USER GUIDE – JEOL JSM-6300 SCANNING ELECTRON MICROSCOPE

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1. BASIC SEM UNIT

The JSM-6300 basic unit (Fig 1) consists of an electron optical column mounted on the main console, a control and display system, a power supply unit, and a pump box. The main console incorporates a vacuum system, and the control and display system incorporates the control panels, keyboard, and display system. The power supply unit is placed at the back of the control and display system. The unit is controlled by an SCO-UNIX-based Scanning Electron Microscope control system designed to make all SEM functions, from microscope operation to image acquisition, manipulation, and storage relatively simple and easy. The basic SEM is connected to an EDX unit, which allows a characteristic X-ray spectrum to be displayed (Fig. 1). Auxiliary attachments, such as a cathodoluminescence (CL) detector, have also been added.

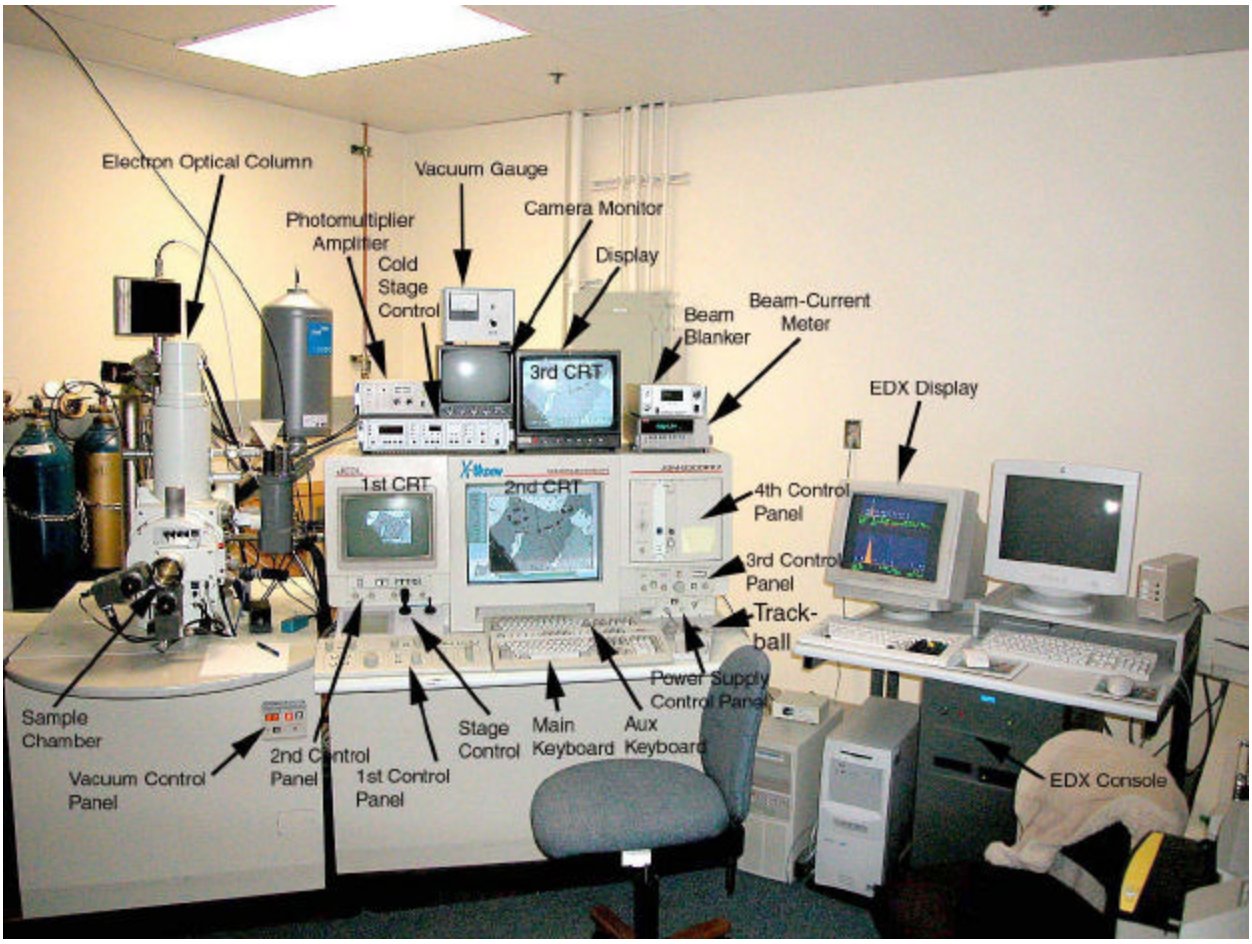

Figure 1. Basic JEOL SEM unit and auxiliary equipment

2. LOGIN PROCEDURE

The first screen that appears in the X-Vision (computer software) system (2nd CRT) is the SCO login windows. [Hit space bar or touch the trackball to turn on the screen] To log in:

Type your login name, using only lower case letters

Press <ENTER>

Type your password
Press <ENTER>

Click on the JSM6301F control icon (see Fig. 15)., which brings up the CONTROL SYSTEMS MENU. Then click on FILE, then EOS PARAMETERS. Click on FILE, LOAD PARAMETERS. Select FILE, then the required parameters from the parameters file, including accelerating voltage. Click OK, then APPLY. Turn on the 3rd CRT to view parameters on screen.

From the DIRECTORY icon (left side of screen), set up date, etc. Either click on "OK" or press <ENTER>

3. OPERATION

Two operational modes, direct operation using knob/switch controls and operation through the keyboard and trackball, are available for the SEM. The switch/knob control mode is convenient for dynamic control and observation. Keyboard control allows the operating parameters to be specified from the basic screen, EOS menus, command window (for selection or entry of commands and arguments, refer to the separate volume, Commands/Messages), parameters menu, or automated function menu. The FIS menu is used for imaging processing (to be discussed).

Some items of keyboard control are linked to knob or switch control and others are completely independent.

3.1 Starting Up

Initial startup procedures (opening nitrogen cylinder, turning on cooling water, etc.) are normally performed by the lab manager (John Donovan). [These procedures are described in Section 5.1 of the JEOL operating manual.]

When evacuation is completed and the column reaches the pressure required for high-voltage application and image observation, the EMISSION-CURRENT meter lamp on the 3nd control panel (Fig1; Fig. 5) lights up (about 20 minutes after startup). Proper vacuum has been achieved when the vacuum gauge (Fig. 2) reaches the 10^{-5} Torr range, with the vacuum-gauge knob turned all the way to the right.

