

UlvacPhi PHI5000 VersaProbe III with AES

Part 0, Manual Operation Sequences

X-ray Laboratory, Nano-Engineering Research Center, Institute of Engineering Innovation, School of Engineering, The University of Tokyo

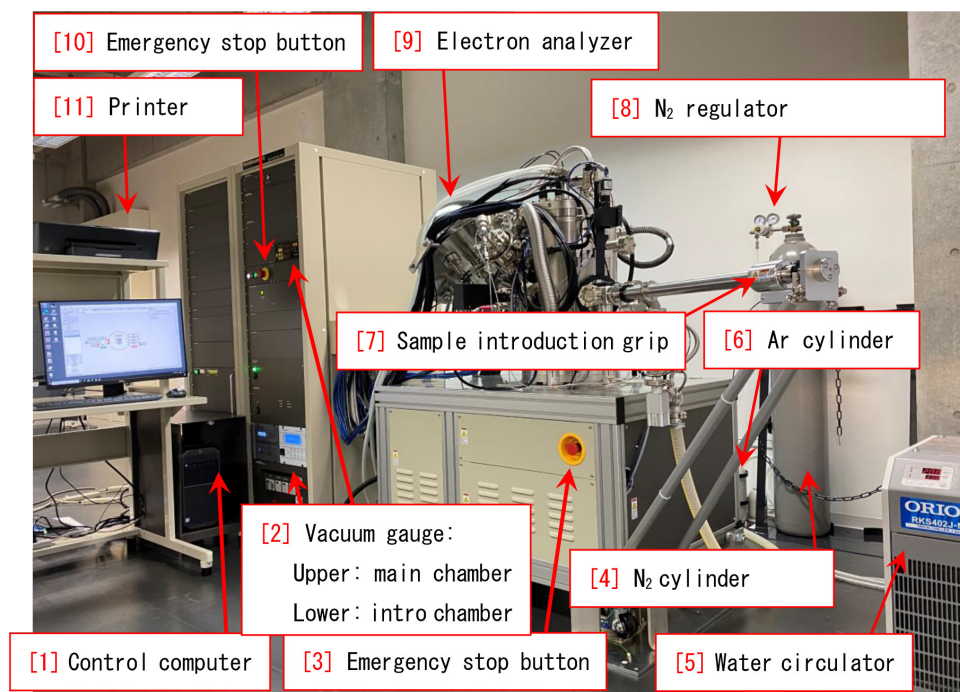


Figure 0: Whole figure of the apparatus.

PHI 5000 VersaProbe III with AES can be controlled by the computer. However, there are several operations that should be done manually: first, setting the sample holder into the vacuum chamber (§1.1), secondly, creation of the ‘Platen’ (sample holder for the one-time experiment) and taking photograph of the samples on the sample holder (§1.2), and thirdly, taking out of the sample holder (§2.1). These sequences do not have to be memorized but should correctly be done referring to this manual.

Appendix A describes how to initialize the sample stage.

The usual usage fee of the VersaProbe III is ¥4,400/h. However, it is discounted to be ¥2,900/h by uploading the raw data files and an excel file that describes the information about the samples, How to upload the data files is described in Appendix B.

The URL for uploading the data files is as follows: https://lcnet.t.u-tokyo.ac.jp/data_system/.

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Chapter 1

Manual operation before starting the experiment



Figure 1.1: Log-in window of SmartSoft-VP

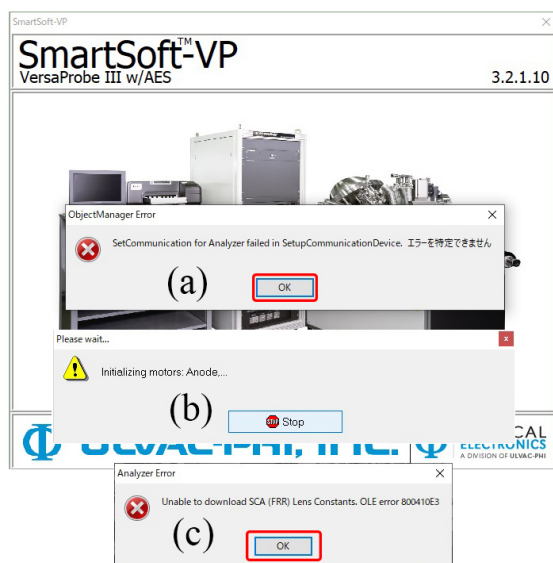


Figure 1.2: Start-up window of the VersaProbe. 'OK' on (a) can be clicked to open (b). After waiting for a moment, (c) is displayed. 'OK' on (c) can be clicked to continue

1.1 Introduction of the sample holder to the introduction chamber

1.1.1 Fill in the experimental notebook

Date, affiliation (laboratory), status (school grade), phone number, and pressure in the main chamber before starting and finishing the experiment, should be described on the experimental notebook.

1.1.2 Start up of the control program 'SmartSoft-VP'

After the photograph of the VersaProbe III is shown, Figs. 1.2 (a), (b) and (c) are displayed. In Fig. 1.2 (a), 'OK' can be clicked to show Fig. 1.2 (b). After waiting for a moment, Fig. 1.2 (c) is displayed. Here, 'OK' can be clicked to start up the control software 'SmartSoft-VP'.

'[2] System' tab on the upper left of Fig. 1.3 should be opened. Then, '[3] Vacuum' tab on the lower right should be opened. '... [4]' can be clicked to enlarge the vacuum gauge.

1.1.3 N₂ purge of the introduction chamber

Fig. 1.4 shows around the regulator of N₂. It should be verified that both the values of '[1] first-order pressure' and '[2] second-order pressure' are zero. After verifying that the

