Scanning Electron Microscope Operation

Zeiss Supra-40

Roger Robbins

9/10/2010
Update: 6/22/2011

Erik Jonsson School of Engineering
The University of Texas at Dallas

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Introduction

[General introduction to the scope and purpose of this document.]

This is a step-by-step operation manual written for the Zeiss Supra-40 Scanning Electron Microscope at the University of Texas at Dallas Cleanroom, including the numerous optional subsystems mounted on this tool. In general the material presented here informs the reader about how the tool works in the order that an operator would proceed in using the tool. Included at the end of each section is a skeletal outline of procedural steps so the newly trained operator can follow the order of operation without having to read the in-depth explanations during real time use.

Survey Description of SEM

[Describe reason for following survey topics and limitation – i.e. describing purpose of sensors and when they would be used.]

SEM Concept

[Describe physics of electron beam formation, control and material interaction creating return electrons. How does the scanning principle create a magnified image?]

EDAX Concept

[Describe physics concept of EDAX operation and what information it acquires – when it would be used, etc.]

Secondary Electron Detectors

[What are secondary electrons, where do they come from? How does electron detector work? Describe principle of operation of both Everhart-Thornton and in-lens detectors. What information do these detectors acquire and how/when are they used.]

Backscatter Electron Detector

[What is a backscattered electron and where does it come from? How are these electrons different from secondary electrons? What information do they contain? How does the backscatter electron detector work?]

Electron Back Scatter Diffraction Detector

[What is back scatter diffraction? What is it for? How is the diffraction pattern detected and what electrons does it acquire?]

Scanning Transmission Electron Microscope System

[Explain what a scanning transmission electron microscope is. How are electrons detected? What electrons are detected? Where is the detector? How do electrons get through material? What does the image look like? What information does it display? When do you use it?]

Our Scanning Electron Microscope has an optional accessory that enables the system to produce Scanning Transmission Electron Microscope (STEM) images. This configuration sets the sample to be imaged on a special sample mount such that the STEM detector, which is mounted on a long rod that extends out of the side of the sample chamber, can align to the underside of the sample. Electrons from the column penetrate the thinned sample and collide with atoms in the sample and then pass through the sample and are detected by diode detectors at the end of the probe.

The STEM can produce very high resolution material density images in both bright and dark field modes with high signal to noise ratio and sharp contrast. It is used to examine the cross section of semiconductor device elements, crystal grain boundaries as well as other material boundaries. The EDAX material analysis system can identify properties of highly localized features in thin samples rather easily.

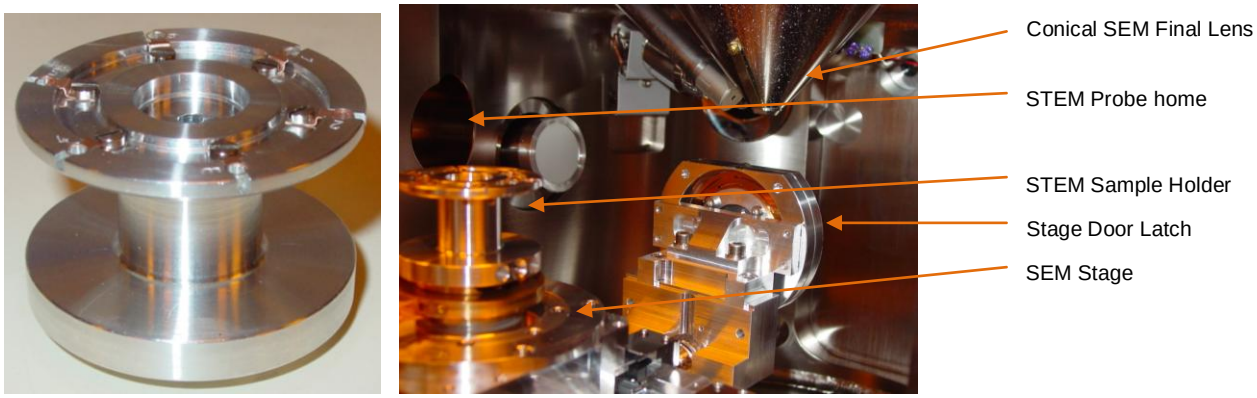

Figure 1. Images of STEM sample holder (left) and SEM stage (right)

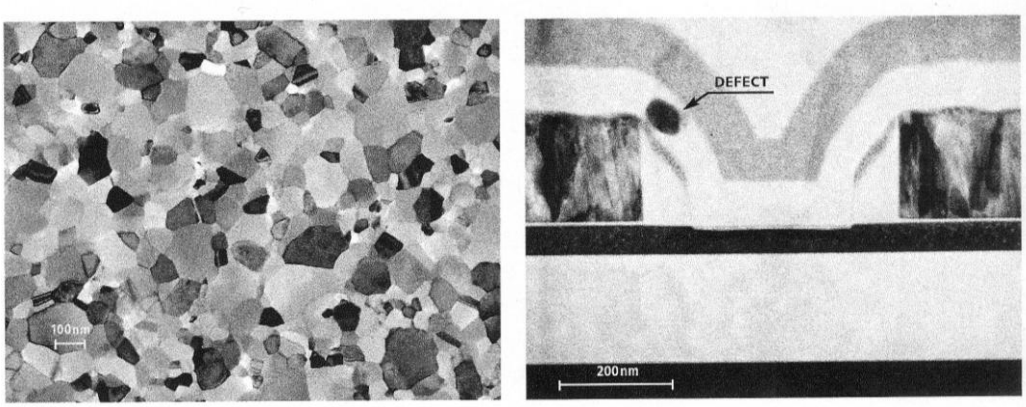

Figure 2. Examples of STEM images: Crystallites (left) and semiconductor device defect (right).

Nano-Manipulator Stage

[Describe the purpose of the nano-manipulator and the structure of the stage. What is special about the probes? In general, how is it used? How small of an object can it touch, probe or move? When do you use it? What is involved in installing and setting it up? Staff only setup.]

Low Noise Electrical Probe System

[General brief description of the low noise probe system. This is a specialty system…]

Electron Beam Lithography System

[Describe what the electron beam lithography system is and how it is controlled. What are the writing specs? i.e. field size, critical dimension limits, substrate capability, alignment capability. What is this specialty system used for?]

Operating Instructions for SEM

Introduction

[Note that this is the detailed description section for operating the SEM imaging system.]

Starting Check List

[Before using the system, determine what condition/configuration the SEM is in – it should be in a standard state. List critical items that must be checked before operating the SEM.]

In the case that you arrive at the SEM for your scheduled time and the SEM has a previous user still logged in to the system, please take a moment to check the status. Is the system powered up? Is someone still using the system? Is there a handwritten sign on the table? Check the log book – look at the Start time/Finish time. The convention has now been established that when a user is finished, the word "DONE" is written in the logbook to show courtesy to the next user that the machine is ready. Are there comments in the logbook noting SEM condition/problems? Is the SmartSEM software active? Also, check to see if any extendable detector probe is inserted into the chamber. Who is logged in? (Check the User ID in the far upper left corner of the SEM screen). If someone is still using the system find them and ask if they are still active on the SEM. We want to avoid destroying someone's analysis setup if they are just briefly away from the SEM. On the other hand if someone has walked away and left the system without logging off, we don't want to delay your time on the system needlessly. If you can't find anyone, and the system is still logged in by someone, contact a staff member for permission to start.

Operating Procedure

[Detailed description of simple SEM imaging operation from a new user's perspective.]

Time Scheduling and LogbookSTEP 1.

In order to acquire authorization to use the SEM, you first must reserve time on the SEM through the SEM scheduling WEB site. It is currently found at the URL address:

http://www.utdallas.edu/~hcf011000/cgi-bin/Login.pl

Return to STEP 1.

There is also a paper logbook at the SEM that users must fill out completely. It is for the purpose of recording the actual SEM use in order to help staff with problem solving as well as administrative details. This gives the staff archival information on what the tool is used for, how long it is to be used, who is using it, and for what subsystem. In addition, the blank Purpose/comments area gives you the space to tell the staff about SEM problems or remind you what you did during this SEM session, etc. Statements on what you were doing also help staff understand how the SEM is being used and enable better support. Figure 3 shows a sample log sheet. Return to STEP 2

Date _____ **Name** _____ **Supervisor** _____

Start Time_____ Circle Use: SEM EDAX EBSD STEM Nabity Litho Bakscatter

Finish Time_____ **Sample**_____

Purpose/Comments:

Figure 3. Sample SEM log sheet kept at the SEM.

Sample Preparation

Your sample can be mounted on one of a number of sample mounts. The simplest mount is the pin disc made of Aluminum and shown in Figure 4.

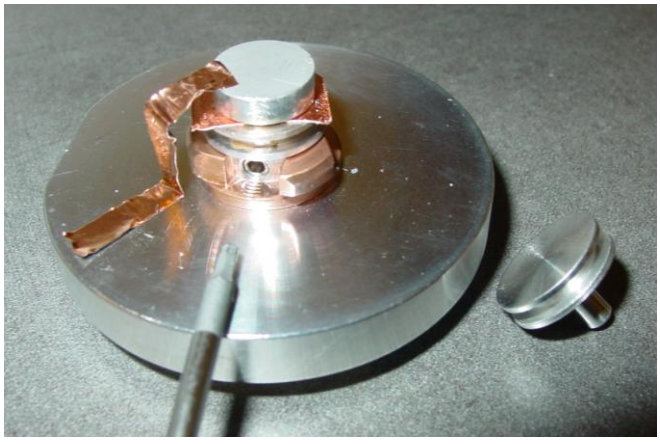

Figure 4. Simple SEM sample mount showing a metal sample attached with sticky conducting tape. The Copper tape affixed to the top of the sample insures a conductive path to ground for the top film, esp. if the substrate is an insulator. Also shown to the right is the removable Aluminum SEM sample Pin Mount. The large round table is the

mounting platform that slides onto the SEM stage. The Allen wrench fixes the little sample mount stem to the mounting platform – do not over tighten.

Sample Holders

There are a huge number of sample holder designs for SEM samples. We have a few, (Figure 5). However, for special sample configurations you should consider purchasing special sample holders that fit your needs, and most importantly, our SEM. Please consult with the Clean Room Staff SEM owner before ordering.

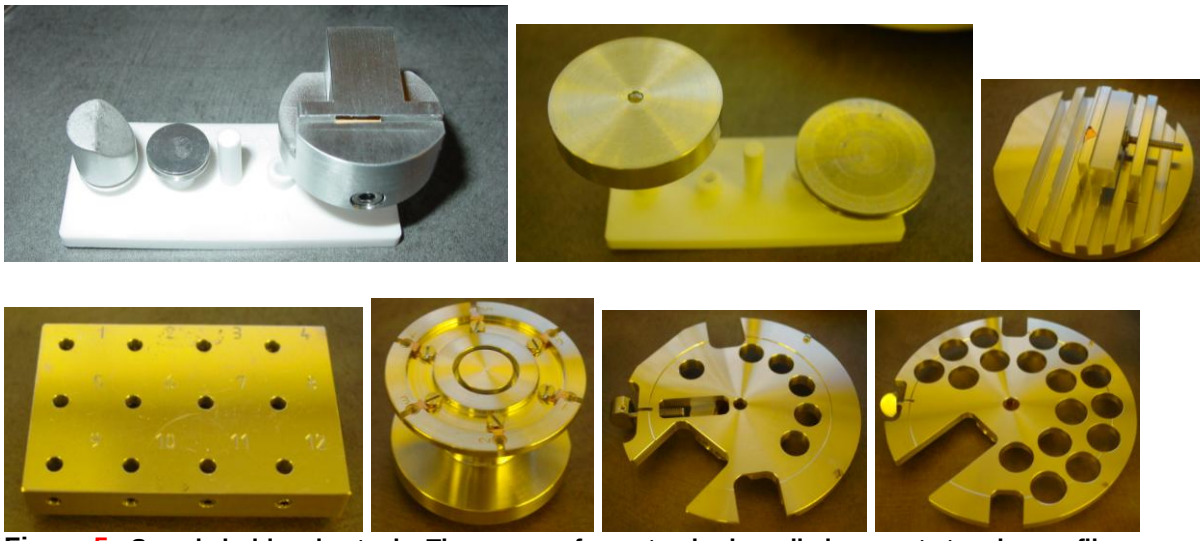

Figure 5. Sample holders in stock. These range from standard small pin mounts to edge profile holders to multiple pin to 4" and 6" wafer holders. There is also a transmission electron microscope sample holder (second from left on bottom row).

Sample Grounding

The sample needs to be electrically connected to the sample holder to prevent the electron beam from "charging" the sample and distorting the image. This is usually done through conductive tape. We have double and single sided Copper sticky tape. If you have a conductive sample this simple attachment will work fine. If you have an insulator substrate with a conductive film on the top, the top film must be electrically connected to the Aluminum sample holder. This can be done with the Copper sticky tape looped around the edge to the top surface of your sample. If your sample is an insulator, then you may need to coat it with a conductive material – i.e. Au/Pd or Carbon.

Sample Coating

Fortunately, we have a "Hummer VI" sputter deposition system[1] located in Bay 5 just for this task. It has a Gold/Palladium target and a sputter deposition rate of about 1.2 Angstroms per second. Thus about 2-4 minutes of sputtering will apply enough metal to conduct the SEM electrons to ground and prevent charging without noticeably altering the topography of your substrate. The "Hummer VI" is shown in Figure 6. Note that if you coat an insulator substrate, you will need to connect the newly conducting top side to ground with a copper tape.

Figure 6. Hummer VI sputter deposition system for coating insulating SEM samples.

Return to STEP 3

SEM Login

You will have a user ID and password assigned to you after initial training. Thus when you arrive at the SEM, the screen will display a standard Microsoft Windows XP scene, waiting for you to execute the SmartSEM software. Click on

[1] Thomas, Brian, "The Hummer VI," www.utdallas.edu/Research/Centers/Cleanroom/Documents/eHummer.pdf , (10/29/2003)

the SmartSEM icon either on the left side of the left screen or the similar but tiny icon in the lower left taskbar of the right screen. This will bring up the Log On window in the left screen, Figure 7. Enter your username and password and click OK. This will bring up the Smart SEM software that runs the SEM.

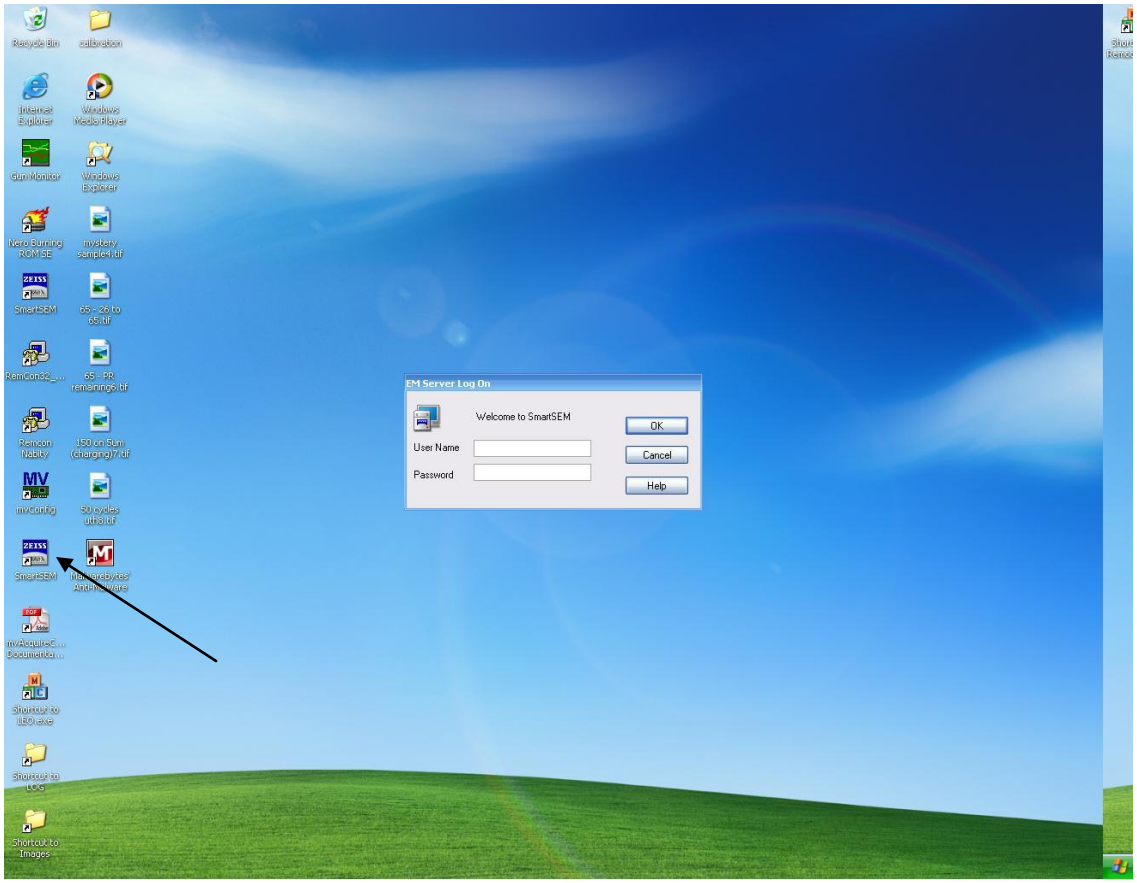

Figure 7. Left screen Log On window. Note the ZEISS Smart SEM icon in the left column 5th icon that calls up the log on window.