Figure 2 Vacuum Gauge

3.2 Sample Loading

Sample loading is made through the specimen exchange chamber, or airlock chamber (Fig. 3), by using the specimen exchange rod, which is integrated with the specimen exchange cap.

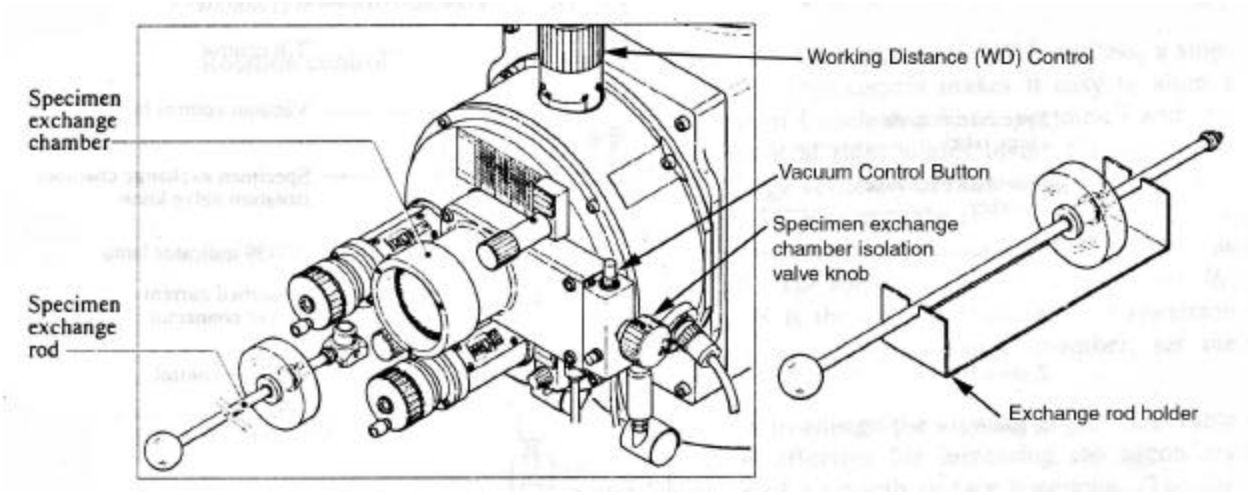

Figure 3 Specimen exchange rod and specimen chamber

Note: the specimen chamber can be viewed on the camera monitor display located beneath the vacuum gauge (see Fig. 1). Turn on the camera monitor (switch in lower, right-hand corner), then push the white camera button on the 4th control panel (see Fig. 1; Fig. 4). When this button is lit, the camera is on.

1. Turn the beam off (insert the Faraday cup) by pushing the PCD button on the 4th Control Panel (Fig. 4). The beam is blocked when the button is IN.

2. Turn on the camera monitor (see Fig 1), with switch in lower right-hand corner of camera-monitor panel, and turn on camera by pushing the white button on the 4th Control Panel (Fig. 4) – on when lighted.

3. Check the Camera Monitor screen to view the sample chamber. The tip of the COLUMN is visible top-center. The SPECIMEN STAGE is just below the tip of the column. Check for two potential problems: (a.) A sample has been left in the sample chamber. Remove this sample following the procedure described in Step 14. (b) The cathodoluminescence detector is in place (a bar can be seen extending across the sample chamber). The detector must be retracted before inserting a sample in the sample chamber (see Cathodoluminescence Microscopy, Section 5.4).

4. Insert a specimen in a specimen holder. Apply conductive paint between the specimen and the holder to prevent specimen charge-up. Screw the specimen-exchange rod into the holder [Keep the rod on its stand when not in use.] Make sure the rod is lubricated with apiezon grease.

5. Set the working distance to 39mm or 48 mm by using the working-distance control (Fig 3).

6. Press SAMPLE EXCHANGE on the TOUCH SCREEN CONTROL to the left of the stage control joystick (see Fig. 6). Make sure that the VIB button located in the vacuum control panel (see Fig. 1) is closed (lit). Tilt, Rotation (Fig. 4) sold be set to 0. Check that the SE detector is off.

7. Close the "GUN ISO" (lation) valve next to the VIB to isolate the stage. The button is lighted (bright) when closed.

8. Mount the specimen exchange rod on the specimen exchange chamber and, while holding the rod, press the vacuum control button (Fig. 3) to evacuate the specimen exchange chamber. The lamp button will go out in about a minute to indicate that evacuation is completed. Do not leave instrument unattended during this operation.

9. After verifying that the specimen exchange chamber evacuation is completed, open the specimen exchange chamber isolation valve by turning the valve knob fully counterclockwise and pulling it out fully (the specimen chamber illumination lamp lights). Use smooth motions.

10. Looking into the specimen chamber through the glass cap of the specimen exchange rod, slide the specimen holder onto the holder mount of the specimen stage (noting the dovetail shape) by pushing the specimen holder with the rod. Then unscrew the rod and retract it fully, using a smooth motion.

11. After verifying that the specimen exchange rod has been fully retracted, close the isolation valve by pushing the valve knob fully and turning it clockwise.

12. While holding the specimen exchange rod, press the vacuum control button to vent the specimen exchange chamber (the button lamp lights), remove the rod and place it on the stand.

13. WAIT UNTIL THE SAMPLE CHAMBER VACUUM IS LESS THAN 8 X 10^{-6} TORR BEFORE OPENING THE GUN ISO VALVE.

13. Verify that the ALARM/AEM switch on the VACUUM CONTROL panel is set at ALARM.

14. To remove a sample from the sample chamber, reverse the procedure used in loading: turn off the accelerating voltage, mount the specimen exchange rod onto the specimen exchange chamber, evacuate the specimen exchange chamber and open the isolation valve following steps 4 and 5, attach the rod to the specimen holder and fully retract the rod, close the isolation valve and remove the specimen exchange rod from the specimen exchange chamber by carrying out steps 7 and 8, remove the specimen holder from the specimen exchange rod.

4. CALIBRATING THE SEM

Before beginning the process of acquiring images, it is necessary to set the desired working parameters and adjust the instrument for maximum efficiency.

[**To avoid overexposure of the sample during calibration, block the electron beam by inserting the Faraday cup (button marked PCD) located on the Fourth Control Panel (Fig 4). The beam is blocked when the button is pushed in.]**

4.1 Setting accelerating voltage.

Turn on the accelerating voltage by pressing the accelerating voltage button (ON, the lamp lights) on the 3rd control panel (Fig. 5). This applies the accelerating voltage to the electron gun and simultaneously turns on the filament heating current. TURN ON EOS MENU. Set the accelerating voltage to the desired value. The accelerating voltage can be selected in 0.1 kV steps from 0.2 to 5 kV and in 1 kV steps from 5 to 30 kV. The value set with this knob is displayed on THE 3RD CRT.

