

# ULVAC PHI PHI5000 VersaProbe Part 0 Important Sequence Manual

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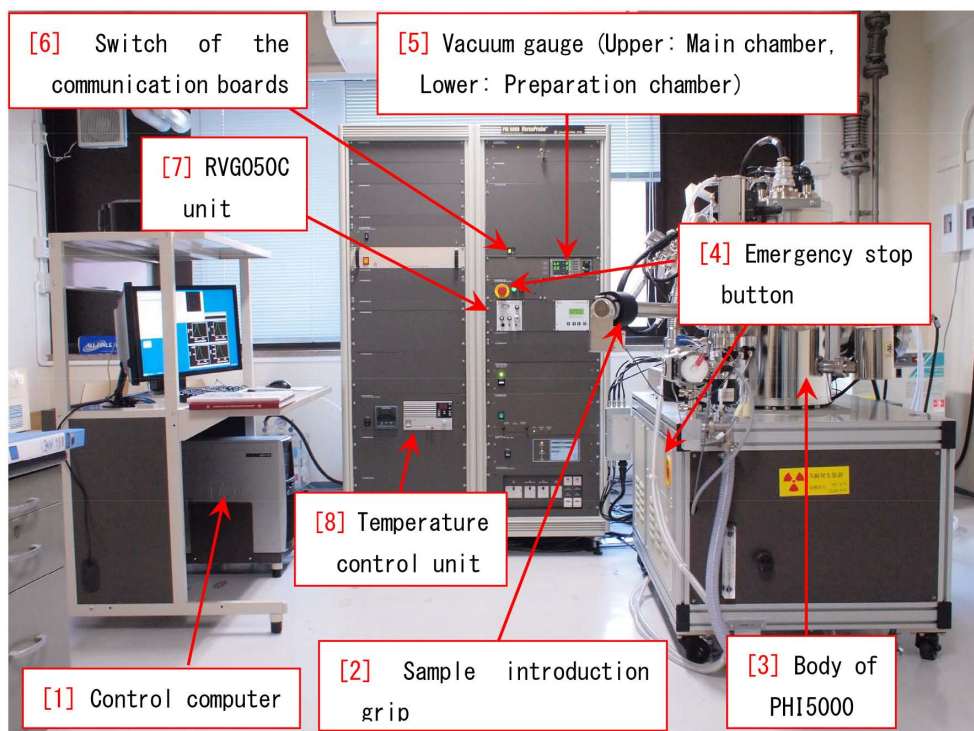


Figure 0: Whole figure of the apparatus.

Most of the operation of ‘PHI 5000 VersaProbe’ can be controlled by a computer. However, the following four operations should be done manually, that is, before starting the experiment, §1.1 [p.1] ‘Introduction of the samples’ and §1.2[p.5] ‘Startup of the Ar ion gun’ and after finishing the experiment, §2.1[p.7] ‘Shutdown of the Ar ion gun’ and §2.2[p.8] ‘Extraction of the samples’. These four sequences are described in this manual. The user does not have to memorize these sequences. However, when doing these sequences, follow the descriptions in this manual, please.

Furthermore, in Appendix A[p.10], the features and usages of three kinds of sample holders are described.

**The AR-XPS holder has been designed only for angle-resolved XPS (AR-XPS). Do not use this for another purpose, absolutely. When using the AR-XPS holder, refer to Appendix A §A.3 [p.12], necessarily.**

The usage of the transfer vessel is described in Appendix B [p.14]. A recommended procedure for preparing the samples without using the transfer vessel is described in Appendix C [p.22]. Appendix D [p.23] describes the procedure to restart the computer when it freezes by a communication error. Appendix E [p.24] describes how to do experiment controlling the temperature. Appendix F [p.29] describes how to do experiment using Mg&Zr X-ray source. Appendix G [p.32] describes how to initialize the sample stage. Appendix H [p.36] describes how to request the remote assistance.

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# Chapter 1

## Important sequences before starting the experiment

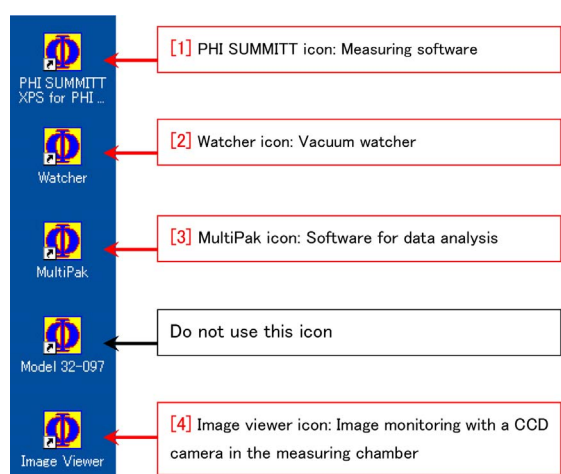


Figure 1.1: Important icons on the desktop.

### 1.1 Introduction of the samples

#### 1.1.1 Filling out in the experimental notebook

In the experimental notebook, date, affiliation, official title (or educational stage), phone number and pressure in the measuring (main) chamber (the upper value [Pa] displayed on ‘[5] Vacuum gauge’ in Fig. 0 on the cover of this manual) before starting the experiment.

#### 1.1.2 Selection of the sample holder

The sample holder on which the samples are mount can be selected from three kinds of holders; one-inch, two-inch and AR-XPS holders referring to the description in Appendix A [p.10]. Use of one-inch holder is most recommended

since it’s usage is simple and safe. When using two-inch and AR-XPS holders, follow the usage described in Appendix A [p.10], please.

**Particularly, AR-XPS holder can be used only for angle-resolved XPS (AR-XPS). It should not be used for another purpose. When doing AR-XPS using this holder, follow absolutely the usage described in Appendix A §A.3 [p.12], please.**

#### 1.1.3 Restart of the computer

When using this apparatus, a communication error rarely happens. The procedure to recover from this problem is described in Appendix D [p.23]. However, this trouble can be avoided with high possibility by doing this procedure before starting the experiment. The user is strongly recommended to do the procedure described in Appendix D [p.23].

#### 1.1.4 Startup of the control program on the computer

At first, turn on the switches of the dual monitors of computer. If any program were running on the computer, close the all softwares, please. Then, open ‘[1] CCD image’ in Fig. 1.2 [p.2] that shows the inside of the measuring (main) chamber by double-clicking ‘[4] Image Viewer icon’ in Fig. 1.1. If you can view nothing on ‘[1] CCD image’ window, verify that the light illuminating in the chamber is turned on and adjust the brightness, please.

Next, startup the control program by double-clicking ‘[1] PHI SUMMITT icon’ to open the



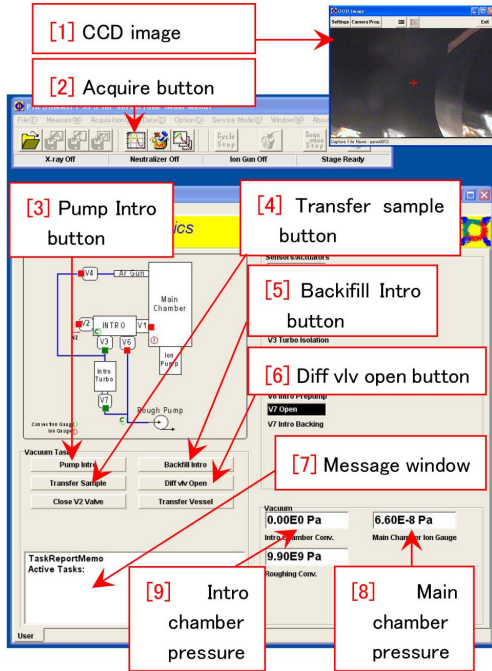


Figure 1.2: CCD image, Main Menu & Vacuum Watcher.

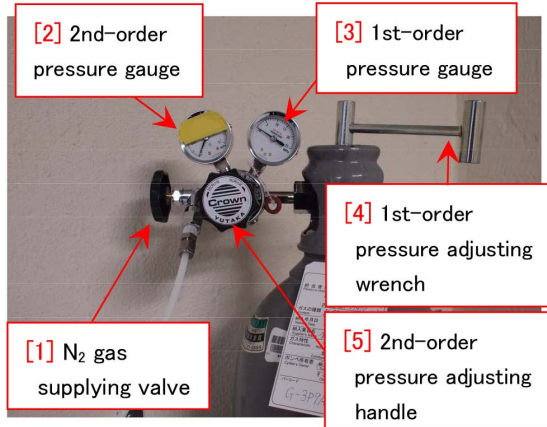


Figure 1.3: Regulator of N<sub>2</sub> gas cylinder.

other two windows, i.e. upper wide window (main menu) and lower big square window (vacuum watcher) as shown in Fig. 1.2.

### 1.1.5 Verification of the Vacuum Watcher

Before N<sub>2</sub>-purging ‘[1] Preparation chamber’ in Fig. 1.4, it should be verified that only V3 and V7 valves open (green) and the other valves close (red) on the Vacuum Watcher window as shown in Fig. 1.2. After that, the preparation

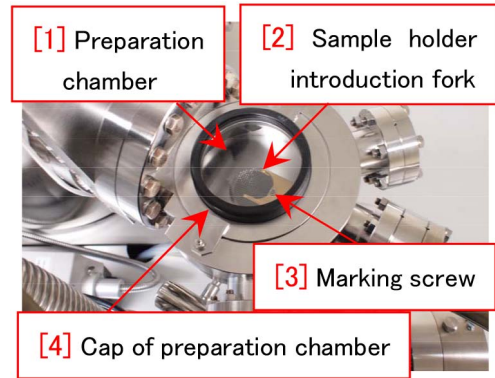


Figure 1.4: Preparation chamber.

(introduction) chamber should be N<sub>2</sub>-purged.

### 1.1.6 Opening the valve of the N<sub>2</sub> gas cylinder

For N<sub>2</sub>-purging the preparation (introduction) chamber, dry N<sub>2</sub> gas should be supplied from the N<sub>2</sub> cylinder.

It should be verified in Fig. 1.3 that both ‘[3] 1st-order pressure gauge’ and ‘[2] 2nd-order pressure gauge’ indicate zero and that ‘[1] N<sub>2</sub> gas supplying valve’ opens. Next, ‘[4] 1st-order pressure adjusting wrench’ should be turned counterclockwise (about 5°) to open. Next turn ‘[5] 2nd-order pressure adjusting handle’ clockwise (increase) slowly and carefully, please such that ‘[2] 2nd-order pressure gauge’ indicates 0.015 MPa.

### 1.1.7 N<sub>2</sub>-purge of the preparation chamber

For N<sub>2</sub>-purging the preparation (introduction) chamber, please click ‘[5] Backfill Intro button’ in Fig. 1.2. V3 valve automatically closes, and then V2 valve opens to start purging the preparation (introduction) chamber. If the pressure in the preparation chamber shown on ‘[5] Vacuum gauge’ in Fig. 0 on the cover of this manual (or ‘[9] Intro chamber pressure’ in Fig. 1.2) stops before reaching the atmospheric pressure (1.0E+5 Pa), ‘[5] 2nd-order pressure adjusting handle’ in Fig. 1.3 should be turned slightly clockwise (increase) slowly and carefully such that the pressure of the preparation (introduction) chamber reaches the atmospheric pres-



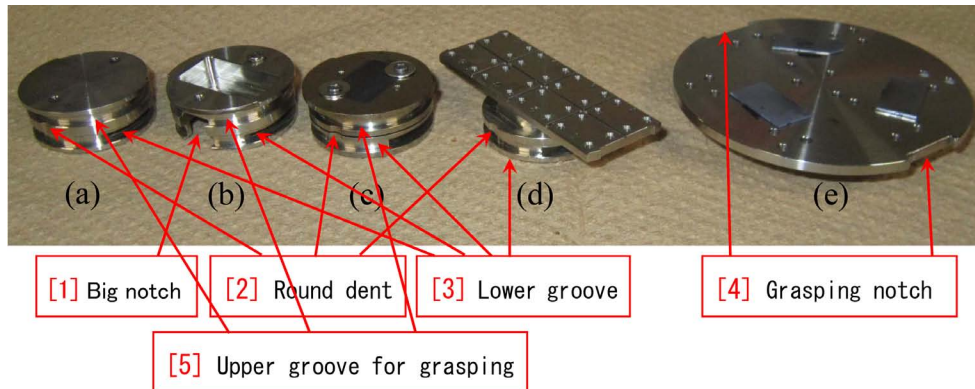


Figure 1.5: Sample holders (refer to Appendix A[p.10] for more detail).

sure. After purging the preparation chamber, ‘[4] 1st-order pressure adjusting wrench’ in Fig. 1.3 should be turned clockwise to close. Please verify that the pressure on ‘[4] 1st-order pressure gauge’ decreases to zero and then the pressure on ‘[2] 2nd-order pressure gauge’ decrease to be zero in Fig. 1.3. The other knobs in Fig. 1.3 should not be touched here.

### 1.1.8 Setting the sample holder

When using the transfer vessel, refer to the description in Appendix B [p.14], please.

When not using it, refer to the following description, please.

‘[4] Cap of preparation chamber’ in Fig. 1.4 can easily be removed after the preparation (introduction) chamber is  $N_2$ -purged. The rim of ‘[4] Cap of preparation chamber’ in Fig. 1.4 should be swiped by using ‘Kimwipe’ with alcohol. The groove in which ‘[4] Cap of preparation chamber’ to be put should also be swiped with ‘Kimwipe’ with alcohol grasped with a plastic tweezer. Here, a metal tweezer should not be used since it may damage the groove.

A sample holder should be set to ‘[2] Sample holder introduction fork’ in Fig. 1.4 such that ‘[3] Lower groove’ in Fig. 1.5 just fits to ‘[2] Sample holder introduction fork’ in Fig. 1.4 and ‘[3] Marking screw’ in Fig. 1.4 just fits to ‘[2] Round dent’ in Fig. 1.5. Here, verify that the sample holder is not tilted, please viewing through a window on the front side of preparation chamber.

Two-inch holder shown in Fig. 1.5(e) and AR-XPS holder in Fig. 1.5(d) do not have ‘[5] Up-

per groove for grasping’ but have ‘[3] Lower groove’ and ‘[2] Round dent’ similarly to one-inch holder.

For more details about the sample holders, refer to Appendix A [p.10], please.

### 1.1.9 Vacuuming the preparation chamber

After setting the sample holder in the preparation (introduction) chamber and putting on the cap of the chamber, ‘[3] Pump Intro button’ in Fig. 1.2 should be clicked to start vacuuming the preparation chamber. About 10 min later than this procedure, a ‘complete’ message appears in ‘[7] Message window’ in Fig. 1.2. After waiting for about 15 min, the sample holder can be transferred to the main chamber. However, because further sufficient vacuuming of the preparation (introduction) chamber is desirable, the user is recommended to have a lunch after starting to vacuum the preparation (introduction) chamber.

