ISSN 2433-2216(Online)
JAXA-RR-21-009E

JAXA Research and Development Report

Development of electrostatically controlled micro-manipulation system in purified ultra-clean nitrogen environment and its performances to handle micrometer-sized asteroidal regolith particles

YADA Toru, FUJIMURA Akio, ABE Masanao, NOGUCHI Takaaki
NAKAMURA Tomoki, OKAZAKI Ryuji, OGAWA Maho, OKADA Tatsuaki
ISHIBASHI Yukihiro, SHIRAI Kei, KAROUJI Yuzuru, UESUGI Masayuki
NAKATO Aiko, KUMAGAI Kazuya, NISHIMURA Masahiro, MATSUI Shigeo
SAKAMOTO Kanako, YOSHITAKE Miwa, YOGATA Kasumi and IWAMAE Ayako

February 2022

Japan Aerospace Exploration Agency

Contents

Development of electrostatically controlled micro-manipulation system in purified ultra-clean nitrogen environment and its performances to handle micrometer-sized asteroidal regolith particles

YADA Toru[*1], FUJIMURA Akio[*1], ABE Masanao[*1], NOGUCHI Takaaki[*2], NAKAMURA Tomoki[*3], OKAZAKI Ryuji[*4], OGAWA Maho[*1,4], OKADA Tatsuaki[*1], ISHIBASHI Yukihiro[*1], SHIRAI Kei[*1], KAROUJI Yuzuru[*1], UESUGI Masayuki[*5], NAKATO Aiko[*1], KUMAGAI Kazuya[*6] NISHIMURA Masahiro[*1], MATSUI Shigeo[*1], SAKAMOTO Kanako[*1], YOSHITAKE Miwa[*1] YOGATA Kasumi[*1] and IWAMAE Ayako[*6]

ABSTRACT

Abstract: Electrostatically controlled micromanipulation system was developed for handling μm-size particles returned by Hayabusa spacecraft and installed in the Clean Chamber 2 of purified nitrogen condition. The system consists of one sample stage, two probe stages equipped with quartz glass probes including electrodes, and three microscopes observing the top of the sample stage from three different angles. Because materials allowed to installed in the Chamber is limited to constrain materials of possible contaminants, we have also developed a procedure for making quartz glass needles including Pt wire. After the rehearsal using simulant particles, we applied this system for the actual Hayabusa-returned particles, and the system works fine for handling more than one thousand of Hayabusa-returned particles of 10-320 μm in size in this decade.

Keywords: curation, return sample, micromanipulation, electrostatically controlled, Itokawa, Hayabusa

1. Introduction

It had been estimated that meteorites found on the Earth should have originated from asteroid, however, it had not been proved until samples would have been returned from the asteroid. Hayabusa spacecraft, which had launched on 12 June 2003, reached the target body, near-Earth S-type asteroid Itokawa (25143), on 20 June 2005, held remote-sensing analyses and touchdown sampling twice and left the body in March 2006 [1, 2]. Then it had returned its re-entry capsule back to the Earth on 13 June 2010 [3]. Since the sampling operation of the Hayabusa had not been successful as it could not have shot a tantalum projectile to blow up asteroid regolith and let them lift up and move into its sample catcher during the touchdown process on Itokawa [2], it was estimated that the weight and size of recovered samples should be small, such as less than 1mg in total and less than 100μm, respectively. Therefore, we had to develop a tool to handle small particles returned by Hayabusa.

---

[*] Received November 30, 2021
[*1] Institute of Space and Astronautical Science, Japan Aerospace Exploration Agency
[*2] Kyoto University
[*3] Tohoku University
[*4] Kyushu University
[*5] Japan Synchrotron Radiation Research Institute
[*6] Marine Works Japan Ltd.

There are several constraints in developing a tool for handling small particles from Itokawa. They are as follows;

(1) less loss rate of sample particles,
(2) non- or less-destructive to particles,
(3) less skill-dependent for operators,
(4) less inorganic and organic contamination to samples,
(5) prevent samples from exposing to terrestrial atmosphere,

Here should be noted that there are several species of materials used for the sampler of Hayabusa; aluminum, aluminum alloy A6061, Teflon, stainless steel 304, Viton, and sapphire glass (Al_2O_3), which could be contaminated to the samples during the sampling process. Moreover, a clean chamber filled with purified nitrogen had been developed for handling returned samples in order to prevent them exposing to terrestrial atmosphere [4].

A method to grab a particle directly, such as manual handling with tweezers, must be unacceptable in (1), (2) and (3). In these points of view, one of the favorable method is electrostatically controlled micromanipulation. As we control voltage for electrode to make an electrostatic force larger than a gravitational force, we can lift up particles from the substrate. In this method, we can handle them avoiding the problems in (1), (2) and (3). In utilizing the electrostatic force, there are several kinds of commercial manipulators, however, (4) is a problem for them, such as a hydraulic drive, a stepping motor with grease, and a piezo motor, which use materials unacceptable for handling the Hayabusa-returned samples.

In order to clear the problem of (4) and (5), an electrostatically controlled micro-manipulation system specially designed for Hayabusa-returned samples is necessary to be developed.

