

# Guide to Operating the JXA-8530F EPMA: Data Acquisition and Analysis

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## I. Specimen Exchange

1. Removing the Specimen Holder
  - a. Click **OFF** button on the observation monitor screen to close valve *VI*.
  - b. Click the **Observation** and **Condition** buttons at the right of the *Observation* display and click the **Exchange Position** button under *SEM Monitor*.
  - c. Verify the **Exchange Position** button is *green* or the **EXCH POSN** lamp at the right side of the specimen-exchange chamber is lit.
  - d. Hold and pull the handle of specimen exchange rod upward so that the rod is held horizontally.
  - e. Push the handle until it stops and then turn it **90° counter clockwise** along the guide – the tip of the specimen-exchange rod joins to the specimen holder.
  - f. Pull the handle to relocate the holder from specimen stage to specimen exchange chamber.
  - g. Click the **VENT** button at the bottom right of the *Observation* display.
  - h. Unfasten the door clamp of specimen-exchange chamber and open the door, and then remove the holder adaptor together with specimen holder.
  - i. Close specimen-exchange chamber door so that dust does not enter inside.
2. Loading the Specimen Holder
  - a. Mount the specimen on the specimen holder.
  - b. Confirm that the specimen-exchange rod is kept horizontal.
  - c. Open specimen-exchange chamber door and fit the tip of specimen exchange adaptor together with specimen holder on the specimen exchange platform.
  - d. Make sure that there is no particles and dust on the O-ring and its mating surfaces of the specimen-exchange chamber.
  - e. Close the door and fasten the door clamp, and then click the **EVAC** switch at the bottom right of the observation display.
  - f. Wait until the vacuum of specimen chamber reaches **<5x10<sup>-4</sup>**.
  - g. Push the handle of specimen-exchange rod until it stops to relocate the holder from specimen exchange chamber to specimen stage and then turn it 90 degree clockwise along the guide.
  - h. Pull the handle to the full extent and pull the locking knob on the lock mechanism of the specimen-exchange rod while holding the handle of the rod, and then slowly lower the handle.
  - i. The **Select specimen holder** window appears in the **Observation** display. Select the specimen holder that you loaded in the specimen stage from the **Select specimen holder** dialog box, and then click the **OK** button.
  - j. Click **Home Stage** button to relocate the stage to home position.

## II. Observing Optical Microscope (OM) Images

*Before performing X-ray analysis with WDS, the analysis point on the specimen must be located at the focal point for the optical microscope.*

1. Observing the OM image
  - i. Use the **Stage Map** tab panel in the observation monitor screen or the trackball to move the stage to a position for observation.
  - ii. Click the **Om Monitor** tab in the analysis monitor screen of the EPMA monitor.
  - iii. Click the **OM lamp** in the **OM Monitor** tab panel.
  - iv. While observing the OM image, rotate the JOG dial to bring the OM image into focus.
  - v. Click the OM lamp button to turn off the illumination when you are ready to do image and X-ray analysis.
2. Saving the OM image

You can save the OM image in an image file. Clicking the capture icon displays the image selection dialog box, so save the image in a necessary directory.

## III. Observing SEI (Secondary Electron Image)

1. Adjust the stage height by turning the JOG dial on the trackball while checking the OM Monitor in the Analysis display, so that the OM image is approximately focused.
2. Select the accelerating voltage depending on the specimen and the purpose of observation.
3. Click the **ON** button on the observation monitor screen to open the gun valve.
4. Set the parameters in the operation panel as follows:

- i. IMAGE SELECT: SEM/SEI
- ii. SCANNING MODE:
  - a) PRB SCAN: ON (lighting)
  - b) QUICK VIEW: Q1 or Q2
5. Confirm that the probe current is about  $5.0 \times 10^{-9}$  A and **SE Detector** in the **Observation Condition** tab in the **Observation** display is **ON** (on).
6. Set **PCD** on the operation panel to “**OUT**”.
7. Click the **ACB** button in the **Observation** display to execute auto contrast/brightness.
8. Click the **AFC** button in the **Observation** display to execute auto focus.
9. Adjust the image focus using the **FOCUS** knob or the mouse.
10. Adjust the astigmatism of the image using the **Stig-X** and **-Y** knobs or the mouse.
11. Balance the contrast and brightness to adjust the image quality.
 

*Note: For high-resolution imaging,*

  - Set the probe current to  $10^{-11}$  to  $10^{-10}$  A using the **PROBE CURRENT** knob on the operation panel;
  - Use the objective lens aperture of 3.
12. Click the **Photo** button to take a photograph and save the image.

#### IV. Observing BEI (Backscattered Electron Image)

1. Observe an image of the specimen in the SEI mode and adjust the focus and stigmatism.
2. Press the **VIEW** button under **IMAGE SELECT** on the operation panel, and set the image mode to **COMPO** or **TOPO**.
3. Adjust the contrast and brightness to get high quality image.

