

ULVAC PHI PHI5000 VersaProbe III

Part 2 Analysis Manual

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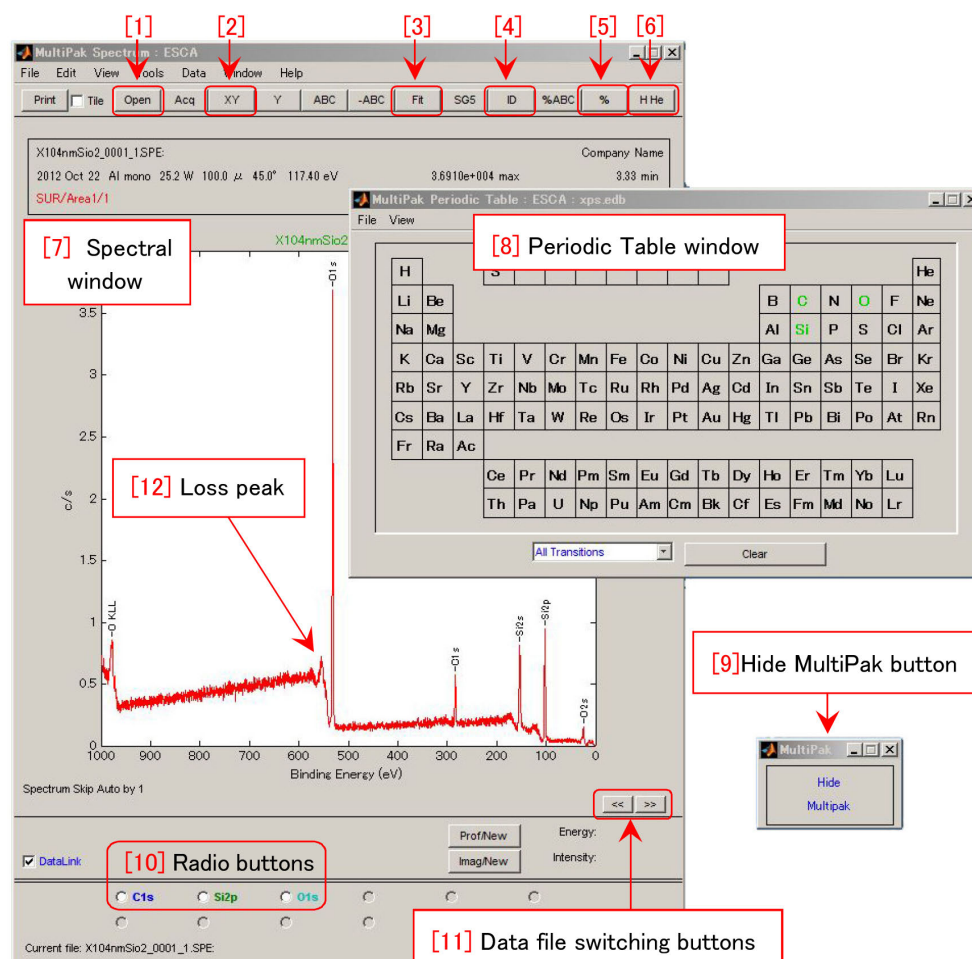


Figure 0: Initial window of 'MultiPak'.

Measured data by using PHI 5000 VersaProbe can be analyzed by a software 'MultiPak'. In this manual, the usage of 'MultiPak' is described.

'[9] Hide MultiPak' button can be clicked to hide 'Multipack' windows and to be 'Show MultiPak' button. This button can be clicked to show 'Multipak' window again. '[11] Data file switching buttons' can be clicked to change data file whose contents are displayed.

Frequently used buttons are as follows, '[1] Open' button to open file(s), '[2] XY' button to let the graph displayed with the original scale, '[3] Fit' button for curve fitting. '[4] ID' button to automatically assign photoelectron peaks. '[5] %' button for quantitative molar ratio evaluation and '[6] H He' button to display the periodic table of elements.

Appendix A [p.21] and Appendix B [p.23] describes about charge-up correction and identification of chemical state, respectively. In Appendix C [p.25], the usage of NIST data base is described.

Contents

1	Analysis of photoelectron spectra	1
1.1	Analysis of photoelectron spectra	1
1.1.1	Reading spectrum data	1
1.1.2	Assignment of wide scan spectrum	2
1.1.3	Quantitative analysis of elements from narrow spectra	2
1.1.4	Start of peak separation	3
1.1.4.1	Clicking ‘Fit’ button	4
1.1.4.2	Adjustment of background	4
1.1.4.3	Making fitting curves	4
1.1.4.4	Adjustment of fitting curves	4
1.1.4.5	Least square fitting	4
1.1.5	Refinement of peak separation	5
1.1.5.1	Fitting with Gaussian curves	5
1.1.5.2	Fitting with Gaussian and Lorentzian curves	5
1.1.5.3	Asymmetric fitting	5
1.1.6	Chargeup correction	6
1.1.7	Identification of chemical state	7
1.2	Quantitative evaluation of chemical states for different elements	7
1.2.1	Concerning PET (poly-ethyleneterephthalate)	7
1.2.2	Loading photoelectron spectra	7
1.2.3	Peak separation and quantative analysis of C1s spectrum	7
1.2.4	Addition of peak separation and quantitative analysis of the O1s spectrum	8
1.3	Loading and drawing of the fitting curve	10
1.3.1	Loading of separated fitting curves on the ‘MultiPak’	10
1.3.2	Export of the separated fitting curves into ascii files	11
1.3.3	Loading and graph-drawing of the separated fitting curves	11
2	Analysis of sputtering spectrum data	13
2.1	Analysis of sputtering spectrum data	13
2.1.1	Display of selected narrow spectra	14
2.1.2	three-dimensional display of sputtering spectrum data by using montage viewer	14
2.1.3	Chargeup correction	16
2.1.4	Identification of chemical state	16
3	Analysis of mapping data	17
3.1	Reading a map data file	17
3.2	Analysis of mapping data from a single peak	17
3.3	Analysis of mapping data obtained for plural peaks	18
3.3.1	Extraction of a map by setting a spectral range	18

3.3.2	Extraction of a map by selecting a region in the map	19
A	About charge-up shift correction	21
A.1	Charge-up shift correction	21
A.1.1	Abstract of charge-up shift correction	21
A.1.2	Charge-up shift correction using contamination carbon	21
B	Identification of chemical state	23
B.1	Identification of chemical state	23
B.1.1	Abstract of chemical-state identification	23
B.1.2	Start of chemical state assignment	23
B.1.3	Setting the range of spectral data	24
B.1.4	Identification of chemical state	24
C	Usage of NIST(National Institute of Standards and Technology) database	25
C.1	Access to NIST database	25
C.2	Usage of NIST database	27
C.2.1	How to search data in NIST URL	27
C.2.2	An example searching Si2p peak	27
C.2.3	Sorting the data	27

List of Figures

0	Initial window of ‘MultiPak’.	i
1.1	File selection window	1
1.2	Narrow spectra.	2
1.3	Default background (a) should be changed as (b).	2
1.4	Incorrect result of quantitative analysis due to selection of both Al2s and Al2p peaks.	2
1.5	By clicking ‘ [1] Al’ (a) in the periodic table with Shift key pressed, ‘Transition window’ (b) has been displayed.	3
1.6	Quantitative analysis of elements from C1s, O1s and Al2p peaks.	3
1.7	Quantitative analysis of elements from C1s, O1s and Al2s peaks.	3
1.8	Start of curve fitting.	4
1.9	Curve fitting, setup window.	4
1.10	Curve fitting, Band limit setting window.	6
1.11	Molecular structure of PET(from Wikipedia)	6
1.12	Narrow spectra measured for PET	6
1.13	C1s photoelectron spectrum for PET	7
1.14	Curve fitting for the C1s spectrum	7
1.15	Peak separation of C1s spectrum	8
1.16	Export of the spectra	8
1.17	Chemical state ratios of the C1s spectrum	8
1.18	Chemical state ratios of carbon and oxygen	8
1.19	Curve fitting of O1s spectrum	9
1.20	Peak separation of the O1s spectrum	9
1.21	File name specifying window	9
1.22	Molar ratio of chemical states of carbon and oxygen	9
1.23	Molar ratio of chemical states of carbon and oxygen	10
1.24	File opening window of the ‘MultiPak’	10
1.25	Folder select window of the ‘MultiPak’	10
1.26	Spectra of O1s and C1s have been loaded	10
1.27	C1s spectrum separated into four curves	11
1.28	File name specifying window	11
1.29	Spectrum load and drawn as a graph on the Excel	11
2.1	File selection window	13
2.2	Spectrum window and profile window	14
2.3	Si2p narrow spectrum.	14
2.4	‘Select spectra’ has been selected from ‘Tools menu’.	15
2.5	Spectrum selection scroll bar has been displayed.	15
2.6	Each spectrum has been displayed.	15
2.7	‘Montage viewer’ in ‘Tools’ menu can be clicked.	16

2.8	Settings of the ‘Montage viewer’.	16
2.9	Spectrum obtained by repeating sputtering can be three-dimensionally displayed.	16
3.1	File type selection window.	17
3.2	File open window.	17
3.3	Spectrum window and map window. Map by a single Si2p peak of elemental silicon.	17
3.4	Map by a single Si2p peak of SiO ₂ .	18
3.5	Mapping data file with plural peaks has been opened.	18
3.6	A spectral range for SiO ₂ peak has been selected from a range including two peaks.	18
3.7	An extracted map for SiO ₂ peak.	18
3.8	A spectral range for elemental silicon has been selected from a range including two peaks.	19
3.9	An extracted map for elemental silicon.	19
3.10	Extraction of maps for SiO ₂ and elemental silicon by selecting region.	19
3.11	A spectrum extracted from selected region.	19
3.12	In Fig. 3.10, ‘[3] Exit’ button has been clicked.	20
3.13	The color of map for elemental silicon has changed to be red.	20
3.14	A map displayed with different colors for SiO ₂ and elemental silicon.	20
A.1	Startup of the charge-up shift correction menu.	21
A.2	Charge-up correction window.	21
B.1	Startup of the chemical state identification menu.	23
B.2	Spectral peak assigned to SiO ₂ .	23
B.3	Spectral peak assigned to p-type silicon.	23
C.1	Input ‘XPS’ and ‘NIST’ as key words in google	25
C.2	Click ‘NIST XPS Database’ found as the first candidate	25
C.3	Click ‘Search Menu’.	26
C.4	Click ‘Retrieve Data for a Selected Element’.	26
C.5	Click ‘Go to Step 2’ with ‘Binding Energy’ checked.	26
C.6	Click an elemental symbol in the periodic table.	26
C.7	Click ‘Search’ with a photoelectron peak checked.	26
C.8	Spectral peak position of Si2p photoelectron for elemental silicon.	26
C.9	Authors and journal are found in the red frame.	26
C.10	Spectral peak position of Si2p photoelectron for SiO ₂ .	27
C.11	‘Click’ in Fig. C.10 has been clicked to display the authors and journal.	27
C.12	‘[1]Spectral Line’, ‘[2]Formula’ or ‘[3]Energy(eV)’ can be clicked to sort the data.	27

Chapter 1

Analysis of photoelectron spectra

1.1 Analysis of photoelectron spectra

1.1.1 Reading spectrum data

‘[1] Open’ button on the upper left corner of Fig. 0 on the cover of this manual, can be clicked to display Fig. 1.1.

At first, ‘[3] Browse’ button on the lower left corner of Fig. 1.1 should be clicked to open ‘[5] Folder Select’ window. After selecting a folder in this window, by clicking ‘[6] OK’ button, data files whose extension has been selected from ‘[2] List Files of Type:’ pull-down menu, are displayed in ‘[1] File Name’ box on the upper left corner of Fig. 1.1. ‘*.spe’ should be selected from ‘[2] List Files of Type:’ pull-down menu to display spectrum data file(s). Exten-

sions of other kinds of data files are as follows, ‘*.pro’: spectrum data measured with sputtering, ‘*.lin’: line profile data, ‘*.map’: mapping data, ‘*.sxi’: SXI (scanning X-ray imaging) data, etc. From ‘[1] File Name’ box, a data file can be clicked to be selected. Plural data files can also be selected by clicking them with [Shift] or [Shift] key pressed. ‘[4] OK’ button can be clicked to open files.