### 1.1.10 Preparation of the sample stage

‘[2] Acquire button’ in Fig. 1.6 [p.4] can be clicked to open three windows, i.e. ‘[3] Image window’, ‘[4] Acquisition setting window’ and ‘[5] Stage controller’ Figure 1.7 [p.4] is a closeup of ‘[3] Image window’ in Fig. 1.6 [p.4].

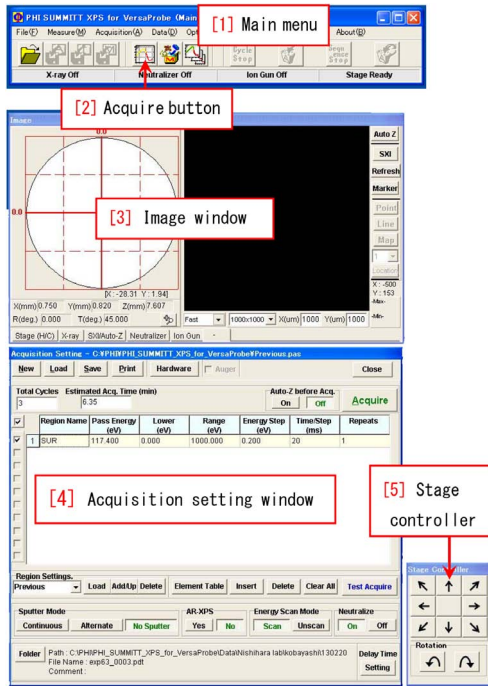


Figure 1.6: Image window, Acquisition setting window and Stage controller.

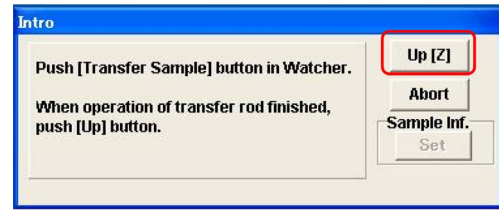


Figure 1.8: Up[Z] button should not be clicked soon.

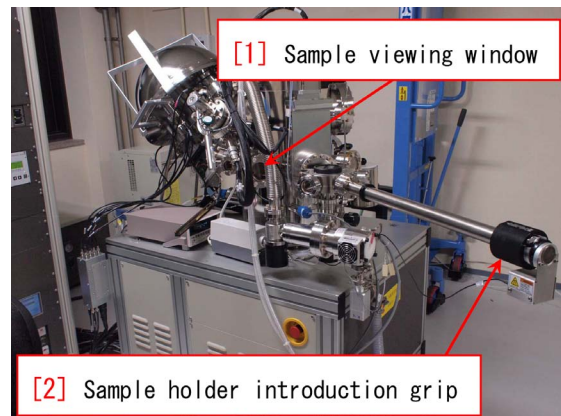


Figure 1.9: Sample introduction grip should be slide on the rod carefully and slowly watching through the viewing window.

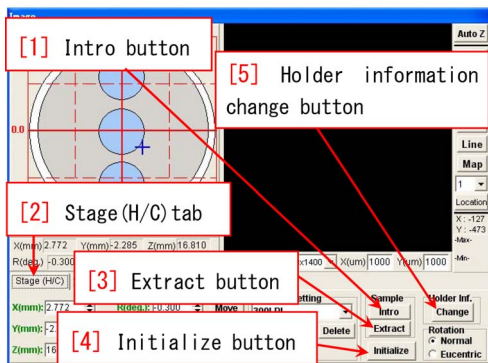


Figure 1.7: Image window. ‘Stage(H/C) tab’ has been clicked.

‘[1] Intro button’ in Fig. 1.7 should be clicked such that the sample stage moves to the introduction position. This situation can be observed on ‘[1] CCD Image’ window in Fig. 1.2[p.2]. After the sample stage moves to the introduction position, ‘Up[Z] button’ appears as shown in Fig. 1.8 However, do not click this button here.

### 1.1.11 Transferring the sample to the measuring chamber

After vacuuming the preparation (introduction) chamber sufficiently, ‘[4] Transfer Sample button’ in Fig. 1.2 [p.2] should be clicked to open V1 valve. Take care of the pressure in the measuring (main) chamber, when the V1 valve opens. If the pressure does not become higher than  $1.0E-4$ , there is no problem.

‘[2] Sample holder introducing grip’ in Fig. 1.9 should be slide slowly and carefully on the rod to move the sample holder from the preparation (introduction) chamber to the measuring (main) chamber watching the sample stage through ‘[1] Sample viewing window’ in Fig. 1.9. It should be confirmed that the sample holder is set on the sample stage. Here, ‘[2] Sample holder introducing grip’ in Fig. 1.9 should touch the black stopper ring on the rod. If the sample holder hits against the sample stage and cannot be set on the sample stage, please contact the manager (Kouhei OKITSU; 27470, 090-2203-8789). If the sample holder is set on the sample stage

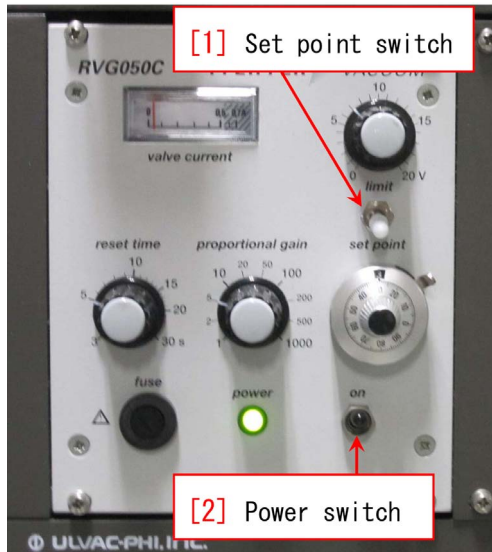


Figure 1.10: RVG050C unit.

in the measuring (main) chamber, ‘Up[Z] button’ in Fig. 1.8 should be clicked here. Now, it should be confirmed on the CCD Image window that the sample holder moves upward.

Next, ‘[2] Sample holder introduction grip’ in Fig. 1.9 should be subtracted completely to be stopped by the stopper ring on the rod. Here, it should be confirmed that only ‘[2] Sample holder introduction fork’ in Fig.1.4[p.2] is extracted. The sample holder should be set on the sample stage but not on ‘[2] Sample holder introduction fork’ in Fig. 1.4[p.2]. V1 valve is automatically closed with a clicking sound. Finally, ‘[5] Holder information change button’ in Fig. 1.7 should be clicked to select one-inch, two-inch or AR-XPS holder to select the sample thickness from a pull-down menu. For more detail about sample holders, refer to Appendix A[p.10], please.

## 1.2 Startup of the Ar ion gun

### 1.2.1 Verification of RVG050 unit for controlling the Ar gas flow

At first, it should be confirmed that ‘[1] Set point switch’ is set to be ‘set point’ (down) and ‘[2] Power switch’ is set to be off (down) on ‘RVG050C unit’ in Fig. 1.10.

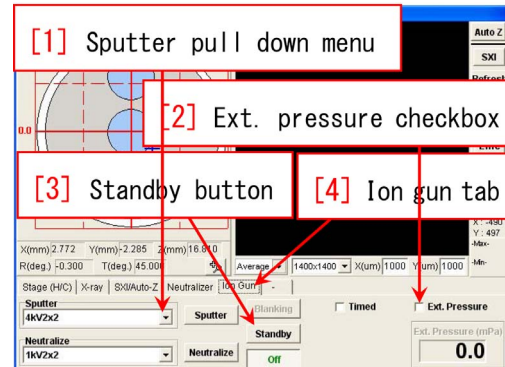


Figure 1.11: Image window. ‘Ion gun tab’ has been clicked.

### 1.2.2 Starting the differential vacuuming

‘[6] Diff vlv Open button’ in Fig. 1.2 [p.2] should be clicked to start the differential vacuuming by opening the V4 valve. It takes  $\sim 10$  sec after clicking ‘[6] Diff vlv Open button’.

Next, ‘[2] Ext. pressure checkbox’ should be checked to display the value of extractor pressure on the lower right corner of Fig. 1.11.

### 1.2.3 Standby of the Ar ion gun

‘[4] Ion Gun tab’ in Fig. 1.11 should be clicked such that a window as shown in Fig. 1.11 is displayed. Then, ‘Neutralize’ should be selected from ‘[1] Sputter pull-down menu’ and also from ‘Neutralize pull-down menu’. Next, ‘[3] Standby button’ should be clicked to be green. Finally, ‘[2] Ext. Pressure checkbox’ should be checked to display the extractor pressure on the right lower corner of the window shown in Fig. 1.11.

### 1.2.4 Adjusting the Ar gas flow

In Fig. 1.12 [p.6], after confirming that ‘[1] Leak valve’ (outer large knob only) and ‘[2] Ar cylinder valve’ (green knob) are completely closed, ‘[2] Ar cylinder valve’ (green knob) should be counterclockwise turned by  $180^\circ$  to be opened for  $1 \sim 2$  seconds and then should be completely closed.

Next, ‘[1] Leak valve’ (large knob only)

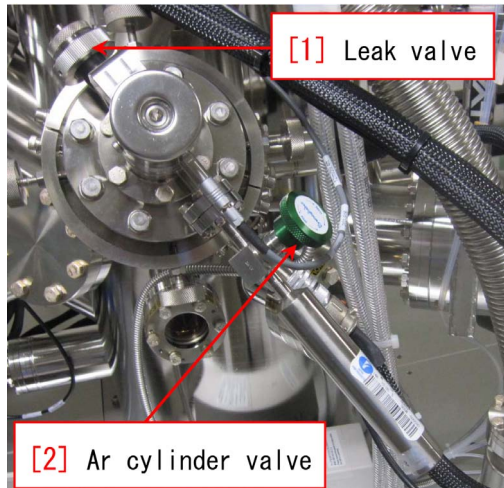


Figure 1.12: Ar cylinder and valves.

in Fig. 1.8 [p.4] should be turned counterclockwise to be opened slowly and carefully watching the values of the extractor pressure displayed on the right lower corner in Fig. 1.11 [p.5] and the pressure in the measuring (main) chamber displayed as '[8] Main chamber pressure' in Fig. 1.2 [p.2]. After turning '[1] Leak valve' in Fig. 1.12 by about two revolutions, the value of extractor pressure and/or '[8] Main chamber pressure' in Fig. 1.2 [p.2] increases. Then the extractor pressure should be adjusted at 32.5 mPa (tolerance: 32.0 ~ 33.0 mPa).

### 1.2.5 Switch on of RVG050C unit

'[2] Power switch' in Fig. 1.10 [p.5] should be on (up) to start controlling the Ar gas

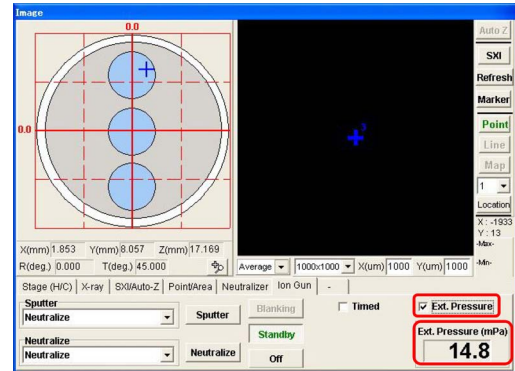


Figure 1.13: Ext.Pressure should be always monitored.

flow. The extractor pressure displayed on the right lower corner of Fig. 1.11 [p.5] decreases to be ~15 mPa after oscillating for a few minutes.

Now, the user can start the experiment.

During the experiment, '[2] Ext. Pressure checkbox' in Fig. 1.13 should be unchecked. If checking it during sputtering the sample with Ar ion, the extractor pressure may decrease. However, this causes no problem. However, after finishing the experiment, it should be checked again to verify that the extractor pressure reaches to ~15 mPa within a few minutes. If Ext.Pressure decreases extremely from 15 mPa, the Ar cylinder may be almost empty. In such a case, contact the manager (K. Okitsu; 27470, 090-2203-8789), please.

With regard to how to do experiment, refer to Part 1 manual, please.



## Chapter 2

# Important sequences after the experiment

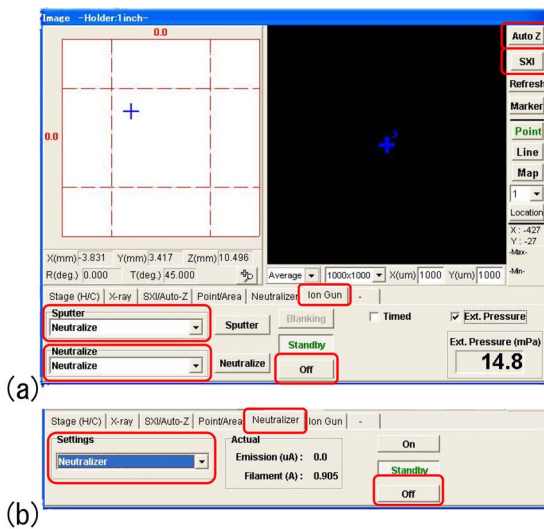


Figure 2.1: (a) ‘Ion Gun tab’ and (b) ‘Neutralizer tab’ has been clicked in the Image Window.

## 2.1 Shutdown of the Ar ion gun

### 2.1.1 Closing the leak valve

If ‘Standby button’ in Fig. 2.1(a) is green, check ‘Ext.Pressure check box’ (when not checked), please. Ext.Pressure should be verified to be  $\sim 15\text{mPa}$  to confirm sufficient Ar gas flow during the measurement.

1 h after finishing the measurement, ‘Standby button’ in Fig. 2.1(a) automatically changes to be black. In such a case, click it such as to be green again, please. Then, Ext.Pressure should be verified to increase to be  $\sim 15\text{mPa}$  within a few minutes.

If the value of Ext.Pressure is extremely

smaller than  $15\text{mPa}$ , the Ar gas cylinder may be empty. In such a case, contact the manager (K. Okitsu; 27470, 090-2203-8789), please.

The following procedure should be done carefully watching the value of the extractor pressure such as not to increase. Here, ‘Neutralize’ should be selected both from ‘Sputter pull-down menu’ and ‘Neutralize pull-down menu’ in Fig. 2.1(a).

‘[1] Leak valve’ in Fig. 1.12 should be turned clockwise to be closed. After closing it completely, open it by  $90^\circ$ .

