

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

Akio FUJIMURA¹, Masanao ABE^{2,1}, *Toru YADA^{1,2},
Tomoki NAKAMURA³, Takaaki NOGUCHI⁴, Ryuji OKAZAKI⁵,
Keisuke NAGAO⁶, Hiroshi NARAOKA⁵, Yukihiro ISHIBASHI¹,
Kei SHIRAI², Michael E. ZOLENSKY⁷, Scott SANDFORD⁸,
Tatsuaki OKADA^{2,1}, Masayuki UESUGI¹, Yuzuru KAROUJI¹,
Maho OGAWA⁹, Shogo YAKAME⁹, Munetaka UENO²,
Toshifumi MUKAI¹⁰, Makoto YOSHIKAWA^{2,1}
and Junichiro KAWAGUCHI¹

¹JAXA's Space Exploration Center, Japan Aerospace Exploration Agency, 3-1-1,
Yoshinodai, Chuo-ku, Sagamihara, Kanagawa 252-5210, JAPAN.

²Institute of Space and Astronautical Science, Japan Aerospace Exploration Agency,
3-1-1, Yoshinodai, Chuo-ku, Sagamihara, Kanagawa 252-5210, JAPAN.

³Department of Earth and Planetary Material Sciences, Graduate School of Science,
Tohoku University, Aoba, Sendai, Miyagi 980-8578, JAPAN.

⁴College of Science, Ibaraki University, 2-1-1 Bunkyo, Mito, Ibaraki 310-8512,
JAPAN.

⁵Department of Earth and Planetary Science, Graduate School of Science, Kyushu
University, Hakozaki, Fukuoka 812-8581, JAPAN.

⁶Geochemical Research Center, Graduate School of Science, The University of Tokyo,
7-3-1 Hongo, Bunkyo-ku, Tokyo 113-0033, JAPAN.

⁷ARES, NASA Johnson Space Center, Houston, TX 77058, USA.

⁸NASA Ames Research Center, Moffett Field, CA 94035, USA.

⁹Department of Earth and Planetary Science, Graduate School of Science, University of
Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-0033, JAPAN.

¹⁰Japan Aerospace Exploration Agency, 3-1-1, Yoshinodai, Chuo-ku, Sagamihara,
Kanagawa 252-5210, JAPAN.

*Corresponding author: e-mail: yada@planeta.sci.isas.jaxa.jp

Keywords: Hayabusa, Itokawa, curation, asteroid, sample return mission,

Abstract: The Planetary Material Sample Curation Facility of JAXA (PMSCF/JAXA) was established in Sagami-hara, Kanagawa, Japan in order 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 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 experienced in a series of processes to be extracted 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, NASA and will be for international announcement of opportunity (A/O), but certain fraction of them will be preserved in vacuum for future analyses.

Note: This technical report is written based on the paper to be published in the journal *Meteoritics and Planetary Science* by Wiley-Blackwell. Thus some parts of sentences and figures in this report are almost identical to those of the paper.

INTRODUCTION

Sample return missions recovering samples from other extraterrestrial bodies have brought us great progresses in understanding their origin, formation and evolution processes. The Apollo missions, accomplished six successful landings on the Lunar nearside and recover samples there, brought back about 382 kilograms of lunar samples. In order to curate the returned lunar samples with lesser terrestrial contamination, the Apollo mission had prepared the Lunar Receiving Laboratory (LRL) in Johnson Space Center (JSC) of NASA to treat the lunar samples in glove boxes (Allton et al., 1998). Almost forty years after the Apollo mission, the Stardust spacecraft successfully returned samples captured nearby comet Wild 2 (Brownlee et al., 2008). They prepared a special clean room to curate the Stardust samples under clean condition (Zolensky et al., 2008). The Stardust clean room is a higher cleanliness level than that the LRL, but it does not contain a glove box to treat the samples in nitrogen condition, as the LRL did.

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. Scientific objectives of MUSES-C spacecraft were to obtain a surface topography, near infrared (IR) spectra, X-ray fluorescence spectra of a target asteroid and to try capturing samples on its surface to return the recovered samples to the Earth. Its target asteroid had been changed from 4660 Nereus, to 10302 1989ML, and finally to 25143 1998SF36, named Itokawa later. Visible and near 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 at 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 Regio for sample collections on 20 and 26 November 2005 (Yano et al., 2006). It returned to the 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 return sample curation was planned before its launch, but it had been reconsidered by an advisory committee for specifications of a Planetary Material Sample Curation Facility of JAXA between Dec. 2005 and Feb.

2007. Detail specifications of the facility and clean chambers and instruments installed in the facility was considered in parallel, and the facility had finished in March 2008. In the followings, we describe details of the facility and curation of the Hayabusa return samples.

SPECIFICATIONS OF THE PLANETARY MATERIAL SAMPLE CURATION FACILITY OF 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 in Japanese Industrial Standards (JIS) code) coated with pure aluminum, stainless steel (SUS304 in JIS code), Viton®, Al₂O₃ 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 Al₂O₃ glass was set inside a sample catcher during the final assemblage for flight model of the spacecraft, thus contamination after that point has been 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 the 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 is estimated to be less than 1Pa for 100 hours 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 returning trip in interplanetary space. During atmospheric entry, the sample container is 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 are kept less than 26°C. The magnetic condition of Hayabusa returned samples should have been disturbed during a return trip to the Earth due to ion engine operation which is one of 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 had not been monitored during the processes in this mission because their effects on the samples are considered to be minor. Hereafter we detail the curation facility and clean chambers, in which the Hayabusa samples were treated.

Clean rooms

We designed the Planetary Material Sample Curation Facility (PMSCF) to diminish

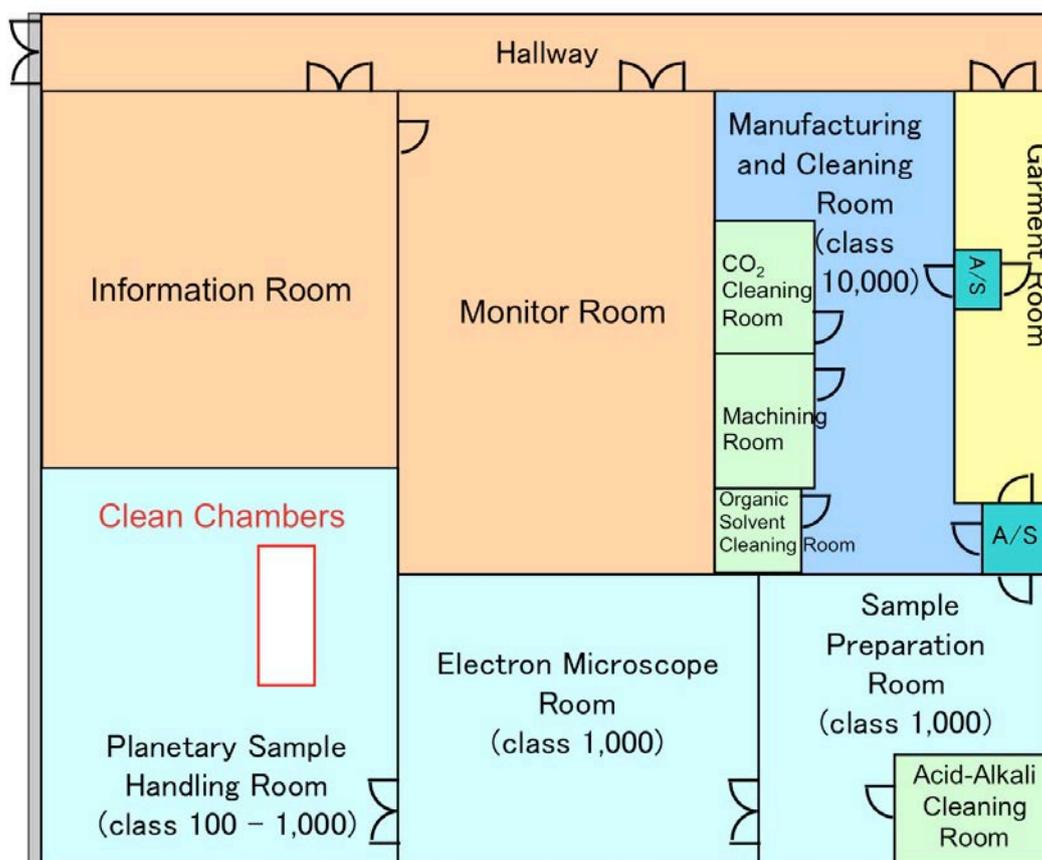


Fig. 1. A schematic viewgraph of cleanrooms of the Planetary Material Sample Curation Facility (PMSCF) of JAXA. They consist of four cleanrooms, the Planetary Sample handling room, the Electron microscope room, the Sample preparation room and the Manufacturing and cleaning room.

terrestrial contaminants from its environment. It equips four clean rooms, the Planetary sample handling room, the Electron microscope room, the Sample preparation room and the Manufacturing and cleaning room (Fig. 1). The clean class level of the room is 100 - 1,000 in Fed. Std. 209D for the Planetary sample handling room, 1,000 for Electron microscope room and Sample preparation room, and 10,000 for the Manufacturing and cleaning room. Because of necessity for cleaning using organic solvent, acid and alkali and instruments which would give off contaminant, four independent air exhaust rooms are equipped in the clean rooms, the Acid and alkali treatment room, the Organic solvent cleaning room, the Machining room and the CO₂ cleaning room. Equipment which cannot be set in the clean rooms, such as roughing pumps and nitrogen purifiers, are also set in the Basement.

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 are mainly made of stainless steel (SUS304 in JIS code), and their inside walls are treated by the method of electrochemical polishing. They were baked in vacuum more than 120°C before and after the installation to lessen contaminant gas absorbed on them. Environment from the ultrahigh vacuum to atmospheric pressure of purified nitrogen condition can be realized in the chamber No.1, in which the sample capsule of Hayabusa was opened. For opening the sample container in the clean chamber No.1, a container opening system is equipped to the clean chamber No.1. It is composed of four movable shafts which can be connected with an inner lid of the container, touch-type displacement sensors and laser displacement sensors to monitor the movement of the shafts and load cells to monitor the stress on them. Residual gas in the container can be collected in residual gas sampling bottles made of stainless steel which are equipped with an introduction line to the clean chamber No.1. A lower part of the sample container will be stored in vacuum condition of cabinet No.1 attached to the chamber No.1. For the transportation of the sample container, a transportation chamber is equipped between the chamber No.1 and cabinet No.1. It has two magnet coupling transfer rods by which the container can be transported from the chamber No.1 to the cabinet No.1 via the transportation chamber both in vacuum and atmospheric pressure nitrogen conditions. The chamber No.1 also has two Viton® gloves equipped with gate valves so that we can handle the sample container and catcher with tools and jigs.

The sample catcher will be sent to the clean chamber No.2 for further handling such as recovery of solid sample grains from the catcher and treated them samples for further sample analysis. An ultra long work distance optical microscope is prepared to be capable to be adjusted to both glass viewports of the clean chamber No.1 and glass windows of the clean chamber No.2. The samples can be handled in an atmospheric pressure condition of purified nitrogen with special tools and jigs in the clean chamber No.2 which is equipped with six Viton® gloves. And samples once cataloged will be stored in vacuum of cabinet No.2 or 3 attached to the chamber No.2. Both the chamber No.1 and No.2 equip ultraviolet (UV) neutralization apparatuses in order to compensate electrostatic charge which should occur in the pure nitrogen condition. Also, an alpha-ray neutralizer of ^{210}Po radioactive source is prepared for the same purpose.



Fig. 2. A photograph of clean chambers in the Planetary Material Sample Curation Facility (PMSCF) of JAXA. They consist of two main chambers, No.1 (left) and No.2 (right) and situate in the planetary sample handling room of 100 – 1,000 in clean class level of Fed. Std. 209D.

Other equipment

Electron microscope

A field-emission type scanning electron microscope (FESEM), Hitachi S-4300SE/N, equipped with energy dispersive X-ray spectrometer (EDS), Oxford Instruments INCA X-act Energy 350, is installed in the electron microscope room. It is prepared for initial description of planetary material samples, and equips low vacuum observation mode for observing insulators such as silicate without conductive coating. We also prepared a custom-made sample holder which is basically made of copper coated by gold and has a cap with a Viton® O-ring to seal samples inside from terrestrial atmosphere. With this holder and an airlock of the FESEM which equips a mechanism to open its cap, we can transfer samples from the clean chamber to the FESEM without exposing to terrestrial atmosphere. There situated another glove box in the electron microscope room. It has two Viton® gloves for handling and an air lock to introduce tools and jigs without breaking the environment inside the box. It can be vacuumed to be exchanged with nitrogen, also flow nitrogen gas and control its inner pressure not only positive to the cleanroom air but also negative to the air.

Instruments for cleaning and checking cleanliness

To keep cleanliness of the clean chamber, a series of cleaning instruments are prepared for cleaning tools, containers and jigs used in the clean chamber. Four ultrasonic cleaners of 38kHz, 35kHz and 95kHz, 100kHz, and 950kHz in frequency are set in the manufacturing and cleaning room and the sample preparation room. They are capable to perform overflow cleaning with ultra pure water supply system in the rooms. In order to perform acid and base treatment cleanly, a clean evaporator is installed in the acid and base 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 CO₂ blast cleaner to remove particles and organics on surfaces of materials, and an UV ozone cleaner to sterilize bacteria and remove organics on materials' surfaces.

In order to check the environment of the clean chambers and attached chambers with them, two types of mass spectrometers are set near by the clean chambers. One is an atmospheric pressure ionization mass spectrometer (API-MS) and the other is a differential pumping quadrupole mass spectrometer (Q-MS). The former is suitable

for analyze impurities in nitrogen gas less than sub-ppm, and the latter is capable to analyze impurities more than sub-ppm. A Fourier transform infrared spectroscopy (FT-IR) and a dynamic contact angle meter are prepared to check cleanliness of surfaces of materials.

Electrostatically controlled micromanipulation system

We prepared three commercial base micromanipulator systems operated in clean rooms and two specially designed micromanipulator systems operated in an ultra pure nitrogen environment of the clean chamber and one system operated in a low vacuum nitrogen condition of FESEM sample room for handling the minute particle one by one. Most of the systems are operated manually and have special designed quartz glass probe with electrode that lifts or releases a particle by electrostatic force. Two of the three commercial base micromanipulators are operated in an atmospheric condition. Another one commercial base micromanipulator system is attached on a microscope with long work distance objective lenses and operated mainly in a glove box filled with highly purified ultra pure nitrogen. For the use in a clean chamber filled with the ultra pure nitrogen, two specially designed micromanipulator systems are prepared. One system is a principal device for handling and consists of single sample stage and dual probe stages. The other system enables us to move a particle and release it on the bottom of small quartz bottle that is one type of sample transfer vessel, and it prevents farther alterations when the mouth of the bottle is welded by a quartz plug in the clean chamber. These two systems are of oil free for the operation in the clean chamber. The last one system is installed in sample room of FE-SEM, which is filled with low vacuum clean nitrogen due to prevent metalize sample surface for discharge. In the FE-SEM, particles on a specially designed sample holder can be lifted and released by the micromanipulator system. This manipulator is not operated manually, but it has two axes (x and y) PC control piezoelectric elements which hold probes each other.

