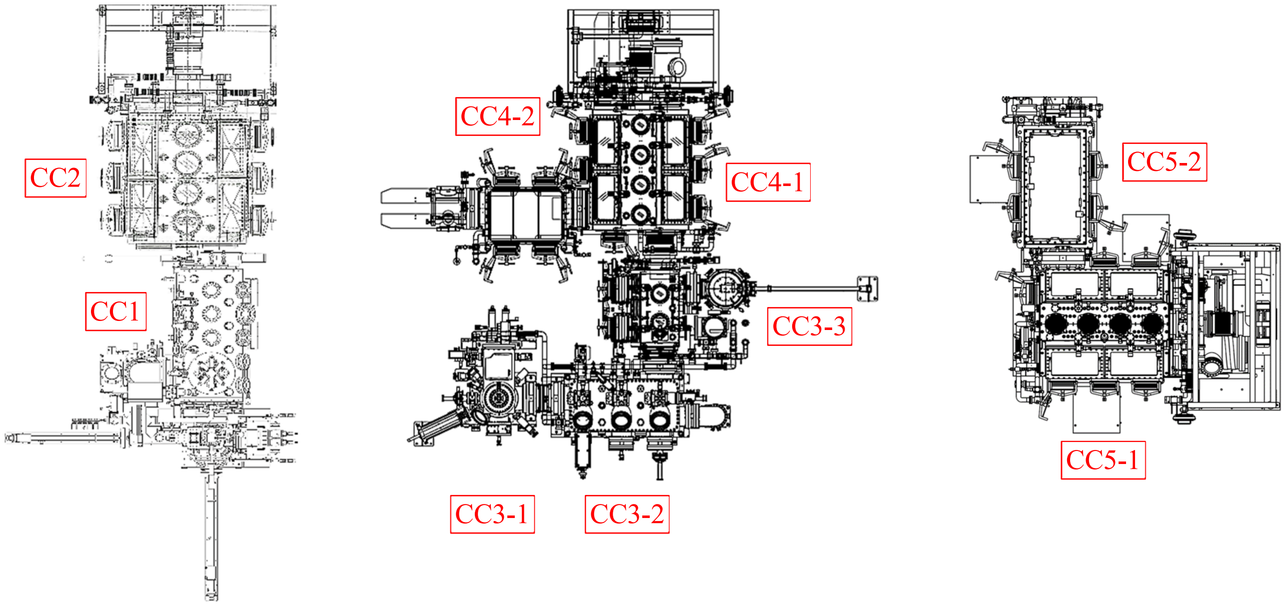

Fig. 2 Clean chambers for a) Hayabusa, b) Hayabusa2, and c) OSIRIS-REx samples.

Sample containers used in clean chambers are prepared for the storage of returned samples and transportation to another research institute. Sample containers basically consist of an inner container made of glass material (synthetic quartz or sapphire) on which the samples are directly touched and an outer holder made of stainless steel (SUS304). On the other hand, the detailed design of the containers differs depending on the size of the samples and the purpose of use. Here, we present the design of sample containers prepared for Hayabusa, Hayabusa2, and OSIRIS-REx samples and the handling of these containers in sample processing.

2 USE OF SAMPLE CONTAINERS FOR RETURNED ASTEROID SAMPLES

2.1 Sample containers for Itokawa samples in Hayabusa mission

The Hayabusa spacecraft sampled at the surface of the S-type near-Earth asteroid Itokawa (25143) by touchdown at two landing sites [7] [8]. Samples at the first and second touchdown sites were collected in Rooms B and A in the Sample Catcher, respectively. The spacecraft returned samples to the Earth in June 2010, and the samples contained in the sample catcher were transported to the ESCuC and installed into the clean chamber CC1 in an ISO class6 clean room at the facility. After opening the sample catcher in CC2, a portion of the samples was moved directly from the sample catcher to a slide glass using the electrostatic micro-manipulator or collected on the Teflon spatula. The remaining samples in Rooms A and B were dropped on the quartz disk slides of 48 mm in diameter and 2 mm in thickness (Fig.3a), which had basically no chemical reaction to the samples. Then, the remaining particles were dropped on aluminum metal disks of 48 mm in diameter and 3 mm in thickness (Fig. 3b) to be able to observe samples without moving them.

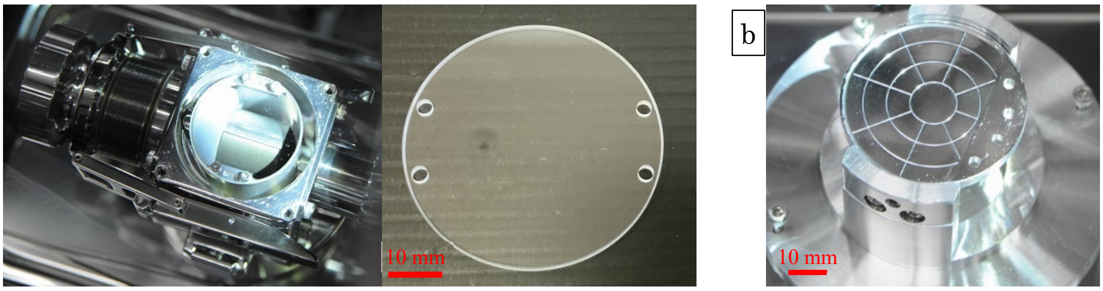

Fig. 3 a) Quartz disk slide and b) aluminum metal disk to drop the Hayabusa samples

Since the collected samples were small particles less than a few hundred microns in size, elemental analysis by SEM-EDS was essential to confirm if the particles were derived from Itokawa or other contaminants. The particles were enclosed in the SEM holder (Fig. 4) within the clean chamber and transferred to the SEM-EDS without exposure to the terrestrial atmosphere. The particles analyzed by SEM-EDS were returned to the clean chamber and moved to the multi-well concavity slides (Fig. 5) made of quartz glass enclosed in an aluminum case for sample storage. The initial observation of collected materials on the Teflon spatula and the quartz disks revealed that Hayabusa samples consisted of a lot of fine particles. The multi-well concavity slide was designed to store the particles as effectively as possible using the electrostatic micro-manipulator.