SEM Image Screen

The next few steps of preparing the SEM for sample loading and image acquisition require commands from the software. The SEM user interface is spread across two monitors. The user interface screen is shown in Figure 8. The icons in the upper command line are used very frequently in operating the SEM and optimizing the image. The set of 6 tabs on the right side of the screen

contain command and parameter setting buttons for setting the SEM up for imaging. The row of icons at the bottom of the screen has commands allowing you to measure or label things on the image. The SEM image appears in the large region in the center.

Return to STEP 4

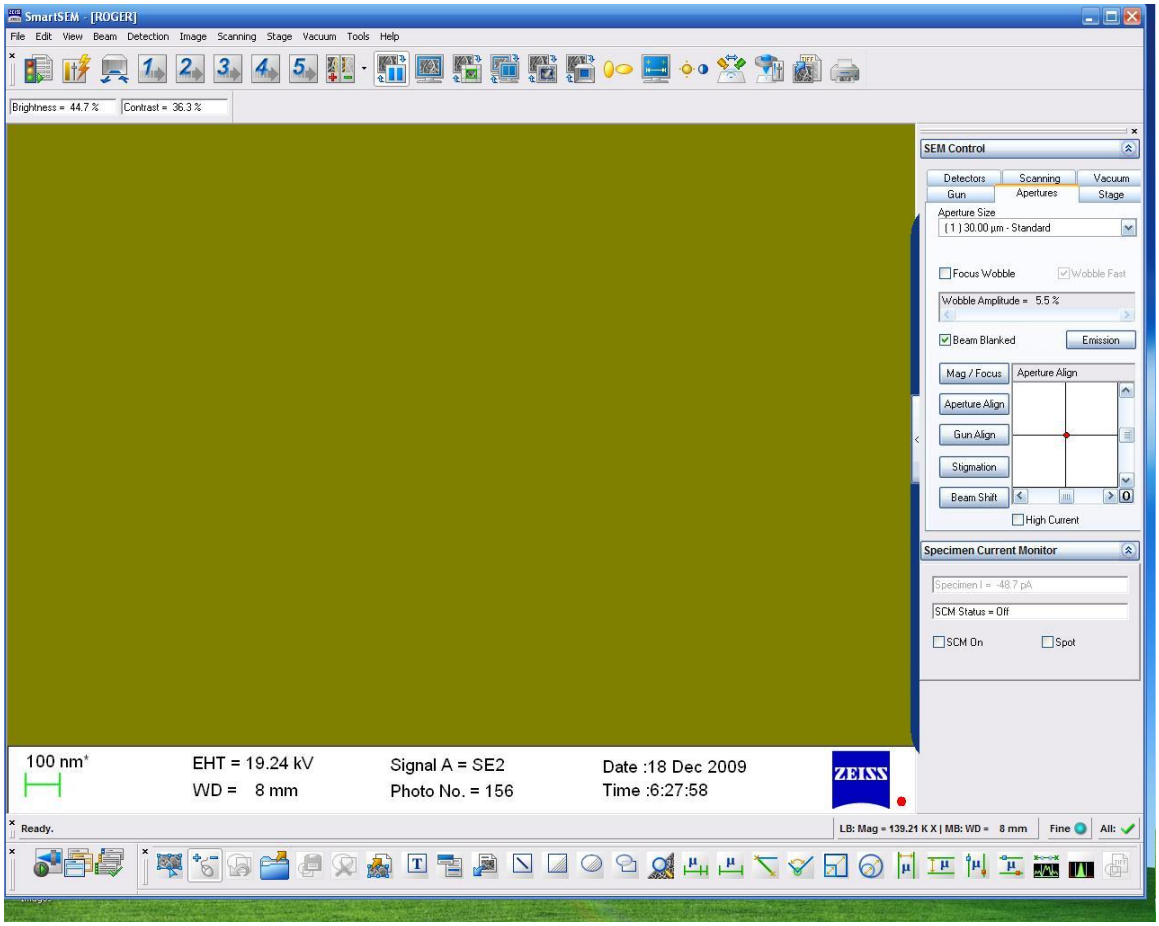

Figure 8. User interface on the Left monitor after Log-On. Note the Imaging control icons in the upper row and the 6 tab command panel at the right side of the screen. Measuring and documentation icons are along the bottom. Also note the dual boxes at the upper left with Brightness = 44.7% and contrast = 36.3%; this is the handy HFP window that shows the value in % range of any adjustment knob on the keyboard. The strip of information at the bottom of the image frame is called the Data Zone and contains the micron bar, EHT, WD, etc – this strip is saved with the image.

One of the handiest tools hidden in the View menu is the Hard Front Panel option. If you pull down the "View" menu and click on the "Toolbars…" command, a new window will appear having an option for "HFP Status" with a check box next to it. Click in the box and close the windows. This will produce a pair of small window boxes with numbers in them. There will be a label assigning these numbers to a parameter pair such as Magnification/Focus which will correspond to the numeric value of the designated knob positions by (+/-) percent

travel from their origin. This conveniently gives you a numeric gauge for the amount of adjustment the knob is providing. If you grab the boundary of this window pair, you can move it to a convenient location in your user interface screen. This way, you will know how much magnification you are requesting, for example. The functions listed in the window match all the adjustment knobs on the keyboard and automatically appear when any of the knobs are just slightly touched.

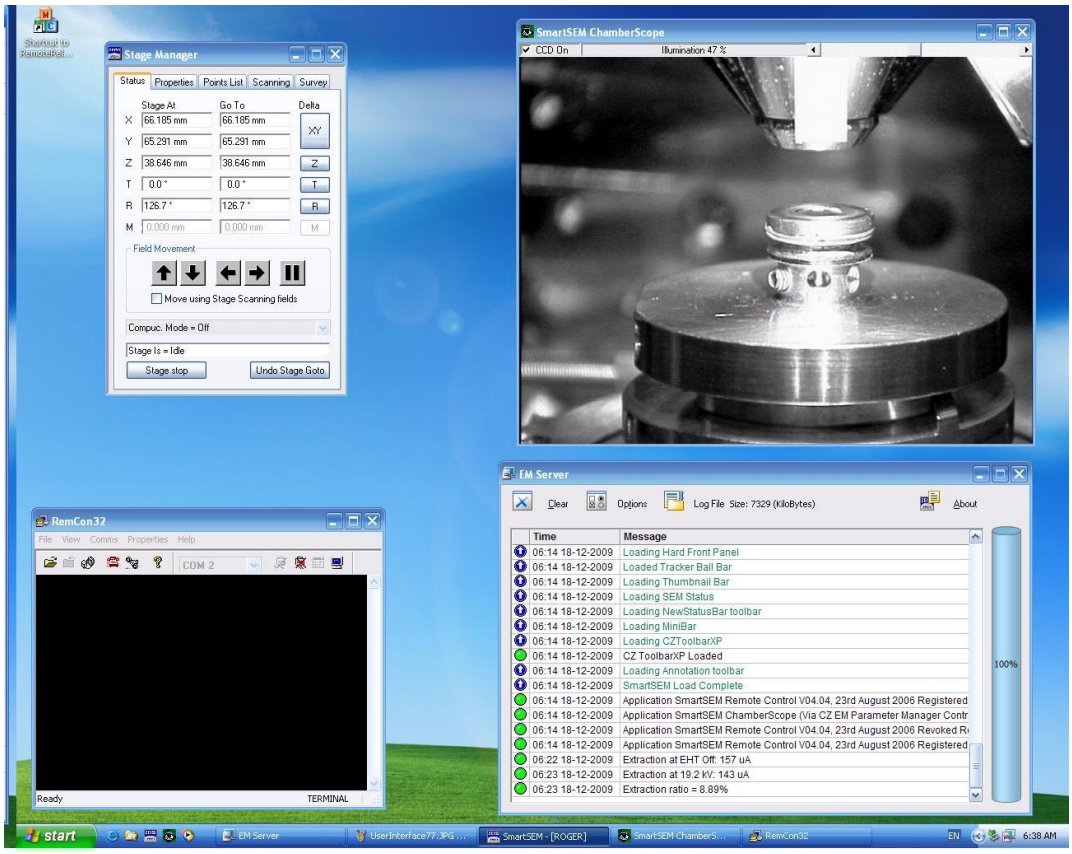

Figure 9. Screen on the right monitor showing the Stage Manager, ChamberScope, RemCon32, and EM Server support windows open. This is a practical assortment of windows for safe operation of the SEM. Note the ChamberScope "Eye" icon at the lower left of the command line.

NOTE: If you change this arrangement of windows during your SEM session, please put them back before you log out so the next user will have a standard display.

Sample Loading

Now that the sample is prepared and mounted on the SEM sample mount, we have to vent the SEM to load it. This is done by clicking on the Sample Exchange icon in the upper left of the SEM image screen, Figure 8.

This will turn off the SEM high voltage and in a little pop-up window ask if you have retracted all extendable probes – (EBSD, STEM, Backscatter detector) – so that the stage will not collide with them and damage the very expensive probes. Before clicking in the popup window, physically go look to see if the probes have been retracted – even if you "know" they have. These "probes" are generally worth as much as a mid-size Mercedes Benz! Make sure of their status.
The stage usually vents in about 1 minute. When it arrives at atmospheric pressure, gently pull the SEM chamber door open with its handy handle. The stage table has a stainless steel disk at the center of a Copper table that has a bevel on its rim that fits the reverse bevel on the underside of the sample holder. Carefully slide the sample holder over this disk until it docks against the cross bar on top of the SEM stage table. Note that the sample holder copper docking disk underneath, has a flat side on it – this is the side that meets the docking bar on the stage table. Take great care in sliding this on or off because there are delicate mechanisms right next to the SEM table and on the stage. See the SEM stage in Figure 10.

Return to STEP 5

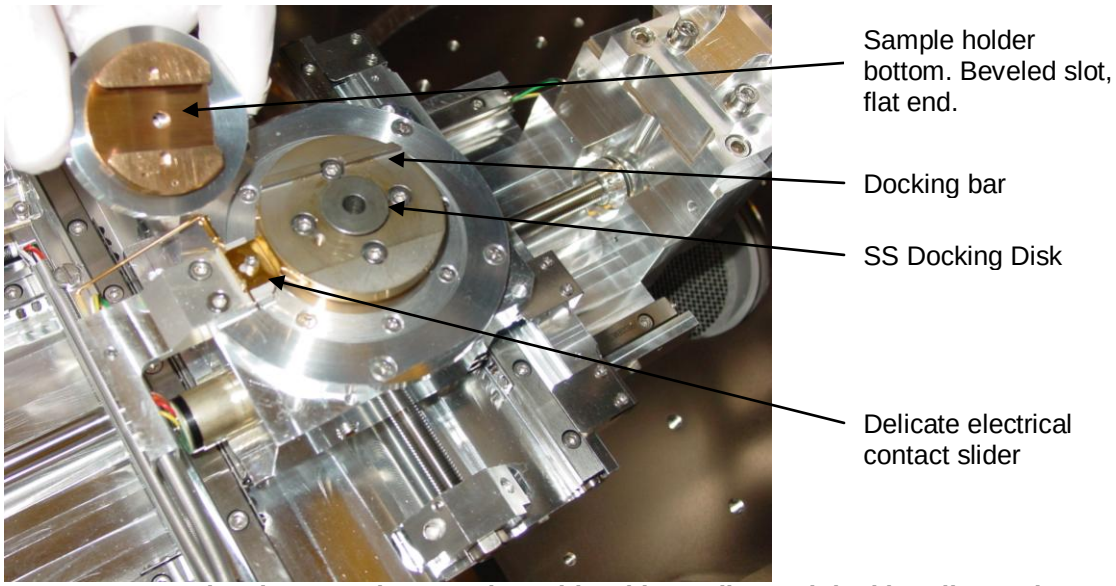

Figure 10. SEM stage showing sample mounting table with small round docking disc and banking bar. Hand holding sample holder shows underside of holder with the reverse beveled slot and flat end that dock against the bar on the SEM stage table. Also note the delicate fixtures around the SEM stage table that can be easily damaged by a wandering thumb.

After the sample is properly mounted onto the SEM stage and inspected, gently close the chamber door. Just as it closes, it will "latch" closed by the force of a

magnet at the back of the stage so that the door will be held closed against the sealing O-ring when the vacuum pump starts, thus preventing an old problem of sucking room air into the chamber.

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Now click OK in the box on the screen asking "Press OK to Pump." The pump down sequence will take several minutes to achieve sufficient vacuum in the chamber for the system to open the column valve and establish a beam. In the meantime, you can click on the "Vacuum" tab in the right hand panel of the screen to view the vacuum level in the chamber and the gun. The gun should be below $8x10^{-10}$ Torr and the chamber vacuum line will be grayed out until the vacuum achieves a measurable level. The chamber will eventually achieve something in the low 10^{-6} Torr range.

Return to STEP 7

Establishing the Electron Beam

As the vacuum level in the chamber drops below $7.5x10^{-5}$ Torr, the column valve will open and the gun EHT (High Voltage) will "Run Up." By clicking on the Gun tab on the right side of the SEM screen, you can see the gun conditions. Normal operating conditions are shown in Table 1 below.

Table 1
Normal Gun Conditions

Parameter	Value
EHT	19.79 kV
Extractor V	4.40 kV
Extractor I	157 micro A
Fil I	2.410 A
Fil I Target	2.410 A
Extractor A Target	4.40 A
EHT Target	219.79 kV

If the electron beam has been on and at high voltage, the system will automatically bring it back on after proper vacuum levels have been reached. If the high voltage does not come back on, you can switch it

back on from the Gun tab window by clicking on the drop down menu under “Beam State=” and click “EHT On.”

Stage Control

Now that a beam is established, the substrate needs to be positioned under the column so the beam can see it. This requires moving the stage from its default loading position to the inspection location which may depend on the sample and its size and shape. Before moving the stage, bring up the “Chamber Scope” window by clicking on the “Eye” icon at the lower left task bar of the right side LCD monitor. This will bring up a window into the SEM chamber viewed from the rear looking toward the front door. The image is an infrared image illuminated by 6 IR LEDs. Note that the image shows the stage in a mirror image presentation, since the video camera is mounted on the back wall of the chamber looking toward you. In any case, this allows you to see the mechanical movements of the stage in real time. This is important to help prevent collisions and improve the speed at which you can find your sample.