All micromanipulator devices possess newly developed quartz glass probe. The probe has a platinum electrode that takes control of sample particle by electro-static force. The probe works as lift tool or release tool when the polarity and voltage of the electrode is changed. The electro-static control method is reasonable, because the electro-static force acting on a particle is essential and gravity force is not effective when particles are small, i.e., less than several tens micrometer. The shape and tip length of the quartz probe vary widely for the actual purposes. Though the electro-static control method is easily realized on the system with metal needle probes, we did not use

metal probes basically. If the metal needle probe touches the other metal probe or metal base with different electric potential, accidental spark will take place and sample particle lifted by the probe will be melted or lost.

In case of very tiny sample particles, visual confirmation is difficult by an optical microscope, and more precise technique such as SEM observation will be required. In the sample room of our FE-SEM, the particles often became stuck the surface of spatula that collects particles from sample container. The metal needle is useful to dig the particles from the surface of the spatula, because it is not break, and because quartz glass probe is easy to brake. We prepare tantalum needle, since the tantalum is a selected material for the projectile of sample collector of Hayabusa spacecraft.

The present tiny sample requires high cleanliness, and chemically clean quartz glass probe is convenient. The conventional borosilicate glass and metal, such as tungsten needle, were not employed because of their pollution source. The electrostatic controlling method developed at present, has remarkable advantages for handling tiny particles. The method provides adequate force for handling every sample particle. As a result, the present returned samples preserve their loose structural character realized at the surface of the asteroid Itokawa, and avoid chemical pollution coupled with cleanliness of quartz glass probe. The electrostatic controlling method produces a potential contamination of the magnetic and electric properties for the virginal asteroid sample. Unfortunately, however, propelling power of Hayabusa spacecraft is provided by ion engines that require large electric current, sample particles in a sample container were affected by this electric current in the spacecraft to a greater or lesser degree.

PERFORMANCE CHECK OF THE CURATION FACILITY

Cleanliness of clean rooms

Particle cleanliness levels of the clean rooms were investigated after finishing their constructions on April 11, 2008. A following table is the results of the analyses of cleanliness for each cleanrooms. The data were acquired by a particle counter PMS LASAIR II 310A. The air suction rate of the analyses was 28.3L/min (1 ft³/min). The cleanliness was analyzed at multiple points for each cleanroom in accordance with its area, and analyzed three times at each point. The unit is N/cf³ and the particle number of >0.5μm corresponds to the cleanliness level in Fed. Std. 209D. This table shows that the actual cleanliness level for each cleanroom is much smaller than its

specification.

Table 1. Results of analyses of particle cleanliness level of each cleanrooms of the facility (unit: N/cf³).

Particle size	Garment room	Manufacturing and cleaning room	Sample preparation room	Electron microscope room	Planetary sample handling room
Analysis points	2	4	5	8	9
>0.3 μ m	55.3	27.0	6.7	4.7	3.1
>0.5μm	17.2	17.1	4.0	2.5	1.6
>1 μ m	9.2	10.5	1.7	1.2	0.7
>5 μ m	1.8	2.4	0.2	0.3	0.1
>10 μ m	1.2	1.4	0.1	0.1	0.0
>25 μ m	0.0	0.1	0.0	0.0	0.0

Cleanliness of clean chambers

The source of the nitrogen gas supplying to the clean chambers is liquid nitrogen whose concentrations of the heavy noble gases are less than 0.1% of terrestrial atmosphere. The vaporized nitrogen gas is purified by two types of gas purifiers. The first is a flow type and the second is a cyclic type. The nitrogen gas purified by the flow type purifier is introduced to the cyclic gas purifiers directly linked to the clean chambers.

The mass spectrum of the nitrogen processed by the flow type purifier is obtained the API-MS (Fig. 3). The major impurities detected in the purified nitrogen gas are ~1 ppb of water and 0.2 ppb of oxygen. The other candidate impurities are under detection limits of CH₄ and CO₂, less than 50 ppt.

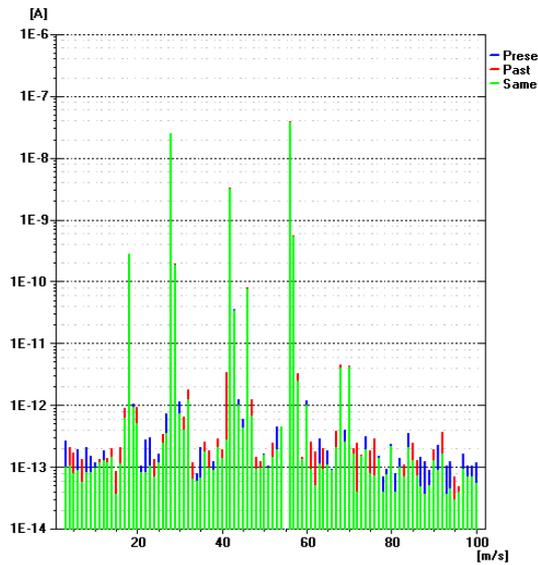


Fig. 3. Electric current vs. m/z relation obtained by API-MS.

The spectrum shows ~ 1 ppb of water, 0.2 ppb of oxygen, and CH_4 and CO_2 of lower than detection limit, less than 50 ppt.

The baking process under the vacuum condition is the most effective clean up procedure for the clean chambers, which has been already cleaned in particle level. We performed the baking at about 120°C for three days, by using 37 heaters and temperature monitor of 48 points for the clean chamber 1, and 6 heaters and temperature monitor of 25 points for the clean chamber No.2.

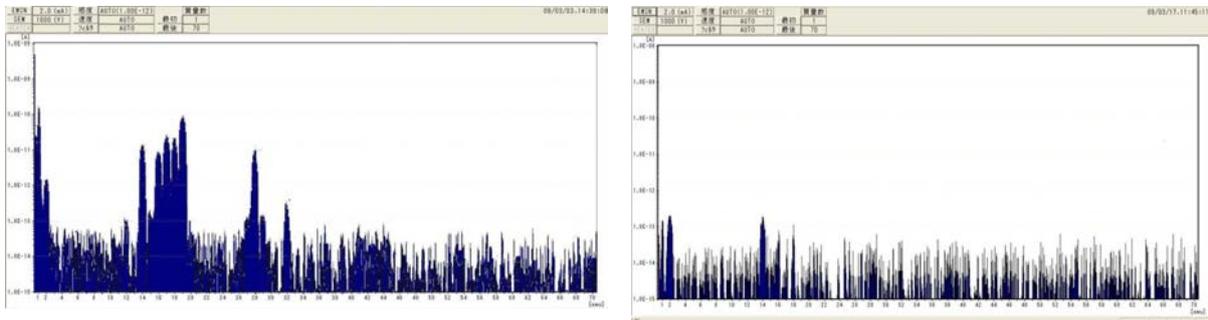


Fig. 4. RGA-QMS spectra obtained in the chamber No.1. Their vertical axes are electric currents and horizontal axes mass numbers (m/z).

Left: Obtained at the vacuum condition of 7.68×10^{-6} Pa realized before baking.

Right: Obtained at the vacuum condition of 7.20×10^{-8} Pa realized after baking. The elements of $m/z=1, 2,$ and 14 are identified. The other elements are not detected, because no apparent mass peak of larger than 1×10^{-14} A can be recognized.

After the baking, the vacuum condition of the chamber No.1 reaches to 7.20×10^{-8} Pa. The residual gas components measured by the residual gas analyzer of Q-MS (RGA-QMS) are lower than the detection limit of the RGA-QMS (i.e., lower than 1.0×10^{-14} A) except mass number 1(H), 2(H_2), 14(N). The observed spectrum is shown in Fig. 4. The vacuum condition of the chamber No.2 reaches to 3.16×10^{-6} Pa that reflects the lower performance of the vacuum condition including the sealing

Viton® gaskets of several components of the chamber and low performance of the vacuum pump attached to it. The lower vacuum performance of the chamber 2 is expected because the chamber is designed for the operation under the atmospheric pressure of nitrogen.

The cleanliness of the chamber No. 2 is examined by the API-MS. The major impurity gas components are of ~ 1 ppb of oxygen and ~ 14 ppb of water in the second room fulfilled by the purified nitrogen supplied by the cyclic gas purifier. Both concentrations of the impurities are less than the nominal specification of the cyclic gas purifier, which are 20 ppb of oxygen and also 20 ppb of water. The mass spectrum obtained by the API-MS is shown in figure 5

The time dependence of the vacuum deterioration was measured on the chamber No.1 and No.2 (Fig. 5). The vacuum condition under the pressure of 1×10^{-5} Pa was realized during 15 minutes in the chamber No.1 at the condition of stopping the e, and under the pressure of 1×10^{-4} Pa in the chamber No.2. The result of the chamber No.1 is explained by the outgas rate of 7×10^{-9} Pa \cdot m³/s/m², which is consistent to the outgas rate of the completely baked and electrically etched clean surface of stainless steel (SUS316L in JIS code) wall of the chamber No.1. The result of the second room is explained by 1×10^{-7} Pa \cdot m³/s/m², which can be interpreted by the outgas rate of baked Viton® elastomer and of baked metal surface wall of the chamber No.2. The chamber No.2 is a glove box and possesses three sets of Viton® gloves and several Viton® O-ring seals. Around half of the outgas may be due to this baked Viton® elastomer

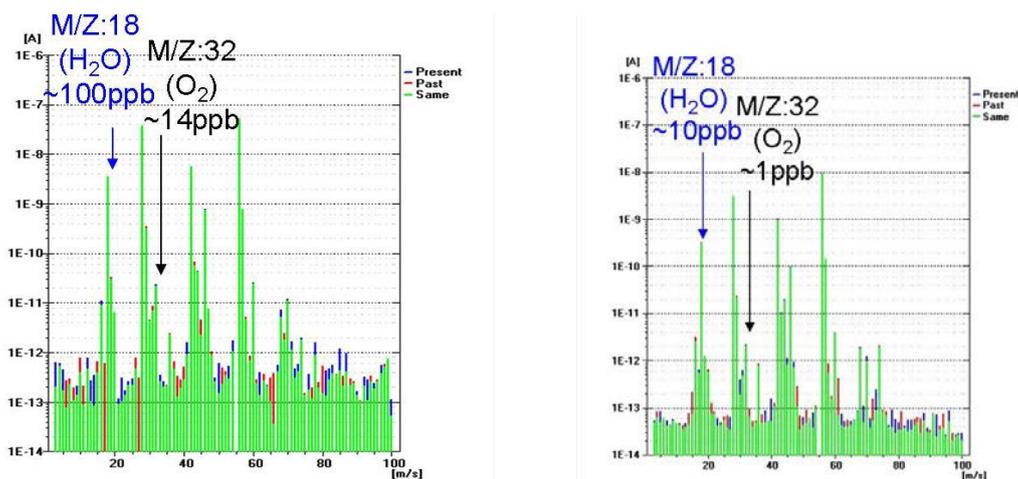


Fig. 5 API-MS spectra obtained in the clean chamber No.2. Their vertical axes are electric current and horizontal mass number (m/z). Left: ~ 100 ppb of water and ~ 14 ppb of oxygen are detected in atmospheric pressure nitrogen of the chamber No.2 (before baking). Right: Detected water content is ~ 10 ppb and oxygen is ~ 1 ppb in the atmospheric pressure nitrogen of the chamber No.2 (after baking). The vacuum condition of 3.16×10^{-6} Pa is achieved just after baking.

and the other half may be caused by the outgas rate of the completely baked and electrically etched clean surface of the stainless steel (SUS304 in JIS code) wall of the chamber No.2.

As the results of the baking, the cleanliness of the chamber No.2 has been improved. The observed impurity components in nitrogen gas of the chamber No.2 supplied by the cyclic gas purifier are 9 ppb of water, 1 ppb of oxygen, 2 ppb of CH₄, and 4 ppb of CO₂. After the 4 hours interval of supply stop of the purified nitrogen gas, the major impurity components are about 90 ppb of water and about 30 ppb of oxygen in the chamber No.2.

Noble gas abundances in high purity nitrogen gas used in the curation facility

Pure nitrogen gas is preferable for use of atmosphere in the chambers No.1 and No.2 to prevent weathering and oxidation of samples by terrestrial water vapor and oxygen. Moreover, low concentrations of noble gases in the nitrogen gas are favorable because the high concentrations of noble gases in terrestrial atmosphere are the most serious source of noble gas contamination to extraterrestrial samples such as meteorites and interplanetary dust particles. We searched 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. 6. The nitrogen gas was sampled from headspaces of tanks for storage of liquid nitrogen in the company: ELF is a small tank of 175L but liquid nitrogen (LN₂) Tank is a large one in which liquid nitrogen produced in the company is stored. Noble gases were measured with a modified-VG5400 (MS-III) at the Geochemical Research Center, University of Tokyo. Concentrations of Ar, Kr, and Xe from both tanks are almost the same among them, and 3 to 5 orders of magnitude lower than those in atmosphere. In contrast to the heavier noble gases, He and Ne concentrations in both tanks are different among them. Concentration of He in the LN₂ Tank is not so depleted, about 1/10 of atmospheric one, while that of Ne is higher than the atmospheric value. The concentrations in ELF are low compared with those of LN₂ Tank. 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 be in liquid at the liquid nitrogen temperature. If this is the case for He and Ne, nitrogen gas with low concentrations of all noble gases will be available from the tank after purging of enough amount of nitrogen gas from the headspace.

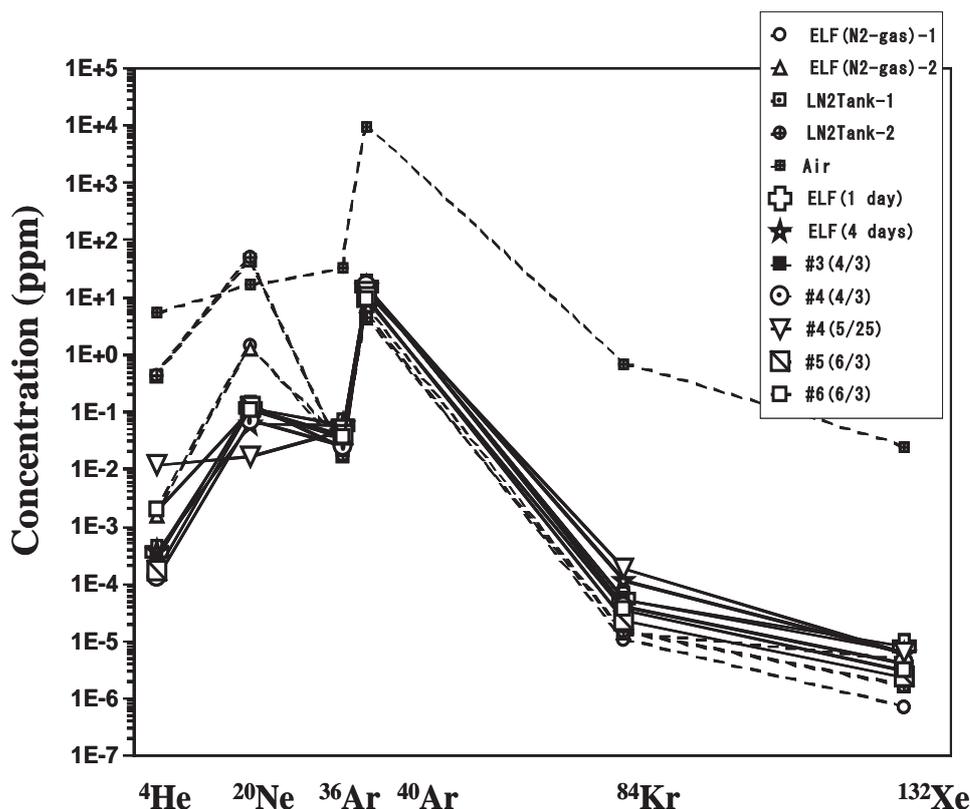


Fig. 6. Noble gas concentrations in nitrogen gas evaporated from liquid nitrogen and accumulated in headspace of tanks for storage of liquid nitrogen. Noble gas concentrations in atmosphere are shown for comparison. Four samples, from ELF (nitrogen gas) to LN₂Tank, were collected in metal bottles from an ELF tank and a large tank (LN₂Tank) at the company (Taiyo Nippon Sanso Corporation) producing the liquid nitrogen. Seven samples, from ELF (1 day) to #6(6/3), were in the curation chamber, to which the nitrogen gas is supplied from ELF tanks after purging the headspace of ELF tanks.

