EURO-CARES

A PLAN FOR EUROPEAN CURATION OF RETURNED EXTRATERRESTRIAL MATERIALS

WORK PACKAGE 4

INSTRUMENTS & METHODS

SPACE AGENCY VISITS

(DELIVERABLE D4.1)

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Report on Space Agency Visits

As part of EURO-CARES our team undertook several visits to space agencies with functional and current curation capacities, to learn from their experience and expertise The only two space agencies that have built recent curation facilities and are currently curating returned sample material are The National Aeronautics and Space Administration (NASA) and The Japan Aerospace Exploration Agency (JAXA). We were welcomed hospitably and given free access in order to view the curation facilities and instrumentation.

The outcomes from the visits are reflected in the deliverables especially for WP3 and WP4 (D4.2, D3.1).

Summary of Visits Undertaken

Location	Personnel	Host	Dates
NASA	Ludovic Ferrière	Michael Zolensky, Ryan Ziegler and Cecilia Satterwhite	March 2015
JAXA	Ludovic Ferrière and Aurore Huzler	Toru Yada and Masanao Abe	November 2015
NASA	Aurore Hutzler and Caroline Smith	Judith Allton, Ryan Ziegler, Nancy Todd, Anne Kascak, Michael Zolensky, Cindy Evans and Lisa Pace	March 2016
JAXA	Yves Marrochi, Mathieu Roskosz and Guillaume Avice	Yoshi Yurimoto	July 2016

Below we summarise some of the observations for each laboratory.

Hayabusa-returned sample curation facility, JAXA

Description

The Planetary Material Sample Curation Facility (PMSCF) at JAXA (Fig. 1) is located in Sagamihara (Kanagawa, Japan) and was completed in March 2008 for the purpose of the Hayabusa 1 space mission that collected samples at the surface of the asteroid Itokawa in 2005 and came back to Earth in 2010.

Design and architecture

The PMSCF was built from scratch, and the design integrates workspace outside of the cleanroom, a comprehensive plan of rooms for curation and storage and a public part for outreach activities. Some of the non-cleanroom parts were built to be easily retrofitted for the Hayabusa 2 sample return mission (currently in progress; i.e., the "Monitor and Meeting room" is equipped with a specially designed grated floor, as the "Sample Handling and Storage Room", see below).

The curation facility consists of a garment room, leading to four main cleanrooms with different levels of cleanliness. An office space ("Monitor and Meeting room") and a viewing room (used for public outreach) are also part of the facility (see Fig. 1). It is about 400 square meters in total.