Fig. 4 SEM holder

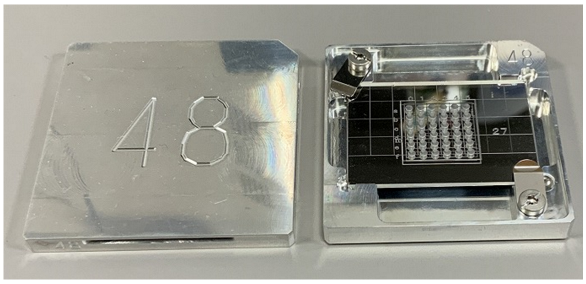

Fig. 5 Multi-well concavity slide assembly

After the return of Hayabusa2 samples, we recognized that the sample holders used to store Ryugu individual grains are also suitable for handling the individual Itokawa particles. Individual particles are transferred from the multi-well concavity slides to single concavity quartz dishes put in the outer holders, the same as those used for Hayabusa2 sample storage (capsule pack 10). The sample transferring process is currently ongoing.

Fig. 6 Single well concavity dish with capsule pack 10

Samples to be allocated to the other research institutes are transferred to another concavity slide with 5 dimples (Fig. 7a), as 5 samples can be requested at a maximum at a time in the Hayabusa Announcement of Opportunities

(AO). The concavity slide was sealed in the NW flange-based stainless-steel (SUS304) container with an aluminum edge seal and a chain clamp (Fig. 7c) to keep the samples in hermetic condition during transportation. The hermeticity of the aluminum edge seal with chain clamp was confirmed with the helium leak rate of less than 1×10^{-11} standard cc/sec using a residual gas analyzer quadrupole mass spectrometer (RGA-QMS) in high vacuum condition. However, the chain clamp contains materials that are unacceptable to use inside the clean chambers. Hence, the container is first sealed using a quick clamp with O-ring sealing (Fig. 7b) inside the clean chamber (CC2) and moved to the glovebox dedicated to handling the Hayabusa samples (GB2). Then, the O-ring and quick clamp are replaced with an aluminum edge seal ring and chain clamp in the GB2. As the Itokawa particles have been stored individually in φ10 mm sample holders since 2021, the transfer container was modified to enclose the sample holder by using the pack holder (see next chapter for details), same as for Hayabusa2 sample container (Fig. 7d).

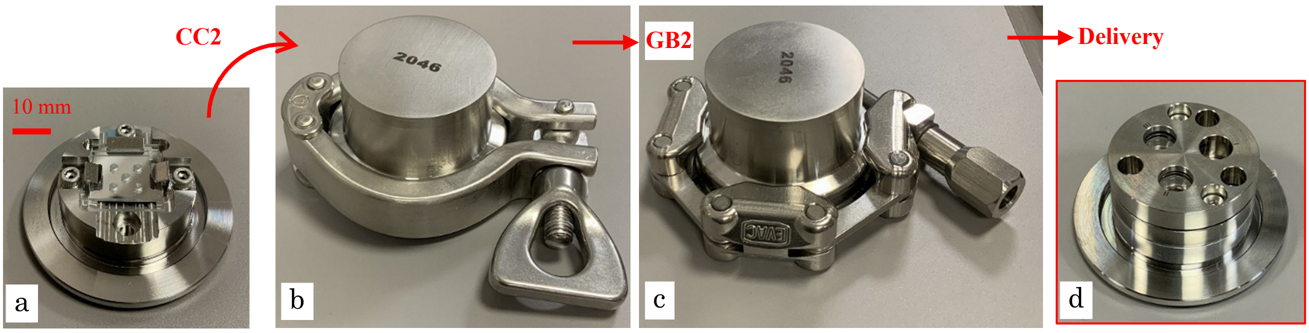

Fig. 7 Sample transfer containers for Hayabusa samples, a) transfer container base with multi-well concavity slide, b) quick clamp with O-ring seal, c) chain clamp with aluminum edge seal, d) transfer container base for φ10mm sample holders

2.2 Sample containers for Ryugu samples in the Hayabusa2 mission

The Hayabusa2 spacecraft sampled at the surface of the C-type near-Earth asteroid (162173) Ryugu by touchdown at two landing sites of TD1 and TD2 [9] [10] [11]. Samples were collected in the sample catcher divided into three chambers, A, B, and C. Chambers A and C collected samples at TD1 and TD2, respectively. In contrast, chamber B was kept open between 1st and 2nd touchdown and expected to collect floating particles. The spacecraft returned samples to the Earth in December 2020, and the samples contained in the sample catcher were transported to the ESCuC and installed into the clean chamber CC3-1 in an ISO class6 clean room at the facility. The sample catcher was then opened in CC3-2, and a small portion of the samples were extracted and dropped on the synthetic quartz petri dish (Fig. 8) in high vacuum conditions. The sample catcher was moved to CC4, consisting of two chambers, CC4-1 and 4-2, and the remaining samples were divided into the bulk sample containers (Fig. 9) in purified nitrogen condition. The standard bulk sample container (deep type) was designed to contain 1 g of samples with particles less than 5 mm in size, which was the estimated maximum sample volume and maximum grain size, respectively. We also prepared bulk sample containers for even fewer samples (shallow type). The detailed design of deep and shallow types of bulk containers is described in Chapter 3.2.1. Samples in chambers A, B, and C were distributed separately to the different bulk sample containers. A total of seven bulk sample containers (three deep types for chamber A, three deep types for chamber C, and one shallow type for chamber B) were used.

