UlvacPhi PHI5000 VersaProbe III with AES Part 1 Control software 'SmartSoft-VP' manual

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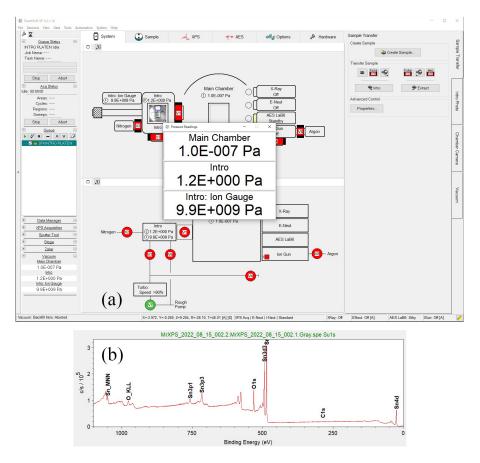


Figure 0: Window of 'SmartSoft-VP'

PHI 5000 VersaProbe III can be controlled with the software 'SmartSoft-VP'. This manual describes the usage of this software. The window can be changed by opening tabs on the upper side and the right side of Fig. 0 (a). Fig. 0 (b) shows photoelectron survey spectrum measured for a tin plate whose surface is oxidized. The contents are as follows, Chapter 1 [p.1] : setting of the 'Platen', Chapter 2 [p.3] : position correction of the crosshair, Chapter 3 [p.7] : registration of the measuring position, Chapter 4 [p.9] : how to obtain the photoelectron spectra, Chapter 5 [p.19] : photoelectron spectroscopy with sputtering, Chapter 6 [p.25] : SEM mode, Chapter 7 [p.29] : Auger electron spectroscopy, Chapter 8 [p.33] : Auger electron measurement with sputtering, Chapter 9 [p.37] : Auger electron line measurement and Chapter 10 [p.39] : Auger electron mapping measurement.

The sample stage sometimes has problems that can be resolved by initializing it. Appendix A describes how to initialize the sample stage.

2024.10.04_001 Kouhei OKITSU http://www.webpark1275.sakura.ne.jp/ DynamicalTheory/

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Creation of the 'Platen'

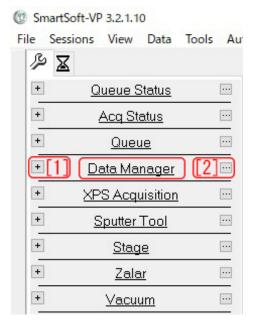


Figure 1.1: Menus shown on the left side of the window of 'SmartSoft-VP'

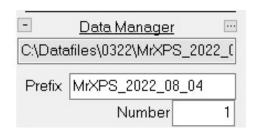


Figure 1.2: + can be clicked to open the 'Data Manager'

The following sequences are described under the assumption that the 'Platen' has been created and the photograph of the sample holder has been taken by referring to the Part0 manual.

Fig. 1.1 displays the menus shown on the left side of the window of 'SmartSoft-VP'. '+[1]' on the left side has been clicked to show Fig. 1.2. Further, ' $[2] \cdot \cdot \cdot$ ' on the right side can be clicked to show Fig. 1.3. Figs. 1.3 (a), (b), (c) and (d) have been opened by clicking the tabs of [a], [b], [c] and [d].

In Fig. 1.3 (a), 'Name [1]': Name of 'Platen' and '[2] Type': 60 mm sample holder mounted, are shown. '[3] Image Overlay' shows the file name of the photograph. If it is already set when taking the photograph of the sample holder, it does not have to be changed.

In Fig. 1.3 (a), 'Max Z Height [4]' is the highest limit of the sample stage that should not be changed.

In Fig. 1.3 (b), 'C:/Datafiles/ ··· [5]' show the name of data file with full path. 'Prefix

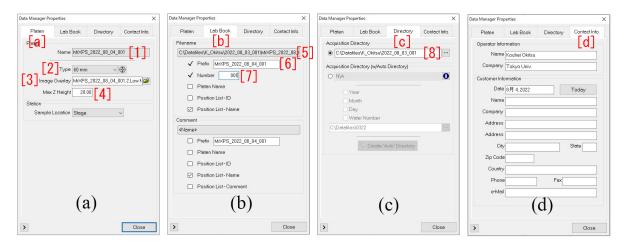


Figure 1.3: Details concerning the data name and folder

[6]' is the header of the data file names. 'Number [7]' is the data file number that increases automatically by repeating the measurement.

In Fig. 1.3 (c), 'Acquisition Directory [8]' is the name of the folder in which the measured data are automatically saved.

In Fig. 1.3 (d), the information concerning the operator can be described, However, this does not have to be described necessarily.

Position correction of the crosshair

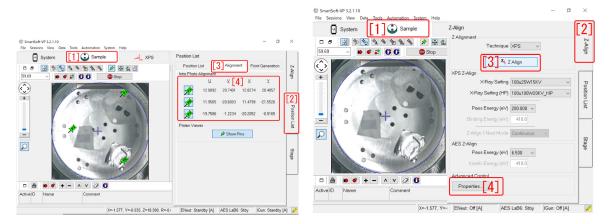
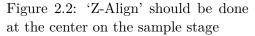


Figure 2.1: Position correction of the crosshair



In general, position of the crosshair is slightly different from the incident point of X-rays. There is no other way to set the measuring position except for setting it on the photograph of the sample holder. Then, the position correction of the crosshair is necessary for precise setting of the measuring positions on the sample holder.

In Fig. 2.1, tabs of '[1] Sample', '[2] Position List' and '[3] Alignment' have been clicked to open in this order. Position of PIN registered as shown in Fig. 2.1 should not differ from the practical position of the X-ray incidence. However, slight discrepancy is usually found. Then, the procedure that the present chapter describes, should be done before starting the experiment.

2.1 Z-Align

In Fig. 2.2, tabs of '[1] Sample' and '[2] Z-Align' have been clicked to open in this order just after the sample holder is introduced to the main chamber. As shown on the photograph of Fig. 2.2, the position of the blue crosshair is at the center of the sample holder. Here, '[3] Z-Align' button should be clicked such that the focus of incident X-rays is just on the surface of the sample holder.

Figs. 2.3 (a), (b) and (c) appear when the Z-Align is being done. Fig. 2.3 (a) shows the emission current of the Ar ion gun. This should not be closed until it automatically closes. Fig.

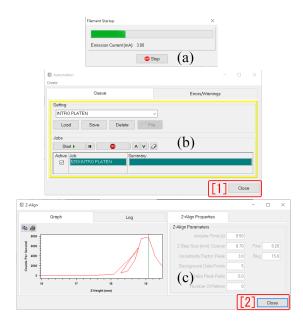


Figure 2.3: Window opened when starting the 'Z-align'

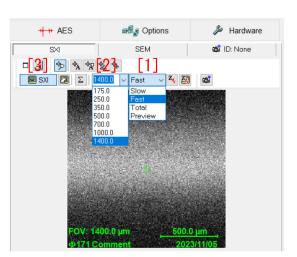


Figure 2.4: Start of SXI (Scanning X-ray Imaging)

2.3 (b) may soon be closed by clicking '[1] Close' just after this window appears. 'Properties [4]' on Fig. 2.2 can be clicked to show the graph whose abscissa and ordinate are the height of sample stage and the electron intensity as shown in Fig. 2.3 (c). When the peak is found and the vertical line appears, '[2] Close' can be clicked to go to the next operation.

