

**JEOL 5800LV
SCANNING ELECTRON MICROSCOPE
OPERATOR'S MANUAL**

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Introduction

INTRODUCTION

This document is an introduction to the operation of the JEOL 5800LV scanning electron microscope and the Oxford Isis 300 analytical system. This manual should be considered a work in progress. With time, we will add sections as operating procedures are developed or improved by user experience. This manual is not intended to be formal instrument documentation, but rather, a hands-on users guide to the integrated operation of both the microscope and analyzer. Details of microscope operation, the EDS analytical system, and the computer software can be found in the manufacturer's manuals kept in the lab. Unfortunately the JEOL reference manual is poorly translated from Japanese and is difficult to understand, and thus is a limited resource. The Oxford software manuals are a useful resource, with reference and tutorial sections for most of the software packages.

The first part of this manual consists of a brief introduction to the JEOL 5800 and the Oxford analytical system. This is followed by a comprehensive checklist for setting up, starting, and shutting down the instrument. This section is divided into two parts: one for operation in high vacuum and the other for operation in low vacuum mode. Sections then follow for EDS analysis, collection and manipulation of images and x-ray maps, and operation of the cathodoluminescence detector. An appendix section is included that deals with emergencies and potential problem areas. Users should proceed stepwise through the sections on loading samples and instrument set up and then proceed to the appropriate section(s) for imaging, X-ray analysis, etc.

THE JEOL 5800LV

The JEOL 5800 SEM is a sophisticated scanning electron microscope that was designed to operate in either high or low vacuum (the LV connotation indicates the presence of the "low vacuum" controls). The electron column contains a pressure differential aperture that allows the specimen chamber to be operated at a low (or poor) vacuum while the rest of the column and gun are maintained at a high vacuum. In this operating mode, two separate rotary vacuum pumps are used: one to back the diffusion pump that pumps the column and another to pump directly on the chamber. When the system is operated in high vacuum mode, the diffusion pump and its rotary pump are used to maintain equal vacuum in the column and chamber.

The microscope is controlled by an internal computer through a menu-driven software interface. The motorized stage is operated from a joy stick, and the single joystick serves several functions which are set by buttons on the keyboard. Likewise, several knobs on the keyboard serve different functions which are selected from keyboard buttons. The computer has a command line interpreter, so that in addition to manual operation from the various knobs, buttons and joystick, many of the functions can be accomplished by commands typed from the keyboard. The instrument's computer can also receive instructions and be controlled by other independent computers through RS232 and SCSI ports.

Introduction

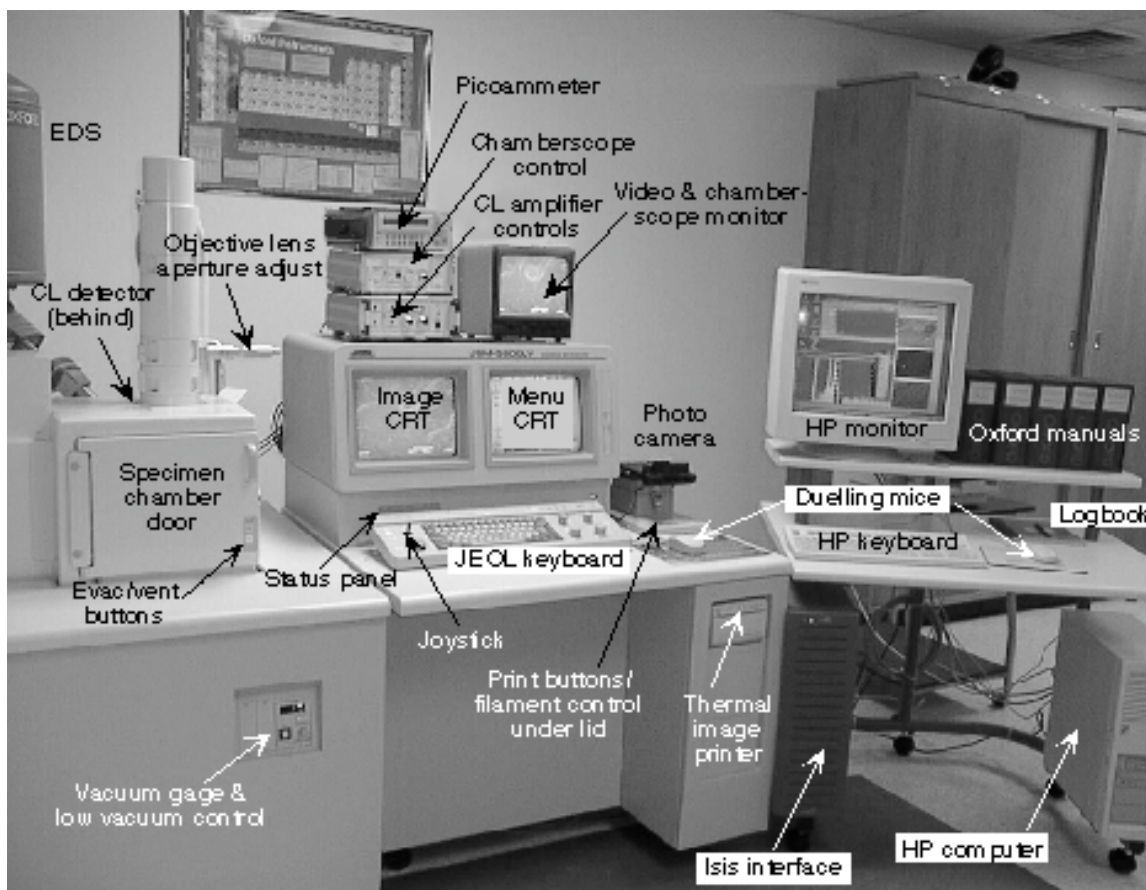


Figure 1. Components of the JEOL scanning electron microscope and Oxford analyzer system.

The SEM is equipped with detectors for imaging of secondary electrons (SE), backscattered electrons (BSE), x-rays, and cathodoluminescence (CL). The SE detector is a highly efficient Everhart-Thornly detector for imaging at all scan rates in high vacuum. The Everhart-Thornly detector cannot be used in the low vacuum mode, and its function is replaced by the BSE detector. This detector consists of an annular ring mounted under the objective lens and another flat detector a short distance from the lens, creating a parallax from which topographic information is derived in the low vacuum mode. The EDS can be utilized in both vacuum modes, but requires a working distance of 15 mm or less. The CL detector is an independent optical device that must be inserted into the electron path with the stage at a specific height. Only the SE detector can be used with the CL inserted.