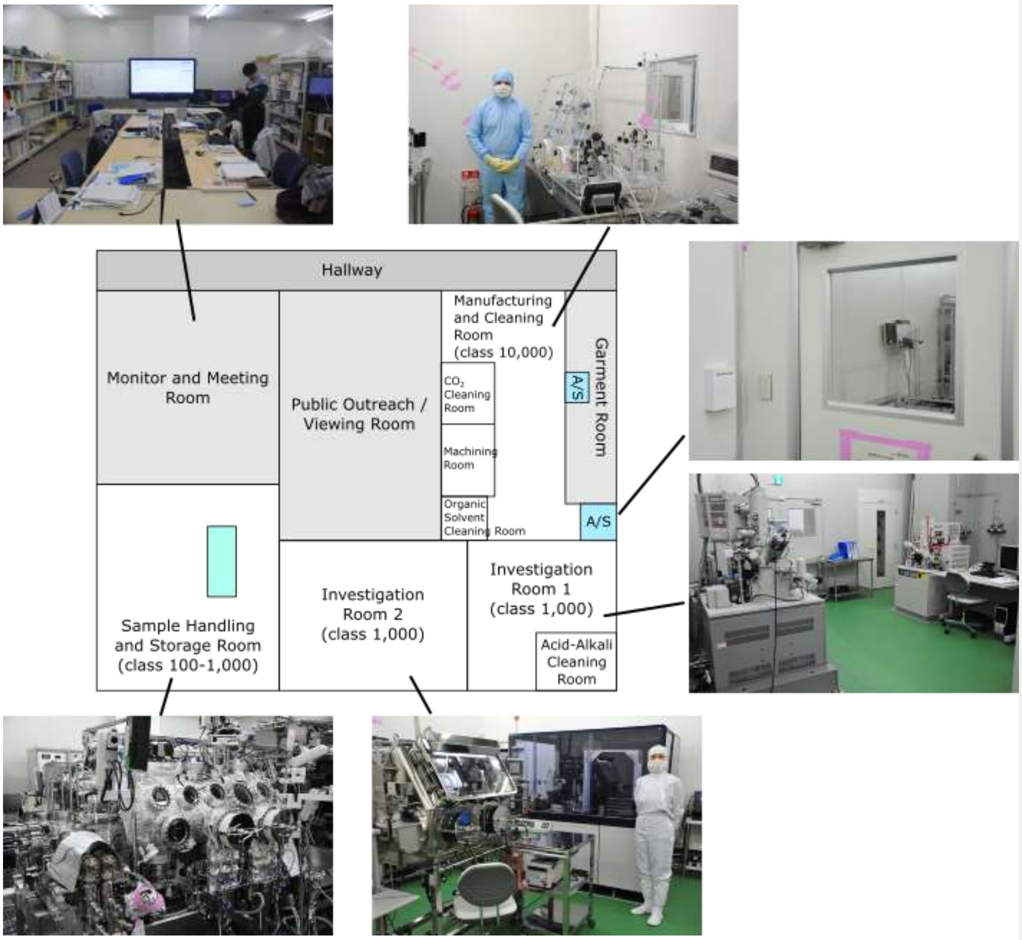

Fig. 1: A schematic viewgraph of the PMSCF/JAXA. Pictures showing (from top, clockwise): (i) the Monitor and Meeting room, (ii) Ludovic Ferrière inside of the Manufacturing and Cleaning Room, wearing a class 10,000 gown, (iii) the air shower between the Garment room and the cleaner part of the complex, (iv) the Investigation room 1, (v) Aurore Hutzler in the Investigation room 2, wearing a class 1,000 gown, and (vi) the clean chambers (used for handling and storage of the samples).

Equipment and instrumentation

Samples are stored and handled in the "Sample Handling and Storage Room", within two clean chambers mainly made of stainless steel 304 (Fig. 2.) These clean chambers are equipped with vacuum systems, pure nitrogen supply systems, optical microscopes, cleaning tools and containers, electrostatically controlled micromanipulation systems, etc. It is also equipped for photographing and weighing samples. Both chambers are kept under positive N_2 pressure relative to the atmosphere with chamber #1 being equipped with a system providing purified evaporated liquid nitrogen. This system allows the contamination to be minimized during the transition from ultra-high vacuum to atmospheric pressure of pure nitrogen. Clean chambers were a bespoke design from the Hitachi company (this company has a deep knowledge of state-of-the-art technologies after years of making clean rooms for manufacturing semiconductors; for special equipment like this one, it is of the utmost importance to find suppliers ready to go through a close collaboration with the curation team). A safe, some storage cabinets (under N_2 atmosphere) and some electronic control panel boards can also be found in this cleanroom. This room is equipped with a specially designed grated floor allowing laminar flow (i.e. air is exhausted via the grated floor).

