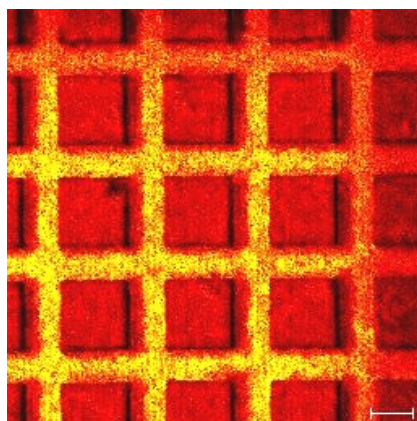


ToF-SIMS Operation Manual



Ion image of the Cu grid,
scale bar 10 μ m

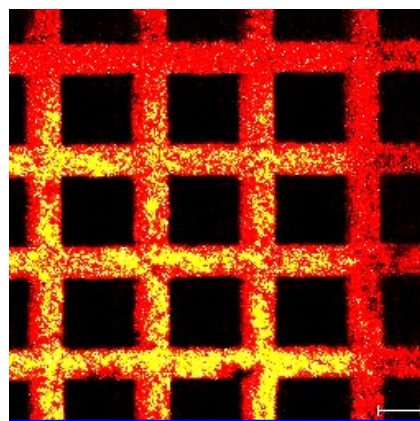
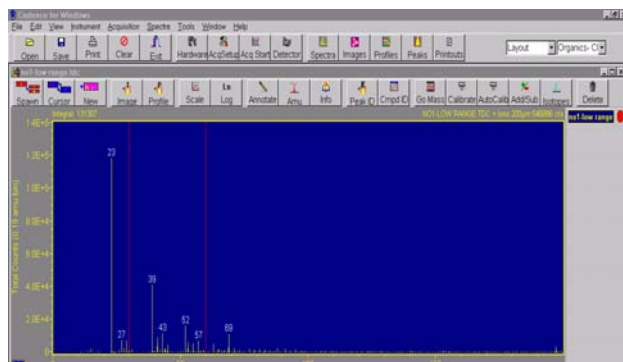


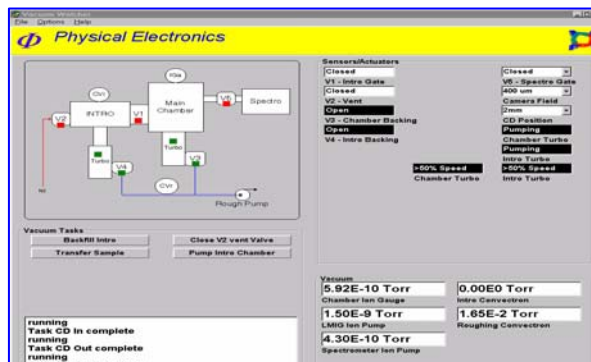
Image of the copper distribution in
the Cu grid, scale bar 10 μ m

Keck Interdisciplinary Surface Science Center
NUANCE
Northwestern University
Room 1149, Cook Hall
2220 Campus Drive
Evanston, IL, 60208

SIMS Operation Manual



WinCadence



Watcher

A. Login

Log in the computer system and write down on the log sheet.

B. Load Your Samples

Note: Make sure the SIMS instrument is in *closed status* (**section G**).

1. Go:
 - to the WinCadence* window
 - to the INSTRUMENT menu
 - to the STAGE CONTROL page
 - click EXCHANGE POSITION.
2. Go:
 - to the watcher window
 - click TRANSFER SAMPLE to open the “V1” gate.
3.
 - Slide the transfer rod to the main chamber.
 - Screw the transfer rod into the sample holder, and retract it to the beginning position.
 - The “V1” gate will close automatically. (Look at the watcher window to *make sure* the “V1” gate is closed.)
4. Open the valve of nitrogen tank.
5. In the watcher window, click BACKFILL INTRO & wait for the pressure in the intro-chamber to reach 760 Torr.

* PHI's WinCadence software system

6. Open the lid of the intro-chamber and take out the sample holder. Put your samples into the holders with clean tweezers.
7. Screw the transfer rod to the sample holder, & close the lid of the intro-chamber.
8. Click **PUMP INTRO CHAMBER**, & wait for the intro-chamber vacuum pressure to reach zero.
9. Close the valve of nitrogen tank.
10. Click **TRANSFER SAMPLE** to open the “V1” gate.
11. —Slide the sample holder to the main chamber from the intro-chamber.
—Unscrew the transfer rod from the sample holder, and slide the rod back to the beginning position.
—The “V 1” gate will close automatically. (Look at the **watcher window** to make sure the “V1” gate is closed.)
12. Go:
—to the **WinCadence window** under the INSTRUMENT menu
—go to STAGE CONTROL
—click **LOAD** & load the file of “4-position holder.plx”
—select one of four samples, such as “the upper left”
—click **DRIVE TO**
—click **OK** to close this page.
Note: You can further move the sample by the joystick in order to find the area of interest after you load instrument file.