#### V. EDS Analysis

##### 1. EDS qualitative analysis (Spectrum mode)

- i. Specify a folder to store the analysis data using the **Image File** tab in the Observation display.
- ii. In the Observation display, click the Observation button from the function buttons and move the position of the specimen to the analysis position using the **Stage Map** tab.
- iii. Set **PCD** on the operation panel to “**OUT**”.
- iv. Perform the focusing and astigmatism correction of the image.
- v. Select **Analysis – Option** from the menu bar.
 

*The Options dialog box appears, check the “The Project folder is specified at the time of creation” check box in the Options dialog box.*
- vi. Set the acquisition conditions for the EDS qualitative analysis:
  - a) Select **Analysis – Condition** from the menu bar.
 

*The Acquisition Condition dialog box appears.*
  - b) Set the process time of the spectrum measurement column to T3, and also set the Preset Value (measurement time) and other parameters. If you want to perform automatic element determination, select AUTO in ‘Quali, Analysis (qualitative analysis)’.
  - c) Right-click DT (dead time) in the EDS window in the Observation display.
  - d) Set the diameter of the X-ray limiting aperture to 1 (open).
  - e) Adjust the absorbed current using the PRB CURRENT knob on the operation panel so that the indication of **DT** becomes about 20 to 30% at a maximum.
- vii. Perform the focusing, astigmatism correction and image-quality adjustment of the observed image. Then set the scan speed to **Fine 1** or **Fine 2**, and press the **FREEZE** button on the operation panel.
 

*After pressing the FREEZE button, do not move the stage position.*
- viii. Click the **Analysis-EDS** buttons. The EDS spectrum display appears.
- ix. Click the **Spectrum** button.
- x. Register the analysis position.
  - a) In the case of point analysis:
    - 1) Click the **Point** button under **Analysis** position; then click an arbitrary position on the image.
    - 2) The window to enter the project name appears. Enter the project name.
    - 3) Repeat the same operation if you want more point analysis.
  - b) In the case of area analysis:
    - 1) Click the **Area** button under **Analysis** position; then drag an area you want to analyze on the image.
- xi. Click the **Start** button: *PCD is set to ‘OUT’ and EDS analysis starts from the preset numbers sequentially.*

- xii. When you want to display an EDS spectrum after finishing the analysis, click the corresponding ‘No.’ in the list of the analysis positions in the Data tab. An EDS spectrum appears in the spectrum window.
- xiii. If you want to print the analysis data, click the Print button at the right of the EDS window. A print preview appears. Image data and spectrum data are displayed in the present layout. You can select **SmileView** for the software to print the data.

**2. EDS area analysis (Map mode)**

- i. Search the field of view, then prepare for EDS analysis (Steps i to iv in Sect. a).
- ii. Select Analysis – Option from the menu bar.
- iii. Check the ‘**The Project folder is specified at the time of creation**’ check box in the Options dialog box.
- iv. Set the acquisition conditions for area analysis:
  - a) Select **Analysis – Condition** from the menu bar
  - b) Set the conditions for **MAP** column: you can obtain a good result by setting the high count rate mode (**T2**) for the **Process Time** of the area analysis.
  - c) Right-click **DT** (dead time) in the EDS window in the Observation display; the set the diameter of the X-ray limiting aperture to **1** (open) and the **Process Time** to the same value as **T2**.
  - d) Adjust the absorbed current using the **PRB CURRENT** knob on the operation panel so that the indication of **DT** becomes about **20 to 30% at a maximum**.
- v. Perform the focusing, astigmatism correction and image-quality adjustment of the observed image. Then set the scan speed to **Fine 1** or **Fine 2**, and press the **FREEZE** button on the operation panel.
- vi. Click the **Analysis – EDS** buttons
- vii. Click the **Map** button.
- viii. Click the Start button to start data acquisition.
  - ❖ When you click the **Qual.** (qualitative analysis) button during the data acquisition, the element determination is executed. Next, when you click the Play button, the area-analysis data of the determined element is displayed in real time.
  - ❖ Then when you click an arbitrary element in the **Periodic Table** and click the Play button again, the specified element map data are added to a real-time display of the data.
  - ❖ When you want to stop the analysis in halfway, click the Stop button.
  - ❖ After acquiring the data, you can reconstruct the mapping data of an arbitrary element. Also, you can reconstruct an EDS spectrum data obtained from an arbitrary area.