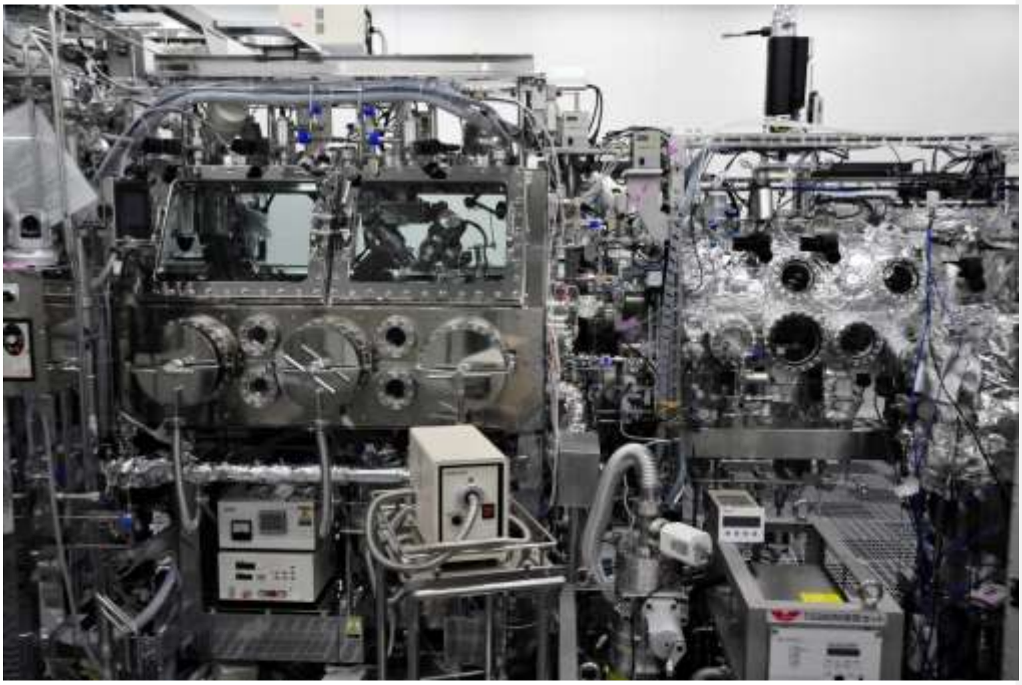

Fig. 2: Specially designed clean chambers used for handling samples and also for storage.

The "Investigation Room 1" is equipped with two Scanning electron microscopes (SEM), including one with the Focused ion beam (FIB) preparation technique. An acid-alkali cleaning room (i.e., a small room located in one corner of this room, see Fig. 1) is used for cleaning different small devices.

The "Investigation Room 2" is equipped with an X-ray diffractometer (XRD), a Fourier transform infrared (FTIR) spectrometer, a microRaman spectrometer, an SEM, and a number of (binocular) microscopes and other smaller devices within gloveboxes.

The "Manufacturing and Cleaning Room" is equipped with a number of (binocular) microscopes and other smaller devices (such as micro-manipulators and others devices allowing polished sections to be prepared, etc.) within gloveboxes as well as some storage cabinets (under N_2 atmosphere). Three additional "small rooms" (i.e., with independent air exhausting systems) are located within this room (see Fig. 1) and equipped with different specific instruments and devices allowing different types of cleaning to be conducted and even a large milling machine for machining specific workpieces of particular sizes and shapes.

Cleanliness

In the garment room, workers put on cleanroom outfits, to cover everything but the eyes. Make-up and jewellery are forbidden. Cleanroom outfits are different for the class 10,000 cleanroom and for the class 100-1,000 cleanroom (see Fig. 1). Workers go through a turbulent air shower to go from the garment room to the cleanrooms.

As previously mentioned, in the case of the "Sample Handling and Storage Room", the air is exhausted via the grated floor. For the other rooms, a standard anti-static flooring is used.

Bringing equipment such as cameras, laptop, or notebooks are not allowed inside of the clean rooms. Instead, such material is already available inside of the clean area. Data are loaded onto a data-sharing server, using computers inside of the cleanroom.

Operations

The daily routine of the PMSCF requires the systematic monitoring of the vacuum conditions. Its software allows the vacuum conditions to be continuously measured and corrected, if necessary. The curation facility is located in the JAXA building complex, close to the control room of the spacecraft Hayabusa 2, thus, allowing direct interventions 24 hours a day, seven days a week, 365 days a year.

Operating the clean chambers requires a lot of practice. Specific people in JAXA were selected and trained for this difficult job. The sample transfer from the clean chamber to the sample holder is performed by two people following a very detailed protocol. The samples located on quartz glass disk are first observed and photographed by two optical microscopes set in the system. Then, the samples are picked up using a micromanipulator system that is composed of a specific glass needle with an internal platinum wire on which a voltage is applied. This allows samples to be grabbed using static electricity and to transfer them to the sample holder for characterization.

Comment [LF1]: Redudant with the two first sentences of this paragraph

For further details, the readers should refer to the following publications:

Abe, M., Yada, T., Uesugi, M., Karouji, Y., Nakato, A., Kumagai, K., and Okada, T. 2015. Current status of JAXA's Extraterrestrial Sample Curation Center (abstract #1245). 46th Lunar and Planetary Science Conference. CD-ROM.

Yada, T., Fujimura, A., Abe, M., Nakamura, T., Noguchi, T., Okazaki, R., Okada, T., Ishibashi, Y., Shirai, K., Zolensky, M. E., Sandford, S., Uesugi, M., Karouji, Y., Ueno, M., Mukai, T., Yoshikawa, M., and Kawaguchi, J. 2011. Hayabusa sample curation at planetary material sample curation facility in JAXA (abstract #5386). 74th Annual Meeting of the Meteoritical Society.