2. Development of Electrostatically Controlled Micromanipulation System

For the first step of developing electrostatically-controlled micro-manipulation, we had checked the behavior of particles of several combination of materials in normal atmospheric condition. Table 1 shows behaviors of borosilicate glass, Teflon, and polystyrene particles of 100μm in size on an aluminum foil handpicked by positively- and negatively-charged borosilicate glass pipette with a cupper electrode inside. As shown in the table, the glass particle which tends to have positive electric charge are easily controlled by the needle of negative electrostatic voltage. The Teflon and polystyrene particles that tend to have negative electric charge, are easily controlled by the needle of positively electrostatic voltage, although charged voltage for the former is slightly smaller than that for the latter. The electrical character of polystyrene stands between that of glass and Teflon, somewhat close to the latter. These results are partially consistent with expected character from material electrification, however Teflon is described as the most negatively charged material whereas the polystyrene tends to be charged moderately negative [5]. This might be explained that surface condition of the conductive material attached on the insulators should affect their charging condition unexpectedly. We had also changed the probe to a tungsten needle, a borosilicate glass needle and a borosilicate pipette needle to check the usability of these probes. Among them, the most appropriate one is a borosilicate pipette with cupper electrode. In conclusion, the most suitable operation voltage of borosilicate pipettes with cupper electrode is -0.5V for glass sample, +0.5V for Teflon particle, and +0.7V for polystyrene sphere. The aluminum dish of electrically grounded is also suitable for the sample stage, because conductive materials should constrain an electrostatic charge on the dish.

As shown in the experiments in normal atmosphere, a borosilicate pipette with cupper electrode inside is the most usable in handling particles with electrostatically controlled condition. However, B, Na and Al contained in borosilicate glass probe might cause contamination to samples because the probe will directly contact with them. Thus, we should avoid to use borosilicate glass and select more suitable material for probe in order to minimize contamination to samples. One of the option is a synthetic quartz glass containing less than 1ppb in every element other than Si. There also exists a puller to make a quartz glass pipette, P-2000 by the Sutter Instrument (Fig. 1a). However, we could not make a quartz glass with electrode inside with the instrument of original configuration. Thus we added a few mechanical arrangements on it. First of all, we had put a platinum wire of 0.03mm in diameter inside a quartz glass tube of 1mm in diameter to be evacuated from its both ends. In this condition, the quartz glass with Pt wire was heated to its softening point to be yielded in the center of the tube and let the Pt wire embedded in the quartz glass (Fig. 1b). Then the tube was heated to its melting point to be pulled from its both sides to end up in making a pair of quartz glass needles with Pt wire inside (Fig. 1c). They were checked by an optical microscope to confirm whether there was any disconnection in the Pt wire inside the needles or not, then the acceptable one experienced additional processes such as gold wire connection to the Pt wire and cut of the end of tubes to align them in appropriate lengths. The angle of quartz glass needle to the stage surface is important for decrease handling difficulty. The shallower the needle angle from the dish surface is, the easier pick up and release of the particle is. However, the munimum angle for the needle should be 45 degree due to the constraint from the angle of dimples of a sample holder of an electron microscope where a particle will be placed by the manipulator. Practically, the angle of the needle is used to be set to 50 degree from the dish surface for the better approach of the needle to the bottom of the dimples of the holder.

As mentioned before, any commercial product for probe and sample moving stages are not appropriate for handling the returned samples due to the material constrains for handling the returned samples. Thus we had developed them using Al alloy 6061, stainless steel 304 and Teflon as main constituent materials and also use joint terminals made of gold and cupper conductive wires coated by polyimide film inside the clean chamber. Fig. 2 shows a schematic viewgraph of a sample moving stage and left and right probe moving stages from the front side. Because a particle sticking to the top of a glass probe could easily fall down from the probe due to vibration in moving the probe-moving stage, we should not move the stage in handling particle but move the sample-moving stage to lift up, transfer and release a sample particle. The sample stage could move ±50mm in X axis, ±100mm in Y axis, ±50mm in Z axis and 360˚ in theta axis. Besides, each X, Y and Z axis of the stage has equipped a micro-motion controlling dial which could allow us to move stage in micrometer scale. The right and left probe-moving stages also could be moved in X, Y, Z, θ XY and θ YZ axes, which could equip probe holders on their upper parts. The stages are mainly used for adjusting a position of the top of the glass probe to the center of sights of microscopes for the manipulator. The quartz glass probe with Pt wire inside is set the end of the probe holder and the gold wire connected with the Pt wire inside the probe is connected to the cupper cable coated by polyimide film involved in the probe holder to be connected to the DC power supply apparatus outside the chamber through the feedthrough flange of the chamber. The top plate of the sample moving stage could be also connected to the DC power supply apparatus so that we could also control charged voltage of each of the sample moving stage and probe moving stages, which can be operated manually via Viton gloves set to the clean chamber. All the parts of the stages are grease-free.