**VI. WDS Qualitative Analysis**

*In the qualitative analysis of all elements, normally four to five analyzing crystals with different lattice spaces are scanned simultaneously, and the existence of elements from B (or Be) to U is examined. With this method, you can obtain the information on both the constituent elements in the specimen and rough concentrations of the constituent elements through simple quantitative analysis.*

- 1. **Checking the configuration of analyzing crystals:** In JXA-8530F EPMA the crystals are configured as following:

Spectrometer	CH1	CH2	CH3	CH4	CH5
Crystal 1	PETJ	TAP	PETJ	LiF	PETH
Crystal 2	TAP	LDE2	LiF	PETJ	LiFH
Crystal 3	LDEB	none	none	none	none
Crystal 4	LDE1	none	none	none	none
Spect Range, mm	71 - 258 mm	62 - 258 mm	62 - 258 mm	62 - 258 mm	88 - 235 mm
Counter Type	Flow, P-10 Ar-CH <sub>4</sub>	Flow, P-10 Ar-CH <sub>4</sub>	Sealed, Xe	Sealed, Xe	Sealed, Xe

When taking into account both the efficient analysis of light elements and the total acquisition time, the conditions of the analyzing crystals are listed in the order of the wavelengths to be measured.

Analyzing crystal	Analyzing elements (range of analysis)
LIFH	Ti-K to Ga-K, REE (rare earth elements)-L line, etc.
PETJ	Si-K to Ti-K, Kr-L to Tb-L, U to M, etc.
TAPJ	F-K to Si-K
LDE1H	N-K to O-K
LDE2	B-K to C-K

- ❖ The analyzing ranges of these crystals overlap each other. Thus, decreasing the analyzing range a little enables you to shorten the total acquisition time.

## 2. Recognizing the analysis conditions

WDS requires relatively large probe currents (a few nA to a few  $\mu\text{A}$ ) for the analysis. In addition, the characteristic X-ray energies of each element range from as wide as 100 eV to 10 keV. Thus it is not easy to set the efficient conditions of qualitative analysis for detecting all analyzable elements. In order to efficiently analyze trace elements, it is necessary to set the optimum conditions (accelerating voltage and probe current) depending on the material (specimen).

Sample material	Accelerating voltage	Probe current
Mainly light elements	10 to 15 kV	-----
Mainly heavy elements	20 to 30 kV	-----
Metals, alloys, ceramics	-----	10 nA to 1 $\mu\text{A}$
Trace elements in metal	-----	100 nA to 10 $\mu\text{A}$
Susceptible to beam irradiation such as resin	-----	1 nA ~ 100 nA

## 3. Setting the analysis conditions

Before the analysis, set general analysis conditions that can be easily applied to various specimens. For example, if the specimen to analyze is SU, set the conditions of the qualitative analysis of all elements as follow: (accelerating voltage: 15 kV, Probe current: 100 nA, Probe diameter: 20  $\mu\text{m}$ )

Ch	Crystal	Start point [mm]	End point [mm]	Step [ $\mu\text{m}$ ]	Dwell time [ms]	SCA setting
5	LIFH	87	230	50	100	Default value
2	TAPJ	70	220	60	80	Default value
2	LDE2	100	220	200	400	Default value
3	PETJ	87	230	50	100	Default value
1	LDE1	90	155	200	400	Default value

In the qualitative analysis under the conditions in the above table, a guideline for the peak-position determination and element identification is about a few thousand ppm. When you want to identify trace elements, set the dwell time longer.

- i. If the user recipe or the recently-used recipe contains the existing analysis conditions, click the **Quick** icon button in the Analysis display, and then double-click these conditions. If not, click the **Qual.** icon button and set the new analysis conditions.
- ii. Check the **Project Name** and the **Path** to save data.
- iii. Click the **Add Spectrum** button *five times* and temporarily create the list of *five* **Spectrum Scanning Conditions**.
- iv. Select the **Detail** check box.
- v. Select each condition from the list and enter the parameters in the relevant boxes under **Detailed Setting** at the bottom left of the **Spectrum Scanning Condition** panel, such as **Crystal**, **Start [mm]** (start point) and **End [mm]** (end point). Then click the **Apply** button for each spectrometer channel to finalize these settings.
- vi. Clear the **Detail** check box after entering all parameters for the spectrum scanning conditions.
- vii. Check the **Execute Semi-Quant.** check box.
- viii. Set the **electron optics conditions**. For example, accelerating voltage to 15 kV and probe current to 100 nA using the **Observation display** (SEM monitor).
- ix. Check the stage height using the OM monitor after moving the specimen stage to the specimen holder position.
- x. Set PCD on the operation panel to 'OUT' and focus the image using SEI; then set PCD to 'IN'.
- xi. Click the **Read Now** button under **Electron Optics Condition** in the Qualitative analysis condition setting panel.
  - ❖ If the Auto Probe Current box is checked, the present electron optics conditions are automatically adjusted and measurement is executed.
  - ❖ If necessary, select the Detail check box to set the detailed electron optics conditions. Normally you do not need to perform the detailed setting of the electron optics conditions.

## 4. Saving the analysis conditions (Optional)

- i. Click the **Save Recipe** button.
- ii. Enter the parameters in the **Recipe name** box and the **Comment** box. Then specify the **Save place** and click the **OK** button.