Radio buttons in the red frames of ‘[7] Spectrum Display:’ and ‘[8] Profile Display:’ can be checked to select the opening mode of ‘Spectrum Display Window’ and ‘Profile Display Window’: ‘New’ for displaying new one(s) on a new window, ‘Replace’ for displaying new one(s) on the current window replacing with new one(s), ‘Overlay’ for overlaying new one(s)

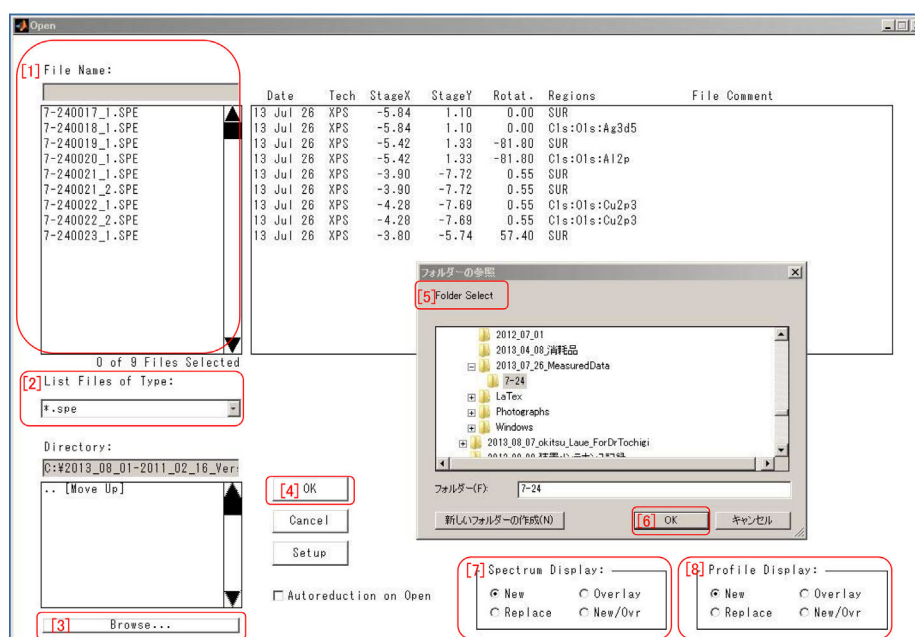


Figure 1.1: File selection window

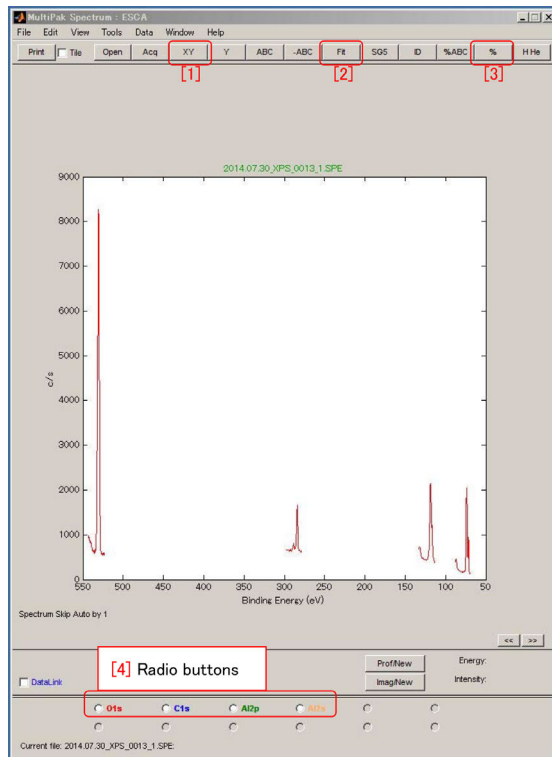


Figure 1.2: Narrow spectra.

on the current window and ‘New/Ovr’ for overlaying new ones on a new window, can be checked.

1.1.2 Assignment of wide scan spectrum

After opening a wide scan (survey scan) spectrum data file, ‘[4] ID’ button in Fig. 0 can be clicked automatically to assign the photoelectron peaks. O2s peak that was not assigned only by clicking ‘[4] ID’ button, has been assigned by clicking ‘O’ in the periodic table twice. Elemental symbol in the periodic table can be clicked again in general to assign other peaks that were not assigned only by clicking ‘[4] ID’ button in Fig. 0.

For assigning peaks that were assigned even with the above procedure in Fig. 0, they can be manually assigned with the following procedure. At first, from the left end to the right end of energy range around the spectral peak to be closed-up should be left-click&dragged horizontally to enlarge it. Then, around the peak to be assigned should be right-click&dragged horizontally such that candidates of spectral peaks

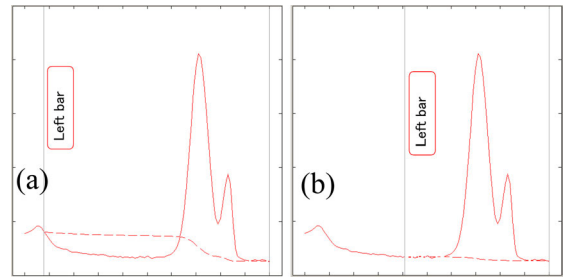


Figure 1.3: Default background (a) should be changed as (b).

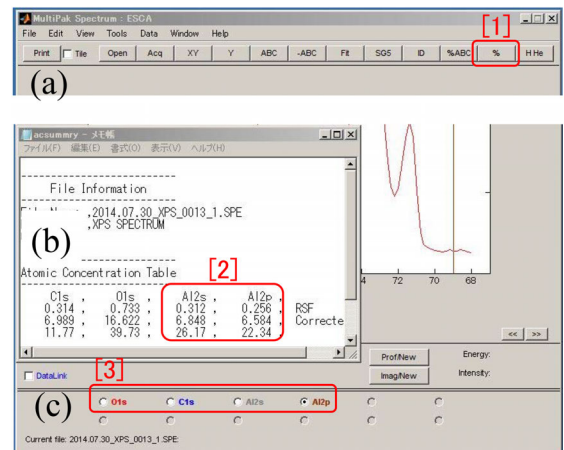


Figure 1.4: Incorrect result of quantitative analysis due to selection of both Al2s and Al2p peaks.

are displayed next to next. After assigning the largest peak, smaller peaks due to the same element can be assigned by clicking the elemental symbol twice in the periodic table in Fig. 0.

1.1.3 Quantitative analysis of elements from narrow spectra

In Fig. 1.2, a file of measured narrow spectra is opened. ‘[4] Radio buttons’ of measured spectral peaks in Fig. 1.2 can be checked to enlarge one of them. The enlarged graph can be redrawn with the original scale by unchecking the radio button or clicking ‘[1] XY’ button in Fig. 1.2. Quantitative analysis of molar ratios of elements can be done by clicking ‘[3] %’ button. Before that, however, the background level displayed with a red dashed curve should be adjusted shown in Fig. 1.3. In the case of Fig. 1.3 (a), spectral region whose level is less than

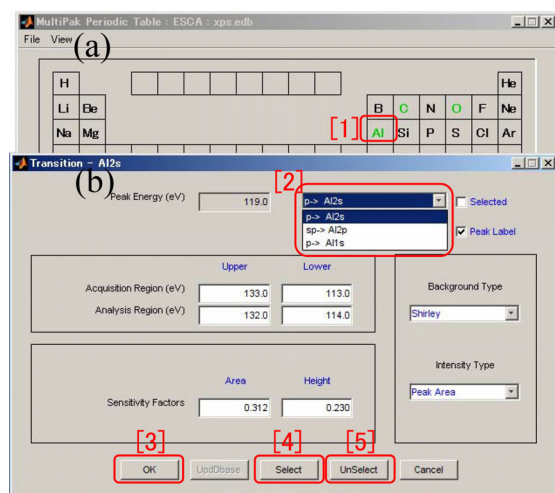


Figure 1.5: By clicking '[1] Al' (a) in the periodic table with Shift key pressed, 'Transition window' (b) has been displayed.

the background is integrated as negative values, which causes underestimation of integrated intensity. Therefore, the background should be changed by click&dragging 'Left bar' (and also 'Right bar' if necessary) as shown in Fig. 1.3 (b). After applying this procedure to all spectral peaks, '[1] %' button on the upper right corner of Fig. 1.4(a) can be clicked for a quantitative estimation of molar ratios of the elements. The molar ratios are evaluated dividing the integrated intensities of spectral peaks by 'Corrected RSF' that is a sensitivity factor of photoelectrons corrected with the machine parameters and the pass energy.

In the case of Fig. 1.4 (b), molar ratio of Al is estimated both for Al2p and Al2s peaks, which should be avoided with the following procedure.

In the periodic table of Fig. 1.5 (a), '[1] Al' should be clicked with [Shift] key pressed to display Fig. 1.5 (b). After selecting 'p-> Al2s' from '[2]' pull-down menu, '[5] Unselect' and then '[3] OK' can be clicked such that 'Al2s' is not found in '[3]' radio buttons in Fig. 1.6 (c).

After adjusting background level as shown in Fig. 1.3 (b) for all spectral peaks, '[1] %' button on the right of Fig. 1.6(a) can be clicked to display Fig. 1.6(b) '[2]', in which molar ratio of Al is estimated only for the integrated intensity of Al2p peak.

For estimating the molar ratios only from the integrated intensity of Al2s peak, the following

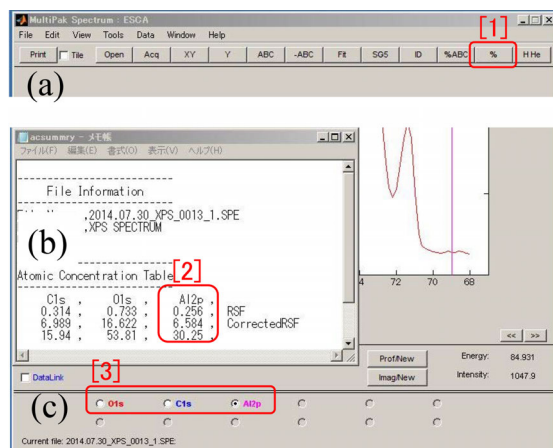


Figure 1.6: Quantitative analysis of elements from C1s, O1s and Al2p peaks.

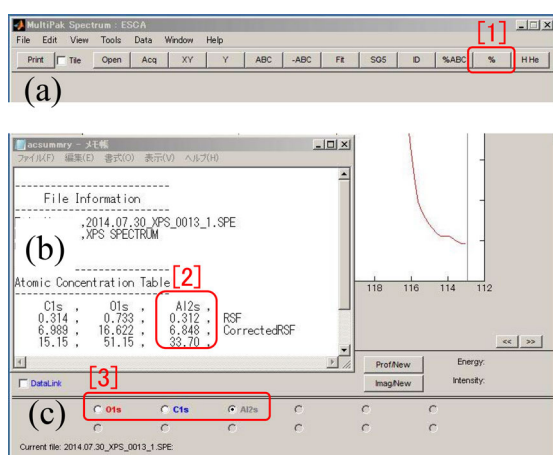


Figure 1.7: Quantitative analysis of elements from C1s, O1s and Al2s peaks.

procedure should be done. At first, after selecting 'p-> Al2s' from '[2]' in Fig. 1.5(b), and then '[4] Select' should be clicked. Next, after selecting 'sp-> Al2p', click '[5] Unselect' and then '[3] OK' button, please, such that only Al2s is found in Fig. 1.7(c) '[3]' radio buttons. After adjusting the background for every spectral peak as shown in Fig. 1.3(b), '[1] %' button on the right of Fig. 1.7(a) should be clicked such that Al molar ratio calculated using only Al2s is displayed as shown in Fig. 1.7(b).

1.1.4 Start of peak separation

Separating spectral peaks is complex. Try the following procedure, please. The pur-

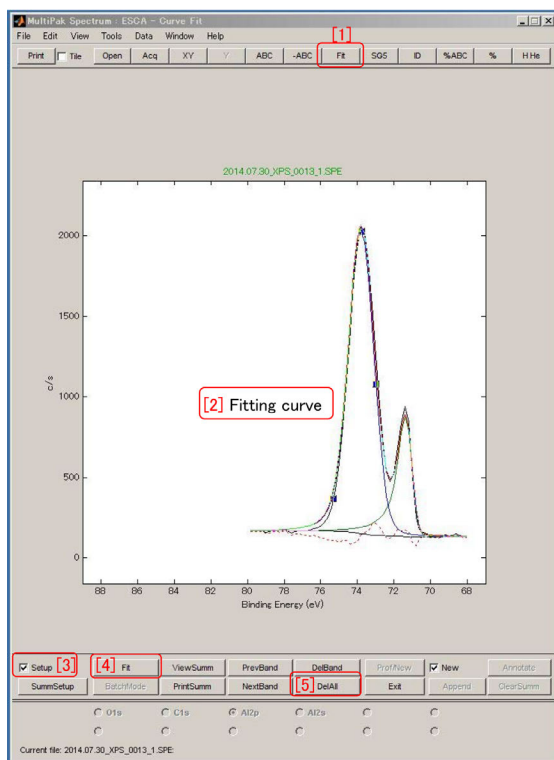


Figure 1.8: Start of curve fitting.

pose of separating peaks is evaluating quantitatively the integrated intensities of plural spectral peaks as shown in Fig. 1.8 as reasonably as possible. Ratios of '[7] Area' in Fig. 1.9 turns out to be molar ratios of the element with two chemical states. The value of '[2] ChiSquared' in Fig. 1.9 approaches to unity when residual error decreases.

1.1.4.1 Clicking 'Fit' button

'[1] Fit' button on the upper part of Fig. 1.8 can be clicked to display Fig. 1.9. If several fitting curves are already displayed in Fig. 1.8, '[5] Del All' button should be clicked to delete all of them.

1.1.4.2 Adjustment of background

'[3] Setup' check box on the lower left corner of Fig. 1.8, should be unchecked to adjust the background.

1.1.4.3 Making fitting curves

After checking '[3] Setup' check box in Fig. 1.8, two peak positions should be right-clicked to

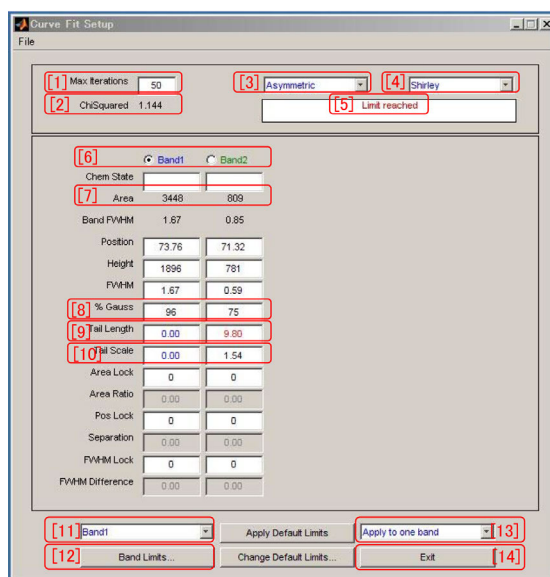


Figure 1.9: Curve fitting, setup window.

make two fitting curves.