THE OXFORD ISIS 300

The Oxford analytical system consists of the EDS detector, an interface box and a standard desk-top computer. The EDS has an ultra-thin window installed and can therefore detect elements from boron and up ($Z \geq 5$). The detector is controlled from the Oxford interface, which contains the electronic hardware for the EDS signal and image acquisition, along with microscope automation hardware. The interface passes

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information to and receives commands from the user through the Oxford software on the PC computer. The PC is an HP Vectra with a 200 MHz Pentium CPU, a 2 GB hard drive, a single 3 1/2" high-density floppy disk drive, a CD-Rom, and a 100 MB Iomega Zip™ drive communicating through a SCSI port. Software is operated in the Windows 95 operating environment. The system is attached to the EPS local area network (LAN) via ethernet connection.

Image and EDS acquisition is accomplished through Oxford Windows software. The software is modular in design, and many of the available modules have been installed. There are four primary modules: X-ray Analysis, Autobeam, Automation, and IMQuant. Two additional modules are Detector Calibration and Notepad. All of these primary modules are accessed through Labbook™, Oxford's information database system. Qualitative x-ray identification, quantitative analysis, and x-ray mapping are available through the X-ray Analysis module. SE, BSE and CL images are acquired through the Autobeam module. This module is also the access point for several image analysis subsystems, including Cameo™, area analysis and phase analysis software. The Automation module controls stage movement and electron column functions for operating the microscope in an automated mode. Finally, the manipulation, processing, and analysis of image is done in the IMQuant module.

Data transfer from the SEM to the "outside world" can take place on a variety of media. Images can be acquired and printed directly from the SEM to Polaroid film or to a thermal image printer. These digital images can be saved directly, in TIFF format, to a floppy disk on the JEOL, which is the highest resolution available (see Appendix A, Table 1). More commonly, however, images are acquired through the Isis system. There the images are stored in Oxford's proprietary format on the hard drive. The images must then be converted to TIFF or BMP for export. Once exported, images may be loaded to floppy or to Zip disk to be taken away. They can also be sent directly to a user's file or a public directory on the Department LAN for access by the user or by anonymous ftp. In addition, a public file exists on the HP computer that may be accessed by anonymous ftp from an outside computer or sent directly by ftp to another computer.

Oxford Instruments Cathodoluminescence Detector

There are three main components to the CL system:

1. A very sensitive photomultiplier tube. This item is derived from instrumentation developed for use in astronomy. It is capable of detecting very low levels of light and can "see" photon emission far below the levels that are directly detectable by humans. Thus, it is possible to study the luminescence character of minerals that are "nonluminescent" in conventional luminoscopes.
2. A parabolic mirror. This part of the system is inserted very close to the sample and sends the photons to the photomultiplier. The scanning electron beam goes through a hole in this mirror.
3. An amplifier and associated electronics that send the photomultiplier signal to the AUX image port of the JEOL 5800 microscope.

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This instrument is designed for detection of low-level cathodoluminescence. Brightly luminescing minerals, especially those emitting in the red-orange portions of the spectrum or having emission that is persistent (phosphorescent) relative to the scan speed, will require special consideration.

Use of the CL detector increases the level of difficulty in navigating around the sample. It is a good idea to familiarize yourself with the sample before doing CL work. If necessary, identify target grains using optical petrographic techniques and use sketches or optical photomicrographs to navigate to the target grains. India ink trails that lead from the edge of the sample to circles around the items of interest are helpful. Next, it is a good idea to obtain some BSE images or other photomicrographs at the same or slightly lower magnifications than those you expect to use with luminescence. The practical limit of resolution with this luminescence system is around 1000x. Many of the landmarks visible in the BSE-image will also be visible in the secondary electron image.

High Vacuum Operation

GETTING STARTED

Note: for the following discussion, **BOLD CAPS** indicate buttons or other controls, *italic type* indicates menu commands that may be issued by selection with the mouse or from function (F1-F15) keys.

Take note! Important warnings are set in boxed text.

OPERATION IN HIGH VACUUM MODE

I. PRELIMINARY PROCEDURES

- A. Check condition of instrument.
 1. Turn up both JEOL monitors using thumb knobs under each monitor.
 - a. On the left monitor, turn up brightness (right knob) all the way, leave contrast (left knob) low.
 - b. On the right monitor, set brightness and contrast to comfortable levels.
 2. On the *Status Panel*, check to see that the previous user has left the instrument at the proper settings:
 - a. The SEI detector should be enabled.
 - b. Accelerating voltage is at 20 kV.
 - c. Stage height is at Z=25 or greater.
 - d. Stage X, Y, T and R should be at approximately 0.

Do not load a sample when the stage is less than 25 mm.

3. If any of the stage parameters are not as shown above, set stage to *Exchange Position*.
 - a. From the Menu Screen, SELECT *F5 STAGE*.
 - b. SELECT "Exchange Position" from the file list (Use "Low Exchange" if large samples will be introduced).
 - c. SELECT **START** from the window to start the stage moving.
- B. Check status of the keyboard:
 - a. Joystick **X/Y** button is activated (it should be green).
 - b. The **TONE** button is activated.
 - c. **COARSE** focus button is activated.
 - d. Beam **BLANK** button is NOT activated (not red).
 - e. **HT** button is NOT activated.
 - f. **L**(eft) screen freeze button is NOT activated.
 - g. **D-MAG** button is not activated.

II. LOAD SAMPLES

- A. Vent the chamber.
 1. PRESS and HOLD the **VENT** button until it activates.
 2. UNLATCH the door latch.
 3. Wait until **VENT** light stops flashing. Instrument will beep once (door may open by itself before that!).

High Vacuum Operation

B. Insert specimen holder.

1. Use the Specimen Inserting Tool to load sample holder into the chamber.
2. CLOSE the door and latch.
3. PRESS and HOLD **EVAC** button until it activates.

III. INITIAL SETUP

Proceed with setup while instrument is pumping down.

A. Set accelerating voltage (guidelines are given in Table 1 below).

1. From the Menu Screen, SELECT *F2 MENU* .
2. SELECT **ACCV**.
3. SELECT the desired accelerating voltage.

Table 1. Appropriate operating parameters for various working situations.

| | Accelerating Voltage | Working Distance | Spot Size | Beam Current |
|--|-----------------------------|----------------------------|-----------------------------------|---------------------|
| High Magnification/ High Res. (>10,000x) | 20-30 kV | Short 8 - 12 mm | Small 5 - 8 arrows | Low <1 nA |
| Low Magnification (to 18x) | 5 -15 kV | Long to 48 mm | Midrange 8 - 16 arrows | |
| Quantitative EDS | 20 kV | 10 – 12 mm | Midrange | 0.5 - 1 nA |
| Low Vacuum (uncoated or charging) | 15 - 20 kV | Short 8 - 12 mm | Midrange 10 - 16 arrows | |
| Low Voltage (uncoated or charging) | 0.5 - 5 kV | Mod. – large 20 - 48 mm | Midrange 8 - 16 arrows | |
| CL Imaging | 10 - 15 kV | 16 mm | Midrange – high 12 - 20 arrows | 1 -10 nA |

B. Set computer to appropriate sample holder.

1. From the Menu Screen, SELECT *F5 STAGE* .
2. POINT to and SELECT **HOLDER**.
3. POINT to and SELECT the picture of the specimen holder that you put in.