By the way, the stage is electrically isolated and if there is a stage collision with anything inside the chamber, the SEM will very softly “Beep” and if the Sample Current Monitor is OFF[2], then the stage motors will turn off at collision. If this happens and you know exactly why, you can manually back the stage away from the collision – if you are uncertain, please call the SEM staff person for immediate assistance.

Joystick Stage Control

The stage movement is controlled manually by a dual joystick desk console shown in Figure 11. In general, the LEFT joystick controls the Z-axis movement and the stage tilt angle, and the RIGHT joystick controls the X, Y motion and the stage rotation – “5-axis stage.”

[2] NOTE: The Sample Current Monitor (SCM) should always be “OFF” unless the user is actively measuring the sample current.

Figure 11. Joystick Stage control. LEFT stick performs stage Z-travel and Tilt: Z-travel – push lever up or down; Tilt – push the lever right or left. RIGHT joystick moves stage in x and y and twisting the right stick causes the stage to rotate.

To raise the stage into the ChamberScope view, push upwards on the LEFT joystick – stage speed is controlled by the amount you push the stick upwards. The stage will travel "up" in Z and the ChamberScope will show its rise. Release the joystick to stop motion. Note that the z-axis velocity is also governed by the image magnification: If the magnification is high, the stage moves very slowly, if it is low, the stage moves fast. When the stage rises to within about a centimeter of the bottom of the conical lens as seen in the ChamberScope, stop. The (x,y) position can then be adjusted with the RIGHT joystick so that your sample is under the electron lens. Note that the stage (x,y) center is about (65,65) mm. If your sample is at the center of the stage, then just move the stage to these coordinates and adjust around the center to find your exact target.

Tilting the stage is effected by tilting the LEFT joystick to the left or right. This also means that while you are raising the stage in the z-axis, and if you also push the joystick right or left, the stage will simultaneously tilt. Watch out for this dual motion (via the ChamberScope) to avoid awkward stage conditions. The stage will only tilt toward the detectors on the left side of the SEM chamber, (which is to the right in the ChamberScope view because the video camera is looking at the stage from the back wall of the SEM chamber). If you reach the tilt limit, the SEM will stop tilting the stage and show an error "TILT LIMIT." By the way, if you run into a stage limit error, just push the lever in the other direction to move away from the limit. Nevertheless, the correct initial direction to push the tilt stick is to your right. You can observe the exact tilt angle in the window executed by the "Stage" tab on the SEM Control panel to the right of the SEM image screen. See **Figure 9**. This Stage Control window is opened under the Stage command menu in the upper left screen command line.

The RIGHT joystick (x,y,theta) works the same as the LEFT joystick: the more tilt you give the joystick, the faster the stage moves. Also, if the magnification is high the x,y motion is slow and vice versa.

Stage rotation is effected by twisting the RIGHT joystick handle one way or the other. Twisting the handle to the right, (clockwise) will cause the stage to rotate about stage center in a clockwise direction. If your target is far away from the stage center, it will appear to translate off the screen because this rotation center is independent of your sample location.

Note that x and y movements can be somewhat confusing because there may be as many as four or five independent coordinate systems involved in the movement. For example: Substrate coordinate system, Stage coordinate system, Beam coordinate system, Joystick coordinate system, Chamber Scope coordinate system, and a software coordinate compensation interpretation system that tries to make user sense out of all the above complications. ☺

Digital Stage Control

If you open the Stage Manager window and place it in the right screen then you will see two sets of coordinate values for each of the stage coordinate axes. The right hand column is a command listing and if you double click in a coordinate box, you can type in a coordinate number, hit enter, and the stage will move to that coordinate value. It will move fast – not coordinated with the magnification – so this method of moving the stage loaded with danger of collision! I recommend that an operator never use this method for moving the stage up to operating height from the loading position – always use the joystick while watching the video camera. The real value in being able to load stage coordinates comes after the stage is in position under the objective lens and the stage needs to be driven in x and y to a known or mapped sample location.

Coordination of Stage and SEM Image Movement

Because of the multiplicity of coordinate systems and apparent conflict of motions, there is an easy way to coordinate the motions of the SEM Image, the Joystick motion and the stage motion in the video camera image. On the SEM Keyboard there is a knob entitled Scan Rotation. If we set this to 90 degrees, then the SEM image, the joystick and the stage as viewed in the video camera image will all move in the “same” direction according to the operator perspective.

To do this we will need the “Hard Front Panel” option active. This pair of windows will show the exact value of each of the knob settings and will change to

which ever knob we turn. To set the 90 degrees simply turn the Scan Rotation knob until 90 deg shows in the window.

Return to STEP 8

SEM Keyboard

Before we discuss finding the sample, we need a short overview of the SEM control keyboard, Figure 12.

Figure 12. Picture of the SEM console keyboard. This console contains a standard (laptop) style keyboard layout in the midst of a number of knobs. The knobs perform repetitive and sensitive operations of SEM control such as focus and magnification among many others.

The SEM keyboard has a number of knobs to give the operator analogue-like control of SEM parameters. The large knob on the left is magnification and the large knob on the right is focus. The two knobs on the upper right are brightness and contrast. And the two knobs on the upper left are stigmators. These are the most used knobs and their functions require frequent, precise analogue adjustments to achieve visible optimization of image quality.

The lesser used knobs will be described later. These give the more experienced operator the ability to operate the SEM much faster.

Finding and Positioning the SEM Image

Sometimes the SEM will start scanning but the screen will be totally dark. How would you find an image from this condition? The normal way would be to reduce the magnification to minimum, and turn up the contrast knob on the SEM keyboard. This will usually bring up a gray scanning field with some fuzzy bright spots in it. If your stage location has been centered (at 65 mm in x and 65 mm in y) and your target sample is at center and about 1 cm below the tapered end of the objective lens, the fuzzy image will show an out-of-focus image of your sample and perhaps some stage parts. Slowly turn the Focus knob on the keyboard to the right (clockwise) to lengthen the working distance – (distance between the lens and the beam focus point in Z). Soon your image will begin to come into focus. Now you can position the stage in (x,y) and working distance (z) appropriate for the sample.

Multi-Sample Holder Map

When you are using the multi-sample holder, this step appears to be a troubling conundrum for new students trying to find the location of each of the samples, (Figure 13). The reason that there is difficulty in this task is that the default coordinate system of the SEM image on the left screen is at 90 degrees from the motion actuated by the joystick and seen in the video camera image of the stage motion in the Chamberscope screen. Also the Chamberscope camera is looking at the sample from the back wall (invoking a left-right reversal) and at a shallow angle so that it is hard to tell where the stage is located in the front-back direction.

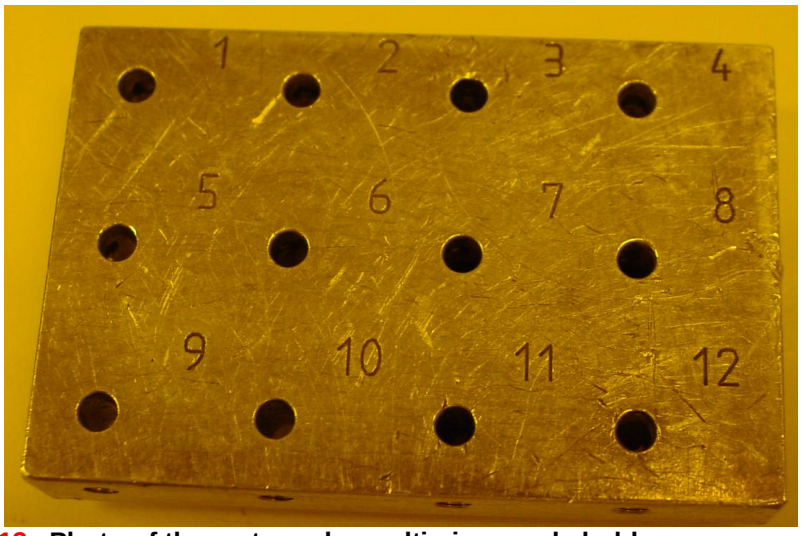
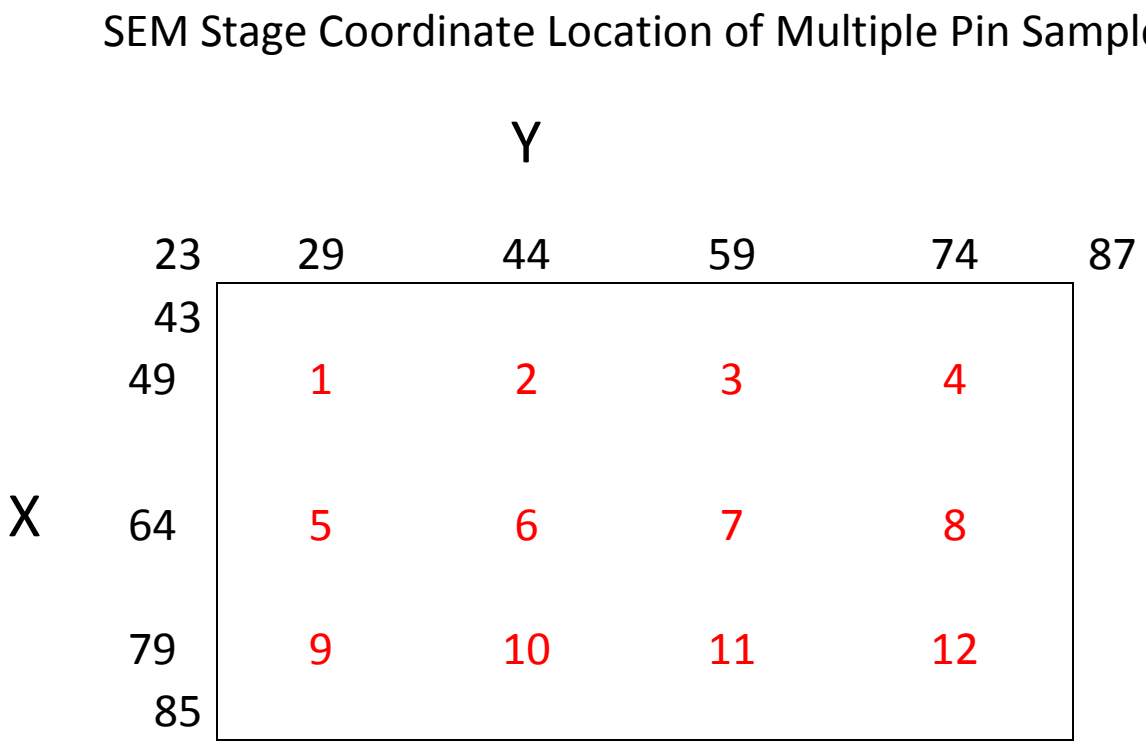


Figure 13. Photo of the rectangular multi pin sample holder.

To compensate for this confusion and perhaps speed up the effort locating a sample, I have created a coordinate location map that sets coordinate

locations for all four corners and each of the 12 pin mounts. This data is only valid if the stage rotation is set to 0 degrees and the sample is mounted on the holding button with the banking bar on the left side of the table as one loads the rectangular multi-pin sample holder onto the SEM stage. The Pin number is the stamped number on the holder as seen in Figure 13 and Figure 14.



*Red numerals identify Sample location marks.

**Read Table for Sample 6: Stage Location = (64,44) millimeters.

***Upper Left corner: Location = (43,23) millimeters.

****Table values are only valid for stage rotation set to 0 degrees.

*****Corner number related to sample positions

Figure 14. Map of pin locations with stage coordinates. Also including corner locations for finding edge profile samples taped to the sidewalls of the holder.

The significance of the Corner Coordinates is that if you are looking at multiple cross section samples taped to the side of the sample holder, you can use the corner values as starting points to find your samples. Then you can move the stage and view samples in a row by moving the SEM image with the Joystick along one axis.

Scan Rotation Coordination with Stage Motion

One more trick can be invoked to make finding things easier. If you set the rotation angle of the electron beam (Scan Rotation) to 90 degrees, then the movement of the SEM image in the left screen will correlate to the apparent motion of the stage in the right hand screen in the Chamber scope window. (Of course the joystick (x,y) coordinate system will translate to (+x=+y') and (+y= -x') where the primed coordinates are the "Stage Manager" coordinates). Setting the Scan Rotation is effected by rotating the Scan Rotation knob on the keyboard and noting the scan angle from the knob readout boxes ("Hard Front Panel") at the top of the SEM image window on the left screen. The 90 degrees scan rotation causes the electron beam scan to rotate from the horizontal direction to the vertical direction as it scans across the sample but is unchanged in the screen image – this consequently causes the SEM image to rotate 90 degrees.

Stage Motion Warning

Note again that moving the stage using the command boxes in the Stage Manager windows is loaded with danger – you need to make sure that the stage will not hit anything along the way to the new coordinates you type into the command windows. Also make sure that the Specimen Current Monitor is "OFF" at all times during stage movement. This gives a small amount of insurance against catastrophic damage if the stage does hit anything in that the collision sensor will turn off the motors upon contact – but not if the current monitor is "ON".

Image Positioning Shortcut

There is another really handy feature of this SEM software that remarkably speeds up the positioning of the sample for a photo. This is needed principally because as you increase the magnification, the portions of the image outside of the center region of the image on the screen expand and move off screen. So after you have obtained the SEM image of your sample by using this handy trick, you can drive a specific spot on the image to the center of the screen where a magnification change will not move it again. This is accomplished by sequentially holding down the "Ctrl" key and then pressing the "Tab" key on the keyboard. This action causes a big green crosshair to appear on the screen. Move the crosshair with the mouse to the location in the image that you want centered in the screen and click the left mouse button. The SEM then either moves the stage or the beam, depending on the magnification, so that the image at

the crosshair moves to the center of the screen. This is a magnificent tool to speed operation.

Return to STEP 9

Focus and Stigmation

To sharpen the nominal low magnification focus used to find the sample, gradually increase the magnification and select successively slower scans to decrease the image noise (snow). If the image feature moves off-screen as you increase the magnification move it back onto the screen with the (x,y) joystick or the neat shortcut outlined in the previous paragraph. As the magnification increases and fine details come into view, select the "Reduced Raster" button at the top of the screen. This will bring up a small, size-changeable active box on the image that displays a small portion of the SEM image. This allows you to switch to slow scan to bring out sharp details but simultaneously increase the frame rate to make it easier to observe the image focus in relation to focus knob changes. Click on scan rate icon #4 to obtain the best image to focus at high magnifications. You can now further increase the magnification to finely focus the image.

Focus technique requires astute observation of the degree of "fuzziness" in the image. However if you turn the focus knob until the image goes distinctly out of focus, note the knob position, and then turn the focus knob the other way until the focus is distinctly out of focus by about the same amount as the previous position, then focus will be about in the center of the two knob positions. Turn the knob back to this center position and proceed to further increase magnification. If the image is again fuzzy, repeat the focus adjustment until the focus is clear.

Stigmation is observed as focus distortion in the image which gives the image a directional fuzziness. This occurs because the electron beam cross section is shaped oblong like an ellipse when it is stigmatic. The axis of the oval spot can be in any orientation and the result is that edges of the image parallel to the long axis of the elliptic spot are sharply focused and the perpendicular image edges are fuzzy and axially stretched. This gives the image a uniform fuzziness orientation.

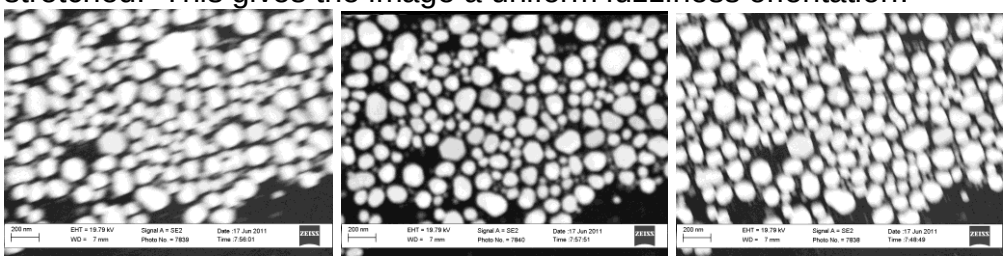


Figure 15. Example of opposite astigmatism orientation on either side of center focus (Left and Right images), and fuzzy center focus due to residual astigmatism at focus. Astigmatism correction is the next step to clear the fuzziness at focus.

