TOF SIMS Procedure

TOF SIMS Lab



Daily procedure:

- **1. Record pressure**
- 2. Start LMIG source
- 3. Data acquisition
- 4. Shut down
- 5. Data interpretation

Loading Samples into Loadlock



- **1.** If sample holder is in the main chamber, click "exchange position" from the drop down menu in Navigator window and press Go button.
- 2. Click Open button to open the gate between main chamber and loadlock.
- **3.** Move transfer rod to the main chamber, turn the rod and lock the sample holder and move transfer rod back to loadlock.
- **4.** Close" button is black when the gate is open, which allows you to close the gate. Click close to close the gate.
- **5.** A stage control window pops up on top of Navigation window, Click OK.
- 6. Click stop to vent the loadlock chamber. It will take about 2 min.
- **7.** Take the holder out and mount samples. Make sure there is no loose powder. Check that screws on the back of the holder are all tightened.
- 8. Insert the sample holder in the transfer rod and close the loadlock door.
- 9. Click Start to pump down the loadlock chamber.



Record Pressure and Load Samples for Analysis

Power On Settings



Load Sputtering Gun

1. This is only needed if acquiring depth profile. Do this step before starting LMIG.

2. Select Load setting

Select DSC-S-A folder: Cs-1Kev.tmt, Cs-3Kev.tmt , Cs-10Kev.tmt **3.** Wait 1 hr for Cs source to stabilize.



IONTOF Power On Settings H HC-BUNCHED.tr E P Automation... Primary Gun 🔽 🛛 LMIG Ŧ Bi3 • Sputter Gun DSC-S/A -Cs Ŧ Charge Comp. 📃 Floodgun Instrument Setup ₽ ₽ Negative 🔻 Cycle Time 100 us • Þ Mass range 1 - 722 u Acquisition ₽ ĸ <u>tailt</u> 0 cts/s Q ► Measurement i 前 Prompt user • Navigation ÷ N Screen Layouts -Vacuum Main 1.1E-8 mbar LL 5.1E-8 mbar Start Stop GateMain closed Open Close

🔓 LMIG - Instrume	ent ? X
Analyzer/Main	Emission
LMIG	Standhu 🔻 🖯 0.00 uá
Source	Emission Current Set 0.50 uA
Pulsing	Heating 2.80 A Save
	Summer 01/
	Suppressor 0 v
Mass Filter	Extractor 8000 V
	· · /
	Energy 25000 V
	< Z •
	Start LMIG.
	Stop LMIG
DSC-S/A	Target Current Measurement
Flood Gun	Manual 🔻 Start 🖨
Loadlock	
Vacuum	
Expert	Autorange V Average
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LMIG - Instrume Analyzer/Main LMIG Gun Gun Pulsing Focus Raster Mass Filter	ent Emission Auto 0.52 uA Emission Current Set 0.50 uA Emission Current Set 0.50 uA Heating 2.80 A Save Heating 2.80 A Save Check Suppressor 1230 V Extractor 8000 V
LMIG - Instrume Analyzer/Main LMIG Source Gun Pulsing Focus Raster Mass Filter Vacuum	ent Emission Auto 0.52 uA Emission Current Set 0.50 uA Emergy 2.80 A Extractor 8000 V Extractor 8000 V
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LMIG - Instrume Analyzer/Main LMIG Source Gun Pulsing Focus Raster Mass Filter Vacuum	ent Emission Auto 0.52 uA Emission Current Set 0.50 uA Emission Current Set 0.50 uA Heating 2.80 A Save Heating 2.80 A Save Check Suppressor 1230 V Extractor 8000 V Energy 25000 V Energy 25000 V Start LMIG Stop LMIG
LMIG - Instrume Analyzer/Main LMIG Source Gun Pulsing Focus Raster Mass Filter Vacuum	ent Emission Auto 0.52 uA Emission Current Set 0.50 uA Emission Current Set 0.50 uA Heating 2.80 A Save Heating 2.80 A Save Check Suppressor 1230 V Extractor 8000 V Extractor 8000 V Energy 25000 V Energy 25000 V Energy 25000 V Target Current Measurement
LMIG - Instrume Analyzer/Main LMIG Source Gun Pulsing Focus Raster Mass Filter Vacuum	ent Emission Auto 0.52 uA Emission Current Set 0.50 uA Emission Current Set 0.50 uA Heating 2.80 A Save Heating 2.80 A Save Check Suppressor 1230 V Extractor 8000 V Extractor 8000 V Energy 25000 V Energy 25000 V Start LMIG Stop LMIG Target Current Measurement Manual Start
LMIG - Instrume Analyzer/Main LMIG Source Gun Pulsing Focus Raster Mass Filter Vacuum DSC-S/A Flood Gun Loadlock	ent Emission Auto 0.52 uA Emission Current Set 0.50 uA Emission Current Set 0.50 uA Heating 2.80 A Save Heating 2.80 A Save Check Suppressor 1230 V Extractor 8000 V Extractor 8000 V Energy 25000 V Energy 25000 V Energy 25000 V Energy 25000 V Energy 25000 V Energy 25000 V
LMIG - Instrume Analyzer/Main LMIG Source Gun Pulsing Focus Raster Mass Filter Vacuum DSC-S/A Flood Gun Loadlock Vacuum	ent Emission Auto O.52 uA Emission Current Set 0.50 uA Emission Current Set 0.50 uA Heating 2.80 A Save Heating 2.80 A Save Check Suppressor 1230 V Extractor 8000 V Extractor 8000 V Energy 25000 V