C. Set the stage height.

1. Switch on the chamber scope.
 - a. PRESS the white button at the right side of the chamber scope control (GW Electronics box on top of the SEM console).
 - b. PRESS the **POWER** button at the far right side of the chamber scope monitor (Sony monitor on top of SEM console).
 - c. Make certain the A/B channel button is **OUT** (A-position on push-button switch at left side of Sony monitor controls).

When raising the stage, set the desired working height at the highest part of the highest sample. DO NOT allow the sample to contact the objective lens or the BSE detector.

High Vacuum Operation

2. Manually set the stage height using the joy stick.
 - a. PRESS the **T/Z** button to change to the "Z" the function of the joystick.
 - b. Raise stage by pushing joystick away from you (positive Y direction), lower by moving joystick toward you (negative Y direction).
 - c. On the chamber scope monitor, observe the position of the sample relative to the bottom of the objective lens.
 - d. PRESS the **X/Y** button to change back to the "X-Y" the function of the joystick.
- D. Move the sample position to an appropriate place on the sample or holder. (For the most part, beam saturation and alignment can be done anywhere on the sample. However, to avoid damaging the sample, it is recommended that setup be done on the rim of the sample holder.
 1. TURN the **MAGNIFICATION** knob counterclockwise to the lowest magnification.
 2. Make sure the **X/Y** button is lit on the keyboard.
 3. The Menu Screen should be on *F5 STAGE* menu.
 - a. Point and click the mouse on some part of the sample holder rim.
 - b. A dialog box will appear. SELECT "Start" to move to the point.

IV. INSTRUMENT STARTUP

You may proceed with this section once "HT Ready" is displayed in the Status Panel.

- A. Filament saturation.
 1. PRESS **LSP1** button.
 - a. ADJUST the **BRT** knob to approximately center the line on the monitor.
 - b. As you reach filament saturation, it may be necessary to adjust both **BRT** and **CONT** knobs to maintain the line within the middle portion of the monitor.
 2. PRESS **HT** button. The **FIL**ament light will flash until you begin heating the filament.
 3. PRESS the lower left corner of the Photo Panel to release the lid and access the Filament Panel.
 - a. Slowly TURN the **FILAMENT** knob clockwise to heat the filament.
 - b. TURN the knob past the false peak until the line profile no longer rises. Leave the knob just at the point of saturation (Do not turn past 3:00 o'clock).
 4. PRESS **TV2** button on keyboard.
 5. TURN magnification all the way down.

If the FILament light begins flashing after you have reached saturation or if it never stops flashing, the filament is burned out. Notify the lab manager.

High Vacuum Operation

B. Current axis (gun) alignment).

Note 1: You will need a sample area suitable for magnification up to 20,000x.

You can probably find a small milling mark or other discernible feature on the sample holder (bright spherical objects work best).

Note 2: This procedure brackets the most commonly used current range (from 8 to 16). If lower spot size will be used, e.g. <8 for high resolution imaging, bracket gun alignment range from 1 to 8.

1. ADJUST the **FOCUS** knob (coarse focus) and initially focus at low magnification on the surface of the sample holder.
 - a. Gradually increase magnification while focusing to find a small feature.
 - b. At 20,000X, approximately center the object in the monitor.
 2. PRESS **SPOT SIZE** button. Use **CRS** knob to set spot size to 8 (>>>>>>>>)) on the Status Panel.
 3. PRESS **RESET** button on keyboard.
 - a. Refocus. (You will probably have to turn the magnification down to find the object again).
 - b. PRESS **RESET** button again and refocus if necessary.
 - c. Repeat reset until there is no movement or change in focus.
 4. Closely center object on the monitor using the joystick.
 - a. Place left index finger on object at center of monitor and keep it there.
 - b. ADJUST **SPOT SIZE** to 16 (>>>>>>>> >>>>>>>>)).
 - c. While keeping finger on object, PRESS **SHIFT** button on Filament Panel.
 - 1) ADJUST **X** and **Y** gun alignment knobs on Filament Panel to re-center object under finger.
 - d. Observe object while turning **SPOT SIZE** back to 8.
 - 1) While turning spot size down, object may loop out from under finger and return back. This is a function of the microscope itself and is ok.
 - 2) If object has shifted from center (under finger) repeat from step 4 above until object returns to within 0.5 μm of center.
 - e. Set **SPOT SIZE** back to midrange (you can lift your finger off the screen now!).
 - f. PRESS **SHIFT** button off.
 5. PRESS **LSP1** button on keyboard to check gun tilt.
 - a. ADJUST **X** and **Y** gun alignment knobs on Filament Panel to maximize height of line profile on screen.
 - b. Close Filament Panel lid using lower left corner of Photo Panel. Be careful not to take a picture by pressing one of the Photo buttons.
 - c. PRESS **TONE** button.
- ### C. Objective aperture alignment.
- Aperture sizes are as follows: #1=20 μm , #2=30 μm and #3=100 μm
1. PRESS **TV1** button on keyboard.
 2. TURN **MAGNIFICATION** to 1,000-5,000x and center the monitor on a clearly discernible object.
 3. PRESS **WOBB** button on keyboard and observe any apparent motion of object.

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- a. If object moves around on screen, the objective aperture must be aligned (any apparent stretching of the object will be eliminated later)
 - b. Adjust aperture alignment control knobs (one at a time) until object remains centered while wobbling in and out of focus.
 - 1) Side knob controls SW-NE motion.
 - 2) End knob controls NW-SE motion.
 4. PRESS **WOBB** button off.
- D. Stigmatism adjustment
- Note:** You will need an area of the sample holder that has fine details (a cluster of bright, spherical objects is best).
1. PRESS **D-MAG** button on keyboard.
 2. Increase magnification to an appropriate level (5,000-20,000x).
 3. ADJUST the **FOCUS** knob (coarse focus) to best focus.
 4. PRESS **COARSE** button off, and carefully focus as well as possible.
 5. PRESS **STIG** button.
 - a. Adjust **X** and **Y** stigmatism knobs one at a time until image becomes as clear and sharp as possible.
 - b. Readjust fine focus.
 - c. Repeat until image is sharp and clear.
 6. PRESS **TONE** button.
- E. Begin work on your sample.
1. Use joystick to move to your sample.
 2. Refer to other sections of the manual for other procedures.
 3. After completion of work, return to the next section below for shut-down procedures.

High Vacuum Operation

V. INSTRUMENT SHUTDOWN

- A. Turn filament off.
 1. Raise Photo Panel lid and TURN **FILAMENT** knob fully counterclockwise.
 2. PRESS **HT** button off.
 3. Close Photo Panel lid.
- B. Return stage to exchange position.
 1. Turn on the chamber scope light and monitor if it is not already on.
 2. From the Menu Screen, SELECT *F5 STAGE* menu.
 3. SELECT "*Exchange*" from File List. Use "*Low exchange*" if large samples are in the chamber.
 - a. SELECT "Start."