2.2 SXI(Scanning X-ray Imaging)

At first in Fig. 2.4, '[1] Fast' and '[2] 1400' should be selected such that the image whose size is $1400 \times 1400 \ \mu \text{m}$ is refreshed with a rate of 1 shot / 1 sec. The SXI image can be clicked such that the clicked position is at the center of the view field. When the field size become very small and the SXI image disappears, '[2] 1400' can be clicked to select again for recovery.

2.3 Moving to the registered position

In Fig. 2.5 (a) 'mouse pointer [1]' can be right-clicked to show the pull-down menu. Then, '[2] Drive to Click' can be clicked to move the sample stage such that the X-rays are incident on the clicked position. In Fig. 2.5 (d), a big hole is found on the SXI image.

Fig. 2.6 shows the enlarged view of the photograph (left) and the SXI (right). At first on the SXI, the center of the hole should be clicked such that it is at the center of the SXI image. By clicking [2] + or scrolling the mouse wheel, the photograph of the hole can be enlarged. [3] can be right-clicked to open the pull-down menu in which 'Create point [4]' should be clicked.

2.4 Registration of the PIN positions

Arbitrary three PIN positions can be selected from the surface of the sample holder.

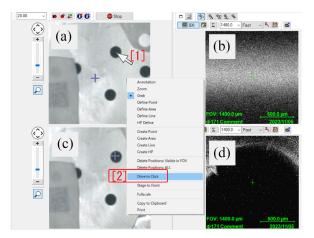


Figure 2.5: 'Drive to Click' has been clicked such that the hole is found on the SXI

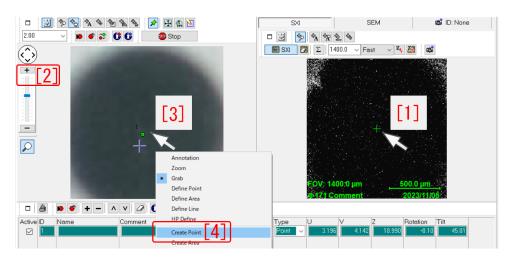


Figure 2.6: Central position of the hole should be adjusted at the center of the SXI (right) and the right-clicked position on the photograph (left)

Since the holes can easily be found on the sample holder, these are recommended to select as PIN positions. If the sight of the second position is lost, it comes to be difficult to find it again. The third position is easily found after the first and second position have been registered.

With Fig. 2.6 shown, [1]-[5] in Fig. 2.7 should be clicked in this order to register the position of (PIN1). After unchecking the three positions of PIN as shown in Fig. 2.7 [4], (PIN1) should be checked again as in the frame of [5] of Fig. 2.7.

As shown in Fig. 2.8 and 2.9, the second position (PIN2) and the third position (PIN3) can be checked in the same manner as for the first position (PIN1).

The discrepancy between the registered and practical positions is caused by the setting error when mounting the sample holder on the sample introduction fork. Then, the procedure described in this chapter should not be omitted.

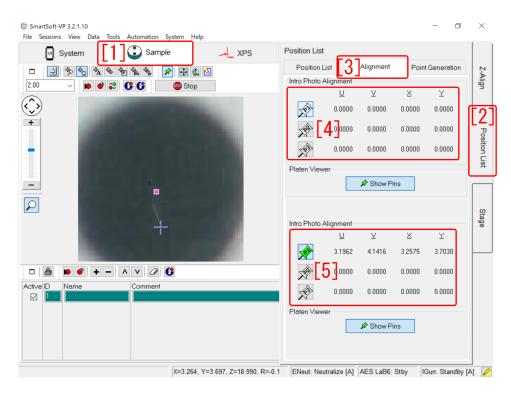


Figure 2.7: After unchecking PIN1, PIN2 and PIN3, PIN1 should be checked again

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Figure 2.8: After registering a second position, PIN2 should be checked

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Figure 2.9: After registering a third position, PIN3 should be checked

Registration of the measuring positions

Fig. 3.1 has been opened by clicking '[1] Sample' tab on the upper side. The scale of the image can be changed by clicking '[2] +/-' on the left side or scrolling the mouse wheel. Further, the central position of the image can be changed by click&dragging the image.

In the image of the photograph, any point can be right-clicked to show the pull-down menu. 'Create Point [3]' can be clicked to create the position. As shown in '[4]' of Fig. 3.1, its name and comments can be given. The position can be deleted by clicking '[4] -' in Fig. 3.3.

At first, for adjusting the height of the sample stage, the created position can be clicked to change the color to be green. '[2]' red button can be clicked to let the blue cross mark move to the created position. Next, '[5] Z-Align' tab can be clicked to open in Fig. 3.1 or 3.3. Then, '[6] Z-Align' button can be clicked to automatically adjust the height of the sample stage. '[7]' of Fig. 3.1 or 3.3 can be clicked to change the condition of incident X-rays. However, it does not have to be changed necessarily. With similar procedure, '[5] Z-Align' should be done for all

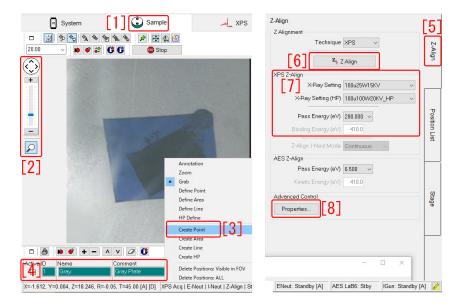


Figure 3.1: Position registration

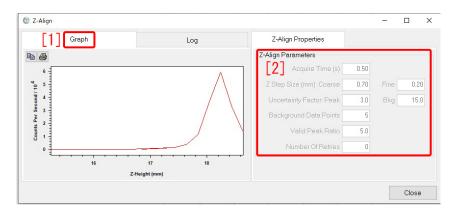


Figure 3.2: This window has been opened by clicking 'Properties [8]' in Fig. 3.1. '[1] Graph' on the upper left can be clicked to open the graph

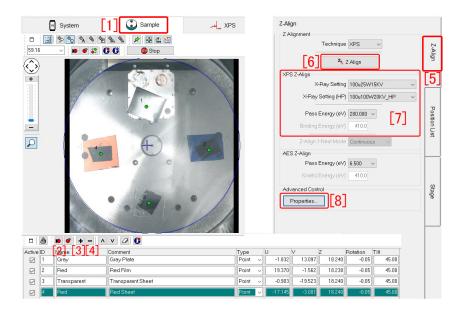


Figure 3.3: Four positions have been registered

created position.