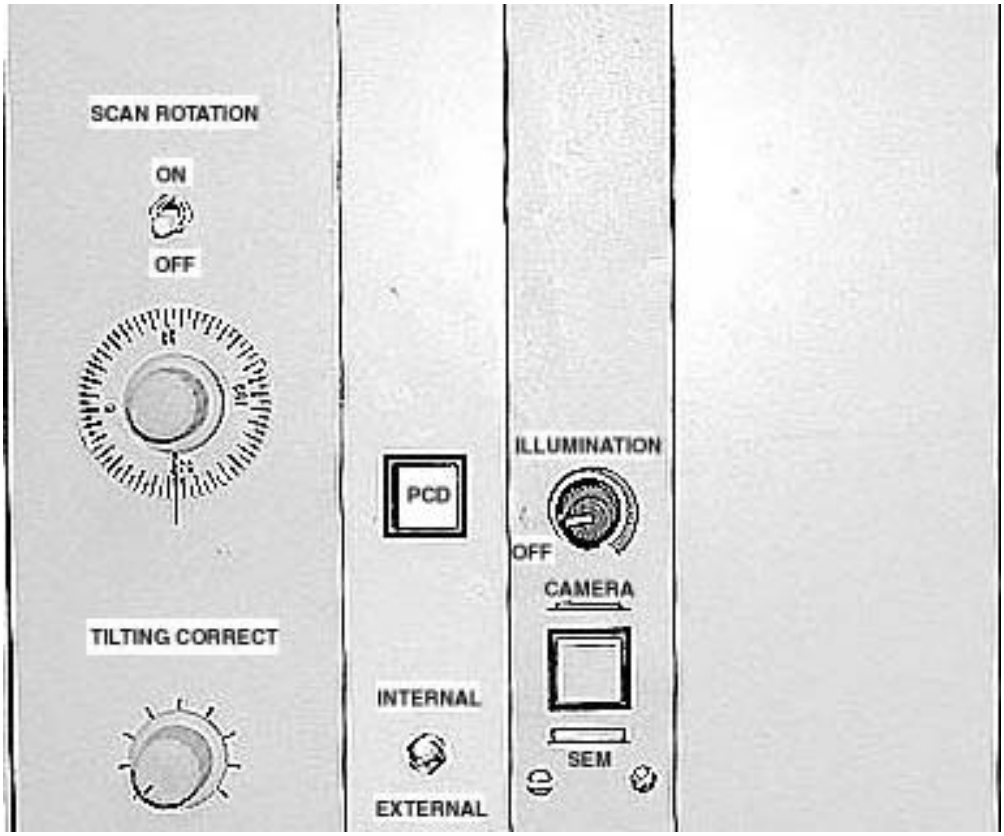

Figure 4. Fourth Control Panel

Figure 5. Third control panel.

knob reduces amperage, the column is aligned.

4.2 Saturating the Filament

The emission current meter (Fig. 5) indicates the filament heating current when the switch is ON (4.0 A at full scale) or emission current when the switch is OFF (400 μ A at full scale). Normally, these values are set at 2.9 A and 60-100 μA. The current meter lamp lights when the column has been evacuated enough for accelerating voltage application. The EMISSION/FILAMENT button switches the current meter reading between the emission current (button lamp off) and the filament heating current (FILA, button lamp on [LIT]).

Saturating the filament involves increasing the filament current until the filament is saturated. Saturated means that increasing the filament current results in no further increase in beam current. [Turning the filament current beyond the saturated position will shorten filament life.]

4.2.1 Saturating Method 1 – no sample loaded

With the beam current blanked, turn up the current (viewed on the beam current meter, Fig. 1) using the EMISSION/FILAMENT KNOB on the 3rd Control Panel (Fig. 5) until the current reaches a value beyond which turning the knob produces little additional change. Turn the current back down, then repeat this process a few times.

4.2.2 Saturating Method 2 – sample loaded

Use the PROBE CURRENT KNOB (see Fig. 10) to adjust the amperage to a suitable value for the sample (range 2-40 nA). Switch the SEM to either BACKSCATTER MODE or SECONDARY ELECTRON MODE. Adjust the brightness/contrast so that the sample is visible. Aim for a middle gray scale.

Turn down the filament current by using the EMISSION/FILAMENT knob (Fig. 5) until the brightness of the image drops off a bit. Then turn it up until further increase does not produce significant increase in brightness of the image. By doing this several times, a point will be reached where further increase in amperage does not cause an increase in brightness of the image. The filament is now saturated.

Note: Once the filament has been saturated, either the PROBE CURRENT KNOB or the CONDENSER LENS SELECTOR BAR (EOS Menu Fig. 16) can be used to set the current to the desired value.

4.3 Aligning the gun (column)

Gun alignment is performed by using the TILT/SHIFT button and the X and Y knobs on the 3rd control panel (Fig. 5). The TILT/SHIFT button switches the GUN ALIGNMENT (X,Y) knob functions (TILT/ SHIFT). In the SHIFT mode, the button lamp lights and the GUN ALIGNMENT knobs are used for horizontally shifted beam correction (GUN SHIFT). In the TILT mode with the button lamp off, the knobs are used for tilted beam correction (GUN TILT). Note: adjust SHIFT at high current; adjust TILT at low current.

In the shift position when the button lamp is lit, the X and Y knobs are used to maximize the amperage (viewed on the BEAM-CURRENT METER ,see Fig. 1). Adjust these knobs, one at a time, to maximize the beam current. Keep adjusting the probe current and readjusting for maximum. Now, turn the PROBE CURRENT KNOB clockwise until the CONDENSER LENS COARSE slider bar (viewed in the basic EOS COLUMN CONTROL WINDOW, see Fig. 16) reads approximately 8-11. This procedure will reduce the amperage into the pA range, as displayed in the BEAM-CURRENT METER. If the BEAM-CURRENT METER hangs, push the AUTO button twice.