Watch the sample on the chamber scope monitor. If the sample appears that it might, for any reason, contact the BSE detector, immediately move the joystick in any direction to abort the move.

4. If necessary, set image mode to SEI.
 - a. From the Menu Screen, SELECT *F2 MENU*.
 - b. SELECT *IMS* and then option *#1 SEI*.
 5. Wait for a few minutes for filament to cool before continuing.
 - C. Remove sample holder.
 1. PRESS and HOLD the **VENT** button until it activates.
 2. UNLATCH the door latch.
 3. Wait until **VENT** light stops flashing. Instrument will beep once (door may open by itself before that!).
 4. Use the Specimen Inserting Tool to remove sample holder from the chamber.
 5. CLOSE the door and latch.
 6. PRESS and HOLD **EVAC** button until it activates.
 7. Remove sample holder from Tool by pressing rubber buttons on Tool handle.
 - a. Remove sample adapter from holder; return holder to shelf.
 - b. Remove sample stubs from adapter; return adapter to shelf.
 - c. Return Inserting Tool to drawer.
 - D. Secure the instrument.
 1. Turn down both JEOL monitors by turning two thumb knobs under each monitor fully counterclockwise.
 2. PRESS **SL3** button on keyboard.
 3. PRESS **D-MAG** button off.
 4. SET magnification to 300,000x.
 5. Turn off chamber scope and monitor.
 - E. Shut down computer.
 - F. Fill out logbook
 - G. Clean up around instrument and sample prep counter.

Low Vacuum Operation

OPERATION IN LOW VACUUM MODE

I. PRELIMINARY PROCEDURES

- A. Mount samples in the appropriate holder.
1. Securely mount the sample to a sample stub.
 - a. Use carbon paint, carbon or double-sided Scotch tape, or Duco™ cement to secure the sample to a sample stub.
 - b. Modeling clay or aluminum foil may be used to prop up a sample in holder.
 2. Secure the sample stub into the specimen adapter with a set screw.
 3. Place the adapter into the specimen holder and secure with one or more set screws.
- Note:** You will need to take special precautions if the sample protrudes more than 1 cm above the height of the specimen holder. **Only experienced users should attempt to operate with samples taller than 1 cm.**
- B. Check condition of instrument.
1. Turn up both JEOL monitors using thumb knobs under each monitor.
 2. On the *Status Panel*, check to see that the previous user has left the instrument at the proper settings:
 - a. The SEI detector should be enabled.
 - b. Accelerating voltage is at 20 kV.
 - c. Stage height is at Z=25 or greater.
 - d. Stage X, Y, T and R should be at approximately 0.

Do not load a sample when the stage is less than 25 mm.

3. If any of the stage parameters are not as shown above, set stage to *Exchange Position*.
 - a. From the Menu Screen, SELECT *F5 STAGE*.
 - b. SELECT "Exchange Position" from the file list (Use "Low Exchange" if large samples will be introduced).
 - c. SELECT **START** from the window to start the stage moving.
- C. Check status of the keyboard:
- a. Joystick **X/Y** button is activated (it should be green).
 - b. The **TONE** button is activated.
 - c. **COARSE** focus button is activated.
 - d. Beam **BLANK** button is NOT activated (not red).
 - e. **HT** button is NOT activated.
 - f. **L**(eft) screen freeze button is NOT activated.
 - g. **D-MAG** button is not activated.

II. LOAD SAMPLES

- A. Vent the chamber.
1. PRESS and HOLD the **VENT** button until it activates.
 2. UNLATCH the door latch.

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3. Wait until **VENT** light stops flashing. Instrument will beep once (door may open by itself before that!).
- B. Insert specimen holder.
 1. Use the Specimen Inserting Tool to load sample holder into the chamber.
 2. **CLOSE** the door and latch.
 3. **PRESS** and **HOLD EVAC** button until it activates.
- C. Turn on the LV mode at this point for faster evacuation.
 1. Lift the plastic lid on the LV control and **PRESS** the white button.
 2. **PRESS** the **AUTO** button.
 3. Dial in the desired pressure on the **CONTROLLER** knob.
 - a. Start at a low number, e.g. 30 Pa, and try to image at this pressure.
 - b. If charging is a problem, you may increase the pressure at any time by pressing the Auto button and dialing in a higher pressure.
 - c. The pressure reading on the vacuum gage will flash until that pressure is achieved in the specimen chamber.

III. INITIAL SETUP

Proceed with setup while instrument is pumping down.

- A. Set accelerating voltage.
 1. From the Menu Screen, **SELECT F2 MENU** .
 2. **SELECT ACCV**.
 3. **SELECT** the desired accelerating voltage.
 - a. Use 20 kV for most applications, including EDS analysis, at working distance 8-15 mm.
 - b. For greater working distance, increase accelerating voltage to 25-30 kV.
- B. Set computer to appropriate sample holder.
 1. From the Menu Screen, **SELECT F5 STAGE** .
 2. **POINT** to and **SELECT HOLDER**.
 3. **POINT** to and **SELECT** the picture of the specimen holder that you put in.
- C. Set the stage height.
 1. For low vacuum microscopy, a short working distance of 15 mm or less to the top of the sample should be used (the upward limit of z is 8 mm).
 2. Switch on the chamber scope.
 - a. **PRESS** the white button at the right side of the chamber scope control (GW Electronics box on top of the SEM console).
 - b. **PRESS** the **POWER** button at the far right side of the chamber scope monitor (Sony monitor on top of SEM console).
 - c. Make certain the A/B channel button is **OUT** (A-position on push-button switch at left side of Sony monitor controls).
 3. Manually set the stage height using the joy stick.

When raising the stage, set the desired working height at the highest part of the sample. DO NOT allow the sample to contact the objective lens or the BSE detector.

- a. **PRESS** the **T/Z** button to change to the "Z" the function of the joystick.

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- b. Raise stage by pushing joystick away from you (positive Y direction), lower by moving joystick toward you (negative Y direction).
 - c. On the chamber scope monitor, observe the position of the sample relative to the bottom of the objective lens.
 - d. PRESS the **X/Y** button to change back to the "X-Y" the function of the joystick.
- D. Move the sample position to an appropriate place on the sample or holder. (For the most part, beam saturation and alignment can be done anywhere on the sample. However, to avoid damaging the sample, it is recommended that setup be done on the rim of the sample holder.
1. TURN the **MAGNIFICATION** knob counterclockwise to the lowest magnification.
 2. Make sure the **X/Y** button is lit on the keyboard.
 3. The Menu Screen should be on *F5 STAGE* menu.
 - a. Point and click the mouse on some part of the sample holder rim.
 - b. A dialog box will appear. SELECT "Start" to move to the point.