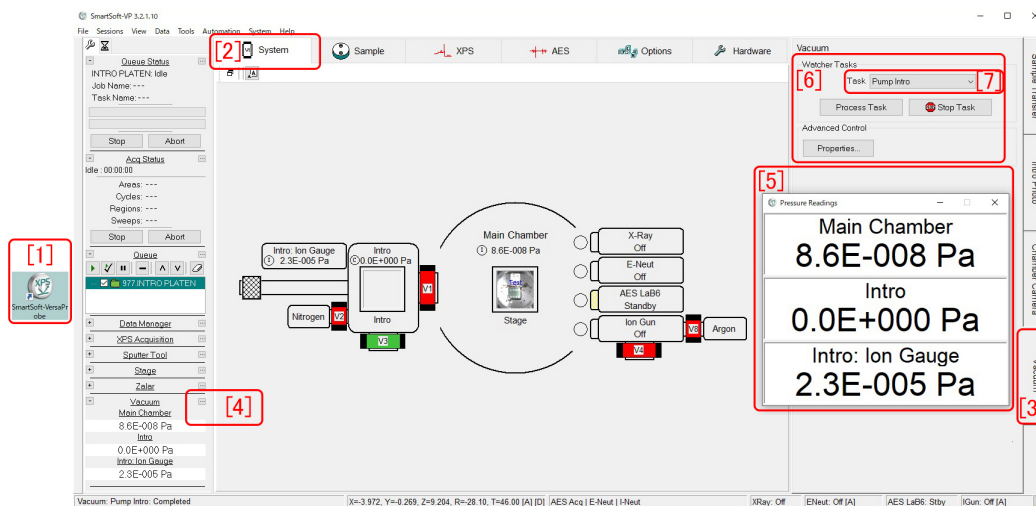


Figure 1.3: VersaProbe III Initial window of the SmartSoft-VP for controlling the VersaProbe III

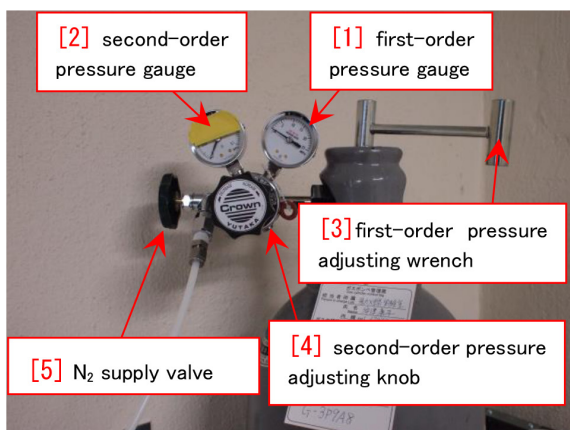


Figure 1.4: N₂ flow regulator

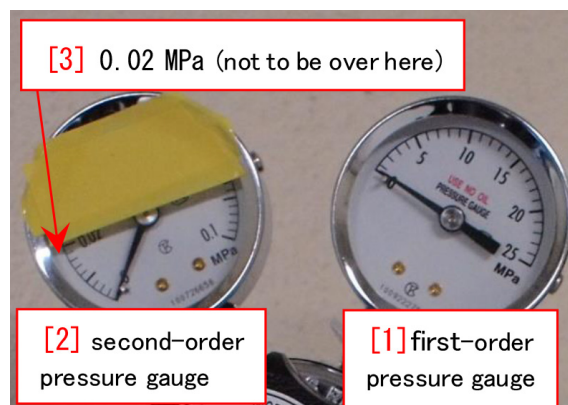


Figure 1.5: N₂ flow regulator (enlarged)

‘[5] N₂ supply valve’ has been opened, ‘[4] second-order pressure adjusting knob’ should be turned counterclockwise such that it can be turned easily. ‘[3] first-order pressure adjusting wrench’ should be opened by turning it counterclockwise to increase the first-order pressure quickly. Next, ‘[4] second-order pressure adjusting knob’ in Fig. 1.4 should be slowly and carefully such that it slight increases to be around 0.015MPa (less than here 0.02 MPa).

Fig. 1.6 is an enlargement of ‘[6]’ on the up-

per left of Fig. 1.3. With ‘Backfill Intro [1]’ selected in the pull-down menu, ‘[2] Process Task’ can be clicked to N₂-purge the introduction chamber and change ‘Nitrogen V2’ to be green. The values of ‘Intro’ and ‘Intro: Ion Gage’ increase to be ‘1.0E+005 Pa’ in Fig. 1.3 ‘[5]’. After N₂-purge is finished, ‘[3] first-order pressure adjusting wrench’ should be closed by turning it clockwise. It should be verified that the ‘first-order pressure’ and ‘second-order pressure’ decrease to be zero. The other knobs do not have to be touched.

1.2. CREATION OF NEW 'PLATEN' AND TAKING PHOTOGRAPH OF THE SAMPLE HOLDER3

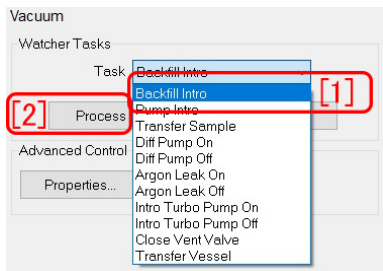


Figure 1.6: Window of SmartSoft-VP

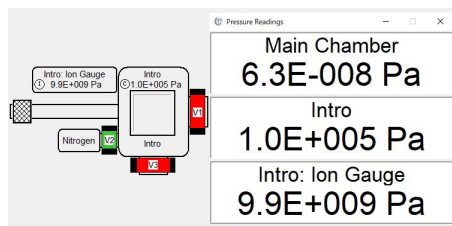


Figure 1.7: N₂ purge finished

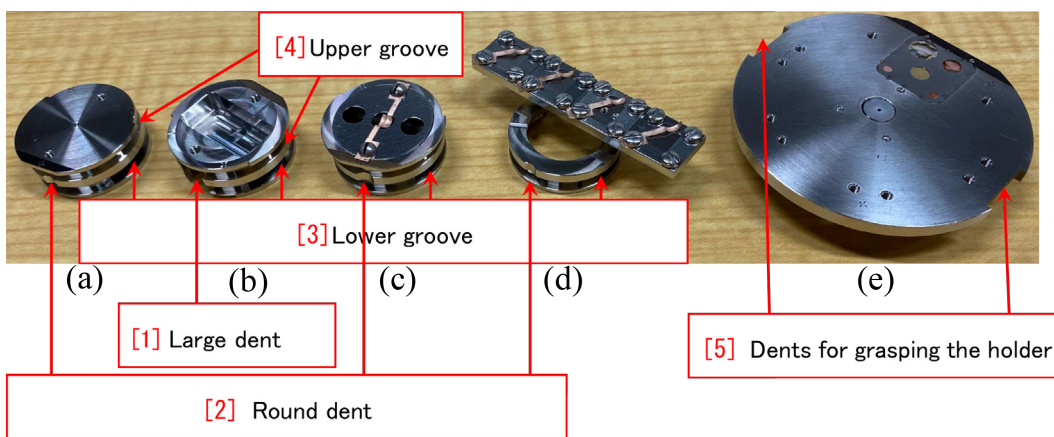


Figure 1.8: Sample holders

1.1.4 Choice of the sample holder

Samples are usually pasted on the sample holder with conductive both-sided carbon tape. The height limit of the sample is 7 mm. Fig. 1.8 shows sample holders for the VersaProbe III. 60mm sample holder [Fig. 1.8 (e)] is frequently used since it can hold many samples. Fig. 1.8 (a), (b) and (c) are 25mm sample holders. (a) is a flat normal holder. (b) with a spot boring is prepared for samples whose height is large. The samples can also be fixed by using screws and metal fixtures as shown in (c). The use of (d) AR-XPS holder is prohibited **since this holder caused a serious trouble.**

1.1.5 Set of the sample holder in the introduction chamber

In Fig. 1.9 (a), a 25mm sample holder is set to the sample introduction fork. As shown in Fig.