The nitrogen gas is used in the curation chamber by supplying it from the headspace of ELF tanks set outside the curation building through a metal pipe line. To certify the purity of nitrogen in the curation chamber, nitrogen gas was sampled several times by using ultra-clean metal bottles connected to the curation chamber, and then transferred to University of Tokyo for noble gas analysis. The measured noble gas concentrations are depicted in Fig. 6. The lower concentrations of He and Ne compared with those in nitrogen gas collected from ELF and LN₂ tanks in the company must be due to the purging of gas in headspace before supply to the curation chamber. Heavy noble gases are also 4 orders of magnitude lower than those in atmosphere. The low concentrations of noble gases would reduce atmospheric contamination to the Hayabusa samples.

Isotopic ratios of noble gases are close to those of atmospheric ones except for Ar isotopic ratios as shown in Fig. 7. The $^{38}\text{Ar}/^{36}\text{Ar}$ and $^{40}\text{Ar}/^{36}\text{Ar}$ ratios are as low as 0.175 and 255, respectively, and are plotted on a mass fractionation line passing the atmospheric Ar. The degree of fractionation is ca. 3.5 %/amu. The large mass fractionation effect on Ar isotopes may be attributable to co-evaporation of a small fraction of Ar with nitrogen gas from liquid nitrogen-oxygen mixture at the separation of nitrogen from oxygen. Because condensation temperature of Ar is close to those of nitrogen and oxygen, only Ar is affected by fractionation effect. In contrast to Ar, He and Ne are always as gas phase, while Kr and Xe are as liquid phase during the fractional evaporation process of liquid nitrogen-oxygen mixture.

A simple Rayleigh distillation model indicates that a single step distillation causes only 1.3 %/amu of enrichments in lighter isotopes in distilled Ar phase, i.e., from

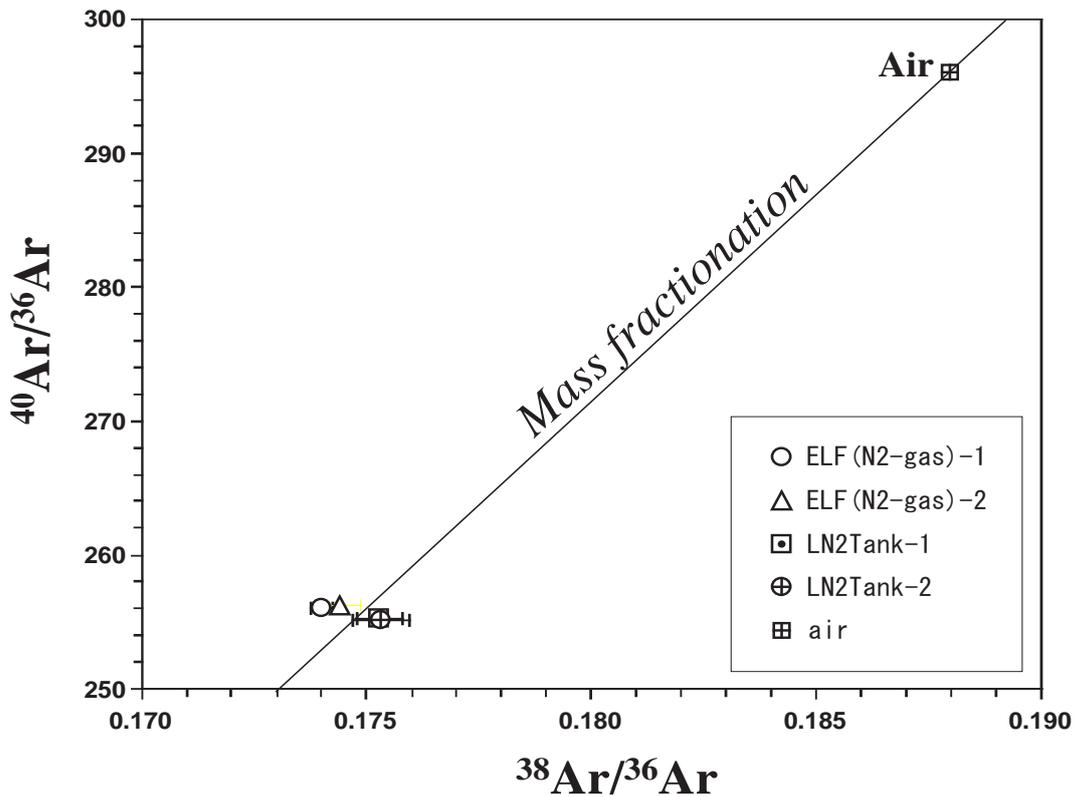


Fig. 7. $^{40}\text{Ar}/^{36}\text{Ar}$ ratios in the nitrogen gases from the ELF and LN₂ tanks, showing heavy mass fractionation from atmospheric value (296) favoring lighter isotopes by ca. 3.5 %/amu.

the atmospheric $^{40}\text{Ar}/^{36}\text{Ar}$ of 296 to ≈ 281 . If 10-20 % of Ar is distributed in nitrogen gas while remaining Ar is in liquid oxygen during a single stage distillation of nitrogen, three steps of distillation can account for the observed $^{40}\text{Ar}/^{36}\text{Ar}$ of 255 in the nitrogen

gas.

Viton® glove permeability to noble gas

Degassing rates of noble gases from a Viton® sheet and noble gas permeability through it were investigated. One side of the 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

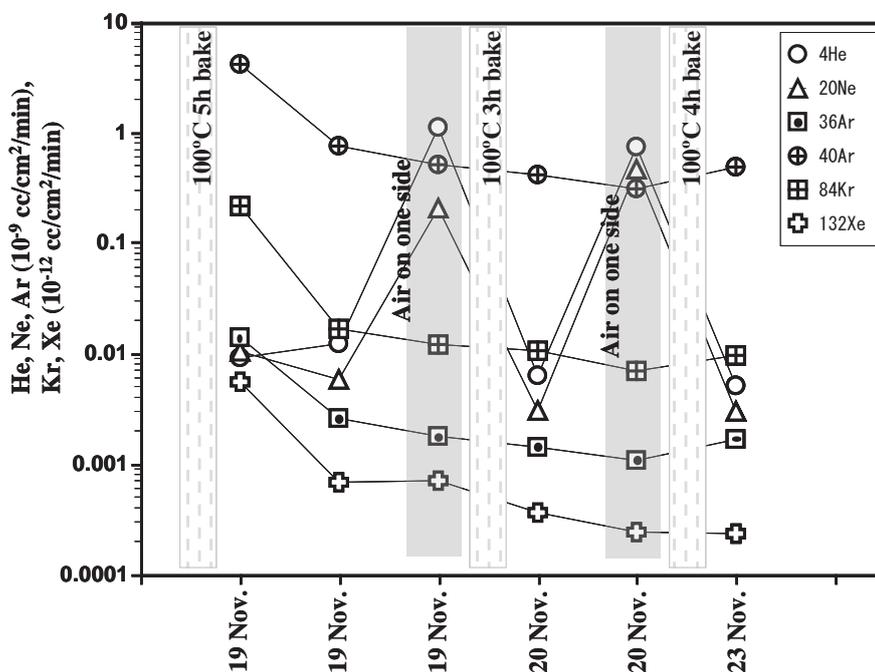


Fig. 8. Increasing rates of noble gases in a vacuum chamber, in which a Viton® sheet was set. The increase is due to degassing of noble gases from the Viton® sheet or permeating atmospheric noble gases through the sheet. The Viton® sheet is used for gloves in the curation chamber.

atmosphere. Measured rates in $\text{cc}/\text{cm}^2/\text{min}$ are plotted in Fig. 8. The case 1) shows degassing rates from the Viton® sheet. It is shown that baking of the Viton® effectively reduces degassing rates. 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 $\leq 10^{-9} \text{ cc}/\text{cm}^2/\text{min}$. Permeation of heavier noble gases (Ar, Kr, and Xe) seems to be much smaller than those of He and Ne. These results show that the Viton® glove can be safely used for the curation chamber if it is degassed by baking

under vacuum condition.

Chemistries of the Viton® gloves for the clean chambers

As described before, the chamber No.1 and No.2 have two and six Viton® gloves, respectively. The gloves are products of North® by Honeywell International Inc. They are mainly composed of Viton® elastomer, but they also contain some amounts of small talc particles to obtain more stable electrostatic condition in pure gas condition free from H₂O. Before attached to the clean chambers, they had been washed by distilled water and baked in vacuo around 110 °C to decrease adsorbed H₂O on their surfaces.

Viton® is stable in water and organic solvent, and the characteristics of the Viton® gloves used for the clean chambers were checked in Kyushu Univ. A small sheet of Viton® trimmed from the gloves was involved in dichloromethane, and the

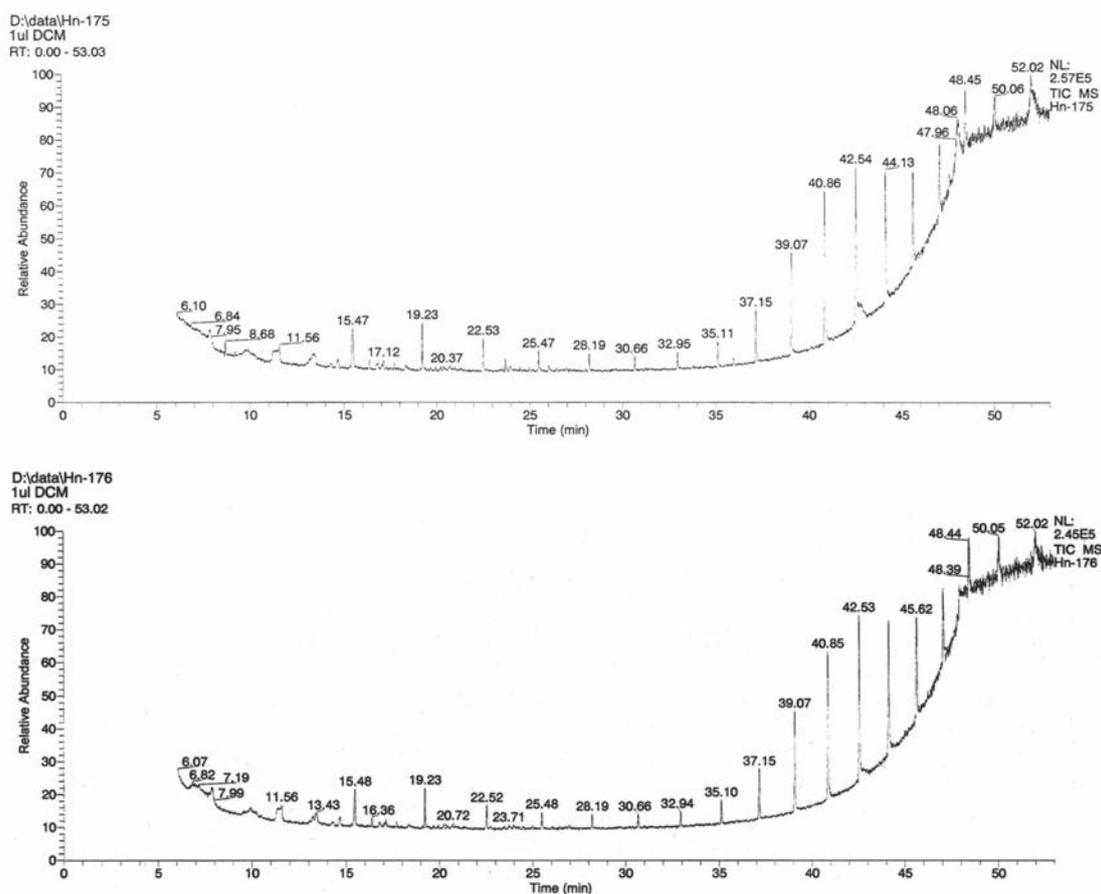


Fig. 9. A GC-MS spectrum of dichloromethane with Viton® sheet trimmed from a Viton® glove used for the clean chamber (upper) and that of dichloromethane blank (lower). There is no different peak in the Viton® spectrum from the blank.

sample solution was analyzed as same as a blank solution by gas chromatography mass spectrometer (GC-MS). An analytical procedure is detailed in Oba and Naraoka (2009). As shown in the analysis results (Fig. 9), there was no difference between the sample and blank, which means that no significant organic contamination other than Viton® itself should not occur from the Viton® gloves.

Cleanliness level accomplished by the cleaning methods in the facility

Every equipment used in the clean chamber is prepared after series of cleaning processes. Their procedures are distinguished based on composed materials. The most typical procedure is a series of ultra sonic cleanings (2-propanol at 40kHz band in frequency, then ultra pure water at 35, 95 and 950 kHz bands respectively in frequency) for all materials such as metals, glasses and Teflon, resistant to cleaning solvents and ultra sonic vibrations. For the quartz glass, an additional cleaning using acid and alkali is performed after the series of ultra sonic cleanings.

We checked the cleanness of the material after its cleanings. Contaminant particles on the material are checked with optical microscopic observations. Almost no contaminant particle larger than a few μm is found just after its cleaning. Such observations are also made with the cameras of the micromanipulation system in the

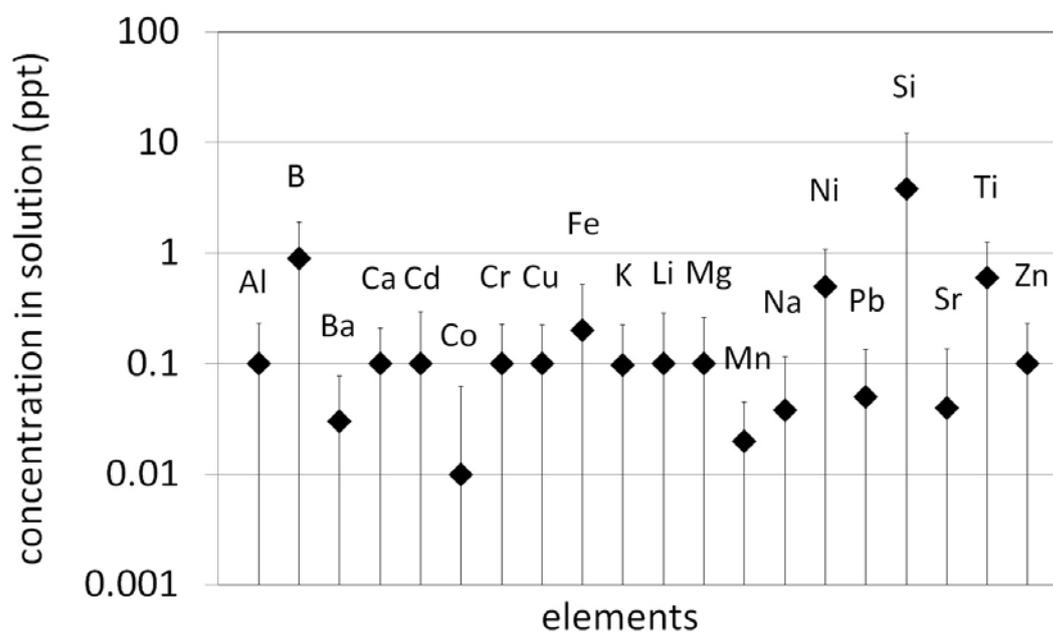


Fig. 10. The concentrations of metal ions in the decocted solution of test quartz pieces in ultrapure water, after acid and alkali cleanings. They are analyzed with ICP-MS. The concentration of 1 ppt in vertical axis corresponds that they are less than 10^{-12} g on 1 cm^2 of glass plate.

clean chamber. SEM observation is also done for the sample holder for SEM. When contaminant particles are found on the holder, additional cleaning such as CO₂ blast cleaning is performed to remove them.