Yada, T., Fujimura, A., Abe, M., Nakamura, T., Noguchi, T., Okazaki, R., Nagao, K., Ishibashi, Y., Shirai, K., Zolensky, M. E., Sandford, S., Okada, T., Uesugi, M., Karouji, Y., Ogawa, M., Yakame, S., Ueno, M., Mukai, T., Yoshikawa, M., and Kawaguchi, J. 2014. Hayabusa-returned sample curation in the Planetary Material Sample Curation Facility of JAXA. Meteoritics and Planetary Science 49(2):135–153, doi: 10.1111/maps.12027.

Lunar Sample Laboratory Facility, NASA

Description

We were given full tours of the Lunar Sample Laboratory Facility (LSLF), a repository and laboratory facility located at NASA's Lyndon B. Johnson Space Center (JSC) in Houston (Texas, USA). This laboratory was obviously designed, and not adapted to an empty space. It is the largest suite of cleanrooms and ancillary laboratories of the JSC. The laboratory was built in 1979, to host a total of 382 kg of lunar rocks, cores and soil brought back by the Apollo missions.

Design and architecture

The complex is composed of a large curation room ("Pristine Lab"; Fig. 3), a large vault (both ISO 6), a laboratory for invited researchers working on disseminated samples, and a smaller vault for returned samples, hosting part of the Genesis samples as well (these parts are ISO 7).

The complex includes a network of corridors and pass boxes to have several levels of cleanliness, and to avoid moving samples by hand, and changing rooms for workers to move progressively to the more pristine areas.

Fig. 3: Overview of the large curation room of the Lunar Sample Laboratory with several of the glove boxes.

Cleanliness

Workers go through a changing room to put cleanroom gowns on. The face is not covered (Fig. 4).

Fig. 4: A. Hutzler storing back a sample box in the Lunar Vault. Note the full coverage except for the face.

The Ultra-Pure Water system is monitored daily. Particles counts are performed each week in laboratories. Each batch of delivered N_2 is checked for cleanliness and isotopic composition. The content of O_2 and H_2O in the lunar glove boxes is measured four times per hour, to allow a prompt reaction in case of an incident.

Equipment and instrumentation

Storage and curation are undertaken in the same type of stainless steel glove boxes, under a dry N_2 atmosphere flow.

Inside the curation glove boxes, a small selection of instruments and tools are available, including specially modified scales, tools allowing manipulation and splitting of the samples (including hammers!), a bag heat sealer, etc. In case an instrument or consumables should be added inside, this can be done using a specific air lock chamber. Lighting and binocular microscopes are placed outside of the glove boxes and can be used through windows. On new glove boxes, a video camera system was added on top (outside the glove box; Fig. 5).

No specific investigation instruments are available within the laboratory.

Fig. 5: A typical stainless steel glove box from the Lunar Laboratory (note the camera on top).

Operations

Samples are mostly handled to prepare sub-samples for dissemination, according to the requests of external laboratories.

To take a sample in or out of storage, the curator and another person needs to be there, to assess that the sample was taken or returned to the right place. The samples are stored with an aluminium tag with a unique number (no bar code system is used).

Each sample is weighed before and after anything happens to it. When sub-sampling is done, the total mass needs to be the same. If not, particles are hunted down.

All disposable material in contact with lunar samples (such as Teflon bags) is kept in a special bin, and inspected for loose particles before being destroyed. Laboratory access is restricted, and protected with a code. The storage rooms (vaults) are protected by a huge steel vault door with a code requesting two people (the entry code is split between different people).

Genesis Processing and Sample Storage Facilities, NASA

Description

The Genesis Laboratory is a retrofitting of a part of the Lunar building (former visitor part). The laboratory was built before the launch of the mission, to assemble the Genesis container under clean and monitored conditions, in 1999.

Design and architecture

Although a retrofitted space, the Genesis Curation Laboratory is large enough to show an organized design with successive rooms of increasing cleanliness (Fig 6).

A non-clean hallway ("Receiving") gives access to the ante-room (ISO 7). Items stored in the ante-room include: consumables and public outreach samples (in left over lunar cases), either on shelves or in a laminar air-flow cabinet.