In Fig. 3.1 or 3.3, 'Properties [8]' on the upper left can be clicked to show Fig. 3.2 whose ordinate and abscissa are the photoelectron intensity and the height of the sample stage, respectively., In '[2]' on the upper right of Fig. 3.2, parameters when doing the 'Z-Align' are shown.

[3] + can be clicked to add further measuring positions. <math>[4] - can be clicked to delete the registered positions.

Photoelectron spectroscopy

4.1 Normal (not using the queue) spectrum measurement

4.1.1 Setting of the survey spectrum measurement

Fig. 4.1 has been opened by clicking tabs of '[1] XPS' on the upper side and '[2] Spectrum' on the right side. '[3a] Position List' tab on the lower left can be clicked to summarize the registered positions.

In Fig. 4.1, 'XPS Setup [10]' on the lower right can be clicked to show Fig. 4.2. Figs. 4.2 (a) and (b) have been opened by clicking tabs of '[a]' and '[b]', respectively. In Fig. 4.2 (a), '[1]' and '[2]' have been checked such that the neutralizations with both electron and Ar ion are automatically done. In Fig. 4.2 (a) '[3]' has been checked automatically to adjust the height of

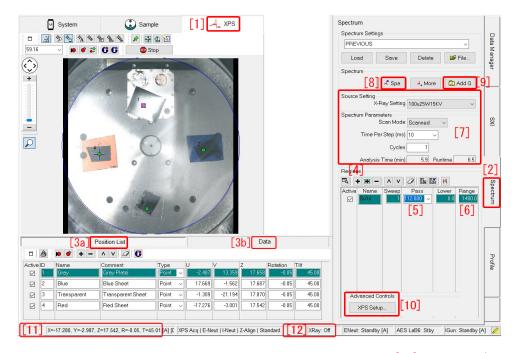


Figure 4.1: Spectra setting window. Sample stage information in '[11]' and ON/OFF of the incident X-rays in '[12]' are shown

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[a] XPS Setup Region		XPS Se	tup	[b] Reg	gion
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>	Close	224.000 1.000	0.8000 ~ 10000 ~ [4]	0.8000	2 1 Close

Figure 4.2: Acquisition setting window

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Li	Be		[2]									в	С	N	0	F	Ne
Na	Mg											AI	Si	Ρ	s	CI	Ar
к	Ca	Sc	Ti	۷	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	K
Rb	Sr	Y	Zr	Nb	Мо	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Те	Т	Xe
Cs	Ba	La	Hf	Та	w	Re	Os	Ir	Pt	Au	Hg	TI	Pb	Bi	Po	At	Rr
Fr	Ra	Ac															
			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr	

Figure 4.3: [Su] [2] can be clicked to set survey spectrum measurement

the sample stage (Z-Align) before the XPS measurement. If the Z-Align has already been done, '[3]' does not have to be checked.

From the pull-down menu of Fig. 4.2 (b) '[4]', energy scan step can be selected such as to be $(1/100 \sim 1/200) \times$ (pass energy).

In Fig. 4.1, [4] + can be clicked to show the periodic table of elements as shown in Fig. 4.3. If [1] Acquisition Setup' tab has been clicked to be opened, [2] SU' button is found on the periodic table. After clicking it, [3] OK' can be clicked to set the survey spectrum measurement (wide-range scan).

In Fig. 4.1, 'Pass [5]' and 'Range [6]' on the right side can be set by selecting from the pull-down menu or typing. The pass energy is the velocity energy of electrons flying in the orbit just between the inner and outer spherical electrodes of the electron analyzer. When the pass energy is set to be a large value, intensity of electrons captured with the detector increases but

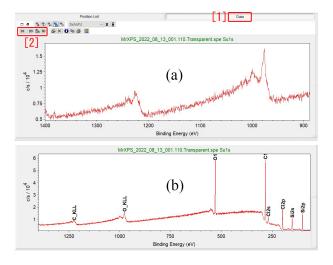


Figure 4.4: Survey spectra being measured

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[1] [2][3][4] [5][6]			

Figure 4.5: Enlarged view of the upper left of Fig. 4.4

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デスクトップ	MrXPS_2022_08_13_001.105.Blue.spe	2022/08/	13 20:17	SPE 7	7
「 ドキュメント	MrXPS_2022_08_13_001.106.Transparent.spe	2022/08/	13 20:20	SPE 7	7.
■ ピクチャ	MrXPS_2022_08_13_001.107.Red.spe	2022/08/	13 20:22	SPE 7	7
■ ビデオ	MrXPS_2022_08_13_001.108.Gray.spe	2022/08/	13 20:38	SPE 7	7.
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Figure 4.6: file selection window

yields poor energy resolution. Here, 'Pass [5]' (pass energy) has been set to be 112.0eV. The energy range has been changed to be 1400 eV from the default value (1100 eV).

On the right side of Fig. 4.1, parameters when measuring the spectra are set in the frame of (7). X-ray Setting '100 μ m25W15kV' means that the focus size and generating power of X-rays are these values. (8) SPE' on the upper part of Fig. 4.1 can be clicked immediately to start the survey spectrum measurement for the four checked positions shown at the lower left of Fig. 4.1.

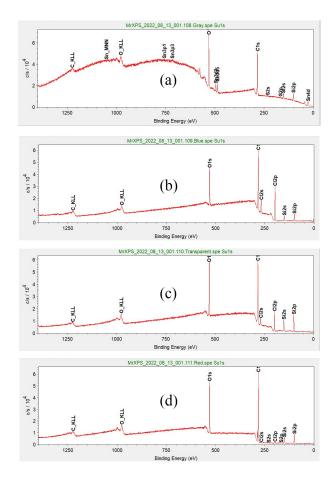


Figure 4.7: Survey spectra

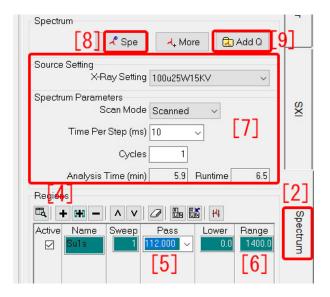


Figure 4.8: Enlarged view of the right side of Fig. 4.1

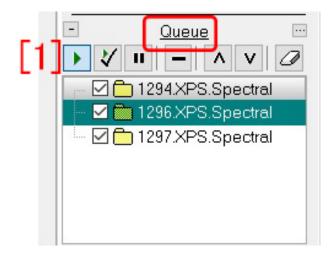


Figure 4.9: Queue window on the left side

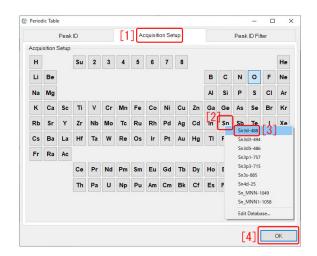


Figure 4.10: Setting of narrow spectrum measurement

4.1.2 Graph drawing of the survey spectra

In Fig. 4.1, '[3b] Data' tab can be clicked to open such that the spectrum being measured is plotted on the graph as shown in Fig. 4.4.