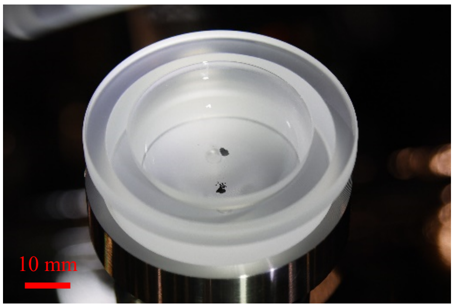

Fig. 8 Synthetic quartz petri dish in CC3-2

Fig. 9 Bulk sample container

For initial description and sample allocation to other research institutes, individual grains and aggregate samples were extracted and transferred to smaller sample containers, consisting of a sapphire dish and outer holder, called the capsule pack (Fig. 10). The sapphire dish was selected because it has more than 90% of transmittance through visible and infrared wavelength up to 5 µm without any absorption band, which is suitable for the NIR spectrometry such as FTIR and MicrOmega (NIR hyperspectral microscope). The sample holder of a capsule pack with a sapphire dish is basically designed to store an individual particle, and there are three types depending on the height of samples: capsule pack 10 (<1 mm), 15-1 (<3 mm), and 15-2 (<6 mm). In addition, the sapphire dish for capsule pack 10 is made for micro-manipulator accessible to sub-mm-sized fine particles. The largest grain collected in the Hayabusa2 mission (C9000) was larger than the inner diameter of the capsule packs. Therefore, a specially customized container for C9000 was made (Fig. 11). We also prepared shallow φ60 mm synthetic quartz petri dishes (φ60 quartz petri dish) to collect grains larger than 1 mm in length (Fig. 12). Bulk sample containers originally contain various sizes of particles in the range of several millimeters to fine powder. In comparison, aggregate samples extracted from bulk sample containers should be composed of particles less than 1 mm in length. Hence, particles larger than 1mm were extracted from the bulk sample container and transferred to φ60 quartz petri dishes before extracting aggregate samples.

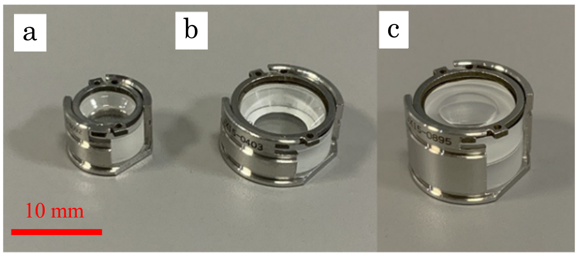

Fig. 10 Capsule packs a) 10, b) 15-1, and c) 15-2

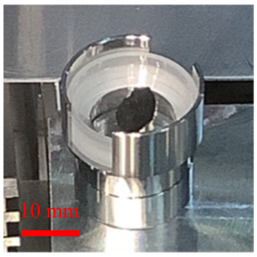

Fig. 11 Sample container for C9000

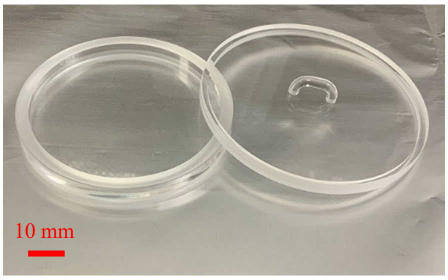

Fig. 12 φ60 quartz petri dish

The transfer container for Hayabusa2 samples, called facility-to-facility transfer container (FFTC, Fig. 13), was originally designed and developed in cooperation with Hayabusa2 Phase2 Kochi Team for the transportation of millimeter sized extraterrestrial samples between instruments and between institutes without exposure to the terrestrial atmosphere [12]. The inner holder of an FFTC, a pack holder (Fig. 13), was designed to fix a capsule pack in an FFTC securely. Hermeticity of FFTC was tested by monitoring pressure stability in positive and negative pressure conditions, and the pressure inside the FFTC could be kept at 72.7±0.8 kPa for a month and at -60.7±0.2 kPa for a half-day [12]. The results confirm that FFTC provides enough hermeticity although it is less hermetic than the sample transfer container for Hayabusa samples. In addition, all the parts of FFTC are made of materials acceptable to be used inside the clean chambers, and the screw-type O-ring seal makes it easier to handle the FFTC inside the clean chambers.