1.8, the sample holder should be set on the sample introduction fork such that '[2] Round dent' on the '[3] Lower groove' just fits the screw of the sample introduction fork. It should be verified viewing through the front window in Fig. 1.9 (d) that the sample holder does not inclined as shown in Fig. 1.9 (e)

1.2 Creation of new 'Platen' and Taking photograph of the sample holder

In UlvacPhi, the sample holder for one experiment is called 'Platen'

1.2.1 Naming the 'Platen' and setting the data folder

In Fig. 1.10, after opening the tabs of 'System[1]' and 'Sample Transfer[2]', '[3] Create

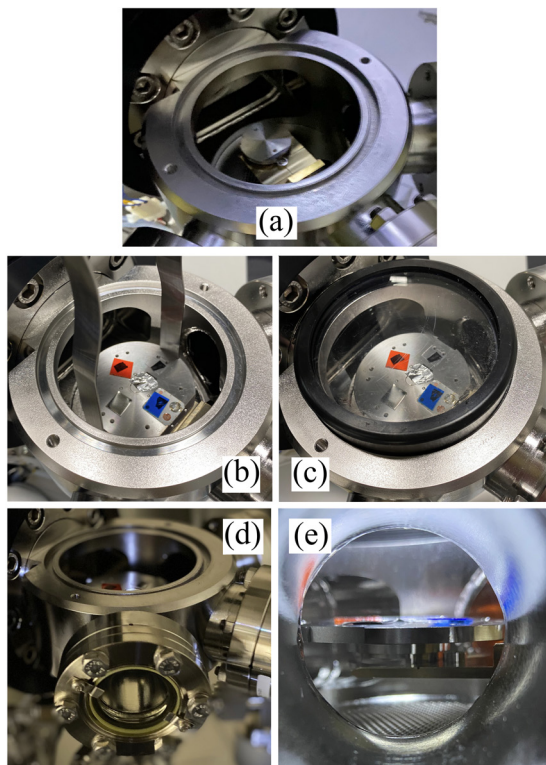


Figure 1.9: (a) a 25 mm sample holder has been set on the sample introduction fork, (b) a 60 mm sample holder has been set in the sample introduction chamber, (d) viewing through the front window, (e) the sample holder to be horizontal

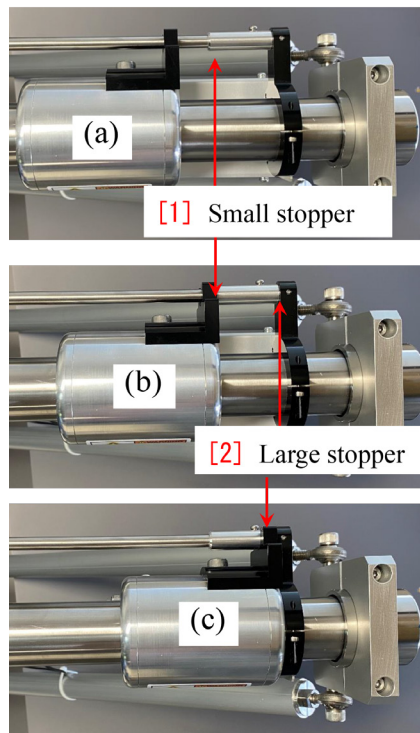


Figure 1.11: The status of the sample introduction grip is (a) free, (b) stopped at the small stopper and (c) stopped at the large stopper

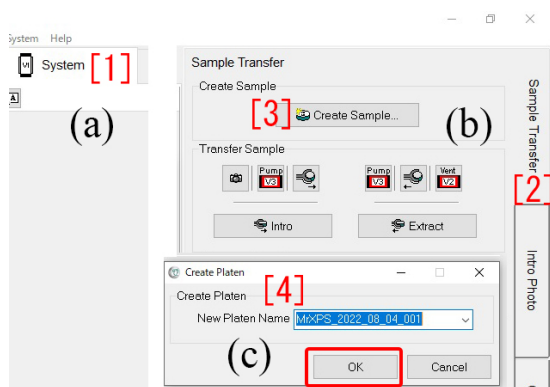


Figure 1.10: After opening the (a) '[1] System' tab and then (b) 'Sample Transfer [2]' tab, '[3] Create Sample' should be clicked. When (c) is opened, '[4] New Platen Name' should be typed to click 'OK'

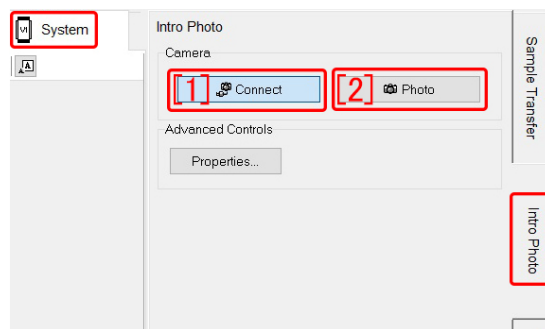


Figure 1.12: 'System' tab on the upper-left and 'Intro Photo' tab on the right have been opened. After clicking '[1] Connect' and then waiting for ten second or so, '[2] Photo' can be clicked to take a photograph of the sample holder

1.2. CREATION OF NEW 'PLATEN' AND TAKING PHOTOGRAPH OF THE SAMPLE HOLDER5

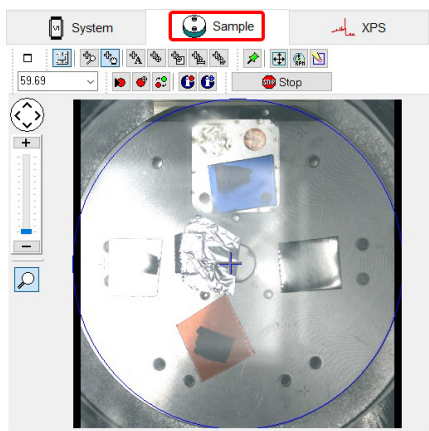


Figure 1.13: With a tab other than 'System' opened, the photographs of the sample holder can be observed

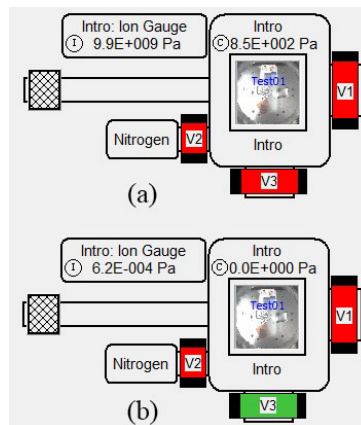


Figure 1.15: After waiting around forty seconds or so, (a) vacuuming mode using only the rotary pump changes to (b) that using both the rotary and turbo-molecular pumps

Sample' has been clicked. In Fig. 1.10 (c), '[4] New Platen Name' can be typed to click 'OK'. Alphabetical characters and number can be used. Kanji, Kana and other special characters cannot be used.