Fig. 4.5 is an enlarged view of the upper left part of Fig. 4.4. 'S9 [1]': smoothing, 'D9 [2]': differentiation and 'ID [4]': peak identification can be clicked to apply these operations for the spectra. '[3]' can be clicked to show the periodic table as shown in Fig. 4.3. The element symbol can be clicked to show peak positions of the photoelectron spectrum. '[6] \times ' in Fig. 4.5 can be clicked to delete the graph.

'[5]' in Fig. 4.5 can be clicked to open the file selection window as shown in Fig. 4.6. After selecting the file, 'Open' can be clicked to show graph of saved spectral data as shown in Fig. 4.7 (a)-(d).

Symbols of photoelectron peaks can be deleted by drag&dropping to out of the frame.

Fig. 4.7 (a), (b), (c) and (d) are survey spectra measured for 'gray', 'blue', 'transparent' and 'red' samples.

4.2 Spectrum measurement using the queue

Fig. 4.8 is an enlarged view of the right part of Fig. 4.1. 'Add Q [9]' button on the upper right of Fig. 4.8 can be clicked to add the measurement to the queue. [1] > button on the left side of Fig. 4.9 can be clicked to start the measurement by using the queue.

4.2.1 Setting of narrow spectrum measurement

Fig. 4.11 is an enlarged view of the lower left part of Fig. 4.1. Only 'Gray' sample has been checked such that the following schedule is applied only for this sample.

	[3a] Position List [3b] Data										
Active	₽	▶ ● + - Name	∧ v ⊘ ઉ Comment	Туре		U	V	Z	Rotation	Tilt	
	1	Gray	Gray Plate	Point	~	-2.487	13.359	17.658	-0.05	45.00	
	2	Blue	Blue Sheet	Point	~	17.669	-1.562	17.687	-0.05	45.00	
	3	Transparent	Transparent Sheet	Point	~	-1.309	-21.194	17.870	-0.05	45.00	
	4	Red	Red Sheet	Point	~	-17.276	-3.001	17.542	-0.05	45.00	

Figure 4.11: The lower left of Fig. 4.1 is enlarged (#1). First position has been selected

At first, '[4] +' button of Fig. 4.8 can be clicked to open the periodic table of Fig. 4.10. '[2] Sn' button has been right-clicked to open the pull-down menu. Spectrum regions are shown in a decreasing order of their integrated intensities. In Fig. 4.10, '[3] Sn3d' can be left-clicked to add this narrow spectrum measurement to the schedule. In similar way, the other spectral region can be added to the schedule. '[4] OK' on the lower right of Fig. 4.10 can be clicked to set this spectral region.

In Fig. 4.12, the second and third positions have been checked such that the same measurement schedule is applied to these two positions.

Fig. 4.13 is an enlarged view of the right part of Fig. 4.1. '[8] Source Setting' and '[9] Spectrum Parameters' in the frame of '[2]' of Fig. 4.13, have been opened by clicking '[4]+'. The

	3a Position List [3b] Data B 6 + A V Ø											
Active	ID	Name	Comment	Туре		U	V	Z	Rotation	Tilt		
	1	Gray	Gray Plate	Point	~	-2.487	13.359	17.658	-0.05	45.00		
	2	Blue	Blue Sheet	Point	~	17.669	-1.562	17.687	-0.05	45.00		
	3	Transparent	Transparent Sheet	Point	~	-1.309	-21.194	17.870	-0.05	45.00		
	4	Red	Red Sheet	Point	~	-17.276	-3.001	17.542	-0.05	45.00		

Figure 4.12: The lower left of Fig. 4.1 is enlarged (#2). Second and third positions has been selected

Spectrum					
[8]	🦨 Spe	<mark>⊿</mark> ₊ Mor	e 🗔	Add Q	1]
Source Setting	ay Settin	q 100u25W1	5KV/		
		100023001	UNV	~	
Spectrum Parame	can Mod	e Scanned	~		
	Cton (m			[2]	
Time Per	Step (m:		<u> </u>		
	Cycle	es 20	[3]		
Analysis	Time (mii	n) 11.6	Runtime	13.3	
Regions 4	5			[6]	
Regions 4	5 ^ v	0 6 6	1	[6]	
	5 ^ v	Pass	Lower	[6] Range	
■ + + -	5			[6] Range 20.0	
Active Name		Pass	Lower		
Active Name	1	Pass 27.000 ~	Lower 523.0	20.0	
Active Name		Pass 27.000 ~ 27.000 ~	Lower 523.0 278.0	20.0	

Figure 4.13: Settings of the spectral measurements

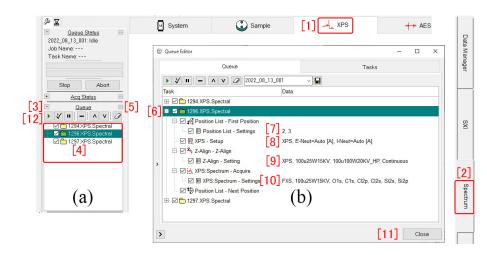


Figure 4.14: Verification and start of the queue. Settings of measurements for the second and third positions have been shown

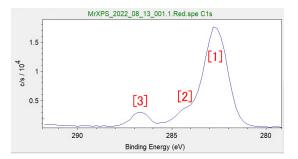


Figure 4.15: C1s photoelectron spectral peaks corresponding to different chemical states

setting is applied to the spectrum of 'Sn3d [7]'. 'Cycles 20 [3]' means that this measurement is repeated by 20 times. Times shown in '[6]' is the measuring time to complete the schedule. The 'Smart-Sift VP' unfortunately dose not have the function to estimate the total measuring time. Then, the time displayed on '[6]' should be added to estimate the total measuring time.

Fig. 4.13 ' $[[5] \land \lor]$ ' can be clicked to move upward or downward the selected blue spectral region. After completing the setting, Fig. 4.13 'Add Q [1]' can be clicked to add this setting to the queue.

4.2.2 Start of narrow spectrum measurement

In Fig. 4.14, [1]-[12] should be verified or clicked in this order. Tabs of '[1] XPS' on the upper side and '[2] Spectrum' on the right side have been opened. In Fig. 4.14 (a), '[3] –' on the left side of 'Queue' can be clicked to show that [1294], [1296], [1297] have been scheduled. [1294]: for gray sample, [1296]: for blue and transparent samples and [1297]: for red sample of the photograph of in Fig. 4.1, have been scheduled.