Next, ‘[2] Power switch’ of RVG050C unit in Fig. 1.10 [p.5] should be off (down) to stop controlling the Ar gas flow. The user should wait for about 10 min to cool down the  $\text{Al}_2\text{O}_3$  crystal that controls the Ar gas flow.

After that, ‘[1] Leak valve’ in Fig. 1.12 should be completely closed. Here, even if the value of extractor pressure is  $0.3 \sim 0.4 \text{ mPa}$ , there is no problem.

### 2.1.2 Stop of the Ar ion gun

When finishing the experiment, ‘Off button’ below ‘Standby button’ in Fig. 2.1(a) should be clicked such that ‘Standby button’ changes to be black.

### 2.1.3 Stop of the electron neutralizer

In Fig. 2.1(b), ‘Neutralizer tab’ has been clicked to be opened in the Image Window.

1 h after finishing the measurement, ‘Standby button’ in Fig. 2.1(b) automatically comes to be black. In such a case, let it be as it is, please. If not, click ‘Off button’ below ‘Standby button’,

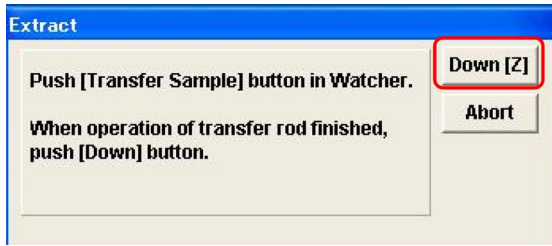


Figure 2.2: ‘Down[Z] button’ should not be clicked soon.

please such that ‘Standby button’ comes to be black.

### 2.1.4 Stop of the differential vacuuming

‘[3] Pump Intro button’ in Fig. 1.2 [p.2] should be clicked to start vacuuming the preparation (introduction) chamber. V4 valve for differential vacuuming automatically closes. Now, the procedure for shutting down the Ar ion gun has been completed.

## 2.2 Extraction of the samples

### 2.2.1 Start vacuuming the introduction chamber

The vacuuming of the introduction chamber has been started at the end of the previous process and should be continued for  $\sim 10$  min such that the pressure in the preparation (introduction) chamber becomes sufficiently low.

The switch of cooling-water circulator should not be turned off such that the stable generation of X-rays is supplied for the next user.

When the transfer vessel was used and the user wants to recover the sample in it, refer to Appendix B [p.14], please.

When it was not used or used but the user does not want to recover the sample in it, refer to the following sections, please.

### 2.2.2 Transferring the sample holder to the introduction chamber

After waiting for about 10 min, the sample holder can be transferred from the measuring (main) chamber to the preparation (introduction) chamber. ‘[3] Extract button’ in Fig. 1.7

[p.4] should be clicked to move the sample stage to the extract position watching carefully ‘CCD image’ window. ‘Down[Z] button’ appears as shown in Fig. 2.2 after the sample stage moves to the extract position. However, ‘Down[Z] button’ should not be clicked here.

Next, ‘[4] Transfer Sample button’ in Fig. 1.2 [p.2] should be clicked to open the V1 valve. When V1 valve is opened with a clicking sound, it should be confirmed that ‘[8] Main chamber pressure’ in Fig. 1.2 [p.2] is less than  $1.0\text{E}-4$  (less than  $1.0\text{E}-5$  is desirable).

‘[2] Sample holder introduction grip’ in Fig. 1.9 [p.4] should be slide slowly and carefully on the rod watching the sample stage through ‘[1] Sample viewing window’ in Fig. 1.9 [p.4]. It should be confirmed that ‘[2] Sample holder introduction fork’ in Fig. 1.4 [p.2] has been inserted to the sample holder. Here, ‘[2] Sample holder introduction grip’ in Fig. 1.9 [p.4] should touch the stopper ring. If ‘[2] Sample holder introduction fork’ in Fig. 1.4 [p.2] hits the sample stage and cannot be slide in, contact the manager (Kouhei Okitsu; 27470, 090-25203-8789).

Here, ‘Down[Z] button’ in Fig. 2.2 should be clicked to move the sample stage downward. This situation should be watched on ‘CCD Image’ viewer in Fig. 1.2 [p.2].

Next, ‘[2] Sample holder introduction grip’ in Fig. 1.9 [p.4] should be extracted slowly and carefully watching that the sample holder is on ‘[2] Sample holder introduction fork’ in Fig. 1.4 [p.2]. When ‘[2] Sample holder introduction grip’ in Fig. 1.9 [p.4] is completely extracted to touch the stopper ring, V1 valve automatically closes with a clicking sound.

When the transfer vessel was used and the user wants to recover the sample holder in it, refer to Appendix B §B.7 [p.19], please. If not, refer to the following sections, please.

### 2.2.3 Opening the valve of the $\text{N}_2$ cylinder

$\text{N}_2$  gas should be supplied from the  $\text{N}_2$  cylinder for purging the introduction chamber. It should be confirmed that ‘[1]  $\text{N}_2$  gas supplying valve’ in Fig. 1.3 [p.2] is not closed. If closed, it should be opened. ‘[5] 2nd-order pressure adjusting handle’ in Fig. 1.3 [p.2] should be turned coun-

terclockwise such that it can easily be turned. Next, ‘[4] 1st-order pressure adjusting wrench’ in Fig. 1.3 [p.2] should be turned counterclockwise by about  $5^\circ$  such that the pressure indicated in ‘[3] 1st-order pressure gauge’ in Fig. 1.3 [p.2] increases. Then, ‘[5] 2nd-order pressure adjusting handle’ should be turned clockwise (increase) slowly and carefully. the pressure indicated in ‘[2] 2nd-order pressure gauge’ should be adjusted to be 0.015 MPa.

#### 2.2.4 N<sub>2</sub>-purging of the introduction chamber

‘[5] Backfill Intro button’ in Fig. 1.2 [p.2] should be clicked to start purging the preparation (introduction) chamber. If the increase of the pressure in the preparation (introduction) chamber stops before reaching the atmospheric pressure (1.0E+5 Pa), ‘[5] 2nd-order pressure adjusting handle’ in Fig. 1.3 [p.2] should be turned clockwise (increase) slowly such that ‘[8] Intro chamber pressure’ reaches 1.0E+5 Pa. After finishing the purge, ‘[4] 1st-order pressure adjusting wrench’ in Fig. 1.3 [p.2] should be turned clockwise by about  $5^\circ$  to be completely closed. It should be confirmed that the pressure indicated in ‘[3] 1st-order pressure gauge’ in Fig. 1.3 [p.2] decreases to zero and then the pressure indicated in ‘[2] 2nd-order pressure gauge’ decreases to zero.

When the transfer vessel was used and the user does not want to recover the sample in it, refer to Appendix B §B.6 [p.18].

When it was not used, refer to the following sections, please.

#### 2.2.5 Extracting the sample holder

After finishing the N<sub>2</sub> purge, the sample holder can be extracted by removing the cap of the preparation (introduction) chamber. The samples should be removed from the sample holder and brought back to the user’s laboratory. The sample holder should be put back into the drawer of the desk.

‘[3] Pump Intro button’ in Fig. 1.2 [p.2] should be clicked to vacuum the preparation (introduction) chamber after putting the cap over the preparation (introduction) chamber to keep inside the chamber clean for the next user.

#### 2.2.6 Filling out in the experimental note

Fill in the pressure [Pa] of the measuring (main) chamber after the experiment. If the user had any trouble, write it in the experimental note, please.

Keep things on the desk tidy and in order, please.



# Appendix A

## Usage of the sample holders

In this chapter, the features and usages of [(a), (b), (c)] one-inch, (e) two-inch and (d) AR-XPS sample holders shown in Fig. A.1 are described.

For any kind of sample holder, the samples are usually fixed on it with a conductive carbon double sided tape with a width of 5 mm cut into a dimension about  $5 \times 5$  mm. It is not recommended to use too long size tape since it will be difficult to remove the samples after the experiment.

Figure A.2 shows a message window that appears just after mounting the sample holder on the sample stage. ‘Set button’ on the lower right corner of this figure should be clicked to set the kind of sample holder. As described before, ‘[5] Holder information change button’ in Fig. 1.7 [p.4] can also be clicked to set the holder information.

**AR-XPS holder can be used only for angle-resolved XPS (AR-XPS). It should not be used for another purpose. When doing AR-XPS using this holder, follow absolutely the usage described in Ap-**

**pendix A §A.3, please.**

### A.1 One-inch sample holder

Figures A.1(a), (b) and (c) show one-inch sample holders. The top surface of the sample should be lower than 7 mm upon the top surface of sample holder.

In the case of (b), a sample with a height less than 14 mm can be used since there is a contour bore with a depth of 7 mm.

Figure A.1 (c) is a one-inch holder designed for measurement with temperature controlled. Do not this holder for another purpose, please. For more detail, refer to Appendix E [p.24], please.

On the side of the one-inch sample holder, there are two grooves; upper and lower grooves. The upper groove is for grasping the holder with a jig. The lower groove is for inserting the holder to ‘[2] Sample holder introduction fork’ in Fig. 1.4 [p.2].

A communication error between the apparatus and the computer rarely happens. In many

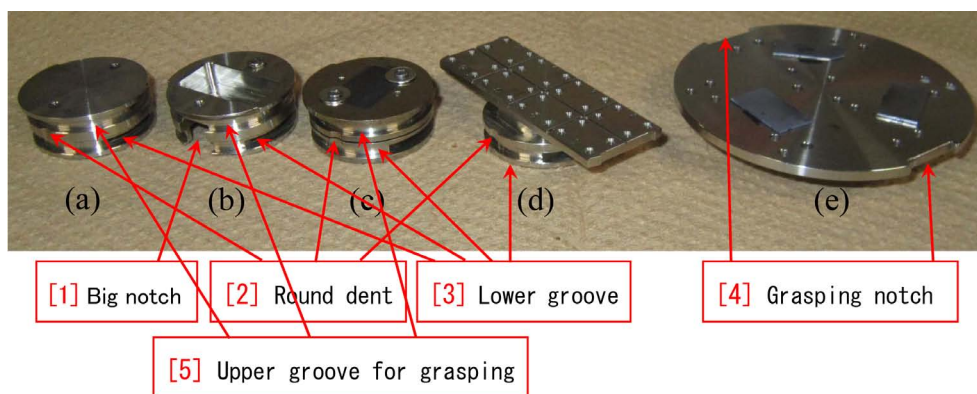


Figure A.1: Message that appears just after setting the sample holder.

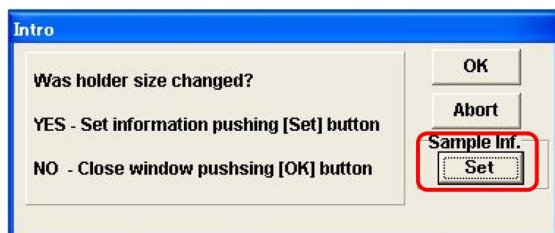


Figure A.2: Message that appears just after setting the sample holder.

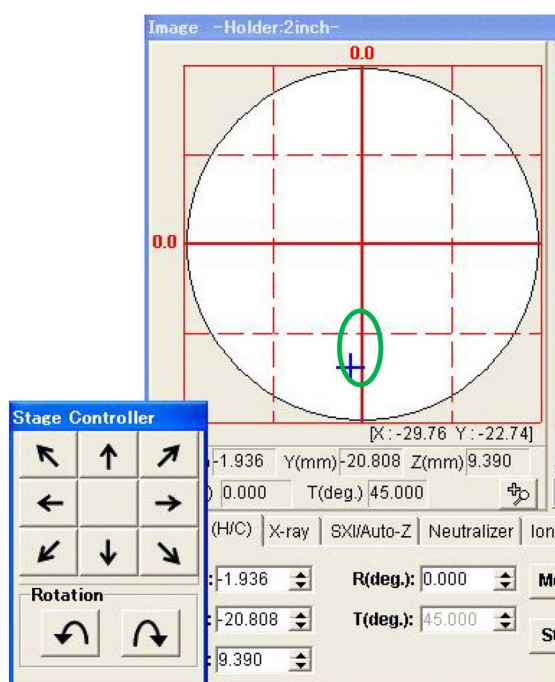


Figure A.3: In the case using the two-inch sample holder, the blue cross mark should be placed in the green elliptical region.

cases, the problem can be solved by initializing the sample stage clicking ‘[4] Initialize button’ in Fig. 1.7 [p.4]. However, this procedure can be done only when a one-inch sample holder is used. Further, all buttons on ‘[3] Stage controller’ in Fig. 1.6 [p.4] to translate and rotate the holder can be used. Then, this is the most recommended sample holder if all the samples to be measured can be mounted on it. The sample stage can be translated also by click&dragging a blue cross mark displayed in a round field in Fig. A.3.

Furthermore, angle-resolved XPS (AR-XPS) can also be done in a rage  $-4.0\text{mm} \leq Y \leq -2.0\text{mm}$  where  $Y$  is the vertical position on the

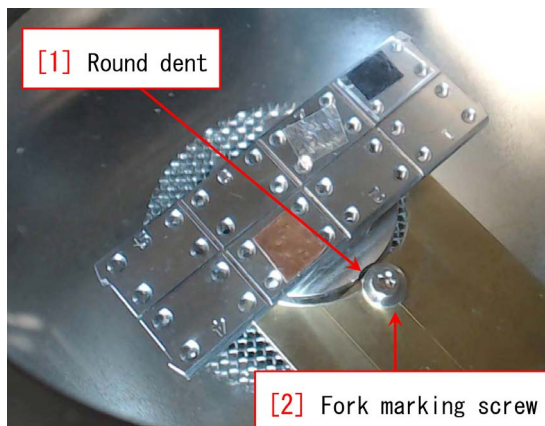


Figure A.4: A sample holder for AR-XPS in the preparation chamber.

image window as shown in Fig. A.3. Then, for any place whose distance from the central position of the one-inch sample holder is less than 2 mm, AR-XPS can be done.

## A.2 Two-inch sample holder

Figure A.1(c) shows a two-inch sample holder. This sample holder is most frequently used since many samples can be mounted on it. However, when using this sample holder, follow the following description, please.

Figure A.3 shows the stage controller and the left half of the image window. The blue cross mark (X-ray incident position) should be in a green elliptical region, that is to say, use only Y-translation buttons and rotation buttons to move the sample position, please.

A communication error rarely happens as described above. In the worst case, the sample holder may conflict ‘[1] Aperture of electron analyzer’ in Fig. A.5 [p.12] that may follow a serious accident. When the blue cross mark is in the green elliptical region, the central position of the sample holder is distant from just below the ‘[1] Aperture of electron analyzer’ in Fig. A.5 [p.12]. Therefore, the worst case can be avoided. However, put the sample such as to be over 5mm distant from the edge of the two-inch holder. This region is outside the movable range of the sample stage.