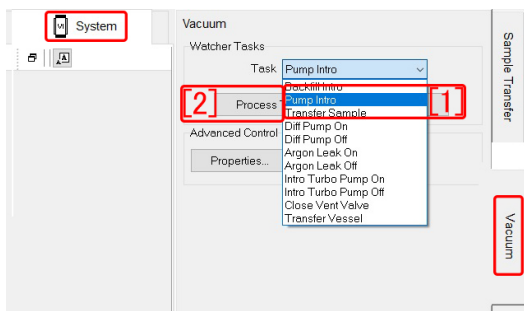


Figure 1.14: After verifying that the cover is put on the introduction chamber, 'Pump Intro[1]' should be selected from the pull-down menu. Then, '[2] Process Task' can be clicked to start vacuuming the introduction chamber

1.2.2 Taking a photograph of the sample holder

While the photograph can be taken during vacuuming the introduction chamber through the transparent cover as shown in Fig. 1.9 (c), it can be also taken before putting the cover on the introduction chamber.

At first, the sample introduction grip should be stopped at the 'Small stopper' as shown in Fig. 1.11 (b). In Fig. 1.12 with 'System' tab on the upper-left and 'Intro Photo' tab on the right opened, '[1] Connect' should be clicked. After waiting for 10 second or so, '[2] Photo' can be clicked to take a photograph of the surface of the sample holder. After waiting for 10 second or so, it can be observed if a tab other than 'System' is opened.

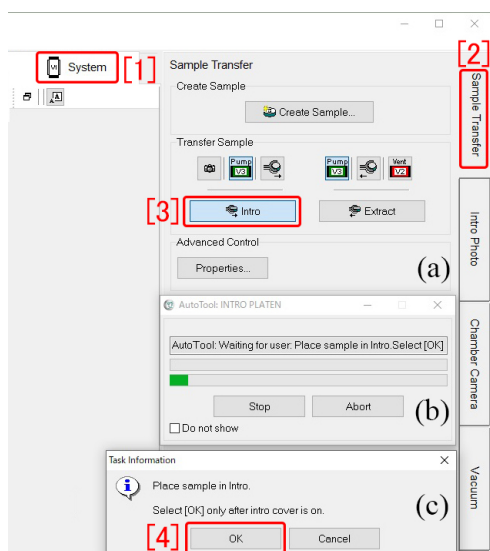


Figure 1.16: By clicking '[3] Intro' of (a), (b) and (c) are displayed. Then, '[4] OK' of (c) can be clicked to continue

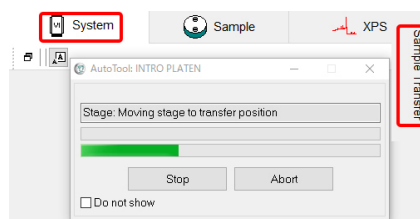


Figure 1.18: The sample stage is moving to the transfer position

1.2.3 Vacuuming the introduction chamber

Before putting the cover on the introduction chamber, inside the rim of the cover and the groove on the introduction chamber for putting on it should be swiped with ethanol since the vacuum level sometimes comes to be poor by dust.

After putting the cover, the sample introduction grip should be stopped at the 'Small stopper' as shown in Fig. 1.11 (b). After selecting 'Pump Intro' from the pull-down menu in Fig. 1.14, '[2] Process Task' should be clicked to start vacuuming the introduction chamber

After the red display of V3 in Fig. 1.15 (a) changes to be green as shown in Fig. 1.15 (b), the upper value of the vacuum gauge should become 0.0E000 within 2 seconds or so. After waiting 1 h or so, when the pressure gauge of the introduction chamber is less than 2.8E-004, the sample holder can be introduced to the main chamber from the introduction chamber.

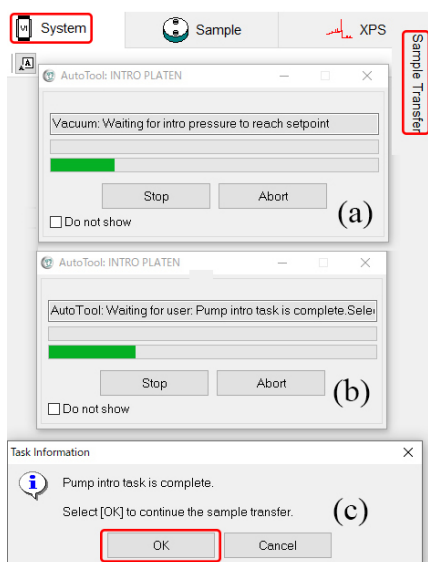


Figure 1.17: After vacuuming the introduction chamber for 1 h, 'OK' can be clicked to continue

1.3 Introduction of the sample holder to the main chamber

There are two ways of sample introduction to the main chamber: manual introduction and introduction by dragg&dragging the photograph.