1.1.4.4 Adjustment of fitting curves

Small square marks found on the top and side of the fitting curve in Fig. 1.8 should be click&dragged to adjust the height and width of it. The focus on '[6]' radio buttons and '[11]' pull-down menu in Fig. 1.9 move simultaneously with that on the square marks in Fig. 1.8. The focus can be changed by clicking the fitting curve in Fig. 1.8, rechecking '[6]' radio buttons or changing the selection from '[11]' pull-down menu in Fig. 1.9.

A red broken curve found under the fitted spectra in Fig. 1.8 shows the residual between the measured spectrum and that calculated from the sum of fitting curves. The heights and widths of fitting curves should be adjusted manually such that the residual becomes minimum.

1.1.4.5 Least square fitting

After that, '[4] Fit' button on the lower left corner of Fig. 1.8 should be clicked to do least square fitting such that the result are displayed in Fig. 1.9.

1.1.5 Refinement of peak separation

In '[1] Max Iterations' on the upper left corner of Fig. 1.9, how many times at most the least-square fitting to be repeated by clicking '[4] Fit' button once, can be set. The fitting should be repeated such that the deviation of '[2] ChiSquared' value from unity comes to be as small as possible. The value to be estimated is the ratio of '[7] Area'. From '[3]' pull-down menu in Fig. 1.9, three ways of fitting, i.e. 'Gauss' for fitting with Gaussian curves, 'Gauss-Lorentz' for fitting with a summation of Gaussian and Lorentzian curves and 'Asymmetric' for fitting taking into account the asymmetry of spectral curves. In '[4]' pull-down menu on the upper right corner of Fig. 1.9, 'Shirley' has been selected as a background estimation method. Here, 'Shirley' or 'IteratedShirley' is recommended in general.

1.1.5.1 Fitting with Gaussian curves

Gaussian curve gives distribution of error. When the spectrum to be fitted is regarded as error distribution, 'Gauss' is recommended. When 'Gauss' is selected for a fitting curve in Fig. 1.9, three items, '[8] % Gauss' (ratio of Gaussian), '[9] Tail Length' and '[10] Tail Scale' cannot be changed since these items are not included in the fitting parameters.

1.1.5.2 Fitting with Gaussian and Lorentzian curves

When 'Gauss-Lorentz' is selected from '[3]' pull-down menu in Fig. 1.9, the spectral curves are fitted with summation of Gaussian and Lorentzian curves.

The top of Lorentzian curve is more acute than that of Gaussian curve. It is empirically known that a symmetric spectral peak can be approximated with a summation of Gaussian and Lorentzian curves better than Gaussian only. '[8] % Gauss' in Fig. 1.9 can be changed since it is included in the fitting parameters. '[9] Tail Length' and '[10] Tail Scale' cannot be changed since the spectral peak is assumed to be symmetric.

1.1.5.3 Asymmetric fitting

When 'Asymmetric' is selected for fitting method in '[3]' pull-down menu in Fig. 1.9, the least-square fitting is done under an assumption that the spectral peak is asymmetric. '[9] Tail Length' and '[10] Tail Scale' can be changed since these values are included in the fitting parameters.

Photoelectron spectral peaks have tails on the left (higher energy) side in general. Photoelectrons found in the vicinity of the spectral peak position are emitted from atoms whose depth is less than 10 nm without being interacted with other electrons.

In X-ray photoelectron spectroscopy, binding energy E_B (strictly speaking $E_B + \text{work function}$) can be calculated by subtracting the measured velocity energy E_V of photoelectrons from the photon energy of X-rays, $h\nu$, i.e. $E_B = h\nu - E_V$. Therefore, if the velocity energies of photoelectrons are lost by interacted with other electrons, the binding energy E_B is overestimated. Photoelectrons emitted from atoms whose depth from the sample surface is large, are interacted with other electrons and then the velocity energies are lost, which causes overestimation of the binding energies.

However, the asymmetry of photoelectron spectral peak is not only due to the above description but also due to more complex reasons.

While photoelectron spectrum for an insulator has a symmetric shape, that for metal has an asymmetric shape in general. The reason is considered to be that photoelectrons emitted from metal atoms are interacted with many free electrons and then their velocity energies are lost.

Free electrons in conductors (metals) are electrons that can be accelerated even by an infinitesimal electric field. Fermi energy is an energy at which the possibility of existence of electrons is just 0.5. In the case of conductor (metal), Fermi energy exists in conduction band. In the case of insulator, Fermi energy exists in the forbidden band with a large band gap. In the case of semiconductor, the situation is intermediate between the cases of conductor and insulator. Electrons in an insulator can receive only energy larger than the band gap whereas free electrons in a conductor (metal) can receive

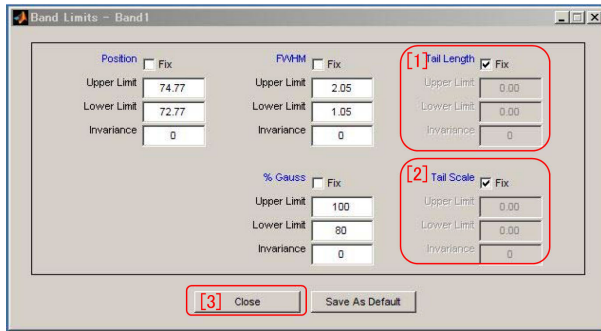


Figure 1.10: Curve fitting, Band limit setting window.

an infinitesimal energy from electric field made by an emitted photoelectron.

Asymmetric fitting takes into account the above situations in conductors and semiconductors. Therefore, the spectral peak has a tail on the left (higher energy) side of it whose '[9] Tail Length' (width) and '[10] Tail Scale' (height) are included in the fitting parameters.

Figure 1.8 [p.4] shows a photoelectron profile measured for an Al plate whose surface is oxidized. The left and right peaks can be assigned to Al_2O_3 and elemental aluminum, respectively.

For the left peak due to Al_2O_3 , '[9] Tail Length' and '[10] Tail Scale' in Fig. 1.9 [p.4] are fixed to be zero since there are no free electrons that can receive infinitesimal energy from a small electric field made by a photo-emitted electron in Al_2O_3 (insulator). In this case, after setting both '[9] Tail Length' and '[10] Tail Scale' to be zero and selecting 'Apply to one band' from '[13] pull-down menu' in Fig. 1.9 [p.4], '[12] Band Limits' button should be clicked to display Fig. 1.10. After checking '[1] Tail Length Fix' and '[2] Tail Scale Fix' in Fig. 1.10, '[3] Close' button should be clicked.

On the upper right of Fig. 1.9 [p.4], '[5] Limit reached' is displayed with red characters and also value of '[9] Tail Length' is displayed with red numbers. These red characters or value mean that further least-square fitting can not be done since this value reached to 'Upper Limit' or 'Lower Limit' set in Fig. 1.10. In Fig. 1.8 [p.4], a slight discrepancy is found between measured spectrum drawn with a black curve and sum of two fitting curves drawn with a red curve around middle position of two peaks. However,

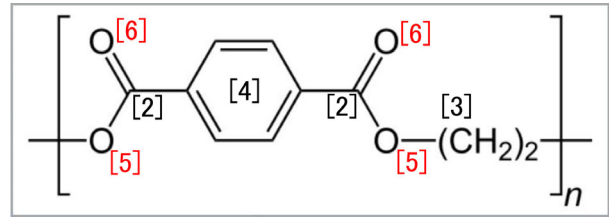


Figure 1.11: Molecular structure of PET(from Wikipedia)

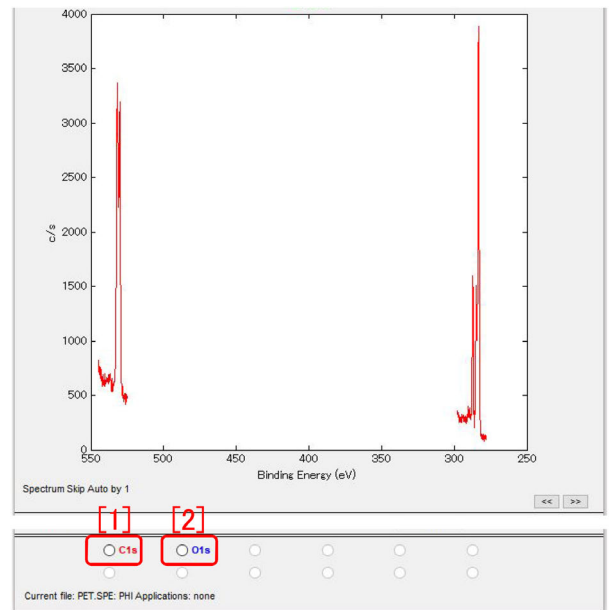


Figure 1.12: Narrow spectra measured for PET

the least square fitting stopped since '[9] Tail Length' of Band2 (light blue curve) reached to the limit value. The limit values can be changed by selecting 'Apply to one band' from '[13] pull-down menu' in Fig. 1.9 [p.4] and then clicking '[12] Band Limits' button to display Fig. 1.10. Further least-square fitting can be done by clicking '[3] Close' button in Fig. 1.10 and then '[4] Fit' button on the lower left corner of Fig. 1.8 [p.4].

If not going well, '[14] Exit' button in Fig. 1.9 [p.4] can be clicked to retry the fitting.

1.1.6 Chargeup correction

If the user want to do charge-up correction, refer to Appendix A, please while it is not necessary usually in the case of this apparatus.

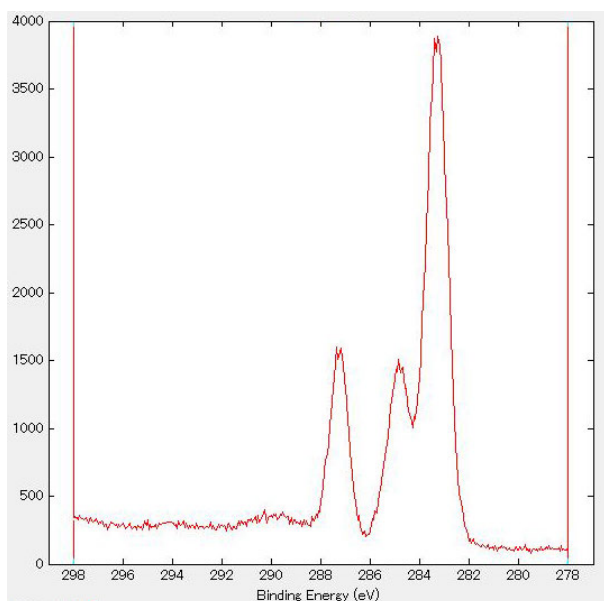


Figure 1.13: C1s photoelectron spectrum for PET

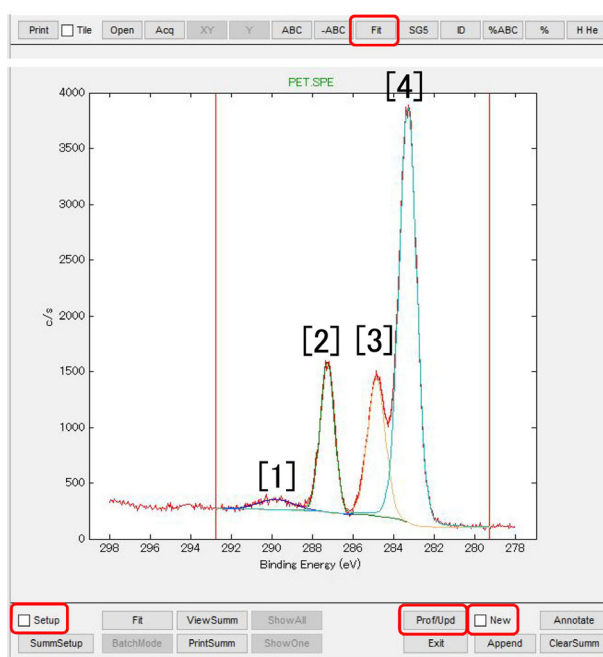


Figure 1.14: Curve fitting for the C1s spectrum

1.1.7 Identification of chemical state

For identifying the spectral peak position to chemical state, refer to Appendix B [p.23], please. Furthermore, Appendix C [p.25] can be referred to use the data base of the NIST (National Institute of Standard and Technology).

1.2 Quantitative evaluation of chemical states for different elements

1.2.1 Concerning PET (polyethyleneterephthalate)

PET (polyethyleneterephthalate) is a widely used high molecular compound as the material of PET bottles and others. Fig. 1.11 shows one unit molecular structure of PET. It has '[2] carbons of C-O double bond in carbonyl group', '[3] carbons of methylene group', '[4] carbons of benzene ring', '[5] oxygens bonded with two carbon atoms' and '[6] oxygens of C-O double bond in carbonyl group'. They are contained with an atomic ratio of 2 : 2 : 6 : 2 : 2 as shown in Fig. 1.11.

1.2.2 Loading photoelectron spectra

Fig. 1.12 shows photoelectron spectra of O1s and C1s loaded on the 'MultiPak'. By checking '[1] C1s' radio button in Fig. 1.12, C1s spectrum can be enlarged as shown in Fig. 1.13.

1.2.3 Peak separation and quantative analysis of C1s spectrum

Fig. 1.14 shows four curves separately to fit the C1s spectrum. This has been done by referring Fig. 1.8 [p.4] and the description of §1.1.4 [p.3]. By unchecking 'Setup' on the lower left, unchecking 'New' on the lower right and then clicking 'Prof/Upd' button in Fig. 1.14, Fig. 1.17 [p.8] can be displayed.

When [1], [2], [3] and [4] are checked in Fig. 1.17 [p.8], molar ratios of the elements corresponding to [1], [2], [3] and [4] of Figs. 1.11 and 1.14, are plotted in the graphic region of Fig. 1.17 [p.8]. However, [1] does not exist in Fig. 1.11 since it is owing to π - π^* transition and cannot be assigned to any atom.