## 5. Setting the analysis position and starting the analysis

- i. Determine a rough field of view using the **Stage Map** tab in the Observation display or the trackball, and adjust the specimen height using the OM monitor. Then, finally adjust the position of the specimen stage according to the magnification while observing SEI or BEI, and move the field of view for the qualitative analysis to the center of the observation screen.
- ii. Confirm that the **Stage** button is turned on in the **Analysis Position Condition** panel (simple panel), and enter the parameters in the No. box and the Comment box. Then sequentially click the **Read Position** button and the **Apply** button to save the analysis position.
- iii. Check the **Detail** check box. The panel is switched to the **Analysis Position Condition** panel. *In this panel you can independently select the scan mode (rectangular, circle) and the probe diameter for each stage No. When you want to set the analysis field in specific distance steps, select the **Line input** tab or the **Grid input** tab at the bottom of this panel. When you finish entering the parameters, clear the Detail check box.*
- iv. Return to the analysis start panel and click the **Acquire** button. The qualitative analysis starts and the progress of the analysis is displayed in real time.

## 6. Qualitative-Analysis Data Processing

- i. Click the **Epma Data** explorer.
- ii. Double-click the acquired qualitative-analysis data icon to open the file.
- iii. Changing the layout of the spectra data.
  - a) Select View-Layout from the menu bar. The layout changing tab appears.
  - b) Changing the layout using the menu boxes on the right.
- iv. Smoothing a spectrum.
  - a) Select Operate-Calculate... from the menu bar. The smoothing tab appears on the right.
  - b) Change the parameters for the smoothing using the menu boxes on the right.
- v. Enlarging a spectrum
  - a) Right-click the spectrum you want to enlarge and select Zoom.
  - b) Change the parameters for the zooming using the menu boxes on the right.
- vi. Peak identification and KLM marker
  - a) Select Operate – Peak ID or Operate – KLM Marker from the menu bar. The Peak ID and KLM Marker tabs appear.
  - b) In the Peak ID tab, clicking an arbitrary peak position displays the list of possible characteristic X-rays.
  - c) You can finely adjust the cursor position by using the arrow keys on the keyboard.
  - d) The elements that were already identified are displayed in yellow or blue.
  - e) When you select characteristic X-rays in the list and click the **Apply** button, the symbol of the element, which corresponds to the selected characteristic X-rays, is displayed.
  - f) Click an arbitrary element in the **KLM Marker** tab and confirm the position of the corresponding characteristic X-ray peak that appears in the spectrum.
  - g) As in the Peak ID tab, the identified elements appear in separate colors.
  - h) You may want to Save Identified Elements via Save ID Elements tab.
- vii. Semi-Quantitative Analysis
  - a) Select **Operate – Semi-Quant Analysis**; the Semi-Quant. Analysis window appears.
  - b) Select the identified elements, and then click the Apply button. The semi-quantitative results appear either in a separate window or on the spectrum window.

## VII. WDS Line Analysis

*In line analysis, the EPMA software program acquires the signal from WDS or EDS while scanning the stage or beam and display the line profile. The X-ray intensity data can be converted into concentration data using calibration curve data.*

1. If the line analysis conditions are available in a user recipe or a recently used recipe, double-click on those conditions. If the conditions are not available, click the Line button in the toolbar to set new conditions in the **Line Analysis** screen.
2. Confirm the project name and save path, making changes as necessary.
3. On the observation monitor screen, set the accelerating voltage and probe current, and then move the specimen stage to the analysis position while observing the SEI.
4. Use the jog dial to adjust the specimen height so that the image on the OM monitor becomes focused.
5. Perform the settings for the electron optics conditions