Start the LMIG Bi Source

Click Start LMIG

 (Liquid Metal Ion Gun)
 Then window shown
 on the right pops up.

 Select appropriate file

 and click Open to start
 the LMIG source:
 HC-bunched (high mass
 resolution);
 BA-image (high spatial
 resolution imaging).

 The start LMIG recipe

 will fire the LMIG. It
 takes about 5 min.

Load Settings File							×
🔾 🗢 📕 🕨 Operator	My Documents ► settings ► LMIG			▼ 49	Search LMIG		٩
Organize 👻 New folder					E ·		0
☆ Favorites	Name	Date modified	Туре	Size			
📃 Desktop	BA-IMAGE.tmt	6/29/2016 12:00 PM	ION-TOF Settings	2 KB			
〕 Downloads	E HC-BUNCHED.tmt	6/28/2016 3:06 PM	ION-TOF Settings	2 KB			
Recent Places	EX-IMAGE.tmt	8/11/2015 12:28 PM	ION-TOF Settings	2 KB			
	HC-BUNCHED no buncher voltage.tmt	2/24/2015 11:21 AM	ION-TOF Settings	2 KB			
🥽 Libraries	R-BUNCHED_40 ns_a.tmt	4/11/2014 3:42 PM	ION-TOF Settings	2 KB			
Documents	HC-BUNCHED_60 ns.tmt	7/24/2013 11:10 AM	ION-TOF Settings	2 KB			
🎝 Music	HC-BUNCHED_40 ns.tmt	9/28/2011 11:03 AM	ION-TOF Settings	3 KB			
Pictures	HC-BUNCHED_100 ns.tmt	7/23/2010 3:32 PM	ION-TOF Settings	2 KB			
😸 Videos	HC-BUNCHEDa.tmt	11/11/2009 4:17 PM	ION-TOF Settings	2 KB			
	HC-unBUNCHED.tmt	4/21/2009 10:40 AM	ION-TOF Settings	2 KB			
🌉 Computer	C-BUNCHED.tmt	4/21/2009 10:37 AM	ION-TOF Settings	2 KB			
🚢 Local Disk (C:)							
💿 DVD RW Drive (D:) I(
🖵 tofsims (\\152.14.54.							
年 Network							
File na	me: HC-BUNCHED.tmt			•	Settings files (*.tmt)		-
						C	
					<u>U</u> pen 🔻	Cancel	