§1.3.1 and §1.3.2 describe the former and the latter methods, respectively

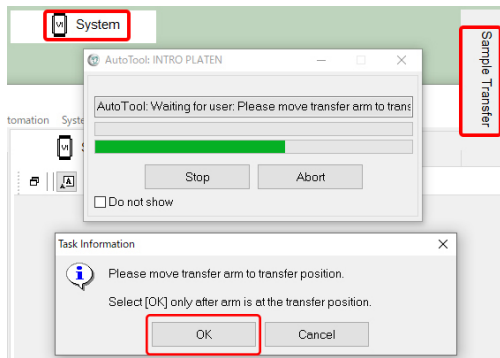


Figure 1.19: Here, the sample introduction fork should be slid into the main chamber. After completely sliding it as shown in Fig. 1.20, 'OK' can be clicked

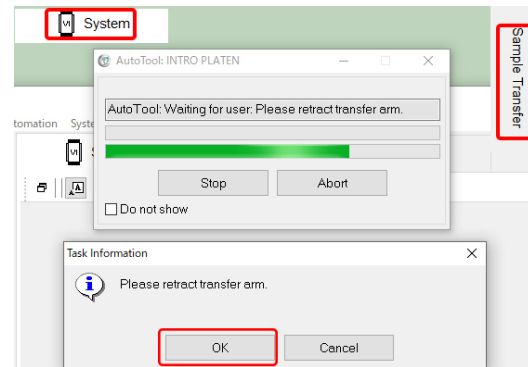


Figure 1.22: Here, the sample introduction fork should be slowly drawn after clicking 'OK'. Only the fork can be extracted without the sample



Figure 1.20: After inserting the sample introduction fork completely, 'OK' in Fig. 1.19 should be clicked

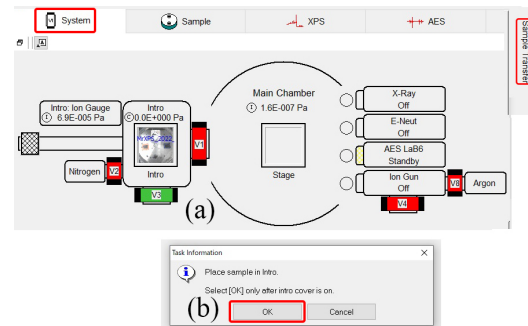


Figure 1.23: Here, the sample introduction fork should be slowly drawn. Only the fork can be extracted without the sample

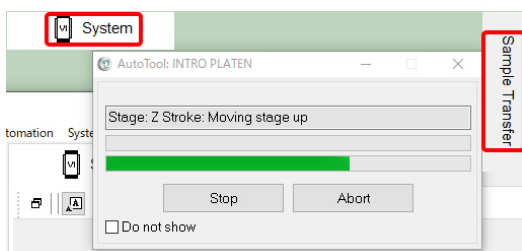


Figure 1.21: Wait for the sample stage moves upward such that the sample holder is captured by the sample stage

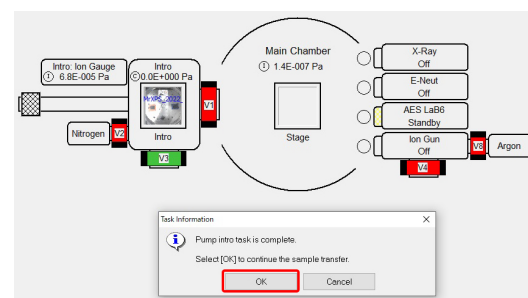


Figure 1.24: 1 h later after the introduction chamber is started to be vacuumed, 'OK' should be clicked for the message

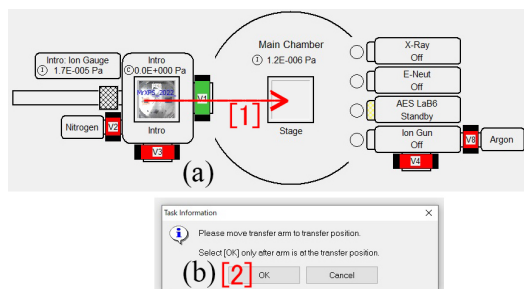


Figure 1.25: (a) After waiting for V1 valve to be opened, the photograph of the sample holder should be drag&dropped on the sample stage in the main chamber. After inserting the grip completely as shown in Fig. 1.20, (b) ‘[2] OK’ should be clicked

1.3.1 Manual introduction of the sample holder

‘[1] System’ and ‘[2] Sample Transfer’ tabs in Fig. 1.16 should be opened when introducing the sample holder to the main chamber. The user should wait for 1 h during the introduction chamber is being vacuumed. The pressure in the introduction chamber should be less than $2.8E-004$ in Fig. 1.7 when introducing the sample holder into the main chamber. ‘[3] Intro’ in Fig. 1.16 (a) should be clicked to introduce the sample holder to the main chamber. After waiting for a minute or so, V1 valve opens with a sound of compressed air. After clicking ‘[4] OK’ in Fig. 1.16 (c), the sample introduction grip should be slowly inserted such as to stop at the stopper. If it cannot be inserted completely to the stopper, it should be drawn to close V1. Then, the sample stage should be initialized by referring to the description in Appendix A [p.15].

After Figs. 1.17 (a) and (b) are shown, ‘OK’ in Fig. 1.17 (c) can be clicked. With this operation, the sample stage moves to the transfer position to show Fig. 1.18.

When Fig. 1.19 is shown, the sample holder can be transferred to the main chamber from the introduction chamber to be set on the sample stage by siliding the ‘Sample introduction fork’ stopped at position (b) of Fig. 1.11.

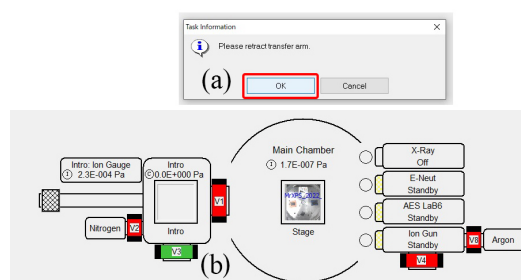


Figure 1.26: Here, the sample introduction fork should be slowly extracted. Only the sample introduction fork without the sample can be drawn

The sample introduction grip should be inserted such as to stop at the stopper as shown in Fig. 1.20. If it cannot be inserted completely, it should be drawn such that the sample holder is returned back to the introduction chamber to close V1. Then, the sample stage should be initialized referring to the description of Appendix A. After that, the sample transfer procedure should be retried.

With regard to the XPS measurement after the sample transfer, the description in Part1 manual should be referred.

1.3.2 Introduction of the sample holder by click&dragging the photograph

When the pressure in the introduction chamber in Fig. 1.7 is less than $2.8E-004$ after waiting for 1 hour, ‘OK’ can be clicked for the message in Fig. 1.24. The photograph of the sample holder can be clicked&dragged as shown in ‘[1] →’.

After inserting the sample introduction fork completely to stop at the stopper as shown in Fig. 1.20, ‘OK’ in Fig. 1.25 (b) should be clicked.

After clicking ‘OK’ for the message of Fig. 1.26 (a), the sample introduction fork should be drawn slowly such that the sample holder comes back into the introduction chamber. With this procedure, the sample introduction finished. Then, the XPS measurement can be

started referring to the description in Part1 manual.