Workers access then a garment room (ISO 7), where staff and visitors are required to change into an ISO 4 gown. The HEPA filtered ISO 4 part is composed of a corridor (ISO 6), a cleaning room and a characterization/storage room (initially used to assemble the Genesis container). A pass-box was built between cleaning and characterisation rooms, but it disturbs the airflow too much. Samples are hence transported by hand through the corridor.

In the corridor, a cabinet on wheels can be used to extract samples in case of an emergency (for example, hurricanes). The emergency door opens directly to a corridor, with HEPA filters and plastic curtains activated in case of a transfer (Fig. 6). This transfer procedure is rehearsed at least once a year.

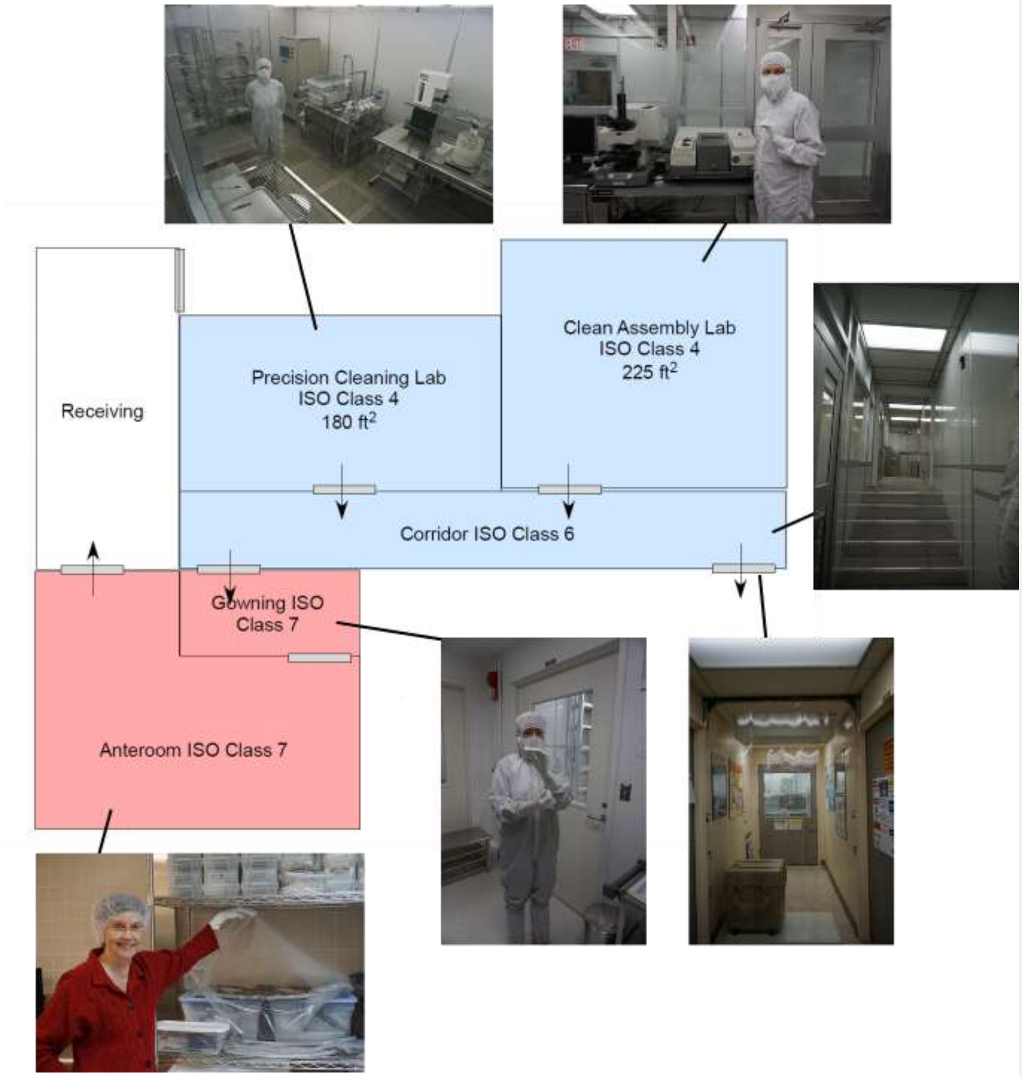

Fig. 6: Schematic viewgraph of the clean rooms of the Genesis Facility. Pictures showing (from top, clockwise): (i) Aurore Hutzler in the Precision Cleaning Lab, (ii) Judith Allton in the Clean Assembly Lab, in front of a FT-IR, (iii) the corridor in the cleaner part of the lab, (iv) the emergency exit from outside, (v) Aurore Hutzler in the garment room, adjusting an ISO 4 gown, and (vi) Judith Allton in the Anteroom, wearing only minimal protection.