5. On Instrument window, click LMIG + SHIFT together to expand LMIG parameter window

Source	Gun	Focus	Raster	Pulsing
Emission Current 0.49 uA	Lens Source 2818 V	X Blanking -16.6 %	Sensitivity X 78.9 %	Cycle Time 100 us
	 ► 	< _ ►	۰ •	I 🗌 🗌
Heating 2.80 A	X Source -10.1 %	Y Blanking 6.7 %	Sensitivity Y 57.9 %	Width 19.9 ns
<	<	۰ F	↓ ►	 I
Suppressor 1231 V	Y Source -9.8 %	Lens Target 69.310 %	X Target 30.1 %	DC 📃
<	< _ >	۰ ا	I → I	Voltage 2170 V
Extractor 8000 V	Lens Mag 15101 V	X Stigmator 28.4 %	Y Target -19.4 %	•
<	+	< 📄 🕨		Delay 279.7 ns
Eperau 25000 V	X Mag 15.9 %	Y Stigmator 33.9 %		< □
<	K	< _ >		🔲 Burst Mode
	Y Mag -10.2 %			Burst Delay 0 %
Aperture 1 17.8 nA	۲ E	Aperture 2 -0.0 nA		•

Bi Beam Alignment for HC_bunching



Emission current set to 0.5 µA when it is selected as Auto: Suppressor voltage will automatically control emission to the set value.

1. Emission current should be around 0.5μ A but can be up to 1 μ A. The current is set by emission current set (select auto).

2. Click Aperture 1, center the beam by clicking center for both X source and Y source (shape should be plateau + sharp slope). Close the scan window after centering. Same for Aperture 2 (trapezoid).

3. Apt1 should have current > 18 nA ideally at 0.5 μA current. The worst case should be > 16 nA. If current in Apt 1 is below <16 nA, see note in next slide or look for Elaine

4. Record the current in Apertures 1 and 2. Need aperture 2 window open to read aperture 2.

5. Note: values of heating, suppressor, extractor, lens source displayed in the manual may be different from current setting, which is fine.

If current in Apt 1 is low <16 nA, try following steps:

1. Uncheck emission control in LMIG-Instrument window

2. Drag Suppressor bar to maximum voltage 2000V. Normally you will see the emission current increase to its maximal current 12.5 μA. The current in Apt 1 is 62.5 nA. If you do not see the current goes to its maximum, increase Extractor little by little until the maximal current is reached.

3. Decrease Suppressor voltage until the current is around $1 \mu A$.

4. Check the Apt 1, Apt 2 current and center the beam.

5. If you change the Extractor voltage, you also need to change the lens source voltage. The rule is: the higher the extractor, the higher the lens source.

6. Adjust lens source with Apt 1 scan window open. Adjust lens source until a plateau + sharp slope is obtained.

7. If this still does not solve the problem, please contact Elaine for further assistance.

Bi Beam Alignment for BA_image



1. Emission current should be around 1.0μ A. The current is set by emission control (check box is checked).

2. Click Apt 1, increase width to 40% center the beam by clicking center for both X source and Y source (plateau + slope). Close the scan window after centering. Same to Apt 2 (plateau + sharp slope).

3. Apt1 should have current > 17 nA ideally at 1.0 μA current. The worst case should be > 15 nA. If current in Apt 1 is below <16 nA, see note in next slide or look for Elaine)

4. Record the current in Apt 1 and 2.

5. Note the value of heating, suppressor, extractor, lens source displayed in the manual is different from current setting, which is fine.