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Chapter 2

Operation after finishing the experiment

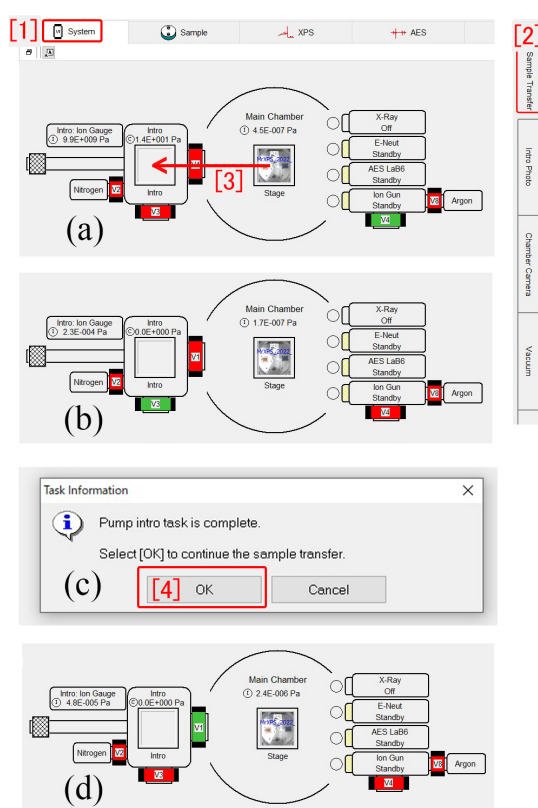


Figure 2.1: With ‘[1] System’ tab on the upper-left and ‘[1] Sample Transfer’ tab on the upper-right opened, (a) the photograph of the sample holder can be click&dragged to show (b) ‘[4] OK’ in (c) can be clicked to show (d). When (d) is shown, the sample transfer grip should be slid in as shown in Fig. 2.2.



Figure 2.2: After V1 valve is opened, the sample introduction grip should be drawn such as to stop at ‘[1] Small stopper’ as shown in Fig. 1.11 (b)

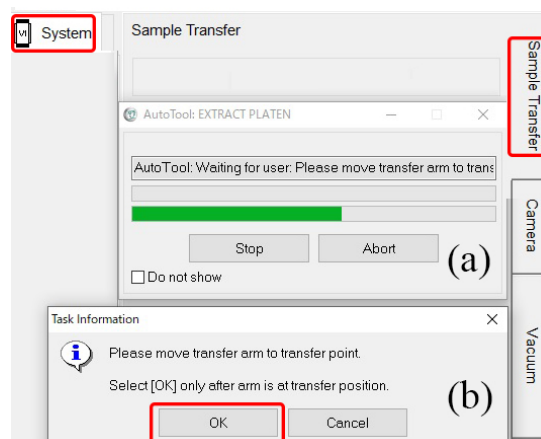


Figure 2.3: After (a) and (b) are shown, ‘OK’ on (b) should be clicked

2.1 Extraction of the sample holder

2.1.1 Vacuuming the introduction chamber

11

After selecting ‘Pump Intro [1]’ from the pull-down menu of Fig. 1.14, ‘[2] Process Task’

should be clicked to start vacuuming the introduction chamber.

After waiting for 15 min or so such that the pressure in the introduction chamber is less than $2.8E-004$, the process to extract the sample holder can be done.

2.1.2 Extraction of the sample holder from the main chamber to the introduction chamber

In Fig. 2.1 (a), with '[1] System' tab and '[2] Sample Transfer' tab opened, the photograph of the sample holder can be click&dragged as shown in '[3] ←' to close V4, open V3 and show Fig. 2.1 (b). In Fig. 2.1 (c), '[4] OK' should be clicked to open V1 and show Fig. 2.1 (d). Here, the sample introduction grip should be slid in such that it stops at the stopper as shown in Fig. 2.2.

If the sample introduction fork cannot be completely slid to the stopper position, it should be drawn to close V1. In such a case, the sample stage should be initialized referring to the description in Appendix A. After that, the procedure of Fig. 2.1 should be retried.

When Fig. 2.3 is shown, it should be verified that the sample introduction fork has been inserted completely as shown in Fig. 2.2. Then, 'OK' should be clicked. With this operation, the sample stage moves downward such that the sample holder is transferred to the sample introduction fork.

When Fig. 2.4 is shown, the sample introduction fork should be drawn slowly and carefully such that the sample holder is returned to the introduction chamber as shown in Fig. 2.5 (a) and (b).

2.1.3 N₂ purge of the introduction chamber

Fig. 2.6 shows around the regulator of the N₂ cylinder. Fig. 2.7 shows the enlarged one. It should be verified that the values of both '[1] First-order pressure gauge' and '[2] Second-order pressure gauge' are zero. At first, '[4] second-order pressure adjusting knob'

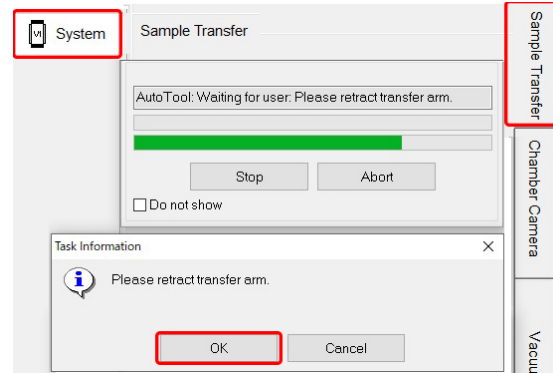


Figure 2.4: When this message is shown, after verifying the sample holder inserted, the sample introduction grip should be slowly drawn

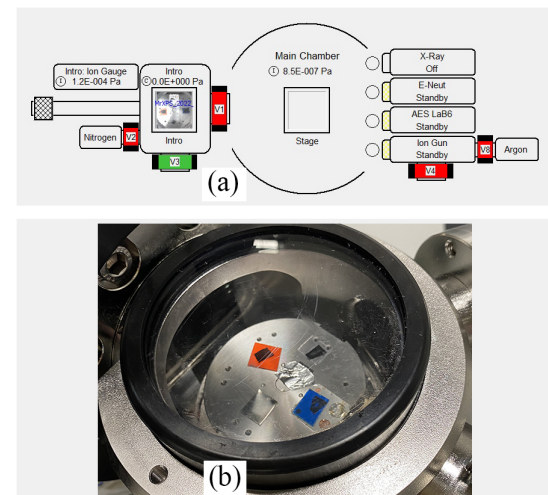


Figure 2.5: The sample holder has been returned back to the introduction chamber

should be turned counterclockwise such that it can easily be turned. Next, '[3] first-order pressure adjusting wrench' should be turned counterclockwise slightly such that the value of '[1] First-order pressure gauge' increases rapidly. Then, '[4] second-order pressure adjusting knob' should be turned clockwise slowly such that the second-order pressure is around 0.015 MPa. It should not be over 0.02 Pa.