Watch the sample on the chamber scope monitor. If the sample appears that it might, for whatever reason, contact the BSE detector, immediately move the joystick in any direction to abort the move.

IV. INSTRUMENT STARTUP

You may proceed with this section once "HT Ready" is displayed in the Status Panel.

A. Filament saturation.

1. Switch off the chamber scope light.
2. PRESS **SPOT SIZE** button.
 - a. Use **CRS** knob to set spot size to midrange on the Status Panel.
 - b. PRESS **TONE** button.
3. PRESS **LSP1** button.
 - a. ADJUST the **BRT** knob to approximately center the line on the monitor.
 - b. As you reach filament saturation, it may be necessary to adjust both **BRT** and **CONT** knobs to maintain the line within the middle portion of the monitor.
4. PRESS **HT** button. The **FIL**ament light will flash until you begin heating the filament.
5. PRESS the lower left corner of the Photo Panel to release the lid and expose the Filament Panel.
 - a. Slowly TURN the **FILAMENT** knob clockwise to heat the filament.
 - b. TURN the knob past the false peak until the line profile no longer rises. Leave the knob just at the point of saturation (Do not turn past 3:00 o'clock).

If the **FILament light begins flashing after you have reached saturation or if it never stops flashing, the filament is burned out. Notify the lab manager.**

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7. PRESS **TONE** button.
- E. Begin work on your sample.
 1. Use joystick to move to your sample.
 2. Refer to other sections of the manual for other procedures.
 3. After completion of work, return to the next section below for shut-down procedures.

V. INSTRUMENT SHUTDOWN

- A. Turn filament off.
 1. Raise Photo Panel lid and TURN **FILAMENT** knob fully counterclockwise.
 2. PRESS **HT** button off.
 3. Close Photo Panel lid.
- B. Return stage to exchange position.
 1. TURN on the chamber scope monitor and light.
 2. From the Menu Screen, SELECT *F5 STAGE* menu.
 - a. If tall samples are present, manually move the stage down until all samples are well below from the objective lens.
 - b. SELECT "*Low exchange*" from the File List if large samples are in the chamber, otherwise SELECT "*Exchange*" from File List.
 - c. SELECT "Start."

Watch the sample on the chamber scope monitor. If the sample appears that it might, for any reason, contact the BSE detector, immediately move the joystick in any direction to abort the move.

4. Wait for a few minutes for filament to cool before continuing.
- C. Remove sample holder.
 1. PRESS and HOLD the **VENT** button until it activates.
 2. Lift the plastic lid on the LV control and PRESS the white button (it will go off shortly).
 3. UNLATCH the door latch.
 4. Wait until **VENT** light stops flashing. Instrument will beep once (door may open by itself before that!).
 5. Use the Specimen Inserting Tool to remove sample holder from the chamber.
 6. CLOSE the door and latch.
 7. PRESS and HOLD **EVAC** button until it activates.
- D. Secure the instrument.
 1. Turn off the LV mode.
 - a. Lift the plastic lid on the LV control and PRESS the white button.
 2. Set image mode to SEI.
 - a. From the Menu Screen, SELECT *F2 MENU*.
 - b. SELECT *IMS* and then option *#1 SEI*.
 3. Turn down both JEOL monitors by turning two thumb knobs under each monitor fully counterclockwise.
 4. PRESS **SL3** button on keyboard.
 5. PRESS **D-MAG** button off.
 6. SET magnification to 300,000x.

Low Vacuum Operation

7. Turn off chamber scope and monitor.
 8. SWITCH the **MET/GND** switch (little blue box behind column) to **MET**.
- E. Fill out logbook.
- F. Turn off computer.

Procedures

MICROSCOPE PROCEDURES

PRIMARY IMAGING

I. SECONDARY AND BACKSCATTERED ELECTRON IMAGING

- A. The secondary electron (SE) detector can be used in any mode of operation EXCEPT low vacuum.
1. The SE detector will be automatically turned off and the backscattered detector will be switched on in low vacuum operation
 2. To switch between detectors in high vacuum mode:
 - a. From the Menu Screen, SELECT *F2 MENU* .
 - b. SELECT **IMS**.
 - 1) SELECT the **SEI** (#1) detector for secondary electron imaging.
 - 2) SELECT the **BSE Compo** (#2) detector for compositional or "shadowed" topographical backscattered electron imaging (BSE Topo [#3]) is used to emphasize the DIFFERENCES in topography rather than produce a topographic image).
 - 3) SELECT **AUX** (#8) for cathodoluminescence imaging (see below).
 - 4) No other imaging modes can be activated from this menu.
- B. The BSE detector can used in any mode of operation.
1. During low vacuum operation, it is the ONLY image detector available.
 2. For collection of topographical images (especially in low vacuum mode), the "shadow" detector must be switched on.
 - a. From the Menu Screen, SELECT *F2 MENU* .
 - b. SELECT **BEIW**.
 - c. In the BEIW window, SWITCH SHADOW to **ON**.
 - d. On the SHADOW ENHANCE slider, set the degree of topographic enhancement (settings between 5 and 7 seem to work best).
 - 1) Higher values produce more topographic contrast, but darken the image.
 - 2) Lower values result in less topographic contrast and brighter images.
 3. For pure BSE images, e.g. working on polished thin sections, switch the shadow detector off.
 4. SELECT scan speed SL1.

II. OPERATION OF THE CL DETECTOR

Read the introductory section for tips on how to use the CL detector.

- A. Setting up the SEM for CL work.
1. Mount the sample.
 - a. Use polished, carbon-coated samples (probe sections or polished slabs).
 - b. Mount your sample in the thin section holder as you would with a probe section, or mount directly on an appropriate size aluminum sample stub with carbon tape (don't forget to provide a conductive path from the sample surface).
 - c. The sample should not protrude above the rim of the aluminum sample holder.

Procedures

2. Load the sample.
 - a. Make sure that:
 - 1) The stage is not tilted ($T=0$).
 - 2) Stage X, Y, and R should be at approximately 0.
 - 3) Stage height is at $Z=25$ or greater.
 - 4) The CL detector is withdrawn (large silver knob fully clockwise).
 - b. Vent the chamber as normal.
 - c. Insert the sample into the SEM.
 - d. Set sample height.
 - 1) While the door is open, insert the CL mirror by turning the large silver knob on the luminescence detector counterclockwise. Turn the knob gently until it stops.
 - 2) Watch while you move the stage upward until the sample surface is about 1 mm below the mirror. The Z setting should now be around 15-17 mm depending on sample height.

Do not drive the stage into the mirror! !

- 3) Retract the mirror by turning the silver knob clockwise. The mirror is fully retracted when you come to a gentle "bump" and the knob will turn no farther.

- e. Close the door and latch. Evacuate the chamber as normal.