- i. In the observation monitor screen, adjust the focus and stigmator for the SEI and then click the **Read Now** button on the **Electron Optics Condition** panel.
6. Set the analysis position conditions
  - i. with **stage** scan
    - a) Two Points mode:
      - 1) Select the **Detail** check box in the **Analysis Position Condition** panel to open the analysis position condition detailed settings.
      - 2) Click the **STAGE** button and select the **Two points** button.
      - 3) Set the position for the start point (**Start**): after adjust the specimen height with the job dial while observing the image on the OM Monitor, finely adjust the stage position while observing the SEI or BEI with a scanning speed of 5 (**Fine1** button) or higher and move the center of the observation image to the start point for line analysis.
      - 4) Select **1:Start** from the list and click the **Register present stage position** button.
      - 5) Set the position for the end point (**End**) in the same way as *steps iii*.
      - 6) Select **2:End** from the list and click the Register present stage position button.
      - 7) Select the scan mode: default is Spot mode.
      - 8) Select whether to set priority with **Lock pixel** or **Lock size**.  
*When you give priority to the number of pixels, Pixels is fixed and Size [um] is set automatically, and vice versa.*
      - 9) Set **Dwell Time [msec]**, **Accumulation**, **Pixels**, and **Size [um]**.
      - 10) Click the **Set Scan** button, and then click the **Apply** button.
      - 11) To add an analysis position, select the analysis coordinate number No. 2 or a later number and repeat Steps ii to x.
      - 12) After completing the settings for all of the analysis position, clear the Detail check box.
    - b) Direction mode
      - 1) Click the **STAGE** button and click the **Direction** button.
      - 2) Set **Dwell Time [msec]**, **Pixels**, **Size [um]**, and **Accumulation**.
      - 3) Select **Point to set (Center/Start/End)** from the pull-down menu.
      - 4) Click the directional buttons to set the direction.
      - 5) While observing the image on the OM monitor, move the specimen stage to the position for the point selected by Point to set (Center/Start/End) in Step c.
      - 6) Click the **Register present stage position** button.
      - 7) Click the **Confirm** button to confirm the Z coordinates for the start, center, and end points, and perform registration again.
      - 8) Click the **Apply** button to register the analysis position conditions to the list.
  - ii. In **Beam** scan mode (*Magnification > 5,000×*)
    - a) Click the **BEAM** button.
    - b) Adjust the specimen stage position while observing the image and the OM monitor. Move the stage so that the center of the observation image is aligned with the center position for the beam scan.
    - c) Click the **Register present stage position** button.
    - d) Use the direction button to specify the direction for analysis.
    - e) Set **Dwell Time [msec]**, **Pixels**, and **Accumulation**.
    - f) Use the **Confirm** button to confirm the Z coordinates and perform registration again as necessary.
    - g) Set **Probe Tracking** as necessary.
    - h) Click the **Apply** button to register the analysis position conditions to the list.
7. Perform the settings for the **Analysis Element Conditions**:
  - i. Click the **Periodic Table** button to bring the Periodic table panel and Element panel to front. And then register the analysis element:
    - **Optimization**: Select the **Optimization** in the **Element** tab and click the button of the element that you use for measurement.
    - **Select from data base**: select **Select from data base** in the **Element** tab. Select the analysis element from the periodic table to display the analyzable X-rays for analysis in the list. Select the X-ray to use for analysis from the list. Click the **Apply** button to register the selected conditions into the analysis element list.
  - ii. Drag the atomic symbol into the analysis element list and change the measurement order or spectrometer channel for measurement.
  - iii. Perform peak search as necessary:

- a) Select the analysis element and click the **Peak Search** button to start peak search.
  - b) Click the **Rd** button to read the peak position.
  - c) Register the edited analysis element conditions into the database as necessary: Enter a **name** and click the **Add in the database** button to register the spectrometer channel, X-ray, analysis crystal peak position, and PHA conditions to the analyzing element database.
  - iv. When performing **EDS** analysis at the same time, select the EDS check box in the EDS column.
  - v. When acquiring **IMS** (image signals), perform the settings in the IMS column.
  - vi. After completing the settings for all of the analysis elements, clear the **Detail** check box.
8. Save the analysis conditions (recipe) as necessary.
  9. Click the **Acquire** button to start the line scan and the display appears in real time.

## 10. Line Analysis Data Processing

### i. Calibration Factor

You can specify the factors A and B when you convert the X-ray intensity image into concentration. If the data of standard specimen, quantitative analysis results, or semi-quantitative analysis results have already been obtained, you can set the factors A and B based on these data; otherwise, you can set them from the database set in the instrument (Cal-STD).

Factors A and B are defined as the following:

$$I = A * C + B$$

I: X-ray intensity

C: Concentration

Factor A: corresponds to slope and X-ray intensity per 1% concentration. Units are counts/(uA.ms.%). The current is the probe current measured by PCD.

Factor B: corresponds to thin section and background. Units are counts/(uA.ms). For image signals, the division by msec is not included.

### a) Method for setting factors A and B from standard specimen data

In order to obtain factors A and B from standard specimen data, you must acquire standard specimen data beforehand using the same channel, analyzing crystal, and accelerating voltage.

- 1) Select the element in the calibration factor dialog box and click the **Default...** button.
- 2) In the **Default...** dialog box, click the **...** button to specify the location (folder) of the standard specimen data.

*Select Cal-STD to set factors A and B from the database set for the instrument.*

- 3) Select the standard specimen data to use from the list and click the **OK** button. Factors A and B are set from the selected data.

### b) Method for setting factors A and B from quantitative (semi-quantitative) results

- 1) Select the element in the calibration factor dialog box and click the **Default...** button.
- 2) Click the **Read from Quantitative Results...** button.
- 3) Select **Quantitative Analysis** or **Semi-quantitative, Peak** or **Net**, and **Mass%** or **atomic%**.
- 4) Click the select **Group/Sample** button and select the quantitative results to use. *You must specify two quantitative results with different concentrations.*
- 5) Click the **OK** button in the Read from quantitative results dialog box.

## VIII. WDS Area Analysis (MAP)

*This section explains the procedure for performing the area (map) analysis by constructing its analysis condition from the beginning. The JXA-8530F that includes an EDS enables you to ordinarily perform WD/ED combined analysis. In addition, using the EDS, you can perform the spectrum mapping.*

### 1. Recognizing the analysis conditions.

You need to obtain the information on the specimen to analyze and the size of the analysis area in advance.