To correct astigmatism, rotate one of the stigmator knobs until the fuzziness increases noticeably and then rotate it the other way until the fuzziness again increases. The proper stigmation correction position is in the middle of these two extreme knob positions, (See Figure 15). Repeat this approach with the other stigmator knob, and then refocus with the big focus knob. This should bring the image to a sharp focus and you are done.

So now that we have covered the manual focus procedure, the SmartSEM software in some cases can make this an effortless button-push procedure. On the row of icons at the top of the screen, Figure 16, is a special "Auto Focus + Stigmate" function that is activated by the middle mouse button (Roller wheel). By clicking on this icon with the roller wheel on the mouse, the system will snap into an auto focus mode where the scan is reduced to a small rectangle in the center of the image and a window with a progress bar pops up showing you the focus status. Allow this to complete and observe the results in your image. The little focus and status box will disappear when the function is complete. If the image has sharp details, the auto function was successful. If the image appears way out of focus, it got lost. So the auto focus function depends on the target to have sharply contrasted features to be successful. If the image is low contrast, the auto focus function can twiddle until it is lost and you will have to manually re-focus. Practice is required to improve your skill at this focus task.

Figure 16. This is the panel of action icons at the top of the SEM image page. The third icon from the right is the auto-focus button. The numbered boxes are standard scan rates. Button #1 is used for finding the image and stage movement, Button #2 for stage movement, Button #3 for focusing, Button #4 for image capture, and Button #5 for image capture.

Return to STEP 10

Aperture Align

Any time a different aperture has been selected, the beam needs to be realigned. After you establish focus at a reasonably high magnification (>20KX), click on the "Aperture" tab in the menu set at the right of the SEM image window. Then click on the "Focus Wobble" command and set the "% Wobble" to about 5%. This will automatically change the focus +/- 5% in a repeating fashion. The image will appear to go in and out of focus as well as oscillate in its position. Your task to align the beam to the

aperture is to adjust the x and y “Aperture” knobs on the keyboard until the image does not move (wobble) any more – it should just go in and out of focus in the same location. When you have stabilized the image wobble just click on the “% Wobble” command and it will stop. Then repeat the focus and astigmatism correction.

Brightness and Contrast

The gray level and contrast of the image are set by the two knobs on the upper right corner of the keyboard. The concept in setting these parameters is to have a brightness level that shows dim objects, but a contrast level that avoids white saturation. Contrast also determines the edge definition of objects when looking for sharp, high contrast edges. Electrically, the brightness knob sets the zero offset of the gray level and the contrast knob sets the gray level gain. These knobs are linear in effect, but affect each other – i.e. increasing the contrast makes the image brighter, but so does the brightness knob.

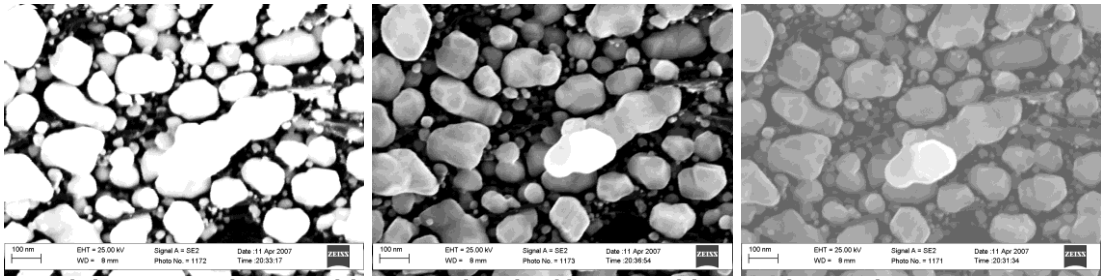


Figure 17. High contrast image with saturation (Left), Normal image, (Center), Low contrast image (Right).

Scan Speed

Scan speed is an important parameter with many ramifications. In general, for a fast scan speed, the image contains more noise (snow), and for a slower speed, the image becomes less noisy and more highly resolved. However, the fast scans reflect real time movement of the stage better than the slow scans. This gives the operator an easier view of stage movement and allows a faster positioning ability. Because the SEM imaging technique is inherently slow, any speed improvement will become highly appreciated by the operator as experience level increases. When the stage is positioned with the desired image in position, the scan rate can be slowed to set the focus and reduce noise for image capture. The software can be trained to automatically impose the fast scan if you move the stage. This command is found in:

“Tools\User Preferences\stage\Fast Scanning\ (ON or OFF).”

There are other digital manipulation techniques to reduce noise in the image. Under the scan tab there is a box that allows the user to choose various noise reduction methods such as pixel averaging, frame averaging and the extent of averaging. In the pixel averaging technique, the SEM captures a settable number of brightness samples at each spot the electron beam moves to in a frame, then averages them all together and displays the spot as the average. This technique reduces the image noise by reducing the spot noise at each spot and results in a real time moving image if the stage is moving. The frame averaging sweeps an entire frame, stores it, and then averages it with the next frame for a selectable number of frames. This creates a dynamic image that produces ghost images for a moving stage that slowly coalesce into the final image when the stage stops moving – not so useful for motion situations.

Return to STEP 11.

Image Capture

Image capture is accomplished by "freezing" the image (at the "End of Frame")[3] and clicking on the Microsoft "File" menu at the far upper left of the screen. This brings up a reduced size window with commands to save the image, Figure 18.

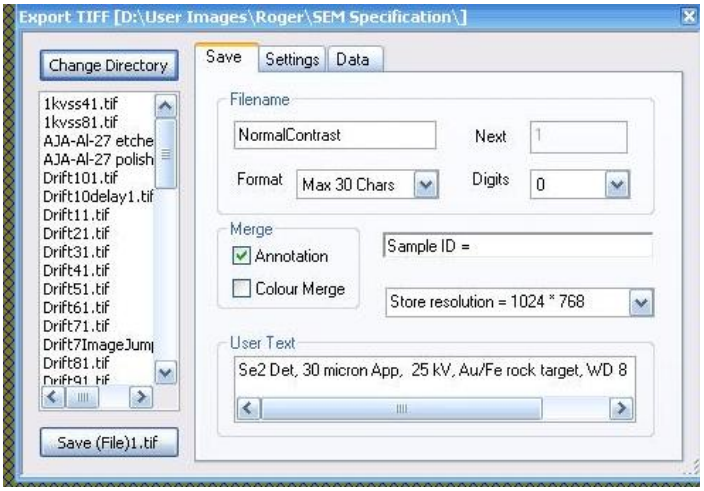


Figure 18. Image capture screen.

To save an image with support data, select your directory by clicking on "Change Directory" and selecting your directory. It will nominally default to your assigned directory on the D: drive, but if you want to store directly to

[3] Set "Freeze on end of Frame" by opening the top row "Scan" Menu and selecting "Freeze on End of Frame."

a memory stick, you will have to select it. Type in the name of the image file you want to store. Click a check mark into the box titled “Annotation” to save any screen text annotation with the image. Fill in any special text explanation you want to save with the file into the “User Text” box. This text will save into the image file so that you can see it in a directory listing but will not imprint on the image.

Special Imaging Techniques

Charging

Many times during your career at the SEM, occasions will arise when you would like to image an insulator material. Unfortunately, SEM physics objects to that… If you were to try to image an insulator, the image would appear to contort into strange conditions of brightness, shape, and aurora-like movement. This would be due to electrons congregating in ever larger numbers on the surface or body of the insulating substrate, thus building up a space charge which severely affects the electron beam. This phenomenon can cause many strange and dynamic effects in the picture too numerous to describe here.

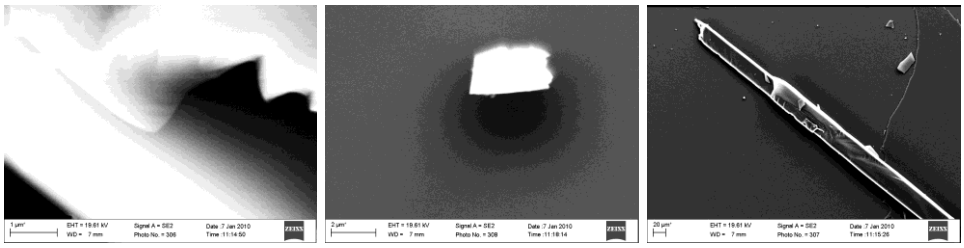


Figure 19. Examples of objects charging in SEM

To avoid charging, we normally sputter coat substrates with a very thin film of Gold and Palladium mix[1] so that the coated surface will be able to discharge excess electrons to ground and thus not cause the imaging beam difficulty.

Charging in a sample is highly complicated and depends on many things, but the balance of electrons hitting the target and those escaping the target is the key to controlling the consequences. Ideally the sample potential should remain at ground level. The potential above the sample is set by the voltage of the substrate and as electrons from the beam enter this field, they are deflected. This can shift the beam and thus move the SEM image.

In some cases you might be able to take advantage of a physical characteristic of electron collision with a tilted surface to help balance the incoming electron charge with the electron charge leaving the surface via scattering. From the data in Figure 20, you can see that as the sample surface is tilted more parallel to the beam, setting a shallow grazing angle, that more electrons scatter out of the surface. If the substrate drain path current can be balanced with the escaping electron charge rate, then the surface could be brought to neutral voltage and the image would become non-charging.

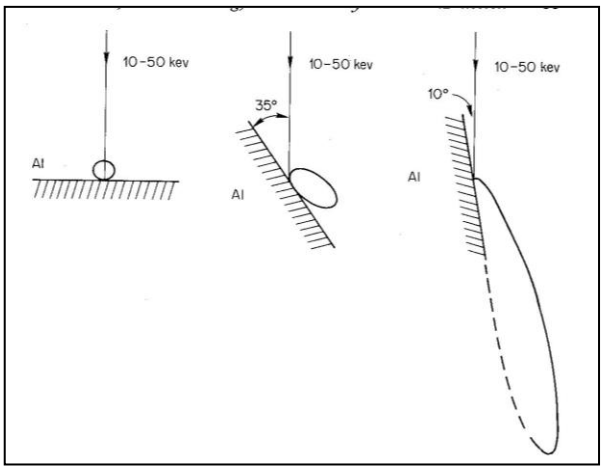


Figure 20. Backscatter electron emission as a function of substrate angle with the primary beam. This shows that as the angle between the electron beam and the surface tilts, more electrons escape. (Data from Kanter, H. (1957)).

Other things can happen on a more local basis as electrons are captured in local regions. These regions will cause the SEM image to "blossom" into bright, random shaped phantom objects in the picture due to clouds of excess electrons bleeding off of the surface and entering the electron detector signal. Many other consequences can also happen, but they all prevent a true image from being captured. Therefore considerable effort is required in some cases to prevent charging.

Electron Penetration Effects

The SEM imaging technique sometimes determines whether you can see the aspects of your substrate that you are interested in. For example, if you need information from the topography of the sample, you should consider the penetration depth of the probe electrons. As electrons enter the sample surface, they penetrate into the body of the material a distance depending on several parameters, chiefly the energy of the electron itself and the material. The deeper they penetrate, the less surface information is contained in the image, and the more material parameter information they convey. However, the higher energy electrons produce a higher geometric image resolution. This occurs because higher energy electron beams are distorted less by the SEM hardware than lower energy beams. Therefore you will need to find an appropriate compromise to meet your requirements.

Backscattered Electron Image

By collecting mostly backscattered electrons from elastic collisions with atoms in the substrate, you can obtain an image with contrast more from the material type than from the topography, (See the topic of Backscatter Electron Detector). Figure 21 shows a graph of backscattering coefficient as a function of Z (Atomic Number). As the atomic number goes up, the material becomes more efficient at producing inelastically backscattered electrons which can be captured by backscattered electron detector. This will produce an image with material contrast where material consisting of heaver atoms show up as brighter regions ions in the image.

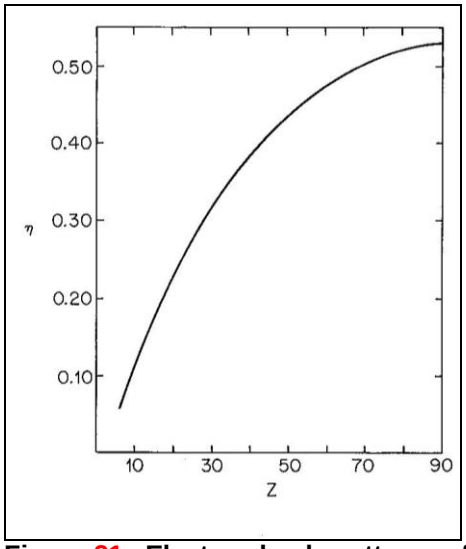


Figure 21. Electron backscatter coefficient vs. Atomic number, Z. This characteristic increase in electron emission with Z creates image contrast in Z, or "mass contrast."

Secondary Electron Image

The standard SEM image is generated by "secondary' electron emission from the substrate. These are low energy electrons (less than 50 eV) produced both by the primary electron beam and the high energy backscattered electrons. However while the primary electron beam can

penetrate the substrate by several or many microns, the escape depth for the secondary electrons is only on the order of tens or hundreds of angstroms. Thus they are generated very close to the surface, (Figure 22).

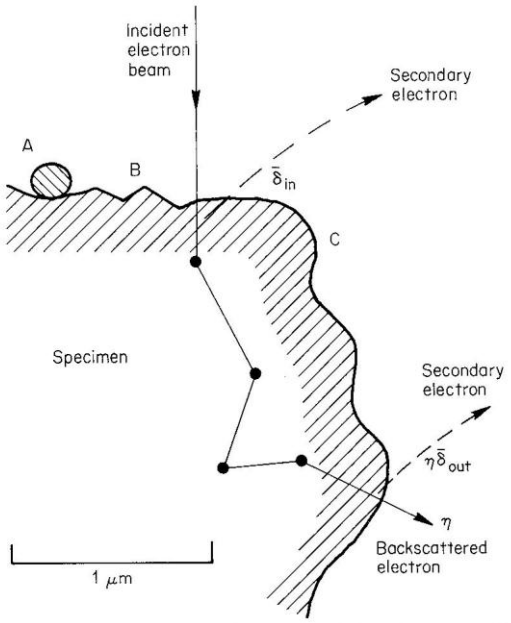


Figure 22. This schematic depicts generation of secondary electrons near the surface from the primary beam and backscattered electrons.

To the first order, the energy distribution of the secondary electrons is independent of the energy of the primary beam. Because of this characteristic, a "universal" curve can be generated for all metals, Figure 23. The curve rises from a zero emission coefficient for zero secondary electron energy, to a peak between 1.3 and 2.5 eV for most metals, and then falls to a low value for secondary energies greater than 10 eV. For insulators, the peak falls at a lower secondary electron energy, because there is a lower potential barrier at the surface of insulators than there is with metals, and this allows a larger number of slower secondary electrons to escape. The SEM video system produces a brighter pixel where the secondary electron current striking the electron detector is greater.

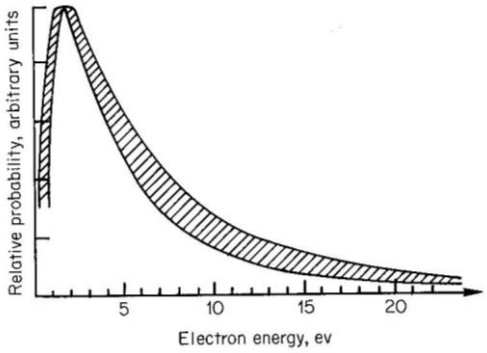


Figure 23. This graph shows the envelope of probabilities of secondary electron escape from the surface of metals as a function of the secondary electron energy. The data shows a probability of escape peak between 1.3 and 2.5 eV. This peak denotes the general energy level of the secondary electrons.