The micro-manipulation system includes three microscopes equipped outside and inside the clean chamber (Fig. 3). The first one is set outside the clean chamber, on the top of the chamber to observe the top of the sample-moving stage. This microscope is revised version of UWZ-500 made by Union corporation, which equips with electronic zooming and focusing system and could observe object in magnification from x0.23 to x2.8 in 500mm of working distance, 62μm to 10μm in spatial resolution. Because the viewport where the microscope is set above is offset from the center of the sample stage, a series of mirror units is equipped inside the chamber for the microscope to observe the center of the sample-moving stage through the viewport of the chamber. The second and third ones are set inside the clean chamber from the back side of the left and right probe-moving stages. The second one is based on Nikon stereomicroscope objective lens P-Plan Apo 1.5x and a CCD camera and the third one is based on Nikon microscope CM-20L with an objective lens of CFI LU Plan Flour EPI 5x and a CCD camera. Each of them is fixed focus and magnification and 1μm and 2μm in spatial resolution, respectively. They are combined and sealed inside container made of the aluminum alloy 6061, a quartz glass window and a Viton O-ring. The image signals from the CCD cameras attached to the microscopes inside the chamber are transmitted by USB cables inside and outside the chamber to be connected to the PC which can control the CCD cameras and monitor the images of the microscopes through the feedthrough flange of the chamber. The USB cables inside the chamber are products for vacuum, composed of cupper cables coated by polyimide film. The LCD monitors of the PCs for three microscopes are located beside the chamber and the operator of the manipulator could observe them during handling the manipulator (Fig 4(a)). With these three microscopes observing a top of the glass probe from three different angles, the operator of the manipulator can recognize its position three-dimensionally and also observe the target particles in detail from three different angles (Fig. 4(b)-(d)).

3. Results - Rehearsal and Performances

The micro-manipulation system had been established at the end of January 2010. We had held manipulation tests on simulant particles since then until the asteroidal sample returned to the Earth in June 2010. In order to avoid the contamination of the simulant particles to the returned samples, synthetic olivine grains including NiO had been used for the test because they could be easily distinguished from the real Itokawa grains with their weird chemical compositions. The simulant particle of 100μm in size in a petri dish made of quartz glass could have been lifted up in the condition of +/- 100V in probe voltage and 0V in stage voltage. It could have been released on the glass surface in the condition of +/- 50V in probe voltage and +/-30V in stage voltage. It was also possible to lift up the particle from the surface of the aluminum alloy A6061 and release it on the material. In this way, the electrostatically-controlled micromanipulator system was ready for the performance of Hayabusa-returned samples.

After the sample container including samples returned from S-type asteroid Itokawa was carried into cleanrooms of JAXA, it was finally introduced into the Clean Chamber (CC) #1 and opened in static low vacuum condition [4]. After the opening of the sample container, gaseous sample released from the container was separated into gas cylinders and then the chamber was evacuated to high vacuum and purged with purified nitrogen. Then a sample catcher was extracted to be transferred into the next Chamber, CC2. The sample catcher is a cylinder made of aluminum alloy 6061 and sizes 80mm in height and 48mm in diameter [4]. The catcher is divided into three chambers, chamber A, chamber B and rotational cylinder. During the first touchdown onto Itokawa by the Hayabusa spacecraft, regolith particles should have been recovered in the chamber B and those by the second touchdown should have been recovered in the chamber A.

The samples in the chamber of the sample catcher had been recovered on the quartz and aluminum disks by tapping. Then the particles on the disk have been picked up with the electrostatically-controlled micromanipulator system one by one and transferred to the sample holder for the scanning electron microscope (SEM). After the initial description with the SEM, the particles have been moved to gridded quartz glass slides for the sample preservation. Particles returned by Hayabusa are grouped into four categories [4]. Category 1 and 2 are composed of olivine, pyroxene and/or plagioclase as major phases with accessary phases of Fe sulfide, Fe-Ni metal, chromite and/or Ca phosphate, which originated from asteroid Itokawa. Some of them are monomineralic, and others are polymineralic. Their sizes range from less than 10μm up to more than 300μm. Category 3 particles consist mainly of carbon, oxygen and nitrogen, which should be some kind of organics. They are so far identified as terrestrial origin [6]. Category 4 particles are fragment of terrestrial artificial materials like aluminum, quartz glass, stainless steel, sapphire glass and metallic particles like iron, zinc, titanium, and etc.

The electrostatically-controlled micromanipulation system has been utilized for removing particles from the quartz glass disks into SEM holders and transferring them from the holder to gridded glass slides after the SEM observation, as shown in Fig. 4(b)-(d). In the most cases, the system worked well for the particles transfer. Fig. 5 shows viewgraphs that actual charged voltage on the probe for lifting-up Itokawa particles from substrates made of conductive and non-conductive materials. Note that particles lifted from each substrate are identical, although either values in either substrates are missed in some cases. There is a tendency that larger particles need larger absolute voltage than smaller particles' cases. As compared data for substrates made of conductive and non-conductive materials, it is also implied that absolute voltages for particles lifted up from non-conductive substrate, like quartz glass slides as shown Fig. 5(a), are larger than those for particles lifted up from conductive substrates, like aluminum and gold as shown Fig. 5(b). Thus it can be noted that conductive substrates like metal are favorable for electrostatically-controlled micromanipulation system.