'... [5]' on the right side in Fig. 4.14 (a) can be clicked to show Fig. 4.14 (b). By clicking '[6] 1296 XPS Spectral', it can found that this schedule has been set for '[7]' for the second and third positions, [8] with automatic neutralizer settings for both electron and Ar ion, [9] with the

height of the sample stage adjusted automatically with X-rays before the spectral measurements in the vicinity of photoelectron peaks of [10] O1s, C1s, Cl2p, Cl2s, Si2s and Si2p with X-rays whose fucus size is 100μ m. After clicking '[11] Close' on the lower right of Fig. 4.14 (b), '[12] \triangleright ' in Fig. 4.14 (a) can be clicked to start the queue measurement.

Fig. 4.15 shows C1s spectrum taken for the 'red' sample. In this figure, the narrow spectrum cannot be clearly separated as shown in '[1]' and '[2]'. In such a case as this, a smaller value of pass energy can be set to separate the peak clearly with higher energy resolution. However, decreasing the value of pass energy causes the weak electron intensity. There is a trade-off relation between the electron intensity and the energy resolution.

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Sputtering measurement

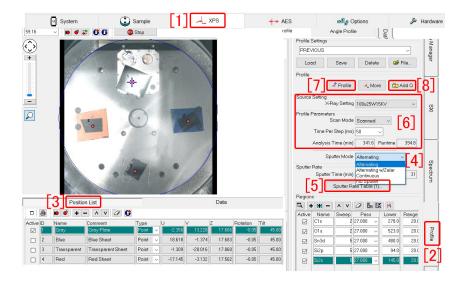


Figure 5.1: Sputtering setting window has been shown with '[1] XPS' tab on the upper side and '[2] Profile' tab on the lower right opened

5.1 Sputtering measurement

The XPS is a measurement for surface analysis to determine the kinds of elements whose depth from the sample surface is less than $2 \sim 3$ nm and their chemical states. However, depth profile of them can be obtained by repeating the measurement of XPS and Ar ion sputtering alternately. In this chapter, how to do the sputtering measurement is described to obtain depth profile of the elemental ratios and their chemical states.

5.2 Setting of the sputtering measurement

In Fig. 5.1, '[1] XPS' tab on the upper side and '[2] Profile' tab on the lower right have been opened. In '[3] Position List' on the lower left of Fig. 5.1, 'Gray'sample has been checked to do

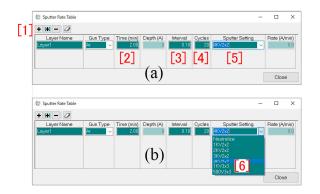


Figure 5.2: This window has been opened by clicking '[5] Sputter Rate Table' in Fig. 5.1

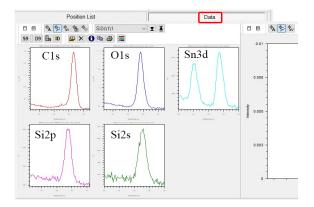


Figure 5.3: Survey spectrum before sputtering

the sputtering measurement whose schedule is set as shown on the right part of Fig. 5.1. Here, '[5] Sputter Rate Table' has been clicked to open as shown in Fig. 5.2.

The sputtering schedule can be added by clicking '[1] +' on the upper left of Fig. 5.2 (a). 'Time (min) 2.00 [2]' is the total sputtering time by repeating the sputtering of 'Interval [3] 0.1 min by 20 times ('Cycles [4] 20'). The sputtering voltage and scanning region (2×2 mm) of the focus of the ion beam on the sample is set to be 'Sputter Setting 3kV 2 × 2 [5]'. This sputtering condition has been set by choosing from the pull-down menu of Fig. 5.2 (b) [6]. Further sputtering schedule can be added by clicking '[1] +' on the upper left. The sputtering rate for SiO₂ has been estimated to be 7 nm / min in the case of 3kV 2 × 2.

In the frame of '[6]' of Fig. 5.1, conditions of incident X-rays can be set. '[7] Profile' can be clicked to start the usual measurement (not using 'Queue'). 'Add Q [8]' can be clicked to add the sputtering schedule to the 'Queue' (automatic measurement). The usage of the 'Queue' is described in §4.2 [p.14].

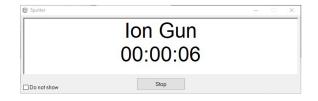


Figure 5.4: Timer shown during sputtering

	Data
	8 🔦 ୭ 🐦 🗙 🖨 📾 🛎
	MrXPS_2022_08_15_002.1.MrXPS_2022_08_15_002.1.Gray_1.pro
Intensity / 10 ⁴	Legend C1s O1s Sn3d Si2p Si2s O
	Cycles

Figure 5.5: Graph showing integrated electron intensity of each spectrum

5.3 Performing the stuttering measurement

5.3.1 Spectral measurement before the first sputtering

In Fig. 5.3, narrow range spectra around C1s, O1s, Sn3d, Si2p and Si2s photoelectron peaks are shown. The XPS spectra before the first sputtering (Presputter measurement) is usually taken with the default setting.

5.3.2 Display during the sputtering

Fig. 5.4 shows the timer displayed during the sputtering. It counts down the residual sputtering time.

5.3.3 Display during the XPS measurement

Fig. 5.5 shows the changes of integrated intensities of photoelectron spectra. This graph is displayed on the right side of Fig. 5.3. The integrated intensities of photoelectron spectra other than C1s have increased. The intensity of C1s peak due to the contamination materials in the atmosphere has decreased since the contamination has been removed by sputtering.

Fig. 5.6 (a), (b) and (c) show the photoelectron spectra that have been measured just after the first, second and third sputtering. The binding energy of the contamination carbon is known to be 284.8 eV. This energy value is frequently used for the energy calibration. In Fig. 5.6 (a), (b) and (c), it can be found that the integrated intensity of C1s has gradually decreased.

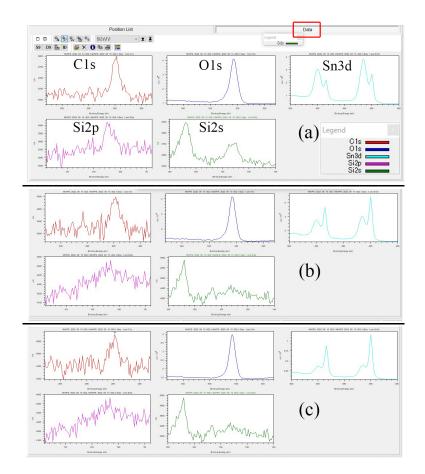


Figure 5.6: Spectra changing by repeating the sputtering. Narrow spectra (a) obtained after the first, (b) second and (c) third sputtering

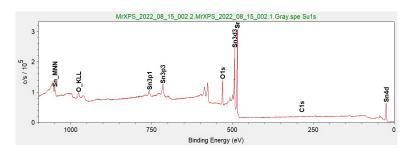


Figure 5.7: Survey spectrum measured after repeating the sputtering by three times

In Fig. 5.3, two peaks of Sn3d3 (left) and Sn3d5 (right) are found. In Fig. 5.6 (a), (b) and (c), these peaks are further separated to be two peaks respectively that can be assigned to be tin oxide and elemental tin. It can be found that the oxidized layer of the surface is removed by the sputtering and the integrated intensity of elemental tin increases gradually.