Specimen: e.g. Ceramic specimen

Specimen pre-treatment: e.g. cross-sectional mechanical milling after embedding in resin. About 10 nm thick carbon coating on surface.

Accelerating voltage: e.g. 15 kV

Probe current: e.g. 100 nA

Scanning method: e.g. stage scan

Number of points to analyze: e.g. 400x400

Pixel size: e.g. 0.5 μm x 0.5 μm

Analysis area: e.g. 200 μm x 200 μm (defined by the pixel size and numbers)

Dwell time: e.g. 20 ms

Elements and characteristic X-ray to analyze: e.g. Fe (Fe-Ka), Si (Si-Ka), Al (Al-Ka), O (O-Ka)

## 2. Setting the analysis conditions

- i. If the user recipe or the recently-used recipe contains the existing analysis conditions, click the **Quick** icon in the Analysis display, then double-click the saved recipe. If not, click the **MAP** icon button and set new analysis conditions.
- ii. Confirm the **Project Name** and the **Path** to save the data. If necessary, change these parameters.
- iii. Click the **Periodic Table** button.
- iv. Select the **Optimization** button; then click the WDS button and click the element to analyze.
- v. Click the element that has appeared in the analysis element list. The analysis conditions are displayed under **WDS parameter**.
- vi. (optional) Carry out the peak search for the selected element, and register the peak search result. Conduct peak searches of other elements if necessary.
  - When you want to acquire **EDS** data at the same time, check the **EDS** check box.
  - When you want to acquire **SEI** and/or **COMPO**, check the corresponding check boxes.
- vii. Set the accelerating voltage, e.g. 15 kV, and the probe current e.g. 100 nA, in the Observation display.
- viii. After moving the specimen stage to the specimen holder position, check the specimen height using the **OM** panel and focus the SEI.
- ix. Click the **Read Now** button under **Electron Optics Condition** panel.

## 3. (optional) Saving the analysis conditions

*When you register various analysis conditions by clicking the Save Recipe button, you will not have to set the parameters again the next time.*

- i. When you finish setting the necessary parameters for the analysis, click the Save Recipe button to save these parameters with an appropriate name.
- ii. When you want to use the same analysis conditions as the last time, call the saved recipe from the **EPMA Quick Start** panel, set the analysis position, and the start the measurement.

## 4. Setting the analysis position and starting the analysis

- i. Enter the parameters in the **Comment** box in the **Analysis Position Condition** panel. Then sequentially click the **Read** button and the **Apply** button to register the analysis position.
- ii. Click the **STAGE** button, then set the relevant parameters as follows:
  - Enhancing condition:** Normal Area
  - Pixels:** e.g. 400 x 400
  - Size:** e.g. 0.50  $\mu\text{m}$  x 0.50  $\mu\text{m}$
- iii. Sequentially click the **Read** button and the **Confirm** button.
- iv. Adjust the specimen height using the **OM Monitor** tab and click the **Register Z axis and go to next position button**.
  - v. Repeatedly carry out the Z axis correction for points B, C, D.
  - vi. When you finish the Z axis correction of the four points, sequentially click the **OK** button and the **Apply** button.
- vii. Click the **Start** button. The area (map) analysis starts and the progress of the analysis is displayed in real time.

## 5. Map Data Processing

- i. Double-click the map data icon you want to process in the data display panel in the **Epma Data** explorer to open the map data window.
- ii. Changing the layout of selected map data.
  - a) Select **Display – Display Attribute** from the menu bar to open the Property window.
  - b) Change the layout you want.
- iii. Changing the color levels
  - a) Select **Display – Level Modify** from the menu bar to open the color-level changing window.
  - b) Change the color level you want to.
- iv. Enlarging Map data
  - a) Select **Display – Zoom/Rotation** from the menu bar to open the **Scale/Rotation** window.
  - b) Select **Outline** from the **Scale/Rotation** window.
  - c) Drag the mouse over any area in any map (the area is highlighted); then click **Apply** button in the **Scale/Rotation** window.
- v. Analyzing Map data
  - a) Select **Operation – Map Analysis** from the menu bar to open the map analysis window.
  - b) Select the option you want to analyze and click **Apply** button to display the results.
- vi. Calibration Factor



You can specify the factors A and B when you convert the X-ray intensity image into concentration. If the data of standard specimen, quantitative analysis results, or semi-quantitative analysis results have already been obtained, you can set the factors A and B based on these data; otherwise, you can set them from the database set in the instrument (Cal-STD).

Factors A and B are defined as the following:

$$I = A * C + B$$

I: X-ray intensity

C: Concentration

Factor A: corresponds to slope and X-ray intensity per 1% concentration. Units are counts/(uA.ms.%). The current is the probe current measured by PCD.