Contaminant ions and organics such as machine oil, grease, and human sebum are checked by the other method. Their abundances are evaluated with the decocted solution of test quartz pieces in ultrapure water, after the acid and alkali cleaning. 20 of cations (Al, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Si, Sr, Ti and Zn) are analyzed with inductively coupled plasma mass spectrometry (ICP-MS) and atomic absorption spectrometry (AAS). Most of them are less than 1 ppt (Fig. 10), corresponding that they are less than 10⁻¹² g on 1 cm² of glass plate. 6 of anions (F, Cl, NO₂, NO₃, PO₄, and SO₄) are also evaluated with ion chromatography (IC). The result is shown in figure 12. PO₄ is less than 1 ppb, but the other anions are around 10 to 20 ppb, corresponding that they are less than about 10⁻⁸ g on 1 cm² of glass plate. Upper limit of total organic carbon (TOC) is ~36 ppb, based on the TOC meter analysis (Fig. 11).

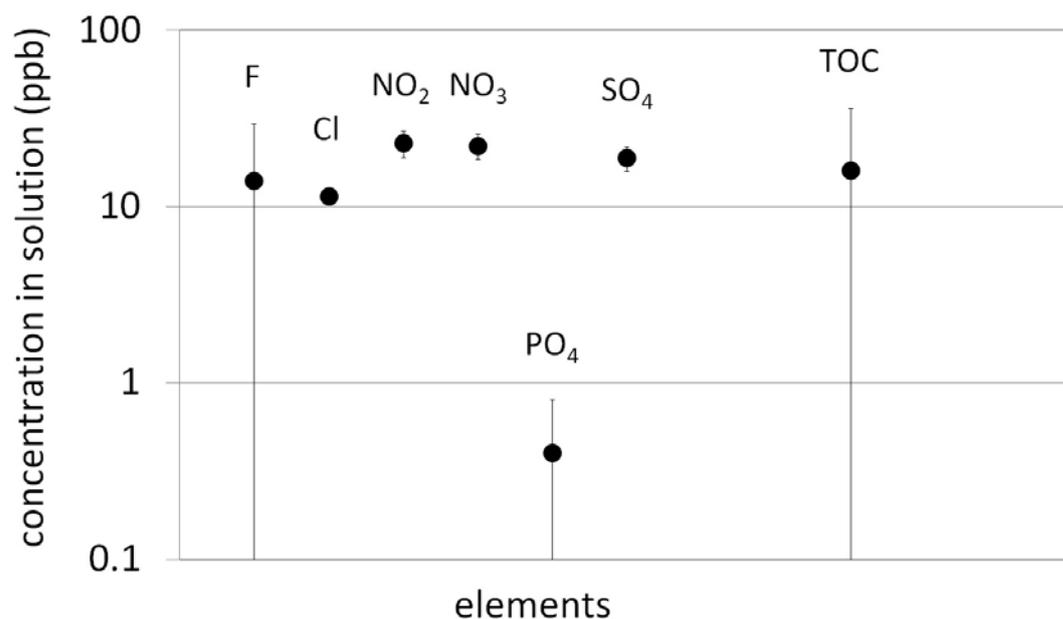


Fig. 11. The amount of anions and organic carbon (TOC) in the decocted solution of test quartz pieces in ultrapure water, after the acid and alkali cleaning. They are analyzed with IC for anions and TOC meter for carbon. The amount of 1 ppt in vertical axis corresponds that they are less than 10⁻⁹ g on 1 cm² of glass plate.

REHEARSAL OF ACCEPTANCE OF A REENTRY CAPSULE AND SAMPLE RECOVERY FROM A SAMPLE CONTAINER

Structures of the reentry capsule and the sample container of Hayabusa

The Hayabusa spacecraft equipped a reentry capsule in which sample recovered from Asteroid Itokawa had been enclosed. The capsule is saucer-shaped 30cm in width and 15cm in height. It is covered with front- and back-side ablators made of carbon fiber reinforced plastic (CFRP) to prevent the sample container, a parachute and related devices from atmospheric entry heating. The sample container is set in the center of the reentry capsule. Figure 12 shows the structure of the sample container. It shapes like up-side down silk hut and sizes 12cm in width 15cm 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. They are mainly made of aluminum alloy (A6061 in JIS code) coated by pure aluminum, and surfaces of the parts which should cause frictions with other parts are coated with Teflon in order to obtain smooth movements, which is to treat the surface of aluminum alloy to be alumite and coat it with Teflon in order to obtain smooth movements (McMurtrey, 1985).

The initial position of the sample catcher together with the lids was on the top

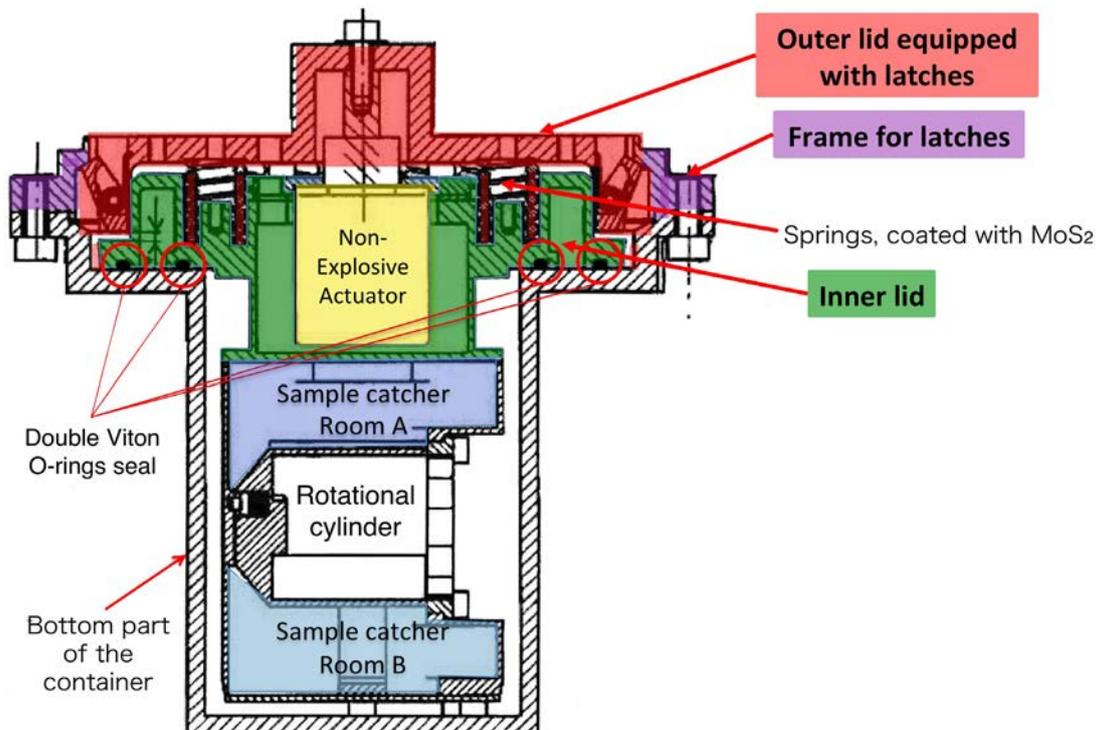


Fig. 12. A schematic view of the sample container of Hayabusa. It mainly consists of an outer lid equipped with latches, an inner lid, a frame for latches, a non-explosive actuator and a sample catcher. The sample catcher is divided into three rooms, room A, room B and a rotational cylinder.

of a sampler horn, and it was transferred from the initial position to the sample container set in the reentry capsule before the departure from Itokawa to the 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 unclosed in the clean chamber No.1. The sample catcher is divided to three rooms, room A, B and a rotational cylinder (Fig. 12). Initial position of the rotational cylinder had been opened for the room B, and the first touchdown of the Hayabusa 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 the room A and B.

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

Rehearsal of a series of processes of the sample container in the facility

We had performed a series of rehearsal 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 Hayabusa reentry capsule return to the Earth in June 2010. A flowchart of the rehearsal is shown in Figure 13. Each step of the processes was repeatedly performed in the same setting as the real part until the technical problems were clarified. Basically all the procedures of the rehearsal were described as procedure manuals for rehearsals, and they were revised after the rehearsals based on the results of the rehearsals to make up procedure manuals for real part of the Hayabusa sample return.

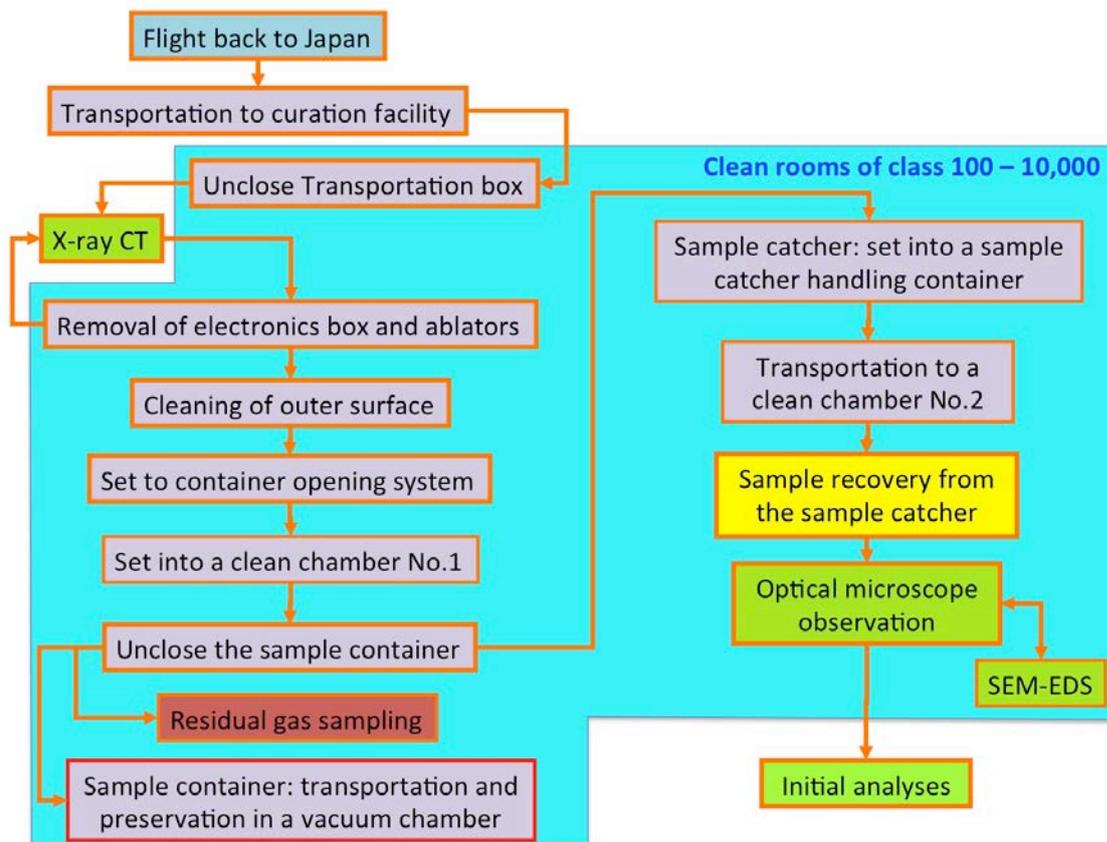


Fig. 13. A flowchart of the rehearsal for a series of processes from an acceptance of the reentry capsule of Hayabusa to a recovery of samples. It took around a year to perform each process of the rehearsal repeatedly.

X-ray computed tomography (CT)

The first step after the acceptance of the reentry capsule is an X-ray computed tomography (CT) imaging of whole the capsule. With this process, the condition of latches and seal of the container and the position of the rotational cylinder could be confirmed. After removal of the electronic box and ablators, the container would be examined with the X-ray CT again in order to confirm existence of samples inside the sample catcher. The X-ray CT used for the rehearsal and the actual Hayabusa returned capsule is Toshiba TOSCANER 24200RE, situated in Chofu Aerospace Center Aerodrome Branch of JAXA. We brought the simulated sample container in which San Carlos olivine grain of several mm and those particles and stainless steel spheres of 200-250 μm was preset and performed test investigations with the X-ray CT instrument.

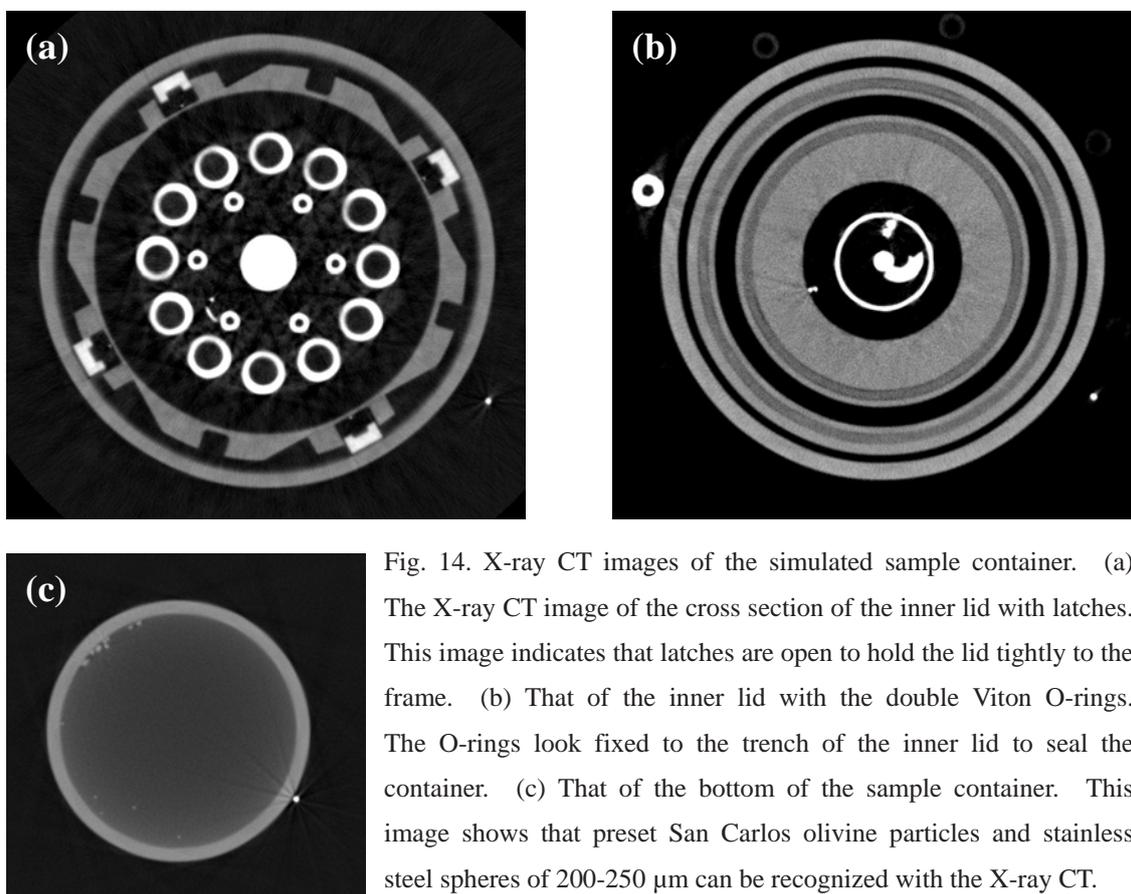


Fig. 14. X-ray CT images of the simulated sample container. (a) The X-ray CT image of the cross section of the inner lid with latches. This image indicates that latches are open to hold the lid tightly to the frame. (b) That of the inner lid with the double Viton O-rings. The O-rings look fixed to the trench of the inner lid to seal the container. (c) That of the bottom of the sample container. This image shows that preset San Carlos olivine particles and stainless steel spheres of 200-250 μm can be recognized with the X-ray CT.