3. Setup the SEM.
 - a. Make sure the Chamberscope IR light is off.
 - b. Check initial settings on the PA1 amplifier (Oxford Instruments box on top of the SEM console):
 - 1) Gain = 5.0
 - 2) Offset= 5.0
 - 3) X100/X1000 switch in up (X100) position.
 - 4) Invert/Normal switch between Amplifier Overload light and Input Overload light is set to the left (Normal image).
 - 5) The unit is switched off (green LED light should not be lit).
 - c. Set the accelerating voltage at 10 to 15kV (or less).

B. Operation.

1. Bring up the beam as normal.
 - a. Optimize the gun and column alignment for a spot size at midrange or above (>>>>>>>>> >>>>>).

It is important to have the beam in optimum alignment for CL work—don't shortcut this step! Refer to IV.B. through IV.D. in the HIGH VACUUM section above.

2. Navigate to the area of interest on your sample.
3. Obtain a focused good secondary electron image on the sample.
4. Carefully insert the mirror.
 - a. Set the magnification to its lowest setting and scan rate to **TV1**.

Procedures

- b. Rotate the large silver knob slowly counterclockwise while watching the SE image.
 - 1) If the SE image of the sample moves during insertion (mirror has bumped the sample), retract the mirror, lower the stage slightly, and try again.
 - 2) At low magnification (<100x) you should be able to see the hole in the mirror when it is fully inserted.
- c. Center the hole in the mirror vertically in the SE image by finely adjusting the large silver knob.
 - 1) The secondary electron image will be seriously degraded by inserting the mirror. This is normal.
 - 2) Obtain a reasonable secondary electron image by adjusting SEM contrast and brightness.
 - 3) Increase the magnification to a point where you can no longer see the hole in the mirror.
5. Acquire an image from the CL detector.
 - a. Turn on the power to the PA1 amplifier (black button at the lower right).
 - b. Switch to the CL image.
 - 1) From the Menu Screen, SELECT *F2 MENU* .
 - 2) SELECT **IMS**, then SELECT **AUX (#8)** from the IMS window.
 - 3) SELECT **SL1** or **SL2** on the keyboard.

Do not allow the detector to be flooded with too much light. If the chamberscope IR light is left on, the door opened with the PA1 left on, or if samples luminesce too strongly, the amplifier will switch off and the red overload light will come on. Correct the problem before resetting the amplifier.

- c. Carefully jog the stage up or down by 1 or 2 mm maximum to find the focal point of the mirror. Look for an increase in brightness at the focal point.
- d. The image contrast and brightness is controlled by:
 - 1) Gain and offset knobs on the PA1 amplifier.
 - 2) Increasing or decreasing beam current.
 - a) If image screen is flooded white, turn gain down or drop the beam current. For extreme CL, set accelerating voltage to a lower kV.
 - b) If image screen is dark, turn gain up or try turning beam current up. For weakly luminescing samples, switch amplification factor to X1000 (use X100 for most things).
 - c) Set gain until an image of bright spots (pixels) can be seen on the screen. Adjust offset down (usually) until individual pixels can be seen and they are not flaring or smearing into each other.
- f. Use a slow scan for correct exposure.
 - 1) Use **SL2** or **SL3** on the JEOL to collect an image on the screen.
 - 2) To collect images on the Isis system, use the Slow scan with the dwell set at 400 μ s or greater. For persistent CL (phosphorescence), which

Procedures

will appear as streaks in the image, increase the dwell time to as high as necessary (up to 6400 μ s) to eliminate streaking.

- 3) It is best to use the smallest, or lowest resolution (Medium scan), image on the Oxford to set up the exposure, then switch to Fine or Ultrafine scan to collect the final image.
- g. For moving about your sample you will probably find it most convenient to retract the mirror, returning to SE or BSE imaging.
 - 1) Because the mirror blocks electrons from reaching the BSE detector, BSE imaging cannot be used with the mirror inserted.
 - 2) Likewise, the mirror also blocks x-rays from reaching the EDS detector, so it cannot be used with the mirror inserted.

Do not rotate or tilt the stage or move Z closer than 15 mm with the mirror inserted!

- C. Shut off the CL detector and retract the mirror.
 1. Switch the PA1 amplifier off.
 2. Retract the mirror by rotating the large silver knob fully clockwise.
 3. If you are finished with the CL detector, return PA1 settings to initial values (see section A.3. above)

Do not open the chamber without first turning the CL detector off!

OXFORD SYSTEM PROCEDURES

DIGITAL IMAGING

The Oxford Isis system consists of specialized programs that are accessed as program modules selected from the control and database program (“Labbook”). The programs available are: X-Ray Analysis, Autobeam (digital image acquisition), Detector Control, Autostage (automated stage control), IMQuant (digital image analysis) and Notebook (report writing). From within X-ray Analysis, the available programs are Quant, SpeedMap, Quantmap, Linescan and Autocolumn. Cameo and Phase Measurement are available in Autobeam.

I. ELECTRON AND CATHODOLUMINESCENCE IMAGING

A. Start Isis imaging software.

1. Turn on the HP computer and monitor.
 - a. PRESS any key (spacebar) to start the computer (Windows™ will load automatically).
 - b. If green or yellow light is on present at the lower right the of the monitor, it is already on; otherwise turn monitor on at switch (lower right front).
2. Start and setup Isis “Labbook”
 - a. SELECT (double-click on) the “Link ISIS” shortcut icon from the desktop.



- 1) After a short time, the “Welcome to Link ISIS” window will appear; SELECT your name from the list of operators.
- 2) CLICK the small blue labbook icon or double-click on your name.
- b. Next the “Labbok” window will appear (Fig. 3).
 - 1) Your last job file will appear by default, but if necessary, change to the appropriate job file or create a new one (see section c. below). All your spectra and images should be stored in your own job file--don’t use someone else’s.
 - 2) CLICK **Autobeam button** (second from left).
- c. To change or setup new jobfile.
 - 1) CLICK **JOBS...** button.
 - 2) Double-click on one of your available job files or setup new.
 - a) PULL DOWN “**Jobs**” menu.
 - b) SELECT “**Create a new job...**”
 - c) PRESS “**Yes**” on the Job Creation dialog box.
 - d) Type in Job Description and Client Name.
 - e) PRESS “**OK**”
 - 3) Your job file is now available for use.