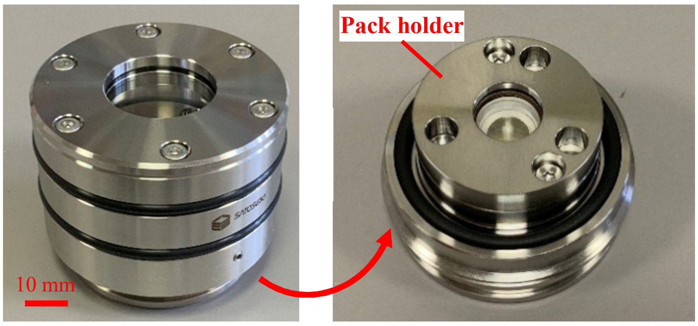

Fig. 13 FFTC for transfer of Hayabusa2 samples

2.3 Sample containers for Bennu samples received from NASA in OSIRIS-REx mission

The OSIRIS-REx (Origins-Spectral Interpretation-Resource Identification-Security-Regolith Explorer) spacecraft collected at the surface of the B-type near-Earth asteroid (101955) Bennu and returned the samples to the Earth in September 2023 [13] [14]. The returned Sample canister [15] was transported to the NASA Astromaterials Curation Facility at Johnson Space Center (JSC) and first installed into the Canister Glovebox in the OSIRIS-REx clean room of ISO class 6. The sample canister enclosed the Touch-and-Go Sample Acquisition Mechanism (TAGSAM) containing collected samples and other devices. TAGSAM was dismounted from the sample canister inside the Canister Glovebox and transferred to the TAGSAM Glovebox for the following processes. TAGSAM has 24 contact pads at its bottom, which touched on the asteroid's surface during the sampling and are expected to capture the most surface materials of Bennu. Each contact pad was removed and containerized in the dedicated containers used for both sample storage and delivery. Then, after the TAGSAM was opened, collected samples were transferred to the pie-shaped aluminum sample trays [4].

JAXA will receive 0.5% of the total sample mass by the Memorandum of Understanding (MOU) between NASA and JAXA. A portion of samples in the sample trays is extracted and transferred to the OSIRIS-REx bulk sample containers (Fig. 14) prepared by JAXA curation, which consist of sapphire dishes and outer metallic (SUS304) holders. The sapphire dish is same as the deep-type bulk sample dish used to contain Hayabusa2 samples up to 1.2 g. According to XX, initial estimated mass, approx. 250 g was collected in the TAGSAM. Based on this initial estimated mass, approx. 1.25 g of samples will be allocated to JAXA. The JAXA curation prepared 5 bulk sample containers with capacity of collecting more than 5 g of samples considering each selected area of samples might be necessary to be stored in a different container. The maximum grain size for the bulk sample container is approx. 10 mm in length and 5 mm in height. The outer metallic holder was re-designed from the one used for Hayabusa2 samples to handle the bulk sample container more easily and safely. The bulk sample holder is enclosed in the sample holder attachment (Fig. 15) and then assembled to the sample transfer container prepared by NASA JSC curation. After the bulk sample containers are transported to ESCuC, they will be installed into the clean chamber CC5 consisting of two chambers, CC5-1 and 5-2, in an ISO class6 clean room. For initial description and sample allocation to other research institutes, individual grains and aggregate samples will be extracted and transferred to sample holders, the same as the ones used for Ryugu samples in the clean chamber CC4.

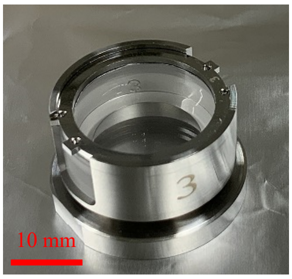

Fig. 14 O-REx bulk sample container

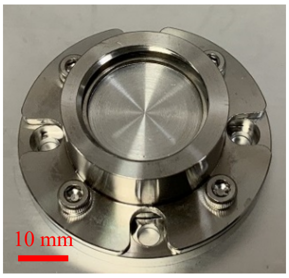

Fig. 15 Sample holder attachment

JAXA also receives one contact pad sample by the Letter of Agreement (LOA) between NASA and JAXA. The contact pad has already been stored in the contact pad container, which can be used for transportation as well. The contact pad container will be installed into a purified nitrogen glovebox prepared in the same clean room with CC5. Each particle captured in the contact pad will be extracted and transferred to “capsule packs” as main mass samples.

3 DESIGN OF SAMPLE CONTAINERS

3.1 Sample containers for Hayabusa samples

3.1.1 Sample storage containers

3.1.1.1 Multi-well concavity slide assembly (Fig. 16)

The multi-well concavity slide (40 mm×28 mm×t1 mm) made of quartz glass has multiple wells, each 1 mm in diameter and 0.5 mm in depth. This glass slide is placed on a mirror-polished stainless-steel plate to see the particles clearly under the microscope. A set of concavity slide and mirror polished plates is placed in the flat aluminum box (54 mm×54 mm×t9 mm when closed) and fixed with a leaf spring plate made of stainless steel. The aluminum box is closed with the aluminum lid for sample storage.

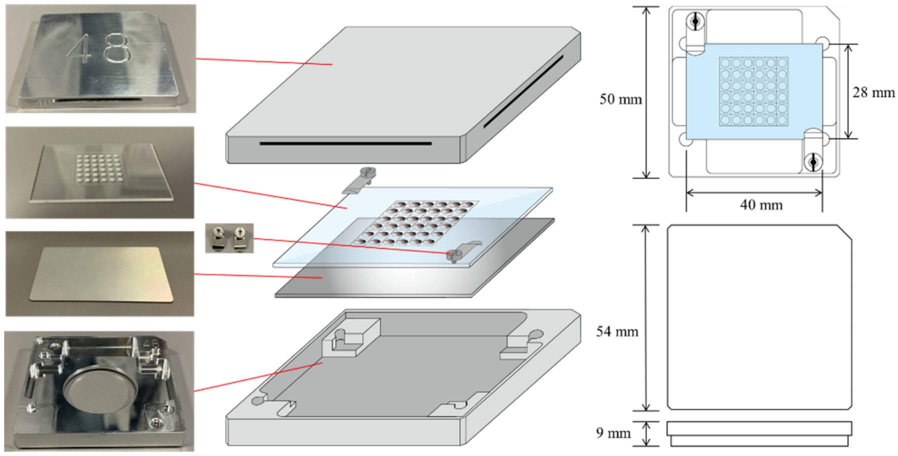

Fig. 16 Design of multi-well concavity slide assembly

3.1.1.2 φ10 mm individual sample holder (Fig. 17)

Particles stored in the multi-well concavity slides have been transferred to sample containers for individual particles since 2021. The outer holder of the sample container is identical to the one used for Hayabusa2 samples made of stainless steel. The sample dish is made of synthetic quartz, whose size is 10 mm in diameter and 4.5 mm in thickness. There is a concavity of 1 mm in diameter and 0.5 mm in depth at its center to mount a particle. Notches are made on both sides of the sample dish to hold it with tweezers.