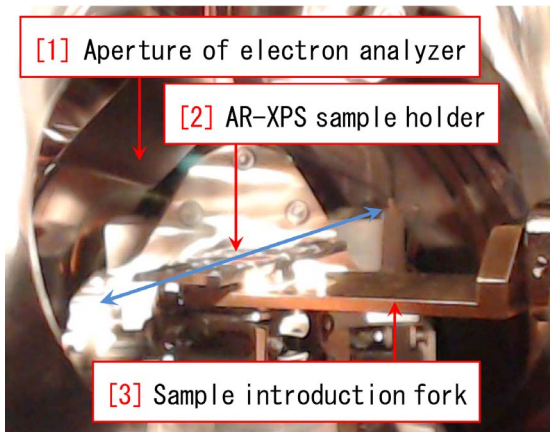


Figure A.5: A sample holder for AR-XPS has been inserted to the sample stage.

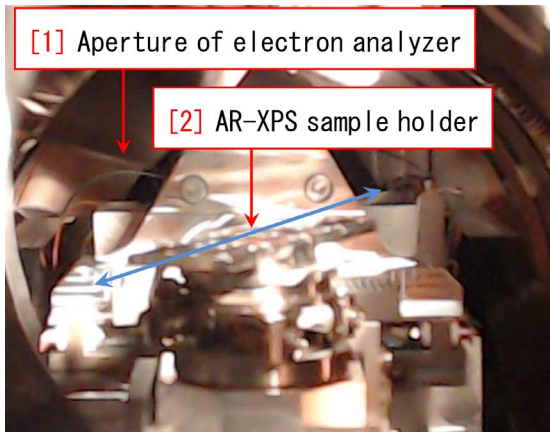


Figure A.6: The sample holder introduction fork has been extracted.

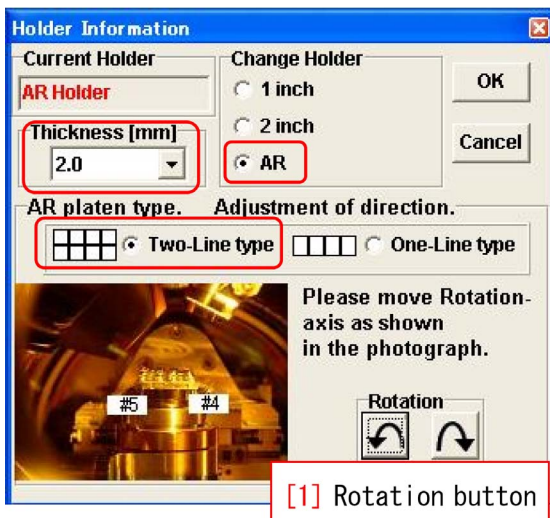


Figure A.7: Setting of the sample holder for AR-XPS.

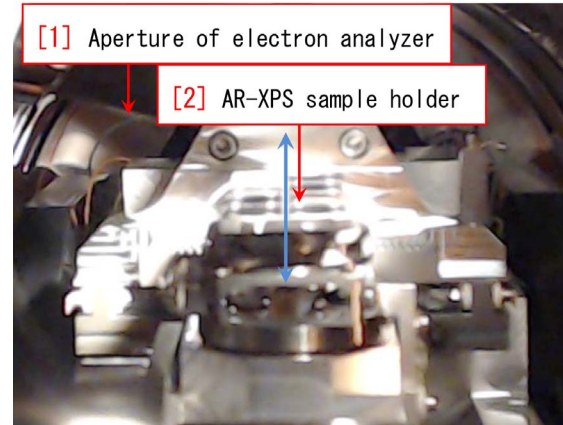


Figure A.8: The sample stage has been rotated such that the longitudinal direction (a blue arrow) of the holder is parallel to the viewing direction.

### A.3 Sample holder for AR-XPS

Figure A.4 [p.11] shows an AR-XPS sample holder in the preparation (introduction) chamber. The position of ‘[1] Round dent’ fits to that of ‘[2] Fork marking screw’. While there is a slight gap between the fork and the holder, it does not matter.

For translating the sample holder to the measuring (main) chamber, the sample stage should be moved to the intro position at first. Then, V1 valve between the preparation (introduction) and measuring (main) chambers should be opened. Figure A.5 shows a situation that an AR-XPS sample holder has been just inserted to the sample stage viewed through the window. In this figure, the longitudinal direction (shown as a blue arrow) of the sample holder is inclined from the viewing direction.

‘Up[Z] button’ on the computer display should be clicked to mount the sample holder on the sample stage. Next, the sample introduction fork should be extracted as shown in Fig. A.6 Then a message window as shown in Fig. A.2 [p.11] appears. Here, by clicking ‘Set button’, the kind of sample holder should be set.

Figure A.7 shows a window that appears just after doing the above procedure. The bottom half of this window has been displayed by checking ‘AR-XPS button’ to select the AR-XPS



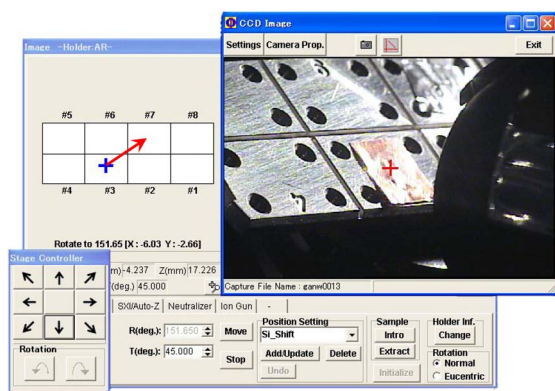


Figure A.9: The blue cross mark should be click&dragged to move from position #3 to #7.

holder. Setting the sample thickness is identical to the cases of one- and two-inch sample holders. ‘AR platen type’ should necessarily be set to be ‘Two-Line type’.

Then, ‘[1] Rotation button’ in Fig. A.7 should be clicked to rotate the sample holder such that the longitudinal direction of it (a blue arrow in Fig. A.6) becomes parallel to the viewing direction as shown in Fig. A.8. Here,  $R(\text{deg})$  displayed on the lower center of Fig. A.3 [p.11] should be about  $-28^\circ$ .

‘OK button’ on the upper right corner of Fig. A.7 should be clicked after adjusting the rotation angle. After that, the rotation buttons become gray and impossible to click.

Figure A.9 shows ‘CCD image’, ‘Image window’ and ‘Stage controller’ with a cascade style. X (horizontal) and Y (vertical) values of position have been adjusted and ‘Auto-Z’ has also been done such that the X-rays are incident approximately on the central position of

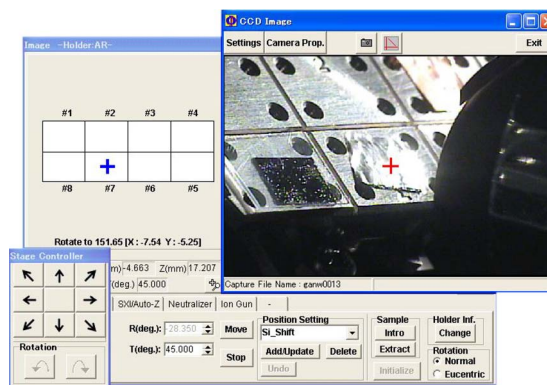


Figure A.10: The sample holder has been rotated by  $180^\circ$ .

the copper sample. This sample is placed at position #3. On positions on the left and upper of position #3, an upside-down number ‘4’ and number ‘6’ can be seen, respectively. The blue cross mark is in position #3.

An aluminum foil has been placed at position #7. To move the sample stage to position #7, the blue cross mark should be click&dragged to position #7 as shown as a red arrow in Fig. A.9

Once, the sample stage moves downward and then rotates by  $180^\circ$ . After that, it moves upward again such that the blue cross mark is on the aluminum sample at position #7 as shown in Fig. A.10. A more precise adjustment can be done by clicking XY translation buttons. Then, ‘Auto-Z’ should be done for adjusting the height of the sample stage.

**In Fig. A.7, ‘[1] Rotate button’ should not be clicked absolutely except when just mounting the AR-XPS holder on the sample stage at first, whereas it can be clicked.**

# Appendix B

## Usage of the transfer vessel

If the user's laboratory has a glove box, a transfer vessel can be used. The samples on the sample holder prepared and mounted in the glove box can be brought in the transfer vessel to the preparation (introduction) chamber of PHI5000 VersaProbe without exposing them in the atmosphere.

The usage of the transfer vessel is described in this chapter. Only a one-inch sample holder can be brought in the transfer vessel.

### B.1 Lending the transfer vessel

Figure B.1 shows items that should be brought from the room 330 in the 9th building of the faculty of engineering to the glove box in the user's laboratory, i.e. '[1] Transfer vessel' (in the lower right drawer of the desk), '[2] one-inch sample holder' (in the upper left drawer of the desk) and some length of '[3] Carbon double-sided tape' (in the upper right drawer of the desk). These items should be put into the glove box in the user's laboratory. Do not use other kinds of double-sided tape, please. They should be brought out wrapping in a new aluminum foil.

Before bringing out them, contact the manager (K. Okitsu; 27470, 090-2203-8789, okitsu[atmark]soyak.t.u-tokyo.ac.jp) by phone or E-mail. Also after returning them, contact him via E-mail, please.

### B.2 Setting the sample holder in the transfer vessel

The following procedure should be done in the glove box. Figure B.2 shows the situation when



Figure B.1: Transfer vessel, sample holder & double-sided tape.

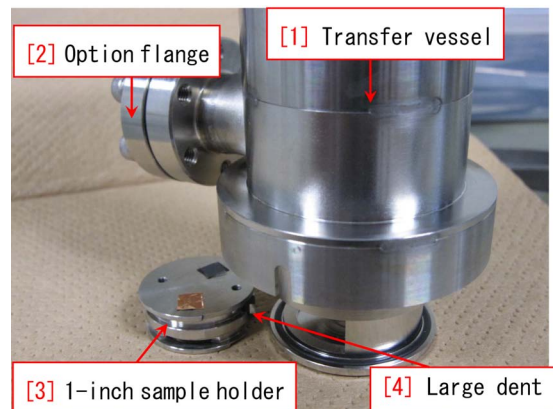


Figure B.2: Accommodation of the sample holder into the bottom of the transfer vessel.



Figure B.3: The bottom of the transfer vessel has been closed.

setting the sample holder on which the samples have been pasted into the bottom of the transfer vessel. Open and pull down the bottom of ‘[1] Transfer vessel’ by turning counterclockwise the knob on the top of the vessel, please. The height of the top surface of sample pasted with a double-sided tape should be lower than 5mm from the top surface of the sample holder. Put the sample holder under ‘[2] Option flange’ such that ‘[4] Large dent’ faces the vessel to set the sample holder on the bottom of the transfer vessel. Then, turn clockwise and close the knob on the vessel to accommodate the sample holder in the vessel as shown in Fig. B.3. Bring the sample holder in the vessel to room 330 of the 9th building, please.

## B.3 N<sub>2</sub>-purging the preparation chamber

### B.3.1 Verification of ‘Vacuum Watcher’

Before N<sub>2</sub>-purging the preparation chamber, verify that only valves V3 and V7 have been opened (green) and all other valves are closed (red), please.

### B.3.2 Operation of the regulator of N<sub>2</sub> cylinder

For N<sub>2</sub>-purging the preparation (introduction) chamber, dry N<sub>2</sub> gas should be supplied from the N<sub>2</sub> cylinder.

It should be verified in Fig. B.4 that both ‘[3] 1st-order pressure gauge’ and

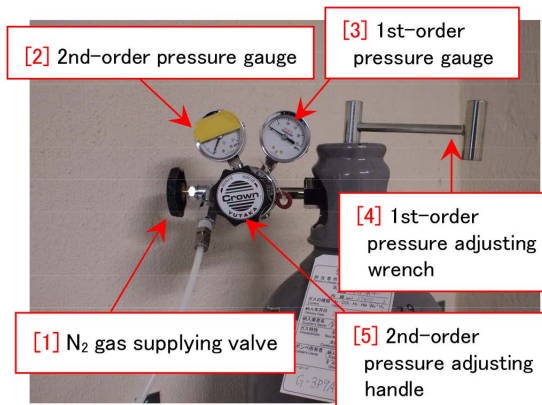


Figure B.4: Around the regulator of N<sub>2</sub> cylinder.

‘[2] 2nd-order pressure gauge’ indicate zero and that ‘[1] N<sub>2</sub> gas supplying valve’ is open. Then, the ‘[5] 2nd-order pressure adjusting handle’ should be turned counterclockwise (decrease) such that it can easily be turned. Next, ‘[4] 1st-order pressure adjusting wrench’ should be turned counterclockwise to open (about 5°). Next, turn ‘[5] 2nd-order pressure adjusting handle’ clockwise (increase) slowly and carefully, please such that ‘[2] 2nd-order pressure gauge’ indicates 0.015 MPa.

### B.3.3 N<sub>2</sub>-purging the preparation chamber

To N<sub>2</sub>-purge the preparation (introduction) chamber, please click ‘[5] Backfill Intro button’ in Fig. 1.2 [p.2]. V3 valve will be automatically closed, and then the V2 valve will be opened to start purging the preparation (introduction) chamber. If the value of the preparation chamber pressure shown on ‘[5] Vacuum gauge’ in Fig. 0 on the cover of this manual and/or ‘[9] Intro chamber pressure’ in Fig. 1.2 [p.2] stops before reaching the atmospheric pressure (1.0E+5 Pa), ‘[5] 2nd-order pressure adjusting handle’ in Fig. B.4 should be turned slightly clockwise (increase) slowly and carefully such that the pressure of the preparation (introduction) chamber reaches the atmospheric pressure (1.0E+5 Pa). After purging the preparation chamber, ‘[4] 1st-order pressure adjusting wrench’



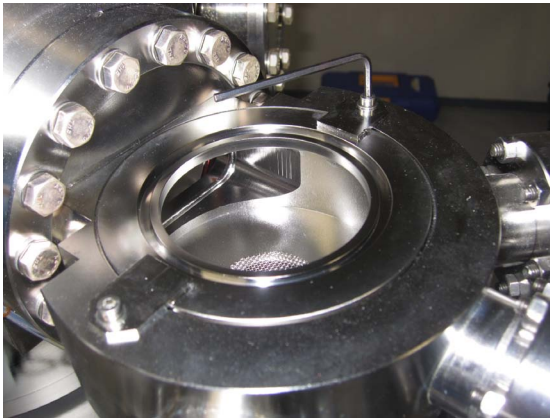


Figure B.5: The jig for the normal cap on the preparation chamber should be removed.