Push the SHIFT/TILT selector button, turning off the light. Maximize the amperage as before. When maximized, turn the amperage back up and light the switch again (SHIFT mode). Repeat this process of maximizing at high and low amperage until no change is observed when switching. For example, if a switch is made from low to high and the high amperage is already maximized and any change to either

4.4 Centering the Aperture

The aperture is centered by using the WOBBLER BUTTON in the 2ND CONTROL PANEL (Fig. 6). With an image on the 1st CRT Display, select some small feature in the image and center it on the 1st CRT screen with the X and Y controls of the specimen stage. Focus the image of the feature with the FOCUS knob (COARSE/FINE) on the IST CONTROL PANEL (see Fig 1 and Fig. 9). Press the WOBBLER button on the 2nd Control Panel (upper, left button in Figure 6); lamp lights when ON. If the image does not shift as the focus is varied, the aperture is centered. If it shifts, adjust the aperture centering knobs (Fig. 7), located on the gun column, to minimize the shift. Carry out centering with magnification increased to 5000x –20,000x. Turn WOBBLER button OFF.

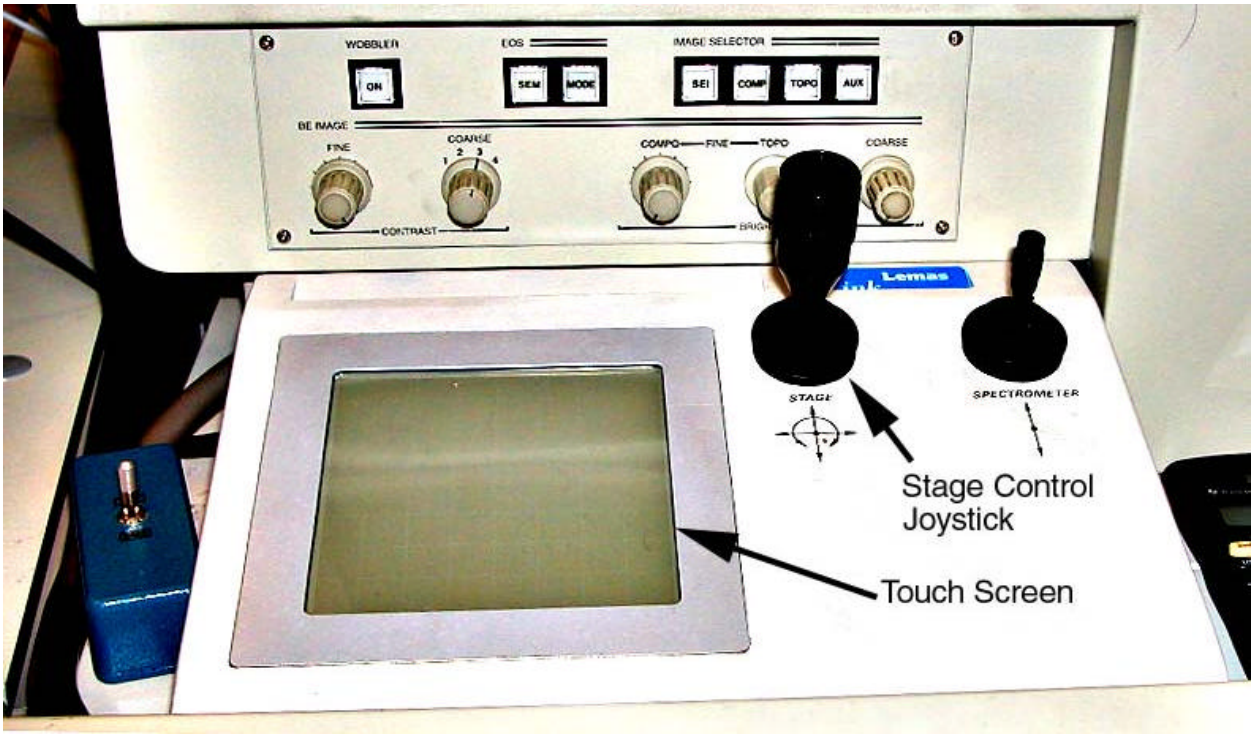

Figure 6. Second Control Panel (top panel). The panel below shows the sample stage control (large black joystick) and the touch screen .

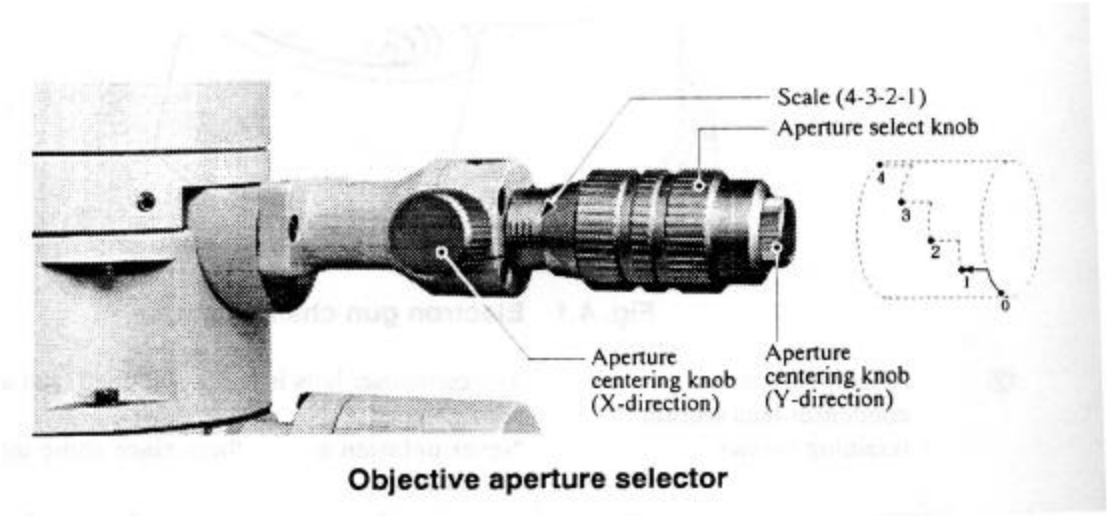

Figure 7. Aperture centering knob

4.5 Astigmatism Adjustment

To correct astigmatism, adjust the X and Y astigmatism knobs until the sharpest images is obtained.