4. Concluding Remarks

Because we have to avoid contamination and loss in handling for such small sized samples, we developed an electrostatically-controlled micromanipulator system installed into the clean chamber for Hayabusa-returned particles. In this decade, the system works so fine that more than one thousand of particles in 10~320 μm of major axes have been successfully handled with the system. This system was applicable for Hayabusa-returned samples, and can be applied for future sample return mission in the case samples obtained by the mission is limited to small amount, or small in size.

However, the electrostatic condition of the particles are very unstable in some cases, we spent more than a week to remove some particles from the glass substrates. Possible cause of this difficulty should be material or shape of the particles, or any unknown parameter related to the electrostatic condition of the chamber, gloves, and substrates. In this sense, we still cannot totally control the electrostatic condition. One of the future works for improving this system is to clarify the unknow parameter to make particle handling more stable. One of the keys to improve the system is to establish an electric neutralization method which can work effectively on target particles inside the clean chamber.

REFERENCES

[1] Fujiwara A. et al. (2006) Science 312, 1330–1334.
[2] Yano H. et al. (2006) Science 312, 1350–1353.
[3] Abe M, et al. (2011) LPSC LXII, #1685.
[4] Yada T. et al. (2014) Meteoritics Planet. Sci. 49, 135–153.
[5] Lee B. W. and Orr D. E., Web page of AphaLab, Inc.
(https://www.alphalabinc.com/triboelectric-series/ Retrieved on Jan. 12, 2022)
[6] Uesugi M. et al. (2014) Earth Planet Space 66:102.

Table 1. Results of experiments of electrostatically controlled manipulation for three different material particles on an earthed aluminum foil in normal atmosphere.

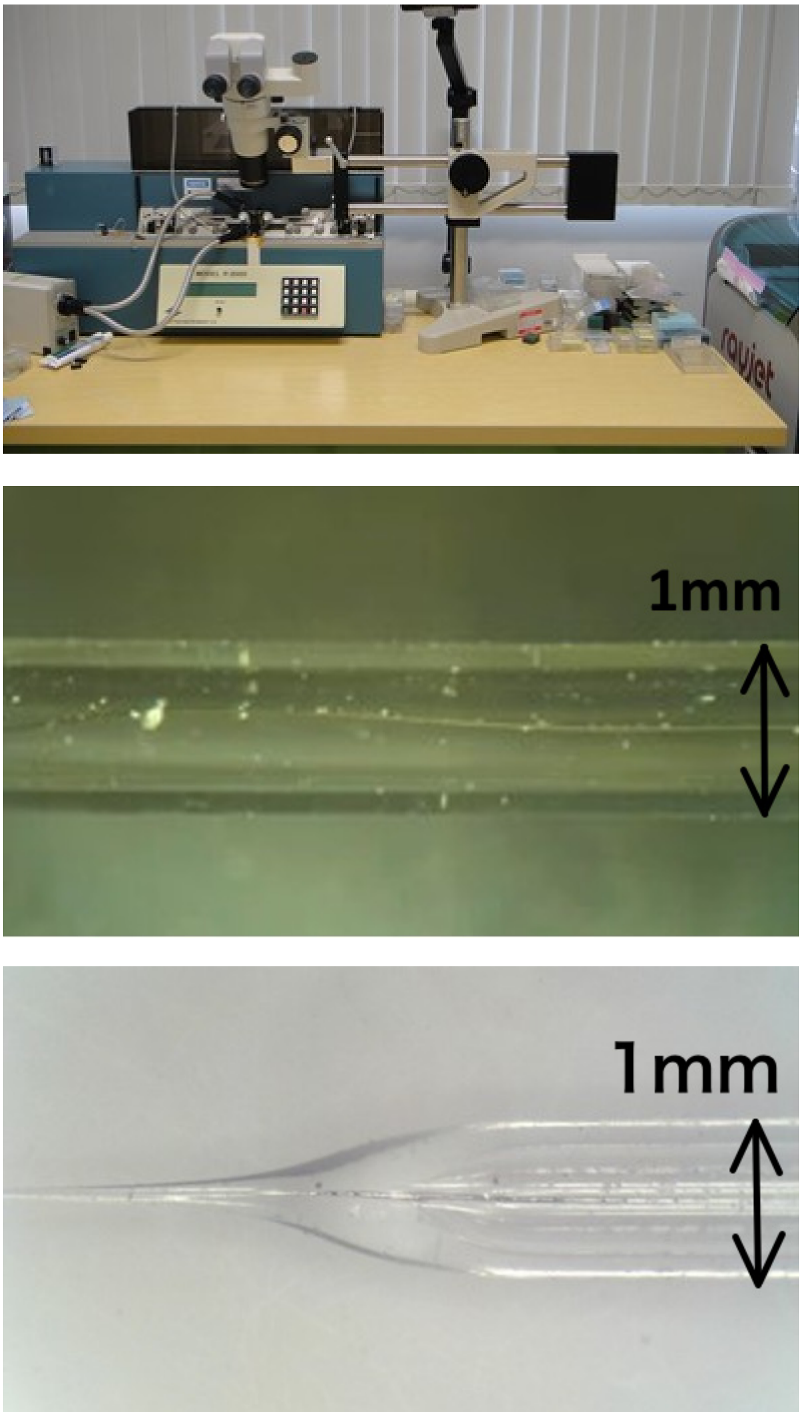
Voltage applied for the glass probe (V)	Glass	Teflon	Polystyrene
+0.7	×	○	◎
+0.5	×	◎	×
+0.3	×	□	×
0	×	□	×
-0.3	×	×	×
-0.5	◎	×	×
-0.7	○	×	×

Symbol ◎ indicates the condition by which every sample particle can be controlled reliably.