Factor B: corresponds to thin section and background. Units are counts/(uA.ms). For image signals, the division by msec is not included.

a) Method for setting factors A and B from standard specimen data

In order to obtain factors A and B from standard specimen data, you must acquire standard specimen data beforehand using the same channel, analyzing crystal, and accelerating voltage.

- 1) Select the element in the calibration factor dialog box and click the **Default...** button.
- 2) In the **Default...** dialog box, click the **...** button to specify the location (folder) of the standard specimen data.  
*Select Cal-STD to set factors A and B from the database set for the instrument.*
- 3) Select the standard specimen data to use from the list and click the **OK** button. Factors A and B are set from the selected data.

b) Method for setting factors A and B from quantitative (semi-quantitative) results

- 1) Select the element in the calibration factor dialog box and click the **Default...** button.
- 2) Click the **Read from Quantitative Results...** button.
- 3) Select **Quantitative Analysis** or **Semi-quantitative, Peak** or **Net**, and **Mass%** or **atomic%**.
- 4) Click the select **Group/Sample** button and select the quantitative results to use. *You must specify two quantitative results with different concentrations.*
- 5) Click the **OK** button in the Read from quantitative results dialog box.

## IX. Standard Specimen Analysis

*Standard specimen analysis is necessary for the quantitative analysis of the constituent elements in the target specimens.*

1. Recognizing the analysis conditions

For the standard specimens, you can select the pure-element standard specimens. When you finish selecting the standard specimens, you need to determine what kinds of characteristic X-rays to use for the analysis. The most reliable method is to acquire qualitatively-analyzed spectra of the unknown specimens and examine the presence of the spectral overlap from the constituent elements.

2. Setting the analysis conditions

- i. Place a standard specimen, which is necessary for the quantitative analysis, on the specimen holder.
- ii. Set the electron optics conditions using the Observation display. For example, accelerating voltage: 20 kV, probe current: 10 nA.
- iii. Click the **Std** (standard specimen) icon button in the Analysis display. The **Standard Specimen Analysis** panel appears.
- iv. If necessary, change the **Project Name** and the **Path** to save the data.
- v. Click the **Read Now** button in the **Electron Optics Condition** panel.
  - a) Using the **Stage Map** tab in the **Observation** display, move the specimen stage to the position of the standard specimen and then adjust the specimen height while observing the **OM Monitor** in the Analysis display.
- vi. Click the **...** button in the **Standard Analysis Condition** panel. The standard specimen data management panel appears.
  - a) When you want to register a new group, click the **+** button at the lower left of the standard specimen data management panel; then enter a new name.
  - b) Click the **Add** button.
  - c) Register the standard specimen data. For example:
    - 1) Standard (specimen) name: *e. g. Fe* (arbitrary name)
    - 2) Comment: (Arbitrary)

- 3) Compositional information: *e. g.* Metal, Mass%
- 4) Element/Value: *e. g.* Fe/100.0

When a standard specimen is a compound, if the abundance ratio of each element is the same as its chemical formula, you can enter the data in the following way:

- 1) Standard (specimen) name: *e. g.* CuFeS2 (arbitrary name)
- 2) Comment: (arbitrary)
- 3) Compositional information: *e. g.* Metal, Atom ratio
- 4) Element, Value: *e. g.* Cu 1 Fe 1 S 2

- d) Check the position of the standard specimen by the observation of the **OM Monitor**, **SEI** or **BEI**, and then click the **Read** button.
  - e) Click the **Successive Input** button and repeat steps c) and d) to register other element data.
  - f) When you finish entering the data, click the **OK** button. The standard specimen data management panel appears.
- vii. From the list of registered elements, select the data of the element, *e.g.* **Fe** and click the **OK** button.
- viii. If necessary, select the **Detail** check box in the **Standard Analysis Condition** panel and set the detailed analysis conditions.
- a) Peak search: *Always* (conditions of each channel are preferred)
  - b) Background (measurement): *Always*
  - c) Method of WDS movement: *Asynchronous* measurement.
- ix. Move the specimen stage to the position of the standard specimen, and finely adjust the specimen height using the OM Monitor. Then, sequentially click the **Read Position** button and the **Apply** button in the **Analysis Position Condition** panel – the specimen position is registered.
- x. If necessary, check the **Detail** check box; then determine the **electron-probe diameter** and the **number of accumulations** for the stage position.
- xi. Set the parameters in the **Analysis Element Condition** panel.
- a) Click the **Periodic Table** button in the **Analysis Element Condition** panel. The periodic table tab appears.
  - b) Open the **Element** tab, click the **Select from data base** button, and click the element standard, *e.g.* **Fe** in the **Periodic Table**. The list of the analyzing crystals that can analyze characteristic X-rays for the element standard, *e.g.* Fe is displayed.
  - c) Double-click one in the list to select the conditions.
  - d) As steps a) to c), register the necessary parameters for the spectrometer channel, X-ray and crystal. **Detail** check box enables you to finely change the analysis conditions. The default values of the main analysis conditions are:
 