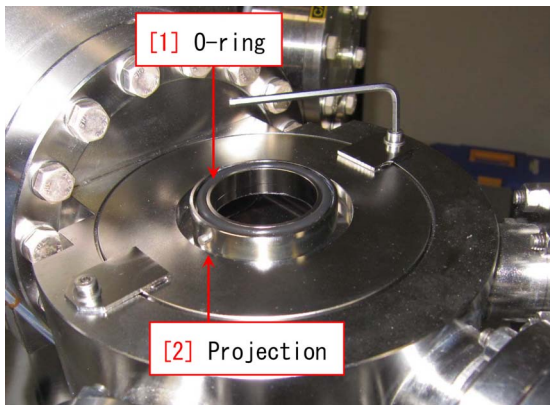


Figure B.6: The jig for the transfer vessel should be set on the preparation chamber.

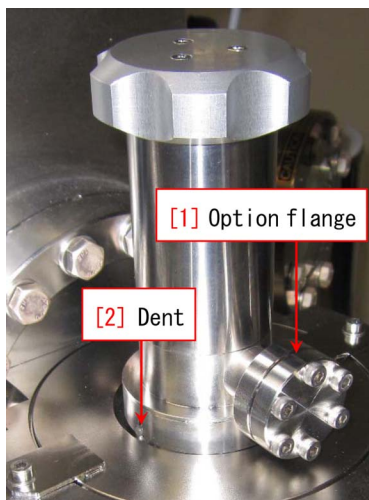


Figure B.7: The transfer vessel has been put on the preparation chamber.

in Fig. B.4 [p.15] should be turned clockwise to close. Please verify that the value in '[3] 1st-order pressure gauge' decrease to zero and then the value in '[2] 2nd-order pressure gauge' decrease to zero in Fig. B.4 [p.15]. The other knobs in Fig. B.4 [p.15] should not be touched here.

## B.4 Setting the transfer vessel

### B.4.1 Exchange of the jig on the preparation chamber

Figure B.5 shows the jig for the normal cap on the preparation chamber. Unfasten two screws with a 2.5mm hex wrench to remove it, please.

Figure B.6 shows a situation that the jig for the transfer vessel has been set on the preparation chamber. Two screws should be fasten with a 2.5mm hex wrench. Do not touch '[1] O-ring' with a bare hand, please.

### B.4.2 Setting the transfer vessel

Figure B.7 shows the transfer vessel set on the preparation chamber. '[1] Option flange' should face the near side such that '[2] Dent' fits '[2] Projection' in Fig. B.6.

## B.5 Transfer of the sample holder into the preparation chamber

### B.5.1 Vacuuming the preparation chamber

In this stage, 'Vacuum Watcher' should be as shown in Fig. B.8. Here, start vacuuming the preparation chamber by clicking 'Pump Intro button', please. About 15 min after starting to vacuum, a 'complete' message appears on the lower left corner of 'Vacuum Watcher'.

### B.5.2 Clicking 'Transfer vessel button'

In Fig. B.9, 'Transfer vessel button' has been clicked after vacuuming sufficiently the preparation chamber. All valves have been closed.



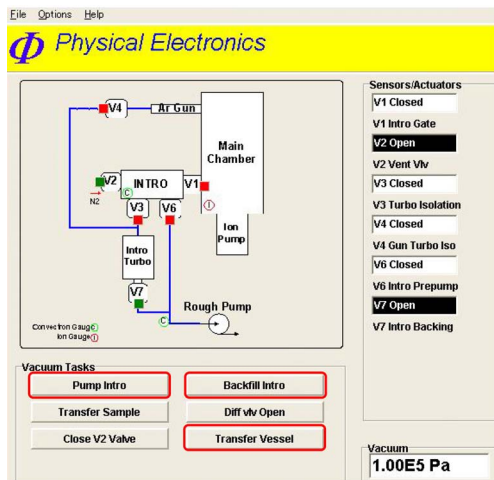


Figure B.8: Vacuum Watcher. The preparation chamber has been N<sub>2</sub>-purged.

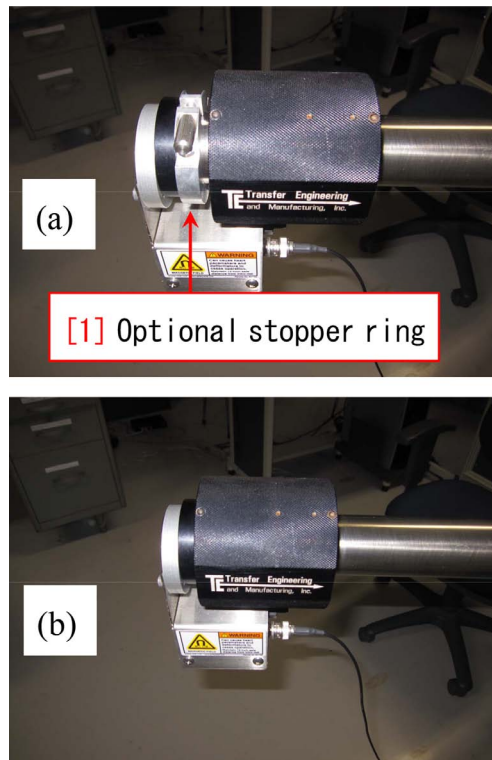


Figure B.10: Optional stopper ring should be removed to subtract the grip further.

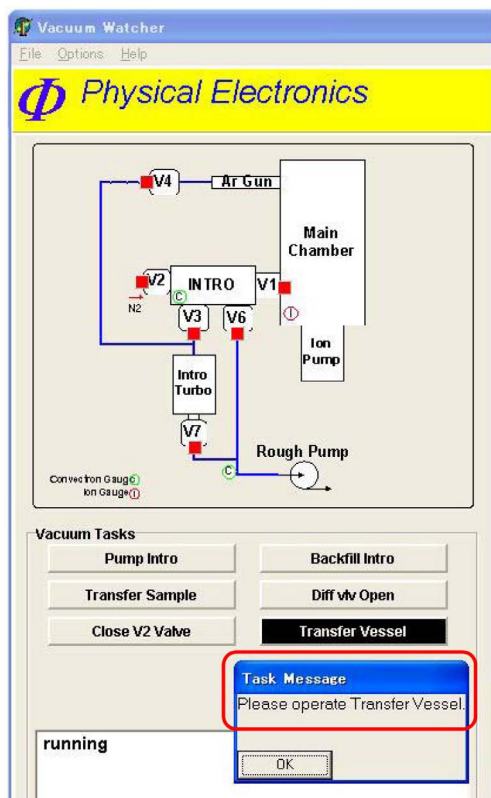


Figure B.9: Vacuum Watcher. Transfer Vessel button has been clicked.

A message ‘Please operate Transfer Vessel’ appears. However, do not click ‘OK button’, here.

### B.5.3 Removing the optional stopper ring

Here, the black sample introduction grip has been almost subtracted. However, it should be further subtracted to pull down the bottom of the transfer vessel in the preparation chamber. An aluminum ‘[1] Optional stopper ring’ in Fig. B.10(a) should be removed to subtract the black grip further.

Take care not such that ‘[1] Optional stopper ring’ is lost, please.

### B.5.4 Transfer of the sample holder to ‘sample introduction fork’

The procedure described in this section should be done viewing through the window of the preparation chamber by illuminating inside the chamber with a light (an emergency light hung inside the door of the room may be used. But do not forget to return it, please).

In Fig. B.11 the knob on the transfer vessel has been rotated counterclockwise and opened such that the height of the lower groove of ‘[2] Sample holder’ is identical to that of ‘[3] Sample introduction fork’. Here, the pressure in the preparation chamber has increased to  $1.0 \sim 7.0E+3$  Pa.

Insert ‘[3] Sample introduction fork’ to the lower groove of the sample holder, please. Next, the bottom of the vessel should be further pull down by turning counterclockwise the knob upon the vessel such that ‘[3] Sample introduction fork’ can be subtracted with the sample holder on it.

After transferring the sample holder to ‘[3] Sample introduction fork’, the knob upon the vessel should be turn counterclockwise to lift the bottom of the vessel and close it.

### B.5.5 Start vacuuming the introduction chamber

Click ‘OK button’ in Fig. B.9 [p.17], please such that the preparation chamber starts automatically being vacuumed.

After this procedure, refer to the description in §1.1.10 [p.3], please.

## B.6 Procedures after the experiment (when the samples can be disposed)

When the samples can be disposed after the experiment, do the following procedure, please, after the procedure described in §2.2.4 [p.9].

### B.6.1 Removing the transfer vessel

After  $N_2$ -purging the preparation chamber, the transfer vessel can easily be lift up to remove.

### B.6.2 Exchange of the jig upon the preparation chamber

From the situation in Fig. B.6 [p.16], remove the jig upon the preparation chamber by unfastening two screws with a 2.5 mm hex wrench. Next, put the jig for the normal cap on the

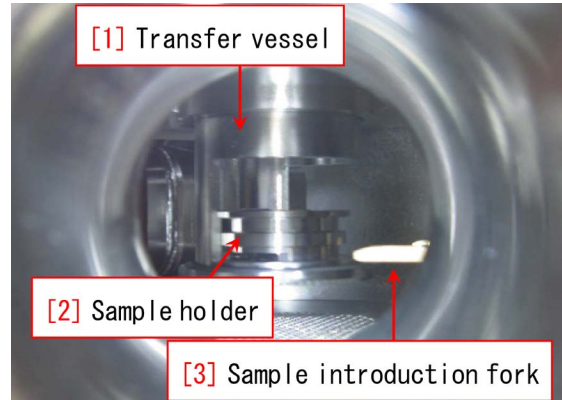


Figure B.11: The bottom of the transfer vessel has been pull down in the preparation chamber.

preparation chamber as shown in Fig. B.5 [p.16]. Fasten two screws with a 2.5 mm hex wrench, please.

The removed jig for the transfer vessel should be wrapped in a new aluminum foil and be returned to the lower right drawer of the desk.

The aluminum optional stopper ring should be attached again as shown in Fig. B.10(a) [p.17].

### B.6.3 Extraction of the samples

After extracting of the sample holder, retrieve the samples.

The transfer vessel should be wrapped in a new aluminum foil to return it to the lower right drawer of the desk. Return the one-inch sample holder into the upper left drawer of the desk, please.

After that, send an E-mail to the manager (K. Okitsu; okitsu [atmark] soyak.t.u-tokyo.ac.jp), please.

The samples should be brought back to the user’s laboratory.

### B.6.4 Filling out in the experimental note

Fill in the pressure [Pa] of the measuring (main) chamber after the experiment. If the user had any trouble, write it in the experimental note, please.

And keep things on the desk tidy

and in order, please.

## B.7 Procedures after the experiment (when the sample is to be used again)

If the samples are retrieved into the transfer vessel, they can be used again in another experiment. After the procedure in §2.2.1 [p.8], do the following procedure, please.

### B.7.1 Vacuuming the transfer vessel

Since the inside of the transfer vessel is just separated by an O-ring from the atmosphere, it may be polluted after long hours of the experiment. Then, turn counterclockwise the knob on the vessel slightly to open the bottom of the vessel such that it is vacuumed for about 15 ~ 20 min, please.

### B.7.2 Release of the bottom of the transfer vessel

Here, ‘optional stopper ring’ has been removed such that the black grip is completely subtracted as shown in Fig. B.10(b) [p.17].

The knob on the transfer vessel should be turned counterclockwise such that the bottom of it is pull down in the preparation chamber.

### B.7.3 Transferring the sample holder to the bottom of the transfer vessel

After pulling completely down the bottom of the transfer vessel, the sample introduction fork on which the sample holder is, can be inserted onto the bottom of the transfer vessel.

The knob on the transfer vessel should be turned clockwise to lift up the bottom of it such that the sample introduction fork can be subtracted as shown in Fig. B.11. next, the fork should be subtracted completely.

After that, turn further clockwise the knob on the transfer vessel to close it completely. Now, the sample holder on which the samples are mounted has been accommodated in the vacuumed transfer vessel.

### B.7.4 Verification of ‘Vacuum Watcher’

Before N<sub>2</sub>-purging the preparation chamber, verify that only valves V3 and V7 have been opened (green) and all other valves are closed (red) in Fig. B.8 [p.17], please.

### B.7.5 N<sub>2</sub>-purging the preparation chamber

To N<sub>2</sub>-purge the preparation (introduction) chamber, please click ‘[5] Backfill Intro button’ in Fig. 1.2 [p.2]. V3 valve will be automatically closed, and then the V2 valve will be opened to start purging the preparation (introduction) chamber. If the value of the preparation chamber shown on ‘[5] Vacuum gauge’ in Fig. 0 on the cover of this manual and/or ‘[9] Intro chamber pressure’ in Fig. 1.2 [p.2] stops before reaching the atmospheric pressure (1.0E+5 Pa), ‘[5] 2nd-order pressure adjusting handle’ in Fig. B.4 [p.15] should be turned slightly clockwise (increase) slowly and carefully such that the pressure of the preparation (introduction) chamber reaches the atmospheric pressure (1.0E+5 Pa). After purging the preparation chamber, ‘[4] 1st-order pressure adjusting wrench’ in Fig. B.4 [p.15] should be turned clockwise to close, completely. Please verify that the value in ‘[3] 1st-order pressure gauge’ decreases to zero and then the value in ‘[2] 2nd-order pressure gauge’ decreases to zero in Fig. B.4 [p.15]. The other knobs in Fig. B.4 [p.15] should not be touched here.

### B.7.6 Removal of the transfer vessel

After N<sub>2</sub>-purging the preparation chamber, the transfer vessel can easily be lift up and removed.

### B.7.7 Exchange of the jig upon the preparation chamber

From the situation in Fig. B.6 [p.16], remove the jig upon the preparation chamber by unfastening two screws with a 2.5 mm hex wrench. Next, put the jig for the normal cap on the preparation chamber as shown in Fig. B.5 [p.16]. Fasten two screws with a 2.5 mm hex wrench, please.

The removed jig for the transfer vessel should

be wrapped in a new aluminum foil and be returned to the lower right drawer of the desk.

The aluminum optional stopper ring should be attached again as shown in Fig. B.10(a) [p.17].

### **B.7.8 Filling out in the experimental note**

Fill in the pressure [Pa] of the measuring (main) chamber after the experiment. If the user had any trouble, write it in the experimental note, please.

Keep things on the desk tidy and in order, please.

### **B.7.9 Extraction of the samples**

The samples in the transfer vessel should be

retrieved in the glove box of the user's laboratory. Turn slowly and carefully counterclockwise the knob on the transfer vessel to open the bottom of it since the vessel has been vacuumed.