Symbol ○ shows the condition where several grains of sample particles can be handle safely.

Symbol □ shows the condition where tip of needle collect many grains of sample particles, and individual handling of the sample grain is difficult.

Symbol × shows that the sample particles cannot be handled.



(a) (b) (c)

Fig. 1. Technique for making quartz glass needles including electrodes made of Pt wire. (a) A photo of P-2000 by Sutter Instrument. With this instrument, quartz glass tubes with Pt wires inside is melt by laser and pulled and stretched to be needles. (b) A micrograph of a platinum wire embedded in quartz glass inside the quartz glass tube before laser ablation. (c) A micrograph of quartz glass needle with Pt wire inside after the laser ablation.

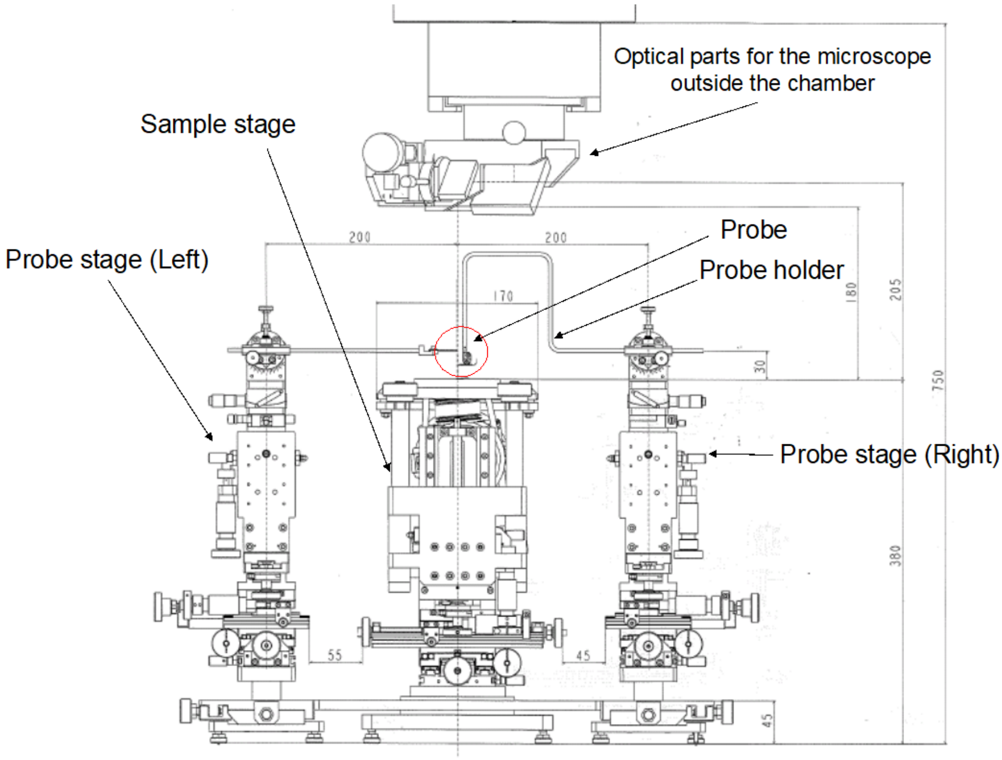


Fig. 2. A schematic viewgraph of a sample stage and left and right probe stages from the front side.