5. ACQUIRING IMAGES

5.1 Nature of SEM Emissions

When an electron beam from the electron gun encounters a specimen in the specimen chamber, several important kinds of emissions take place (Fig. 8). The most useful emissions are emissions of backscattered electrons, secondary electrons, photons (cathodoluminescence), and X-rays
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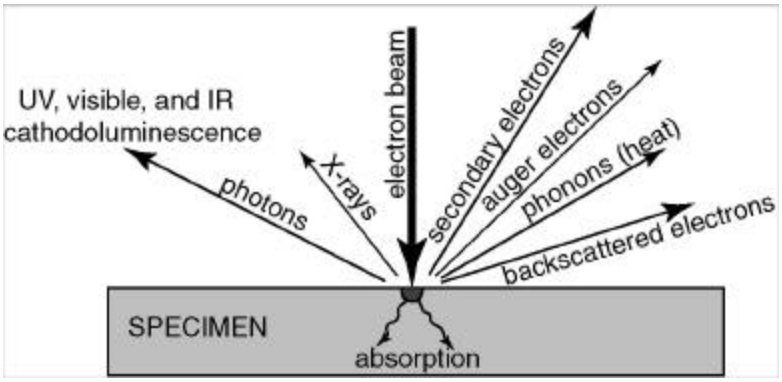
Figure 8. Effects produced by electron-beam interaction with a specimen in the SEM



The number of backscattered electrons emitted from a material bombarded by the electron beam (and thus the brightness of the backscattered image) is a function of the average atomic number of the material. For example, backscattered (BSE) images of quartz appear relatively dark compared to those of iron-rich minerals such as pyrite, which would appear very bright. Therefore, BSE images are useful for distinguishing among different minerals in a specimen. The emission of secondary electrons is related to topography of the specimen. Consequently, secondary electron images are useful for studying the shapes of crystals or other objects. The intensity of CL (photon) emissions is related in a complicated way to the presence of trace elements and lattice defects in minerals. SEM-CL images commonly display textural features, not visible in other kinds of images, that have a variety of geological applications. X-rays are emitted with characteristic energies that make possible identification of the chemical elements responsible for their emission.

5.2 Secondary Electron Microscopy

1. Ascertain that the accelerating voltage is on and the lamp of the EMISSION-CURRENT meter is on.

3. Push the SEI button, located on the 2nd Control Panel (Fig 6) to ON (lit), which turns on the SE detector.

4. Push FAST SCAN button on the 1st Control Panel (Fig 10) to view the image on the 1st CRT (see Fig. 1). 2. Turn the SE detector on by using the auxiliary keyboard (Fig. 9).

5. Use the COARSE/FINE button in the 1st Control Panel (Fig10) allows selection of either fine or coarse focusing. The focusing knob can then be used to obtain the best focus. Commonly, focusing is done at high magnification.

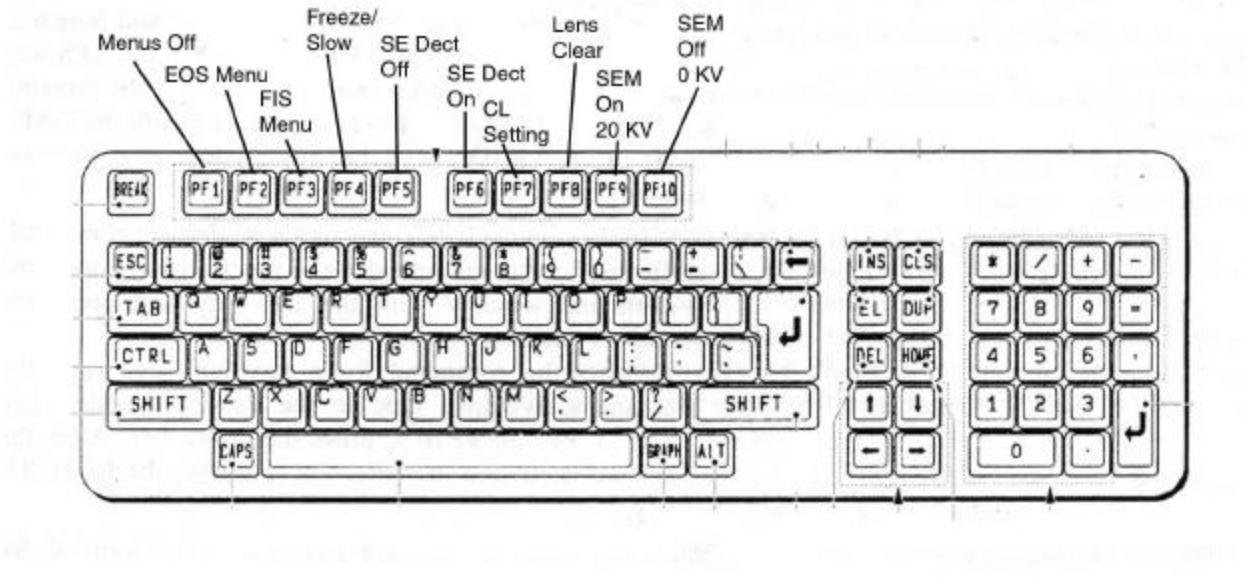


Figure 9 Auxiliary Keyboard (see Fig. 1)

6. Brightness and contrast can be adjusted by the BRIGHTNESS and CONTRAST knobs on the 1st Control Panel (Fig. 10).

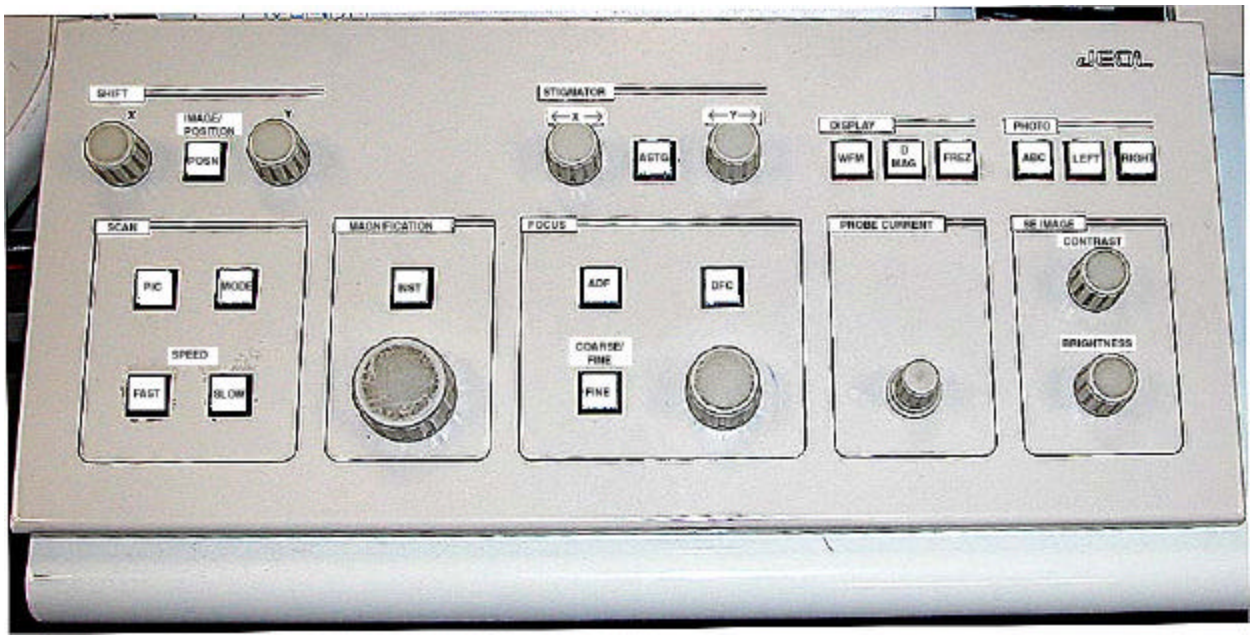


Figure 10. First Control Panel.