The retrieved samples can be used in another experiment.

### **B.7.10 Return of the transfer vessel and the sample holder**

The transfer vessel should be wrapped in a new aluminum foil to return it to the lower right drawer of the desk. Return the one-inch sample holder into the upper left drawer of the desk, please.

After that, send an E-mail to the manager (K. Okitsu; okitsu [atmark] soyak.t.u-tokyo.ac.jp), please.

*B.7. PROCEDURES AFTER THE EXPERIMENT (WHEN THE SAMPLE IS TO BE USED AGAIN)21*

To be continued.

## Appendix C

# Guide for sample preparation without using the transfer vessel

Because the sample surface is contaminated with organic materials by exposing it in the atmosphere, carbon 1s peak is necessarily found in the spectra. This carbon 1s peak disappears after sputtering briefly. When photoelectron peaks of carbon contained in the sample are measured, the user should make efforts to prevent the sample surface from carbon contamination.

The use of transfer vessel is recommended if the user's laboratory has a glove box. However, the user does not have it, the following way of sample preparation is recommended.

### C.1 Case of samples prepared with dry process *e.g.* vacuum deposition

Wrap the sample in an aluminum foil just after it is taken into the atmosphere, please. A new aluminum foil should necessarily be used.

### C.2 Case of samples prepared with wet process *e.g.* synthesis in a liquid

After washing the sample with acetone (or alcohol) and then with ion exchanged water, the water should be blown away with an air duster or a dry nitrogen gun. Avoid to use an air duster near a fire if it includes burnable gas, please. Just after blowing the sample, it should be wrapped with a new aluminum foil.

### C.3 common procedures for both dry and wet processes

Keep wrapping the samples until just before the measurement, please, to prevent them from contamination with organic carbon in the atmosphere. When mounting them on a sample holder and then vacuuming, do this procedure as quickly as possible, please.

## Appendix D

# Reset of the communication system

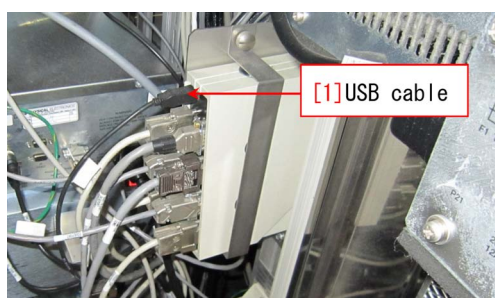


Figure D.1: The USB cable should be removed and inserted again

This chapter describes a procedure of recovery when a communication error occurs.

### D.1 Shut down of the program

Shut down all programs running on the computer at first, please. '[1] CCD image', 'Main menu' and 'Vacuum Watcher' in Fig.1.2 [p.2] can be closed by clicking '× button' on the right upper corner of the windows. If 'complete' message is displayed in the message window of 'Vacuum Watcher', it can also be closed. If any valve is opening or closing under the control of 'Vacuum Watcher', wait for a moment, please, until the operation of the valve finishes.

If any program is hung-up, shut down it by opening the task manager of windows by right-clicking the time display on the lower right corner of the display.

If the computer can not be shut down, please contact the manager (Dr. K. Okitsu; 27470, 090-2203-8789).

### D.2 Shut down of the computer

The computer cannot be shut down with the normal procedure due to its defect. After finishing all programs, it should be forcibly shut down by pressing the power switch for several seconds.

### D.3 Reset of the communication boards

Turn off '[6] Switch of the communication boards' in Fig. 0 on the cover of this manual, please, after shutting down the computer. Then, the back door behind the '[5] Vacuum gauge' in Fig. 0 on the cover of this manual should be opened. A black USB cable is found as shown in Fig. D.1. It should be removed and then inserted again. Next, turn on the '[6] Switch of the communication boards' in Fig. 0.

### D.4 Start up of the computer

After switching on the '[1] Control computer' in Fig. 0 on the cover of this manual, log in the windows with an account of Phiuser' with a password 'phiphi'.

### D.5 Start up of the control program

Restart the experiment by starting the control program referring to the description in §1.1.4 [p.1], please.



## Appendix E

# Measurement with the temperature controlled

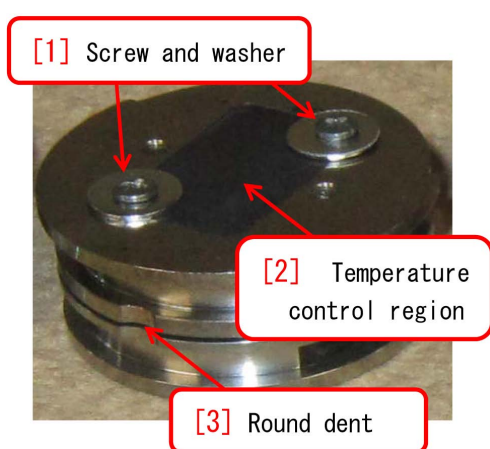


Figure E.1: Sample holder for temperature control.

**Liquid N<sub>2</sub> is necessary for the XPS measurement with the temperature controlled. The user should let the manager (Dr. K. Okitsu: 090-2203-8789, Okitsu[atmark]soyak.t.u-tokyo.ac.jp) know when the user want to do this kind of measurement at least a week before the experiment such that the manager can prepare liquid N<sub>2</sub>.**

### E.1 Abstract of the temperature control system

PHI5000 VersaProbe has a function to control the temperature of samples. The temperature range is  $-120^{\circ}\text{C}\sim 500^{\circ}$ .

Figure E.1 shows a sample fixed on the sample holder with screws and washers. Both-side tape should not be used in general since its thermal resistance is not confirmed.

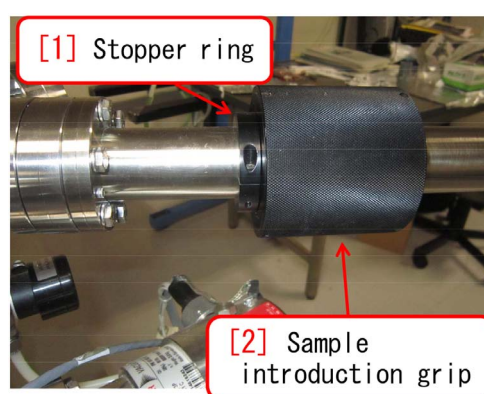


Figure E.2: Sample introduction grip should be pushed such that it touches the stopper ring completely.

The temperature can be controlled with ‘[8] Temperature control unit’ in Fig. 0 on the cover of this manual.

After doing the procedures described in §1.1.1 [p.1] - §1.1.10 [p.3], do the procedures described in the following sections, please.

### E.2 Introduction of the sample holder

The touch feeling when inserting the sample holder onto the sample stage is slightly tighter than the normal one-inch holder. It should be inserted such that the sample introduction grip completely touches the stopper ring completely.

After setting the sample holder in the measuring chamber, Ar ion gun should be started up referring to §1.2 [p.5].

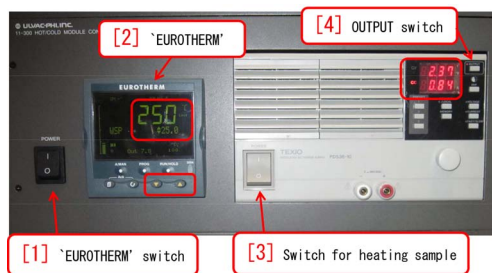


Figure E.3: HOT/COLD module

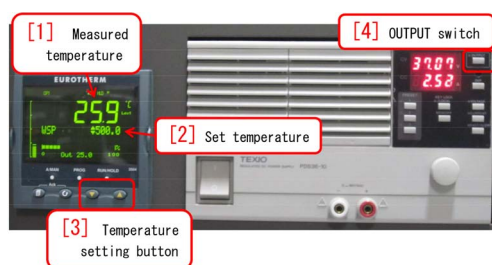


Figure E.4: Temperature is set at 500°C.

### E.3 How to set the temperature

Figure E.3 is a close-up of ‘[8] Temperature control unit’ of Fig. 0 on the cover of this manual.

At first, in Fig. E.3, ‘[1] EUROTHERM switch’ should be turned on. Here, do not turn on ‘[3] Switch for heating sample’, yet. After several messages are shown on the display of ‘[2] EUROTHERM’, As shown in a red frame of the figure of ‘[2] EUROTHERM’, measured temperature (upper large number) and set temperature (lower small number) are displayed.

Next, turn on ‘[3] Switch for heating sample’, please in Fig. E.3. In Fig. E.4, ‘[2] Set temperature’ has been adjusted to be 500°C by pressing ‘[3] Temperature setting button’. ‘[4] OUTPUT switch’ can be pressed to increase ‘[1] Measured temperature’ with 1°C/sec. When the temperature is increasing, the pressure in the measuring chamber increases due to emitted gas from the sample holder. Before the temperature increases over  $10^{-4}$ Pa, press ‘[4] OUTPUT switch’ again to stop applying the current to the sample holder, please. After the pressure in the main chamber decreases to be lower than  $10^{-5}$  Pa, ‘[4] OUTPUT switch’ should be pressed to apply the current again. By repeat-

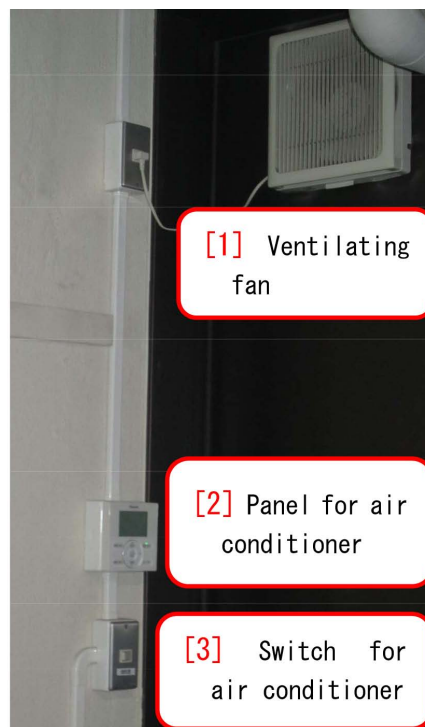


Figure E.5: Fan should be confirmed to ventilate.

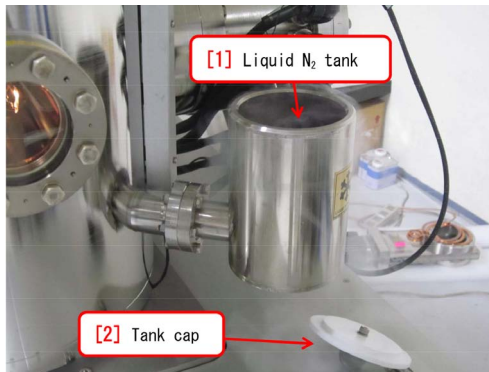
ing this procedure several times for  $\sim 3$  minutes, ‘[1] Measured temperature’ reaches to 500°C.

### E.4 Preparation of liquid N<sub>2</sub>

When using liquid N<sub>2</sub>, it should be confirmed necessarily that the fan on the left-hand back of room #330 is ventilating, referring to Fig. E.5.

Even when doing the experiment with a high temperature, liquid N<sub>2</sub> is necessary to cool the sample holder to room temperature after finishing the experiment.

The temperature can be set arbitrarily in a range of  $-120 \sim 500^\circ\text{C}$  by pressing ‘[3] Temperature setting button’ in Fig. E.4. When doing an experiment with a high temperature, the temperature is adjusted usually by keeping balance between heating with the heater and cooling with liquid N<sub>2</sub> while the temperature can sometimes be adjusted to be a high temperature without using liquid N<sub>2</sub>. In a case of low temperature, liquid N<sub>2</sub> is necessary for cooling the sample.

Figure E.6: Vessel of liquid N<sub>2</sub>Figure E.8: Dipper for liquid N<sub>2</sub>Figure E.7: Liquid N<sub>2</sub> tankFigure E.9: Dippers for liquid N<sub>2</sub>.

## E.5 How to supply liquid N<sub>2</sub>

Figure E.6 [p.26] shows ‘[1] Liquid N<sub>2</sub> dewar’ and ‘[2] Liquid N<sub>2</sub> vessel’. ‘[1] Liquid N<sub>2</sub> dewar’ can also be brought to room #330 from the user’s laboratory. It can also be prepared by the X-ray laboratory if the user contact the manager (K. Okitsu: 27470, 090-2203-8789, okitsu[atmark]soyak.t.u-tokyo.ac.jp) three days before the experiment.

In Fig. E.7, liquid N<sub>2</sub> has been supplied into ‘[1] Liquid N<sub>2</sub> tank’ from ‘[2] Liquid N<sub>2</sub> vessel’ in Fig. E.6. ‘[1] Liquid N<sub>2</sub> tank’ can be covered by ‘[2] Tank cap’ to prevent liquid N<sub>2</sub> from vaporization for four hours. However, when doing an experiment with a high temperature, it should be supplied more frequently. When doing an experiment with a low temperature, the sample can be kept to be an arbitrary temperature by keeping balance heating with the heater

and cooling with liquid N<sub>2</sub>.

## E.6 How to restore the sample to the room temperature

When retrieving the sample holder, it should necessarily be restored to room temperature for safety.

Before retrieving the sample, ‘[2] Set temperature’ in Fig. E.4 [p.25] should be set to be  $\sim 25^\circ\text{C}$ . After doing an experiment with a high temperature, a small quantity of liquid N<sub>2</sub> should be supplied to ‘[1] Liquid N<sub>2</sub> tank’ in Fig. E.7 for rapid cooling. After doing an experiment with a low temperature, residual liquid N<sub>2</sub> should be retrieved to ‘[2] Liquid N<sub>2</sub> vessel’ in Fig. E.6 by using ‘[1] Liquid N<sub>2</sub> dipper’ in Fig. E.8 or dippers as shown in Fig. E.9.

‘[1] Measured temperature’ in Fig. E.4 [p.25] should be  $25^\circ\text{C}$  even after turning off ‘[4] OUTPUT switch’ at last. Blowing inside the liquid N<sub>2</sub> tank with ‘[1] Dryer’ as shown in Fig. E.10,

is effective for removing the residual liquid N<sub>2</sub> rapidly.

### E.7 Procedure to return the sample holder back to the preparation chamber

Similarly to a normal experiment at room temperature, the sample can be recovered referring to chapter 2 [p.7]. Follow the description in §2.2.2 [p.8], please to recover the sample holder from the main chamber to the preparation chamber. When subtracting the sample holder from the sample stage, take care such that it is slowly subtracted from the sample stage since the subtracting feeling is slightly



Figure E.10: Inside the tank should be blown by a dryer.

tight compared with the normal one-inch sample holder.

To be continued.