C. Fire LMIG Gun and Set Up Parameters

1. Make sure the pressure in the main chamber is $<2 \times 10^{-8}$ Torr.
2. Go:
—to the **WinCadence window**.
—Click the **HARDWARE** button
—Go to the LMIG tabbed page
—Click the **LOAD INS FILE** button to load the instrument file, such as “15kvga3.ins” (good for spectra) or 25kvga3.ins (good for mapping) or 5kvga2.ins (good for depth profiling).
Fire the LMIG gun:
—After you load the INS file, you can see the EXTRACTOR = 7500. Change Suppressor to -500.
—Press the “+” key to increase the EXTRACTOR value to *increase* the extractor current to 8-12 for a few seconds. Then use the “-” key to *decrease* the EXTRACTOR value to 7500.

—Adjust Suppressor by pressing the “+” or “-” key in order to control the extractor current within the range of 1.2-1.6. Finally *lock* the extract current by **clicking** the **LOCK** button behind the extractor current.

(Before firing the LMIG gun, make sure the “V5” valve is closed and the data acquisition stopped. If you have difficulty in firing the gun, please contact the lab manager.)

4. Set the desired RASTER SIZE (i.e. 100 μ m, typically <800 μ m).
5. **Click** **ACQ_SETUP** to open the ACQUISITION SETUP page.
 - if your sample is *conducting*, “analysis beam only”
 - if your sample is *insulating*, “analytical with chg. comp”.
6. Go to the **watcher window**, select **OPEN** in the “V5-spectro Gate” column located on the upper right of the window.

Note: Don’t close the **watcher window** at any time.
7. **Click** the **VIDEO** button and switch it to the detector mode.
8. **Click** **ACQ_START**.
9. Observe the SONY monitor screen to make sure that the scanning secondary ion beam is located inside the square and the intensity is reasonable.
 - If not, go to the “Hardware-LMIG” page to adjust the X beam Position, Y beam position, and Sample voltage by pressing the “+” or “-” key to get reasonable counts.
 - After finishing, click the **OK** button to close the “LMIG” page.

D. Acquire Your Data (Spectra, Mapping, Depth Profiling, and Helpful Tips)

1. Spectra Acquisition

Click the “Spectra” on the top menu of the **WinCadence window**.

Notes: You can stop data acquisition by **clicking** **ACQ_STOP**.

You also can set a certain time for data acquisition in the basic setting of ACQ SETUP page.

2. Mapping

a) How to generate an image

—Click the **IMAGES** button.

—In images-tabbed window, use the mouse to **click** **TOTAL IONS** in the column on the left side and **drag** it to the blank square on the right side. An image of “total ion” will display.

Note: If you are interested in the distribution of some specific peaks, you need to make new peaks. (Refer to Peaks Section “helpful tips 2”.)

- b) Imaging mode: To eliminate artifacts on mapping, you need to open “Acquisition Setup” window, and click “Advanced Settings” tab, and choose “imaging mode 16x16” or “imaging mode 32x32” or “imaging mode 64x64” from “Detector Scan”. The default is “Fast Mode”
- c) How to use the inspector function (examine the details of a single image)
- Click the arrow of the **MULTI** button and select the mode **INSPECTOR**.
 - Select the name of the species from the table on the left side of window. The corresponding image will appear on the right side, along with detailed information.
- d) How to use the line scan function (reveal the chemical distribution along a line)
- Click the arrow of the **MULTI** button in the **image window** and select the mode *LineScans*. A LINESCANS page will display.
 - Select the name of the species from the table on the left side and drag it to the blank square on the lower right of the window. An image will occur.
 - Hold the left button of the mouse to draw a line. A chemical line distribution will occur.

3) Depth profiling

Note: First you need to create two different instrument files: one for analysis, the second for sputtering.

- a) Create the *analysis instrument file*
- Use the joy stick to move the sample and find an area of interest.
 - Load the instrument file, such as “5kvga2analysis.ins”.
 - Fire the gun and lock the extractor current within the range of 1.2-1.6.
 - Change the mechanical movable aperture to ring_2 (**ask facility manager to help**)
 - Set the raster size to 25µm.
 - Click **SAVE INS FILE**, & save in *your own folder* as “xxx-analysis.ins”.
DO NOT overwrite the original file.
- b) Create the *sputtering instrument file*
- Set the raster size to 100 µm.
 - Click **SAVE INS FILE** and save in *your own folder* as “xxx-sputter.ins”.
 - Load the analysis file you created in step a.
- c) Pick up the peaks of interest
- Make peaks that interest you in the depth profiling. (Refer to **helpful tips 2**)
- d) Set up and start the depth profiling

Analyze Phase		Sputter Phase		Charge Comp. Phase	
Settle time	1	Settle time	1	Settle time	1
Analyze time	15 or 23 or others	Sputter time	5-100	Comp. time	0 for conductive 1-10 for insulating
Comp. duty cycle	0 for conductive 55 for insulating	Sputter INS file	Load your own sputter file		
		Sputter gun	LMIG		
		Sample has	H.V.		