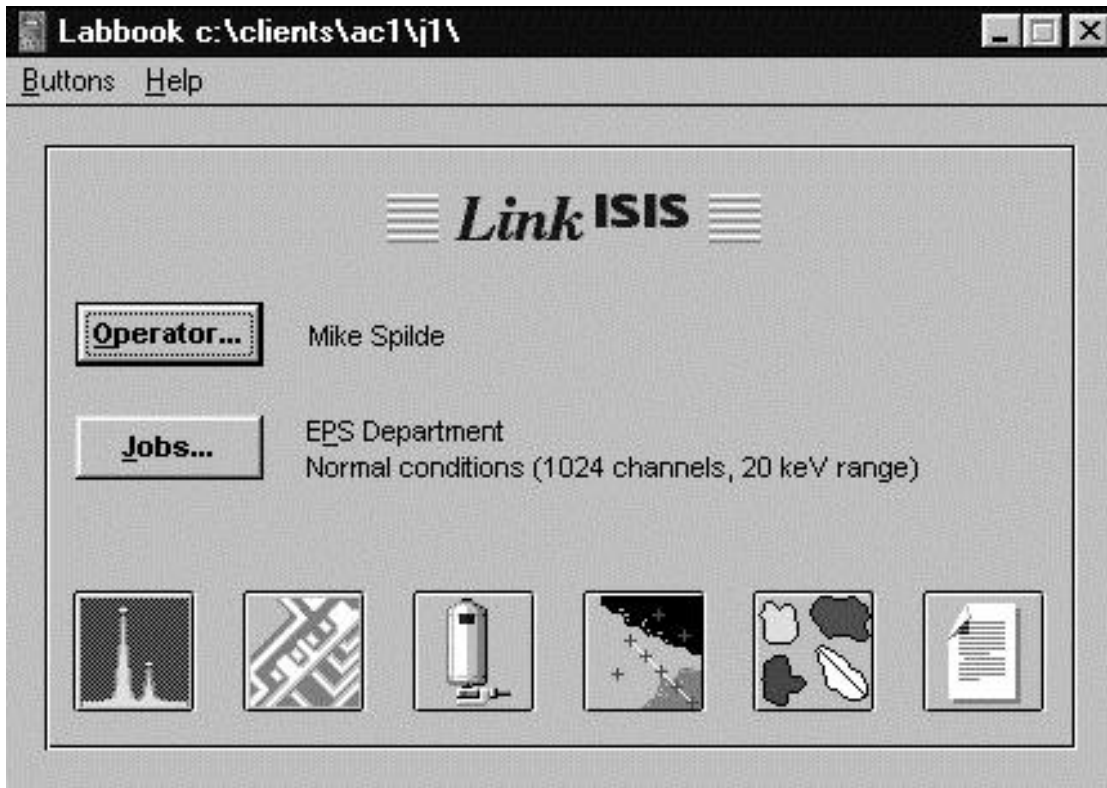


Figure 3. Oxford Labbook window. Bottom row of symbols are buttons leading to the various programs for X-Ray analysis, digital imaging, EDS detector setup, auto point analysis, feature analysis and a notebook program.

- B. Set image and acquisition mode.
 1. The image mode (SE, BSE, etc.) must be selected at the microscope using the IMS Menu (See Section I under Microscope Procedures).
 2. Setup image acquisition mode.
 - a. PULL DOWN **Edit** menu and SELECT “**Image setup...**”
 - b. Set image size and scan speed according to type of imaging and image quality (See Tables 1 and 2).
 - 1) Larger image sizes provide the highest resolution (least pixellated) but require longer time for acquisition. 512 x 368 is generally a good compromise; however, if you anticipate projecting or publishing an image, 1024 x 736 is the most desirable.
 - 2) Slower scans provide sharpest and most noise-free images but require more time for acquisition. Medium and slow scan speeds are required for low current (high magnification) and BSE images.
 - c. Make sure “Mains synchronize” is checked.
 - d. CLICK “**OK**”
 3. Adjust contrast and brightness on microscope.
 - a. PULL DOWN **View** menu and SELECT “**Signal calibration...**”
 - b. Adjust contrast and brightness controls on the microscope to get the proper image intensity.

Table 1. Image resolutions available on the SEM.

| | Horizontal | Vertical | No. of pixels | Storage Requirement |
|--------------------|------------|----------|---------------|---------------------|
| Oxford Isis System | | | | |
| Medium | 128 | 92 | 11,776 | 13 KB |
| Fine | 256 | 184 | 47,104 | 52 KB |
| Ultrafine | 512 | 368 | 188,416 | 200 KB |
| High | 1024 | 736 | 753,664 | 775 KB |
| JEOL TIFF Format* | 1280 | 960 | 1,228,800 | 1.4 MB |

*JEOL format can only be saved on floppy disk.

- 1) In the image window, red areas are oversaturated and green are midlevel gray.
- 2) Viewing the line trace, contrast controls the amplitude and brightness controls the level of the line.
- 3) Generally you want the greatest contrast range without oversaturation or undersaturation in most image areas (e.g. epoxy mounting medium could be undersaturated black without loss of real data).

c. CLICK “Close”

C. Collect and store image.

1. CLICK **start button** (the left-most of the menu buttons, with round dot).
2. Immediately after image is acquired, CLICK **stop button** (second from left with square dot).
3. Save the image.
 - a. PULL DOWN **File** menu and SELECT “**Save image...**”
 - b. Type a title and CLICK “**OK**”
4. Scale bar may be switched on/off by selecting “Scale Bar...” from the View menu.

Table 2. Image acquisition time on the Oxford Isis System.

| Resolution | Pixel Size | Scan Speed | Time | Comments |
|------------|------------|------------|--------------|---|
| Medium | 128 x 92 | Fast | 3 sec | Use to check initial image setup (scan speed) |
| | | Medium | 5 sec | |
| | | Slow | 8 sec | |
| | | Kalman* | 3 sec/frame | |
| Fine | 256 x 184 | Fast | 5 sec | Good for mapping, quick initial images |
| | | Medium | 8 sec | |
| | | Slow | 25 sec | |
| | | Kalman | 5 sec/frame | |
| Ultrafine | 512 x 368 | Fast | 8 sec | Good for mapping and general purpose imaging |
| | | Medium | 28 sec | |
| | | Slow | 1.5 min | |
| | | Kalman | 8 sec/frame | |
| High | 1024 x 736 | Fast | 20 sec | Use for publication-quality images and slides |
| | | Medium | 1.5 min | |
| | | Slow | 5.5 min | |
| | | Kalman | 20 sec/frame | |

*Kalman averaging acquires and mathematically averages together any number of fast scan frames so total time depends on the number of frames collected.

II. EXPORTING DIGITAL IMAGES.

Digital images are saved in Oxford's image format and must be converted to tiff or bitmap format and exported before they can be read by other image processing or printing programs.