## Appendix F

# Usage of Mg & Zr X-ray source

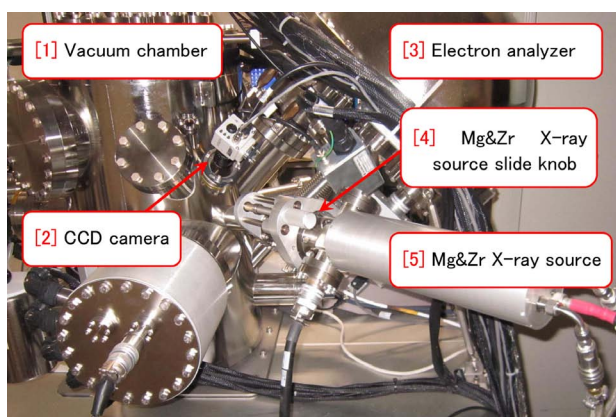


Figure F.1: Behind PHI5000VersaProbe

In addition to monochromatized Al  $K\alpha$  X-ray source (1486.6 eV), PHI5000 VersaProbe is equipped with Mg  $K\alpha$  (1253.4 eV) and Zr  $L\alpha$  (2042.4 eV) X-ray sources without monochromator. In those cases, however, only 1-inch sample holder can be used and only spectroscopy can be done.

In the XPS measurement, binding energy ( $E_B$ ) is estimated by an equation;  $E_B = h\nu - E_V$ . Here,  $h\nu$  is photon energy of X-rays and  $E_V$  is velocity energy of photoelectrons emitted from the surface of the sample. However, Auger electron peaks are also observed in the spectra. For example,  $KLL$  Auger electron is emitted when an  $L$  electron directly receives  $K-L$  energy difference from another  $L$  electron that transit to  $K$  energy level from which a photoelectron is emitted. The Auger transition is a transition process other than X-ray fluorescence. The velocity energy of Auger electron does not change even if changing X-ray source whereas it of photoelectron changes depending on the photon energy of incident X-rays. When a spectral overlap of photoelectron and Auger electron is

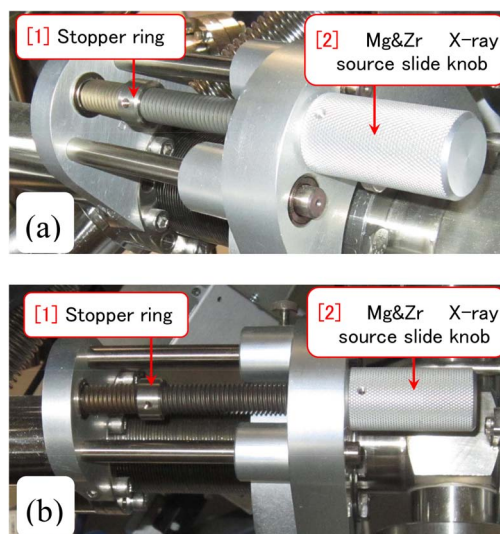


Figure F.2: Setting of ‘[2] Mg&Zr X-ray source slide knob’ in Fig. F.1 when using only Al X-ray source.

observed, it can be avoided by changing the X-ray source. For this purpose, Mg  $K\alpha$  (1253.4 eV) and Zr  $L\alpha$  (2042.4 eV) X-ray sources have been prepared.

## F.1 Preparation of Mg&Zr X-ray source

### F.1.1 Position and slide mechanism of Mg&Zr X-ray source

Figure F.1 is a back view of PHI5000 VersaProbe. Closeups of ‘[4] Mg&Zr X-ray source slide knob’ in Fig. F.1 are Figs. F.2 (a) and F.2 (b). When only Al X-ray source is used, ‘[2] Mg&Zr X-ray source slide knob’ in Fig. F.2 should necessarily be set like this.

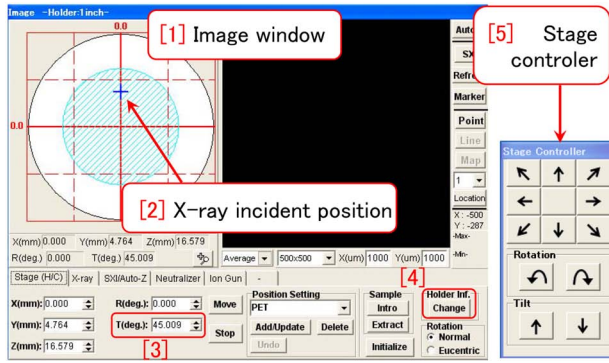


Figure F.3: Image window and stage controller

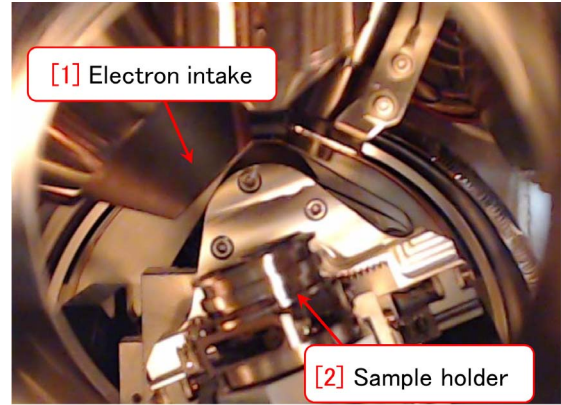


Figure F.5: Tilt angle is 60°.

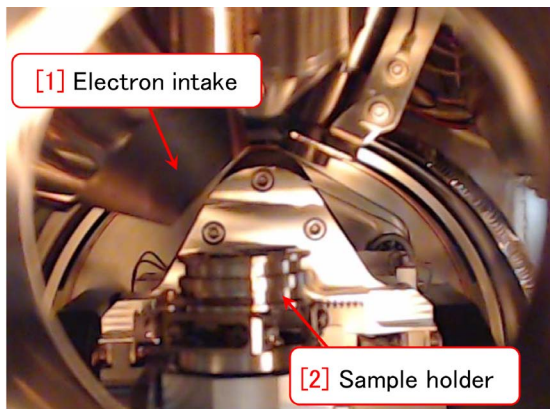


Figure F.4: Tilt angle is defined to be 45° when the surface of sample holder is horizontal.

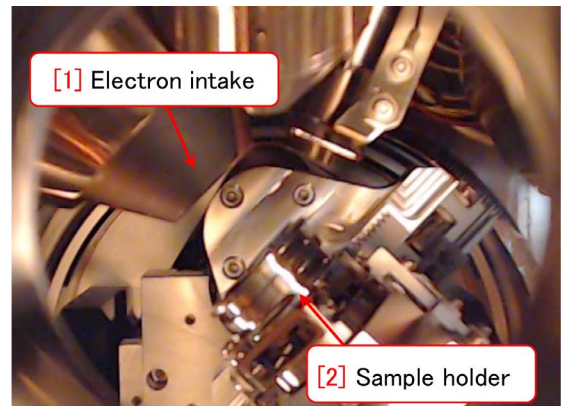


Figure F.6: Tilt angle should be 90° when using Mg&amp;Zr X-ray sources.

### F.1.2 Position adjustment of the sample stage

After setting a 1-inch sample holder in the main chamber referring to the descriptions in §1.1.2 [p.1] ~ §1.1.11 [p.4], it should be verified that 1-inch sample holder is correctly selected by clicking ‘[4] Holder Inf. Change button’ in Fig. F.3. The sample stage should be moved such that the blue cross mark indicating ‘[2] X-ray incident position’ is at the position in the upper half of circular region as shown in Fig. F.3. The sample stage can be moved by click&dragging the blue cross mark in Fig. F.3 or clicking arrow buttons on ‘[5] stage controller’ in Fig. F.3. Tilt buttons should not be clicked here.

When taking photoelectron spectra with the Mg&Zr X-ray sources, the incident position of X-rays indicated by the blue cross mark should

be on the upper half of the circular region in Fig. F.3. Arrow buttons and rotation button can be clicked to adjust the sample position. However, tilt buttons should not be clicked.

### F.1.3 Setting the tilt angle of sample stage to be 90°

Figure F.4 has been taken through the side window of the main chamber after the operations described above. Here, press [Enter] key after typing 90.0° in Fig. F.3 ‘[3] T(deg) text box’, please. Then, the sample holder starts tilting as shown in Fig. F.5 and stops at the situation as shown in Fig. F.6. Spectral measurement with Mg&Zr X-ray source should be done with the tilt angle of 90.0° like this.



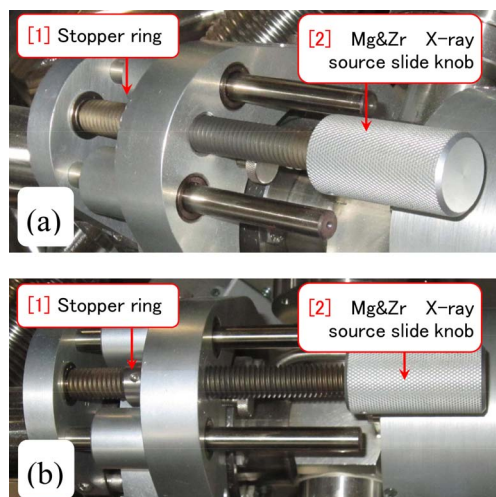


Figure F.7: Mg&Zr X-ray sources have been slid in (behind the VersaProbe).

#### F.1.4 Slide in of Mg&Zr X-ray source

Figure F.2 ‘[2] Mg&Zr X-ray source slide knob’ should be rotated counterclockwise such that it moves to stop at ‘[1] Stopper ring’ as shown in Fig. F.7 [p.31]. Figure F.8 is a closeup near the sample holder in the main chamber. Note that ‘[3] Mg&Zr X-ray source’ has been slid in whereas it was not observed in Fig. F.6 [p.30].

With this situation, spectral measurement can be done by using the Mg&Zr X-ray source.

## F.2 Shutdown of Mg&Zr X-ray source after the experiment

### F.2.1 Slide out of Mg&Zr X-ray source

After finishing the experiment with Mg&Zr X-ray source, ‘[2] Mg&Zr X-ray source slide knob’ in Fig. F.7 should be rotated clockwise such that the Mg&Zr X-ray source

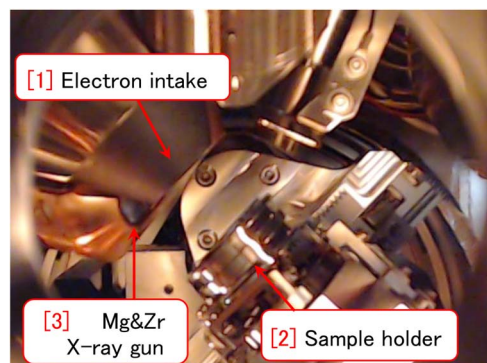


Figure F.8: Mg&Zr X-ray sources have been slid in (through the window).

slides out as shown in Fig. F.2 [p.29]. **It should be verified by viewing through the side window of the main chamber that it has been slid out as shown in Fig. F.6 [p.30].**

### F.2.2 Setting the tilt angle of sample stage to be 45° again

In Fig. F.3 [p.30], after typing 45.0° in ‘[3] T(deg)’ text box, press [Enter] key, please. Then, it should be verified that the surface of sample holder comes to be horizontal as shown in Fig. F.4 [p.30].

With this situation, measurement can be done by using Al X-ray source. To finish the experiment, refer to descriptions in the following sections, please.

### F.2.3 Shutdown of the ion gun

Shut down the Ar ion gun, please by referring to the descriptions in §2.1.1 [p.7] ~ §2.1.4 [p.8]

### F.2.4 Recovery of the sample

Finish the experiment, please, after recovering the samples referring to the descriptions in §2.2.1 [p.8] ~ §2.2.5 [p.9]

## Appendix G

# Initialization of the sample stage

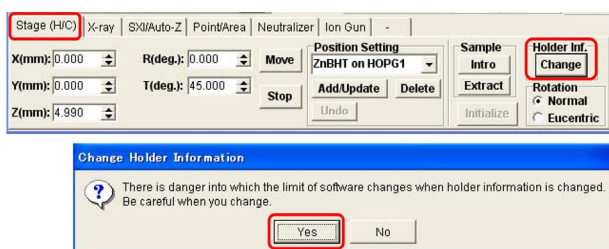


Figure G.1: Stage(H/C) tab has been opened in the image window.

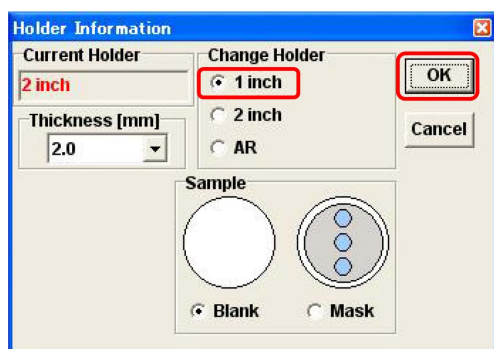


Figure G.2: Holder Information Window

When the sample stage has any trouble, it may be solved by initializing the sample holder. If the sample holder cannot be successfully set onto the sample stage in the main chamber, check that it has been not tilted or not rotated by 180°, at first, please.

Next, try to reset the communication system referring to the descriptions in Appendix D [p.23], please.

Even when the problem cannot be solved by doing the above procedure, try to initialize the sample stage referring to descriptions in the present chapter.

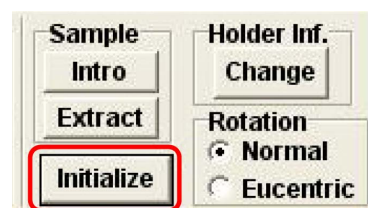


Figure G.3: Initialization of the sample stage

### G.1 Switch back of sample holder setting to 1-inch again

When 1-inch sample holder has been selected, procedure described in this section is not necessary. Go to the next section, please.

