Standard Operating Procedure

Leica ACE 600 Sputter Coater



Yale West Campus Materials Characterization Core *ywcmatsci.yale.edu*

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Leica ACE 600 Sputter Coater SOP

1. Log FOM to turn on Sputter Coater (Leica ACE 600 High Vacuum).

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    Sputter Coater (Leica ACE 600 High Vacuum)
Equipment status: Available
Mark status as DOWN Turn Relay ON OFF Detect relay status

Note to users
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2. Open N₂ valve, make sure the pressure is around $7.5 \sim 12$ psi. If possible, do not tune the flow rate knob.



Only open this valve. Avoid tuning flow rate.

3. Click **Vent** to vent the chamber. Wait until the pressure reaches at least **8E+2 mbar** to open the chamber window.

Pressure: 6.6E-7 mbar Pump	Sputtering	Glow discharge	Sequence		S	Varning Service interval gauge Service gauge	e exceeded	·///0014
Standby Vent		¥.	Process 1			Gold		
	Open 🕥	Open 🕥	Open 🕥	ent	Ar	Source:	left Tilt	
	Aenu 🔳 Light	*	10:05 2019-05-23	N	[mbar]	[mm]	m	

Please ignore the warning message "Service interval gauge exceeded Service gauge".

- 4. Mount your sample on the sample stage using double side scotch tape.
 - Each small stage can self-rotate. If you want to rotate samples, make sure your sample is not touching nearby stage.
 - For large sample, like glass slide, you can put your sample on more than one small stage, **but make sure the stage is not rotating** (Rotation speed is 0, shown in Stage setup).
 - Check and make sure each small stage sits in the large stage properly. (Otherwise Stage Motor Error may happen.)
- 5. Clean the chamber using N2 gun in fume hood if the chamber is dirty.

- 6. Lock the door/window.
 - Note: please use N2 spray gun or Vacuum to clean the chamber if it is dirty. The small flakes in chamber may contaminate your samples.
- 7. Check and Change target if needed.

Note: Target change must be done in Vent chamber state. Please check 'Comments' of logbook for target information.

1) Open the sputter head cover, disconnect the power cable shown below.



- 2) Loose the two screws to release the Sputter Head.
- 3) Take off the head, flip it and put on a clean paper.
- 4) Rotate the standard ring and take off the target foil using tweezers.
- 5) Please put the target into the **Correct** bag.

Note: please wear the gloves to avoid the contamination of target.



- 6) Put the new meal target and lock using standard ring.
- 7) Put the sputter head back, make sure the target orientation is correct because the cable has different pins.

Note: Never ever force push if you feel any resistance. The pin is easily bent.



- 8) Tight screws (finger-tight is enough) and connect the power cable.
- 9) Close the sputter head cover.
- 8. Click **Pump** to vacate the chamber.
- 9. Turn on the small fan to cool the sputter head if you are coating more than 50nm thick film.
- 10. Set up program.



1) Click the **Sputtering Open** , you will reach the sputter program shown below.

sure: mbar	Sputtering			Nickel	
mp	Characteristic	s		Source:	right
ndby	Material	Current [mA]	Ar [mbar]	WD [mm]	Tilt เๆ
	Ni	70	2.0E-2	99	19

- 2) Click the top-right block **Nickel** to choose the material you want to coat. Here we take Ni as an example.
- 3) Click on Characteristics window

to modify program.

Name	Src.	Mat.	Method TH/TI, CUR, PRE	Stage WD, SH, TI, ROT	Vacuum [mbar] BA, WO, VT, PU
Gold	L	Au	5.0nm, 30mA, no	96mm, 1mm, - 10°, 3	1.0E-4, 5.0E-2, no, 1x
Chrome	L	Cr	4.0nm, 110mA, no	50mm, 3mm, 0°, 5	6.0E-5, 8.0E-3, no, 1x
Nickel	R	Ni	1.0nm, 70mA, no	99mm, 1mm, 19°, 1	2.0E-5, 2.0E-2, no, 1x
Aluminum	R	AI	9.0nm, 100mA, no	50mm, 3mm, 0°, 3	2.0E-5, 1.0E-2, no, 1x
Cobalt	R	Co	4.0nm, 100mA, no	50mm, 3mm, 0°, 3	2.0E-5, 1.0E-2, no, 1x

- 4) **Src.** Click R (or L) to set sputter head you are going to use (R: right sputter head, L: left sputter head).
- 5) Mat. Double check the Material.
- 6) Method. Highlight Method column, set up the deposition Thickness and Current; check the following table for the best Current value. Click Save when done.