This data sets a value for the energy of the secondary electrons collected by the secondary electron detector in the SEM. Thus the Everhard-Thornley electron detector with its collection grid has to accelerate the secondary electrons to several hundred eV to cause a phosphor photon to be generated and then converted to an electron current that is multiplied many times by the photo multiplier and video pre-amp. It is this current that produces the standard SEM image on the computer screen.

Image Storage

Note that even if you save your images onto your assigned disc space on Drive "D:" on the SEM computer, it is subject to removal without notice by staff. This must be because if the disc becomes full, then the operation of the SEM is compromised and the Clean Room Staff needs to be able to restore operation by removing files. Therefore, it would be prudent to save your images to a memory stick instead of the SEM hard drive. Normally images would only be removed if a problem arose with the SEM operation. But you don't know when that will happen and you wouldn't want to lose critical information.

Return to STEP 12

Sample Removal

Sample removal is accomplished by clicking on the "Specimen Change" icon at the top left of the SEM image screen. This action will automatically turn off the high voltage and close the column valve before venting the chamber. Before you click on this icon, please manually drive the stage down in z to a height of 10 mm, then move the stage in x and y to the center at (65,65) mm. for the x,y move you can type into the "go to" box in the stage manager menu the

65,65 locations for an auto drive move. This will move the stage to a safe position in a safe manner so that there will not be any collisions during the move or the door opening action. When you remove your sample and click on the ensuing “Press OK to Pump” button on the popup window, the SEM will re-establish vacuum and bring up the High Voltage (EHT). If you are finished, follow the EHT Shutdown Procedure in the next section.

Return to STEP 13

Shutdown Procedure

If you are done and ready to leave, remove your sample and pump down the sample chamber. When the pump down starts, you can log off to end your session and sign out of the logbook. The EM Server program will take care of the SEM hardware after you log out. The logoff will stop the use timer and log you off so that the tool is ready for the next user. At logoff, the high voltage for the gun is automatically shut down. This will preserve the life of the field emitter tip that produces the electron beam. It is expensive – many thousands of dollars.

Completion Check List

[Before leaving SEM, set the machine to its standard idle configuration - mandatory.]

Job Completion Checklist

1	EHT off
2	Return software to standard SEM configuration
3	Remove Sample
4	Re-establish vacuum
5	Log out of SEM software
6	Complete Log Book entries
7	Make sure that the SEM table is clean and free of samples
8	Return tools to tool home

Rules of SEM Operation

The official rules pertaining to time, cost, and operation follow:

- Time Allocation: Because this tool is a high use system, we have established a sign-up protocol to ensure that the tool is used fully and to alleviate schedule overlaps and conflicts on the UTD web page,
  - [http://www.utdallas.edu/~hcf011000/cgi-bin/Login.pl]
  - There is an SEM time allotment page that you can log in to and capture time on the machine.
  - Users are permitted to sign up for 10 prime time hours per week (Prime time hours are Monday – Friday 8:00 AM to 6:00 PM).
  - Signup times are set at 1 hour time blocks (min time allowed).
  - Users are charged for the time they reserve the tool for unless they cancel at least 24 hours before the start of their session. You may swap time with another user if necessary.
  - Non prime time hours (Weekends and weekdays from 6:00 PM to 8:00 AM) have no restriction on the length of signup time.
- Fill out the logbook at the start of your session.
- Do not store or leave samples at the machine. Samples can be thrown away by staff without notification.
- Do not use SEM computer to check e-mail or browse the internet or play games.
- Specimen current Monitor (SCM) should be OFF at all times except for the short time it takes to measure specimen current.
- Never unplug the Pico Amp Meter cable from the front door while the electron beam is on – stage could charge up to thousands of volts and zap the pico ammeter when the cable is plugged back in.

You will be given a password after your specific training on the SEM. If you sign up, you are expected to keep your appointment with the SEM. Remember, the current rule is that if you sign up you and you need to cancel, you must cancel 24 hours before your scheduled time. This means that if you sign up the day before, and discover you can't make it, you may pay for the time anyway – unless you make arrangements with another user to take your place and make note of that in the logbook at the reserved time slot. The person taking your time must sign in the paper logbook for the time used.

Standard Configuration

When you complete your operation, return the machine to standard SEM configuration. If you have been trained on one of the several subsystems and you leave the tool set up for that system, the common SEM user may not know how to return to the standard operation.

Remove Sample

When you complete your time on the machine, remove your sample. Also whisk your sample away from the SEM – don't leave it lying around on the table, etc. It will be subject to destruction by the staff cleaning up the table.

Logbook

Fill in the logbook. If you leave blanks, you could be subject to denied use of the SEM.

Purpose/Comments

The Purpose/Comments section in the logbook is intended for recording what you did and note any problems encountered. This is helpful to staff in diagnosing problems and noting what type of work is done in the SEM for logistic support data. Please fill this in!

Consolidated SEM Operation Instructions

This is an abbreviated list of operating instructions that guide you through the "what to do next" confusion that may ensue after initial training for a new user or an encounter with the SEM after a long absence from SEM operation. It is essentially a step-by-step listing of the operating sequence tasks with reference links to the full explanation in the instruction sections in case the abbreviated listing does not trigger full understanding.

In this list of operating steps, there will be a blue "Jump to" word that you can click on and the document will jump back to the full explanation section. When you finish reading the explanation, click the blue "Return to Step n" link and the document will return you to the Consolidated Instruction step that you started from.

STEP 1.

Schedule SEM time on-line. Jump to:Time Scheduling

STEP 2.

Fill out paper Logbook at the SEM. Jump to logbook

STEP 3.

Prepare Sample (Cu Tape, Hummer Au dep., Size sample, etc. …can be done before scheduling SEM), and Mount Sample on Pin Mount Jump to Sample

STEP 4.

Log-in to SEM software Jump to Login

STEP 5.

Vent SEM sample chamber. Jump to Loading

STEP 6.

Open door and mount sample on stage. Jump to Loading

STEP 7.

Close door and pump down chamber. Jump to Loading

STEP 8.

Position stage under objective lens with joystick – watching stage in the Video window.
Use joystick to raise stage to focus point to avoid collision with lens.
Jump to Stage Control

STEP 9.

When system is ready (Scanning operational at EHT), drive stage to target location at low magnification.
Jump to SEM Image

STEP 10.

Focus and correct astigmatism.
Jump to Focus

STEP 11.

Optimize image (brightness, contrast, scan speed).
Jump to Brightness

STEP 12.

Capture image to Disc. Remember the strange rule that Staff has the power to erase any data that they need to without notice to the owner, so transfer your images to a memory stick before leaving.
Jump to Image capture

STEP 13.

Exchange Sample (Vent, open door, change or remove sample, close door, pump down chamber). Never leave sample chamber at atmospheric pressure for very long.

Jump to Sample Removal

STEP 14.

Log-Off SEM software to stop your session cost timer.

STEP 15.

Log out of Logbook.

STEP 16.

Clean up the SEM and the sample prep table. Note that Staff can toss your samples if they are left on the table.

Operating Instructions for EDAX Material Identification System

Introduction

[Note that this is the detailed description section for operating the SEM/EDAX imaging system.]
The "EDAX" system (Figure 24) mounted on our Supra-40 Scanning Electron Microscope is an X-Ray analysis system capable of producing an energy spectrum from X-Rays emanating from a specimen material that is struck by energetic electrons and analyzing the data to determine what elements are producing the X-rays. This principle is termed Energy Dispersive (X-ray) Spectroscopy (EDS). It is a type of X-ray Fluorescence Spectroscopy.

The EDAX X-ray detector is a Lithium Drifted Silicon detector. To avoid Li migration under the influence of bias and temperature, the detector is chilled to liquid nitrogen temperatures. If the liquid nitrogen evaporates and the detector warms up above a threshold set in the software, the detector is turned off to reduce the bias to zero. This helps prevent deterioration of sensitivity because of the thermal and bias drift of the Lithium impurities. Filling the LN_2 Dewar is a responsibility of the Clean Room staff. If you find that the Dewar is empty, ask a staff member to fill it. It will take at least an hour before the sensor is stable enough to capture reliable data.

Figure 24. EDAX LN2 Dewar and pre-amplifier mounted on the Gemini electron microscope column.

Basically, the X-ray emission is stimulated by electrons from the SEM electron beam striking atoms in the sample material and knocking out inner shell electrons and creating electron holes where the ejected electron used to be. Then another electron from an upper shell of the same atom “falls” into this hole and liberates a photon having energy equal to the difference between the falling electron’s home level and the lower level it falls into. The energy of this liberated photon is in the range of X-ray photons; Figure 25.

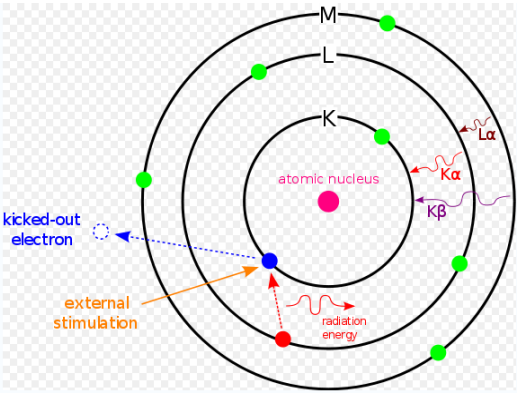


Figure 25. Sketch of atomic energy levels and x-ray photon generation from SEM electron bombardment excitation.

By counting and sorting each X-ray into software bins according to the energy level of each X-ray, a graph can be fashioned that shows X-ray counts vs. energy (Figure 26). This results in a spectrum characteristic of the quantum energy differences in the electron energy structure of the atoms in the sample material. This spectrum can then be analyzed by the software and the elements contained in the material can be identified. With more analysis, the ratio of elements can also be identified.

This document is intended to convey operating instructions to perform elementary material analysis at a basic level. It follows the step by step procedure for obtaining the data and generating a simple analysis. Students must obtain Staff training on this tool before attempting operation. The directions in this section assume that you have been trained and have some experience running the SEM itself.

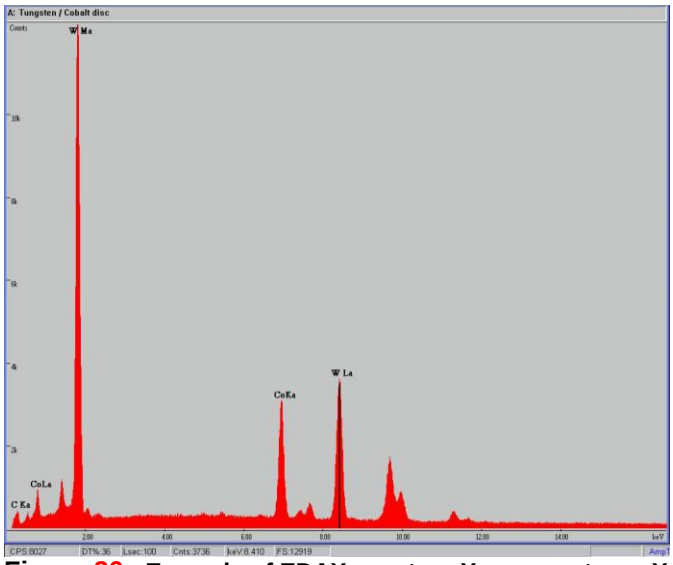


Figure 26. Example of EDAX spectra: X-ray counts vs. X-ray energy. The pattern of energy levels is analyzed to determine what element emitted the X-rays.

Starting Check List

[Before using the system, determine what condition/configuration the SEM/EDAX is in – it should be in a standard state. List critical items that must be checked before operating SEM/EDAX.]

1. Check the LED status indicator on the back of the EDAX preamplifier box just under the big liquid Nitrogen Dewar. If it is red, the sensor is warm and needs LN2 added to the Dewar. If it is green, all is well. If it is not lit then the power is not on – activate the EDAX software and re-check.
2. How do you tell if the LN2 tank is empty? There are several tell-tale observations that suggest that the tank is empty and the sensor is warm:
    1. Lift the lid at the top of the EDAX LN2 tank – if it is freezing cold and steaming, then it is likely there is still LN2 in the tank.
    2. If the EDAX Genesis program sees no x-rays then it is likely that the X-Ray detector is shut off because it is above the operating temperature limit.
    3. If the “Status” LED on the upper end of the EDAX signal amplifier at the base of the LN2 tank is illuminated in Red the amplifier is turned off because the temperature is out of spec.

EDAX Spectral Analysis Setup Procedure

[Detailed operating procedure from the perspective of a new operator.]

Scheduling the tool

This tool falls under the SEM scheduler at the following web site:

http://www.utdallas.edu/cgi-bin/cgiwrap/hcf011000.Login.pl

To get started on this scheduler, you must create an account following the link on the web page. Additionally you must have turned in an application form and have been given permission to create a schedule. Ask staff about this if you need further information. The time you reserve on the scheduler is used to calculate the charges your supporting project is charged for your time on the SEM.

Fill out Logbook

The paper logbook sitting on the SEM table must be filled out completely.

Sample Loading

Sample loading is similar to the SEM loading procedure. The only difference from the SEM procedure is that the Working Distance (WD) for the EDAX should be set to 8 mm in order to optimize the capture of the X-rays emitted from the sample.

Finding Image

Follow the standard SEM imaging procedure to find a standard resolution image and focus on the region of interest for EDAX analysis. See the section on SEM imaging.

Establishing X-ray Capture

Setting Working Distance

To set the working distance, double click on the Working Distance (WD) label in the Data Zone at the bottom of the SEM image screen. A small window containing the current working distance will pop up and allow you to type in 8 for the new WD. This will more than likely throw off the focus you have already established, so manually (joystick) move the sample stage up or down to bring the focus back. When you achieve reasonable focus with the stage movement, you can perform fine adjustment with the focus knob.

Beam Energy

If you know what elements to expect in your sample, look up the EDAX voltage on the wall chart for the K, L, and M shell electrons for the sample atoms and determine the highest energy of all elements in the sample. Then set the electron beam voltage (EHT) at twice the highest electron binding energy – limited of course by the 30 kV max beam voltage.

Turn off Chamber LED Illumination

Before executing the EDAX software, you must turn off the LEDs that illuminate the interior of the stage vacuum chamber. The light from these LEDs causes the X-ray detector to count large numbers of phantom X-rays, and confuses the energy calibration. You can turn off the LEDs by sliding the illumination slider at the top of the Chamber Scope window all the way to the left. The screen will go totally dark. If after that, the window still shows a small spot of light, it indicates that the stage End of Travel Sensor is illegally active. The only way to cure this is to re-initialize the stage, which consequently requires re-establishing the SEM image at your target.

Illumination Error Recovery

Recovering from the Stage End-of-Travel-Sensor illumination error requires re-initializing the stage. To do this, manually drop the stage back to a safe distance from the objective lens (Z~10 mm) and then open the "Stage" menu from the top row of command menus. Find the Initialization command at the top of the list and click on it. This will cause the stage to automatically drive to the zero end of each axis and reset the stage zero location. When it finishes this operation, it will have turned off the end of travel sensor illumination. This fixes the interference with the X-Ray detector and the EDAX system can work now. However, you have to move the stage to the previous location and find the spot on your sample that you want to record a mass spectrum from.

WARNING: Please do not drive the stage in Z by inputting numbers into the digital move boxes – move it manually while watching the video camera so that you won't hit anything.

Aperture Selection

The beam current in the Zeiss Supra 40 SEM is set by choosing different sized apertures. The EDAX signal is more dependent on beam current

than it is on image resolution. A slightly fuzzy image is usually OK. But the optimum EDAX signal is such that the EDAX X-ray counting rate (CPS) is sufficient to allow the computer's Dead Time (DT) to run between 20% and 40% of the capture time. This DT number is found at the bottom of the spectra screen next to the left most number in the data line. The X-ray counting rate (counts per second (CPS)) is the actual left most number in the data line.