Bi Beam Focusing for BA_image

🖶 Navigator - Backmount V1.shi		
Navigator SE/SI Video Stage		
16:59:21		
	Source Emission Current 0.97 w Heating 2,80 A	Gun Focus Raster Putsing Lens Source 3054 V X Blanking -11.3 % Sensitivity X 53.7 % Cycle Tim X Source -6.2 % Y Blanking 4.9 % Sensitivity Y 62.5 % Width 2 Y Source 35.6 % Lens Target 69.520 % X Target 76.7 % DC Y Lens Man 15216 V X Stimmator 291 % Y Lens H-4 4 %
⊢ 1 μm 20.0 × 20.0 μm 100 μm 750 ×; SE / SI Image 4 defunt SI	750 µm 10 mm 126 ×122 mm Aperture 1 3.3 n ⁴	X Mag 13.9 % Y Stigmator 9.8 % Y Mag ·14.9 % Aperture 2 0.0 nA
Se Victoria Since Action Sectoria Secto	A CT C C C C C C C C C C C C C C C C C C	

1. Find "A Grid" from the drop down list in stage control window and click go.

2. If holder contains samples higher than the surface, move Z handle UP a little bit, watch through the main chamber window to see the distance between holder and extraction cone. Click A grid position on stage control and then hit go. After the stage moves to "A grid", repeat step 1 (this will move to the right Z height).

- **3.** Right click mouse, change field of view to 50 μm x 50 μm.
- **4.** Check the box before DC in LIMG window.
- 5. In SE/SI image window, change SI to SE, pixel size to 256 x 256, scanning type to Sawtooth. Increase SE multiplier to 8000V.
- 6. Click black arrow to start SE image acquisition, tune Lens Target, X and Y stigmator for best image resolution.
- 7. Change filed of view to smaller area and repeat step 6.
- 8. Click square button (the same position as the black arrow). Uncheck DC, reduce SE multipler to OV, change SE to SI.

Measure Current



💁 Analyzer/Main - 🛛	Instrument
Analyzer/Main	Extractor 0 V
Extraction	× 📄 🔹 🕨
TOF	Energy 0 V
	•
	Lens 0 V
Detector	< <u>⊢</u> ►
	× 0.0 %
Timing	K 📄 🕨
	Y 0.0 %
	< 📄 🕨
H/C	VDP 0.0 V
	< 📄 🕨
	Reflector 0.0 V
	< 📄 🕨
	Linked
LMIG	
DSC-S/A	Target Current Measurement
Flood Gun	Manual 🔻 Start 🖨
Loadlock	I Target -0.023 🗚 25 uA 🗸
Vacuum	Autorange V Average
Expert	

- Move the stage to Faraday cup: select faraday cup from the drop down menu and click go (Make sure no "protruding objects" on the holder otherwise the extraction cone will be destroyed)
 IMPORTANT: DO NOT MOVING THE HANDLE DOWN TOO FAST (Z direction) OR THE EXTRACTION CONE WILL HIT THE STAGE AND BE DESTROYED.
- **2.** Turn off illumination. Illumination check box should be unchecked.
- **3.** Make sure polarity in instrument panel is in positive.
- **4.** Right click mouse on SE/SI image window. Select specify raster field of view and change to 20 μ m x 20 μ m.

- 5. Autorange and Average boxes are checked.
 6. The box in front of LMIG is checked if measure Bi current. Thebox in front of Cs should not be checked.
- 7. Click start from the instrument window. Right down the Target current and then click stop.
 (Start button changes to Stop when measuing the current.)



Load Setting files

1. Select Load setting

2. Select appropriate folder for the

respective species and open file

LMIG folder:BA_image.tmt (for High resolution image)
HC_bunched.tmt (for High mass resolution spectrum)DSC-S-A folder:Cs-10Kev.tmtAnalyzer folder:Analyzer for HC.tmt

Flood Gun folder:

- 3. Flood gun is needed only if the sample is not conductive.
- 4. DSC-S-A is needed only if you want to acquire depth profile. Wait 1 hr for Cs source

Flood Gun.tmt





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Vacuum

Check Mass Resolution



Mass (u)