By clicking '[1] %' button on the upper right corner of Fig. 1.17 [p.8] with '[2] AC%' checked, Fig. 1.18 [p.8] can be shown. '[1], [2], [3], [4]' displayed in Fig. 1.18 [p.8] are molar ratios of these chemical states. The relative sensitiv-

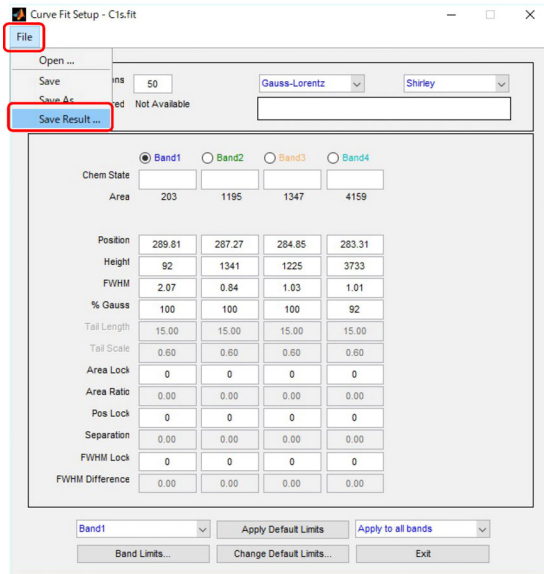


Figure 1.15: Peak separation of C1s spectrum

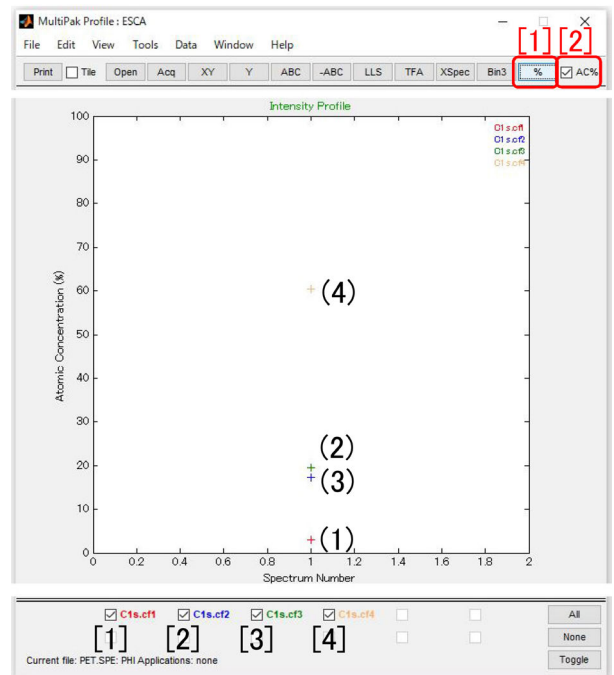


Figure 1.17: Chemical state ratios of the C1s spectrum

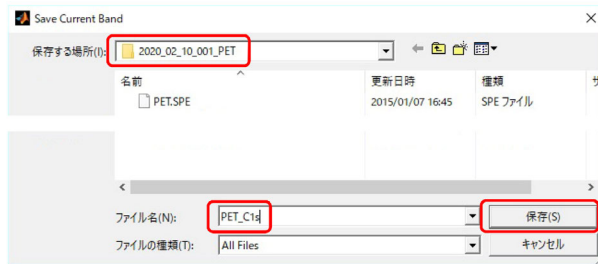


Figure 1.16: Export of the spectra



Figure 1.18: Chemical state ratios of carbon and oxygen

ity factors (RSF) in the small red frame of Fig. 1.18 are the emission sensitivities of photoelectron compared with that for F1s (RSF for F1s is unity). Corrected RSF are the corrected values of sensitivity with the machine parameter of the VersaProbe and the pass energy. However, the RSF is considered not to depend on the chemical state and is an identical value for the different chemical states. The molar ratios (%) have been calculated by dividing the integrated intensities of their peaks by the corrected RSF such that the summation of them is 100 (%).

1.2.4 Addition of peak separation and quantitative analysis of the O1s spectrum

A peak separation window for the O1s spectrum as shown in Fig. 1.19 can be opened by checking the radio button of '[2] O1s' on the lower left

of Fig. 1.12 [p.6]. When clicking 'Fit' on the upper part of Fig. 1.19, Fig. 1.20 is displayed. Then, curve fitting can be done referring to the description in §1.1.4 [p.3].

After the above procedure, by opening 'File' and then clicking 'Save Result...' on the upper left corner of Fig. 1.20, Fig. 1.21 can be displayed. After selecting the folder, 'Save' can be clicked to save the data into the file named as 'PET_O1s'. Five files as shown to be '[O1s]' in the red frame of '[1]' in Fig. 1.24 [p.10] are saved separately. With regard to the following procedure, refer to the description in §1.3 [p.10], please.

Similarly to the case of C1s, 'Prof/Upd'

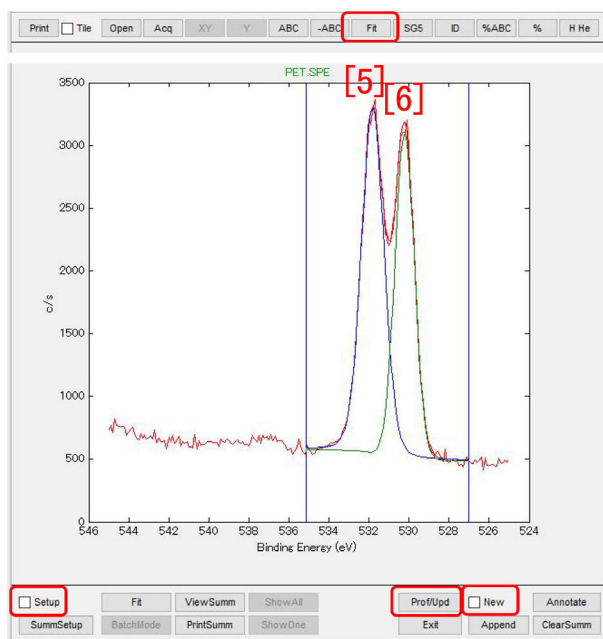


Figure 1.19: Curve fitting of O1s spectrum

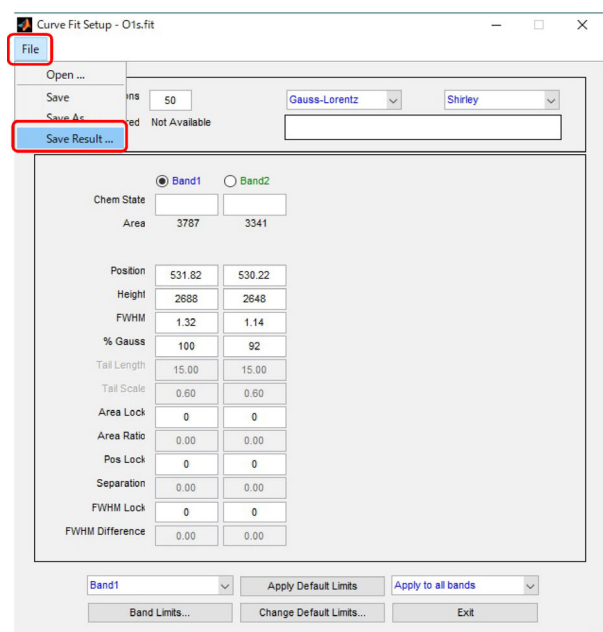


Figure 1.20: Peak separation of the O1s spectrum

should be clicked in Fig. 1.19, with ‘Setup’ on the lower left and ‘New’ on the lower right corners unchecked to display Fig. 1.22.

‘[1]’ has been unchecked on the lower left corner of Fig. 1.22 since this peak is due to $\pi\text{-}\pi^*$ transition and cannot be assigned to any atom.

When ‘[2]AC%’ is checked on the upper right

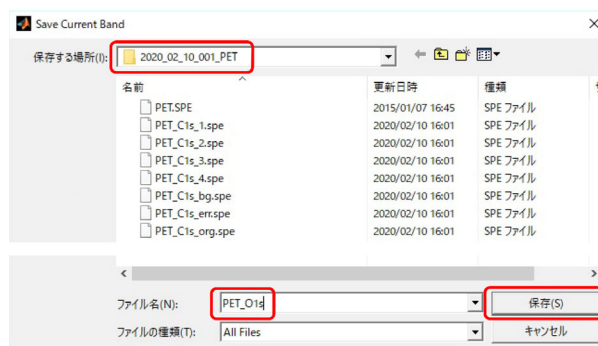


Figure 1.21: File name specifying window

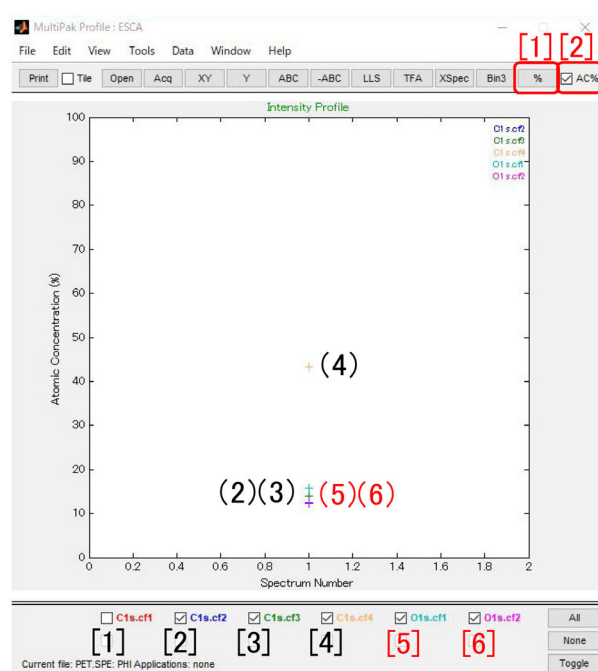


Figure 1.22: Molar ratio of chemical states of carbon and oxygen

of Fig. 1.22, ‘+ mark’ of ‘(2), (3), (4)’ and ‘(5), (6)’ are displayed to show the molar ratios of chemical states of carbon and oxygen. The molar ratios of ‘(2), (3), (5), (6)’ and ‘(4)’ are found to be approximately equal to 1 : 3 as shown in Fig. 1.11 [p.6].

Here, by clicking ‘[1] %’ on the upper right of Fig. 1.22, Fig. 1.23 [p.10] can be displayed. ‘[2], [3], [4]’ and ‘[5], [6]’ in the lowermost part of Fig. 1.23 [p.10], shows molar ratios of carbons with chemical states of ‘[2], [3], [4]’ and oxygens with those of ‘[5], [6]’ in Figs. 1.11 [p.6], 1.14 [p.7] and 1.19 [p.9].

Abcissa	C1s.cf2	C1s.cf3	C1s.cf4	O1s.cf1	O1s.cf2
1.00	2.008	2.008	2.008	5.342	5.342
	12.62	14.28	44.17	16.36	12.57

Figure 1.23: Molar ratio of chemical states of carbon and oxygen

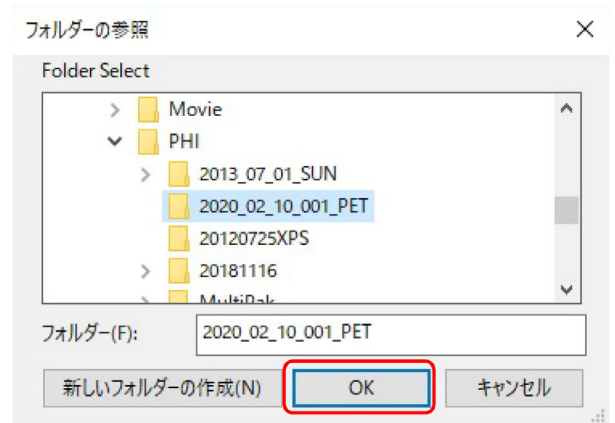


Figure 1.25: Folder select window of the 'MultiPak'

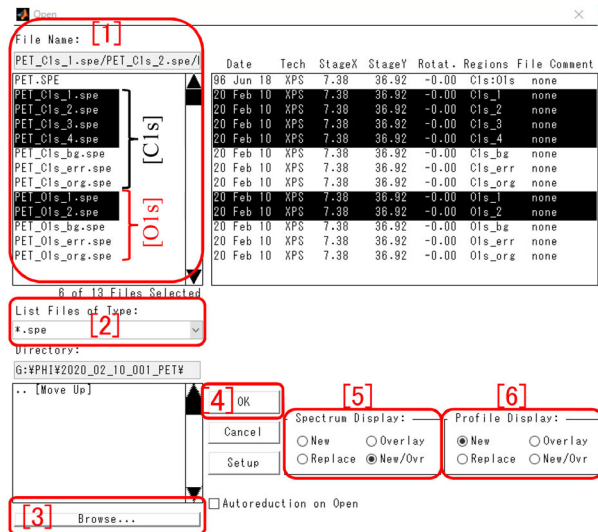


Figure 1.24: File opening window of the 'MultiPak'

1.3 Loading and drawing of the fitting curve

1.3.1 Loading of separated fitting curves on the 'MultiPak'

'Open' on the upper left corner of Fig. 0 on the cover of this manual, can be clicked to let Fig. 1.24 be shown. After selecting an extension of '*.spe' in the red frame of '[2]' in Fig. 1.24, '[3] Browse' on the lower left can be clicked to display Fig. 1.25.