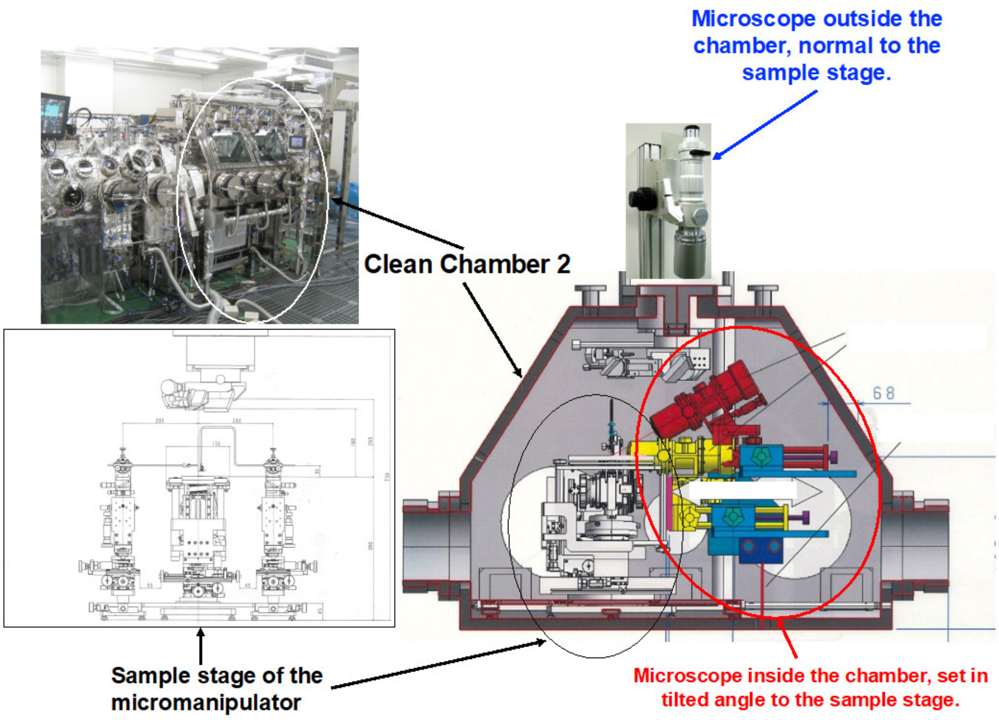
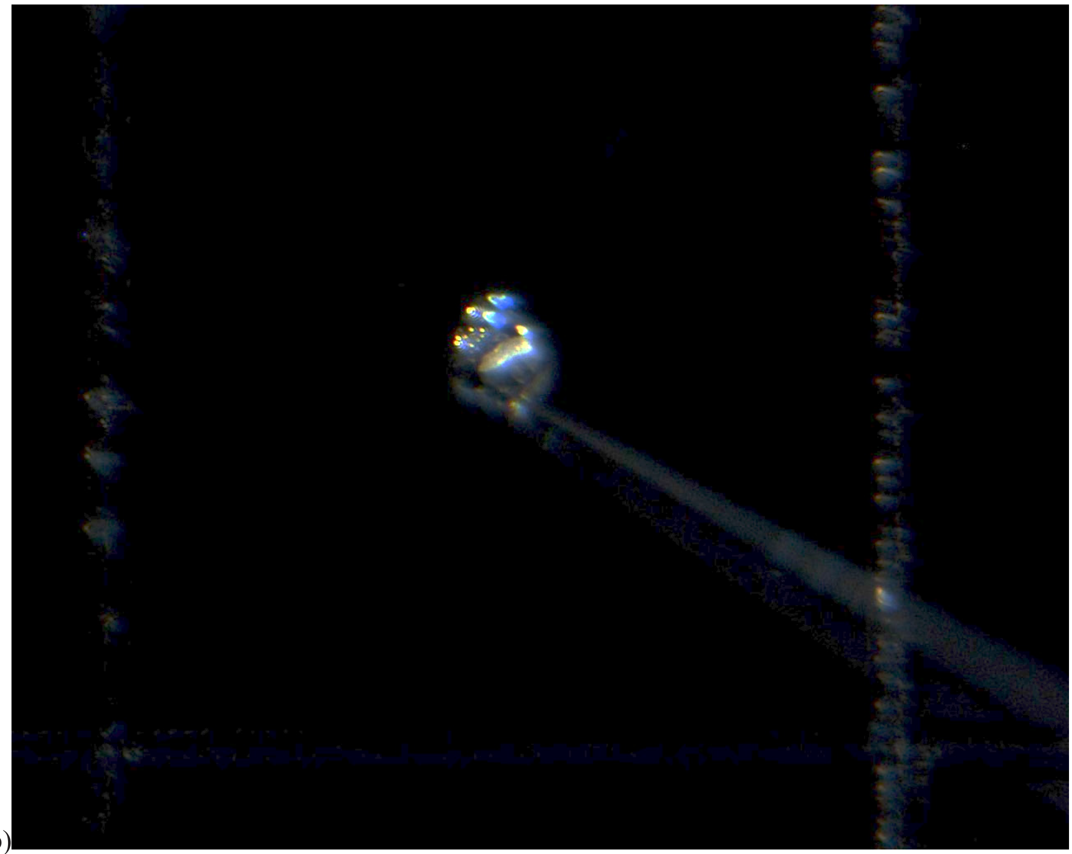