Fig. 2.8 is an enlargement of around '[6]' of Fig. 1.3 [p.2] 'Backfill Intro [1]' should be selected to click '[2] Process Task' for N₂-purging the introduction chamber. When purging, 'Nitrogen V2' on the left of vacuum circuit changes

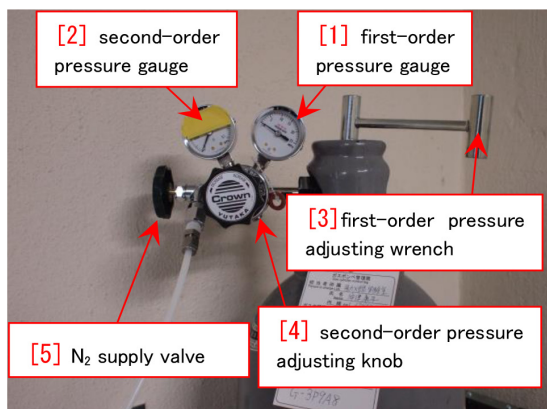


Figure 2.6: N₂ flow regulator for purging the introduction chamber

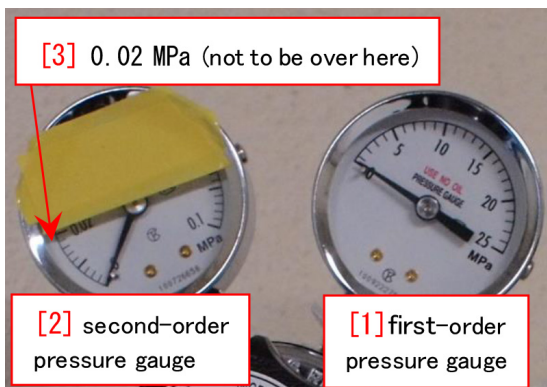


Figure 2.7: N₂ flow regulator for purging the introduction chamber (enlarged)

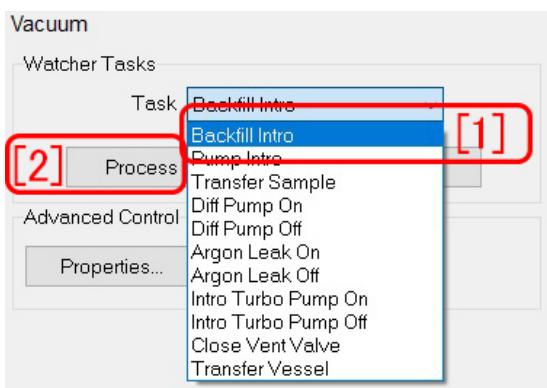


Figure 2.8: With tabs of ‘System’ and ‘Vacuum’, ‘Backfill Intro’ should be selected to click ‘Process Task’

to be green from red. The value of ‘Intro’ in Fig. 2.9 increase to be 1.0E+005 Pa.

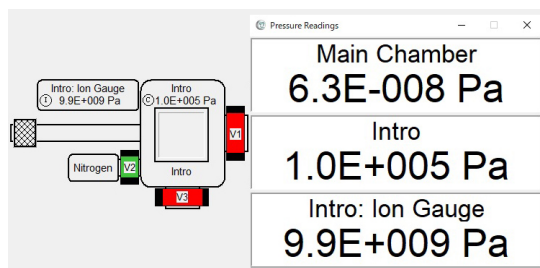


Figure 2.9: After N₂-purging the introduction chamber

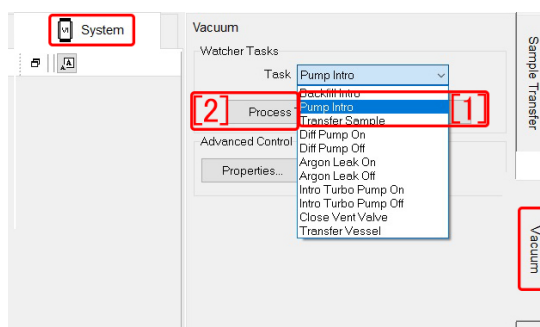


Figure 2.10: After N₂-purging the introduction chamber

After N₂-purging, ‘[3] first-order pressure adjusting wrench’ should be turned clockwise to close. It should be verified that the first-order pressure and then the second-order pressure decrease to be zero. The other knobs do not have to be touched.

2.1.4 Vacuuming the introduction chamber

After putting the transparent cover on the introduction chamber, in Fig. 2.10, ‘Pump Intro [1]’ should be selected to click ‘[2] Process Task’ for vacuuming the introduction chamber.

After finishing the experiment, the final value of the pressure in the main chamber should be filled in on the experimental notebook.

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Appendix A

Initialization of the sample stage

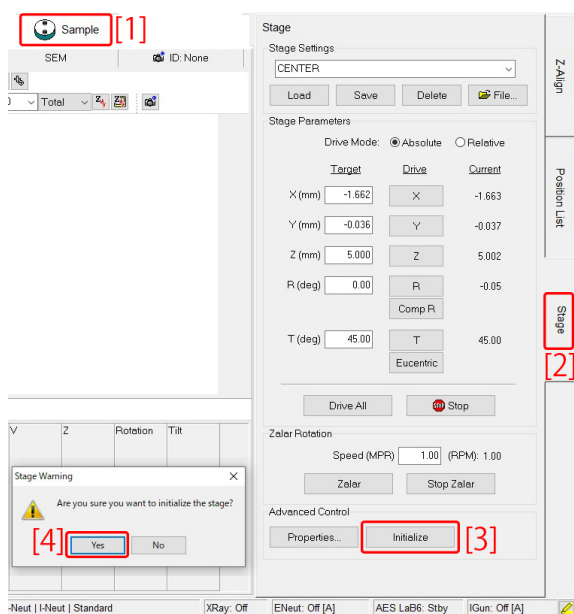


Figure A.1: With 'System [1]' on the upper-left and 'Stage [2]' on the right opened, 'Initialize [3]' can be clicked to initialize the sample stage. Before clicking '[4] Yes' on the lower verification window, it should be verified that the sample introduction fork has not been inserted in the main chamber

The sample stage of the VersaProbe III sometimes has trouble that can be resolved by initializing it.