7. Acquire slow-scan image.

a. Access the FRAM INTEGRATION SYSTEM menu by clicking on the CONTROL bar menu and then on FRAME INTEGRATION SYSTEM. THE FRAME INTEGRATION SYSTEM (FIS) menus will appear (Fig. 11).

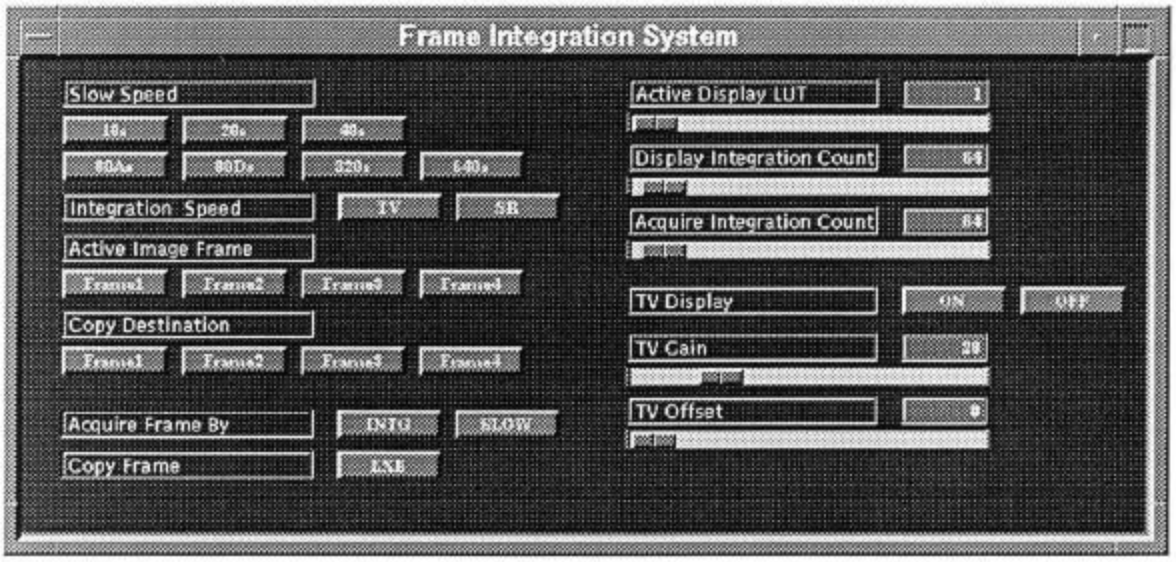


Figure 11. Frame Integration System (FIS) Menu.

c. Make selections for FRAME INTEGRATION and SCAN SPEED (e.g., 20s, 80s) on the FRAME INTEGRATION SYSTEM menu. Then click SLOW. In a few seconds, the image will be acquired on the 2nd CRT.

8. Storing Image

a. Double-click on the JSM IMAGING SYSTEM icon (see Fig. 14). This opens the IMAGING CONTROL WINDOW (Fig. 12).

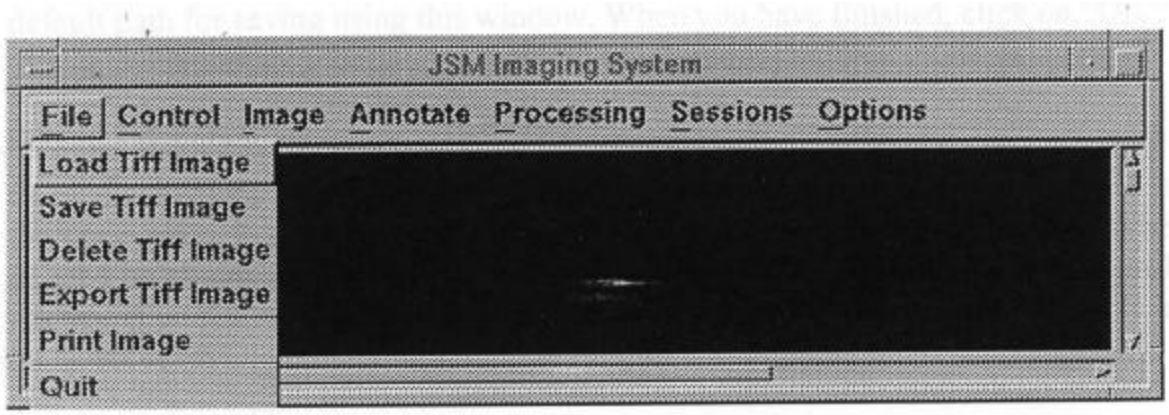


Figure 12. JSM Imaging System Menu

b. Click on FILE (Fig. 12), then on SAVE TIFF IMAGE. The file saving window appears. Select a path and create a file name for the new image, or save it over an old one by keeping the path and file name then save. When finished, click O.K.

9. Print Image. Click on the FILE menu bar and then on PRINT IMAGE. A window displaying the printer path will appear. Click on PRINT to go ahead, or CANCEL to cancel the print job.

5.3 Backscattered Electron Microscopy

After a secondary image has been acquired and stored, a backscattered (BSE) image can be obtained by pushing the COMP button on the 2nd Control Panel (Fig. 6) to the ON position. Focusing is performed in the same manner as described for secondary microscopy. Brightness and contrast are adjusted by using the brightness and contrast knobs on the 2nd Control Panel. BSE images are saved in the same manner described under secondary microscopy.

5.3.1 X-ray Analysis

Qualitative chemical information about minerals in a specimen can be obtained in the BSE mode by using the EDX (x-ray spectrometer) unit to capture X-ray emissions and display them as an energy spectrum (see EDX display in Fig. 1).