<u>Cleanliness</u>

In the hallway, before entering the facility, workers are equipped with basic protections (hair, feet and gloves). Items that have to be brought inside the laboratory (such as a camera) are wiped clean with Isopropyl alcohol (IPA)/water wipes before entering the laboratory.

The floor of the ISO 4 part is nickel-coated, raised by 40 cm above the ground, and perforated to allow a laminar air flow from ceiling to floor. The Ni contamination was not seen as an issue at the time of building it (as organic contamination was of higher importance). The building itself is completely air-tight, which causes water to condense onto the floor. There are fire incipient detectors on the lower floor.

Air is brought through HEPA filters on the ceiling. The temperature is higher in the ISO 4 part, because of the friction of the air through the filters. There is no system to modify the temperature of the laboratory. The 18.2MΩ water system is the same for the whole building, with continuous flow. Dry N_2 gas for storage cabinets is delivered to JSC, and monitored for cleanliness. Before entering the cabinets, it goes through a final purifying system.

The laboratory is cleaned every week with IPA wipes, and particle counts are performed every week. Each person entering the room writes his/her name, purpose, and hours of visits, on a sheet of paper outside of the room. Jewellery is not allowed inside. A full cleanliness protocol is undertaken prior to entering the lab, and each worker and visitor has to sign an agreement beforehand.

Visitors are rarely accepted inside of the laboratory, and public outreach is limited.

Equipment and instrumentation

In the ante-room the is a UV Ozone Cleaner (to clean Genesis wafer, but not allowed in ISO 4 part because of high particle shedding).

In the Precision Cleaning lab is housed the following equipment:

- Ultrapure Water (UPW) System: E-1 Grade or better (ASTM D5127-13) & UPW Heater
- 72 kHz Ultrasonic UPW Cascade Tank and Baths
- 1 MHz Megasonic Pulse UPW Cleaning

In the Clean Assembly lab are:

- Gaseous Nitrogen System: Boil-off grade C LN2 (MIL-PRF-27401G) & GN2 Heater
- GN2 storage desiccators
- Liquid Particle Counters (> 1 µm)
- Optical Automated Scanning Microscopes
- Stereomicroscopes
- FT-IR with Continuum Microscope
- Clean tools for handling and assembly

Operations

Access is controlled by badges.

The main function of this laboratory is to characterise and catalogue the Genesis wafers and to allow dissemination to research institutions. A limited number of instruments (e.g. FT-IR microscope to sort between two wafer manufacturers and imaging tools) are used – all are inside of the laboratory.

Wafers and tools are cleaned using UPW in ultrasonic and megasonic baths. The water temperature is controlled, to limit corrosion of materials. Beakers are made of glass, since boron is not a contamination issue. Some instruments are not laboratory designed.

Stardust Laboratory, NASA

Description

The mission Stardust aimed at collecting particles from the coma of comet Wild 2, by trapping them into aerogel. The samples were returned in 2006.

Design and architecture

The Stardust laboratory is in a retrofitted room of the Lunar Laboratory. A large glass window allows a full view of the curation room from the anteroom (Fig. 7).

Fig. 7: View of the Stardust curation room from the anteroom through the glass window.

It is composed of an anteroom, a changing room and the curation and storage room (Fig. 8).

Fig. 8: View from the inside of the Stardust curation lab (ISO 5). On the left, the door to the garment room, on the right, the glass window overlooking the anteroom.

Cleanliness

Workers are equipped with a full cleanroom gown (Fig. 9). There is no air-shower before entering the curation laboratory. The anteroom is a class 10,000 area served by one air handler with six HEPA fan filter units. The curation laboratory is a class 100 cleanroom, with conditioned air from the outer space with 36 fan filter units mounted on the ceiling (each of the fans can be controlled remotely from the control panel located in the change room).