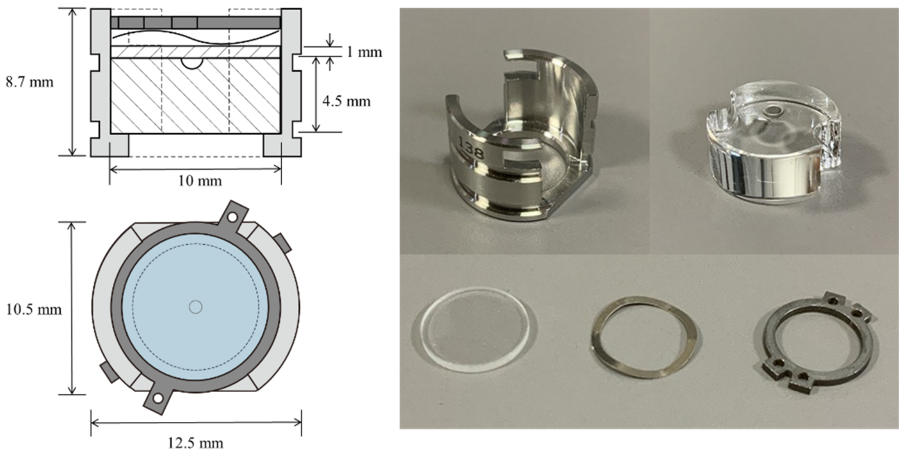

Fig. 17 Design of φ10 mm individual sample holder

3.1.2 SEM holder (Fig. 18)

SEM holder is used for transferring samples between the clean chamber and SEM without exposure to the terrestrial atmosphere, which consists of SEM holder base, sample holder, SEM holder lid, and sealing clamp. The SEM holder base is made of aluminum, and two titanium pins are attached to the side of the circular base plate. The sample holder consists of three portions – holder base, holder cap, and holder plate made of copper, aluminum, and gold, respectively – and is fixed to the SEM holder base. The SEM holder lid is made of stainless steel with an O-ring to seal the SEM holder.

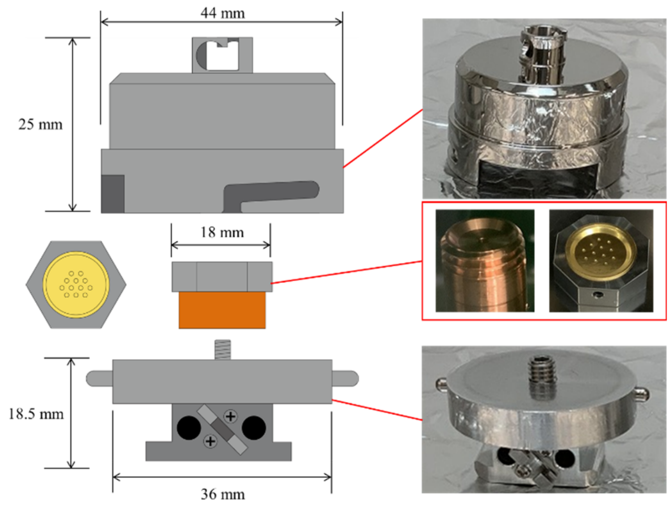

Fig. 18 Design of SEM holder

3.1.3 Sample transfer container (Figs. 19 and 20)

The sample transfer container for Hayabusa samples consists of an NW40 flange base (55 mm in diameter), inner attachment (35 mm in diameter), and silk-hat-shaped lid (55 mm at the base, 44 mm on top in diameter, and 28 mm in height), all made of stainless steel. The inner attachment is fixed to the NW flange base with a hex screw. A cover glass covers samples mounted on a multi-well concavity slide fixed to the inner attachment. Both concavity slide and cover glass are made of synthetic quartz. The sample transfer container is sealed using a quick clamp with an O-ring seal inside the clean chamber and moved to the glovebox (GB2). The quick clamp with an O-ring seal is replaced with a chain clamp with an aluminum edge seal to provide better hermeticity inside the GB2. Then, the sealed container is delivered to the other research institutes.