After choosing the same folder as selected in Figs. 1.16 [p.8] and 1.21 [p.9], 'OK' can be clicked in Fig. 1.25 to let file names be shown in the red frame of '[1]' in Fig. 1.24. Seven files of '[C1s]' in Fig. 1.24 correspond to the spectra separated into '[1], [2], [3], [4]', the background (_bg.spe), the residual (_err.spe) and the

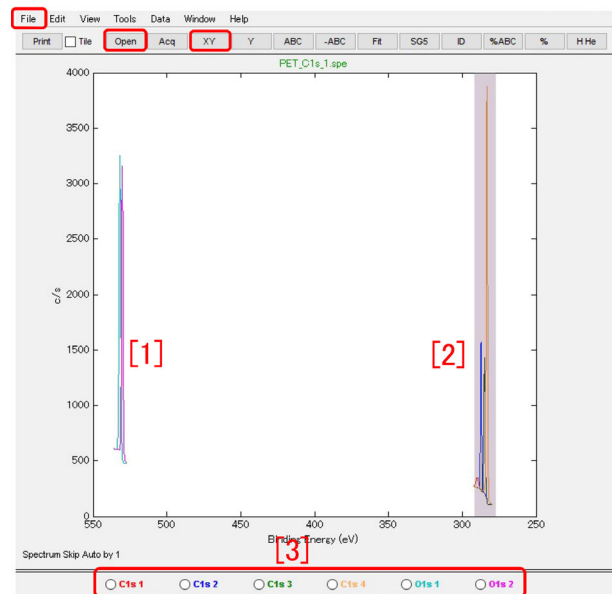


Figure 1.26: Spectra of O1s and C1s have been loaded

as-measured spectrum (_org.spe).

By checking 'New/Ovr' in the red frame of '[5]' in Fig. 1.24 and then clicking '[4] OK', Fig. 1.26 can be displayed. The radio button of '[3]' can be checked to show an enlarged spectrum and unchecked again to show the spectra with the original scale.

In Fig. 1.26, '[1]': separated O1s spectrum' and '[2]': separated C1s spectrum' are shown. By horizontally dragging the vicinity of '[2]' an enlarged spectrum can be displayed as shown in Fig. 1.27.

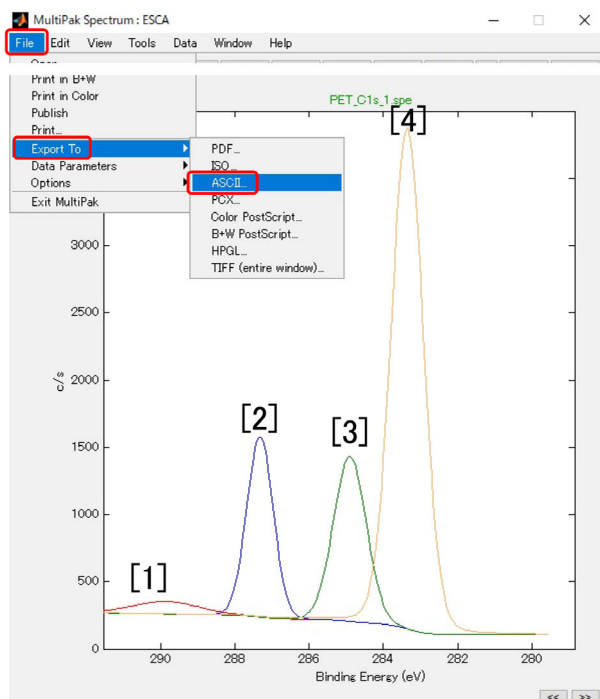


Figure 1.27: C1s spectrum separated into four curves

1.3.2 Export of the separated fitting curves into ascii files

On the upper left of Fig. 1.27, by opening 'File', selecting 'Export To' and then clicking 'ASCII', Fig. 1.28 can be opened. By specifying 'PET_C1s_1234.csv' as the file name and then clicking 'Save(S)', the file that can be loaded on the Excel is saved with this file name.

1.3.3 Loading and graph-drawing of the separated fitting curves

Fig. 1.29 has been opened by double-



Figure 1.28: File name specifying window

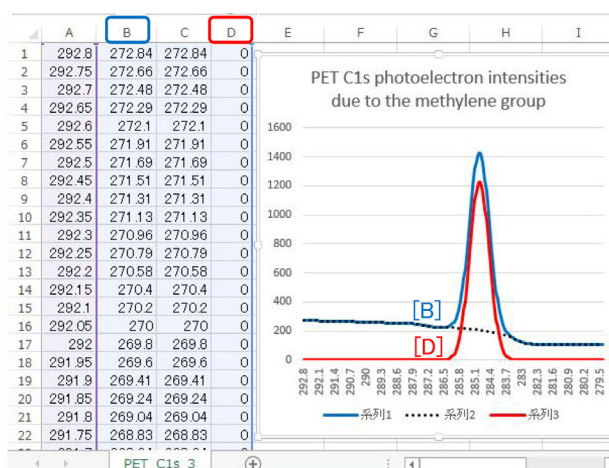


Figure 1.29: Spectrum load and drawn as a graph on the Excel

clicking the saved file. The values on the columns of [A], [B], [C] and [D] on the left of Fig. 1.29 are '[A]: binding energy (eV)', '[B]: curve of [3] in Fig. 1.27' '[C]: background' and '[D]: [B] - [C]'.

Please, note that the curve of '[3]' is not the fitting curve itself fitted with Gaussian and Lorentzian curves, but the summation of fitting curve and background.

To be continued

Chapter 2

Analysis of sputtering spectrum data

2.1 Analysis of sputtering spectrum data

‘[1] Open’ button in Fig. 0 on the cover (‘Open’ button on the upper left of Fig. 2.1) can be clicked to display Fig. 2.1

‘[3] Browse’ button on the lower left of Fig. 2.1 can be clicked to open an explorer. After selecting a folder, ‘OK’ button can be clicked to show files whose extension has been selected from ‘[2] List Files of Type:’ pull-down menu. In ‘[1] File Name’ box on the upper left of Fig. 2.1. For opening a data file of sputtering spectrum data, ‘*.pro’ should be selected from ‘[2] List Files of Type:’ pull-down menu. Plural data files can be selected by clicking file names

with [Shift] button pressed. ‘[4] OK’ button can be clicked to open the selected data file(s).

File opening mode can be selected by checking ‘[5] Spectrum Display’ radio button for opening the spectral window (on the left of Fig. 2.2 [p.14]) and by checking ‘[6] Profile Display’ radio button for opening the profile window (on the right of Fig. 2.2 [p.14]). In the profile window, molar ratios of the elements depending on the depth are shown. If ‘New’ radio button has been checked, the selected data file is displayed in a new window. If ‘Replace’ radio button has been checked, it is displayed by replacing the old data with it. If ‘Overlay’ radio button has been checked, it is displayed by overlaying it with old one. If ‘New/Ovr’ radio button has

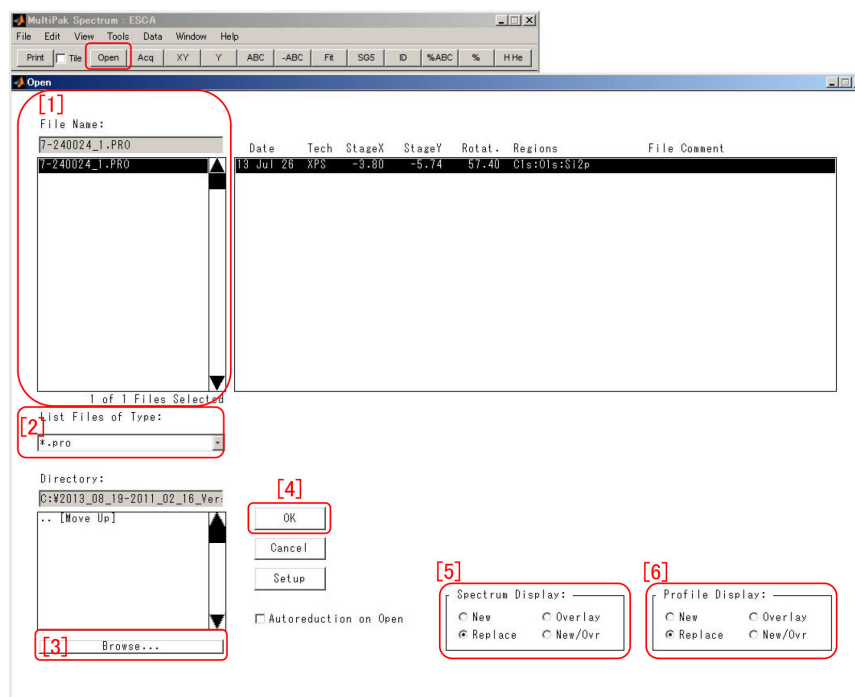


Figure 2.1: File selection window

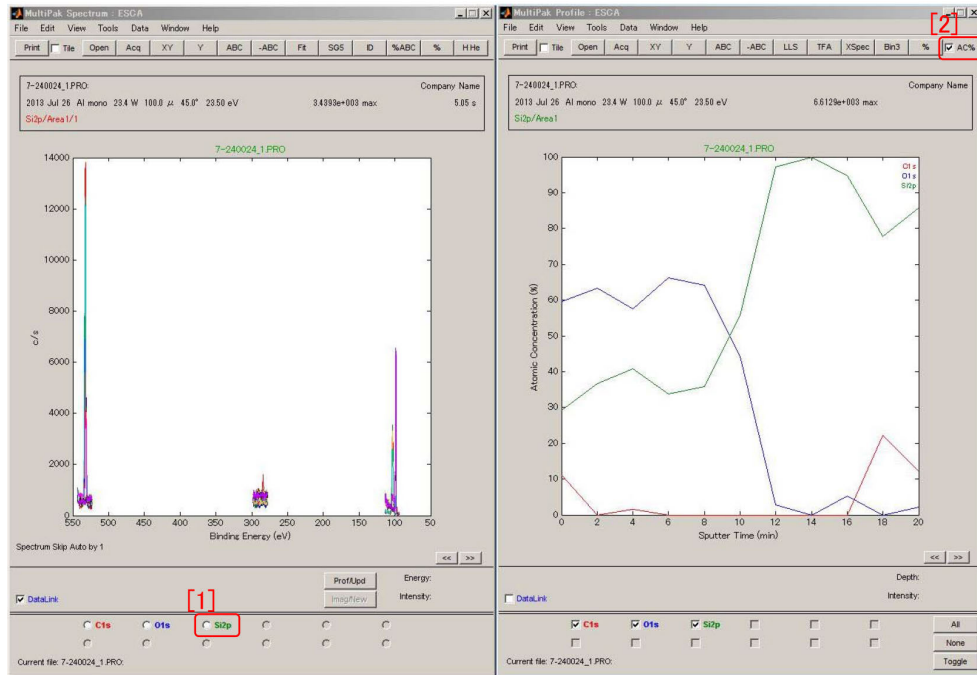


Figure 2.2: Spectrum window and profile window

been checked, the selected plural data are newly displayed overlaying them simultaneously.

‘[1] Si2p’ radio button on the lower left of Fig. 2.2 can be clicked to display only Si2p spectra measured by repeating the sputtering as shown in Fig. 2.3. When ‘[2] AC%’ check box in Fig. 2.2 has been checked, the right ordinate is displayed in molar ratio while it is displayed in integrated intensity when ‘AC%’ check box is unchecked.

2.1.1 Display of selected narrow spectra

In Fig. 2.3, all spectra taken by repeating the sputtering are overlaid. However, as shown in Fig. 2.4, ‘Select Spectra’ in ‘Tools’ menu can be clicked such that selected spectra can be displayed. After clicking ‘Select Spectra’, a scroll bar is displayed on the right side of the spectra as shown in Fig. 2.5.

‘Select One’ button in Fig. 2.5 can be clicked to display only one spectrum. The ordinal number of spectrum that is displayed, can be changed by scrolling the slider from up to down or by clicking small green triangles upper and lower of the scroll bar. In Figs. 2.6(a), 2.6(b), 2.6(c) and 2.6(d), 1st, 5th, 6th and 10th spectra are displayed, respectively.

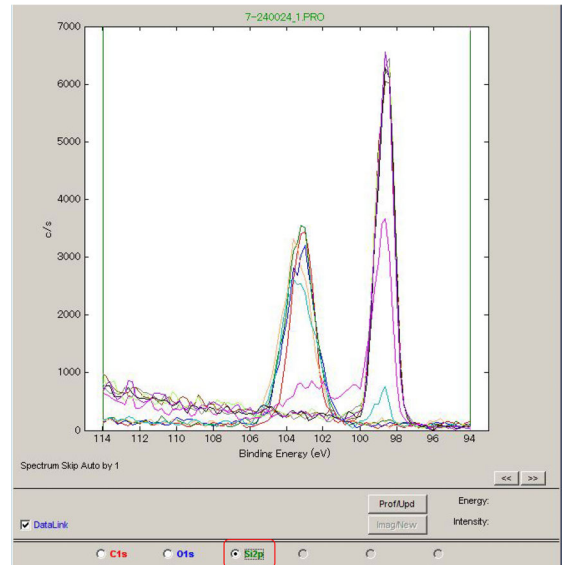


Figure 2.3: Si2p narrow spectrum.

2.1.2 three-dimensional display of sputtering spectrum data by using montage viewer

As shown in Fig. 2.7 [p.16], ‘Montage Viewer’ in ‘Tools’ menu can be clicked to display Fig. 2.8 [p.16]. ‘Montage’ can be selected from ‘[2] Mode’

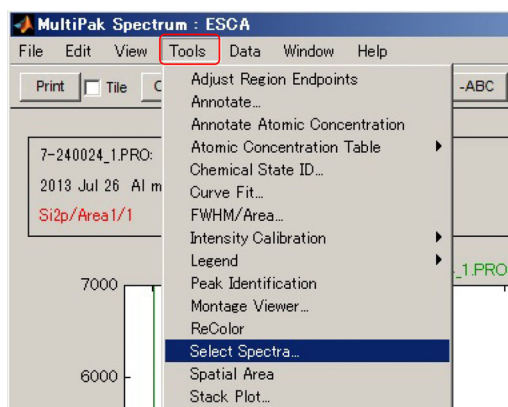


Figure 2.4: 'Select spectra' has been selected from 'Tools menu'.