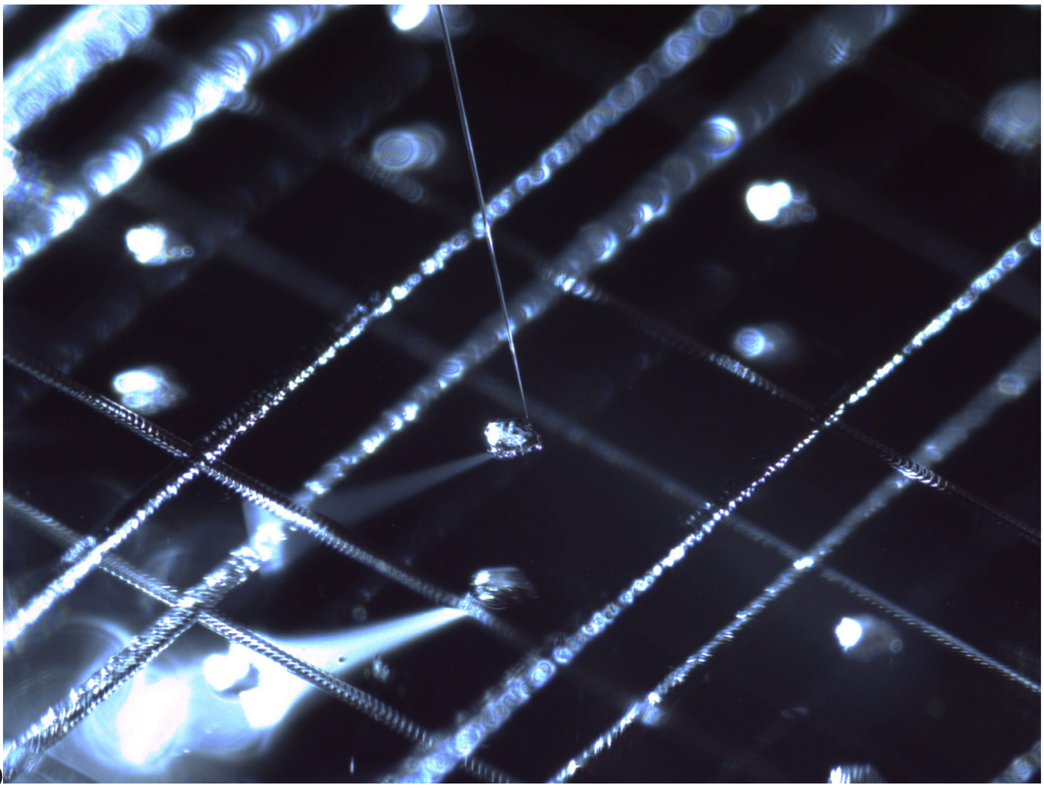
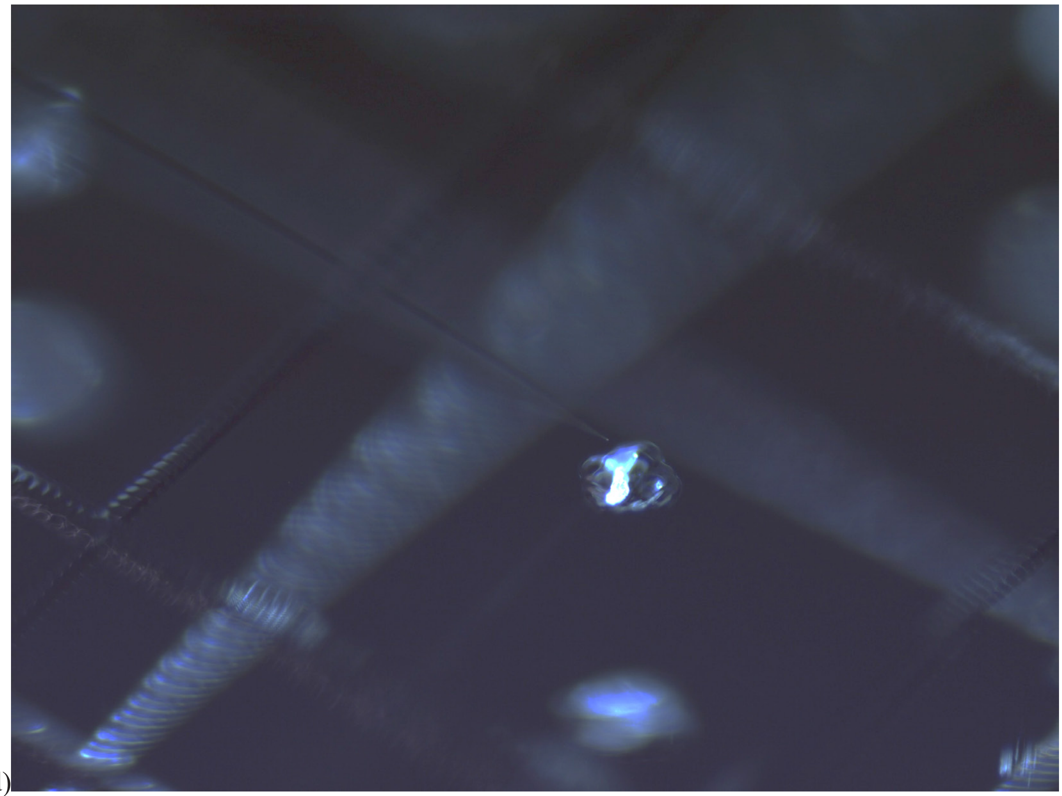