Figs. 14 shows the X-ray CT images of the test investigations. These X-ray CT images indicate that conditions of latches and double Viton® O-rings could be confirmed with these images and silicate particles larger than 200 μm could be recognized with the image.

Removal of the ablators and cleaning the container and check the cleanliness

A back-side ablator could be removed only after machining to expose tops of eight screw bolts which connect the ablator with the outer lid of the container and 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 milling work using a simulated ablator in April 2010, and succeeded in drilling to expose the top of the screw. For the machining at the real part, we prepared special jigs to sustain 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 clean cleaning to be transferred to the quartz slide glasses, then checked 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 the clean chamber No.1. At first, we removed the outer lid, the frame for latches, springs, a non-explosive actuator (NEA) and screw bolts which combine such parts together from the sample container. We prepared special jigs to remove such parts with keeping seal of the container (Fig. 15). After the removal of the parts mentioned above, we connected a joint jig on the inner lid in order 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 in order 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.



Fig. 15. A snapshot of a rehearsal for removal of the outer lid, the frame for latches and NEA from the sample container. The simulated container equips a vacuum flange, though which we could check the condition inside the container.

Opening the sample container and recovering the residual gas

The inner pressure of the sample container of real part will be unknown, thus we have to estimate the pressure and adjust that of the clean chamber to the estimated pressure not to disturb samples in the container due to the gas flow from or to the container because of the difference of pressure between them. For convenience of multiple trials for the pressure estimation, we prepared a cover chamber which can be applied for the vacuum flange of the container opening system and can be set its condition from high vacuum to atmospheric pressure. We had changed both inner pressure of the container and the pressure of the cover flange, and monitored loads and displacements of the four shafts of the opening system.

Because the power to press the inner lid with the springs above is around 160kgf, we pressed 40kgf for each of the four shafts on the container opening system for an initial position of opening. After 150 μm displacement released from the initial position, the load at that position was analyzed. Because the difference of pressure between that in the container and that around it reacts loads on the four shafts which press the inner lid with double Viton® O-rings. Figure 16 is a diagram of external pressure for the container versus mean released load of the four shafts, plotting results

in inner pressure of the container ranged from <100 Pa to 10^5 Pa in each of different settings. The lower the external pressure was, the lower the load was, because the pressure force on the inner lid decreases as the external pressure decreases. Additionally, the load on the inner lid increased as the inner pressure of the container decreased in the same external pressure condition, because the inner pressure of the container reacts to decrease the load on the inner lid of the container. Although the reproducibility of the load in the same condition was not stable, it can be said that the difference of the inner pressure of around $>5 \times 10^4$ Pa could be distinguished from the difference of the load in the different external pressure. Also, the graph shows that the load obtained in the cover chamber were almost reproduced in the clean chamber No.1. Just in case, we also performed the rehearsal of the opening container in conditions that the double Viton® O-ring should have harmed by accident or hardened due to severe conditions in interplanetary space during the round trip to the Earth. As a result, those differences of initial conditions did not seem to make differences in values of loads from the case of Viton® O-rings in the normal condition.

As the container would be opened in the clean chamber No.1, residual gases which should be released from the container would be captured in gas cylinders for capturing residual gas which are connected to the clean chamber No.1. The gas cylinders have been baked >180 °C to decrease the background interferences. The background interference of the baked gas cylinders were checked in the same noble gas mass spectrometer in the University of Tokyo as described above. They resulted in their background were good enough for the noble gas analyses.

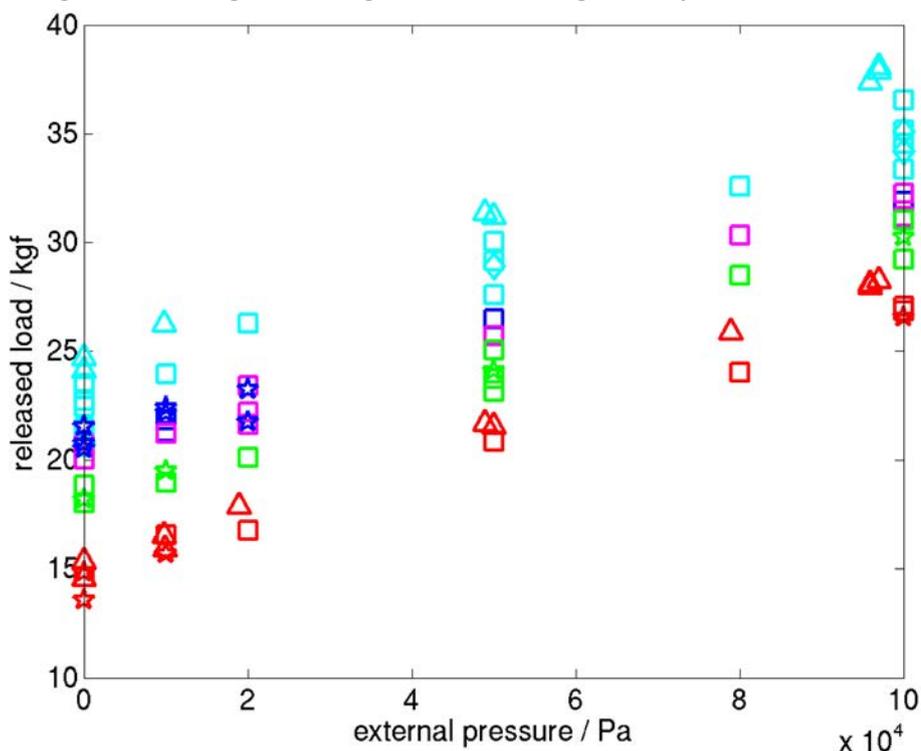


Fig. 16. Mean released load from four shafts after displacing 150 μm from the point initially loaded to 40kgf. Color of each symbol indicates a container inner pressure as follow: < 100 Pa [cyan], 10^4 Pa [blue], 2×10^4 Pa [magenta], 5×10^4 Pa [green] and 10^5 Pa [red]. Symbols represent data acquired with apparatus conditions as follow: chamber No.1 [triangle], cover chamber with the sample container of normal O-rings [square], cover chamber with the sample container of purposely damaged O-rings [diamond] and that of baked [pentagram].

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 next to the clean chamber No.1 to transfer the sample container from the sample opening system to the cabinet No.1 via the transportation chamber both in the atmospheric pressure nitrogen condition and the vacuum condition. In the

transportation chamber, we also check images inside the container through 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 condition of the clean chamber No.1 was set to atmospheric pressure nitrogen, and the sample catcher together with the inner lid was transported with a special rod handled by two Viton® gloves and set to the a 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 remove them from the bolts with a plier made of stainless steel before we unscrewed the bolts. With this procedure, we will get the first look of the catcher room A in real part, we therefore checked images of inside the room A through a glass viewport of the top of the chamber with the ultra-long working distance microscope which was set above the viewport. Then the sample catcher was enclosed into the sample catcher handling container, and sent to the clean chamber No.2 via a gate valve between the chamber No.1 and 2. In the 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 firstly tried to handle simulated particle (San Carlos olivine and synthetic nickel olivine of 30-500 μm is size) in the cleanroom air condition using a reflecting and polarizing microscope with an oil hydraulics micromanipulator setting a quartz glass probe with a platinum wire connected with a direct-current (DC) power source. The simulated particles were put on a quartz slide glass whose back side was coated by gold and connected to another DC power source. We tried to lift up the particles on the glass plate and release it on a different place of there. Although the largest nickel olivine of 500 μm cannot be handled, San Carlos olivine of 30-100 μm can be handled with the manipulator in the electronic condition between -12V and +16V DC for the probe voltage.

Next, we brought all the instruments above into the glove box in the electron microscope to have a rehearsal of manipulation in the same hardware setting with the first one in nitrogen condition. We could handle San Carlos olivine particles of 50-150 μm in the electric condition of $\pm 50 \sim \pm 100\text{V}$ DC for the probe and $\pm 30\text{V}$ DC. We also had a rehearsal in the same condition but the simulated samples were put on a PTFE plate with a stainless steel plate underneath. As a result, it was more difficult in

releasing the particle than lifting it up, maybe because of the difference of the material of the plate.

Finally, we had a rehearsal of the electrostatically controlled micromanipulation system in the 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 of $\pm 50 \sim \pm 100\text{V}$ DC for the probe voltage and $0 \sim \pm 30\text{V}$ DC for the stage. We also tried to handle the

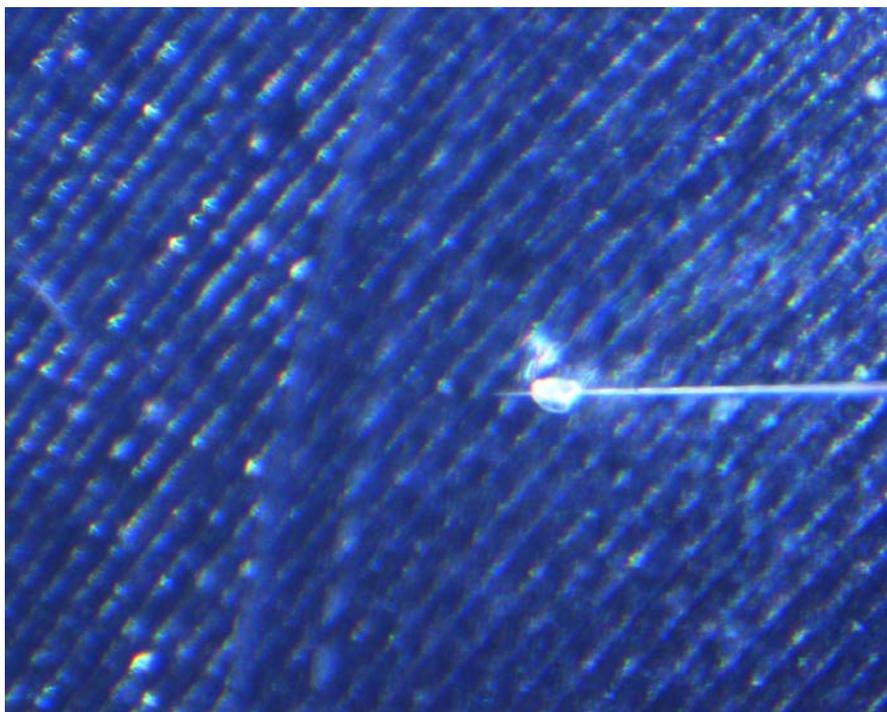


Fig. 17. An optical microscope image of a nickel olivine particle of $>100 \mu\text{m}$ lifted up with a quartz glass probe of an electrostatically controlled micromanipulator system in the clean chamber No.2. The background is a surface of inside wall of a simulated sample catcher.

nickel olivine particle inside the simulated sample catcher and confirmed to lift up and release the particle with the manipulator system (Fig. 17).

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 have been performed, following the flowchart of the rehearsal as shown previously (Fig. 13).

First, the transportation box was opened to extract reentry capsule sealed in plastic bag with nitrogen. The nitrogen gas filled in the bag was recovered in a glass cylinder just in case. After the container was back to the clean room in Sagamihara from the second X-ray CT, a thermosensor, which had been attached bellow 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 up with a special vacuum cleaner. Then the outer surface of the container was cleaned with a series of processes described bellow. First, it was wiped with clean cloths wetted by isopropanol 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. For the confirmation of cleanliness of the surface of the container, it was checked via swabs for clean cleaning and/or clean slide glasses with an optical microscope, a dynamic contact angle meter and a Fourier transform infrared spectroscopy. All the three tests showed no sign of contamination. Next, the container was sent to class 1,000 clean room and fixed to the container opening jigs, which was designed to hold its inner lid keeping its seal and remove its frame for latches and its outer lid. After the series of processes related to the jigs, its inner lid was held with claw jigs, and the gap between the inner lid and the container's flange was 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 was removed from the container's flange. Then the container opening system with the container fixed joined the clean chamber No.1, in which ultra-pure nitrogen was continuously purged during the joint. All the process described above had been finished until June 20th. The environment of the clean chamber No.1 was purified with a circulated nitrogen purifier equipped with the chamber for an all night of June 20th.

Opening the sample container

Here we describe processes to estimate the pressure inside the container and set the pressure of the clean chamber No.1 close to that of the container as much as we could, in order to minimize a disturbance caused by gas flow between inside and outside the container.

The container opening system contains sets of load cells and displacement sensors for four axes. Rehearsals of the opening of the container had been performed with the opening system and an imitation container in a positive, negative or equal pressure inside the container compared to the outside, before the actual container return. During the rehearsals, trend of data of the load cells and the displacement sensors had been recorded as shown in Fig. 18.

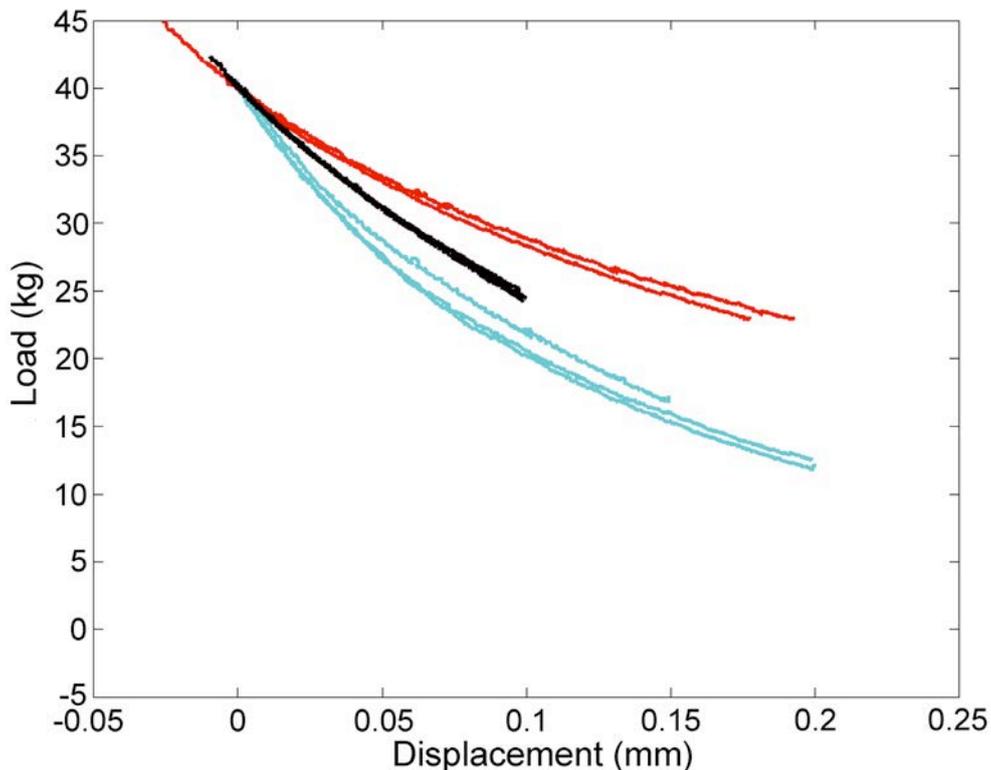


Fig. 18. Correlation curves between displacements and load for the container opening system in <math><100\text{Pa}</math> pressure of the clean chamber No.1. Red curves represent those obtained when the container was atmospheric pressure, and blue curves represent those obtained when it was vacuum in rehearsals. Black curves represent those obtained when the actual container of unknown inner pressure was slightly pulled for its lid.