- A. Open the image to export.
 1. Scale bar may be switched on/off by selecting "**Scale Bar...**" from the **View** menu.
 - a. Note that once the scale bar is added to an exported image, the bar and its text cannot be manipulated, it becomes part of the image.
 - 1) An alternative to the less-than-nice-looking Oxford scale bar is to export 2 images, one with a scale and one without.
 - 2) A scale bar of your liking can then be added to the unscaled in a more functional program, such as Photoshop, using the scaled image for reference.
 - b. A scale can also be calculated from the formula $L = 130/M$ where L is image width (in mm) and M is magnification.
- B. Export the image in either a TIFF format or BMP format.
 1. PULL DOWN **File** menu and SELECT "**Export image...**"
 2. In the dialog window, select the file destination (a folder outside of the current Oxford folder that you are in).
 - a. For ftp download or upload, select D:\!Downloads and select a file.
 - b. For direct transfer to Zip-disk, select drive H.
 3. Enter a file name, 8 characters or less (the .TIF or .BMP extension will automatically be added).
 4. CLICK "**OK**"

X-RAY ANALYSIS

The X-ray analyzer provides access to the EDS system for X-ray collection. Available from the analyzer are programs for auto-identification of peak, quantitative analysis, normal and quantitative EDS mapping, linescans, and beam control for automated analysis.

I. QUALITATIVE EDS ANALYSIS

- A. Start Isis software and select your job file (See Section I.A. under Digital Imaging).
- B. Set process time and acquisition time.
 1. PULL DOWN **Edit** menu and SELECT “**X-ray setup...**”
 2. Type in acquisition time (default is 50 seconds).
 3. Check “Acquisition Preset” window for the process time used.
 - a. For fast acquisition but low resolution (Process Time 1), PRESS the “Optimum” button.
 - b. For best resolution of light elements (Process Time 6),PRESS the “Best Resolution” button.
 - c. If quantitative analysis will be proformed, CLICK the “**More...**” button and select Process Time 4 form the pull-down menu
 4. CLICK “**OK**”
- C. Select target and begin acquisition.
 1. Place the electron beam on the target area by one of these methods:
 - a. Move stage to the area of interest and zoom the magnification (e.g. 100,000x or greater),
 - b. Collect an image on the SEM on the right-hand CRT.
 - 1) From the Menu Screen, SELECT *F2 MENU* .
 - 2) SELECT **EDX** and drag the cursor cross with the left mouse button depressed to the point of interest.
 - 3) SELECT **SET** to position beam on point; select again to relaese.
 - c. Collect an image with the Oxford Autobeam software.
 - 1) CLICK the left mouse button with the curser on the image at the point of interest.
 - 2) Release beam when finished by clicking on right mouse button.
 3. CLICK the round button on the analyzer button bar to begin acquisition.
 4. Use ?-button to find and label peaks.
- D. Exporting Spectra
 1. To export as a graphic plot:
(Note: the TIFF export under the File Menu copies the solid-color spectrum may be problematic for some graphics software.)
 - a. CLICK printer button.
 - b. PULL DOWN **File** menu and SELECT “**Export as TIFF (line spectrum)**”
 - c. Select destination and enter file name; CLICK “**OK**”
 2. To export as an ASCII file:

(Note: this method produces a comma separated text file of the spectrum with a header. There are 1024 data points in 5 columns. It can be read by Excel but works best with the freeware program Desktop Spectrum Analyzer from NIST.)

- a. PULL DOWN **File** menu and SELECT “**Spectrum Conversion**”
 - 1) SELECT “**Conversion Type ...**”
 - 2) CLICK the EMSA/MAS button and CLICK “OK”.
- b. PULL DOWN **File** menu again and SELECT “**Export**”
- c. Select destination and enter file name; CLICK “OK”

II. QUANTITATIVE ANALYSIS

III. X-RAY MAPPING

- A. X-Ray Map Setup
- B. Map Data Analysis
 1. Contrast and brightness adjustments
 2. Map mixing and overlays
 3. Phase Measurement

IV. X-RAY LINESCANS

V. AUTOMATION

IMAGE AND DATA ANALYSIS

I. X-RAY MAP ANALYSIS

- A. Basic Map Analysis
- B. Cameo Analysis
- C. Phase Map Analysis

Open Autobeam

Press Cameo button

Select open image from File menu to open speed map or quantmap data.

Select SE/BSE image (must select one)

Hold Ctrl key and select 2 or 3 x-ray maps

- Selecting 2 maps results in X-Y plot of 2 colors with lower 2 plotted on X-axis and higher 2 on Y axis.
- Selecting 3 maps results in ternary plot of 3 colors lowest 2 is assigned to red in lower right apex
Next 2 assigned green at upper apex
Highest 2 assigned blue at lower left apex

Press Phase Map button an Open File dialog window

Press the triangle button on the Autobeam window (A ternary scatter diagram will be plotted in the Phase measurement window. The various phases should

appear as groups of clusters. If an x-ray map has low counts, empty diagonal areas or lines will appear on the plot.

Select 1st phase, define a name (if desired) Define phase by clicking left mouse button on ternary diagram and draw a series of line segments (hold left mouse button and draw) around phase or cluster of interest. Double click to enclose phase completely. Area % of that cluster will be displayed.

To add a new phase click on Phase button in Area Measurement window. Define name and outline phase. Up to 8 phases may be defined.

To change phase color, click on color box in Phase Measurement window.

It is useful to identify all phases in a map area first, then paint each phase individually by selecting the phase name then press Paint button to identify and paint just that phase in the autobeam window.

Once phases are defined, named, etc. use the Paint All button to paint the Autobeam window with the defined phase colors. Note: if white saturated areas are present in the BSE image (e.g. from high-2 phases), it may be necessary to open the image first and lower contrast and brightness slightly (with Cameo button off) before starting this process. Otherwise the colors do not show up in the white areas of the image.

Use Measure button to print phase proportion data. To print graphical phase map, hit Copy button to save to clipboard and paste as picture into a program such as Word or a graphics program (can also be saved as clipboard File .CLP).

II. IMQUANT

APPENDICES

A. General Information

- I. Calibrate stage to sample height
(Focus at the top or highest point of sample.)
 1. Clear lens by pressing Reset OL button (Refocus if necessary).
 2. Enter command: AFT -OFF [Rtn] (turns off auto focus).
 3. Enter command: AFC-I [Rtn] (initialize stage).
 4. Enter command: WD [Rtn] (set working distance) .
 - a. Displays WD without decimal point
 - b. Use coarse and find focus to set reading to 200.
 - c. Press “E.”
 5. Clear the lens again.
 6. Use joystick to set mechanical Z to focus
 - a. Press T/Z button.
 - b. Move joystick up or down to bring sample into focus.
 7. Enter command: Stage - IZ [Rtn] (sets stage to WD height, which should now be 20 mm).

II. Load Current Jump

1. If the load current in the status monitor window should suddenly jump above 100 uA, it **MUST** be reset or the filament will burn out.
2. Press command F1, type: “Bias”, and hit Return.
 - a. The status monitor will display:

| | |
|--------------------------------|--------------------|
| <code>cmd>bias E/I/D</code> | <code>#####</code> |
| <code>XXuA</code> | |

3. Note the hexadecimal number in the upper right.
4. Turn focus knob one or two clicks to the right and then back to where it was (hexidecimal number should change and then return to original number)
5. Hit “E” to exit.