- Click the **ACQ_SETUP** on the top menu in the **WinCadence window**, and check “Phased Profile”. **Set up the parameters** as outlined in the above table.
- Click **ACQ_START** and then click the **PROFILES** button on the top menu to observe the depth-profile curves.
- Click the **ACQ_STOP** button to stop the measurement.
- **Select** all peaks and save together. The data is saved as “.dat” file and “.txt” file. (The “.txt” file can be opened in MS Excel.)

Helpful Tips To Improve Your Data Acquisition Results

1. Negative ion mode

Click **ACQ_STOP**, go to INSTRUMENT menu, then “Negative(-) ions”. The spectrum will switch to the negative ion mode.

Press the “F4” key to refresh the data. After you finish the measurement under the negative ion mode, go back to the positive ion mode.

2. Peaks

If you are interested in a specific peak, such as “27”, click **PEAKS** on the top menu.

A **peak window** will display.

Click the **NEW PEAK** button, and write the mass “27” in the “formula” column.

Adjust low and high mass to cover the whole peak.

-For *mapping*, check IMAGING

-For *depth profiling*, check PROFILING.

You can also select a peak with the two cursors and then click “image” or “profile” buttons on spectrum window

3. SEM imaging with the SIMS (the resolution is bad)

The SIMS can image surface morphology, the same function as performed with a scanning electron microscope (SEM). Follow the steps below:

- Switch the “positive ion mode” to the “negative ion mode”.
- Click **ACQ_SETUP**. In the ACQUISITION SETUP page
- Check the “SED image”. Click **OK** to close this page.
- Go to the LMIG page to increase the “SED gain” by 300-500.
- Press “F4”. Go to the **images window** described in Tip #2 on page 5. You can see the name *SEM* in the left column. Use the mouse to **select SEM** and **drag** it to the right blank square. A SEM image will display.
- After you have finished the acquisition of the SED image, reduce the “SED gain” to the original value of around 1000 and switch “negative ion mode” to “positive ion mode”.

4. Clean the sample surface using the “sputter tool”

- Move sample to the area of interest using the joy-stick.
- Close the “V5” gate valve in the **watcher window**.
- Select the “sputter tool” in the INSTRUMENT menu of the **WinCadence window**.
A **sputter tool window** will pop up.
- Set the raster size and the sputter time (s), and **click** the **START SPUTTER** button to sputter the sample surface.
- After finishing the sputtering, click the **OK** button to close the **sputter tool window**.
- Open the “V5” gate valve in the **watcher window**.

Note: The sputter area should be at least three times bigger than the analysis area.

E. Analyze Your Data

1. Calibration

To accurately calibrate the mass axes of the spectrum, **click** the **CALIBRATION** button. A calibration–tabbed window will display.

Select “CH3” in the list and **move the double cursor** to “hug” the peak of “CH3”.

Select “C2H3 to calibrate the peak, & **then select** “C3H5” to calibrate the peak.

Click **OK** to close the window.

2. Spawn

The **SPAWN** button is used to generate a subsequent spectrum from the existing spectral data.

Use the mouse to move two cursors in the existing spectrum to a narrower field of view, & then **click** the **SPAWN** button to generate a second spectrum which displays a more detailed spectrum.

3. Peak ID

The **PEAK ID** button is used to identify the chemical formula for a peak of interest.

When you **click** the Peak ID button, a window will display.

Click the **ELEMENTS** button.

Double click the element involved in your sample, & the checked elements will become **red**.

Click the **OK** button to close the window.

Move the double cursors to “hug” a peak of interest, & then **click** **PEAK ID**.

The Peak ID database displays all the possible chemical formulas with exact mass in good agreement with the peak of interest.

4. Blank Peaks

If you do not want a certain peak to show in the spectrum, you can run this function.

Click **PEAKS** on the top menu, & a **peak window** will display.

Move your cursor to select the peak you do not want, & check **BLANKING**.

Return to the **spectrum window** to see what has happened.

5. Evaluating Peak Intensity Using “Job Wizard” (*Quantitative Analysis*)

This function is to read automatically the peak intensity and the normalized peak intensity using “WinCadence” software. For example, there are three Si-wafer samples with different contents of impurities such as *B*, *Mg* and *K*. You want to quantitatively compare the content of impurities in the three Si-wafer samples.

a) Obtain and save the SIMS spectra for all the samples.