In the case of the actual returned container, the inner lid was pulled slightly with the sensors recorded on 21 June and the data was compared with the rehearsal data. A comparison with the data obtained in rehearsals and those in the actual case indicates that the inner pressure of the container would be between vacuum and air pressure. Based on this estimation, the pressure condition of the clean chamber No.1 was set to vacuum, not in a dynamic but a static condition.

Simultaneously, residual gas sampling cylinders which had equipped to the clean chamber No.1 were prepared to capture gas released from the container at the timing of its opening. Since it was estimated that gas inside the container should

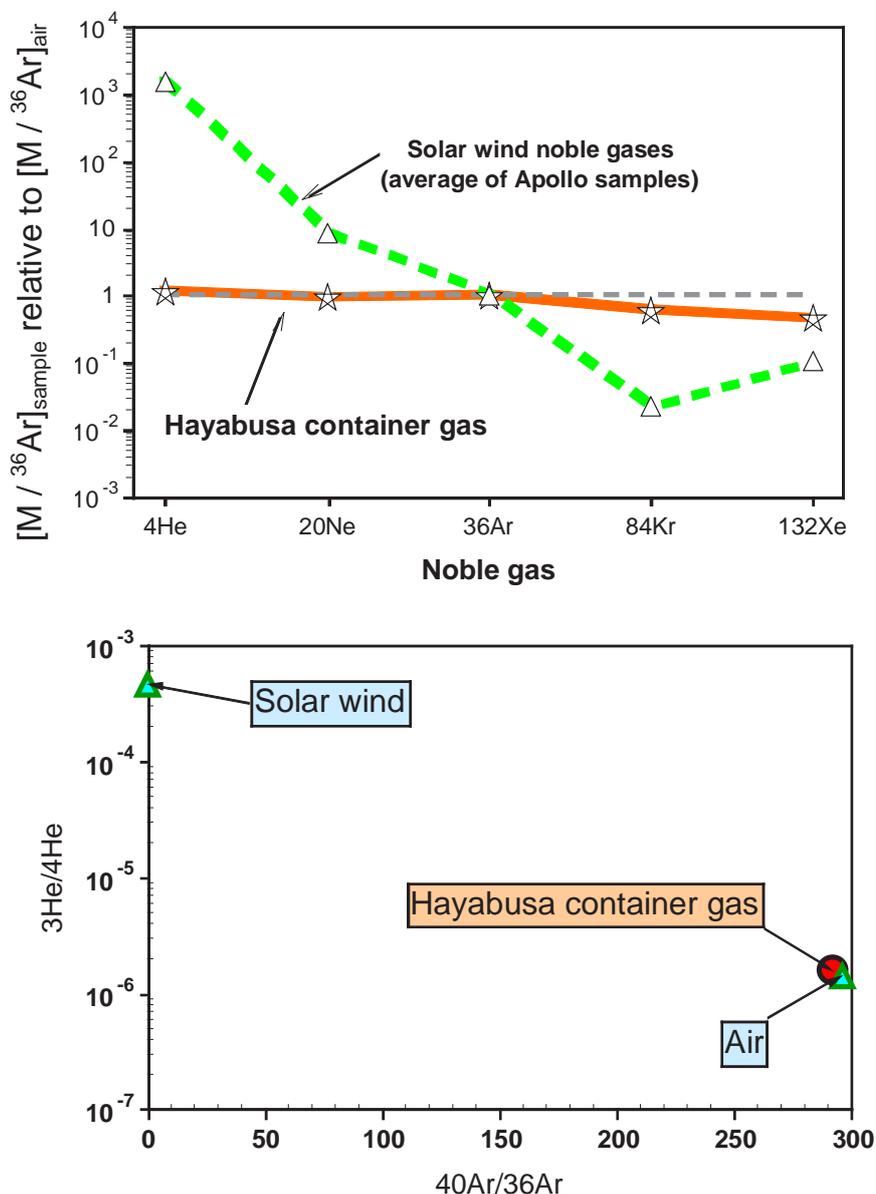


Fig. 19. Graphs showing elemental (top) and isotopic (bottom) ratios of the noble gas collected from the sample container. The top graph shows relative elemental abundances in $^4\text{He}/^{36}\text{Ar}$, $^{20}\text{Ne}/^{36}\text{Ar}$, $^{84}\text{Kr}/^{36}\text{Ar}$ and $^{132}\text{Xe}/^{36}\text{Ar}$ of Hayabusa container gas normalized by those of terrestrial air. They are very close to 1 (black dashed line) and different from those of solar wind noble gases. The bottom graph shows a diagram of $^{40}\text{Ar}/^{36}\text{Ar}$ versus $^3\text{He}/^4\text{He}$, plotting Hayabusa container gas with terrestrial air and solar wind as references. The Hayabusa container gas almost overlaps terrestrial air, which plots far from solar wind.

contain terrestrial atmosphere to some extent, O₂ could be also a sign to distinguish the opening of the container.

Therefore, the opening of the container was performed under monitoring the data of the load cells and displacement sensors of the opening system and those of a differential pumping quadrupole mass spectroscopy, which can detect >1ppm of O₂ in air pressure nitrogen, analyzing the environment of the clean chamber No.1 in order to recognize the opening of the container. 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 both the monitored data showed the signs of the opening, the gas inside the clean chamber No.1 was captured in the gas cylinders. The processes to open the container had been finished in 22 June, and the clean chamber No.1 was evacuated for a whole day of 23 June.

Noble gases sampled in the gas cylinders were analyzed at the Univ. of Tokyo (Okazaki et al., 2011). Elemental and isotopic ratios of the noble gases collected from the sample container are essentially identical to those of terrestrial atmosphere (Fig. 19). There is no sign showing the enrichment of solar gas as found in lunar samples (Fegley and Swindle, 1993).

Sample recovery from the sample catcher

On 24 June, the container was transferred to another chamber which attaches to the clean chamber No.1 with a transfer rod, then the inner lid and the sample catcher was 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 a catcher handling container, and the container include the catcher was transferred to the clean chamber No.2, which was designed for sample handling in purified nitrogen condition and equipped with an electrostatic micro-manipulator combined with optical microscopes. The inner surface of the catcher room A was observed in detail by the optical microscopes, and it is figured out that not many particles larger than a few hundred μm were found inside the room A.

First, we tried to pick up particles from the catcher room A directly with micro-manipulator, although it was unsuccessful because of difficulty in recognizing particles on the cutting-work surface of the interior of the sample catcher room made of aluminum alloy coated by pure aluminum (Fig. 20).

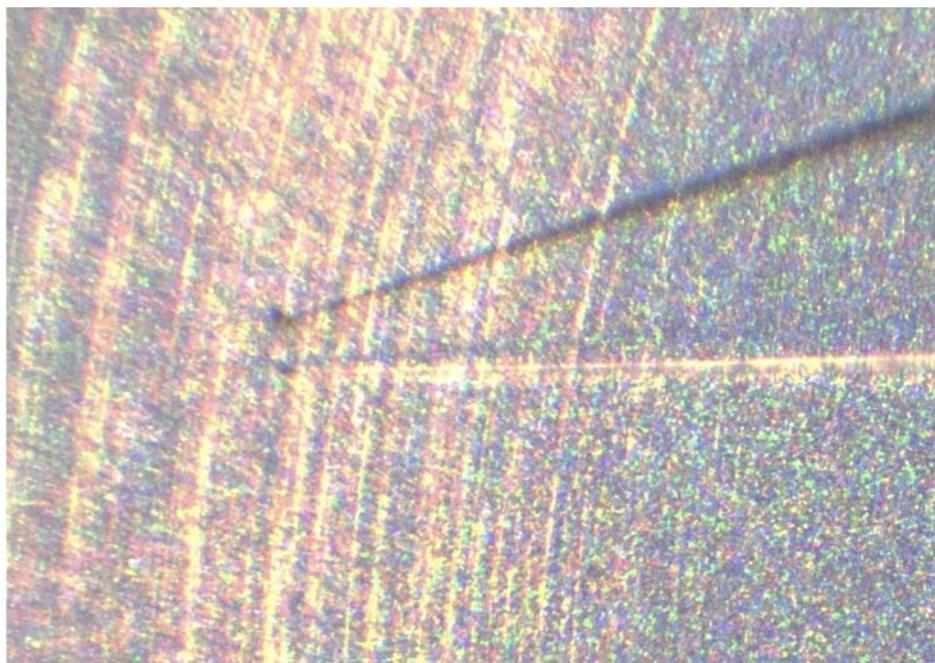


Fig. 20. An optical microscopic image of a small platy particle picked up from the inside surface of the sample catcher room A of Hayabusa. It is difficult to recognize the real particle in the image, its presence is confirmed by its shadow.

Second, we prepared a special Teflon spatula to scoop the interior surface of the 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 large amount of small particles, but we could not pick up them and distribute for initial analyses because they are too small and fixed to the surface of spatula.

Third, we prepared a silica glass plate which can be attached to the opening of the catcher room A and make particles compulsory fall on it by tapping catcher handling container. In this method, we succeeded to recover particles on the plates and picked them up to analyze with FESEM-EDS and distribute for initial analyses. We also tried this method for the catcher room B. Figures 21 show microscopic images of the quartz disks gathering particles from the catcher room A and B. First results of the initial analyses concluded that the particles recovered from catcher room A should originate from Asteroid Itokawa (Nakamura et al., 2011; Noguchi et al., 2011; Tsuchiyama et al., 2011; Ebihara et al., 2011; Nagao et al., 2011; Yurimoto et al., 2011).

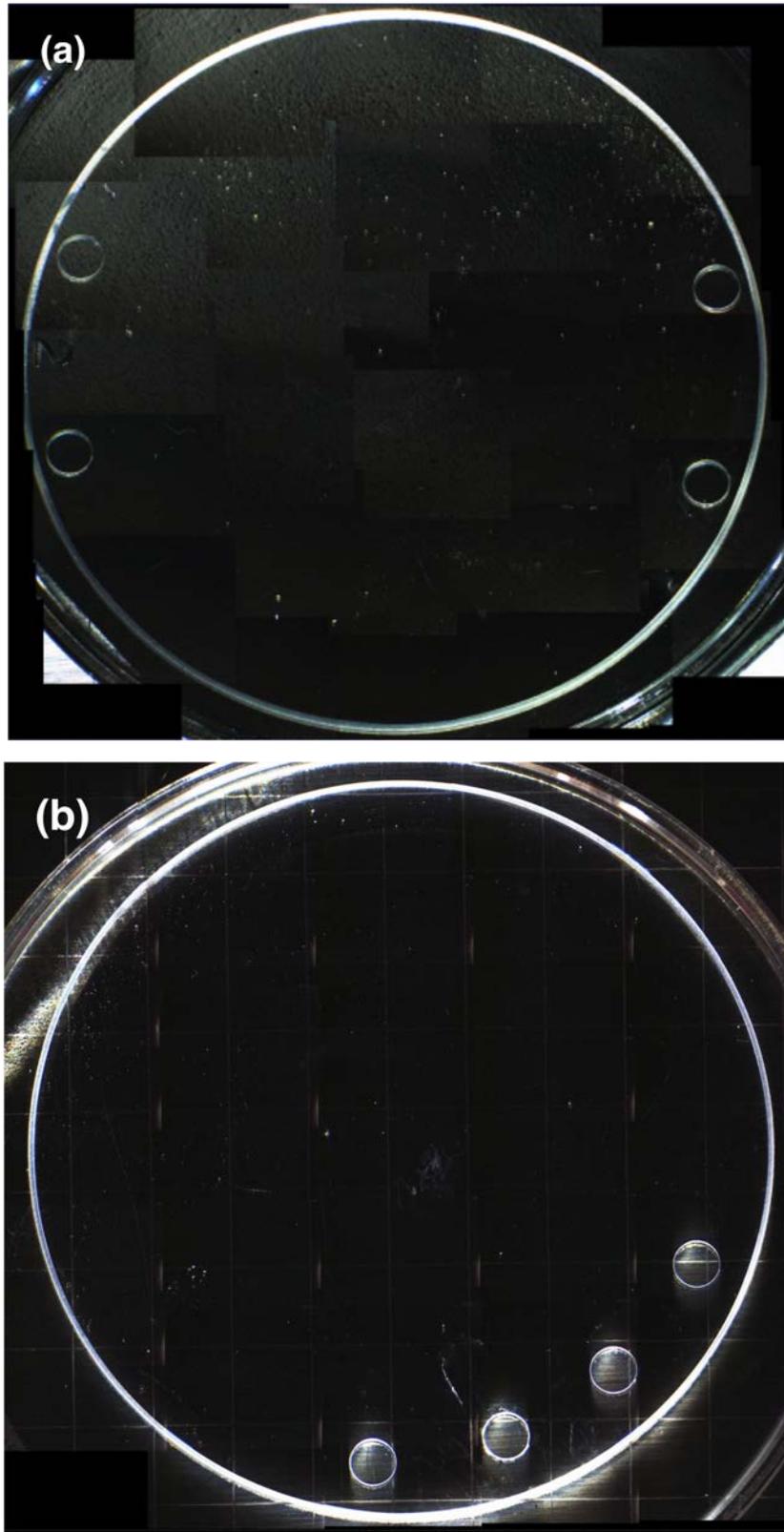


Fig. 21. Mosaic images of quartz glass disks set to the openings of the sample catcher room A (a) and B (b). Each of them sizes 48mm in diameter. The numbers of particles recognized on the room A disk is larger than those of room B.

MANAGEING THE HAYABUSA RETURN SAMPLES

Initial description (optic microscope, FESEM-EDS)

In this section, the operation procedure for the HAYABUSA return samples of compulsory free fall method on quartz disk glass is described.

The samples on quartz disk glass are picked up by the electrostatically controlled micromanipulation system as described above. The picked up samples are observed by two optic microscopes put in the system, and recorded to photograph (Fig. 22). These optic microscopes are observed the samples at immediately above and aslant around 45°, the information of describing data are the positions on the quartz disk slide, the sample sizes and the sample colors.