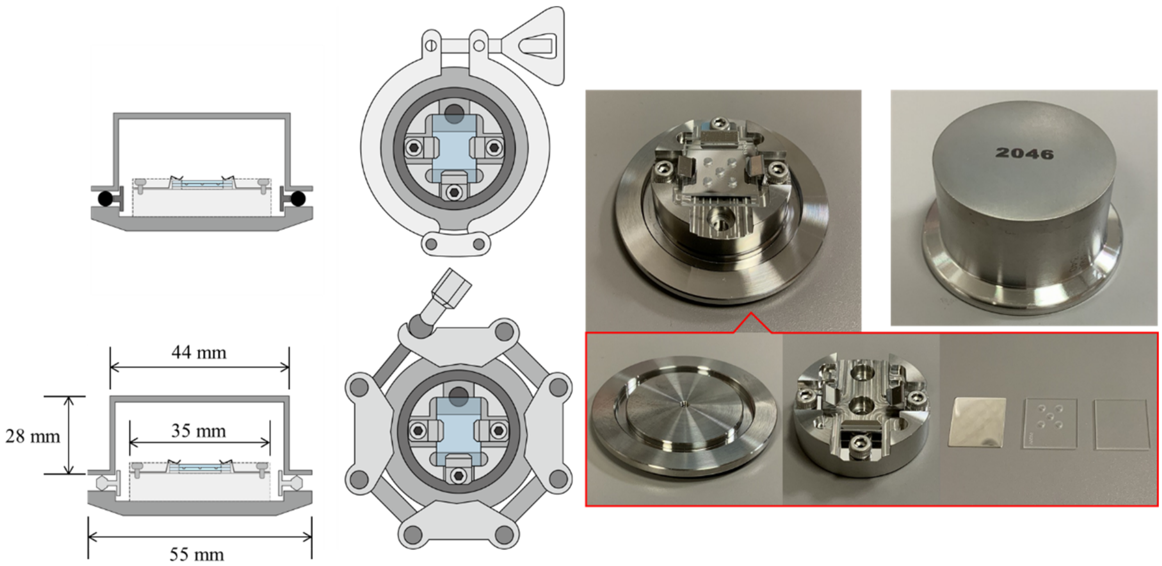

Fig. 19 Design of sample transfer container for Hayabusa samples

Fig. 20 Clamp sealing of Hayabusa samples transfer container

Due to the change of the sample storage container from a multi-well concavity slide to φ10mm single sample holder, the inner attachment of the sample transfer container is modified. The pack holder for Hayabusa2 samples (see next chapter for details) is also used with another attachment with 36 mm in diameter and 6.5 mm in thickness connecting the pack holder and the NW flange base (Fig. 21).

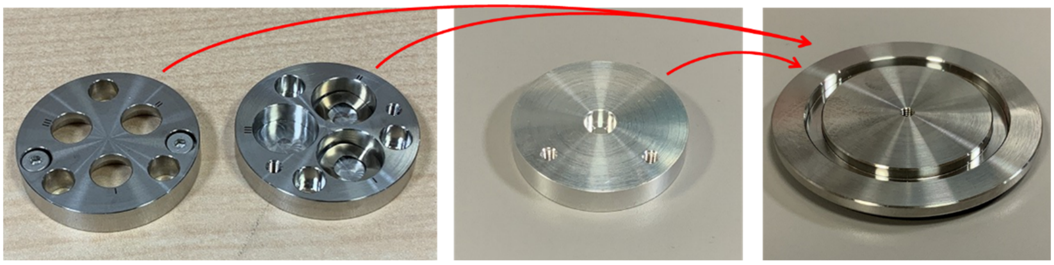

Fig. 21 Inner attachment for φ10mm single sample holder

3.2 Sample containers for Hayabasa2 samples

3.2.1 Bulk sample container (Fig. 22)

A bulk sample container is designed to store samples subdivided from the sample catcher. This container consists of a sapphire dish, a synthetic quartz window, an outer holder, and a lid made of SUS304. Viton O-ring is inserted into the inner margin of the lid. Two types of bulk sample containers, deep or shallow, are used depending on the volume of returned samples. Consequently, three deep containers were used for samples in chamber A, three other deep containers for samples in chamber C, and one shallow container for samples in chamber B. Bulk sample containers have room to place a substrate beneath the sapphire dish. A stainless steel (SUS304) or aluminum substrate with sandblasting surface is used for optical microscopic observation and FT-IR analysis to reduce reflection from an incident light. A gold mirror substrate is used for MicrOmega (near-IR hyperspectral microscope) analysis to prevent the detection of reflected incident light.

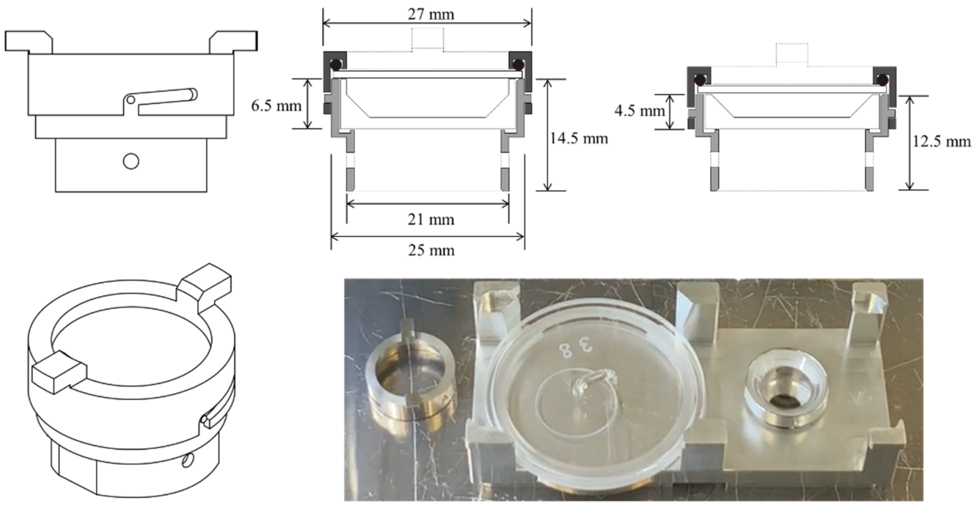

Fig. 22 Design of bulk sample containers for Hayabusa2 samples

3.2.2 Capsule pack (Fig. 23)

A capsule pack is designed to store an individual grain or aggregate sample extracted from a bulk sample. Three types of containers are prepared depending on the size of samples: capsule packs 15-1, 15-2, and 10. Capsule packs 15-1 and 10 consist of a sapphire dish, a synthetic quartz window, an outer holder, a wave washer, and a fix ring made of SUS304. A capsule pack 15-2 uses a sapphire dish as a lid of the sample dish instead of a quartz window. Capsule packs are stored with sets of a sapphire dish, an outer holder, and a quartz window inside CC3-3, 4-1, or 4-2. They are fixed with a wave washer and a fix ring within the clean chamber for sample transportation by using a dedicated opening/closing tool.

