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# Hayabusa-returned sample curation in the Planetary Material Sample Curation Facility of JAXA

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**Abstract**–The Planetary Material Sample Curation Facility of JAXA (PMSCF/JAXA) was established in Sagamihara, Kanagawa, Japan, to curate planetary material samples returned from space in conditions of minimum terrestrial contaminants. The performances for the curation of Hayabusa-returned samples had been checked with a series of comprehensive tests and rehearsals. After the Hayabusa spacecraft had accomplished a round-trip flight to asteroid 25143 Itokawa and returned its reentry capsule to the Earth in June 2010, the reentry capsule was brought back to the PMSCF/JAXA and was put to a series of processes to extract recovered samples from Itokawa. The particles recovered from the sample catcher were analyzed by electron microscope, given their ID, grouped into four categories, and preserved in dimples on quartz slide glasses. Some fraction of them has been distributed for initial analyses at NASA, and will be distributed for international announcement of opportunity (AO), but a certain fraction of them will be preserved in vacuum for future analyses.

## INTRODUCTION

Sample return missions recovering samples from other extraterrestrial bodies have allowed us to make great progress in understanding their origins, formation, and evolution processes. The Apollo missions accomplished six successful landings on the lunar near side, recovered samples there, and brought back 382 kg of lunar samples. To curate the returned lunar samples with lesser terrestrial contamination, the Apollo mission had prepared the Lunar Receiving Laboratory (LRL) at the Johnson Space Center (JSC) of NASA to treat the lunar samples in glove boxes (Allton et al. 1998). Almost 40 years after the Apollo mission, the Stardust spacecraft

successfully returned samples captured nearby comet Wild 2 (Brownlee et al. 2006). They prepared a special clean room to curate the Stardust samples under clean conditions (Zolensky et al. 2008). The Stardust clean room is at a higher cleanliness level than that at the LRL, but it does not contain a glove box to treat the samples in nitrogen condition, as the LRL does.

A sample return mission from an asteroid was first planned in 1985 in Japan. It was accepted as a mission named MUSES-C in 1996. The scientific objectives of MUSES-C spacecraft were to obtain a surface topography, infrared (IR) spectra, X-ray fluorescence spectra of a target asteroid, and try capturing samples on its surface to return the recovered samples to Earth. Its target asteroid had been changed from 4660 Nereus, to 10302 1989ML, and finally to 25143 1998SF36, later named Itokawa. Visible and infrared spectroscopic measurements revealed that the 25143 Itokawa belonged to an S (IV) type asteroid, mainly composed of olivine/pyroxene (Binzel et al. 2001; Dermawan et al. 2002). The spacecraft was launched on 9 May 2003, and traveled to Itokawa to reach an altitude of about 20 km (Gate position) of the asteroid in September 2005 (Fujiwara et al. 2006). Observational results from a near-infrared spectrometer and an X-ray fluorescence spectrometer on Hayabusa indicated that asteroid Itokawa was mainly composed of olivine and related to ordinary chondrites, LL5–6 (Abe et al. 2006; Okada et al. 2006). After remote sensing analyses around Itokawa for about two months, Hayabusa carried out touchdowns twice onto the surface of MUSES-C for sample collections on 20 and 26 November 2005 (Yano et al. 2006). It returned to Earth to release its reentry capsule on 13 June 2010, after a long round trip from Itokawa (Abe et al. 2011).

A basic concept for Hayabusa-returned sample curation was planned before its launch, but it had been reconsidered with help from an advisory committee for specifications of the PMSCF/JAXA between December 2005 and February 2007. Detailed specifications of the facility and clean chambers and instruments installed in the facility were considered in parallel, and the facility was completed in March 2008. In the following section, we describe the concept of the curation facility and details of the actual procedures being carried out in the facility.

## SPECIFICATIONS OF THE PMSCF/JAXA

### The Concept of Sample Condition Controls of Hayabusa Mission

Chemical and physical conditions of returned samples are affected by gas, liquid, and solid terrestrial contamination on the ground before launch, and after atmospheric reentry, as well as temperature, magnetic, electric condition, shock pressure, and acceleration they would experience in space and during atmospheric entry. For solid contamination, material constituents of a sample container of Hayabusa were limited to aluminum alloy (A6061) coated with pure aluminum, stainless steel (304), Viton, $Al_2O_3$ glass, and Teflon coated onto the aluminum surface. Before launch, every part of the sample container was cleaned in 2-propanol using an ultrasonic cleaner, installed in a class 10,000 clean room in Fed. Std. 209D. A contamination coupon made of $Al_2O_3$ glass was set inside a sample catcher during the final assemblage for flight model of the spacecraft, thus contamination after that point was monitored with the coupon. We designed the PMSCF/JAXA to minimize contaminations from natural detritus, artificial particles containing especially boron, rare earth elements (REEs), and lead, for analyses of returned samples from Itokawa. Terrestrial gas and liquid contamination on the samples might have happened after the touchdown sampling. Thus, the sample container was designed to seal the samples after the sampling as described later. The rock samples and gases possibly released from them were expected to have maintained their original conditions in the container. After returning to Earth, terrestrial atmosphere, however, could be permeable through the double O-rings seal. The pressure increase due to gas permeating through the double O-rings seal was estimated to be $<1$ Pa for 100 h after the reentry. The temperature condition of the returned samples was managed to be kept as low as possible. It should have been less than 0 °C during the return trip in interplanetary space. During atmospheric entry, the sample container was designed to be preserved under 80 °C within the reentry capsule protected by its ablators made of carbon fiber-reinforced plastic (CFRP). The thermal condition after the recovery on the Australian desert until introduction to the PMSCF/JAXA was monitored with a temperature logger attached to a transportation box for the reentry capsule. The data of the logger showed that the sample container had been kept under 30 °C. The PMSCF/JAXA clean rooms were kept at less than 26 °C. The magnetic condition of Hayabusa-returned samples should have been disturbed during a return trip to Earth due to ion engine operation, which is one of the propulsion systems on Hayabusa, because the microwave ionization system of the ion engine causes strong magnetism. Among those conditions, electric disturbance and shock from atmospheric entry, landing and transportation, which might affect the samples, are poorly understood. In the following section, we detail the curation facility and clean chambers in which the Hayabusa samples were treated.

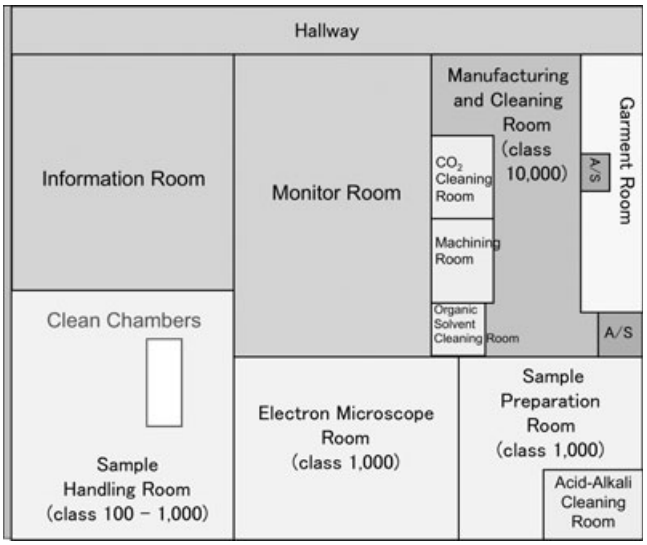


Fig. 1. A schematic viewgraph of clean rooms of the PMSCF/JAXA. They consist of four clean rooms: a sample handling room, an electron microscope room, a sample preparation room, and a manufacturing and cleaning room.

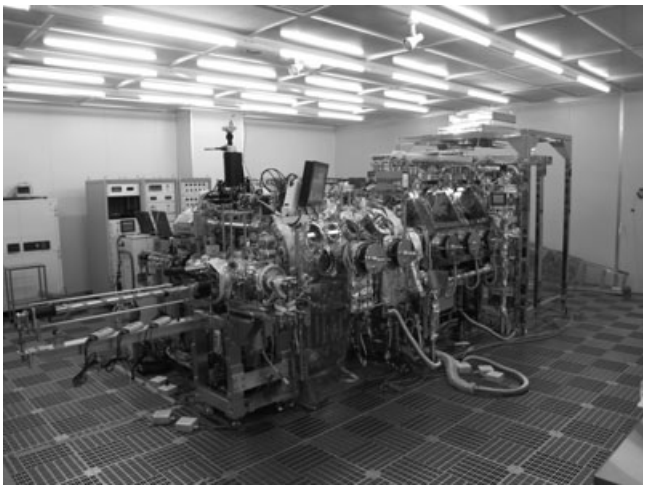

Fig. 2. A photograph of clean chambers in the PMSCF/JAXA. They consist of two main chambers: No. 1 (left) and No. 2 (right) in the planetary sample handling room of 100–1000 in clean class level of Fed. Std. 209D.

## Clean Rooms

We designed the PMSCF/JAXA to diminish terrestrial contaminants from its environment. It consists of four clean rooms of different clean levels: a planetary sample handling room (class 100–1000), an electron microscope room (class 1000), a sample preparation room (class 1000), and a manufacturing and cleaning room (class 10,000) (Fig. 1). All filters used in filter fan units of the clean rooms were made of polytetrafluoroethylene (PTFE), and a chemical filter is set to absorb acid gases such as halogen, sulfate, and nitrate and elemental boron and borate from ambient air introduced into the clean rooms. Thus, such chemical gases and boron are minimized in the clean rooms.