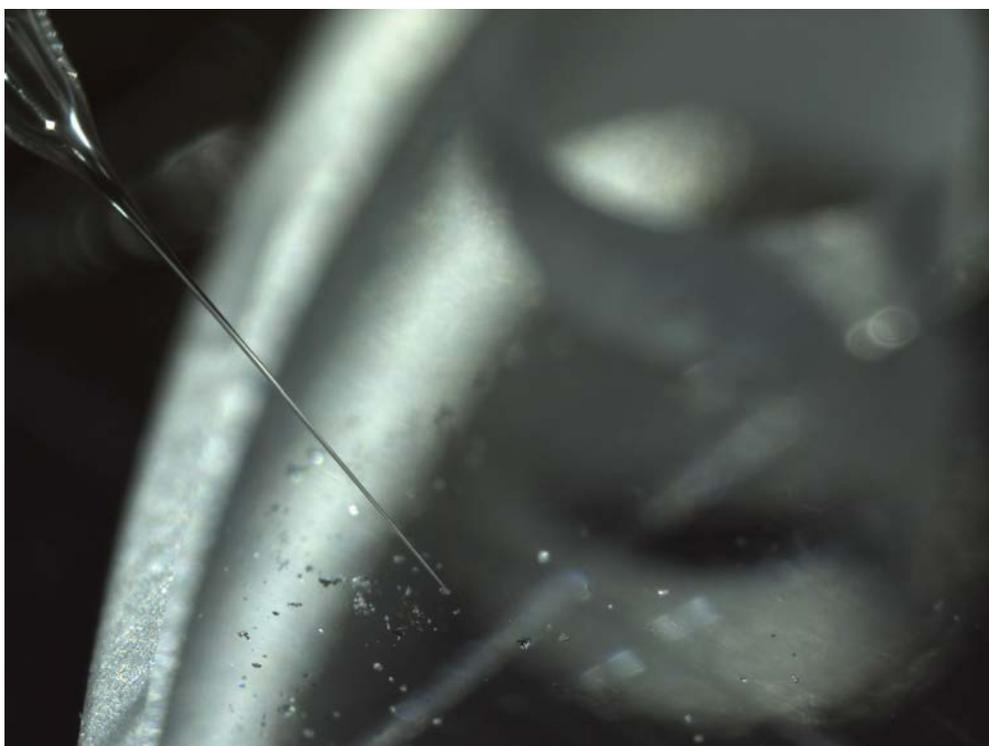


Fig. 22. An optical microscopic image of RA-QD02-0070 picked up with the micromanipulator from the quartz glass disk of catcher room A. This image was taken by the microscope observing the samples from 45° angle. The particle looks translucent in the image.

The picked up samples are transferred to SEM holder by the micromanipulation system. The SEM holder for the sample observation is constructed by gold coated copper, and the gold disk with dimples is laid in the place where samples are put. The SEM holder is covered with a cap, after having moved the samples on the dimple of the gold disk in the SEM holder. The cap is sealed with a Viton® O-ring,

and the samples come to be surrounded by purification nitrogen environment of the clean chamber by covering the SEM holder with the cap in the clean chamber.

The samples transferred to the SEM holder are observed by FESEM-EDS. The sample holder is put in a sample introduction room of the FESEM in a state kept atmosphere interception and purification nitrogen environment, and the nitrogen gas in the introduction room is decompressed carefully slow so that the very small samples are not blown. The samples are observed with a low vacuum mode as no vapor deposition. The configuration of whole sample are observed by BSE and ESED images, and the samples are analyzed the chemical composition for approximately five points by EDS (Fig. 23).

Based on qualitative chemical analyses of the EDS, particles recovered from the sample catcher are grouped into four categories. Category 1 particles represent olivine, pyroxene and feldspar (Fig. 23a). Category 2 particles consist of not only

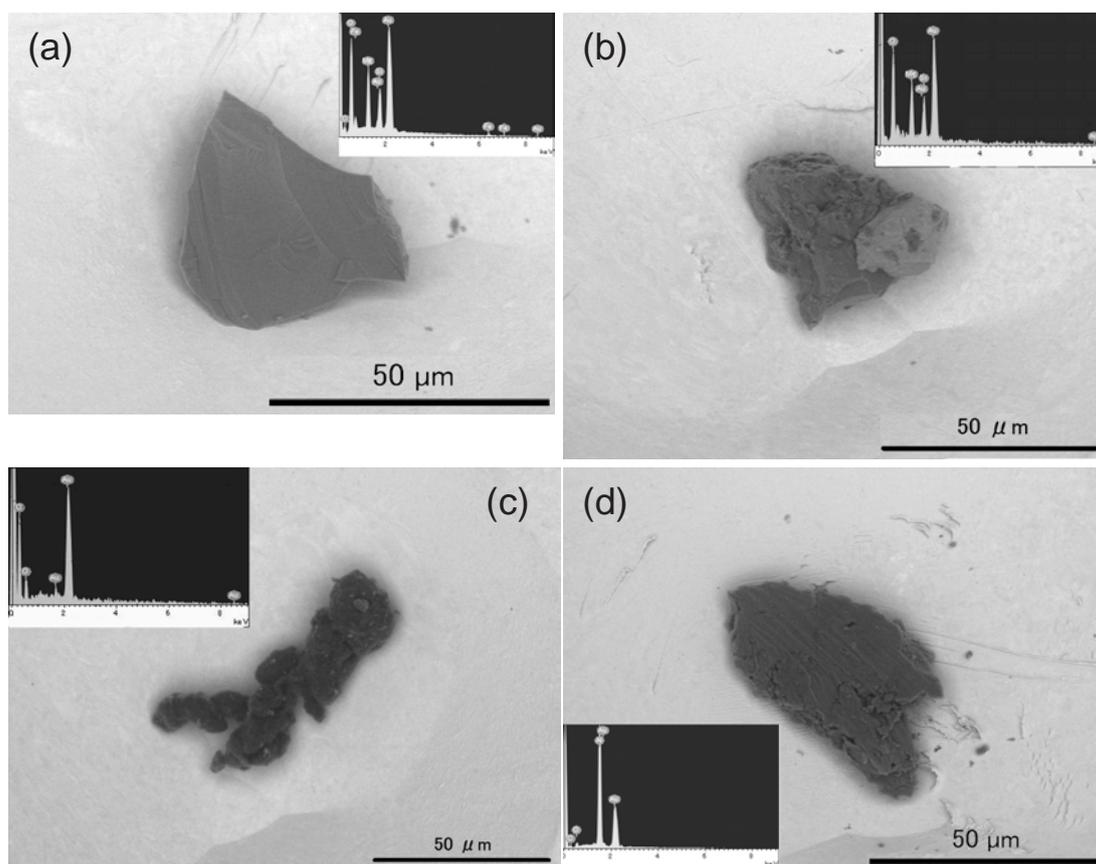


Fig. 23. BSE images and EDS spectra of RA-QD02-0144 grouped as category 1 (a), RA-QD02-0088 as category 2 (b), RA-QD02-0091 as category 3 (c) and RA-QD02-0227 as category 4 (d), respectively. As shown in their BSE images and EDS spectra, category 1 and 2 particles are mainly composed of ferromagnesian 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, chip of stainless steel and etc.

transparent minerals as described in Category 1 but also opaque minerals such as iron sulfide, iron-nickel metal, chromite and etc. (Fig. 23b). Category 3 particles are mainly composed of carbon often containing oxygen, nitrogen and sulfur, supposed to be organic carbon (Fig. 23c). Category 4 particles are artifacts such as aluminum flakes, fragments of quartz glass, stainless steel etc. (Fig. 23d).

Principles of giving sample ID

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

The room name becomes RA or RB, means room A and room B, respectively. If the samples were directly picked up from the catcher, the room name is 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 are 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 new ID name to the samples. In the case of ultrathin sections, pristine mass of the particles is 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 sub-numbers are given to the ultrathin sections, such as RA-QD02-XXXX-YY, where YY is the sequential number of ultrathin sections or transmission electron microscope (TEM) grids. On the other hand, if the samples are divided or broken into multiple pieces due to other method or reasons, the pieces of the sample are named as RA-QD02-XXXX-YY, YY is the sub number 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 data archive section. The sub numbers are adhered to the end of the sample name if the samples are divided or broken again. For example, if ultrathin sections are formed from a previously divided sample,

then the ID name of the ultrathin sections becomes RA-QD02-XXXX-YY-ZZ, where ZZ is the sub-number for the ultrathin sections.

Preservation of the ID given samples

The samples after observation of SEM-EDS analyses are moved with the SEM holder under atmospheric pressure purification nitrogen to the clean chamber. After having moved to the clean chamber, the cap of the SEM holder is opened and the holder is put on the stage of the manipulation system. The particles are picked up while confirming a position of the sample on the gold disk of SEM holder with the microscopes, and the samples are moved on the gridded quartz slide glass which has been washed into as described above. The particles larger than 50 μm are moved in a hollow of the dimple slide glass, and the particles which are smaller than 50 μm are put on the flat slide glass (Fig. 24).

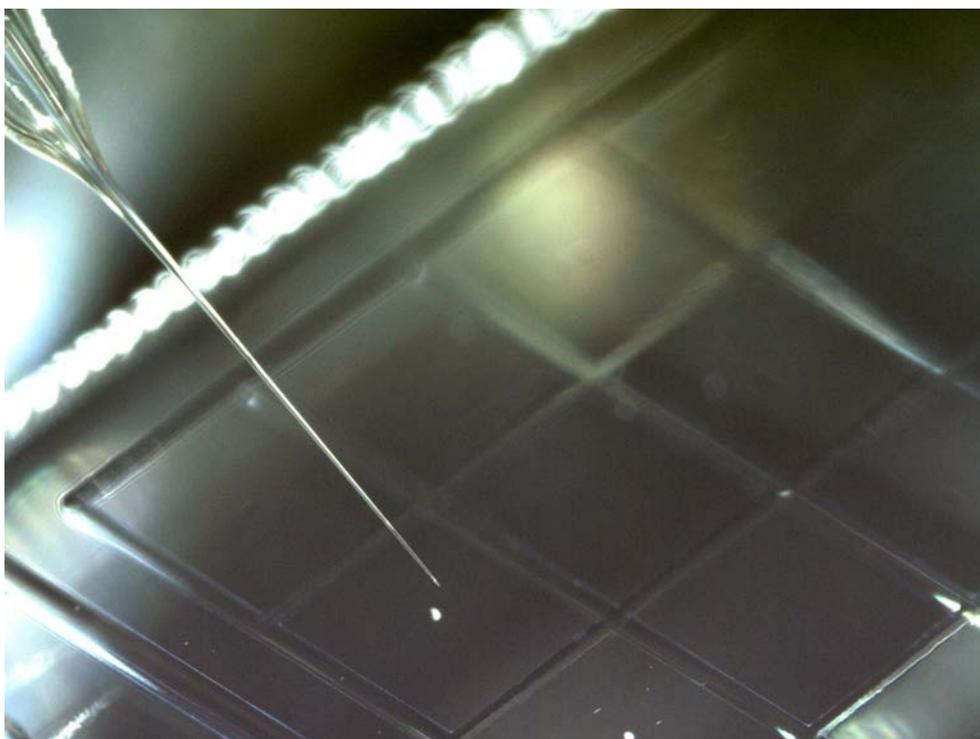


Fig. 24. An optical microscopic image of the Hayabusa returned sample RA-QD02-0070 released on the gridded slide glass. Each particle is put on the center of one of the grid one by one.

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 works, such as images of optical microscope and EDS

spectrum and BSE images, is stored to the database workstation via the closed network in the curation facility. We can quickly check and/or retrieve the daily data from the database. The data relating to the samples are automatically converted into the sample database, which additionally includes sample information such as current status, size, mineral phases and category, transfer history and comments. The database and original data are stored to backup HDDs in a certain periods separately. The database management is basically operated by the curation staff and curator. These databases are basically operated for the curation works as shown in this section. However, the data provided to the researcher in electric media or paper as necessary. Results of the initial analysis and A/O analysis are also stored to the database according to the samples.

SAMPLE DISTRIBUTION FOR INITIAL ANALYSES AND NASA

Designs of sample containers for initial analyses

Because of the differences in analyses conditions of initial analyses, containers of the sample containers, their environments and their cleaning methods were different each other. The samples which were planned to be analyzed by a scheme called “mainstream,” which includes synchrotron radiation CT and diffraction, electron microprobe and ion microprobe, were mounted on the top of glass fibers with crystal bond and set in styrol cases in the cleanroom air condition and transported within a stainless steel container filled with nitrogen. Those for space weathering analysis, operated mainly by transmission electron microscope analyses were mounted in epoxy resin in a special glove box of nitrogen condition and transported within the same container as mentioned above, thus they were processed without exposing to the air. Those for noble gas analyses were set in holes on stainless steel base in a special stainless steel flange which had been baked beforehand to decrease its background. This process was done in the clean chamber No.2, thus the samples should have been kept in nitrogen condition until they arrived to 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 cleanroom air, set in a stainless steel shield, covered with aluminum foil and transported in the same container used for the mainstream. And those for the solar wind analysis, including FESEM-EDS, focused ion beam manufacturing system and ion microprobe, were put on quartz glass container coated with gold in the cleanroom 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 (Fig. 25). All parts of the container are separately cleaned and brought in the clean chamber before their assemblage. The sample is 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 the clean chamber No.2. A metal plate is set under the lower plate in order to increase the ability of the electrostatic control of the sample handling with the micromanipulator. After the sample is put in the dimple, the lower plate is covered with the other plain glass plate. The sample can be observed though the cover plate. The pair of the plates is hold with clips and screws in the machined lower flange, and covered with the other normal flange. The flanges are sealed with six screw bolts and oxygen-free copper gasket coated by gold. The sealing is also performed in the clean chamber, so the inside of the container is filled with atmospheric pressure nitrogen in the clean

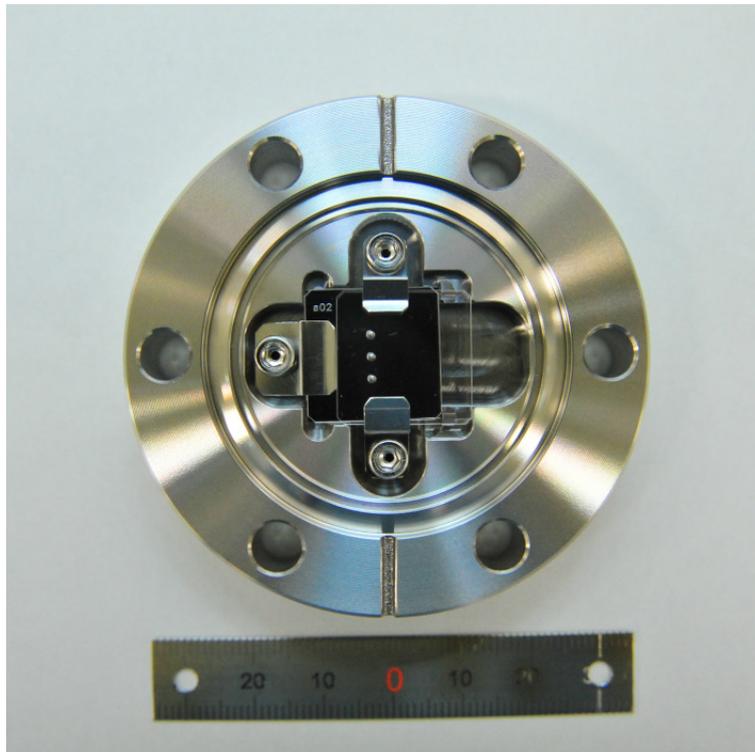


Fig. 25. A photograph of a sample container used for the NASA sample distribution. Its outer container is made of stainless steel, and its inner container is a pair of glass slide made of synthetic quartz glass, on which sample particles are enclosed.

chamber. We use only three materials for the sample container, synthetic quartz, gold and stainless steel. All the parts of the container are cleaned beforehand in the same manner described in previous session.

FUTURE PLANS

Plans for international A/O

The sample distribution of international A/O 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 A/O. The recovered amount is small and the total amount is still uncertain, however, about one hundred particles, including particles returned from the initial analyses, with the preliminary examined ones were opened to the public as the distributed samples of international A/O. Investigators who propose research of the distributed samples submit proposals. The proposals were reviewed by reviewers and A/O committee members. The first international A/O was released in 21 January 2012 and closed in 7 March 2012. Distribution of these samples started in June 2012. Distributed samples in the first international A/O 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 category 3 and 4, those processed by FIB or sectioned by an ultramicrotome in the future international A/O or other announcement.