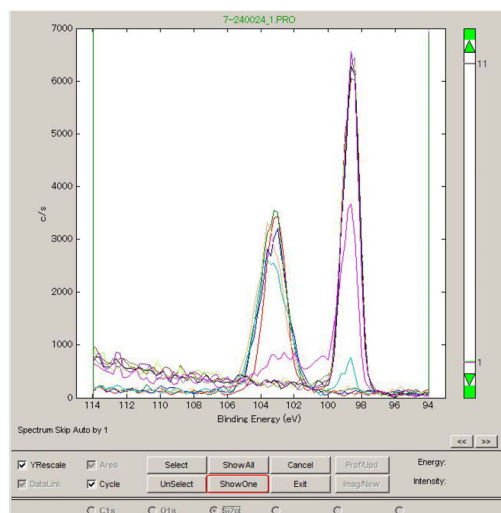


Figure 2.5: Spectrum selection scroll bar has been displayed.

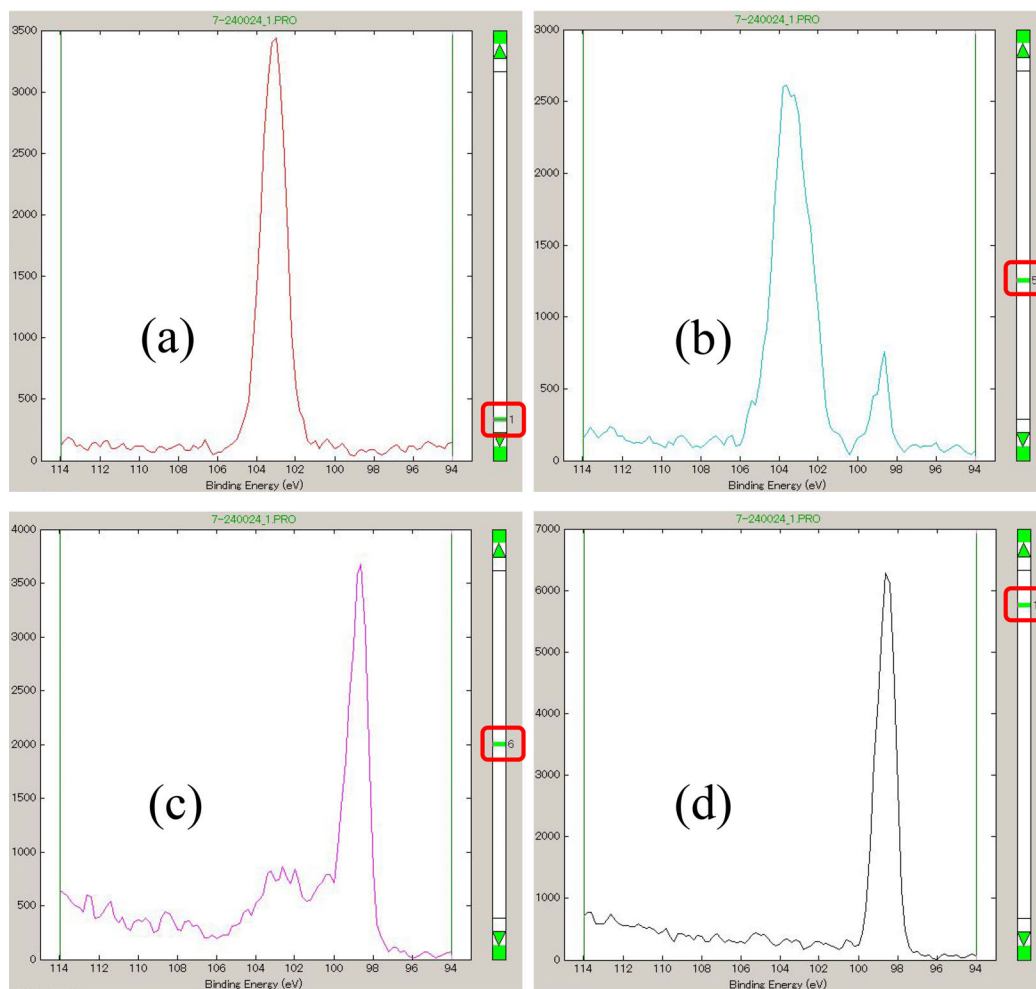


Figure 2.6: Each spectrum has been displayed.

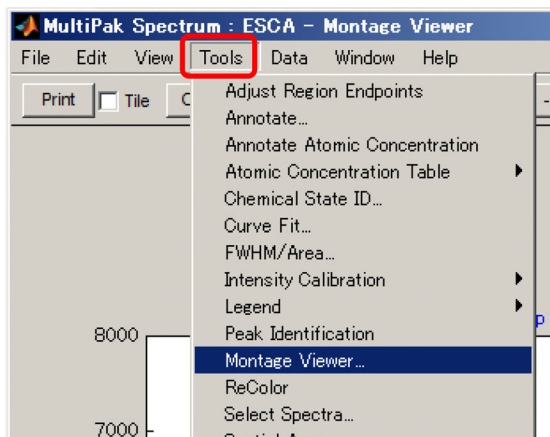


Figure 2.7: 'Montage viewer' in 'Tools' menu can be clicked.

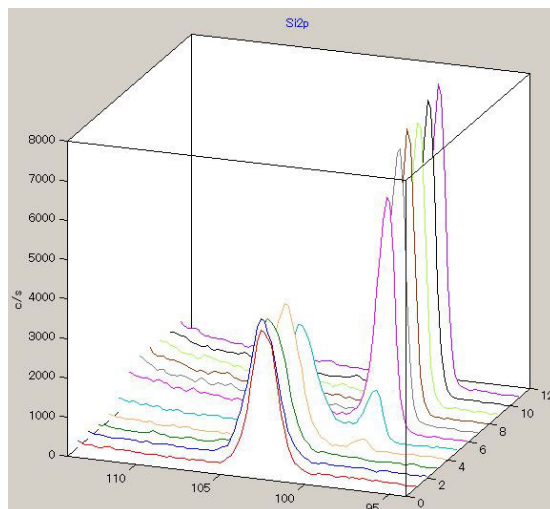


Figure 2.9: Spectrum obtained by repeating sputtering can be three-dimensionally displayed.

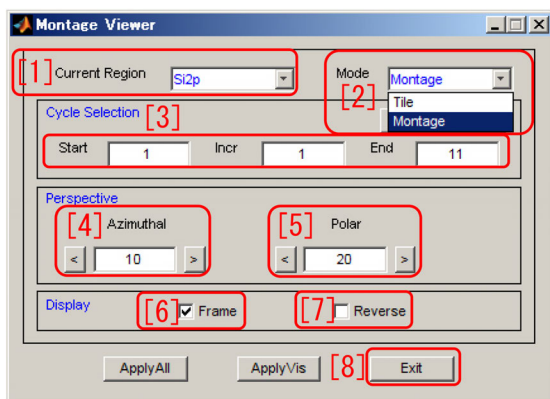


Figure 2.8: Settings of the 'Montage viewer'.

pull-down menu in Fig. 2.8 to display spectra taken by repeating the sputtering three-dimensionally as shown in Fig. 2.9.

If plural spectral region have been measured, one of them can be selected from '[1] Current Region' pull-down menu such that it is displayed. In '[3] Cycle Selection' box in Fig. 2.8, initial

[Start], step [Incr] and final [End] number of spectra can be set. In '[4] Azimuthal' and '[5] Polar', rotation angle around vertical and horizontal axes, respectively, can be set by typing angles or clicking buttons. '[6] Frame' check box can be unchecked such that the frames are not drawn. '[7] Reverse' check box can be checked to reverse the ordinal number of the displayed spectra. '[8] Exit' button can be clicked to exit the montage viewer.

2.1.3 Chargeup correction

If the user want to do charge-up correction, refer to Appendix A, please while it is not necessary usually in the case of this apparatus.

2.1.4 Identification of chemical state

For identifying the spectral peak position to chemical state, refer to Appendix B, please.

Chapter 3

Analysis of mapping data

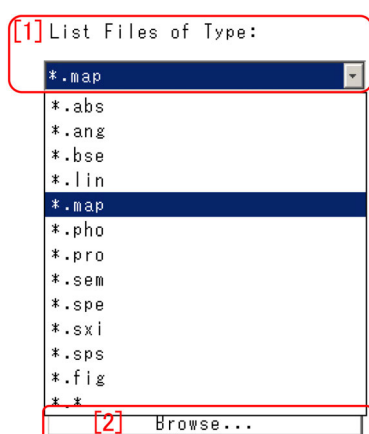


Figure 3.1: File type selection window.

3.1 Reading a map data file

[1] Open' button in Fig. 0 on the cover of this manual ('Open' button on the upper left of Fig. 2.1 [p.13]) can be clicked to display 'File type selection' window as shown in Fig.3.1. In Fig. 3.1, '[1] List Files of Type:' pull-down menu. has been opened. After selecting folder by clicking '[2] Browse' button, File type '*.map' should be selected from the '[2] List Files of Type:' pull-down menu. In Fig. 3.2, mapping data files are shown, from which file(s) can be selected to open it (or them) by clicking '[1] OK' button.

3.2 Analysis of mapping data from a single peak

Figure 3.3 shows 'MultiPak Spectrum' window (left) and 'MultiPak Map' window (right) displayed after inputting data file(s). A map has

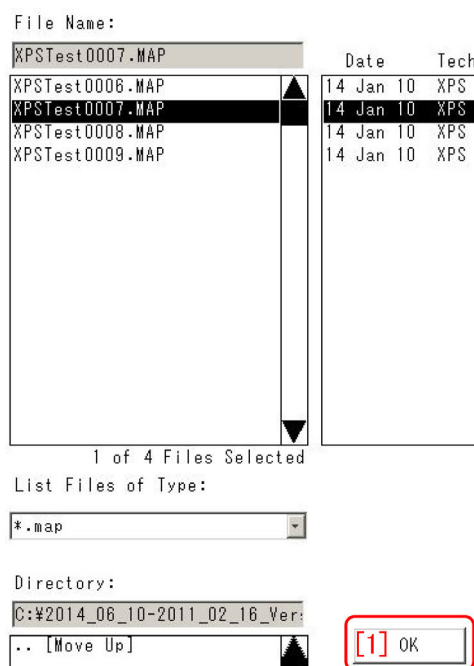


Figure 3.2: File open window.

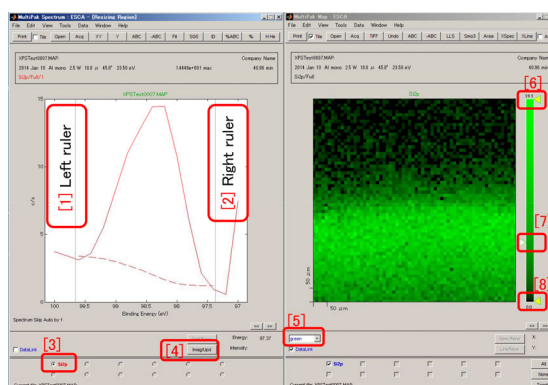


Figure 3.3: Spectrum window and map window. Map by a single Si2p peak of elemental silicon.

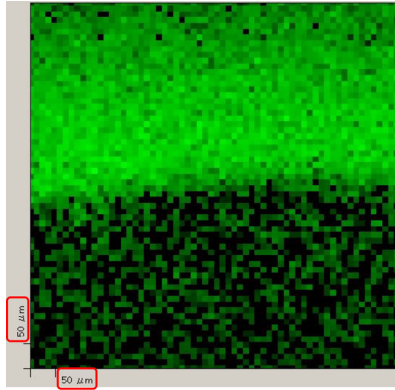
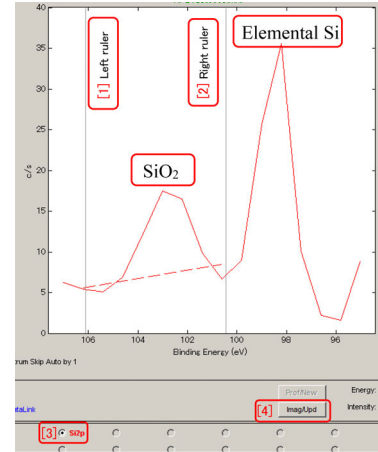
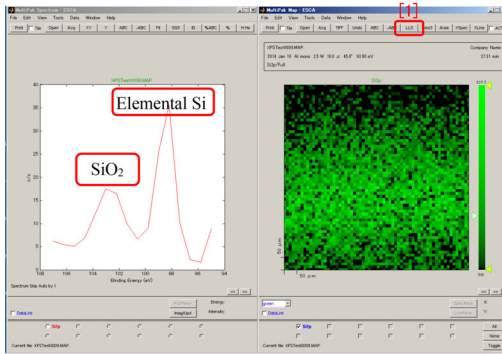
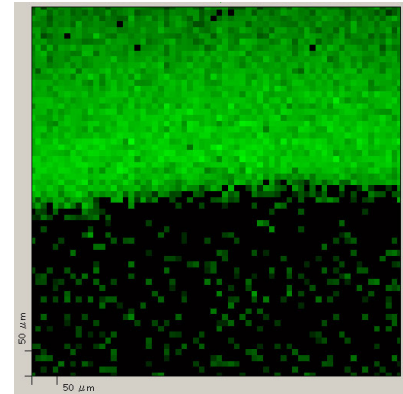
Figure 3.4: Map by a single Si2p peak of SiO₂.Figure 3.6: A spectral range for SiO₂ peak has been selected from a range including two peaks.