Before saving the spectra, you *must* calibrate the spectrum.

Saved file names might be, for example, “test.tdc”, “test 1.tdc” and “test 2.tdc”.

b) Open one of the saved files (for example, “test 1.tdc”).

Click the **PEAKS** button.

In the **peak window**, make new peaks such as *B*, *Mg*, and *K*, as described in Tip #2 on page 5. Select all the peaks you have picked, & **click** the **SAVE** button.

Name the peak file (example: “metals_si wafer.pk”).

c) **Select** the **JOB WIZARD** in the TOOL menu. A **job wizard window** will open. Find the saved spectra files in the left column, and **click** the **ADD FILES=>** button to add the files to the right column.

d) **Click** the **NEXT>>** button, the SPECTRA FILES page will switch to the PEAK FILES page. Then **select** your peak file, such as “metals_si wafer.pk” and **click** the **SELECT** button to add the peak file to right column.

e) **Click** the **NEXT>>** button, and the PEAK FILES page will switch to the CALIBRATION page. **Click** the **NEXT>>** button (Do not calibrate here), the CALIBRATION page will switch to the TABLE SETUP page.

f) In the TABLE SETUP page, select the peaks of interest (for example, *B*, *Mg*, and *K*), and **click** **AS COUNTS=>** button to add the above peaks to the right column.

Note: If you want get the normalized peak intensity, first select the reference peak in the TABLE SETUP page and click the NORMALISE TO=> button to add the reference peak. Then select the peaks of interest, and click AS CONC'S=> button to add the above peaks to the right column).

- g) Click the FINISH button.
- h) Click the VIEW RESULTS button. A table, which shows the peak intensity, will display.
- i) Click the CLIPBOARD button and paste the table into MS Excel. Process your data in MS Excel.

F. Save Your Data

1. Save the spectra:
Click SPECTRA and click SAVE. You will get a file with the format "*.tdc".
To export the spectrum data as a text file, click SAVE, & save the file in the "ASCII unit-mass spectrum (*.ASC)" format. (You will be able to use MS Excel to open your saved file.)
2. Save the images:
Click IMAGES & click SAVE. You will get a file with the format "*.ims".
3. Save the depth profile figure:
Click PROFILES and click SAVE. You will get a file with the format "*.DAT".

Note: To copy a single spectrum or image to other software such as MS Word, click the spectrum or image of interest, use the *copy* function, & *paste* the spectrum or image to the Word file.

G. Shut Down the SIMS Instrument (*closed status*)

1. *Make sure* you have stopped the data acquisition
(Tip: You can see the ACQ_START button after you have stopped acquisition).
2. Click the VIDEO button to switch it to SAMPLE.
3. *Make sure* the instrument is under the positive ion mode.
4. Unlock the extract current (the LOCK button is on the lower right of the WinCadence window).
5. Click HARDWARE. Go to the LMIG page & set "suppressor" to -10.
6. Click the LOAD INS FILE button to load the instrument file of "zero.ins".
7. Click OK button to close the LMIG page.
8. Go to the watcher window, & select CLOSED in the "V5-spectro gate" column on the upper right corner of the window.
9. Observe the watcher window to *make sure* the "V1" gate is closed (red color indicates "closed"; the green color indicates "open").

H. Unload Your Samples

Make certain the SIMS instrument stays in the “closed status”, as described in section G.

1. Go to the **WinCadence window** in the INSTRUMENT menu
2. Go to the STAGE CONTROL page, **click EXCHANGE POSITION**.
3. Go to the **watcher window** & **click TRANSFER SAMPLE** to open the “V1” gate.
4. Slide the transfer rod into the main chamber.
Then screw the rod into the sample holder, and slide back the sample holder to the beginning position. At the same time, the “V1” gate will close automatically.
Observe the **watcher window** to *make sure* the “V1” gate is closed.
5. Open the valve of the nitrogen tank.
6. **Click BACKFILL INTRO**, & then wait for the pressure in the intro-chamber to reach 760 Torr.
7. Open the lid of the intro-chamber, and take out the sample holder.
Note: Please use acetone to ultrasonically clean the tweezers, discs, and apertures which contact your samples directly.
8. Put the sample holder into the intro-chamber, and close the lid of the intro-chamber.
9. **Click PUMP INTRO SAMPLE**, and then wait for the intro-chamber vacuum pressure to reach zero.
10. Close the valve of the nitrogen tank tightly.
11. **Click TRANSFER SAMPLE** to open the “V1” gate.
12. Slide the transfer rod to transfer the sample holder to the main chamber from the intro-chamber.
Then unscrew the rod from the sample holder, and slide it back to the beginning position.
At the same time, the “V1” gate will close automatically.

I. Logoff

Log off the computer system and write down on the log sheet.