Four small rooms for special uses were designed to exhaust the air inside their rooms independently to outside of the clean rooms to protect the other clean rooms from chemical and particle contamination. An acid and alkali treatment room was set in the sample preparation room for cleaning using acid and alkali. An organic solvent cleaning room was prepared in the manufacturing and cleaning room for cleaning using organic solvents. A machining room and a $CO_2$ cleaning room were also situated in the manufacturing and cleaning room for milling and drilling machines using motor oil and two different types of cleaners to blow off particles and organics on metal surfaces, respectively. Additionally, there was a basement for equipment, which cannot be set in the clean rooms, such as roughing pumps for vacuum systems, a compressed air supply system, an ultra pure water supply system, and nitrogen purifiers.

## Clean Chambers

The basic concept for solid and gas contamination control was that the sample should be exposed to the lowest particle level in vacuum or nitrogen condition. Thus, we prepared two clean chambers, No. 1 and No. 2, in the sample handling room of the PMSCF/JAXA (Fig. 2). They were mainly made of stainless steel 304, and their inside walls were treated by the method of electrochemical polishing. They were baked in vacuum to more than 120 °C before and after the installation to reduce contaminant gases absorbed on them under high vacuum condition. Both of the chambers were equipped with vacuum systems composed of turbo molecular pumps (TMPs) and dry scroll pumps. They were also equipped with pure nitrogen supply systems by two methods, a cyclic type nitrogen purifier and a flow type nitrogen purifier. The former was directly connected to each of the chambers to circulate nitrogen with them and exclude interference gases such as $H_2O$, $O_2$, and hydrocarbon from the nitrogen gas. The latter was equipped for the whole supplied nitrogen to the facility including the clean chambers, to purify evaporated liquid nitrogen supplied from outside the building. Additionally, the chambers were equipped with pressure control systems to keep their inner pressures positive relative to atmospheric pressure. Due to these systems, environments from ultrahigh vacuum to atmospheric pressure of purified nitrogen can be realized in chamber No. 1. This is the chamber in which the sample container

of Hayabusa was opened with a special container opening system. Chamber No. 1 was equipped with an evacuation system composed of two tandem TMPs and a dry scroll pump for roughing. The container opening system was composed of a set of four movable shafts, which can be connected with an inner lid of the container, touch-type displacement sensors, and laser displacement sensors. The sensors in the system monitor the displacements of the shafts and load cells to estimate stress on them. Residual gas expanding into the chamber from the container was collected in residual gas sampling bottles made of stainless steel, which were equipped with an introduction line to chamber No. 1. A lower part of the sample container was stored in cabinet No. 1 under vacuum condition. For the transportation of the sample container, a transportation chamber was equipped between chamber No. 1 and cabinet No. 1. It had two magnetic coupling transfer rods by which the container could be transported from chamber No. 1 to cabinet No. 1 via the transportation chamber either in vacuum or in atmospheric pressure nitrogen condition. Chamber No. 1 was also equipped with Viton gloves through gate valves so that we could handle the sample container and catcher with special tools and jigs.

The sample catcher was sent to the clean chamber No. 2 for further handling such as recovery of solid sample grains from the catcher and those samples were treated for further sample analysis. An ultra long working distance optical microscope was installed to the clean chambers to observe inside through glass viewports of clean chamber No. 1 and glass windows of clean chamber No. 2. The samples could be handled in an atmospheric pressure condition of purified nitrogen with special tools and jigs in clean chamber No. 2, which was equipped with six Viton gloves. Samples once cataloged were stored in vacuum of cabinet No. 2 or 3 attached to chamber No. 2. Both chambers No. 1 and No. 2 contain ultraviolet (UV) neutralization lamps to compensate electrostatic charge, which should occur in the pure nitrogen condition. Also, an alpha-ray neutralizer of $^{210}$Po radioactive source was prepared for the same purpose. From the contamination control point of view, we managed the clean chambers to restrict materials acceptable for introducing into them only as stainless steel (304 and 316), pure aluminum and aluminum alloy (A6061), quartz glass, PTFE, and Viton. It is acceptable that gold and copper are used for materials of sample holders, borosilicate glass for containers of less important items, and polyetheretherketone (PEEK) for electric connectors.

There was another glove box in the electron microscope room. It had two Viton gloves for handling and an air lock to introduce tools and jigs without breaking the environment inside the box. It could be evacuated to be refilled with nitrogen, also flow nitrogen gas and control its inner pressure not only positive to the clean room air but also negative to the air.

## Other Equipment

### *Electron Microscope*

A field-emission type scanning electron microscope, Hitachi S-4300SE/N, equipped with energy dispersive X-ray spectrometer, Oxford Instruments INCA X-act Energy 350 (FESEM-EDS) was installed in the electron microscope room. It was prepared for initial description of planetary material samples, and can perform low vacuum observation mode for observing insulators such as silicates without conductive coatings. We also prepared a custom-made sample holder, basically made of copper coated with gold and has a cap with a Viton O-ring to seal samples inside from terrestrial atmosphere. With this holder and a sample exchange chamber of the FESEM, which equips a mechanism to open its cap, we transferred samples from the clean chamber to the FESEM without exposing to terrestrial atmosphere. The FESEM contains a specially designed micromanipulator device for handling a minute particle on the sample holder in a sample chamber of the FESEM, observing the particle in an electron image of a high magnification, as described in the following section.

### *Instruments for Cleaning and Checking Cleanliness*

To keep cleanliness of the clean chamber, a series of cleaning instruments were prepared for cleaning tools, containers, and jigs used in the clean chamber. Four ultra sonic cleaners were set in the manufacturing and cleaning room and the sample preparation room. They were 38, 35 and 98, 100, and 950 kHz in vibration frequencies and capable of performing overflow cleaning with ultra pure water supply system in the rooms. To perform acid and alkali treatment cleanly, a clean evaporator was installed in the acid and alkali cleaning room. It consists of two chambers made of polyvinyl chloride equipped with seat heaters covered by PTFE and an independent intake and exhaust fan system with a series of filters for ambient air intake and a water scrubber for the exhaust line. We also prepared an atmospheric-pressure plasma cleaner and a $CO_2$ blast cleaner to remove particles and organics on the surfaces of materials, and a UV ozone cleaner to sterilize bacteria and remove organics on the materials' surfaces.

To check the environment of the clean chambers and attached chambers, two types of mass spectrometers were set near the clean chambers. One was an atmospheric pressure ionization mass spectrometer (API-MS) and the other was a differential pumping quadrupole mass spectrometer (Q-MS). The former was

capable of analyzing impurities in nitrogen gas at ppb levels, and the latter was suitable to analyze those more than sub-ppm. A Fourier transform infrared spectroscopy (FT-IR) and a dynamic contact angle meter were prepared to check cleanliness of surfaces of materials.

### *Electrostatically Controlled Micromanipulation System*

Because the Hayabusa spacecraft did not fire a bullet, to initiate surface sampling, during the first and the second touchdowns onto the surface of Itokawa (Yano et al. 2006), samples captured by Hayabusa were small in both total amount and size. Thus, specially designed micromanipulator systems were added in an ultra pure nitrogen environment of clean chamber No. 2. They were basically composed of a sample stage and right and left probe stages combined with two optical microscopes, which can observe the top of the sample stage from vertical and tilted angles. The sample stage has four axes movements, *X*, *Y*, *Z*, and rotation, and voltage can be applied to the stage from −32 to +32 volts. Both right and left probe stages have five axes movements, *X*, *Y*, *Z*, rotation, and tilt, and were equipped with quartz glass probes with electrodes inside made of platinum wires, through which we can apply voltage from −110 to +110 volts. With this system, we controlled the electrostatic conditions of the probes and the stage to handle a small particle by the electrostatic force acting on it. This method was effective in such a condition like the chamber filled with purified nitrogen in which insulators tend to have electrostatic charges. Some parts of theories for electrostatic particle manipulation were mentioned in Saito et al. (2007).

An electrostatically controlled micromanipulation system composed of commercial based instruments was also prepared outside the clean chamber. It was installed earlier than any other manipulator and used for developing the electrostatic control method. It could be set both in a clean booth of the electron microscope room and in the glove box filled with nitrogen in the same room, and used for tests of the manipulation inside clean chamber No. 2 and particle manipulation outside the chamber.

An electrostatically controlled micromanipulator was also installed in the FESEM of the PMSCF/JAXA, consisting of two probe stages of *X*, *Y*, and *Z* movements powered by piezoelectric motors, which can be operated by a note PC. The probes were the same as those used for the manipulator in clean chamber No. 2, and voltage can be applied from −110 to +110 volts. We are now developing a technique to pick up particles less than 10 μm from a PTFE spatula and the SEM holder.

## PERFORMANCE CHECK AND REHEARSAL

### Cleanliness Checks for the Clean Rooms and the Clean Chambers

Particle cleanliness levels of the clean rooms were investigated after finishing their constructions. Results of the investigation showed that the actual cleanliness level for each clean room was much smaller than its specification.

After the installation, the clean chambers were baked at about 120 °C for three days. After baking, the vacuum condition of chamber No. 1 reached $7.2 \times 10^{-8}$Pa. Abundances of each residual gas component (e.g., $H_2O$, $O_2$, Ar, etc.) measured by the residual gas analyzer of Q-MS (RGA-QMS) were lower than the detection limit of the RGA-QMS (i.e., lower than $1.0 \times 10^{-14}$ amps) except for mass number 1(H), 2($H_2$), and 14(N). The vacuum condition of the chamber No. 2 reached $3.2 \times 10^{-6}$ Pa, which is around two orders of magnitude higher than that of chamber No. 1. One of the reasons for that vacuum difference between chambers No. 1 and No. 2 is the vacuum system difference between them. Chamber No. 1 was equipped with two tandem TMPs, while chamber No. 2 was pumped by a single TMP. The other possible reason might be degassing from six Viton gloves in chamber No. 2, compared with chamber No. 1, which equips gate valves for the glove ports to prevent them from being exposed directly to chamber No. 1. The cleanliness of chamber No. 2 was examined by the API-MS after baking. The major impurity gas components decrease to approximately 1 ppb of oxygen and approximately 14 ppb of water in chamber No. 2.