5.3.4 Survey spectrum measured after sputtering

Fig. 5.7 is a survey spectrum observed after repeating the sputtering by three times. The peak of C1s has disappeared. Peaks that can be assigned to Sn3d and O1s are strong. It has been clarified that 'Gray' sample is tin whose surface has been oxidized and covered with the contamination carbon.

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SEM(Scanning Electron Microscope)Mode

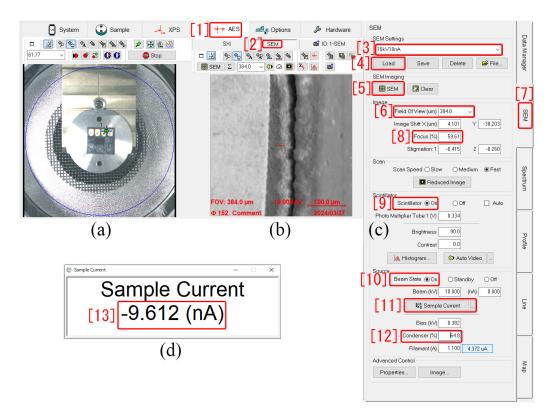


Figure 6.1: The initial window of SEM mode

6.1 Overview of the SEM mode

SEM (Scanning Electron Microscope) is the imaging mode for obtaining the Auger electron image of the surface of the sample. The 60 mm sample holder is not recommended to use for the SEM mode since it is easily affected from outer oscillation caused by the rotary vacuum pump

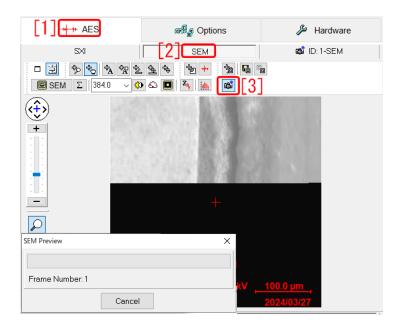


Figure 6.2: 'Camera mark [3]' has been clicked to take the photograph of the SEM image.

etc. Then, the 25 mm sample holder is recommended as shown in Fig. 6.1 (a). This sample is a part of electronic circuit on a printed board.

Here, the position correction of the blue crosshair should already be finished referring to the description in §2 [p.3]. Further, the Z-Align should be done to adjust the height of the sample stage as desribed in §2.1 [p.3].

6.2 Adjustment of the sample current

Fig. 6.1 has been opened by clicking the tabs of '[1] AES' on the upper side and '[7] SEM' on the right side of the window of the 'SmartSoft-VP'.

After selecting '[3] 10kV 10nA' from the pull-down menu, '[4] Load' should be clicked.

By clicking '[11]' Sample Current' on the lower right of Fig. 6.1, a negative value of electric current can be shown. After clicking the mouse on the textbox on the right side of '[12] Condenser', the mouse wheel can be scrolled to change the current. The current should be adjusted such that its absulute value is not over than 10 nm. Then, ' \times ' on the upper right should be clicked to close the window.

6.3 Start of the SEM and fucus adjustment

The secondary electron image can be displayed as shown in Fig. 6.1 (b) by checking radio buttons of '[9] Scintillator ON' and '[10] Beam State ON'.

By right-clicking the textbox on the right side of [8] Focus (%), the mouse wheel can be scrolled such that the surface of the sample is focused on and can be observed clearly.

The visual field size can be changed by selecting from the pull-down menu of '[6] Field Of View' and/or scrolling the mouse wheel with the textbox clicked. '[6] Field Of View 384.0 μ m' is the largest visual field (the lowest magnification).

6.4 Taking the photograph of the SEM image

By clicking 'Camera mark [3]' with the tabs of '[1] AES' on the upper side and '[7] SEM' on the right side clicked to open, the photograph of the SEM image can be taken.

The Auger electron measurement described from chapter 7 [p.29] - chapter 10 [p.39] are obtained for these area on the SEM image.

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Auger electron spectroscopy (AES) mode



Figure 7.1: Setting window of the Auger electron spectroscopy

7.1 Setting of the Auger electron spectroscopy

Fig. 7.1 has been opened by clicking '[1] AES' on the upper left and '[9] Spectrum' on the right side. In this figure, the photograph of the SEM image that has been taken in Fig. 6.2, is shown. Buttons of '[2,3,4]' should be clicked to make [2]: point, [3]: field or [4]: line on the SEM image. In the case of this figure, two field areas with yellow frames have been specified by click&dragging the mouse on the SEM image.

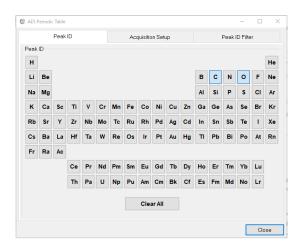


Figure 7.2: Auger electron peak position can be selected from the periodic table

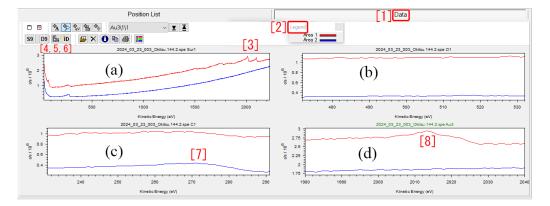


Figure 7.3: Measured Auger electron spectra, (a): Survey spectra, (b): around oxygen Auger electron peak, (c): around carbon Auger electron peak and (d): around gold Auger electron peak.

By clicking the tab of '[17] Position List', '[16]' can be shown. The SEM image on the photograph has been taken at the position '2'. the yellow regions of '2-1' and '2-2' correspond to the two rectangular fields. For these fields, Auger electron spectra are measured.

^{(7]} Time Per Step (ms) 20' is the electron counting time per point. The measurement whose schedule is set as shown in ^{(13]}, is repeated by the number of cycle (⁽⁸⁾ Cycle 1').

[10] Image Registration' should be 'off'.

(12) Periodic table button' can be clicked to show the periodic table of elements as shown in Fig. 7.2. The element symbol can be left-clicked to select the largest Auger electron peak. However, it can be also right-clicked to select an arbitrary Auger electron peak other than the largest peak from the pull-down menu. (11) - can be clicked to delete the selected Augerelectron peak.

'Sweep [14]' is the times of sweep. 'Resolution [15]' is the energy resolution that can be selected to be 0.05-0.4 eV from the pull-down menu.