Figure 3.5: Mapping data file with plural peaks has been opened.

been obtained only for a peak of elemental silicon in Si2p spectrum. In Fig. 3.3 [p.17], '[3] Si2p' radio button can be checked to display '[1] left ruler' and '[2] right ruler'. These rulers can be click&dragged to adjust the range of spectrum. After that, '[4] Imag/Upd' button can be clicked to make the contrast of the map clearer.

In Fig. 3.3 [p.17], the color of the map can be selected from '[5] Color selection' pull-down menu. Tone of the map can be changed by sliding up or down small triangles, '[6]', '[7]', and '[8]'.

Figure 3.4 shows a map obtained only for single SiO₂ peak similarly to that obtained for an elementary silicon peak.

Figure 3.7: An extracted map for SiO₂ peak.

3.3 Analysis of mapping data obtained for plural peaks

3.3.1 Extraction of a map by setting a spectral range

Figure 3.5 shows a map obtained from an Si2p spectral range including both peaks of SiO₂ and elemental silicon.

In Fig. 3.6, after checking '[3] Si2p' radio button, a spectral range including only SiO₂ peak has been selected by click&dragging '[1] left ruler' and '[2] right ruler'.

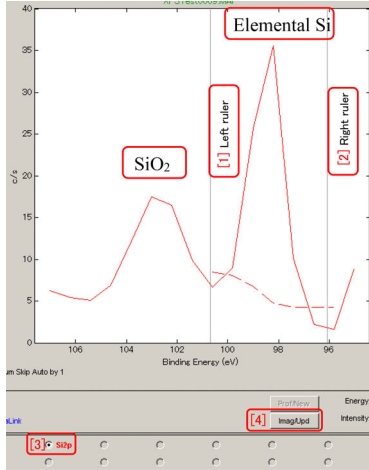


Figure 3.8: A spectral range for elemental silicon has been selected from a range including two peaks.

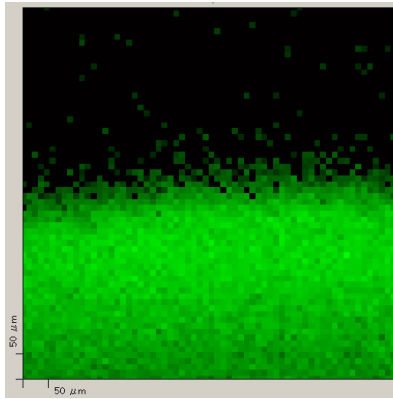


Figure 3.9: An extracted map for elemental silicon.

After that, in Fig. 3.6 '[4] Imag/Upd' button can be clicked to display an SiO₂ map as shown in Fig. 3.7.

In Fig. 3.8, a spectral range including only an elemental silicon peak has been selected in a similar way. '[4] Imag/Upd' button can be clicked to display an elemental silicon map as shown in Fig. 3.9.

3.3.2 Extraction of a map by selecting a region in the map

A map can be extracted also by selecting a region in the map. After clicking '[1] LLS' button on the upper right of Fig. 3.5, square

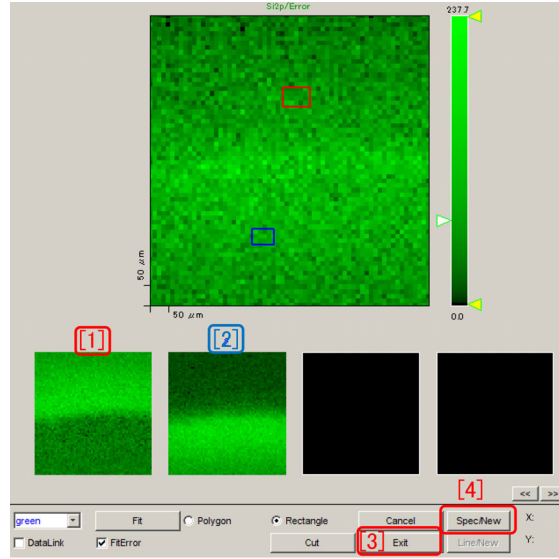


Figure 3.10: Extraction of maps for SiO₂ and elemental silicon by selecting region.

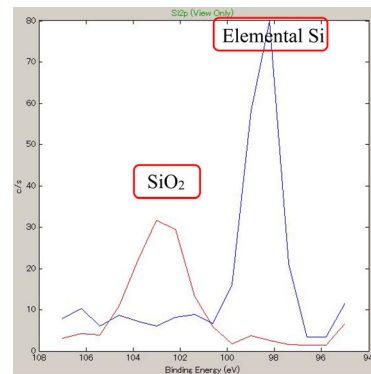


Figure 3.11: A spectrum extracted from selected region.

regions 'red' and 'blue' in Fig. 3.10, can be click&dragged to select them to display maps for peaks in the selected region, i.e. peaks of SiO₂ and elemental silicon.

'[4] Spec/New' button on the lower right of Fig. 3.10 can be clicked to display spectra used to obtain the maps of [1] and [2] as shown in Fig. 3.11.

'[3] Exit' button on the lower right of Fig. 3.10 can be clicked to display Fig. 3.12 [p.20]. Maps of '[1] Si2p', '[2] Si2p.ls1' and '[3] Si2p.ls2' can be displayed or not displayed by checking or unchecking check boxes of '[4] Si2p', '[5] Si2p.ls1' and '[6] Si2p.ls2'.

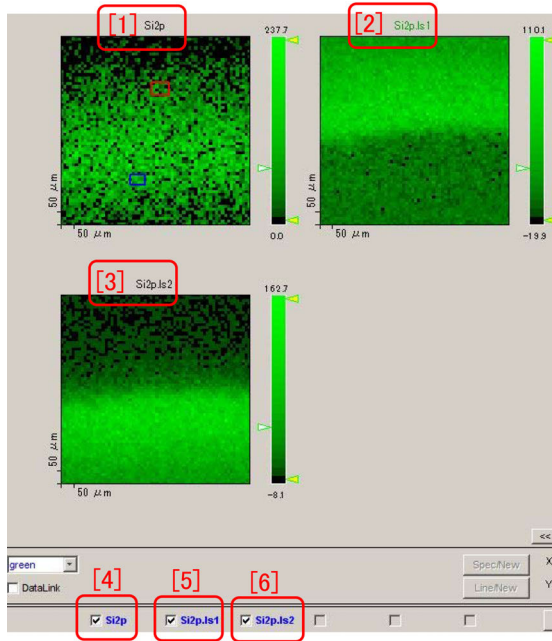


Figure 3.12: In Fig. 3.10, '[3] Exit' button has been clicked.

In Fig. 3.13, maps of '[1] Si2p.ls1' and '[2] Si2p.ls2' have been displayed by unchecking '[4] Si2p' and by checking '[5] Si2p.ls1' and '[6] Si2p.ls2'. Here, the color of elemental silicon map has been changed to be red by selecting 'red' from '[3] color selection' pull-down menu after clicking any position on the elemental silicon map.

In Fig. 3.14, 'RGB Overlay' in 'Tools' menu has been clicked to overlay the green SiO_2 map and the red elemental silicon map.

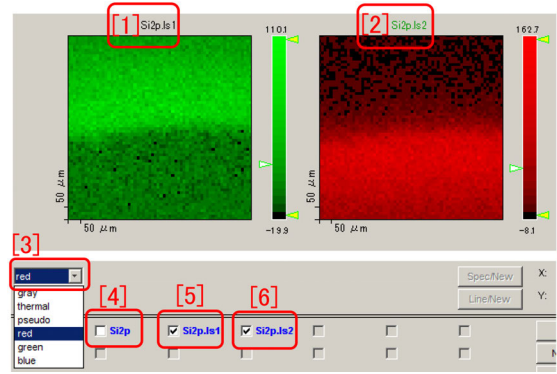


Figure 3.13: The color of map for elemental silicon has changed to be red.

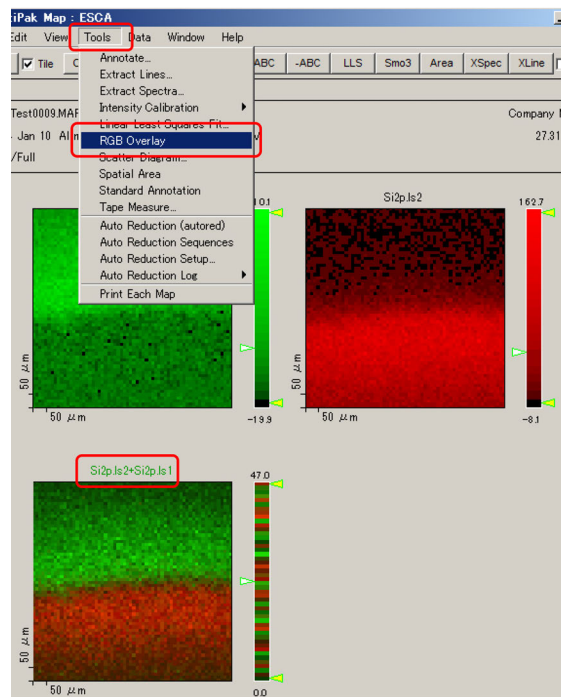


Figure 3.14: A map displayed with different colors for SiO_2 and elemental silicon.

Appendix A

About charge-up shift correction

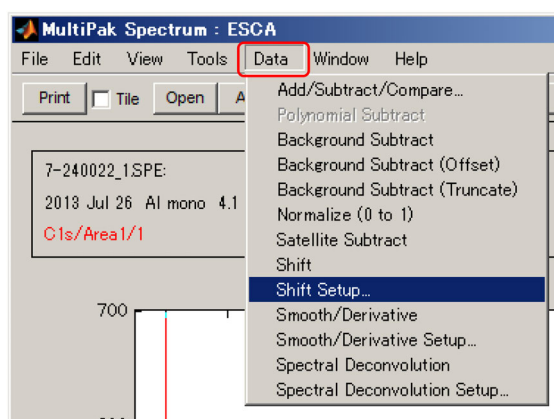


Figure A.1: Startup of the charge-up shift correction menu.

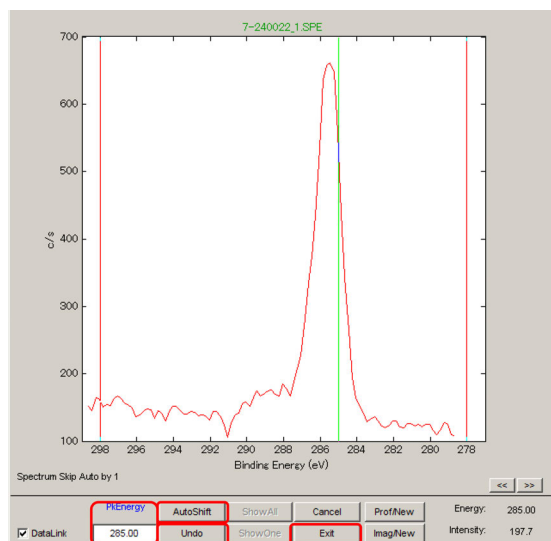


Figure A.2: Charge-up correction window.

A.1 Charge-up shift correction

A.1.1 Abstract of charge-up shift correction

In the case of Phi5000 VersaProbe, by using both low-energy electron gun and low-energy Ar ion gun, charge on the surface of sample is almost completely removed even for an insulator sample. Therefore, correction for charge-up shift is almost not necessary.

However, since 1960's, there were many discussions about correction for charge-up shift for insulator samples.

When a constant plus charge exists on the sample surface due to the emission of electrons, photo electrons (and Auger electrons) lose velocity energy with an identical value. Therefore, if charge-up shift of a spectral peak with a known binding energy is estimated, it can be applied for other peaks.

In general, when a sample is prepared in an

atmospheric condition, the sample surface is contaminated with organic materials in the atmosphere. C1s peak in the contamination is empirically known to be 284.6 ~ 285.0eV. While this method for charge-up correction using the contamination is an evil way, it is described in the following section.

A.1.2 Charge-up shift correction using contamination carbon

In Fig. A.1, after displaying C1s spectrum in the spectral window of 'MultiPak', 'Shift Setup' submenu has been selected from 'Data' menu. By clicking 'Shift Setup', charge-up correction window can be displayed as shown in Fig. A.2.

Here, C1s peak energy is assumed to be 285.0 eV while it is 284.6 ~ 285.0eV as described above. This value has been input in 'PkEnergy text box' in Fig. A.2 [p.21]. When C1s has

a single large peak, the green horizontal line indicating 285.0eV can automatically coincide with the peak by clicking ‘AutoShift button’ in Fig. A.2 [p.21]. The green line can also be moved horizontally by click&dragging it

such that it coincides C1s peak. The above operations can be cancelled by clicking ‘Undo button’ in Fig. A.2 [p.21].

The same value of charge-up shift as C1s can be applied also to other peaks by clicking ‘Exit button’ in Fig. A.2 [p.21].

Identification of chemical state

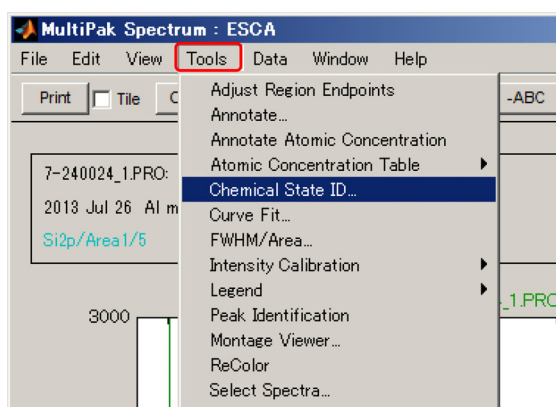


Figure B.1: Startup of the chemical state identification menu.