### Noble Gas Abundances in High Purity Nitrogen Gas Used in the PMSCF/JAXA

The terrestrial atmosphere is the most serious source of noble gas contamination to extraterrestrial samples such as meteorites and interplanetary dust particles. We searched for nitrogen gas with low concentrations of noble gases. Concentrations of noble gases in nitrogen gas evaporated from liquid nitrogen produced by the Taiyo Nippon Sanso Corporation are plotted in Fig. 3; ELF ($N_2$-gas)-1 and -2, and $LN_2$ Tank-1 and -2. Concentrations in air are also plotted in Fig. 3 for comparison. The nitrogen gas was sampled from headspaces of liquid nitrogen storage tanks in the company: ELF was a small tank of 175 L, but the liquid nitrogen ($LN_2$) tank was a large one in which liquid nitrogen produced by the company was stored. Noble gases were measured with a modified-VG5400 (MS-III) at the Geochemical Research Center, University of

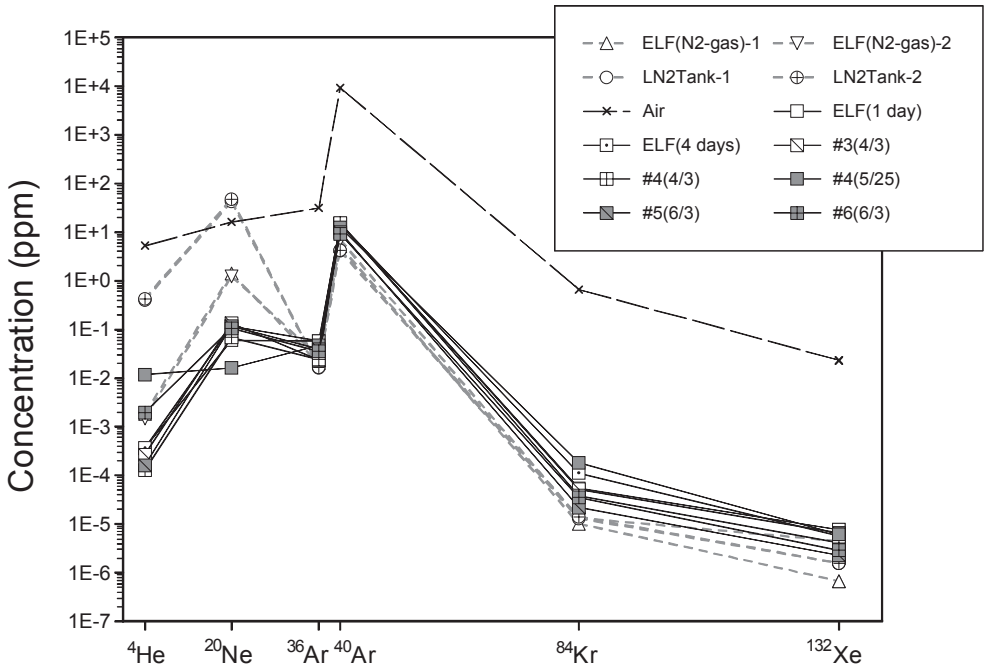


Fig. 3. Noble gas concentrations in nitrogen gas evaporated from liquid nitrogen and accumulated in headspace of tanks for storage of liquid nitrogen. Noble gas concentrations in atmosphere are shown for comparison. Four samples, from ELF ($N_2$-gas) to $LN_2$ Tank, were collected in metal bottles from an ELF tank and a large tank ($LN_2$ Tank) at the company (Taiyo Nippon Sanso Corporation) producing the liquid nitrogen. Seven samples, from ELF (1 day) to #6 (6/3), were in the clean chambers, to which the nitrogen gas is supplied from ELF tanks after purging the headspace of ELF tanks.

Tokyo. Concentrations of Ar, Kr, and Xe in the nitrogen gases from both tanks were almost the same among them, and three to five orders of magnitude lower than those in air. In contrast to the low and similar concentrations of the heavier noble gases, He and Ne concentrations were different among the gases from two tanks and rather high compared with those of Ar, Kr, and Xe, especially Ne, e.g., the concentration of Ne in the nitrogen gas from $LN_2$ Tank was higher than the atmospheric value. We thought that the relative enrichments in He and Ne might be due to accumulation of those gases in the headspace of tanks, because He and Ne would be partitioned in gas phase, while Ar-Xe would be liquid at the liquid nitrogen temperature. This is demonstrated in Fig. 3, where low concentrations of all noble gases measured for nitrogen gas sampled after purging of enough amount of nitrogen gas from the headspace (ELF-1 and 4 days, and #3 - #6). The clean chambers noted above were filled with nitrogen gas with low noble gas concentrations.

Degassing rates of noble gases from a Viton sheet and noble gas permeability through it were also investigated. One side of the Viton sheet was coated with Teflon. The Viton sheet was set to a window of vacuum chamber, and then gases from the sheet were accumulated to measure accumulation rates of noble gases. The accumulation was carried out in two cases (1) both sides of the Viton sheet were connected to the accumulation chamber and (2) one side opposite to the gas accumulation was exposed to atmosphere. The case (1) shows degassing rates from the Viton sheet. It is shown that baking of the Viton effectively reduces degassing rates. However, noble gases can easily dissolve into Viton when it is exposed to atmosphere, and degassing increases again. Moreover, different degassing rates from the Teflon-coated side and non-coated one were not observed. In the case of (2), only He and Ne clearly increased, indicating permeation of He and Ne from atmosphere through the Viton sheet with a rate of $\leq 10^{-9}$ cm$^3$ cm$^{-2}$ min$^{-1}$. Permeation of heavier noble gases (Ar, Kr, and Xe) seems to be much smaller than those of He and Ne. We used the Viton glove for the clean chamber after baking under vacuum condition, because no other convenient materials are available at present.

## Checking Cleanliness Obtained by Cleaning Methods in the PMSCF/JAXA

Equipment used in the clean chamber was prepared by a series of cleaning processes. A typical procedure was a series of ultra sonic cleanings for all materials such as

metals, glasses, and Teflon. The first step of ultrasonic cleaning was to remove mainly organic impurities. Its solvent was 2-propanol, but some other solvents were chosen by necessity. The cleaning in 2-propanol was repeated three times with the 40 kHz frequency band for 15–20 min, changing the solvent each time. The next step of ultrasonic cleaning was to remove particles and ions. Its solvent was ultrapure water, overflowing from the ultrasonic bath to keep providing fresh water. The frequencies for the cleaning are 35, 98, and 950 kHz bands. The cleaning was repeated twice at each frequency for 20–30 min. After the series of ultrasonic cleanings, water on the cleaned parts was removed by the purified nitrogen gas blow or air in clean booth. For quartz glass, an additional acid and alkali treatment was performed after the series of ultra sonic cleanings. They were washed by heated alkali and acid solutions to remove organics, ions, and particles again, as quartz is resistant to strong acid and alkali. The washing method was a batch cleaning, and its procedure was our modified RCA method, originally for washing semiconductor wafers. The alkali and acid solutions were used twice, respectively, with ultrapure water rinse.

We checked the cleanliness of the material after the cleanings. Contaminant particles on the material were checked with optical microscopic observations. So far, we have confirmed that contaminant particles larger than a few micrometer were almost absent after the series of cleanings in most cases. Such observations were also made with the cameras of the micromanipulation system in the clean chamber. FESEM observation was also done for cases of the sample holder for FESEM. When contaminant particles were found on the holder, additional cleaning such as $CO_2$ blast cleaning was performed to remove them.

Contaminant ions and organics such as machine oil, grease, and human sebum were checked by the other method. Their abundances were evaluated with the boiled solution of test quartz pieces in ultrapure water, after the acid and alkali and treatment. Twenty cations, Al, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Si, Sr, Ti, and Zn were examined with inductively coupled plasma mass spectrometry (ICP-MS) and atomic absorption spectrometry (AAS). Additionally, six anions, those are F, Cl, $NO_2$, $NO_3$, $PO_4$, and $SO_4$, were also evaluated with ion chromatography (IC). The estimation of the actual background level after the series of cleaning is still under investigation.

## Structures of the Reentry Capsule and the Sample Container of Hayabusa

The Hayabusa spacecraft carried a reentry capsule in which the samples recovered from Asteroid Itokawa were enclosed. The capsule was saucer-shaped, around 40 cm in width and around 22 cm in height. It was covered with front- and back-side ablators made of CFRP to protect the sample container, a parachute, and related devices from atmospheric entry heating. The sample container was set in the center of the reentry capsule. Figure 4 shows the structure of the sample container. It was shaped like a cylinder with a large flange with sizes of 12 cm in width and around 13 cm in height. It mainly consists of an outer lid equipped with latches, an inner lid, a frame for latches, a sample container, and a sample catcher. Most parts are aluminum alloy (A6061) coated by pure aluminum, and surfaces of the parts which should cause friction with other parts were treated with a special Tufram coating, which treated the surface of aluminum alloy to be alumite and coated it with Teflon to obtain smooth movements (McMurtrey 1985).