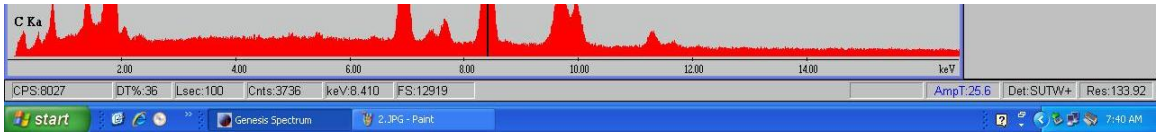


Figure 27. Data bar at the bottom of the spectra screen showing the parameters for EDAX operation.

EDAX Operational Procedure

The EDAX software is executed on the Right side multiplexed monitor and the program start icon is shown in Figure 28. The EDAX computer is selected on the keyboard by rapidly clicking in sequence, the following keys; "Ctrl", "Alt", and "Tab", and then the number 1. When the EDAX screen comes up, click on the "Genesis" icon to start the program.

Figure 28. Genesis icon that starts the EDAX program

Figure 29 shows the full screen spectra capture window with various software control command sub-windows. The following paragraphs will outline the steps required to establish and analyze a spectrum. Note the three tabs at the top of the program opening screen. This is the key to three different aspects of the EDAX program:

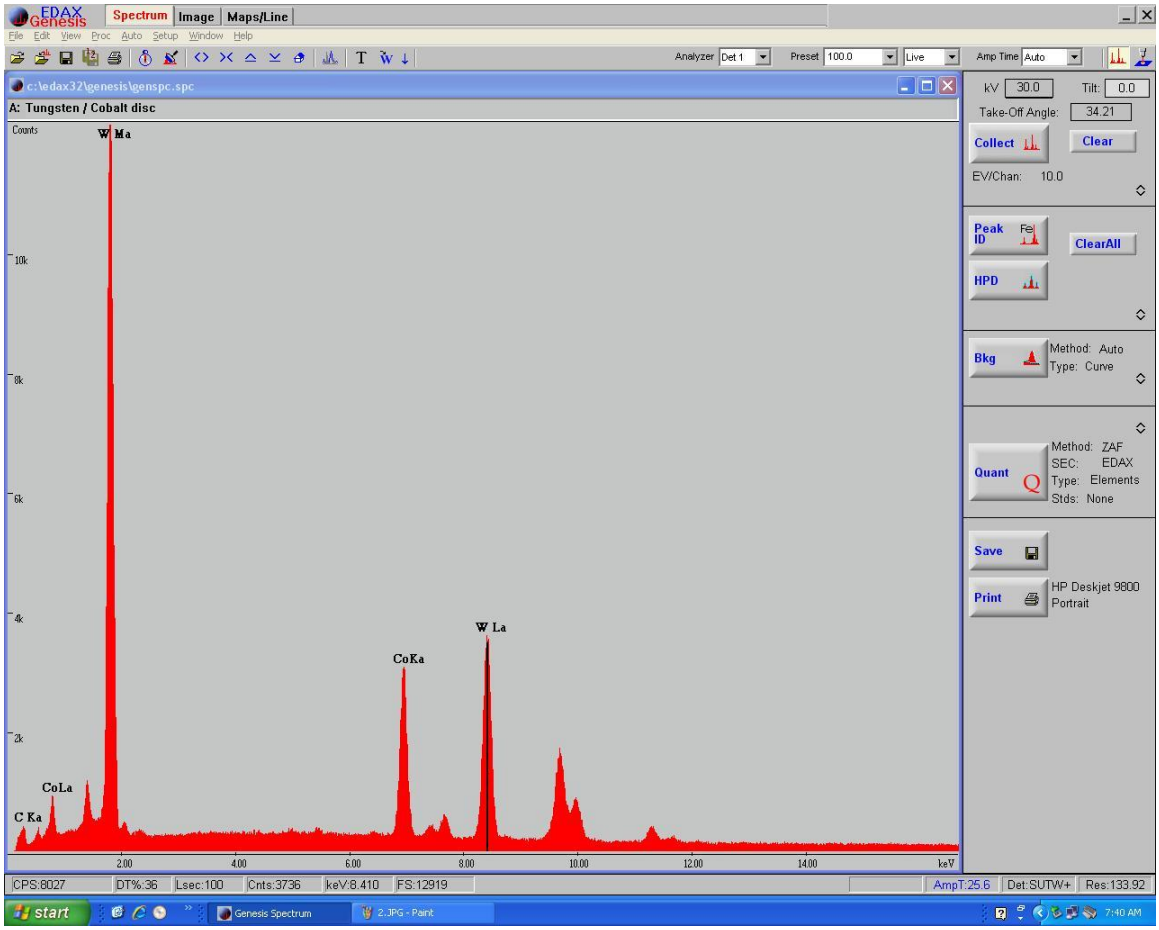


Figure 29. EDAX spectra capture window with some of the spectral peaks identified.

Step 1. Select an Amp Time

Select an "Amp Time" based on the count rate so that the dead time is between 20% and 40%. This is the percentage of the time required by the electronics to count and fill the energy level bins in the software so that a spectrum can be generated. Usually the "Auto" selection will work fine. The window for setting the Amp Time is in the upper right command line as shown in Figure 30. The smaller the number of X-ray counts per second, the larger the Amp Time required.

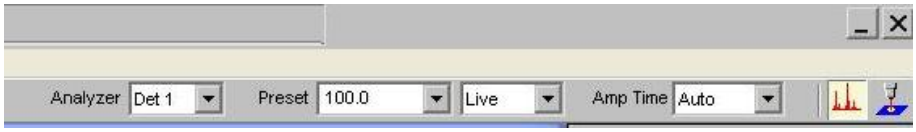


Figure 30. Upper command line showing the Amp Time window

Step 2. Preset a Collection Count

- If desired, set a preset collection count by clicking in the Preset Box (Figure 30 above), and typing a number into the box. This preset count will capture the set number of scans and then automatically stop. The reason for multiple energy scans is to average out any spectral noise that would cause small spikes in the baseline. Usually 100 scans is sufficient to establish a fairly noise free spectrum.

Step 3. Clear the Old Spectra and Peak Labels

- Click the "Clear" in the "Collect box menu area to erase any old spectra that might appear on the screen.
- Click the "Clear All" button in the Peak ID menu area to remove the old peak labels.

Step 4. Adjust the Spectrum Scale

- Set the mouse cursor in the left side of the spectrum graph region, click and hold the left mouse button down and then drag the mouse horizontally to the right to expand the energy scale (at the bottom of the graph) until the max voltage is just beyond the most energetic spectral line expected..
- Alternately, click on the spectrum window and then move the cursor up to the upper left area and click on one of the expand/contract v-shaped arrows to accomplish a similar scale adjustment for the expected upper limit of the spectral data. (Figure 31.)

Figure 31. Location of scale expansion/contraction arrow icons.

Step 5. Start the Spectral Data Collection

- Click on the "Collect" button to start the new data acquisition (off/on toggle switch).

Step 6. Stop the Spectral Data Collection

- If you set the 100 scan limit in the upper right command line, the spectra will stop collecting when it has averaged 100 copies of the spectra data.

- Otherwise, when the spectral data profile has become smooth, you can click on the “Collect” button and the collection will stop, allowing you to start data analysis.

Step 7. Auto Identify the Spectral Peaks

- Click on the “Peak ID” button to start the automatic spectral peak identification analysis.
- Confirm the peak identity by clicking on the “HPD” button located just under the “Peak ID” button. This will draw a thin blue line outlining the spectral peaks according to the theoretical calculation of the proper line shape and location. If this blue line snugly fits the shape of the spectral lines, then the automatic peak identification was successful. If not, then you will have to manually identify the peak.

Step 7a. Manual Identification of the Spectral Peaks

- Manual peak identification can be done several ways using the expanded Peak ID panel, Figure 32. Click on the up and down carat symbol (^) in the right side panel to expand the Peak ID section.

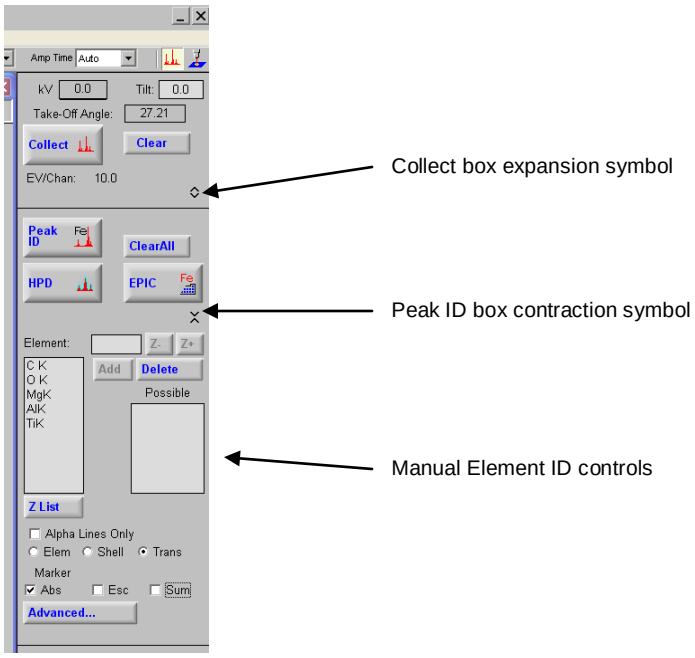


Figure 32. Expanded Peak ID box showing manual peak identify controls and box expansion/contraction symbols (^).

- To manually identify a peak, place the cross cursor over the peak and click the left mouse button. This draws the black vertical marker line to the peak. Quickly after this, the “Possible” box fills with element

symbols representing the most probable element at the black marker location. The top element in the list is the most probable match. Double clicking on this element or clicking on the "Add" button will cause it to jump into the "Element" box and a label will appear on the spectrum. In addition, for your convenience, a set of vertical colored lines representing the most prominent x-ray emission lines will also appear on the spectrum. The lines are color coded and the green color represents the K series. Cyan represents the L series and Yellow represents the M series. The height of these lines represents the theoretical relative peak height of each line. You can erase these lines by right clicking on the spectrum window.

- Confirm the peak identity by clicking on the "HPD" button located just under the "Peak ID" button. This will draw a thin blue line outlining the spectral peaks according to the theoretical calculation of the proper line shape and location. If this snugly fits the shape of the line, then the automatic peak identification was successful. If not, then you may have to guess again.

Step 8. Type in a Label for the Spectra

- In the upper left corner of the spectral data window (Figure 33), highlight the old label and type in a descriptive title for the new data after the "**A:**" character. You are limited to 216 characters in this title.

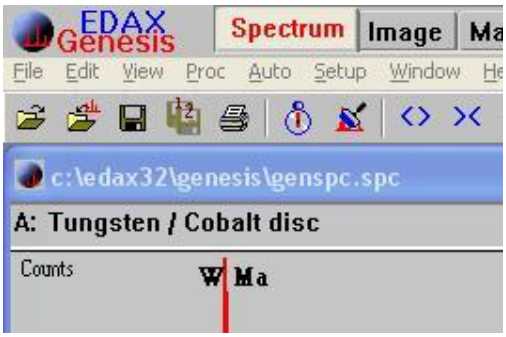


Figure 33. Spectra Title entry after the "A:" character at the upper left corner of the spectra window.

Step 9. Elemental Quantification of a Mixed Material

- Click the "Quantify" button found at the lower right region of the command menu at the right side of the Spectral window. This will produce an overlay window showing a table of normalized material mixture percentages. This is a standardless theoretical calculation based on a set of assumptions and is normalized to 100%.

- Note that this theoretical calculation can be in considerable error because of situational failures of the material to fit the standard parameter value assumptions used by the calculation.

- Material Quantification Assumptions:
  - Sample working Distance = 8 mm
  - Stage tilt = 0 deg
  - Material must be mixed homogeneously to the depth of electron penetration
    - Layered material leads to potential erroneous results up to double digit percentages
    - Homogeneous material analysis can be accurate to <5%

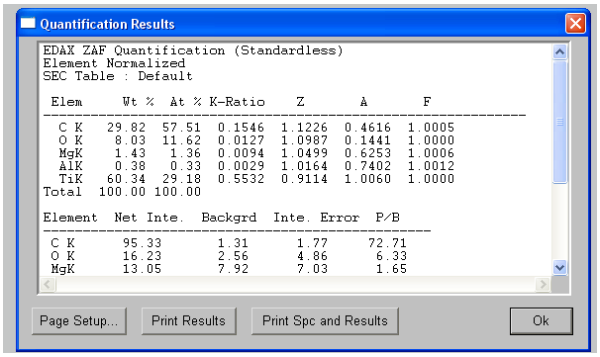


Figure 34. Quantification results window showing the percentages of elements in the EDAX sample.

- Details of high accuracy mixture analysis are beyond the scope of this manual.

Step 10. Printing the Spectrum

- The spectrum and the overlay window showing the quantification results can be printed on a printer sitting on top of the cabinet housing the SEM computers. Click on the appropriate print button located in the overlay window showing the quantification table. This can also be printed to a file (especially to a memory stick).

Step 11. Save the Spectra into a File

- The spectra can be saved by clicking on the “Save” button located at the bottom of the right side panel. You can input a file name and location for the storage.

EDAX X-Ray Mapping Operating Procedure

Overview

X-ray mapping is a technique of acquiring spectral data for each pixel in an SEM image and then coloring a map to indicate what material is located where.

The X-Ray mapping procedure is located under the third tab at the top of the EDAX Genesis window. On the right side of the mapping screen is a command panel containing control buttons located from top to bottom in the order in which they should be used. Figure 35 displays this panel with operational notation.

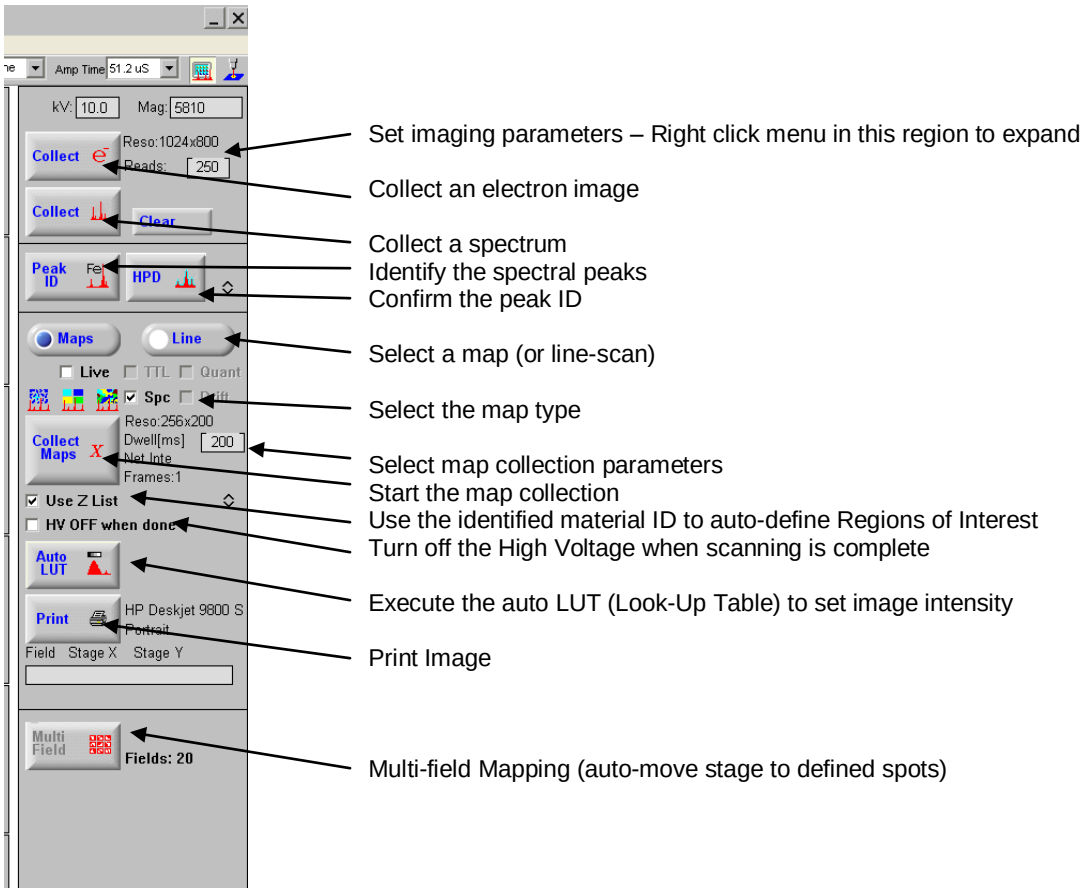


Figure 35. Mapping command panel at right side of mapping screen.