Samples (contained in aerogel inside precision-cleaned quartz-glass containers) and the aerogel sample dust collector tray are stored inside stainless steel cabinets (with glass windows in front) flooded with N_2 gas.

Due to the fact that the samples are embedded in silica aerogel, water should be kept far away and humidity levels well controlled. For this reason, sprinklers were placed in the outer room (above the modular cleanroom) and a sensitive air-sampling fire-detection system was installed within the curation laboratory.

Fig. 9: Ludovic Ferrière inside the Stardust curation room, behind the aerogel dust sample collector tray.

Equipment and instrumentation

Considering that only an extensive photo-documentation and extraction of the cometary samples was conducted in the stardust laboratory (i.e., no characterization), the amount of equipment and instrumentation is very limited (i.e., different types of microscopes and video cameras as well as micromanipulators). Two different methods were designed to extract small volume of aerogel, i.e., the "keystone system" [robotically controlled cutting action consisting of repeated small axial poking motions of the aerogel by two glass microneedles that are mounted on micromanipulators; Fig. 10] and the "quickstone system" [consists of applying ultrasonic frequency oscillations to microblades (either diamond or steel blade) via a piezo-driven holder mounted on a micromanipulator] (more details for both methods can be found in Zolensky et al. 2008).

To be noted is that the room is equipped with an anti-static floor and that samples are processed on vibration isolation tables. In addition, a small handheld radioactive ^{210}Po source is used as local anti-static device.

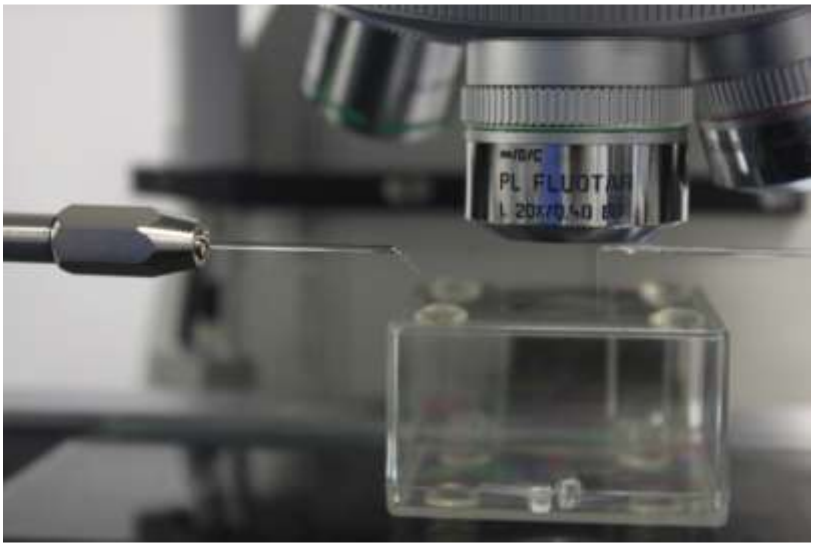

Fig. 10: Thin glass microneedles mounted on micromanipulators and used under an optical microscope to manipulate Stardust samples.

Cosmic Dust Laboratory, NASA

Description

This laboratory hosts a collection of cosmic dust collected from 1981 to the present day. It is designed to locate, prepare, characterize, store and distribute collected particles.