The initial position of the sample catcher together with the lids was on the top of a sampler horn, and it was transferred from the initial position to the sample container set in the reentry capsule during the return from Itokawa to Earth in 2007. With this procedure, the sample container should have been sealed from the environment outside the container with double Viton O-rings attached to the inner lid until the container was opened in clean chamber No. 1. The sample catcher was divided into three rooms, rooms A, B, and a rotational cylinder (Fig. 4). Initial position of the rotational cylinder had been opened for the room B, and the first touchdown to the surface of Itokawa had been performed with this setting. Before the second touchdown, the part of rotational cylinder was rotated to open for the room A to capture particles with the second touchdown. After the second touchdown, the cylinder was rotated to close all the openings to both rooms A and B.

We prepared a simulated model of the sample container for a series of processes rehearsals. In the following section, we describe the rehearsal processes held in the facility.

## Rehearsal of a Series of Processes of the Sample Container in the Facility

We had performed a series of rehearsals for the acceptance of the reentry capsule of Hayabusa, extraction of the sample container, opening of the container, and recovery of samples from its sample catcher from April 2009 to March 2010, before the Hayabusa reentry capsule returned to the Earth in June 2010. A brief flowchart of the rehearsal is shown in Fig. 5. Each step of the processes was repeatedly performed in the same setting as the real part until the technical problems were clarified. Basically, all of the procedures were described as

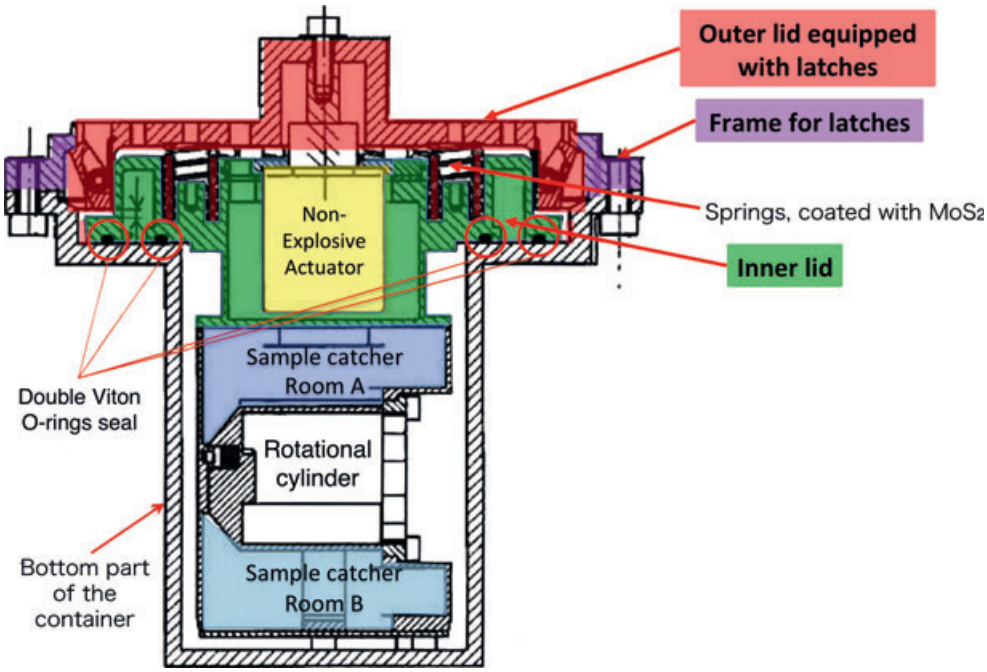


Fig. 4. A schematic view of a cross-section of the sample container of Hayabusa. It mainly consists of an outer lid equipped with latches, an inner lid, a frame for latches, a nonexplosive actuator, and a sample catcher. The sample catcher is divided into three rooms: room A, room B, and a rotational cylinder.

manuals, and the manuals were revised after the rehearsals based on the results to make up the procedures for the actual Hayabusa sample return.

### *X-Ray Computed Tomography (CT)*

The first step after the acceptance of the reentry capsule was an X-ray computed tomography (CT) imaging of the whole capsule. With this process, the condition of the sample container and the positions of screw bolts to connect the backside ablator with the container were confirmed. After removal of the electronic box and ablators, the container was examined with the X-ray CT again to confirm the conditions of latches, seal of the container, and the position of the rotational cylinder. Additionally, existence of samples inside the sample catcher was expected to be confirmed. The X-ray CT used for the rehearsal and the actual Hayabusa-returned capsule was a Toshiba TOSCANER 24200RE, situated in Chofu Aerospace Center Aerodrome Branch of JAXA. We brought the simulated sample container, in which a single grain of several mm and several particles of 200–250 μm of San Carlos olivine and stainless steel spheres of 200–250 μm were pressed together, and test investigations were performed using the X-ray CT instrument. Figure 6 shows the X-ray CT images of the test investigations. These X-ray CT images indicate that conditions of the latches and double Viton O-rings could be confirmed with these images and silicate particles larger than 200 μm could be recognized.

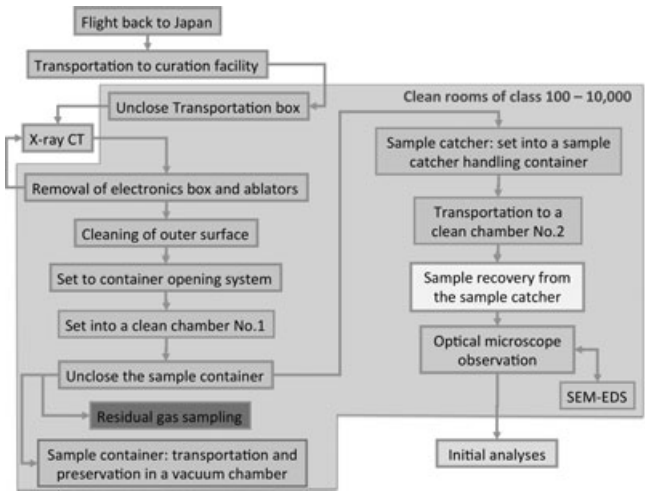


Fig. 5. A flowchart of the rehearsal for a series of processes from an acceptance of the reentry capsule of Hayabusa to a recovery of samples. It took about a year to perform each process of the rehearsal repeatedly.

### *Removal of the Ablators and Cleaning the Container and Check the Cleanliness*

The backside ablator could be removed only after machining to expose the tops of eight screw bolts, which

Fig. 6. X-ray CT images of the simulated sample container. a) The X-ray CT image of the cross-section of the inner lid with latches. This image indicates that latches are open to hold the lid tightly to the frame. b) That of the inner lid with the double Viton O-rings. The O-rings look fixed to the trench of the inner lid to seal the container. c) The bottom of the sample container. This image shows that San Carlos olivine particles and stainless steel spheres of 200–250 μm in the container can be recognized with the X-ray CT.

connect the ablator with the outer lid of the container, and then unscrewing all the bolts. We had installed the milling machine in the manufacturing and cleaning room. With these tools, we performed a rehearsal of the

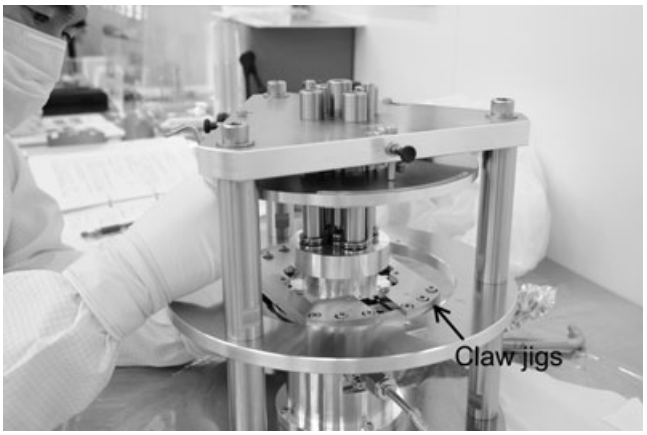


Fig. 7. A snapshot of a rehearsal for removal of the outer lid, the frame for latches, and NEA from a simulated sample container with special jigs and tools. The container was situated in the bottom of the catcher opening jigs, and its top parts were covered by claw jigs. It contains a vacuum flange, through which we could check the condition inside the container.

milling work using a simulated ablator, and succeeded in drilling to expose the top of the screw. For machining the real part, we prepared special jigs to secure the container on the table of milling machine.

We had a rehearsal to clean the surface of the simulated container with dry wiping, wet wiping using ultra pure water and ethanol, dry ice blast cleaning, and atmospheric pressure plasma cleaning. The surface of the container was wiped with special swabs for cleaning, and the swabs were smeared over the quartz slide glasses to check their cleanliness by an optical stereomicroscope, the dynamic contact angle meter, and the FT-IR.

### *Setting the Sample Container to the Clean Chamber No. 1*

We had a rehearsal to perform a series of processes before setting the sample container to clean chamber No. 1. At first, we removed the outer lid, the frame for latches, springs, a nonexplosive actuator (NEA) and screw bolts, which combine such parts together from the sample container. We prepared special jigs to remove such parts while keeping the seal of the container (Fig. 7). After the removal of the parts mentioned above, we connected a joint jig on the inner lid to combine the inner lid to the shafts of the container opening system. Then the container was set to the container opening system, and the inner lid was fixed with the four shafts of the system to keep its seal. The seal condition of the container was checked with the custom-made container, which had a vacuum flange and enabled us to check its vacuum condition through the flange. In the rehearsal, we confirmed that there was no leak during the series of processes.