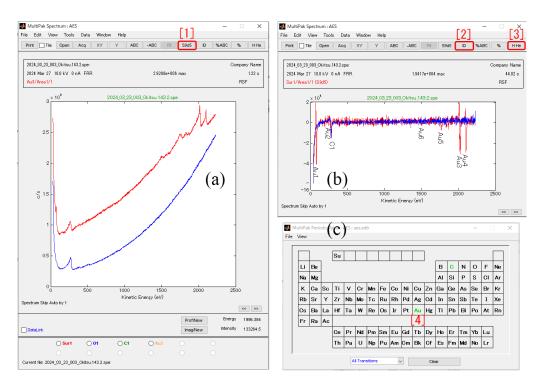


Figure 7.4: Auger electron spectra displayed on the windows of the MultiPak

7.2 Start of the Auger electron spectroscopy

'[6] Spe' can be clicked to start the measurement.

'[1] Data' on the upper right of Fig. 7.3 can be clicked to open for displaying the graphs of spectra. '[2] Legend' shows that the spectra measured for the left (Area1) and right (Area2) areas on the SEM image are drawn with red and blue curves. '[4,5,6]' on the upper right are as follows. '[4] D9' can be clicked to differentiate the spectra. '[5] Periodic table' can be clicked to display the periodic table of elements. '[6] ID' can be clicked to assign the differentiated spectra.

Around the position '[3]' in Fig. 7.3 (a) and the position '[8]' in Fig. 7.3 (d), gold Auger electron peaks are found. However, around the position '[7]' in Fig. 7.3 (c) a carbon Auger electron peak is found.

7.3 Display of the Auger electron spectra with MultiPak

The survey spectral curves in Fig. 7.3 (a) are read and drawn in the window of the Multipak as shown in Fig. 7.4 (a). '[1] S9d5' on the upper side of Fig. 7.4 (a) can be clicked to differentiate the spectra as shown in Fig. 7.4 (b).

^{(2]} ID' on the upper right of Fig. 7.4 (b) can be clicked to automatically assign the spectral peaks. However, ⁽³⁾ H He' can be clicked to display the periodic table of elements. ⁽⁴⁾ Au' on it can be clicked to show the positions of gold Auger electron peaks.

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Chapter 8

Auger electron sputtering measurement

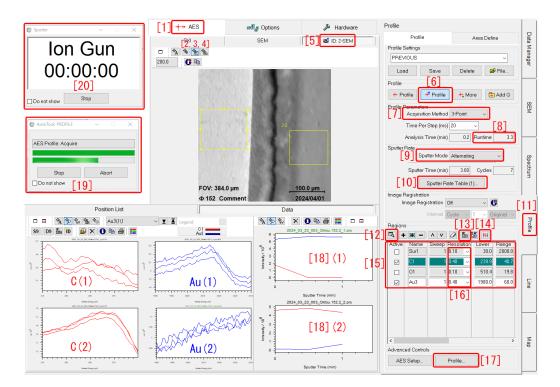


Figure 8.1: Setting window of the Auger electron sputtering measurement

8.1 Area setting of the sputtering measurement

Fig. 8.1 has been displayed by clicking the tabs of '[1] AES' on the upper left and '[11] Profile' on the right side.

'[5] ID:2-SEM' can be clicked to display the photograph of the SEM image. On it, one of '[2,3,4]' can be clicked to specify '[2] point', '[3] rectangle' or '[4] line' for the measurement.

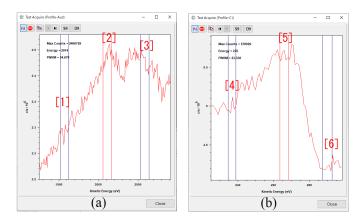


Figure 8.2: Settings of spectral region of sputtering measurement

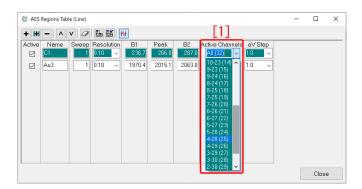


Figure 8.3: Settings of spectral regions of sputtering measurement

In this figure, '[3]' has been clicked to specify two rectangular regions surrounded by yellow frames for sputtering measurement. The sputtering and spectral measurements are repeated alternately.

8.2 Setting of the spectral regions of the sputtering measurement

In Fig. 8.1, after clicking '[13] Periodic table button', element symbols (Sur1, C, O, Au) have been clicked such that the spectral regions around these Auger electron peaks are measured. However, 'Sur1' and 'O1' have been unchecked not to be measured. The element symbol can also be right-clicked to select Auger electron peaks other than the largest peak from the pulldown menu.

Fig. 8.2 can be displayed by clicking '[14]' after selecting '3-points' from the pull-down menu of '[7] Acquisition Method' in Fig. 8.1. When 'Au3' or 'C1' is selected such as to be blue, Fig. 8.2 (a) or (b) is shown.

In each figure, the left, right and center (peak) spectral regions have been set. The vertical bars can be click&dragged horizontally to change the positions of the spectral regions. The widths of the regions can be changed by selecting values of 0.05-0.4 from the pull-down menu

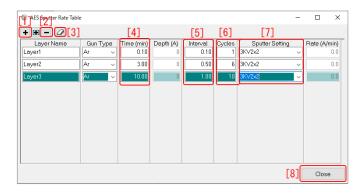


Figure 8.4: Setting of the sputtering schedule

of 'Resolution [16]'. Further, [12] in Fig. 8.1 can be clicked to open Fig. 8.3. In this figure, the number of channels can be changed by selecting from the pull-down menu of '[1] Active Channels'. [Center electron intensities] - [Mean value of the left and right intensities] are plotted in [18] (1) and [18] (2). of Fig. 8.1.

'[10] Sputter Rate Table' in Fig. 8.1 can be clicked to open Fig. 8.4 in which three sputtering schedules have been set. '[1]+' can be clicked to add another schedule. However, '[2]-' can be clicked to delete the schedules. Further, 'Eraser [3]' can be clicked to delete all schedules. '[4] Time' is the total sputtering time. '[5] Interval' is the sputtering time per cycle. '[6] Cycles' is the number of cycles of sputtering. The acceleration voltage of Ar ion can be selected from the pull-down menu of '[7] Sputter Setting'. When '3kV2 × 2' is set, the sputtering rate is 7nm / min for SiO₂. '2 × 2' means that the Ar ion beam is scanned for an area of 2 × 2 mm.

[6] Profile' in Fig. 8.1 can be clicked to start the sputtering measurement.

The spectra measured by repeating the sputtering are shown in C(1), Au(1), C(2) and Au(2) on the lower left of Fig. 8.1. The values of $I[2] - \{I[1] + I[3]\}/2$ and $I[5] - \{I[4] + I[6]\}/2$ of Fig. 8.2 (a) and (b) are plotted as the ordinates of [18](1) and [18](2) of Fig. 8.1. Here, I(x) is the Auger electron intensity at the spectral position x, where $x \in \{[1], [2], [3], [4], [5], [6]\}$.