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. Only the compulsory free fall method was applied for the catcher room B, because destruction of the particles which might have happened in the spatula scooping the inner surface of the catcher room A should be avoided. Since 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 room 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 the catcher room A and B, the rotational cylinder should be removed during the disassembly. After the disassembly, samples in the room A, B and the rotational cylinder will be

completely mixed up, although there is no choice to recover 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 A/O analyses will continue. However, it is sure that some fraction of the Hayabusa samples should be preserved for the future after the distribution to the international A/O analyses and NASA. The samples kept for the future will be divided in some different level due to their processes, such as those kept adhered to the sample catcher without picked up, those observed by FESEM-EDS for classified into the categories, those only picked up without FESEM-EDS observation. The particles other than the Itokawa origin should be also precious samples for sources of information to estimate things 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 the clean chamber No.1 in the state of the vacuum (below 10^{-5} Pa) after its transportation from the clean chamber No.1 since 24 June, 2010. We are planning to send some fraction of the recovered samples to the cabinet attached to the 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 since a 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 feature 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 a big scientific result through our curation activity.

SUMMARY

Since the construction of the PMSCF/JAXA had finished 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 return sample curation. A year before the reentry of Hayabusa capsule, we started to perform a series of rehearsal 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 the curation facility on 18 June 2010, three days after the reentry, and experienced a series of processes, which we performed in the rehearsal. Within seven days after the capsule reentry, the sample container was introduced into the 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, the sample catcher was set to the catcher-handling container and brought to the clean chamber No. 2 in atmospheric pressure nitrogen condition. In the 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 can recover particles in the sample catcher which can be handled with the electrostatically controlled micromanipulation system. Thus far, particles are picked up from the quartz disks of both catcher room A and B, routinely analyzed with the FESEM-EDS, categorized to four groups based on chemical composition, and preserved on quartz slide glasses without exposing 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 for initial analyses, NASA and will be distributed to the international A/O. New particles from the sample catcher will be recovered with other methods in the near future. We are now going to prepare for the sample returned by Hayabusa 2 and other missions.

ACKNOWLEDGMENTS

We owe so much to every member of Hayabusa project team for such a great accomplishment to succeed in the round trip to an asteroid for the world first time and return the very world first asteroidal samples back to the Earth. We are so appreciated to all the members of the advisory committee for specification of the curation facility. We thank Prof. Don Brownlee so much about for reviewing our paper and having valuable advice on it to improve it. Our special thanks go to the all staffs of the maker to establish the curation facility in Sagamihara campus of JAXA. We are so grateful to Prof. Akira Fujiwara and Dr. Hajime Yano for establishing the basic concept of the curation facility. We express best thanks to Prof. Tetsuya Yamada for giving precious information and advice on the reentry capsule and conducting the capsule recovery team to have accomplished a perfect success in recovering the Hayabusa reentry capsule.

REFERENCES

- Abe M., Takagi Y., Kitazato K., Abe S., Hiroi T., Vilas F., Clark B. E., Abell P. A., Lederer S. M., Jarvis K. S., Nimura T., Ueda Y., and Fujiwara A. 2006. Near-Infrared spectral results of asteroid Itokawa from the Hayabusa spacecraft. *Science* 312: 1334-1338.
- Abe M., Fujimura A., Yano H., Okamoto C., Okada T., Yada T., Ishibashi Y., Shirai K., Nakamura T., Noguchi T., Okazaki R., Zolensky M. E., Sandford S. A., Ireland T., Ueno M., Mukai T., Yoshikawa M., Yamada T., Kunihiro H., and Kawaguchi J. 2011. Recovery, Transportation and acceptance to the curation facility of the Hayabusa re-entry capsule (abstract #1638). 42nd Lunar and Planetary Science Conference. USB memory.
- Allton J. H., Bagby Jr. J. R., and Stabekis P. D. 1998. Lessons learned during Apollo Lunar sample quarantine and sample curation. *Adv. Space Res.* 22: 373-382.
- Binzel R. P., Rivkin A. S., Bus S. J., Sunshine J. M., and Burbine T. H. 2001. MUSES-C target asteroid (25143) 1998 SF36: A reddened ordinary chondrites. *Meteoritics & Planetary Science* 36: 1167-1172.
- Brownlee D., Tsou P., Aléon J., Alexander C. M. O'D., Araki T., Bajt S., Baratta G. A., Bastien R., Bland P., Bleuet P., Borg J., Bradley J. P., Brearley A., Brenker F., Brennan S., Bridges J. C., Browning N., Brucato J. R., Brucato H., Bullock E., Burchell M. J., Busemann H., Butterworth A., Chaussidon M., Chevront A., Chi M., Cintala M. J., Clark B. C., Clemett S. J., Cody G., Colangeli L., Cooper G., Cordier P. G., Daghlian C., Dai Z., D'Hendecourt L., Djouadi Z., Dominguez G., Duxbury T., Dworkin J. P., Ebel D., Economou T. E., Fairey S. A. J., Fallon S., Ferrini G., Ferroir T., Fleckenstein H., Floss C., Flynn G., Franchi I. A., Fries M., Gainsforth Z., Gallien J.-P., Genge M., Gilles M. K., Gillet P., Gilmour J., Glavin D. P., Gounelle M., Grady M. M., Graham G. A., Grant P. G., Green S. F., Grossemy F., Grossman L., Grossman J., Guan Y., Hagiya K., Harvey R., Heck P., Herzog G. F., Hoppe P., Hörz F., Huth J., Hutcheon I. D., Ishii H., Ito M., Jacob D., Jacobsen C., Jacobsen S., Joswiak D., Kearsley A. T., Keller L., Khodja H., Kilcoyne A. L. D., Kissel J., Krot A., Langenhorst F., Lanzirotti A., Le L., Leshin L., Leitner J., Lemelle L., Leroux H., Liu M.-C., Luening K., Lyon I., MacPherson G., Marcus M. A., Marhas K., Matrajt G., Meibom A., Mennella V., Messenger K., Mikouchi T., Mostefaoui S., Nakamura T., Nakano T., Newville M., Nittler L. R., Ohnishi I., Ohsumi K., Okudaira K., Papanastassiou D. A., Palma R., Palumbo M. O., Pepin R. E., Perkins D., Perronnet M., Pianetta P., Rao W., Rietmeijer F., Robert F., Rost D., Rotundi A., Ryan R., Sandford S. A., Schwandt C. S., See T. H., Schlutter D., Sheffield-Parker J. A., Simionovici S., Sitnitsky S. I., Snead C. J., Spencer M. K., Stadermann F. J., Steele A., Stephan T., Stroud R., Susini J., Sutton S. R., Taheri

- M., Taylor S., Teslich N., Tomeoka K., Tomioka N., Toppani A., Trigo-Rodríguez J. M., Troadec D., Tsuchiyama A., Tuzolino A. J., Tyliczszak T., Uesugi K., Velbel M., Vellenga J., Vicenzi E., Vincze L., Warren J., Weber I., Weisberg M., Westphal A. J., Wirick S., Wooden D., Wopenka B., Wozniakiewicz P. A., Wright I., Yabuta H., Yano H., Young E. D., Zare R. N., Zega T., Ziegler K., Zimmerman L., Zinner E., and Zolensky M. 2006. Comet Wild 2 under a microscope. *Science* 314:1711–1716.
- Dermawan B., Nakamura T., Fukushima H., Sato H., Yoshida F., and Sato Y. 2002. CCD photometry of the MUSES-C mission target: Asteroid (25143) 1998 SF36. *Publ. Astron. Soc. Japan* 54: 635-640.
- Ebihara M., Sekimoto S., Shirai N., Hamajima Y., Yamamoto M., Kumagai K., Oura Y., Ireland T. R., Kitajima F., Nagao K., Nakamura T., Naraoka H., Noguchi T., Okazaki R., Tsuchiyama A., Uesugi M., Yurimoto H., Zolensky M. E., Abe M., Fujimura A., Mukai T., and Yada T. 2011. Neutron activation analysis of a particle returned from asteroid Itokawa. *Science* 333:1119-1121.
- Fegley B. and Swindle T. D. 1993. Lunar volatiles: implications for lunar resource utilization. In *Resources of near-Earth space*, edited by Lewis J. S., Matthews M. S. and Guerrieri M. L., Univ. Arizona Press, p367-426.
- Fujimura A., Abe M., Yada T., Nakamura T., Noguchi T., Okazaki R., Ishibashi Y., Shirai K., Okada T., Yano H., Zolensky M. E., Sandford S., Ueno M., Mukai T., Yoshikawa M., and Kawaguchi J. 2011. Processes to open the container and the sample catcher of the Hayabusa returned capsule in the Planetary Material Sample Curation Facility of JAXA (abstract #1829). 42nd Lunar Planet. Science Conference. USB memory.
- Fujiwara A., Kawaguchi J., Yeomans D. K., Abe M., Mukai T., Okada T., Saito J., Yano H., Yoshikawa M., Scheeres D. J., Barnouin-Jha O., Cheng A. F., Demura H., Gaskell R. W., Hirata N., Ikeda H., Kominato T., Miyamoto H., Nakamura A. M., Nakamura R., Sasaki S., and Uesugi K. 2006. The rubble-pile asteroid Itokawa as observed by Hayabusa. *Science*, 312: 1330-1334.
- McMurtrey E. L. 1985. *Lubrication handbook for the space industry*. NASA Technical Memorandum – 86556. Huntsville: Marshall Space Flight Center, 458p.
- Nagao K., Okazaki R., Nakamura T., Miura Y. N., Osawa T., Bajo K., Matsuda S., Ebihara M., Ireland T. R., Kitajima F., Naraoka H., Noguchi T., Tsuchiyama A., Yurimoto H., Zolensky M. E., Uesugi M., Shirai K., Abe M., Yada T., Ishibashi Y., Fujimura A., Mukai T., Ueno M., Okada T., Yoshikawa M., and Kawaguchi J. 2011. Irradiation history of Itokawa regolith material deduced from noble gases in the Hayabusa samples. *Science* 333:1128-1131.

- Nakamura E., Makishima A., Moriguti T., Kobayashi K., Tanaka R., Kunihiro T., Tsujimori T., Sakaguchi C., Kitagawa H., Ota T., Yachi Y., Yada T., Abe M., Fujimura A., Ueno M., Mukai T., Yoshikawa M., and Kawaguchi J. 2012. Space environment of an asteroid preserved on micrograins returned by the Hayabusa spacecraft. *Proc. Natl. Acad. Sci.* 109:E624-E629.
- Nakamura T., Noguchi T., Tanaka M., Zolensky M. E., Kimura M., Tsuchiyama A., Nakato A., Ogami T., Ishida H., Uesugi M., Yada T., Shirai K., Fujimura A., Okazaki R., Sandford S. A., Ishibashi Y., Abe M., Okada T., Ueno M., Mukai T., Yoshikawa M., and Kawaguchi J. 2011. Itokawa dust particles: a direct link between S-type asteroids and ordinary chondrites. *Science* 333:1113-1116.
- Naraoka H., Mita H., Hamase K., Mita M., Yabuta H., Saito K., Fukushima K., Kitajima F., Sandford S. A., Nakamura T., Noguchi T., Okazaki R., Nagao K., Ebihara M., Yurimoto H., Tsuchiyama A., Abe M., Shirai K., Ueno M., Yada T., Ishibashi Y., Okada T., Fujimura A., Mukai T., Yoshikawa M., and Kawaguchi J. 2012. Preliminary organic compound analysis of microparticles returned from Asteroid 25143 Itokawa by the Hayabusa mission. *Geochemical J.* 46: 61-72.
- Noguchi T., Nakamura T., Kimura M., Zolensky M. E., Tanaka M., Hashimoto T., Konno M., Nakato A., Ogami T., Fujimura A., Abe M., Yada T., Mukai T., Ueno M., Okada T., Shirai K., Ishibashi Y., and Okazaki R. 2011. Incipient space weathering observed on the surface of Itokawa dust particles. *Science* 333:1121-1125.
- Oba Y. and Naraoka H. 2009. Elemental and isotope behavior of macromolecular organic matter from CM chondrites during hydrous pyrolysis. *Meteoritics Planet. Sci.* 44: 943-953.
- Okada T., Shirai K., Yamamoto Y., Arai T., Ogawa K., Hosono K., and Kato M. 2006. X-ray fluorescence spectrometry of asteroid Itokawa by Hayabusa. *Science* 312: 1338-1341.
- Okazaki R., Nagao K., Miura Y.N., Osawa T., Bajo K., Matsuda S., Nakamura T., Shirai K., Abe M., Yada T., Noguchi T., Ishibashi Y., Fujimura A., Mukai T., Ueno M., Okada T., Yoshikawa M., and Kawaguchi J. 2011. Noble gases recovered from the Hayabusa sample container. (abstract #1829). 42nd Lunar Planet. Science Conference. USB memory.
- Saito S., Sonoda M., Ochiai T., Han M., and Takahashi K. 2007. Micromanipulation of a conductive/dielectric particle by a single probe. *Proceedings of the 7th IEEE International Conference on Nanotechnology*, 733-736.
- Tendero C., Tixier C., Tristant P., Desmaison J., and Leprince P. Atmospheric pressure plasmas: A review. *Spectrochimica Acta Part B* 61: 2-30.
- Tsuchiyama A., Uesugi M., Matsushima T., Michikami T., Kadono T., Nakamura T., Uesugi K., Nakano T., Sandford S. A., Noguchi R., Matsumoto T., Matsuno J., Nagano T., Imai Y.,

- Takeuchi A., Suzuki Y., Ogami T., Katagiri J., Ebihara M., Ireland T. R., Kitajima F., Nagao K., Naraoka H., Noguchi T., Okazaki R., Yurimoto H., Zolensky M. E., Mukai T., Abe M., Yada T., Fujimura A., Yoshikawa M., and Kawaguchi J. 2011. Three-dimensional structure of Hayabusa samples: origin and evolution of Itokawa regolith. *Science* 333:1125-1128.
- Yano H., Kubota T., Miyamoto H., Okada T., Scheeres D., Takagi Y., Yoshida K., Abe M., Abe S., Barnouin-Jha O., Fujiwara A., Hasegawa S., Hashimoto T., Ishiguro M., Kato M., Kawaguchi J., Mukai T., Saito J., Sasaki S., and Yoshikawa M. 2006. Touchdown of the Hayabusa spacecraft at the Muses Sea on Itokawa. *Science* 312: 1350-1353.
- Yurimoto H., Abe K., Abe M., Ebihara M., Fujimura A., Hashiguchi M., Hashizume K., Ireland T. R., Itoh S., Katayama J., Kato C., Kawaguchi J., Kawasaki N., Kitajima F., Kobayashi S., Meike T., Mukai T., Nagao K., Nakamura T., Naraoka H., Noguchi T., Okazaki R., Park C., Sakamoto N., Seto Y., Takei M., Tsuchiyama A., Uesugi M., Wakaki S., Yada T., Yamamoto K., Yoshikawa M., and Zolensky M. E. 2011. Oxygen isotopic compositions of asteroidal materials returned from Itokawa by the Hayabusa mission. *Science* 333:1116-1119.
- Zolensky M. E., Nakamura-Messenger K., Fletcher L., and See T. 2008. Curation, spacecraft recovery, and preliminary examination for the Stardust mission: A perspective from the curatorial facility. *Meteoritics & Planetary Science* 43: 5-21.