### *Opening the Sample Container and Recovering the Residual Gas*

The inner pressure of the sample container of real part is unknown, thus we had to estimate the pressure and adjust that of the clean chamber to the estimated pressure so as not to disturb samples in the container due to the gas flow from or to the container because of the difference in pressure between them. We changed both inner pressure of the container and the loading forces of the cover flange, and monitored loads and displacements of the four shafts of the opening system.

Because the load of the inner lid with the springs above is around 160 kgf, we pressed 40 kgf for each of the four shafts on the container opening system for an initial position of opening. After 150 μm displacement from the initial position, the load at that position was analyzed. The difference in pressure between inside and outside the container should make differences in loads on the four shafts to hold the inner lid with double Viton O-rings. Lower external pressure makes lower load, because the loading force on the inner lid decreases as the external pressure decreases. Although the reproducibility of the load in the same condition was not good, it is possible to detect the difference in the inner pressure more than $5 \times 10^4$ Pa from the difference of the load in the different external pressure. We also rehearsed opening the container in conditions in which the double Viton O-ring could have been harmed accidentally or hardened due to severe conditions in interplanetary space. As a result, those problems of initial conditions did not seem to make differences in values of loads from the case of Viton O-rings in the normal condition.

When the container was opened in clean chamber No. 1, residual gases which should be released from the container were captured in the residual gas sampling bottles in clean chamber No. 1. The gas bottles had been baked $>180$ °C to decrease the background interferences. The background interference of the baked gas bottles was checked in the same noble gas mass spectrometer in the University of Tokyo and was small enough for the noble gas analyses.

### *Handling the Sample Container and the Sample Catcher to Recover Samples*

In the rehearsal, we checked movements of the transfer rods of the transportation chamber. The transportation chamber was set next to clean chamber No. 1 and used for transferring the sample container from the sample opening system to cabinet No. 1 via the transportation chamber either in the atmospheric pressure nitrogen condition or in the vacuum condition. In the chamber, we also checked and photographed the inside of the container though a glass viewport of the top of the chamber with the ultra-long working distance microscope, which was set above the viewport.

After the container transportation, the gate valve between the transportation chamber and clean chamber No. 1 was closed. The condition of clean chamber No. 1 was then set to atmospheric pressure nitrogen, and the sample catcher together with the inner lid was transported with a special rod manually via two Viton gloves and set to the catcher handling jigs to introduce into a sample handling container. Then, the inner lid was separated off from the sample catcher. Screw bolts to connect the inner lid and the sample catcher were locked by stainless steel wires, thus we cut and removed the wires from the bolts with pliers before we unscrewed the bolts. With this procedure, we got the first look of the catcher room A in real part. We therefore watched the inside of room A through a glass viewport of the top of chamber No. 1 with the ultra-long working distance microscope that was set above the viewport. Then the sample catcher was enclosed into the sample catcher handling container with the catcher handling jigs, and sent to the clean chamber No. 2 via a gate valve between chambers No. 1 and 2. In chamber No. 2, we had a rehearsal to scoop out samples inside the catcher to a petri dish made of synthetic quartz glass with a special spatula made of PTFE.

### *Handling Test of Simulated Particles with the Electrostatically Controlled Micromanipulator*

In the rehearsal, we first tried to handle simulated particles in the clean room air condition using a reflecting and polarizing microscope with a commercial based, oil hydraulics micromanipulator setting a quartz glass probe with an internal platinum wire connected with a direct-current (DC) power source. We used San Carlos olivine and synthetic nickel olivine of 30–500 μm in size as the simulated particles. Olivine particles of 30–100 μm can be handled with the manipulator in the electronic condition between $-12$ volts and $+16$ volts DC for the probe voltage. Next, we brought all the instruments above into the glove box in the electron microscope room to rehearse manipulation in the same hardware setting as in the first one in nitrogen condition. We handled olivine particles of 50–150 μm under the electric condition between $-50$ volts and $-100$ volts DC for the probe and $+30$ volts DC for the stage.

Finally, we rehearsed the electrostatically controlled micromanipulation system in clean chamber No. 2. We used a quartz glass particle and a nickel olivine particle of 100 μm in size as simulated particles for handling. They were put on a quartz slide glass, a quartz dish, and an SEM holder made of copper coated by gold. We

could handle the particles in the electric condition between −50 and −100 volts DC for the probe voltage and between 0 and +30 volts DC for the stage. We also checked the handling of the nickel olivine particle inside the simulated sample catcher introduced in clean chamber No. 2, and confirmed that we could pick up and release the particle with the manipulator system.

## ACTUAL HAYABUSA CAPSULE PROCESS

### Processes Before Opening a Sample Container

On 13 June 2010, the reentry capsule of Hayabusa successfully landed in the Woomera Prohibited Area (WPA) of Australia and the recovered capsule and its heat shields arrived at the Planetary Material Sample Curation Facility of JAXA in Sagamihara, Japan on 18 June (Abe et al. 2011). The processes were performed following the flowchart of the rehearsal as shown previously (Fig. 5).

First, the transportation box was introduced into the manufacturing and cleaning room and opened to extract the reentry capsule sealed in plastic bag with nitrogen. The nitrogen gas in the bag was recovered in a glass cylinder just in case. We brought the capsule to the X-ray CT facility in Chofu to confirm the conditions of the latches, the Viton O-rings, and the rotational cylinder in the sample catcher. After it was recognized that there was no trouble in their conditions of the container, the capsule was brought back to the manufacturing and cleaning room in the curation facility. Then, the supporting frame of the capsule and the backside ablator were removed from the container with the hand tools and the milling machine. After the procedure above, the container was brought to the second X-ray CT at Chofu to confirm the condition inside the sample catcher. Unfortunately, the second X-ray CT showed no sign of a recovered sample. After the container was back in the clean room in Sagamihara from the second X-ray CT, a thermosensor, which had been attached below the outer surface of the container's flange, was removed with hand tools. Also, terrestrial dust and chips of the ablator, which existed in the gap between the inner and outer lids and the container's flange, was cleaned with a special vacuum cleaner. Then, the outer surface of the container was cleaned with a series of processes described below. First, it was wiped with clean cloths wetted with 2-propanol and ultra pure water. Second, the surface of the container was blown with a dry ice blast cleaner. Finally, its surface was cleaned with the atmospheric pressure plasma cleaner (Tendero et al. 2006). For the confirmation of cleanliness of the surface of the container, the cleaned surface of the container was wiped with swabs for cleaning, one of the swabs was observed with an optical microscope, and other swabs were wiped on clean slide glasses to be checked by the dynamic contact angle meter and the FT-IR. All three tests showed no sign of contamination. Next, the container was sent to the class 1000 sample preparation room and fixed to the container opening jigs as shown in the rehearsal section and its frame for latches (the purple part shown in Fig. 4) was removed maintaining the seal of the container. Its inner lid was held with claw jigs (shown in Fig. 7), and then its outer lid (the red part shown in Fig. 4) was removed from the container. Finally, the gap between the inner lid and the container's flange was exposed to be cleaned up with the special vacuum cleaner.

Subsequently, the container with the claw jigs was removed from the opening jigs and fixed to the container opening system. After its inner lid was held to the container's flange with four shafts via a joint jig, the claw jigs were removed from the container's flange. Then the container opening system with the container joined clean chamber No. 1, while continuously purging pure nitrogen from clean chamber No. 1. All the processes described above were finished by 20 June, within seven days after the capsule reentry, which had been performed by 24 hours' workflow with eight workers. The environment of clean chamber No. 1 was purified with a cyclic type nitrogen purifier for the night of 20 June.

### Opening the Sample Container

The container opening system contains sets of load cells and displacement sensors for four axes. As described in the rehearsal section, we had data of tendencies of loads and displacements for different pressure levels of the container. The trend of data of the load cells and the displacement sensors had been recorded as cyan and red lines shown in Fig. 8.

In the case of the actual returned container, the inner lid was pulled slightly with the sensors recorded on 21 June and the data were compared with the rehearsal data (black line in Fig. 8). A comparison with the data obtained in rehearsals and those in the actual case indicates that the inner pressure of the actual container was between vacuum and atmospheric pressure. The reproducibility of load to displacement was so poor in the rehearsal, and additionally, the shape of the container of the flight model was different from what we had used for the rehearsal because of a small cutout under the flange of the container where a thermosensor was attached. Therefore, we could only estimate whether it would be vacuum or atmospheric pressure. Based on this estimation, when we opened the container in clean

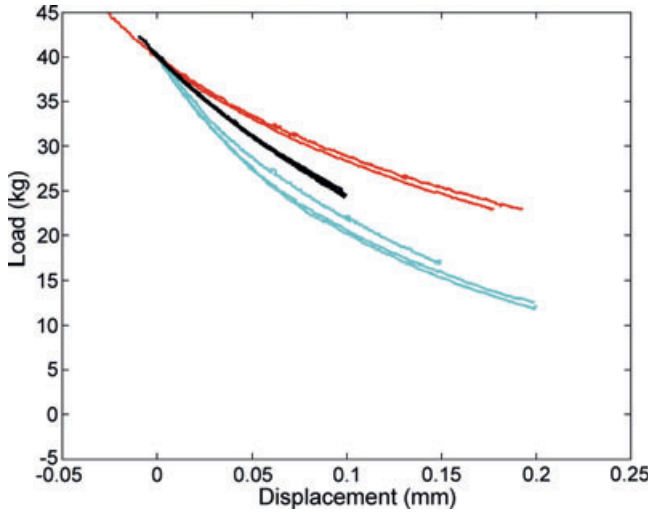


Fig. 8. Correlation curves between displacements and load for the container opening system in < 100 Pa pressure of the clean chamber No. 1. Red curves represent those obtained when the container was with atmospheric pressure, and blue curves represent those obtained when it was vacuum in rehearsals. Black curves represent those obtained when the actual container of unknown inner pressure was slightly pulled for its lid.

chamber No. 1, it was once evacuated and kept in static condition by closing all valves for pumping and/or supplying nitrogen.