When initializing the sample stage, the sample introduction fork should not be



Figure A.2: This window is displayed during initializing the sample stage

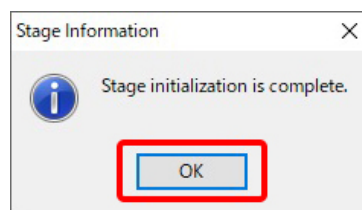


Figure A.3: This window is displayed when the initialization is completed

inserted to the main chamber.

In Fig. A.1, with 'Sample[1]' tab and 'Stage [2]' tab opened, 'Initialize [3]' can be clicked to initialize the sample stage.

When a verification message as shown on the lower left of Fig. A.1, it should be verified that the sample introduction fork has not been inserted to the main chamber before clicking '[4] Yes'.

During the sample stage is being initialided, Fig. A.2 is shown. After the initialization is finished, 'OK' in Fig. A.3 should be clicked.

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Appendix B

Upload of the measured data to the database

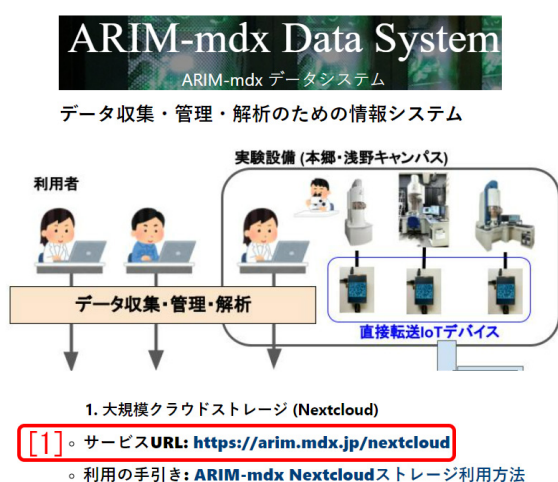


Figure B.1: URL of the hub base of the University of Tokyo of ARIM project. This URL should be clicked to upload the measured data: https://lcnnet.t.u-tokyo.ac.jp/data_system/

The usual usage fee of the VersaProbe III is ¥4,400/h. However, it is discounted to be ¥2,900/h for users who upload the data files on the ARIM database.

This chapter describes how to upload the data files on the ARIM database.

The URL in the figure caption of Fig. B.1 can be clicked to open the login window as shown in Fig. B.2. Here, ID and password can be typed to login the ARIM database. These have been validated when the application for use is accepted.



Figure B.2: Login window to the database. ID and password should be typed to open Fig. B.3

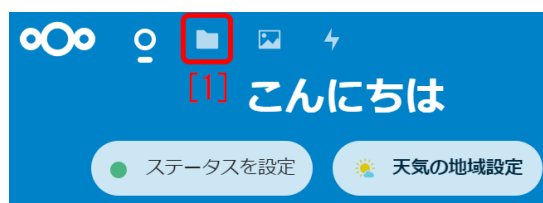


Figure B.3: '[1] icon' can be clicked to show Fig. B.4

From Fig. B.9 to Fig. B.8 can be clicked in this order to upload the raw data files. The data files can be dragg&dropped on the field of 'Here, files should be dragg&dropped' in Fig. B.9.

An excel file 'DataSheetVersaProbeIII_20XX_XX_XX_001.xls' that describes the information concerning the samples should also be uploaded here.

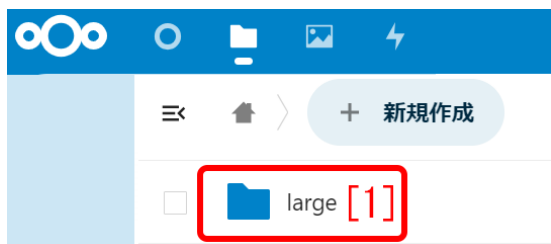


Figure B.4: 'Large [1]' should be clicked to show Fig. B.5

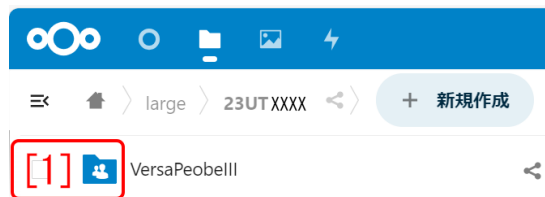


Figure B.7: '[1] VersaProbe III' icon should be double-clicked

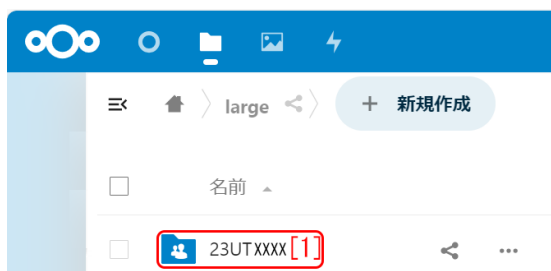


Figure B.5: '23UTXXXX [1]' should be clicked to show Fig. B.6

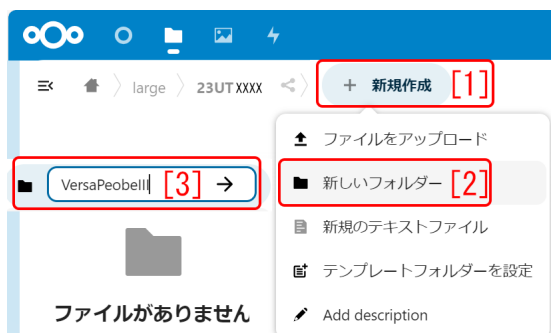


Figure B.6: 'Create [1]' and 'New folder [2]' should be clicked to type 'VersaProbeIII [3]'

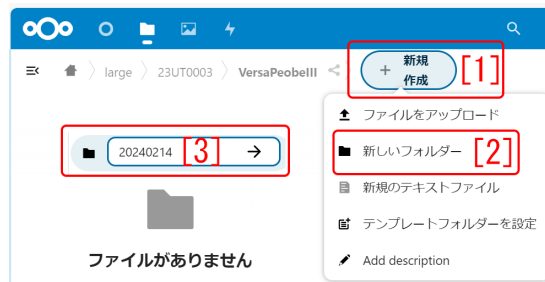


Figure B.8: 'Create [1]' and 'New folder [2]' should be clicked to type the 'date [3]'

After uploading the files, the browser may be closed.

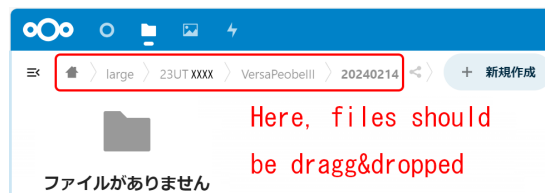


Figure B.9: Files should be drag&dropped on the folder of the date

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