To display an EDX spectrum:

a) Select a grain or crystal to be analyzed from the image displayed in the 1st CRT.

b. Magnify image to maximum level.

c. Push MODE button on 1st Control Panel . [Note: push PIC to turn off mode.

d. Turn on the EDX display using the key on the console located on the floor beneath the EDX display (see Fig. 1).

e. Press the PF-1 key on the keyboard located below the EDX display to start X-ray acquisition. The EDX spectrum will appear. The peaks displayed on the spectrum indicate the characteristic energy of the X-rays generated by a particular chemical element . The chemical element responsible for a particular peak can be identified by using the LABEL function on the EDX keyboard Hit the F3 key twice to bring up READ LABELS. Hit RETURN. Set acquisition parameters (voltage) to 10 Kv.

f. Push key PF-1 again to stop X-ray acquisition.

5.4 Cathodoluminescence Microscopy

The JSM-633 was not equipped initially with a cathodoluminescence detector; however, an Oxford Instrument mirror-type CL detector has been added to the instrument (Fig 13). This detector allows photons to be collected and displayed as cathodoluminescence images. A set of optical filters is available (Fig. 13), which allows CL images to be acquired through a red, blue, or green filter, if desired.

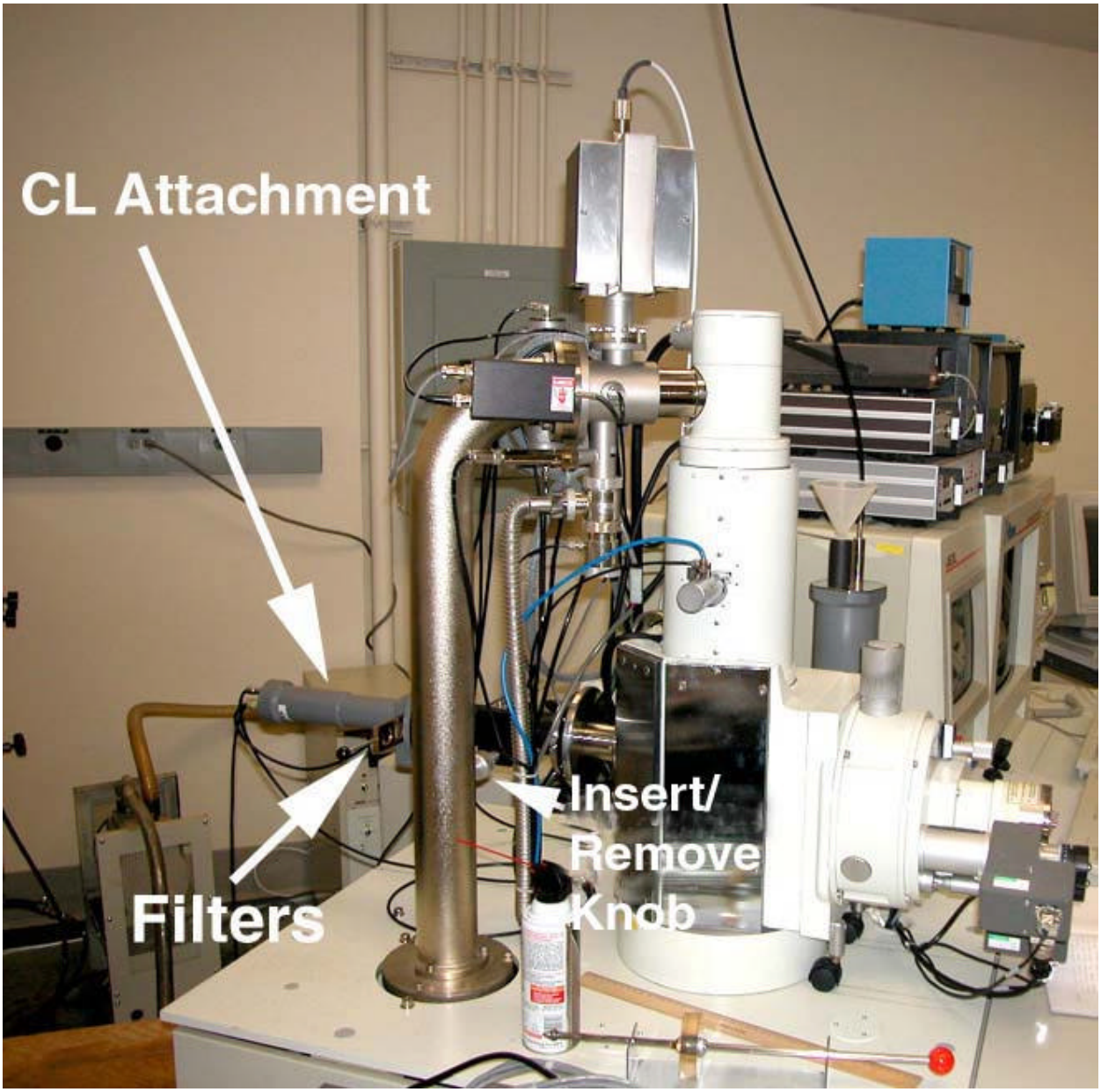


Figure 13. CL detector attached to the JSM-6300.

To obtain CL images:

1. Engage the CL detector by manually turning the INSERT/REMOVE knob (Fig. 13) clockwise until a stop is reached.

2. Set the working distance (Fig. 3) to 25 mm. The position of the detector can be viewed in the sample chamber by turning on the CAMERA MONITOR, as described under "Sample Loading" above.

3. Turn on the CL detector by pushing the AUX button on the 2nd Control Panel (Fig. 6).

4. Focusing: see discussion under “Secondary Microscopy.”

5. Brightness and contrast are adjusted by using the controls on the photomultiplier amplifier (see Fig. 1). GAIN controls the brightness; OFFSET controls contrast (Fig. 14). Note: The PHOTOMULTIPLIER OVERLOAD trips (light glows red) if some portion of the image is excessively bright. Turn down the brightness and reset, or move to a different image and reset.

Figure 14. Photomultiplier Amplifier for cathodoluminescence images.

6. Acquiring, storing, and printing CL images follows the same methods described for secondary and backscattered images.

6. X-VISION CONTROLS

Many of the knob/dial/button operations described in preceding sections can also be carried out by using the X-Vision System software. When Login is completed, icons for the JSM-6301F Control, the JSM Imaging System, and (optional) Stage Control will appear (Fig. 15). Icons are opened into windows by double-clicking on them. Depending upon the application or function group you are opening, you may be presented with either a function window or a window containing another series of icons, from which you may make a more specific choice. If you double-click on one of the X-Vision icons, you will see one of the several windows that control the SEM, imaging, or (optional) stage control.

