

Instructions for Use of **ZEISS 1550VP Field Emission SEM** with **Oxford EDS and HKL EBSD**

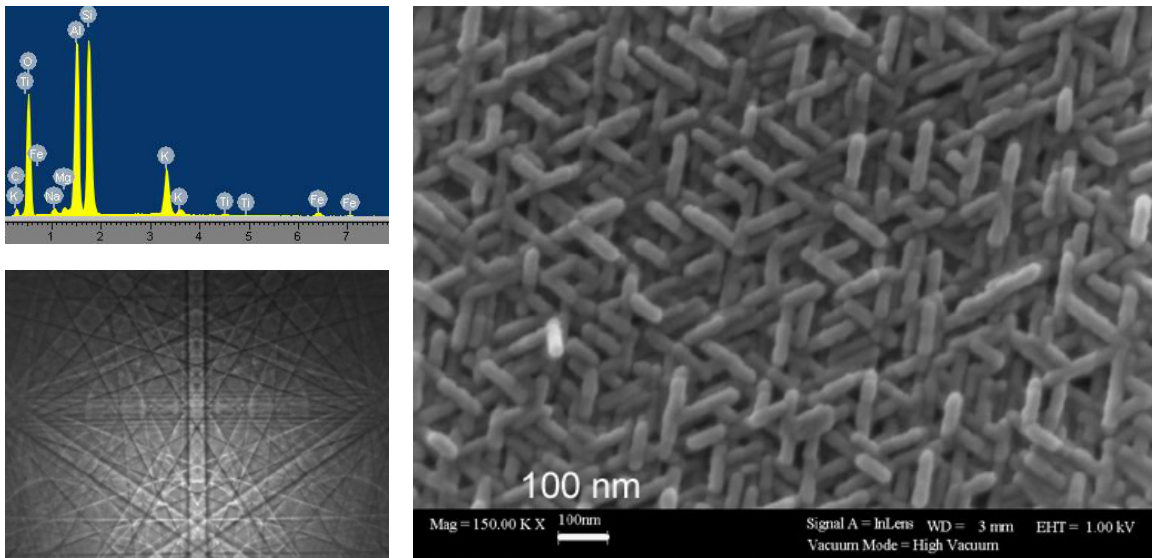
Version 3.0 September 2009

Chi Ma, Analytical Facility, Division of Geological and Planetary Sciences

California Institute of Technology, Pasadena, CA 91125

chi@gps.caltech.edu

<http://www.gps.caltech.edu/facilities/analytical/>



This manual is for users who have already had an introduction to the ZEISS SEM, EDS and EBSD. It is not intended to take the place of training on the instrument. When in doubt, get help.

Sample Preparation

1. Put on a pair of gloves. Samples should be clean, as small as possible, stable in vacuum. If needed, clean samples, stubs and the specimen holder with isopropanol and kimwipe papers.
2. Mount fragment or powder samples firmly on Al-stubs using C-tape. Fix stubs onto the ZEISS specimen holder. 8 stubs may be loaded. Mount thin sections directly on the specimen holder.
3. Make maps if necessary to be used to locate each sample in the SEM.

Warning Salt and oils from fingerprints will contaminate the FESEM vacuum and your samples if you do not wear gloves. Fingerprints can be removed with isopropanol and kimwipe.

Starting ZEISS SmartSEM Program

1. Sign in to the Logbook.
2. Open **SmartSEM** program and log in. Obtain login name and password from Lab manager.
3. Select **ChamberScope** to view the specimen chamber.
4. Key **Control-G** to open SEM Control window. Drag the window to the right monitor.
5. Key **Control-D** to display/hide the data zone.

Info The ZEISS (LEO) 1550VP is a fully PC controlled SEM. All operations, settings and functions are controlled by use of a key board, a mouse and a joystick.

ZEISS Software Manual is available for download on the SEM PC under E:\LEO Manuals\.

Loading the Specimen Holder into the SEM

1. Make sure the specimen holder with samples fixed is ready.
2. Use mouse to click **VAC**, and hit **VENT** (at the lower right of the LEO program), then select **YES** to the question window. N2 gas is being introduced into the chamber.
3. Put on gloves. It takes about 2-3 minutes to vent. Wait and check until the door can be opened. Open the door and slide the holder onto the stage. (If the chamber does not vent after a full 5 minutes, the N2 gas tank used to vent is empty. Get lab manager to change the N2 gas cylinder. Or an experienced user may change the N2 cylinder. If it happens after hours and you do not know how to change the cylinder, pump down the SEM chamber and leave a note on the SEM desk.)
4. Close the door immediately and hold it, then click **VAC** and hit **PUMP**.

Wait for a green tick for **VAC**. The green tick means the chamber vacuum is ready. Vacuum readings are displayed in the SEM Control window (under Vacuum / Gun Vacuum) on the right monitor.

Adjusting the SEM

1. Click **EHT** and hit **EHT on** to turn on the accelerating voltage. EHT stands for electron high tension. EHT value is displayed in the data zone, and in the SEM Control window. The accelerating voltage ranges from 200V to 30 kV. Use 10 kV for imaging, 15 or 20 kV for EDS at the beginning, then tune EHT when needed for different applications.
2. Position one of your samples below the objective lens using the joy stick with caution. **Take great care not to hit the lens or the detectors**. If a detector is damaged by a user, he/she will be billed for replacement parts.
3. Once focused, the WD (working distance between the sample surface and the low portion of the lens) should be greater than 4 mm.
4. Select **In-Lens** or **SE2** detector to display live image. Find your sample.
5. A few parameters (**Magnification / Focus, Brightness / Contrast, Stigmation, Aperture Alignment**) need to be adjusted in order for the SEM to give sharp and high contrast images at a given EHT and WD. Always do corrects and adjustments at a higher magnification, then acquire a image at a lower magnification.