Simultaneously, the residual gas sampling bottles connected to clean chamber No. 1 were prepared to capture gas released from the container at the time of its opening. As it was estimated that gas inside the container could contain terrestrial atmosphere to some extent, $O_2$ and $^{40}Ar$ could be also a sign to distinguish the opening of a leak of the container.

Therefore, the container was opened while monitoring the load cells and displacement sensors by the opening system and $O_2$ and $^{40}Ar$ (and other species with mass numbers of 4, 18, and 44) in the chamber by a differential pumping Q-MS to recognize the opening of the container. The container was opened with the four shafts movement of the opening system. After opening, the chamber pressure became 4 Pa, which corresponds to 5000 Pa as calculated from the volume ratio of the chamber (260 L) and container (0.2 L).

Just before and after the monitored data showed the signs of the opening, the gas inside clean chamber No. 1 was captured in the gas bottles. The processes to open the container were finished on 22 June, and clean chamber No. 1 was evacuated through 23 June.

Noble gases sampled in the gas bottles were analyzed at the University of Tokyo (Okazaki et al. 2011). Elemental ratios of the noble gases collected from the sample container are essentially identical to those of

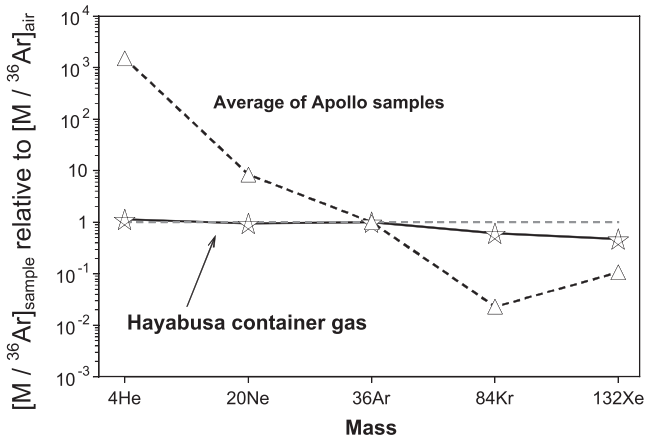


Fig. 9. Graphs showing elemental ratios of the noble gas collected from the sample container. It shows relative elemental abundances in $^{4}He/^{36}Ar$, $^{20}Ne/^{36}Ar$, $^{84}Kr/^{36}Ar$, and $^{132}Xe/^{36}Ar$ of Hayabusa container gas normalized by those of terrestrial air. They are very close to 1 and different from those of solar wind noble gases.

terrestrial atmosphere (Fig. 9). There is no sign showing the enrichment of solar gas as found in lunar samples (Fegley and Swindle 1993).

The inner pressure of the container was much higher than the estimate of that after 100 hours as described before. Possible causes are (1) a continuing but small leak of air through the double Viton O-rings seal, (2) a larger-than-expected permeability of the Viton O-rings, and (3) a temporary leak of air accidentally happening during decomposing the container before the introduction to clean chamber No. 1.

## Sample Recovery from the Sample Catcher

On 24 June, the container was transferred to the transportation chamber from clean chamber No. 1 with a transfer rod; then the inner lid and the sample catcher were set to a catcher handling container. The inner lid, which is combined with a cover of the catcher room A, was removed, and its inner surface was observed and photographed.

The catcher was enclosed into the catcher handling container, and the container including the catcher was transferred to clean chamber No. 2. The inner surface of catcher room A was observed in detail with the optical microscopes, and it was discovered that not many particles larger than a few hundred micrometers were found inside room A.

First, we tried to pick up particles from catcher room A directly with a micromanipulator, although it was unsuccessful because of the difficulty in recognizing particles on the cutting-work surface of the interior of the sample catcher room made of aluminum alloy coated with pure aluminum (Fig. 10).

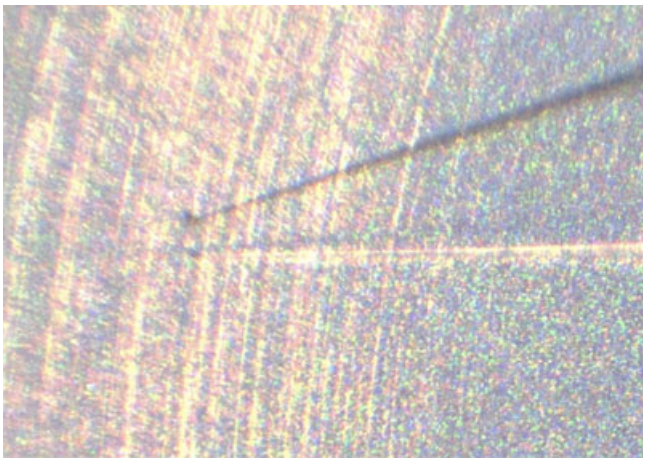

Fig. 10. An optical microscopic image of a small platy particle picked up from the inside surface of the sample catcher room A of Hayabusa. It is difficult to recognize the real particle in the image; its presence is confirmed by its shadow.

Second, we prepared a special PTFE spatula to scoop the interior surface of catcher room A. As we observed the spatula with the FESEM-EDS after the scooping, we identified rocky particles of 1–30 μm in size on it. As described in Nakamura et al. (2011), the EDS spectra of the rocky particles indicate that they originate from the surface of Itokawa. This method was effective for recovering a large number of small particles, but we could not pick them up and distribute for initial analyses because they are too small and fixed to the surface of spatula.

Third, we prepared quartz glass disks, which can be attached to the opening of catcher room A and made particles fall on it by shaking the catcher-handling container. In this method, we recovered larger particles than the previous two methods on the disks. This method was also applied from catcher room B. Figure 11 shows microscopic images of the quartz disks gathering particles from catcher rooms A and B. The particles were picked up from the quartz disk of room A to the multi-holed SEM holder made of copper by the micromanipulation system, and readily observed by the FESEM-EDS without atmosphere exposure. The samples were categorized into four groups according to the results of the FESEM observation, which is described in the following, and some particles composed of ferromagnesian silicates were distributed for initial analyses. The Hayabusa sample preliminary examination team (HASPET) was in charge of the analyses, and each of them prepared tools and containers for transfer of the samples in initial analysis according to the type of analyses (see following sections). First results of the initial analyses concluded that the particles recovered from catcher room A originated from Asteroid Itokawa (Ebihara et al. 2011; Nagao et al. 2011; Nakamura et al. 2011, 2012; Noguchi et al. 2011; Tsuchiyama et al. 2011; Yurimoto et al. 2011; Naraoka et al. 2012).

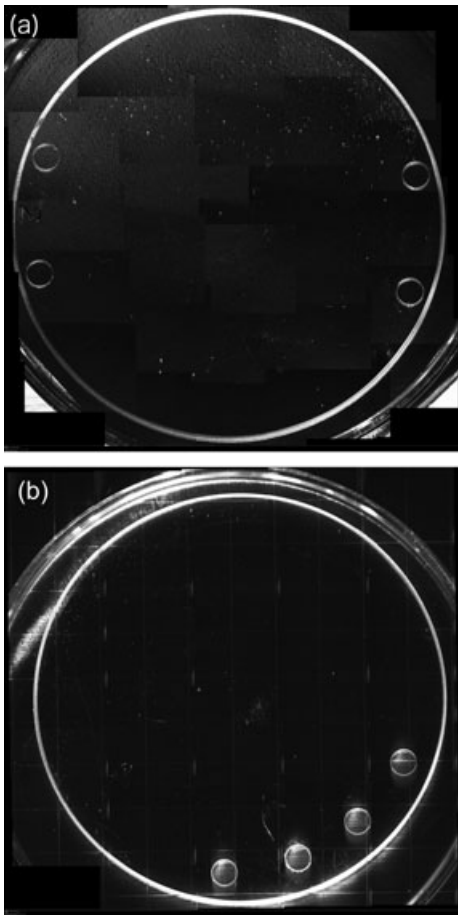


Fig. 11. Mosaic images of quartz glass disks set to the openings of sample catcher room A (a) and B (b). Each of them is 48 mm in diameter. The numbers of particles recognized on the room A disk is larger than those of room B.

## MANAGING THE HAYABUSA-RETURNED SAMPLES

### Initial Description (Optic Microscope, FESEM-EDS)

In this section, current operation procedures for the Hayabusa-returned samples of free-fall method on quartz glass disk are described.

The samples on quartz glass disk were picked up by the electrostatically controlled micromanipulation system as described in the previous section. The picked samples were observed and photographed by two optical microscopes set in the system (Fig. 12). The described data of the particles are their positions on the quartz disk, their sizes, and colors.