Operational Steps for Mapping

1. *Set Preliminary Parameters*

- Make sure that the “Chamber Scope” illumination on the SEM is turned off to prevent problems with the X-ray detector.
- Enter the number of Spectral scans you want in the “Preset” box at the upper right of the EDAX command line - you can have “None” in the box for this operation to produce unlimited scans.
- Set the “Amp Time” to “Auto” to insure that the dead time is between 20% and 40%.
- Make sure the RemCon32_EDAX program is talking to the EDAX program by opening the program and verifying that text is being produced. If it is not, then make sure that port number 2 is set and open. You will have to restart the EDAX program to reconnect.

2. *Set Spectrum Parameters*

- Clear the prior spectrum with the clear button at the right side of the “Collect” button in the right panel.
- Clear the spectral line labels by expanding the Peak ID window and “Del All” from the element ID box.
- Scale the spectrum graph axes. (See Step 5 in the previous section.)

3. *Set Image Parameters*

- The resolution parameter is accessed by right clicking on the numbers to the right side of the “Collect” button. The resolution number is like a digital camera resolution number – higher numbers give clearer pictures, however they take longer to acquire. The “Reads” number sets the number of frames to average together to reduce image noise. The “Resolution” and “Reads” numbers are a little tricky to guess because the combination of these numbers sets the time to acquire the image and could run into hours if they are too big. Suggested nominal starting numbers are 256x200 for the Resolution and between 16 and 32 for the Reads.

4. *Collect Electron Image*

- Press the "Collect e^- " button to start collecting the SEM image over the area of interest for the spectral data collection.
- If the Preset box has a number in it then the image collection will auto stop when the preset number of scans has been reached.
- Otherwise, you will have to click on the "Collect e^-" button again to stop the scanning.
- NOTE: If the spectral lines appear to be high on very light elements, and scrambled elsewhere, the Nabity software may have to be opened and set to "SEM" mode to give control back to the SEM and its EDAX software.
    - To do this, you have to close the RemCon32 program and open the Nabity RemCon32 program [open the SEM "Start" icon to find menu choice].
    - Switch to the Nabity computer by clicking in sequence the buttons: 1)Ctrl, 2) Alt, 3) Tab and then choose the number 2
    - Then open the Nabity software by clicking on the green Nabity icon in the right hand Multiplexed computer screen.
    - Open a "Project" and find the SEM button on the left side of the Nabity window and click on it. This should return control of the SEM column to the SEM which allows the EDAX software to borrow control.
    - Switch the multiplexed computer back to the EDAX computer using the "Ctrl, Alt, Tab" sequence and the number 1.
    - Before you open the EDAX software, close the Nabity RemCon and open the EDAX RemCon software again.

5. *Collect X-ray Spectrum*

- Press the "Collect Spectrum" button to obtain the X-ray spectral data.
- Again, if "none" is entered in the preset box, then press the "Collect" button to stop collecting the spectral data, otherwise, just let the scanning continue until the preset number is reached.

6. *Identify the Spectral Peaks*

- Press the Peak ID button to have the software automatically identify the spectral peaks. If peaks are left without an identity, you may have to manually identify them according to step **8a** in the previous section.

- Confirm the validity of the identification by clicking on the “HPD” (Halographic Peak Deconvolution) button. This will add a blue outline around all the identified spectral peaks. This blue line is the theoretical line location and shape for the element chosen. If it fits snuggly around the spectral line, the identity is probably correct; if it is shifted or does not fit the shape then more effort is needed to identify the material.

7. Select Maps

- Click on the “Maps” button.
- The other option is “Line” which is utilized later after a map is already obtained.

8. Select Map Type

- Standard Map Type – With no boxes checked under the Maps button, the system will capture X-ray data at every pixel for the selected dwell time. The data for that pixel is recorded and immediately updated into the map.
- Live Map Type (Fast mapping) – This map type rasters very quickly over the entire map area set by the SEM magnification and presents the data for the complete map (1 frame). This is repeated and the maps are updated pixel by pixel until the map is stopped by the user or when the set number of frames is reached.
- Spc Type – Live Spectrum Mapping (“Live” and “Spc” boxes checked) rasters frame after frame over the entire map area storing the entire spectral information, while updating the images and the cumulated spectrum one frame at a time. This is the best way to collect data at high speed. After capturing the data, you can access a complete spectrum at each image pixel. You can also build compositional maps of any type, including Quant Maps.
- Quant Mapping – This type collects data in the same way as standard mapping. However at every pixel a complete background subtraction and peak deconvolution is performed. The data could be composed of several types, including Net intensities, k-ratio, and ZAF. In a Quant map with ZAF weight percent as the data type, only one atomic energy level from each element can be used in the map. For example, if Iron K and L lines are in the peak ID list, only iron K will be mapped.

9. *Set Mapping Parameters*

- Roll the cursor to the right of the “Collect Maps” button and click the right mouse button. A new little window will appear with several options as in Figure 36.

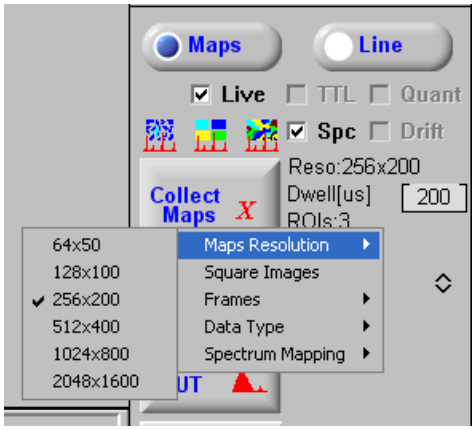


Figure 30.

Figure 36. Expansion of the parameter listing at the “Collect Maps” button.

- The map resolution should be set based on the magnification, size of the sample features, and a nominal time of collection.
  - For fine featured samples a low resolution (64x50) may not be appropriate because the pixel may be larger than the feature.
  - At high magnifications, a higher resolution can result in the pixel size being smaller than the diameter of the beam.
  - The higher the resolution numbers, the longer the collection time.
- The dwell time is the amount of time that the beam will collect data at each pixel. The dwell number for both standard and quant maps is in milliseconds. For live maps, the dwell is in microseconds.
  - The default dwell time for live maps is twice the processing time. The time can be lengthened but not shortened.
  - For Quant maps, a minimum dwell of 50 milliseconds is required. Since the background subtraction and peak de-convolution calculations are performed at each pixel, the dwell must be long enough to allow the calculation.
  - For Live Spectrum maps, the minimum dwell time is 50 microseconds. A minimum dwell of 200 microseconds is

recommended for the 128x100 resolution, but 100 microseconds for 256x200 or higher resolution.
    - The ROI parameter (Region Of Interest) refers to the number of spectral line elements chosen for mapping. This will be the number of maps created.
    - The “Frames” option sets the number of times the field is scanned and data added and averaged to reduce noise.
- Note that a time estimate is shown in the bottom status bar, Figure 37, and will update as the parameters are adjusted. For live and live-spectrum maps, no time estimate is given because the maps can be stopped at any time by the operator. This can be used to help arrive at a practical set of parameter values.

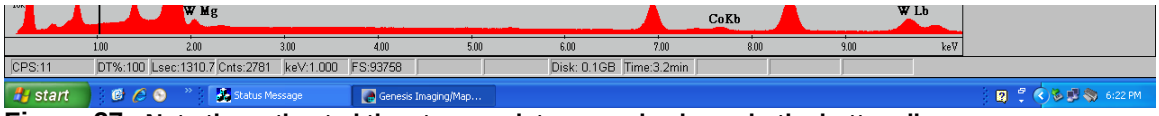


Figure 37. Note the estimated time to complete a map is shown in the bottom line.

10. Collecting a Map

- Click on the “Collect Maps” button to start the map data collection. This will bring up a small window asking you what you want to call the map and where you want to put it. Conjure up a name and stash it in a place you can find later.
- Note that the time estimate in the bottom line will update in real time to help you know when it will be done.
- If you need to stop the map before the end of time, just click on the “Collect Maps” button again. The program will have saved the previous data and will produce a map even though you stopped it.

Map Analysis

Line Scan Analysis

- The Line Scan analysis allows the user to draw a line across an SEM image of a recorded spectrum map (file type “mapname.spd”) and graph the spectral line intensity for each of the ROIs as a function of location on the line. This effectively displays the location of each element along the defined line.
- Line Scan procedure

- Select a saved spectrum map data file (type “name.spd”) in the File Open menu in the map tab screen.
- Expand the Peak ID box by clicking on the up/down carat symbol at the right of the Peak ID button.
- Delete the unwanted elements from the Element box by highlighting them and clicking the adjacent Delete button. (Figure 38) This action selects the elements you want to display in the line scan element intensity graph by throwing out the ones you don’t want.

Figure 38. Expanded Peak ID window showing the Element box and the Delete button.

- Close the expanded Peak ID window and click on the “Line” button. This will exchange the icons below the button to the relevant line scan icons.

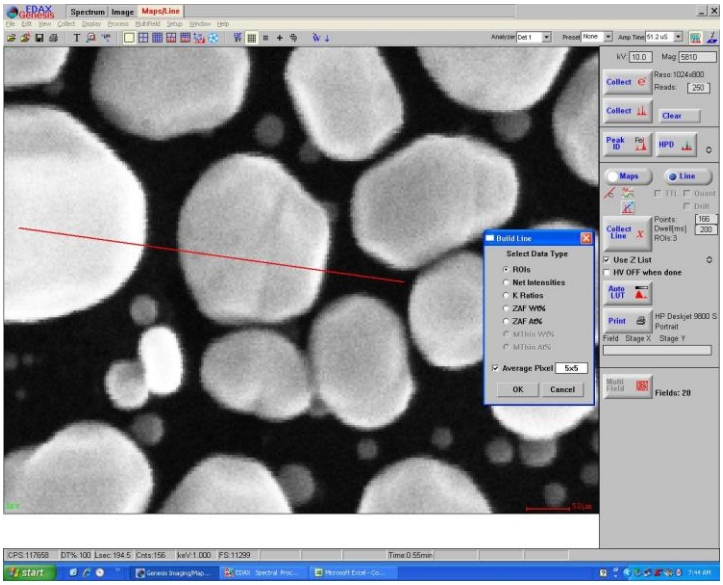


Figure 39. Screen display after the Line button is pushed and the Draw Line icon is clicked on – and the Build Line icon is also clicked on to bring up the little Build Line window box.

- Click on the Draw Line icon to expand the SEM image to full screen size. Draw a line by left clicking and dragging the line across features you want to analyze.
- After the line is created, click on the Build Line icon. This will bring up a little window with choices to make. Select the ROI to display element concentration graphs. Click on the box by the Average pixel label and enter the averaging matrix size, i.e. 5x5. This will help reduce noise in the graph line by using a running average technique. Click Ok and create a name and location to store the resulting "name.csv" file that you can access with MicroSoft EXCEL later.
- The actual line scan graph will appear in a new window with the number of graph lines determined by the remaining elements in the Element box that you set up previously. See the three-band graph in the right window in Figure 40.

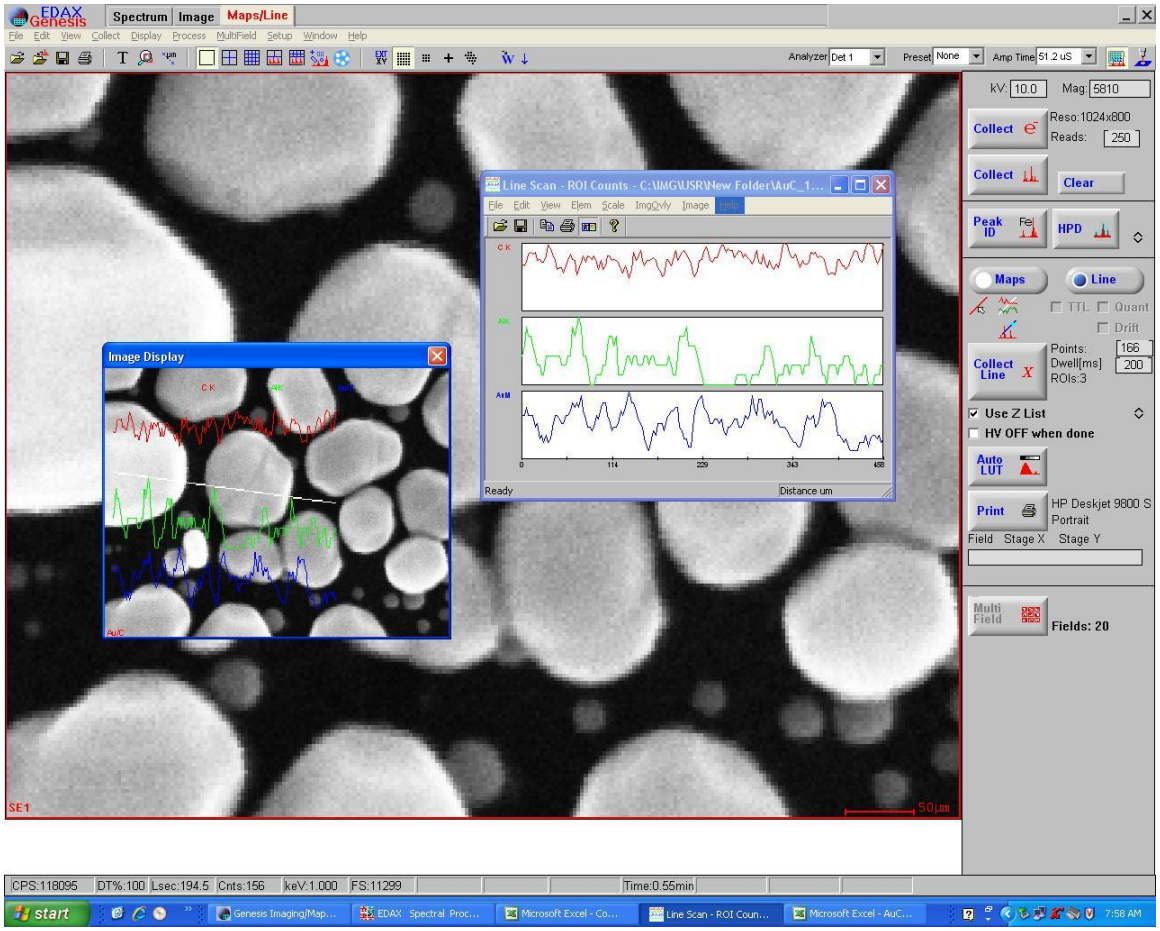


Figure 40. Line Scan graph windows showing the pure graph window (Right) and the overlay graph (Left).

- You can create an overlay graph which puts the graph lines on the SEM image so that you can visually see where the x-ray intensities for the various elements lie on the image. This gives you the visual location of each element you look at. Unfortunately this image appears in a new window that is fixed in size and cannot be saved. Enjoy it while you can, or capture it with the Print Screen button on the keyboard and save it in the Paint program.
- When you are done with the analysis you can return to active data acquisition or just close the EDAX window and go home. Note that this analysis does not require actual SEM operation.