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Chapter 9

Auger electron line measurement

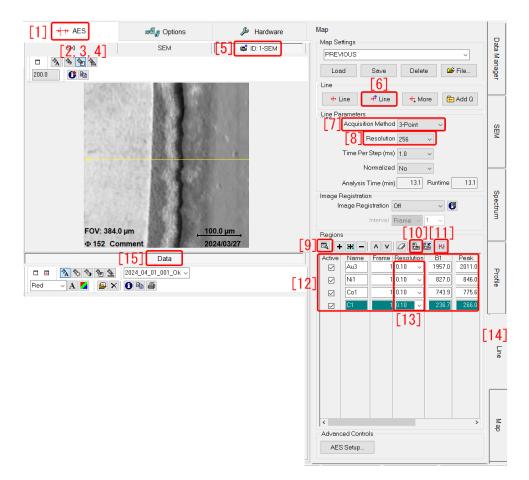


Figure 9.1: Window of the line measurement

9.1 Start of the line measurement

Fig. 9.1 has been displayed by clicking '[1] AES' on the upper left and '[14] Line' on the lower right.

On the SEM image displayed by clicking '[5] ID:2-SEM', '[2]: point', '[3]: rectangle' or '[4]: line' can be specified for measurement. For the line measurement, '[4]: line' should be clicked to draw a line on the SEM image.

The setting of [7-13] should be done in the same way as the mapping measurement. Then, refer to the description in Chapter 10 [p.39].

Chapter 10

Auger electron mapping measurement

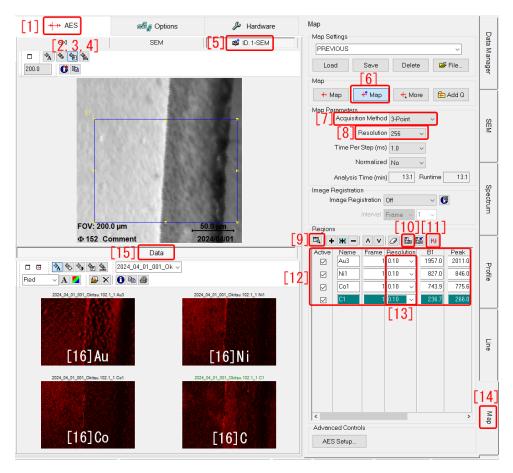


Figure 10.1: Setting window of the mapping

10.1 Start of the mapping measurement

Fig. 10.1 has been displayed by clicking the tabs of [1] AES' on the upper left and [14] Map' on the lower right.

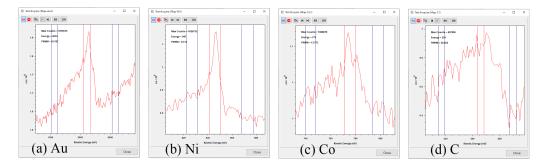


Figure 10.2: Spectral setting of the Auger electron mapping. The values after subtracting the backgounds from the peak intensities are used for mapping. The backgrounds are estimated from the intensities on the left and right sides of the paek

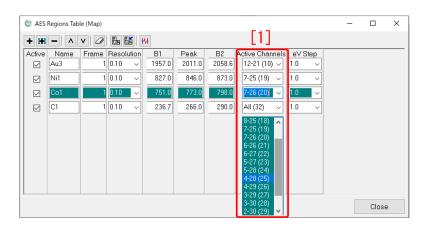


Figure 10.3: Spectral setting of the Auger electron mapping

By clicking '[5] ID-1:SEM', the photograph of SEM image taken in Fig. 6.2 [p.26] is shown. After clicking '[3]' of '[2,3,4]' on the upper left of Fig. 10.1, a rectangular region on the SEM image has been specified by click&dragging the mouse. The mapping measurement is done for this region.

10.2 Setting of the spectral region of the mapping measurement

At first, '3-point' should be selected from the pull-down menu of '[7] Acquisition Method' in Fig. 10.1. Then, number of pixels should be selected from the pull-down menu of '[8] Resolution'.

After clicking '[10] Periodic table' to open the periodic table of the elements, element symbols of (Au, Ni, Co, C) have been left-clicked to set spectral region around the Auger electron peaks as shown in Fig. 10.1 '[12]'. Element symbol can also be right-clicked to open the pull-down menu for selecting other Auger peaks. After clicking Au, Ni, Co or C to select such as to be green from '[12]', '[11]' can be clicked to open Fig. 10.2 (a) Au, (b) Ni, (c) Co or (d) C. In each figure, three spectral regions on the center (peak), the left and the right have been specified. The range of regions can be changed by click&dragging the mouse in Fig. 10.2. The width of regions can be set to be 0.05-0.4 by selecting from the pull-down menu of 'Resolution [13]'. Further,

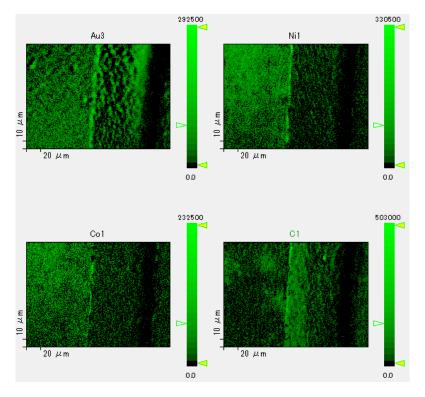


Figure 10.4: Display of mapping data with the Multipak

Fig. 10.1 '[9]' can be clicked to open Fig. 10.3. Then, the energy range can be changed to be smaller by selecting the number of channels from the pull-down menu of '[1] Active Channels'. The values of Auger electron intensities are estimated by subtaracting the background from the peak intensity. Maps of the Auger electron intensities for Au, Ni, Co and C are shown in [16]Au, [16]Ni, [16]Co and [16]C on the lower left of Fig. 10.1.

^{(6]} Map' in Fig. 10.1 can be clicked to start the mapping measurement.

10.3 Display of the mapping data with the MultiPak

Fig. 10.4 shows the mapping data on the window of the Mutipak. The usage of the Mutipak is described in the Part2 manual

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Appendix A

Initialization of the sample stage

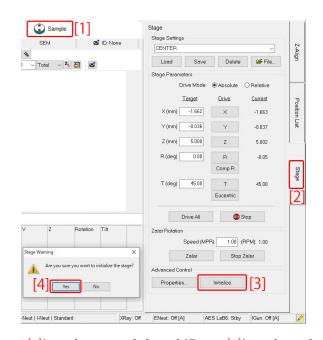


Figure A.1: With 'System [1]' on the upper left and 'Stage [2]' on the right side opened, 'Initialize [3]' on the lower right can be clicked to start initializing the sample stage. Before clicking '[4] Yes' on the lower left confirmation window, it should be confirmed that the sample introduction fork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

The sample stage of the VersaProbe III sometimes has trouble that can be resolved by initializing it. When starting the initialization of the sample stage, the sample introduction fork should not be inserted to the main chamber.

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

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

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

Please wa	ait			128.2	-395	×
<u>.</u>	Initializing motors: Tilt					
		on 2	Stop			

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

Stage Inf	ormation	×
0	Stage initialization is complet	e.
	ОК	

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

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