Mass Calibration

Positive ion spectrum on Contaminated Si



Data Acquisition

<u>Navigator</u> <u>S</u> E/SI Video Sample Holder		→I‡ 🗉 🔧 🗞
<u>1 20 μm</u> 250 x 250 μm	10:01:23	
SE/SI Primary Gun SE Sputter Gun SI V M SE Sputter Gun SI Gain: 20 Cceptance Circle switched OFF	Video Micro Show Crosshair Laser	Stage Control

- 1. Move to the sample to be analyzed by clicking the sample location on the holder in stage control window and then hit go. IF samples are top mounted, move handle Z UP (increase the distance between holder and TOF extraction cone) and do step 1.
- 2. Field of view: change to 100 μm x 100 μm or larger.
- **3.** Start acquisition by clicking the black arrow and check Adjust SI.
- **4.** Adjust the sample holder height by moving the secondary ion circle to the center. (Z handle up to move SI circle to the right and vice verse).
- 5. Uncheck Adjust SI
- 6. Change Field of view to desired value.
- 7. Start acquisition by clicking the black arrow and hit the same button once a spectrum is shown in spectrum window.
- 8. Calibration the mass spectrum.

Data Acquisition for Insulating Samples

- 1. Select load setting in Fpanel and load flood gun setting.
- 2. Change field of view to 500 μm x 500 $\mu m.$
- 3. Start acquisition by clicking the black arrow and check Adjust SI.
- **4.** Adjust the sample holder height by moving the secondary ion circle to the center. The secondary ion circle may appear to be fuzzy due to charging.
- 5. Go to Analyzer/Main in instrument window (shown right), click TOF on left colunm.
- **6.** Check the box before Linked.
- **7.** For positive ion mode, decrease reflector voltage (move to the left) until SI circle disappears on the SE/SI window.
- 8. From this point, increase 20V (move 20 V to the right).
- 9. Uncheck Adjust SI
- **10.** Change Field of view to desired value and start acquisition by clicking the black arrow and hit the same button once a spectrum is shown in spectrum window
- **11.** Calibration the mass spectrum. (note: calibration will be off if reflector voltage is changed).

🔓 Analyzer/Main -	Instrument ? ×
Analyzer/Main	Extractor 0 V
Extraction	
TOF	Energy 0 V
Raster	Lens 0 V
Detector	×
-	× 0.0 %
Timing	+
Vacuum	Y 0.0 %
	I → I
H/C	VDP 0.0 V
	۰ F
	Reflector 0.0 V
	<
	Linked
LMIG	
DSC-S/A	Target Current Measurement
Flood Gun	Manual 🔻 Start \varTheta
Loadlock	I Target -0.024 μ≙ 25 μ≙ ▼
Vacuum	
Expert	Avelage



Depth Profiles: Data Acquisition with Dual Beam

🕂 Navigator - Backmount V1.shi		DSC-S/A					? ×
<u>N</u> avigator <u>S</u> E/SI V <u>i</u> deo S <u>a</u> mple Holder		DSC - Source	DSC - Gun	DSC - Pulsing	DSC - Focus	DSC - Raster	DSC - Heating
-10:08:33	- 0	Emission 0.0 mA	Energy 0 V	Cycle Time 100 us	Lens Crossover 0.00 %	Sensitivity X 30.3 %	-
		Heating 0.00 A	Extractor 0.0 V	Cs Pulser 0 V	X Crossover 0.00 %	Sensitivity Y 27.2 %	Voltage 10.00 V
			Lens Source 0.00 %	El Pulser 0.0 V	Y Crossover 0.00 %	X Target 0.0 %	C60 Heater 0.04 A
		Anode 160.0 V	X Source 0.00 %	Buncher 0 V	Lens Target 0.00 %	Y Target 0.0 %	Cs Heater 0.00 A
			Y Source -0.01 %		Stigmator 0.0 %	RasterSize 0 um	HeaterRelay
	127 x 27 300	Current 1 -0.01 uA	Stigmator Source 0.0 %		Current 2 -0.00 uA		
Sels Prinary Gulf Sels Sputter Gulf Micro Bightness 0 % C St User list User list User list	•	Current 2 -0.00 uA					Voltage 0.00 V <
Si Gain: 20 Laser Faraday Cup	Gol + Edit						Heating Current 0.08 A
Acceptance Circle switched OFF	x:-61.6mm,y:76.7mm		/				