Fig. 3. A schematic viewgraph of microscopes equipped for the manipulation system. One sample stage and two probe stages equipped with quartz glass probes including electrodes are installed inside the Clean Chamber 2. Two optical microscopes observing the top of the probe from tilted angles are equipped inside the Chamber, and a microscope observing the probe from the vertical angle is equipped outside the chamber.


(a)

(b)



(c)



(d)

Fig. 4. (a) A configuration of LCD monitors of the PCs controlling the CCDs of the microscopes. With the three microscopes, we can observe a target particle from three different angles. For example, these are micrographs of RA-QD02-283, which sizes 185 μm in major axis, was placed on the gridded glass slide, shot by (b) microscope 1, (c) microscope 2, and (d) microscope 3. With this system, the operator can recognize accurate position of the top of the probe stereoscopically.

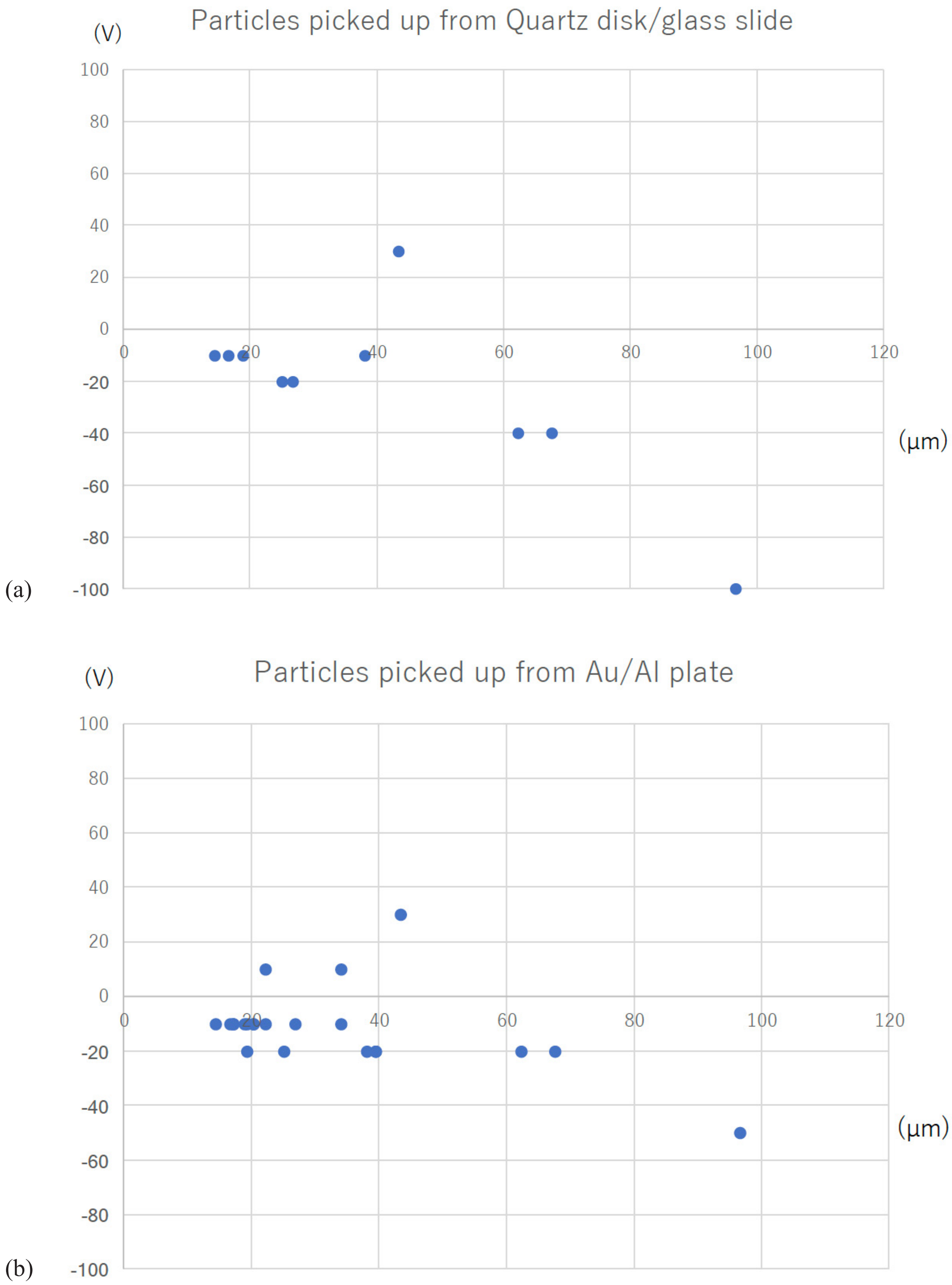


Fig. 5. Viewgraphs showing voltage charged on the probes of electrostatically-controlled micromanipulator in lifting up same type 1 and 2 Itokawa particles of various sizes from substrates of non-conductive and conductive materials (a) disks and slides made of quartz glass and (b) disks made of Al or sample holder plates made of gold. Horizontal axes are major axes of target particles and vertical axes are voltage charged on the probe when the target particles were lifted up from the substrates. Each particle in both viewgraphs is basically identical, although some data are missed in either substrate in some cases.

JAXA Research and Development Report JAXA-RR-21-009E

Development of electrostatically controlled micro-manipulation system in purified ultra-clean nitrogen environment and its performances to handle micrometer-sized asteroidal regolith particles

Edited and Published by:	Japan Aerospace Exploration Agency
	7-44-1 Jindaiji-higashimachi, Chofu-shi, Tokyo 182-8522 Japan
	URL: http://www.jaxa.jp/
Date of Issue:	February 18,2022
Produced by:	Matsueda Printing Inc.

Japan Aerospace Exploration Agency