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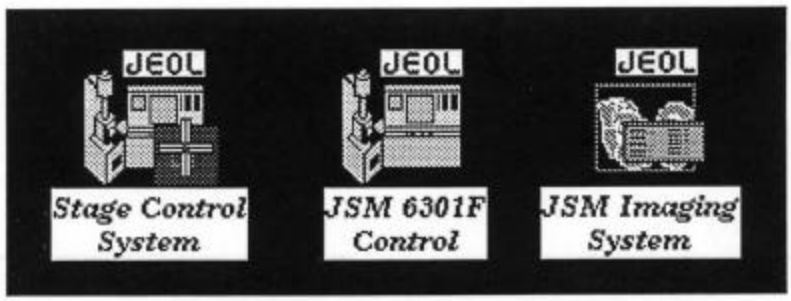

Figure 15. Main X-Vision menu.

6.1 The EOS Windows

Access all EOS Functions by double-clicking on the JSM-6301F Control Icon, which brings up the basic COLUMN CONTROL window (Fig. 16).

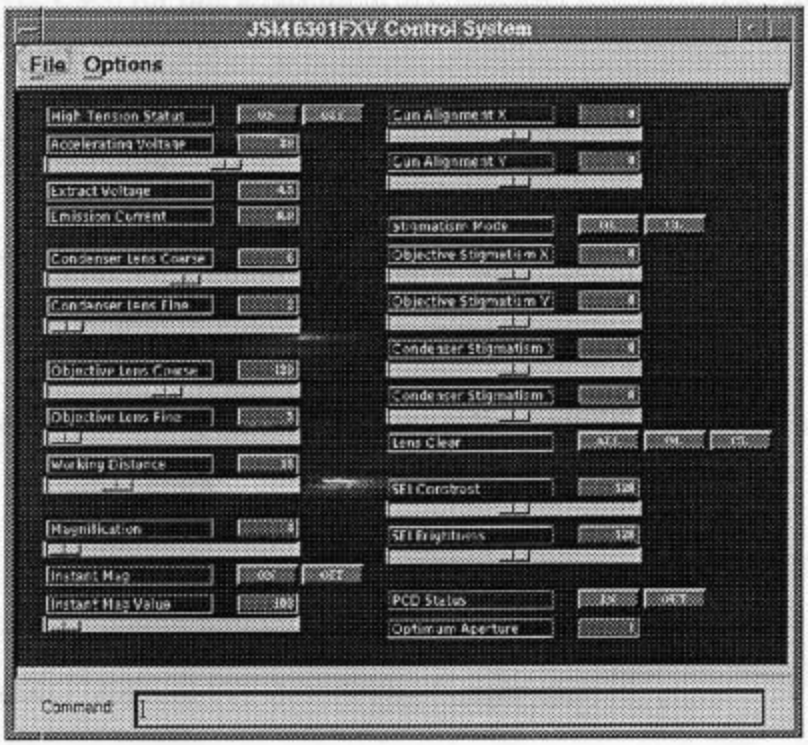

Figure 16. Basic Column Control window.

6.2 Column Control

This window allows all of the column control functions to be accessed, including accelerating voltage, emission current, condenser lens adjustment, magnification, and gun alignment.

6.3 EOS Control Bar

The EOS Control Bar allows quick and direct access to SEM control functions without opening every window that contains these functions. The Control Bar is accessed by pulling down the

FILE menu on the JSM-6301F Control Window. A list of all available functions (Fig. 17) will appear on the left side of the screen.

Function settings on the Control Bar can be changed in two ways: (1) Click once in a value box, then type in the new value. (2) Click on the name of a function. A small window called a "modal" will appear. Within a modal, the number value can only be changed by using the slider bar. To make the change take effect, click on "Close."

Figure 17. EOS Control Bar

6.4 Scan Controls

Pull down the FILE menu on the Basic Column Window by clicking once on the word "File" on the window menu bar. Then click on "EOS Menu 2." This will open the SCAN CONTROL WINDOW (Fig. 18). This window allows scan modes, scan speeds, and Recording Scan Speeds for photos to be adjusted.

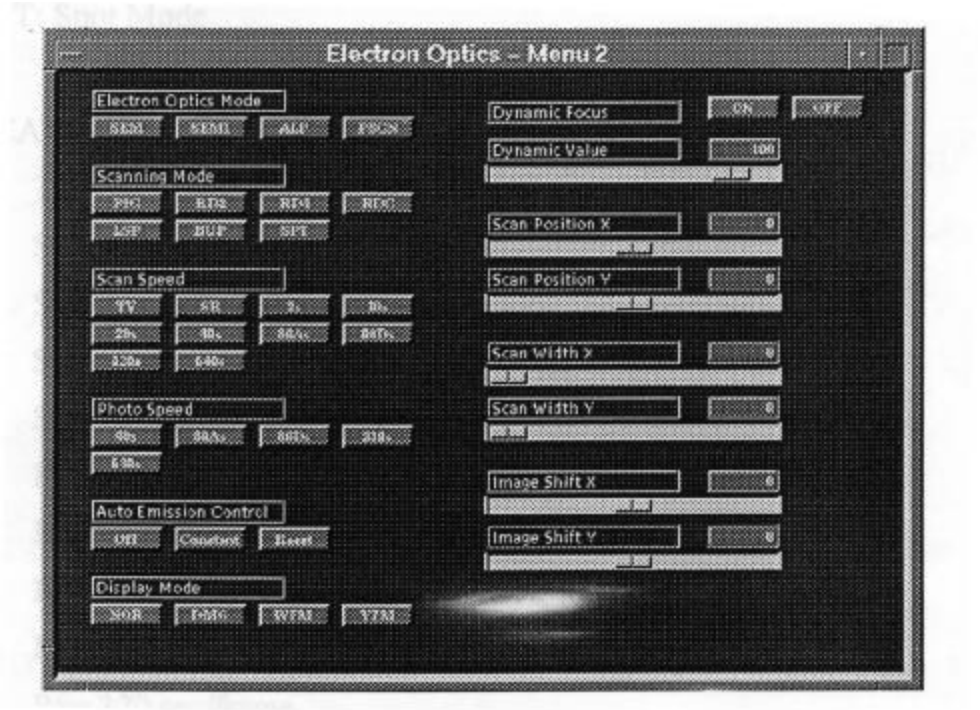

Figure 18. Scan Control Window.

6.5 Imaging control

Double-clicking on the JSM IMAGING SYSTEM icon (see Fig. 15) opens the IMAGING CONTROL WINDOW (Fig. 11). The procedure for accessing imaging functions is described in Section 5.2 (8), which deals with storing images.