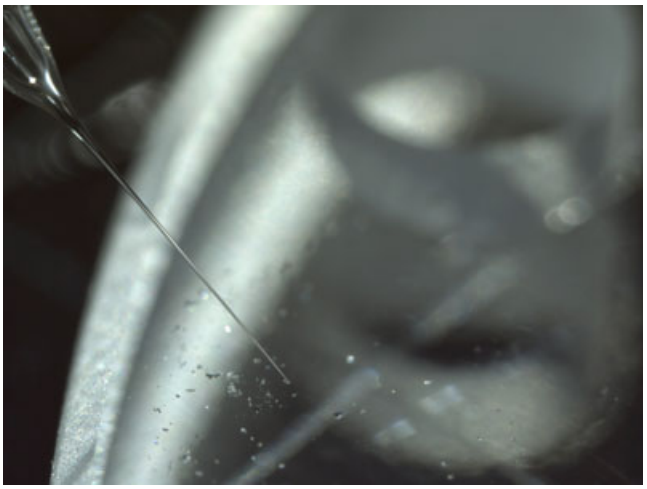

Fig. 12. An optical microscopic image of RA-QD02-0070 picked up with the micromanipulator from the quartz glass disk of catcher room A. This image was taken by the microscope observing the samples from 45° angle. The particle looks translucent in the image.

The picked samples were transferred to the sample holder for FESEM with the micromanipulation system in clean chamber No. 2. The holder was composed of copper coated with gold, and included the gold disk with dimples where samples were set. It was covered with a cap, after setting the samples in the dimple of the gold disk in the holder. The cap was sealed with a Viton O-ring, and inside of the cap was filled by the purified nitrogen environment of the clean chamber.

The samples set on the holder were observed by FESEM-EDS. The sample holder with the cap was transferred from clean chamber No. 2 to a sample exchange chamber of the FESEM and then evacuated to be purged by nitrogen. In the chamber of atmospheric pressure nitrogen condition, its cap was removed with the mechanism equipped in the chamber, and then the holder with samples was vacuumed slowly so that the very small samples were not blown due to intense gas flow. The samples were observed with a low vacuum mode of the FESEM without conductive coating. The configurations of the samples were observed by backscattered electron (BSE) and environmental secondary electron detector (ESED) images, and the samples were analyzed for their chemical composition for approximately five points with the EDS (Fig. 13a). It is indicated that particles analyzed by FESEM might be contaminated hydrocarbon in the sample chamber of FESEM due to electron beam implantation onto the particle surface during SEM observation. However, Naraoka et al. (2012) showed that no hydrocarbon more than blank level could be detected by the time of flight-secondary ion mass spectrometer (ToF-SIMS) in rinsed organic solvent of particles recovered from the catcher, which had been analyzed by FESEM before the organic analysis, thus they are not contaminated by hydrocarbon more than the blank level of the ToF-SIMS during the FESEM analyses.

Based on qualitative chemical analyses of the EDS, particles recovered from the sample catcher were grouped into four categories. Category 1 particles represent those which are composed only from transparent minerals such as olivine, pyroxene, and feldspar (Fig. 13a). Category 2 particles consist of not only transparent minerals as described in category 1 but also opaque minerals such as iron sulfide, iron-nickel metal, and chromite (Fig. 13b). Category 3 particles are mainly composed of carbon often containing oxygen, nitrogen, and sulfur, supposed to be organic carbon (Fig. 13c). Category 4 particles are artifacts such as aluminum flakes, fragments of quartz glass, and stainless steel (Fig. 13d).

**Principles of Giving Sample ID**

Temporary names were given to the samples when they are picked up from the sample catcher, quartz disk, or other place or devices. After the initial description by FESEM-EDS, they were stored on the quartz slide glass. In this stage, official names are given to the samples according to the room of the sample catcher and where they were picked up. The official format of the sample name was given by room name (- pick-up place) - number.

The room name became RA or RB, which means room A and room B, respectively. If the samples were directly picked up from the catcher, the room name was followed by the four-digit number, like RA-0001. If the samples were picked up through other devices, quartz disk for example, the sample name becomes RA-QD02-0001, where QD02 means quartz disk #02. Currently, most of the samples in room A and room B were once recovered onto the quartz disks #02 and #04, respectively, by the compulsory free-fall method. So the sample name becomes RA-QD02-XXXX or RB-QD04-XXXX for room A and room B samples, respectively.

Some samples were divided into several pieces during the processes of pick-up, release, and FESEM observation. In addition, sample divisions like cutting or making ultrathin section were also performed in the initial analysis. In such cases, we gave a new ID name to the samples. In the case of ultrathin sections, pristine mass of the particles was almost preserved as the potted butt. So we gave the same name of the parent sample (before the cutting) to the potted butt. Two-digit subnumbers were given to the ultrathin sections, such as RA-QD02-XXXX-YY, where YY is the sequential number of grids for transmission electron microscope

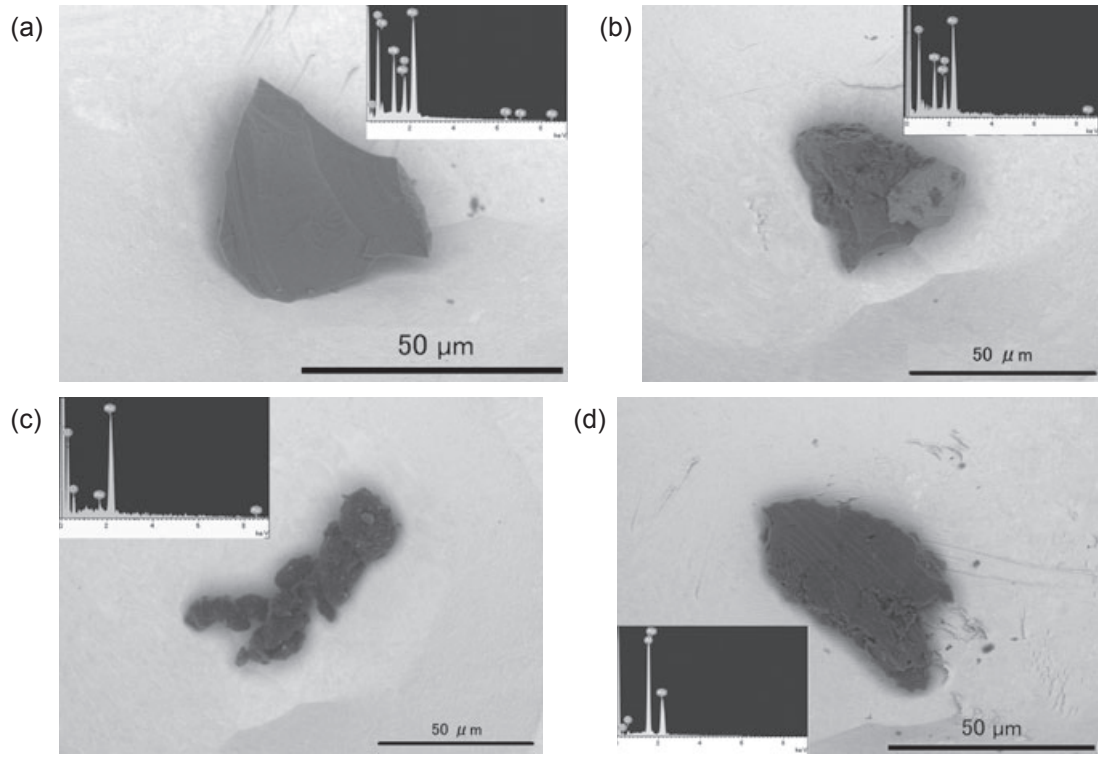


Fig. 13. BSE images and EDS spectra of (a) RA-QD02-0144 grouped as category 1, (b) RA-QD02-0088 as category 2, (c) RA-QD02-0091 as category 3, and (d) RA-QD02-0227 as category 4, respectively. As shown in their BSE images and EDS spectra, category 1 and category 2 particles are mainly composed of ferromagnesian silicate or plagioclase, although different in existence of iron metals or sulfide in category 2. Category 3 particles mainly consist of carbon and oxygen, presumed to be organic materials. Category 4 particles are artificial, such as aluminum flakes, fragments of quartz glass, and chips of stainless steel.

(TEM). On the other hand, if the samples were divided or broken into multiple pieces due to other method or reasons, the pieces of the sample were named as RA-QD02-XXXX-YY, where YY is the subnumber from 01 for the largest pieces, the smaller pieces are named as -02 and larger in the order recognition. Simultaneously, the ID name of the parent sample is removed from the list of available samples, detailed in the data archive section. The subnumbers were adhered to the end of the sample name if the samples are divided or broken again. For example, if ultrathin sections were formed from a previously divided sample, then the ID name of the ultrathin sections became RA-QD02-XXXX-YY-ZZ, where ZZ was the subnumber for the ultrathin sections.

**Preservation of the ID Given Samples**

The samples after observation of FESEM-EDS analyses were moved with the FESEM holder under atmospheric pressure purified nitrogen to the clean chamber. After having moved to the clean chamber, the cap of the holder was opened and the holder was put on the sample stage of the manipulation system. The particles were picked up while confirming a position of the sample on the gold disk of SEM holder with the microscopes, and the samples were moved on the gridded quartz slide, which had been washed as described above. The particles larger than 50 μm were moved in a deeper hollow of the slide glass, and the particles, which are smaller than 50 μm, were put on the flat slide glass (Fig. 14).

The samples put on the slide glass are named to sample ID in addition to slide glass number and grid position (see above), and are preserved in the clean chamber.