- 1. Check sample height
- 2. Right click mouse on SE/SI image window above and change Field of View to 500 μm x 500 μm.
- **3.** Change Raster size in DSC-S-A window to 120 μm x 120 μm.
- **4.** Align LMIG and sputtering gun: start a SI image of the sample in the navigator.
- 5. Enable the sputtering beam for a few seconds (Check the box before sputter gun and then uncheck in a few second), the modification of the SI yield by the sputter beam on the sample should become visible in the SI image. Typically, the sputter beam lowers the SI yield by cleaning the sample from surface contaminants and by reducing the oxygen content at the surface.
- 6. Move mouse to the middle of the crater generated by sputtering beam and drag to about 50 μ m x 50 μ m.
- 7. Right click mouse on SE/SI image window and change Field of View to 50 μ m x 50 μ m.
- **8.** Start acquisition by clicking the black arrow, stop the acquisition by clicking the black stop button.
- 9. Calibrate the spectrum and add the ions of interests to peak lists.
- **10.** Move to a new spot that is close to the alignment spot.
- 11. Open depth profile window.
- 12. Click the red arrow in Fpanel to start the acquisition

13. Select 3D for depth profile. Change raster to random (128 x 128 pixel) and type a desired number in Number of scans and then click OK.

14. Save the measurement after the acquisition is finished.

Shut Down Procedure

A =	Name	Date modified	Type	Size			
💢 Favorites		bate mounied	()pc	Size			
Desktop	Janalyzer	8/11/2015 3:47 PM	File folder				
Downloads	Archive Archive	8/11/2015 3:54 PM	File folder				
Recent Places	DCS-S-A	2/12/2016 9:25 AM	File folder				
_	🎍 depth profile	1/13/2015 2:44 PM	File folder				
📜 Libraries	🎍 Flood Gun	1/13/2015 2:44 PM	File folder				
Documents	LMIG	2/24/2015 11:21 AM	File folder				
J Music	퉬 Setting Start	1/13/2015 2:44 PM	File folder				
Pictures	Cff_LMIG_Anal_FldGun_C60.tmt	2/1/2010 1:51 PM	ION-TOF Settings	4	KB		
😸 Videos	Cff_LMIG_Anal_FldGun_Cs.tmt	5/31/2012 9:31 AM	ION-TOF Settings	4	KB		
🖳 Computer							
🏭 Local Disk (C:)							
DVD RW Drive (D:) IC							
🖵 tofsims (\\152.14.54.							
📬 Network							
·							

IONTOF Power On Settings P

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HC-BUNCHED Im **•**•!

Automation..

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Primary Gun

Bi3

Sputter Gun DSC-S/A

Cs

Charge Comp. 🔲 Floodgun

Instrument Setup

•

Mass range 1 - 722 u

Acquisition

0 cts/s

Q

Measurement

Prompt user

Navigation

Screen Layouts

Main 1.1E-8 mbar LL 5.1E-8 mbar

GateMain closed

÷

Vacuum

Start

Open

Negative 👻 Cycle Time 100 us

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- 📳

Stop

Close

🔽 🖬

1. Click load setting button on Fpanel and then select off LMIG Anal FldGun Cs file. Note: If Cs is on, reduce heating to OA and then wait for 5 min before loading off Cs file.



Categories to load

Analyzer

DSC-S/A

LMIG

Flood Gun

All

Categories stored in the settings file:

2. Select the categories that need to shut down

(ctrl+click for multiple entries) and then click

Selected

3. Change auto to standby and turn heating to 0A from LMIG window.

х

Cancel

4. Turn off illuminator. 5. Select exchange position from drop down list and click go. 6. Open the gate between LL and Main. 7. Move the holder to LL.