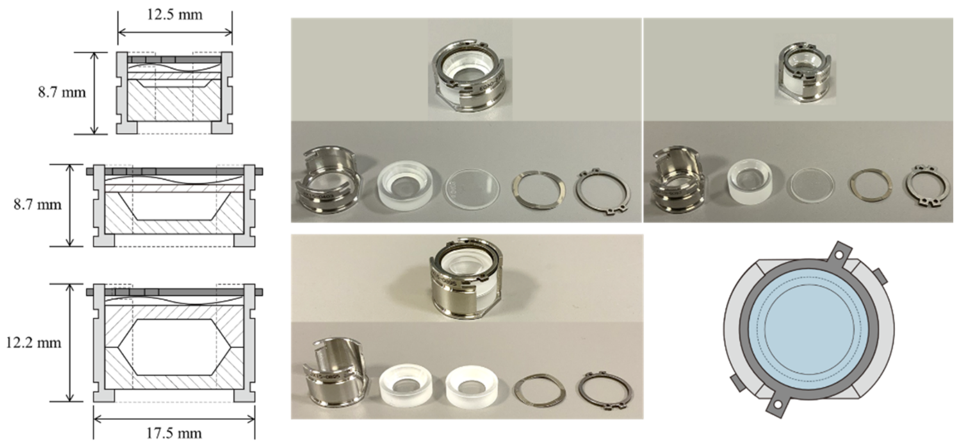

Fig. 23 Design of sample holders (capsule packs) for individual grains and aggregate of Hayabusa2 samples

3.2.3 Specially customized container for C9000, the largest grain (Fig. 24)

The largest grain in the returned samples, C9000, has a length of 10 mm on a longer axis and a height of 4 mm. This grain could not be enclosed in any of the capsule packs prepared for individual grains. Therefore, we designed and made a new container dedicated to C9000, which consists of a sapphire dish, a quartz window, and an outer holder. The size of the sapphire dish is the same as that used for a deep-type bulk sample container.

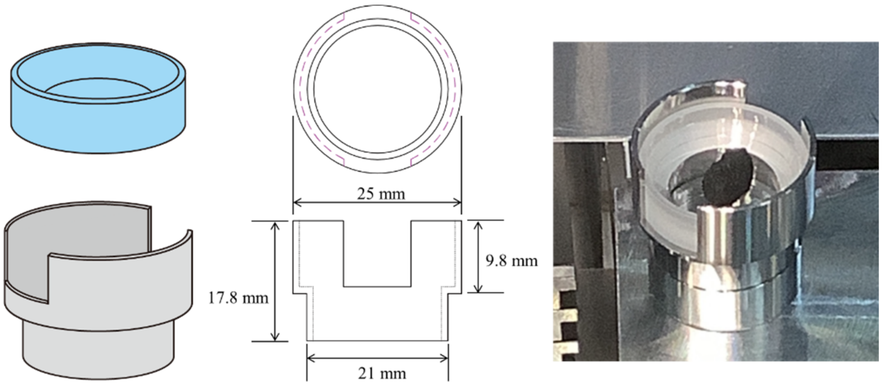

Fig. 24 Design of sample holder for C9000

3.2.4 Facility-to-facility transfer container (FFTC, Fig. 25)

An FFTC was originally designed by Ito et al. (2020) and modified to be fit for Hayabusa2 sample containers. The FFTC has been used to transfer Ryugu samples to research groups, such as Initial Analysis teams, Phase2 curation teams, and scientists via the AO (Announcement of Opportunities for the Hayabusa2 samples). The FFTC consists of a base, a pack holder, and a lid. All the metal parts in the FFTC are made of stainless steel (SUS304). The FFTC base has a Viton O-ring to seal it with a lid. Different types of pack holders were prepared for capsule packs 10, 15-1, and 15-2 and are attached to the FFTC base.

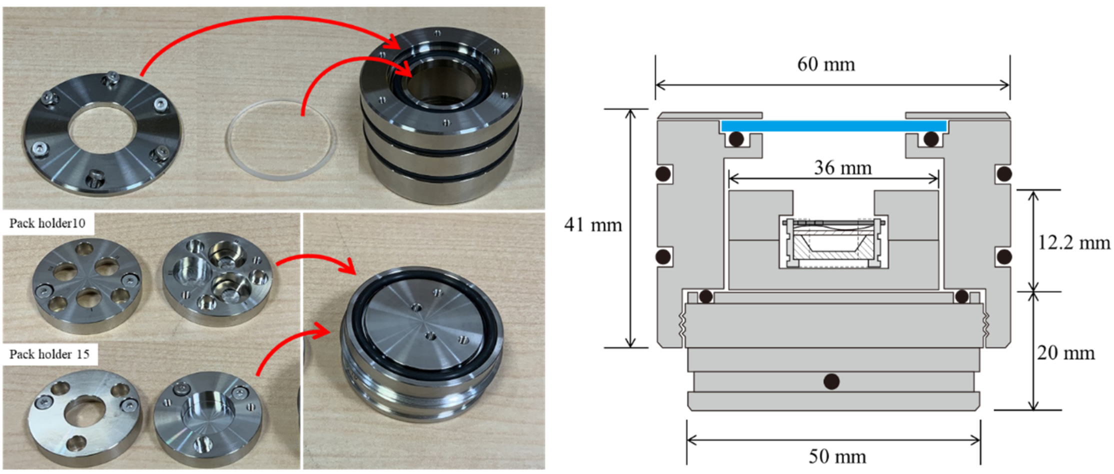

Fig. 25 Design of FFTC for Hayabusa2 sample transfer

3.3 Sample containers for OSIRIS-REx samples

3.3.1 Bulk sample containers (Fig. 26)

The bulk sample container for OSIRIS-REx samples is designed based on that for Hayabusa2 samples, but it is modified with lessons learned from our experience of handling the Hayabusa2 bulk samples. The Hayabusa2 bulk sample container has a heavy lid on top and a smaller diameter in the lower part, which is relatively unstable and risks falling over. The OSIRIS-REx bulk container uses a wave washer and a fix ring as the capsule packs instead of using a heavy lid. In addition, the base of the outer holder has a larger diameter than the upper sapphire dish holder part.