Map Overlays

- A map overlay consists of a mosaic map of up to 6 individual element maps superimposed in a single map. This indicates the x-ray intensity and location of all the identified elements in a single map.
    - To create this overlay, first choose the field of 36 little windows (find this selection in the main row of icons at the top of the window and select the icon with lots of windows in it)
    - Highlight each of the maps to be included by holding down the shift key and clicking on each individual map you want included in the overlay.
    - Then pull down the “Process” menu from the top menu bar and select “Color” and then “Substitution Overlay” from the drop down menu. There must be one open window in which the overlay can be viewed.

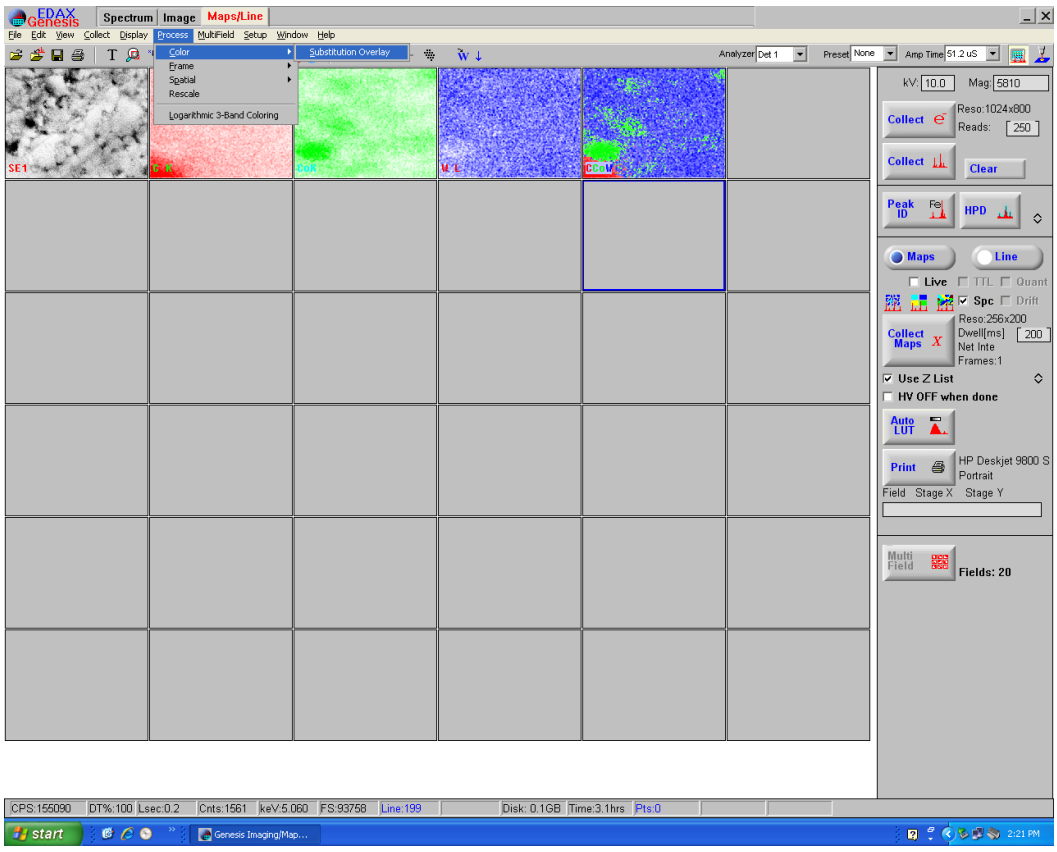


Figure 41. This screen capture shows the Map Overlay (last blue/green/red box at the right end) with the Process menu open showing the Color and Substitution Overlay choices. Note the blue outline below the overlay map. This indicates that that window has been selected – this blue box should appear around each of the normal element images (“shift – click” to select) before choosing the overlay action.

- NOTE: The overlay is created by the software selecting the element with the brightest normalized pixel intensity to represent that pixel in the overlay.

Printing maps

- The collected maps will appear as a collection of individual element maps in small windows with different color dots corresponding to the specific element. This little matrix of elements maps can be printed directly on the HP printer. Highlight each map you want printed and then click on the print button in the upper left command menu. The trick is to hold down the shift key and click on each map you want to get them selected.
  - If the image is too dark and all you get is black ink on the paper, you can reverse the background color to white. This usually gives a better map because the paper is not completely saturated with black ink.
    - Reverse the background color by opening the command line tab "Color" and choose "Reverse".
  - Print the maps by clicking on the "Print" button at the bottom of the right panel.

Completion Check List

- When you are finished with the EDAX operation simply close the EDAX program by clicking on the X in the upper right corner of the window.
- If you need to close out the SEM operation also, then follow the SEM operation completion procedure found under "Completion Check List."

Rules of EDAX Operation

- Only Staff is allowed to fill the LN2 Dewar.
- If you need to use the EDAX system for analysis (not data collection requiring SEM operation), then you can work out an arrangement with the person using the SEM to work in tandem with him/her while not disturbing the SEM operation. Note: E-Beam Lithography uses the EDAX computer screen, so tandem operation with lithographers does not work.
- If you learn an advanced process which uses different program parameters or windows configuration, please return the system to normal conditions before you leave.

Additional EDAX Software Details

[This section can describe the software knobs and buttons in a collected section…]

Special Purpose Subsystems

[Explain that these systems are beyond the scope of this manual and are special purpose systems and users must undergo extensive specialty training by experts in the subsystem – after they become familiar with the standard SEM operation.]

Electron Backscatter Detector Operation

[Briefly describe how the system is used. This is a "survey' level description with generalities rather than detailed knob operation.]

Electron Back Scatter Diffraction System

[Briefly describe how the system is used. This is a "survey' level description with generalities rather than detailed knob operation.]

Sample Preparation Overview

Proper sample surface preparation is paramount in obtaining good Electron Back Scatter Diffraction images that can be accurately analyzed. The EBSD signal is obtained by electron beam interaction with the crystal lattice within 10 to 50 nanometers of the surface. This means that if the surface is composed of contamination to this depth, then the EBSD signal will be of poor quality or even non-existent. Therefore surface preparation must result in surface exposure of the undisturbed crystal lattice. This section will survey several different preparation techniques.

Mechanical Polishing

Mechanical polishing is usually required to remove surface contamination, oxides, and distorted crystal material. However, the methods used to accomplish this are many and vary according to the sample material. There are a number of steps to creating a suitably prepared sample for SEM examination.

1. Sectioning

   This term describes the simple step of making an SEM sample from a parent substrate – i.e. cutting out a small piece from a wafer, etc.

2. Mounting

   This is a preparation step for treating the surface to make it suitable for EBSD imaging. Basically, how you hold the sample to treat the surface (grinding, polishing, etc.) to create the exposed crystal lattice at the surface. This could consist of embedding the sample in resin, or fixing it to a mount that will enable surface treatment.

3. Grinding

   Grinding removes the macroscopic surface contamination, oxides, etc. and exposes the base material in sequential steps that produce a smooth, flat surface ready for further treatment. Typically, grinding starts with something like 240 grit Silicon Carbide (SiC) sandpaper, and goes through succeeding steps of ever finer grit to 800 or 1200 grit sandpaper. Microscopically, the surface will look like fine scratches and will need further work to expose the underlying crystal lattice.

4. Polishing

Polishing is used to remove the scratch deformation caused by the grinding step. It usually consists of using a colloidal polishing grit on a flat table and swirling the sample around on the table in a rotating figure 8 pattern with coarse grit and then stepping to a finer grit until the desired polish quality is achieved. There are many types of grit available, but the literature suggests that a commercially available solution of colloidal silica works best for the final stage of EBSD samples. This stuff consists of negatively charged particles of SiO_2 with a pH value between 8 and 11 (chemically basic). The resulting surface will usually look rather shiny at this point.

The following images show a link between the degree of surface preparation and the resulting EBSD image quality of a standard Nickel substrate.

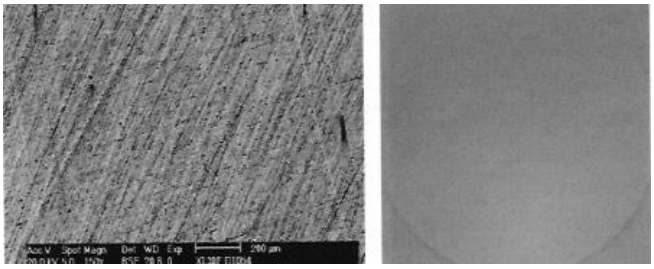


Figure 42. Surface appearance (left) and EBSD pattern (right) after 1200 grit SiC paper (No EBSD pattern visible)

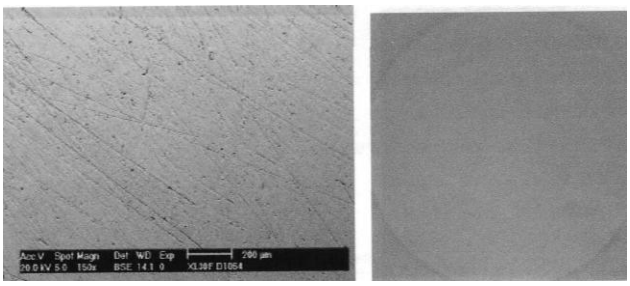


Figure 43. Surface appearance (left) and EBSD pattern (right) after 3 micron diamond paste polish. (no EBSD pattern)

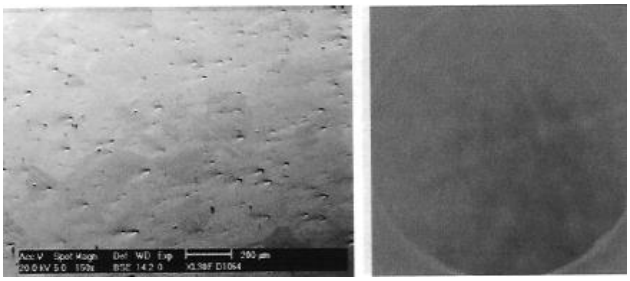


Figure 44. Surface appearance (left) and EBSD pattern (right) after 1 micron alpha alumina polish. (EBSD pattern quality = 28)

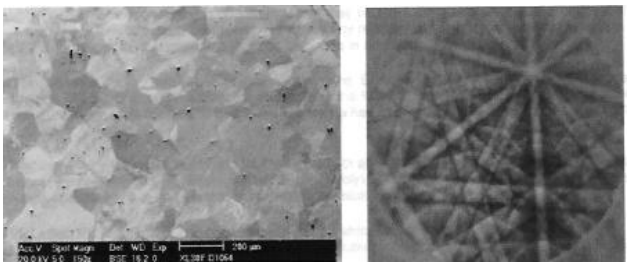


Figure 45. Surface appearance (left) and EBSD pattern (right) after 0.3 micron alpha alumina polish. (EBSD pattern quality = 166)

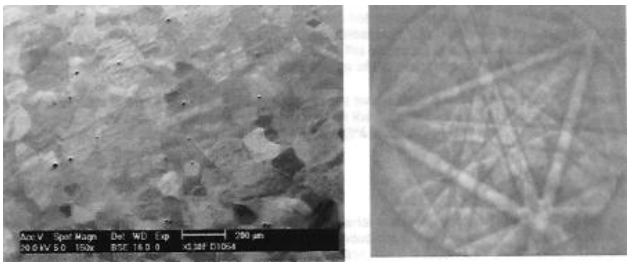


Figure 46. Surface appearance (left) and EBSD pattern (right) after 10 min polish with colloidal silica (EBSD pattern quality = 177)

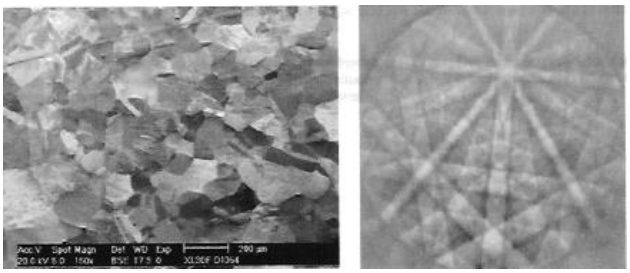


Figure 47. Surface appearance (left) and EBSD pattern (right) after 30 min polish with colloidal silica (EBSD pattern quality = 224)

Even though the above sequence of polishing appears to have produced a good EBSD image quality, there may be some circumstances that require even more preparation before good EBSD images can be obtained. This extra polish step can involve either electro polish or chemical polish. Electro polish involves

immersing the sample in an electrolyte solution and using an electric bias to remove the remnant deformation layers and surface irregularities left after polishing. This method grows an anodic film on the surface and then slowly removes it to reveal the bulk crystal lattice at the surface. Variables that affect this process include the specimen material, the electrolyte solution composition, operating voltage, specimen size, temperature, age of the electrolyte, the circulation rate of the fluid, and the time of contact.

5. Polishing

Chemical etching is another alternative to electro polishing that is less complex and often results in equal EBSD results. This method involves dipping the sample in an appropriate etching solution that merely removes deformed crystal layers because of their higher surface energy compared to the regular crystal lattice. The etchant must not cause surface steps that might interfere with the EBSD signal. In addition the etchant must be selected according to the type of sample involved – this is a very particular choice and determines the degree of success achieved. If the etching solution leaves a surface film behind, then the results will be poor.

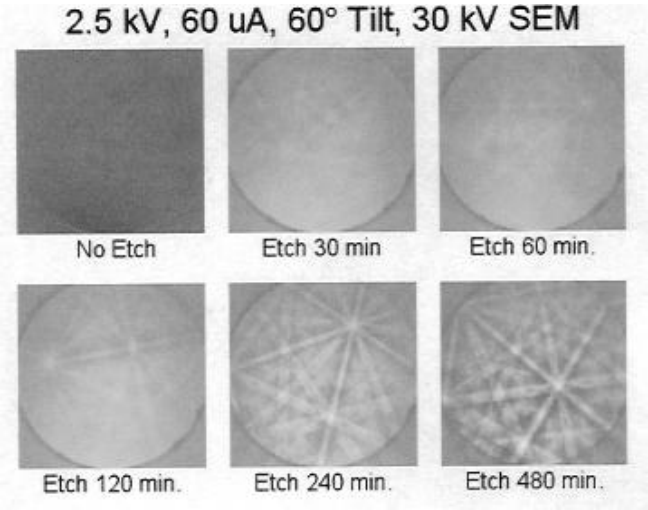


Figure 48. Examples of chemical etch time on EBSD image quality.

SEM Operating Procedure for Obtaining EBSD Images

Scanning Transmission Electron Microscope Detector System

Introduction

The Scanning Transmission Electron Microscope (STEM) detector on our scanning electron microscope consists of a set of diode electron detectors at the end of a long rod that can be positioned under a thinned sample mounted on a special multi-sample holder. Electrons from the standard scanning electron beam column collide with material in the sample but pass through the thinned sample and spread out in a scanning conical pattern. This scanning conical beam is intercepted by 5 planar electron detectors (diodes): 4 independent detectors on the top level and a single detector on a lower level that receives electrons through a central hole in the upper detectors at the intersection of their 4 internal corners. This arrangement allows 6 different modes of observation: 1) Dark Field (DF), 2) Bright Field (BF), 3) DF+DF, 4) BF-DF, 5) Summed Oriented Dark Field (ODF1+ODF2), and 6) ODF1-ODF2.

This arrangement avoids the chromatic aberration (from non homogeneous electron energies) that pure TEM microscopes produce from the refocusing of transmitted electrons. In addition, because of the lower beam voltage compared to the TEM, the STEM system produces higher transmission rates and better signal to noise ratios which in turn create better contrast images with comparable resolution. Furthermore, because of the thin sample and small diameter of the focused beam, the electron collision volume in the sample is very small, which enhances the lateral resolution of EDAX material identification.

This STEM can be used to analyze material morphology in diverse materials like polymers, ceramics, nano-particles, crystalline grain boundaries, and cross sections of semiconductor devices.

Nano Manipulator Stage Operation

Low Noise Nano Prober System

Nabity Electron Beam Lithography System (NPGS)

Scanning Electron Microscope Operation

Zeiss Supra-40

Roger Robbins 16/22/2011

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