**Initial Description and Analysis Data Archive of the Samples**

All data of the curation, such as photographs of optical microscope and EDS spectrum and BSE images, were stored to the database workstation via the closed network in the curation facility. We could quickly check and/or retrieve the daily data from the database. The data relating to the samples were automatically converted into the sample database, which additionally included sample information such as current status (e.g., position on the gridded quartz slide glass), size, mineral

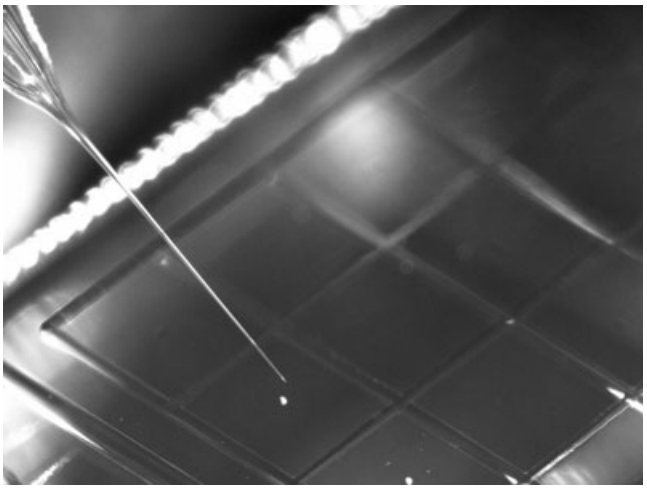

Fig. 14. An optical microscopic image of the Hayabusa-returned sample RA-QD02-0070 released on the gridded slide glass. Each particle is put on the center of one of the grids one by one.

phases and category, transfer history, and comments. The database and original data were stored in a hard disk drive (HDD) and backed up periodically in another HDD. The database management was basically operated by the curation staff and curator. The data stored in the database were basically used for the curation works. However, the data were also provided to researchers in the form of electric media or paper if required. Results of the initial analysis and announcement of opportunity (AO) analysis were also stored in the sample database.

## SAMPLE DISTRIBUTION FOR INITIAL ANALYSES AND NASA

### Design of Sample Containers for Initial Analyses

Because of the differences in the requirements of analyses, sample containers used in the initial analyses were different from each other. The samples analyzed for the mainstream of initial analyses, including synchrotron X-ray CT and diffraction, FESEM and FE electron microprobe analysis (EPMA), and a secondary ion mass spectrometer (SIMS) were completely embedded in epoxy resin and mounted on the glass fibers in the clean room air condition. They were transported within a stainless steel container filled with nitrogen. Those for TEM analyses were mounted in epoxy resin in a special nitrogen glove box and transported within the same container for mainstream analyses. In this case, they were processed without exposure to the air. Those for noble gas analyses were set in holes in a stainless steel base in a special flange of stainless steel, which had been baked beforehand to decrease its background. This process was performed in the clean chamber No. 2, thus the samples should have been kept in nitrogen condition until they arrived at the analyses line of the noble gas mass spectrometer. Those for organic analyses and instrumental neutron activation analysis were put in holes on a diamond plate with a diamond cover in the clean room air. The diamond plate and the cover are set in a stainless steel shield, covered with aluminum foil and transported in the same container of the mainstream analyses. Another special sample container was used for another analyses line, which includes the FESEM-EDS, EPMA, micro-Raman, and SIMS. The samples were put in a quartz glass container coated with gold in the clean room air and transported in the air condition.

### Design of Containers for NASA Samples Distribution

A sample container for the NASA sample distribution consists of a pair of vacuum flanges of stainless steel as an outer container and a pair of synthetic quartz glass plates as a case to enclose the sample. All parts of the container were separately cleaned and brought in the clean chamber after their assemblage. The sample was put in the small dimple, 1 mm aperture and about 0.5 mm depth, on the lower plate one by one with the electrostatically controlled micromanipulator in clean chamber No. 2. A metal plate was set under the lower plate to increase the ability of the electrostatic control of the sample handling with the micromanipulator. After the sample was put in the dimple, the lower plate was covered with the other plain glass plate. The sample could be observed through the cover plate. The pair of the plates was held with clips and screws in the machined lower flange, and covered with the other normal flange. The flanges were sealed with six screw bolts and oxygen-free cupper gasket coated by gold. The sealing was also performed in the clean chamber, so the inside of the container was filled with atmospheric pressure nitrogen in the clean chamber. We used only three materials for the sample container, synthetic quartz, gold, and stainless steel.

## FUTURE PLANS

### Plans for International AO

The sample distribution of international AO is prescribed by MOU (Memorandum of Understanding) of Hayabusa mission. In the MOU, 15% of the whole returned samples were due to be assigned to international AO. The recovered amount was small and the total amount is still uncertain; however, about one hundred particles, including particles returned from the initial analyses, with the preliminarily examined ones were opened to the public as the distributed samples of

international AO. Investigators who proposed research of the distributed samples submitted proposals. The proposals were reviewed by reviewers and AO committee members. The first international AO was released on 21 January 2012 and closed on 7 March 2012. Distribution of these samples started in June 2012. Distributed samples in the first international AO are FESEM-EDS observed particles recovered by free-fall method and classified into the category 1 or 2. We are planning to distribute particles recovered by other method, those classified as categories 3 and 4, those processed by FIB or sectioned by an ultramicrotome from the second international AO.

### Disassembly of the Sample Catcher and Recovery of Samples

At present, the particles in the sample catcher are recovered in three methods, picked up directly, scooped in the PTFE spatula, and recovered by free-fall method from room A. Only the compulsory free-fall method was applied for catcher room B, because destruction of the particles, which might have happened in the spatula scooping the inner surface of catcher room A, should be avoided. As the sample catcher has a complicated structure, it is very difficult to pick up particles from the catcher directly. However, not all particles can be recovered from the sample catcher rooms A and B by the compulsory free-fall method because of adherence of the particles on the surface inside the catcher due to electrostatic charge and the complex structure of the catcher. Therefore, we are planning to recover sample residues in the catcher after its disassembly and taking into pieces on a part level eventually. A partition of catcher rooms A and B, the rotational cylinder should be removed during the disassembly. After the disassembly, samples in the rooms A, B, and the rotational cylinder will be completely mixed up, although there is no choice of recovering the sample residues inside the catcher.

### Future Plan for Managing and Preserving Samples

Although it will take some time to recover all the samples from the catcher and container, we plan to recover the Hayabusa samples and distribute them continuously, as far as international AO analyses will continue. However, some fraction of the Hayabusa samples should be preserved for the future after the distribution to the international AO analyses and NASA. The samples kept for the future will be grouped into some different levels due to their processes, such as those kept adhered to the sample catcher, those observed by FESEM-EDS for classified into the categories, those only picked up with no following FESEM-EDS observation. The particles other than the Itokawa origin should also be precious samples for sources of information to estimate things that had happened in the spacecraft. At present, the lower part of the sample container, which includes many particles other than Itokawa origin, is located inside the transportation chamber next to clean chamber No. 1 in the state of the vacuum (below $10^{-5}$ Pa) after its transportation from clean chamber No. 1 since 24 June 2010. We are planning to send some fraction of the recovered samples to the cabinet attached to clean chamber No. 2 to keep them in a vacuum condition in near future.

### Plans for Hayabusa 2 Sample Curation and Future

In the Hayabusa 2 mission, the same sample recovery method as Hayabusa will be adopted, and the size of a sample container and a sample catcher will be also almost the same. However, Hayabusa 2 should be different from Hayabusa in a container seal system and a gas sampling method after sample return as the scientific importance of the mission is focused on volatile matter, such as water, an organic matter, and noble gas. The acceptance of the reentry capsule of Hayabusa 2 will be also carried out in the PMSCF/JAXA, although revisions and adjustments for it will be necessary. This curation facility has realized the acceptance of the Hayabusa sample by sufficient performance. It is sure that this facility can respond to acceptance of not only Hayabusa 2 but also other extraterrestrial samples. Taking advantages of the features of this facility, in which handling and analyses of the samples without exposing terrestrial atmosphere are possible, we will not only curate and analyze returned samples of Japanese missions but also those of other countries' missions. We expect to contribute to acquisition of significant scientific results through our curation activity.

## SUMMARY

Since the construction of the PMSCF/JAXA was completed in March 2008, we checked the performance of the facility, the clean chamber, and the instruments within the facility in cleanliness and ability to accomplish Hayabusa-returned sample curation. A year before the reentry of Hayabusa capsule, we performed a series of rehearsals for the reentry capsule acceptance, X-ray CT, cleaning and cleanliness check, removal of parts of the sample container, container opening and residual gas sampling, sample catcher handling, and recovery of samples. We utilized the simulated container and particles for the rehearsal. For the real part of Hayabusa, the reentry capsule arrived at the curation facility on 18 June 2010, 3 days after the reentry, and underwent a

series of processes, which we performed in the rehearsal. Within seven days after the capsule reentry, the sample container was introduced into clean chamber No. 1 and the inner pressure of the container was estimated to be between vacuum and atmospheric pressure based on the value of a series of sensors equipped in the sample opening system. Then, the sample container was opened in vacuum condition to recover residual gas in the chamber, although the result was that most gas was from the terrestrial atmosphere. The container was transferred to the transportation chamber in vacuum and the sample catcher was set to the catcher handling container and brought to clean chamber No. 2 in atmospheric pressure nitrogen condition. In clean chamber No. 2, particles in the sample catcher were recovered with the direct pick-up from the catcher, PTFE spatula scooping, and the compulsory free fall on quartz disks. With the third method, we recovered particles in the sample catcher, which can be handled with the electrostatically controlled micromanipulation system. Thus far, particles were picked up from the quartz disks of both catcher rooms A and B, routinely analyzed with the FESEM-EDS, categorized into four groups based on chemical composition, and preserved on quartz slide glasses without exposure to the air. Information for them has been archived in a database established in the PMSCF/JAXA. Some of the recovered particles have been distributed to NASA for initial analyses, and will be distributed to the international AO. New particles from the sample catcher will be recovered with other methods in the near future. We are now going to prepare for the next sample return samples, Hayabusa 2 and other missions.

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