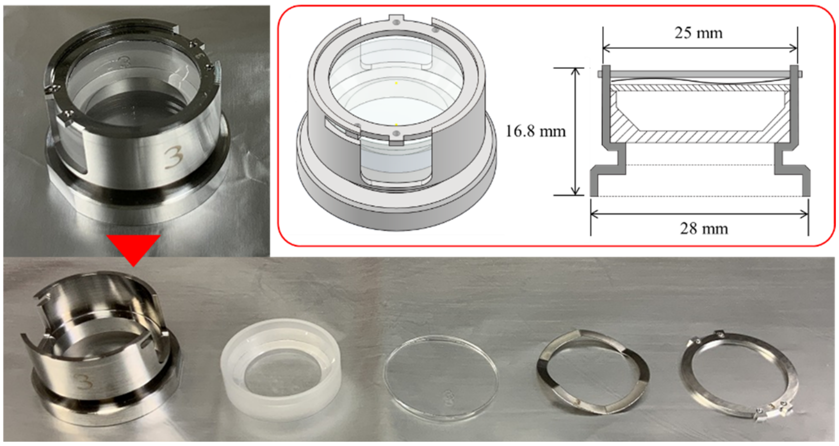

Fig. 26 OSIRIS-REx bulk sample container

3.3.2 Bulk sample transfer container (Fig. 27)

The OSIRIS-REx bulk sample container is enclosed in the sample holder attachment, consisting of the base and lid made of stainless steel (SUS304). The outer holder of the bulk sample transfer container, which is identical to the contact pad holder, was designed and prepared by NASA JSC Curation. We designed the pack holder to fix the capsule pack securely and fit well to the outer holder.

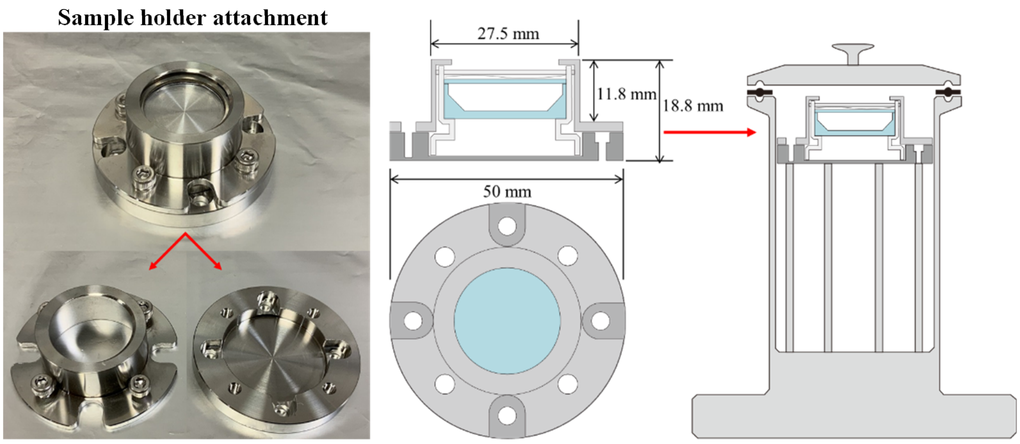

Fig. 27 Sample holder attachment packed in the sample transfer container

4 SUMMARY

The ESCuC received samples collected from the asteroids Itokawa and Ryugu in JAXA sample return missions of Hayabusa and Hayabusa2, respectively, and will receive samples from the asteroid Bennu in NASA OSIRIS-REx mission. Samples are containerized in dedicated sample containers and stored in clean chambers with pure nitrogen conditions. Part of the samples are transferred to other research institutes for detailed analysis without exposure to the terrestrial atmosphere. The ESCuC developed various types of sample containers depending on the purpose of sample handling and the sample size. Samples recovered in the Hayabusa mission are fine particles with tens to hundreds of microns in size. They are first dropped on the quartz or aluminum disks and then transferred to multi-well concavity slides. SEM observation with EDS analysis is essential to confirm if the particles are derived from Itokawa or other contaminants, and the particles are packed in the SEM holder to transfer to SEM-EDS. Samples to be allocated to the other research institutes are moved from the multi-well concavity slides to the other concavity slides in the clamp-sealed transfer container. In recent activities, particles on the multi-well concavity slides are moved to individual sample containers to make the sample storage more permanent and delivery easier.

Samples recovered in the Hayabusa2 mission have variable sizes ranging from fine particles to mm-sized grains with a maximum of 10 mm in longer axis. Three types of sample containers are used to store individual grains and aggregate samples, depending on the sample size. The largest grain is containerized in the specially designed sample container. FFTCs (the O-ring sealed transfer containers) are used to deliver samples to other research institutes. Part of the samples (0.5 wt%) recovered in the NASA OSIRIS-REx mission will be delivered to JAXA in Spring 2024. Bulk sample containers and sample transfer containers were developed in collaboration with NASA JSC curation to contain the OSIRIS-REx samples. The same sample containers with Hayabusa2 samples, capsule packs, and FFTCs will be used to store and deliver the OSIRIS-REx samples.

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