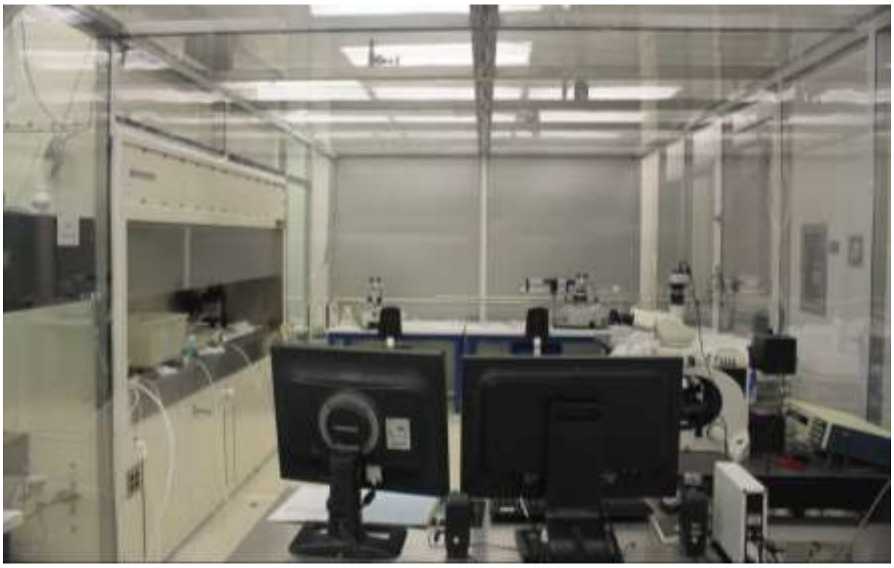

Fig. 11: Overview of the Cosmic Dust curation and preliminary examination room. It is equipped with optical and binocular microscopes; note on the left side the fume hood with ultrapure water system.

Design and architecture

After changing in a garment room, workers go through a laminar air shower (for a few minutes) to lessen the particulate contamination. The laboratory is then composed of two rooms, one for storage of instruments and spare parts, the other for curation and preliminary examination of cosmic dust (Fig. 11). This last room is partly ISO 4, with a special air sweep system.

Cleanliness

For the less clean part of the laboratory, a minimal coverage is required. For the ISO 4 part, a full adapted gown is used.

Equipment and instrumentation

Considering that no detailed characterization is conducted inside the laboratory, it is only equipped with a number of different types of optical and binocular microscopes (some equipped with video cameras) and micromanipulators (Fig. 12). To be noted is that most collected grains remain on their collection surface and are curated (/stored) as they are (i.e. to minimize contamination). The room is also equipped with a ultrapure water system (placed in a fume hood; see Fig. 11).

Fig. 12: Micromanipulator under a binocular microscope in the Cosmic Dust Laboratory.

Hayabusa Curation Laboratory, NASA

Description

The JAXA gave part of the Hayabusa particles (approximately 10%) to NASA. Particles are subject to the loan procedure of JSC.

Design and architecture

The Hayabusa facility is a retrofitted lab, consisting of an anteroom, a changing room and a curation room. It is the smallest of the JSC curation laboratories.

Air and water supply

Ultra-pure water system (Fi. 13): we were advised this should ideally be better on the same floor as the labs, to avoid pumping. If the water supply is located above, there is a risk of flood. The Air Handling for the Lunar Laboratory (Fig. 14), with HEPA filters (changed every 10 years), is located in the same room.

Fig. 13: Ultra-Pure water system. Yellow part is a dam to prevent flood, associated with a flood warning system (orange line).

Fig.14: HEPA filtered air system for the Lunar Laboratory.

Dissemination

To keep track of shipping samples, each has an identification number on an aluminium tag (Fig. 15). If a PI wants a samples, he/she needs to ask the curator who makes the decision or refers the request to the relevant review panel. The curator or technician prepares samples. They first fill out a form, to ask for authorization to disseminate the sample. The transfer is authorized and the status transfer changes. The package is prepared. If the sample is to be taken outside of the US, it needs customs/exports. When the PI receives the sample, he/she must send back the loan form, to acknowledge the reception of the sample, and that it is now under his/her custody.

There are rules governing the size of the package: no more than 10 g of lunar samples in each package, and each package should be on a separate crate with Fedex.

Some thin sections are kept outside the curation rooms, so they can be sent directly to PIs, since they do not required extra clean conditions of storage (mostly meteorites). Each collection has slightly different paperwork requirements.

There is a data pack for everything that happens to a sample in NASA (for example cutting and thin sectioning), with forms and photographs. There is a data pack for everything that happens to a sample outside of NASA, with PI names. A folder is kept for each PI, with each allocation. Dead or retired PI's folders are kept in a separate place.

Fig. 15: Packaged samples (meteorites) with metal tags.

For further details, the readers should refer to the following publications:

Allen, C., Allton, J., Lofgren, G., Righter, K., and Zolensky, M. 2011. Curating NASA's extraterrestrial samples—Past, present, and future. Chemie der Erde 71:1–20.

Allen, C., Allton, J., Lofgren, G., Righter, K., Zeigler, R., and Zolensky, M. 2013. Curating NASA's extraterrestrial samples. Eos 94(29):253–254 [16/07/2013].

Zolensky, M., 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 and Planetary Science 43(1/2):5–21.