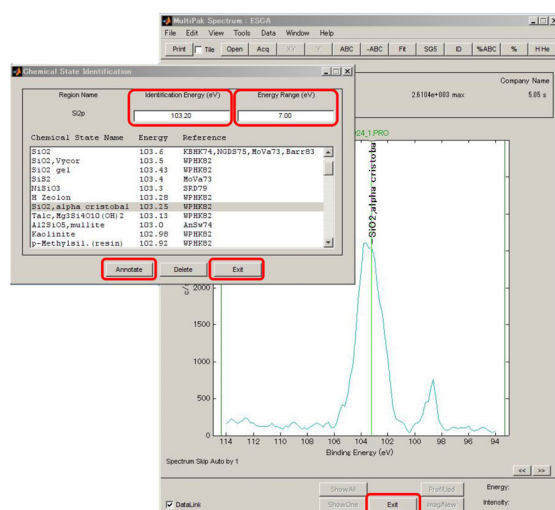


Figure B.2: Spectral peak assigned to SiO₂.

B.1 Identification of chemical state

B.1.1 Abstract of chemical-state identification

‘MultiPak’ has a data base in which peak positions of photoelectron spectra have been summarized. Spectral peaks observed with PHI5000 VersaProbe can be assigned by comparing them with those in the data base.

B.1.2 Start of chemical state assignment

In Fig. B.1, after displaying a spectrum in the vicinity of Si2p peak, ‘Chemical State ID’ sub-menu has been selected from ‘Tools’ menu in the spectral window of ‘MultiPak’.

By clicking ‘Chemical State ID’, ‘Chemical State Identification’ window on the left of Figs. B.2 and B.3 can be opened.

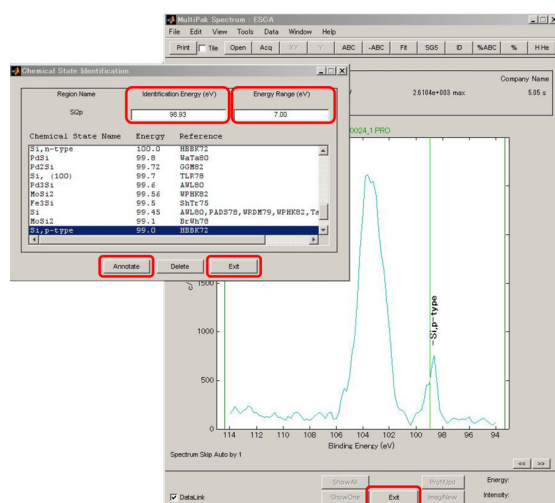


Figure B.3: Spectral peak assigned to p-type silicon.

B.1.3 Setting the range of spectral data

The value in ‘Identification Energy(eV)’ text box is the central value of spectral range and can be typed to be changed. However, default value is recommended in many cases. ‘Energy Range(eV)’ text box is energy range that can be typed to be changed and should not be too small such that spectral data far from the ‘Identification Energy(eV)’ are not looked over.

B.1.4 Identification of chemical state

Peak position in the data base can be displayed in the ‘Chemical State Identification’ window

by click&dragging the vertical green line to adjust it at spectral peak position. By clicking ‘Annotate’ button, the material name can be displayed on the top of the peak. This can be deleted by clicking ‘Delete’ button.

In Figs. B.2 [p.23] and B.3 [p.23], Left and right spectral peaks have been assigned to SiO₂ (α cristobalite) and p-type silicon, respectively.

By clicking ‘Exit’ button on the lower left of ‘Chemical State Identification’ window, it can be closed.

‘Exit’ button at the lower part of the spectral window can be clicked to finish the chemical-state identification mode.

Appendix C

Usage of NIST(National Institute of Standards and Technology) database

C.1 Access to NIST database

the users are recommended to use the database. In the homepage of NIST (National Institute of Standards and Technology) in the United States, there is a database of XPS that everyone can use. Since one of the merit of X-ray photoelectron spectroscopy is that many data

have been stored, The usage of database in 'Multipack' is described in Appendix B [p.23]. This is also recommended to use together with the NIST database.

By inputting 'XPS' and 'NIST' as keywords in Google as shown in Fig. C.1, URL of NIST XPS Database can be found as the first candidate as shown in Fig. C.2.

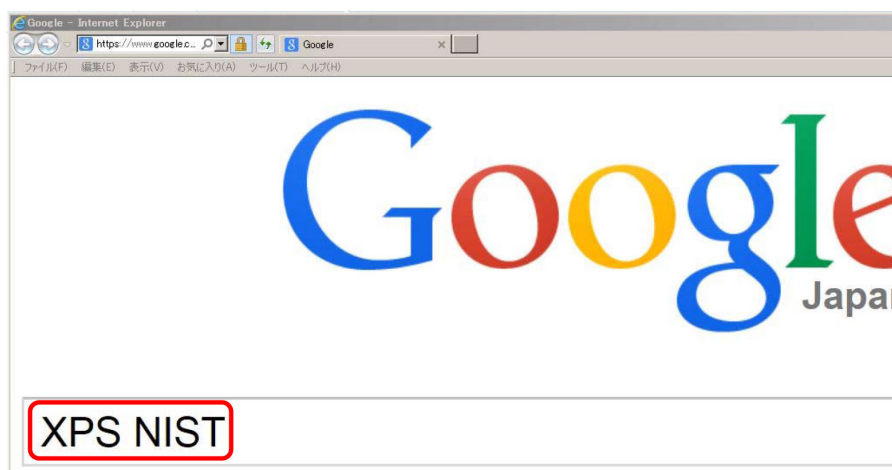


Figure C.1: Input 'XPS' and 'NIST' as key words in google

NIST X-ray Photoelectron Spectroscopy (XPS) Database ..

srdata.nist.gov/xps/ ▼ このページを訳す

The **NIST XPS** Database gives access to energies of many photoelectron and *A* electron spectral lines. The database contains over 29000 line positions, chemical shifts, doublet splittings, and energy separations of photoelectron and ...

Figure C.2: Click 'NIST XPS Database' found as the first candidate



Figure C.3: Click ‘Search Menu’.



Figure C.4: Click ‘Retrieve Data for a Selected Element’.

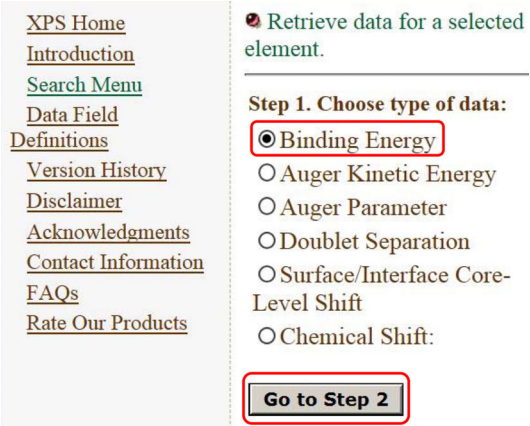


Figure C.5: Click ‘Go to Step 2’ with ‘Binding Energy’ checked.

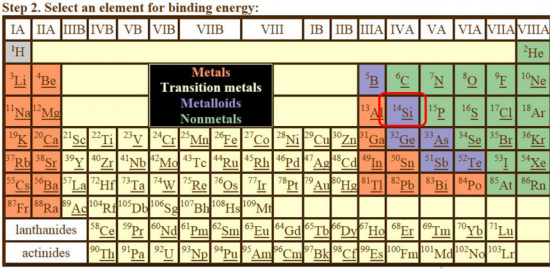


Figure C.6: Click an elemental symbol in the periodic table.

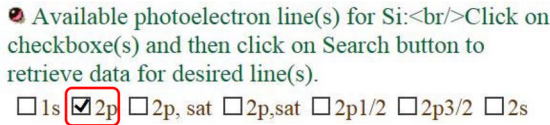


Figure C.7: Click ‘Search’ with a photoelectron peak checked.

Si	2p	SiC	100.80	Click
Si	2p	SiC	100.70	Click
Si	2p	SiC	100.40	Click
Si	2p	Si	99.20	Click
Si	2p	Si	99.60	Click
Si	2p	Si	99.30	Click

Figure C.8: Spectral peak position of Si2p photoelectron for elemental silicon.



Figure C.9: Authors and journal are found in the red frame.

Si	2p	(-Si(CH ₃)(C ₆ H ₅ O)-) _n	102.10	Click
Si	2p	(Si(CH ₃)(C ₆ H ₅ O)-O-) _n	102.10	Click
Si	2p	SiO ₂	103.80	Click
Si	2p	SiO ₂	103.70	Click
Si	2p	SiO ₂	103.40	Click
Si	2p	SiO ₂	103.20	Click
Si	2p	SiO ₂	103.50	Click

Figure C.10: Spectral peak position of Si2p photoelectron for SiO₂.

C.2 Usage of NIST database

C.2.1 How to search data in NIST URL

By clicking ‘NIST XPS Database’ in Fig. C.2, a window as shown in Fig. C.3 opens. Here, click ‘Search Menu’ to display Fig. C.4, please. Next, in Fig. C.4, ‘Retrieve Data for a Selected Element’ in a red frame should be clicked.

C.2.2 An example searching Si2p peak

In Fig. C.5, after checking ‘Binding energy’ radio button, ‘Go to Step 2’ button should be clicked to display Fig. C.6. ‘Si’ can be clicked in the periodic table to display Fig. C.7. ‘Search’ button can be clicked after checking ‘2p’ checkbox to display Fig. C.8

The fourth line in Fig. C.8 shows that Si2p photoelectron peak has been found at 99.20 eV for elemental silicon. ‘Click’ button in this line can be clicked to display Fig. C.9, which shows that this result was published in 1990 by J. Finster, E.-D. Klinkenberg, J. Heeg on page 1586 in *Vacuum* Vol.41.

By scrolling down from Fig. C.8, Fig. C.10 can be found. In the third line, it can be found that Si2p peak has been observed for SiO₂ at 103.80 eV. Here, click ‘Click’, please to display Fig. C.11. Fig. C.11 shows that this result was reported in 1996 by H. Kibel and P.-W. Leech in *Surf. Interface Anal.* Vol.24 p.605.

On the homepage of the library of Tokyo

General:	
Element:	Si
Formula:	SiO ₂
XPS Formula:	
Name:	silicon(IV) dioxide
CAS Registry No:	7631-86-9
Classes:	anhydride, IV-VI semiconductor, oxide
Citation:	
Author Name(s):	Kibel M.H., Leech P.W.
Journal:	Surf. Interface Anal. 24, 605 (1996)

Figure C.11: ‘Click’ in Fig. C.10 has been clicked to display the authors and journal.

Element	[1] Spectral Line	[2] Formula	[3] Energy (eV)	Details ?
Si	2p	Tm/Si	98.00	Click
Si	2p	Tm/Si	98.00	Click
Si	2p	GdSi ₃	98.10	Click
Si	2p	Si	98.40	Click
Si	2p	Si/Ba	98.40	Click
Si	2p	FeSi	98.60	Click
Si	2p	GdSi	98.60	Click

Figure C.12: ‘[1]Spectral Line’, ‘[2]Formula’ or ‘[3]Energy(eV)’ can be clicked to sort the data.

University, there are many electric journals. they can be downloaded on almost all computers in Tokyo University.

C.2.3 Sorting the data

The uppermost part of the database can be displayed by scrolling up it as shown in Fig. C.12. By clicking ‘[1] Spectral Line’, ‘[2] Formula’ and/or ‘[3] Energy (eV)’, they can be arranged in decreasing or increasing order.

End of the document.

Index

Symbols

*.map	17
*.pro	13
*.lin	1
*.map	1
*.pro	1
*.spe	1
*.sxi	1
'ID' button	2

A

Adjustment of background	4
Al ₂ O ₃	6
Analysis of photoelectron spectra	1
Apply to one band	6
Assignment of wide scan spectrum	2

B

Band limit setting window	6
Binding Energy	25, 27
Browse button	13

C

Chargeup correction	6, 16
Chemical state identification	7
Chemical state ratios	7
Corrected RSF	8
Curve fitting	i, 3, 4

D

Data base	27
Database	25

E

Elemental aluminum	6
Elemental silicon	25, 27
Excel	9, 11

F

File opening mode	13
File selection window	1, 13, 17
Fitting curves	4
Folder window	13

G

Gaussian curve	5
Google	25

I

ID button	i
Identification of chemical state	7, 16
Iterated Shirley	5

L

Least square fitting	4
List Files of Type: pull-down menu	13, 17
Lorentzian curve	5

M

Mapping	17
Molar ratio of chemical states	9
Molar ration of elements estimated from photoelectron spectra	3
Montage viewer	14
MultiPak	17

N

Narrow spectra	1
National Institute of Standard and Technology (NIST)	7
National Institute of Standards and Technology	25
NIST	25
NIST (National Institute of Standard and Technology)	7
NIST database	25, 27
NIST(National Institute of Standards and Technology)	25

P

Pass energy	8
Peak separation	3
Periodic table	25
PET	6, 7
polyethyleneterephthalate	6, 7
Profile window	13

Q

Quantitative analysis of elements from narrow spectra	2
---	---

R		<hr/>	
Reading spectrum data	1	Shirley	5
Refinement of peak separation	5	SiO ₂	25, 27
Relative sensitivity factor(RSF)	7	Spectral window	13
Retrieve Data for a Selected Element	25, 27	Sputtering	13
RSF(Relative sensitivity factor)	7	Survey scan (wide scan)	2
S		<hr/>	
Search Menu	25	Wide scan (survey scan)	2