If 2-inch or AR-XPS sample holder has been selected, the setting of the sample holder should be temporarily changed to 1-inch holder. After initializing the sample stage, the setting should be returned such that it coincide with practically used sample holder. The maximum height of the samples should be less than 5 mm. If it is larger than 5 mm, please contact the manager (Kouhei OKITSU; 090-2203-8789)

In Fig. G.1, 'Stage(H/C) tab' on the upper left of the image window has been opened. The image window should be opened not in the macro mode but in the normal (acquisition) mode by clicking 'Acquire button'. At first,

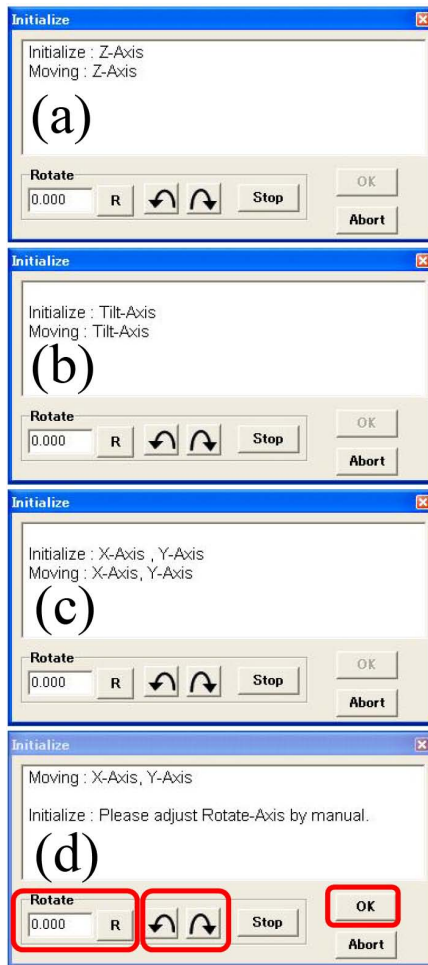


Figure G.4: Message during initializing the sample stage

‘Holder Inf. Change button’ on the right of Fig. G.1 should be clicked such that the bottom half of Fig. G.1. Click ‘OK button’ please to display Fig. G.2. After verifying ‘1-inch radio button’ checked, click ‘OK button’ on the upper right, please.

While ‘Initialize button’ on the right of Fig. G.1 is displayed to be gray and cannot be clicked, in Fig. G.3 it is displayed to be black and can be clicked. Click ‘OK button’, here, to start initialization of the sample stage.

### G.1.1 During initializing the sample stage

Figure G.4 shows messages displayed during the initialization of the sample stage. Initialization of (a) height (Z-axis), (b) tilt angle

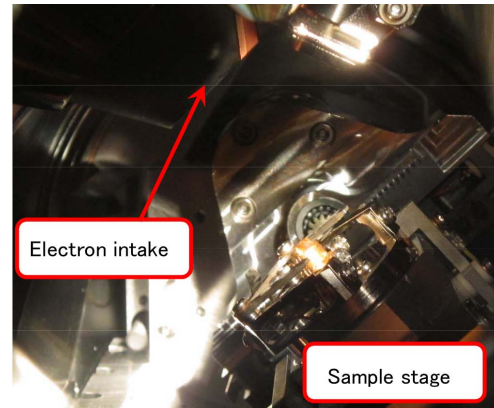


Figure G.5: Message during initializing the tilt angle

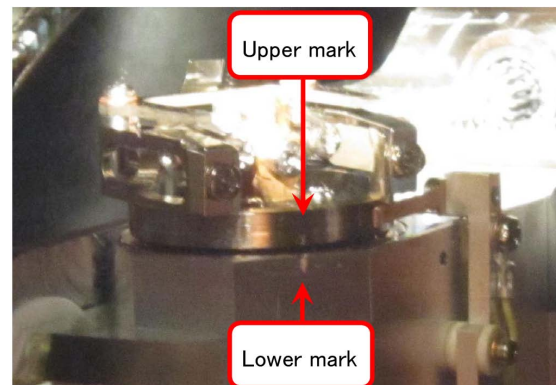


Figure G.6: Initialization of the rotation angle

and (c) horizontal position (X- and Y-Axes) are initialized automatically. However, in Fig. G.4 (d), rotation angle should be manually adjusted as described in the following section. Figure G.5 shows the sample stage viewed from the side window during initializing the tilt angle.

### G.1.2 Manual initialization of the rotation angle

After Fig. G.4(d) is displayed, the rotation angle should be manually initialized.

Figure G.6 is a photograph of the sample stage taken through side front window. The positions of ‘Upper mark’ and ‘Lower mark’ should finally coincide with each other completely.

Adjustment of the rotation angle can be done by typing rotation angle (deg) and then clicking

‘R button’ or by clicking round arrow buttons. Plus angle is clockwise rotation.

After verifying that ‘Upper mark’ and ‘Lower mark’ coincide with each other as shown in Fig. G.6 [p.33], ‘OK button’ on the lower right of Fig. G.4(d) [p.33] should be clicked to finish the

initialization of the sample stage.

After initialization of the sample stage, setting of the sample holder should be returned such that it coincides with the practically used sample holder by clicking ‘Holder Inf. Change button’ on the right of Fig. G.1 [p.32].

To be continued



## Appendix H

# Request of the remote assistance

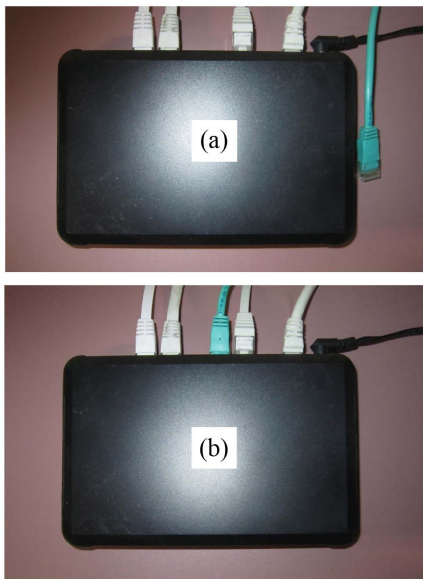


Figure H.1: Connection of LAN cable

Even when the manager (K. Okitsu) is absent on the weekend, he can remote-assist the user to operate the XPS by using the remote assistant function of the Windows. At first, call the manager by phone dialing 090-2203-8789, please. After the manager answer, what to do for the user is as follows.

### H.1 Connection of LAN cable

At first, a green LAN cable should be inserted to the LAN hub placed on the far left on the desk. A green LAN cable in Fig. H.1 (a) should be connected to the hub as shown in Fig. H.1 (b).

After finishing the remote assistance, the green LAN cable should be disconnected again as shown in Fig. H.1 (a).

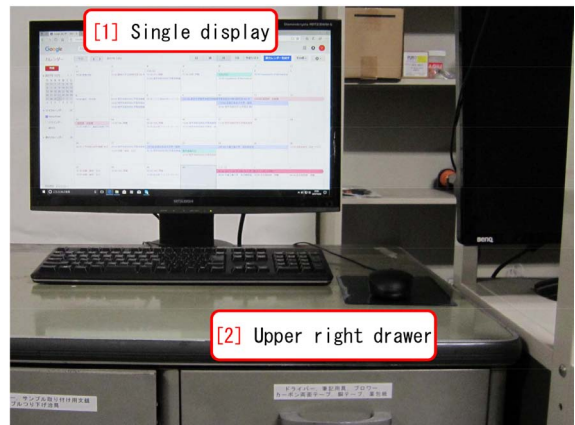


Figure H.2: The computer place on the left of the controlling computer

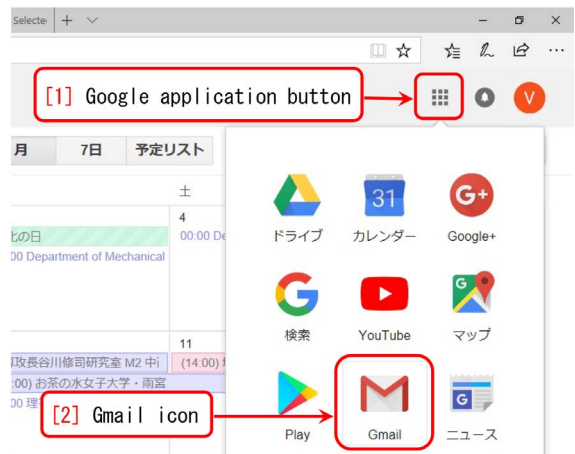


Figure H.3: Open the Gmail in the Google application

### H.2 Preparation of the Gmail

The computer that can be remote-assisted by the manager is that connected with the '[1] Single display' in Fig. H.2 placed on the left of the



Figure H.4: ‘Compose’ should be clicked to type a new e-mail

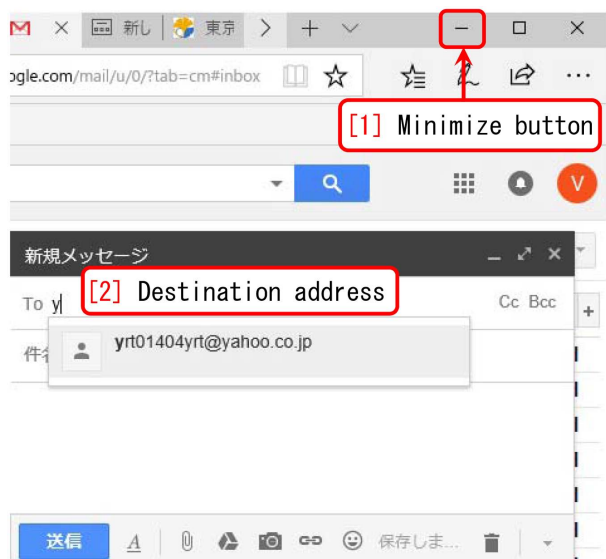


Figure H.5: Just ‘y’ can be typed to display the manager’s e-mail address

dual display.

At first, the internet browser should be opened to let the Google calendar for reservation to use the VersaProbe be shown. In Fig. H.3, the ‘[1] Google application button’ should be clicked to let the ‘[2] GMail icon’ be shown. It should be clicked to open ‘the composing window’ as shown in Fig. H.4. The red ‘[1] Compose button’ should be clicked to type a new e-mail.

In Fig. H.5, only one character ‘y’ can be typed in the ‘[2] Destination address’ to let ‘yrt01404yrt@yahoo.co.jp’ be shown. It should be clicked such that the e-mail is sent to the manager. Then, ‘[1] Minimize button’ should be clicked.



Figure H.6: ‘msra’ should be typed in the search text box.



Figure H.7: ‘[1] Invitation button’ should be clicked



Figure H.8: ‘[1] Save button’ should be clicked

### H.3 Preparation of the file and password of remote assistance

In Fig. H.6, ‘msra [1]’ should be typed in the search box on the lower left of the desktop of the computer. Then, ‘[2] msra’ (micro soft remote assistance) should be clicked to display Fig. H.7. ‘[1] Invitation button’ in it should be clicked to display Fig. H.8. In it, ‘[1] Save button’ can be

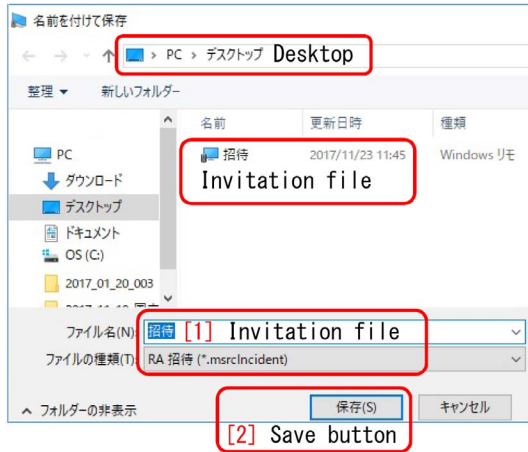


Figure H.9: ‘Invitation file’ should be on the desktop



Figure H.10: ‘[1] Yes button’ should be clicked to overwrite

clicked to let Fig. H.9 be displayed.

‘[1] Invitation file’ as displayed in Kanji characters should be saved on the desktop of the computer. In Fig. H.10, ‘[1] Yes button’ should be clicked to overwrite the file.

By saving the file of the remote assistance, the password is displayed as shown in Fig. H.11. After selecting it, it should be copied to the clipboard by typing [Ctrl]+[C]

## H.4 Sending the file and the password

In Fig. H.12, ‘[1] Browser icon’ is found on the task bar of the computer. It should be clicked to show ‘Compose window’ again as shown in Fig. H.13.

An arbitrary character(s) can be typed as the ‘[2] Title’. The password copied before should be pasted by typing [Ctrl]+[V] as the main body of the e-mail.

In Fig. H.13, ‘[4] File attach button’ can be



Figure H.11: ‘[1] Password’ should be copied to the clipboard

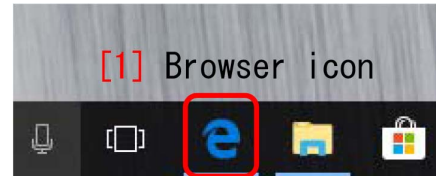


Figure H.12: ‘[1] Browser icon’ on the desktop

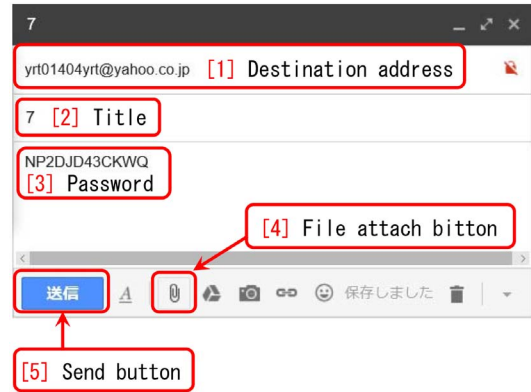


Figure H.13: Type an arbitrary character(s) as the ‘[2] Title’ and paste the ‘[3] Password’ by typing [Ctrl]+[V], please

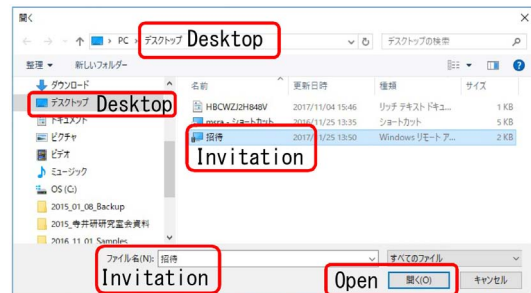


Figure H.14: The invitation file on the desktop should be selected and attached

clicked to open the window of Fig. H.14. The invitation file is found at the center of it. After selecting this, ‘Open’ on the lower right of Fig. H.14 should be clicked.



Figure H.15: ‘[1] Yes button’ should be clicked

## H.5 Starting the remote assistance

When the computer is connected to the manager’s computer with the remote assistance,

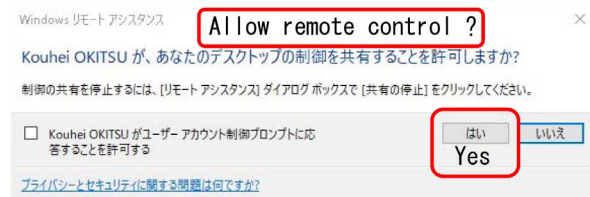


Figure H.16: ‘Yes’ to permit the remote control

Fig. H.15 is displayed. Here, ‘[1] Yes button’ should be clicked to display Fig. H.16. Here, ‘Yes’ should be clicked to permit the remote control.

Both the manager and the user can operate the mouse and keyboard together to operate the computer.

End of the document

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