Peak position:	Theoretical value of characteristic X-rays is corrected
Background position:	<i>e.g.</i> : +5 mm, -5 mm
Measurement method:	Fixed Time method
Peak measurement time:	<i>e.g.</i> : 10 s
Background measurement time:	<i>e.g.</i> : 10 s
Number of peak search:	1
PHA conditions:	Initial values of analyzing crystals set by service engineer
  - e) Click **Save Recipe** button if you want to register the analysis conditions for future use.
- xii. Click the **Acquire** button to start the standard specimen analysis.
- ✚ When you want to serially perform the analysis of the standard specimens, such as Ni and Cr.
    - a) Click the **Add Series Analysis** button and the analysis conditions are registered.
    - b) Set the analysis conditions of other standard elements, such as Ni and Cr according to the procedure in steps above; then click the **...→Start** button to start series analysis.

## X. Quantitative Analysis

### 1. Recognizing the analysis conditions.

The overall analysis conditions were determined in the standard specimen analysis, but it is necessary to set more detailed analysis conditions in the actual quantitative analysis of unknown specimen. For example:

Accelerating voltage:	<i>e.g.</i> 20kV
Probe current:	<i>e.g.</i> 10 nA
Probe diameter:	<i>e.g.</i> 20 um
Analysis time:	<i>e.g.</i> peak position 10 s
	<i>e.g.</i> Background position 5 s
Quantitative analysis method:	<i>e.g.</i> Metal ZAF method

## 2. Setting the analysis conditions.

- i. If there exist analysis conditions, click the **Quick** icon and then select a desired condition from the list of the analysis conditions in the EPMA **Quick Start** panel.
  - ✚ When you want to set a new condition, click the **Quant** icon in the Analysis display. The quantitative analysis condition setting panel appears.
- ii. Confirm the **Project Name** and the **Path** to save the data. If necessary, change these parameters.
- iii. In the **Quantitative Analysis Condition** panel, select ‘**Metal**’ in the **Materials** box and select ‘**ZAF**’ in the **Correction** box.
- iv. Using the *Observation* display toolbar, set the accelerating voltage, e.g. **20 kV** and the probe current, e.g. **10 nA**; then click the **Read Now** button in the **Electron Optics Condition** panel.
- v. In the **Analysis Element Condition** panel, determine the necessary parameters, such as the elements to analyze, the spectrometer to use and the analyzing crystals according to your *Qualitative* analysis.
  - a) *If the analysis conditions are not registered in the database*, activate the **Periodic Table** tab and the **Element** tab, and then sequentially click the **Optimization** button and the symbol buttons for the elements to analyze.
  - b) Drag the symbols of the elements in the list and change the order of measurements or the spectrometer channels to use for the measurements.
  - c) *If there are already-registered analysis conditions*, click the **Select from data base** button in the **Element** tab, select desired analysis conditions from the list of the database, and then click the **Apply** button. *Note: when you carry out this step, you can omit the peak search.*
  - d) Set the detailed analysis conditions of each element, such as background **Upper** and **Lower** positions, *standard specimen data*, etc.
- vi. Set the parameters in the **Analysis Position Condition** panel.
  - a) Enter the parameters in the **Comment** box.
  - b) Click the **STAGE** button and check the specimen position using the trackball or by the observation of the **SEI** and **OM Monitor**. Then, sequentially click the **Read Position** button and the **Apply** button.
  - c) If necessary, select the **Detail** check box to change the **probe diameter** and **positions** that contribute to one spectrum.
- vii. Confirm the status of the **No.** check boxes, and then click the **Acquire** button. The analysis starts.

## 3. Quantitative-Analysis Data Processing

- i. Normal display: double-click the quantitative-analysis data icon in the data selection panel in the **EPMA Explorer**. A text window, called “Normal Display”, appears.
- ii. Obtaining the statistics of multiple data sets
  - a) Click the **Summary** button in the **EPMA Explorer**. The quantitative-analysis summary window appears.
  - b) Select **File-Open** from the menu bar. The **Select Group/Sample** window for quantitative analysis summary appears. Click the **Home** button and select the directory that corresponds to the **Recipe Name**. Then select a group in the **Group** list box and a sample in the **Sample** list box, and the click OK button to display the summary window and the list of the quantitative results.
  - c) Displaying each parameter in text format: select **Mass%** in the **Parameter** box in the quantitative-analysis summary window, and select the data you want to display in text format by checking the **No.** check boxes of these data. Then, select **Row** under **Order** and **Data&Ave.** under **Format**. Finally, click the **Type out** button.
  - d) Exporting the data of each parameter.

## XI. After analysis is done

1. Follow **section I, 2a-j** to remove your sample, re-load the specimen holder, and re-evacuate the specimen chamber.
2. Log off of your instrument session through your NCF FOM account.