Selecting a Detector

- **In-lens SE** detector is good for high-resolution topographic imaging. WD: 4mm-10mm, EHT: 300V–20kV.
- **SE2** detector (the SE detector below the objective lens) is for general propose imaging (low-magnification, high-magnification, SE and BSE imaging). WD: 4-30 mm, EHT: 300V-30kV.
- **VPSE** detector is for variable pressure imaging of non-conductive samples. WD: 6-15mm, EHT: 7kV-30kV, VP: 3Pa-100Pa using N2 gas. It is also a **CL** detector in high vacuum mode.
- **QBSD** back-scatter electron detector is good for compositional contrast imaging. WD: 5-15mm, EHT: 5kV-30kV.
- Forward scatter electron (**FSE**) detector is for orientation contrast imaging. WD: 5-25mm, EHT: 10kV-30kV. This detector may be used for STEM imaging of TEM samples.

Acquiring and Saving Images

1. Click **Photo** from the Toolbar to acquire an image (using scan speed 6 and 30 line integrations).
2. Or select **Scanning** at the SEM Control window. Set **scan speed to 6**, and select **Freeze at end frame, Line Int (N=30)**. It takes 1.3 min to acquire one image (1024 x 768 pixels) at this scan setting. One may choose different settings.
3. Once image frozen (a **blue/red dot** displayed at the lower right corner), right-click the image to send / save it into user's folder under E drive (e:\SEM_users\).
4. Click **2** of the Tool Bar for live image.

Doing EDS analysis

1. Make sure the **RemCon (EDS)** program running on the SEM computer.
2. Obtain live image in the LEO program with WD between 8mm and 18mm. To get high x-ray counts, the sample surface should be 13mm below the objective lens. This means that WD is 13mm when image is focused.
3. Activate the DELL computer on the right. Open the **INCA EDS** program. Select **Point & ID**.
4. Move along **Project -> Sample -> Site of Interest** (ie, acquire image) -> **Acquire EDS -> Quant -> Report**.
5. Save your data into C:\EDS_users\
6. Use **Oxford INCA Help** (i.e., the EDS program manual).

Electron Backscatter Diffraction (EBSD)

- EBSD is for orientation mapping at sub-micron spatial resolution and phase identification.
 - Read **HKL EBSD manual** (i.e. the HKL Channel5 Help).
 - Sample preparation is critical.
1. Make sure the **RemCon (EBSD)** program running on the SEM computer.
 2. Load a small piece of Silicon single crystal wafer along with your samples.
 3. Select **ChamberScope** view. Tilt the stage/sample carefully to 70° from the horizontal towards the EBSD detector. Use the stage to move the sample to about 15mm working distance.
 4. Get live image with SE2 detector, using 20kV EHT, fixed 15mm WD, 120µm aperture with high current mode. The configurations may be changed for different applications.
 5. Find the silicon crystal. Line up Si (110) edge parallel to the SEM x-axis (the tilting axis).
 6. Go back to **ChamberScope** view.
 7. Turn on the EBSD camera control unit and the FSE detector.
 8. Insert the camera. The camera position should be read 171 mm.
 9. **BE CAREFUL. Never ever run the stage and the EBSD detector into each other.**
 10. Go back to ZEISS image mode. Set tilt correction and dynamic focus.
 11. Open **Flamenco** program on the HKL EBSD computer (the DELL pc). Follow Flamenco manual.
 12. Move along **Imaging -> Setup -> Interactive or automatic data collection**.
 13. Do calibrations using Si crystal. Run your samples based on the HKL EBSD manual.
 14. Save your data into C:\EBSD_users\
 15. Once done, go back to **ChamberScope** view.
 16. Retract the EBSD camera fully.
 17. Move the stage down, un-tilt it.
 18. Close the **Flamenco** program. Turn off the HKL camera control unit and the FSE detector.

Shutdown

1. Go to the **ChamberScope** view.
2. If Variable Pressure (VP) mode is used, go back to High Vacuum (HV) mode from VP mode.
3. Click **EHT** and hit **EHT off** to turn off the accelerating voltage.
4. Move the stage down first. Set the stage back to X=50, Y=50, Z=1, T=0, R=0 using **Stage** at the SEM Control window.
5. Click **VAC** and hit **Vent** to vent the SEM specimen chamber.
6. Put on a pair of gloves.
7. Once the chamber vented, open the door and remove the specimen holder off the stage.
8. Close the chamber door immediately. Then click **VAC** and hit **Pump**. **The chamber must be pumped down at the end of a session.**
9. Wait for a green tick for **VAC**.
10. Go to **File** and click **Logoff**. Hit **Yes** to the two questions.
11. Transfer your files (under E:\SEM_users\) now via ftp, memory stick, or CD. Users' data files will be deleted in one month.
12. **Fill out the Logbook, including full name, PTA account,**
13. Remove samples / stubs from the holder. Put the holder back into the top drawer of the wood desk.
14. If you do not want to keep the stubs, remove your samples, then clean the stubs and leave them in the glass baker. We do recycle them.
15. Clean up after yourself.

Use Policy of the SEM computers

1. **Do not change the computers' configurations.**
2. **Do not install programs without approval of Lab manager.**
3. The PCs may be accessed for data processing when the SEM is not being used.