Parameter Suggestions for ACE 600

Material	Current (mA)	Presputtering (Sec)	WD (mm)	Rotation (rpm)	Sputter Vacuum	Base vacuum
					(mbar)	(mbar)
Au	30	-	99	1	5x10-2	5x10-5
Au/Pd	30	-	99	1	5x10-2	5x10-5
Pt	35	-	99	1	5x10-2	5x10-5
Pt/Pd	35	-	99	1	5x10-2	5x10-5
Ag	35	30	99	1	4x10-2	5x10-5
Cr	110	120	99	1	8x10-3	5x10-5
W	90	60	99	1	8x10-3	5x10-5
Ir	15~30	-	99	1	8x10-3	5x10-5
Al	100	60	99	1	1x10-2	5x10-5
Ti	100	60	99	1	1x10-3	5x10-5
Md	90	60	99	1	8x10-3	5x10-5
Ni	100	60	99	1	2x10-2	5x10-5
Cu	60	30	99	1	2x10-2	5x10-5
Со	100	60	99	1	2x10-2	5x10-5

			Recipe of I	r or Au fo	r SEM Ima	ging		
Material	Current (mA)	Thickness (nm)	Pre sputtering (Sec)	WD (mm)	Rotation (rpm)	Tilt	Sputter Vacuum (mbar)	Base vacuum (mbar)
Ir	15	2~3	-	99	1	25	8x10-3	5x10-5
Au	30	2~3	-	99	1	-25	5x10-2	5x10-5

Recipe of Au or other metals for more than 50nm thickness sputtering											
Current (mA)	Thickness (nm)	Pre sputtering (Sec)	WD (mm)	Rotation (rpm)	Tilt	Sputter Vacuum (mbar)	Base vacuum (mbar)				
30~50	> 50	-	50~65	1	-25	5x10-2	5x10-5				
100	> 50	60	50~65	1	25	2x10-2	5x10-5				
	Current (mA) 30~50	Current (mA)Thickness (nm)30~50> 50	Current (mA)Thickness (nm)Pre sputtering (Sec)30~50> 50-	Current (mA)Thickness (nm)Pre sputtering (Sec)WD (mm)30~50> 50-50~65	Current (mA)Thickness (nm)Pre sputtering 	$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	$ \begin{array}{c c c c c c c c c c c c c c c c c c c $				

Please use small working distance (50 ~65mm) if sputtering more than 50 nm.

7) **Stage.** Click Stage column.

• The Maximum Working distance could be 99 mm (the largest value when sample height is 1 mm);

If you sputter more than 50nm, please set **Working distance** to around 60mm. Otherwise, your sputtering process will be terminated due to sputter head over temperature.

- Tilt angle for Right sputter head is 25° if directly facing target is needed;
- Tilt angle for Left sputter head is -25° if directly facing target is needed;

• **Rotation** speed usually is 1. If you have large samples, put 0 as rotation speed. Click **Test** to check your parameter and then click **Init** to recover. Save if you satisfy.

Edit stage - N	ickel			
	source Wo	rking distance	Sample he	ight
sample	d 🗖	99.0 +	- 1.0	+
stage 9	s 1	Tilt	Rotation	
		19° +	Type Speed / Action	+
	Automatic			
Stage: Test	Init	Save	Cancel	×

8) Vacuum. Set up vacuum by clicking Vacuum.

Check the above table for the best **Sputter vacuum** and **Base vacuum**. For Base vacuum, 1.0E-5 to 5.0E-5 is good.

Note: The better vacuum, the longer waiting time. Click **Save**.



9) Click **Back** when all parameters are set up.

Name	Src.	Mat.	Method TH/TI, CUR, PRE	Stage WD, SH, TI, ROT	Vacuum [mbar] BA, WO, VT, PU
Gold	L	Au	5.0nm, 30mA, no	96mm, 1mm, - 10°, 3	1.0E-4, 5.0E-2, no, 1x
Chrome	L	Cr	4.0nm, 110mA, no	50mm, 3mm, 0°, 5	6.0E-5, 8.0E-3, no, 1x
Nickel	R	Ni	1.0nm, 70mA, no	99mm, 1mm, 19°, 1	2.0E-5, 2.0E-2, no, 1x
Aluminum	R	AI	9.0nm, 100mA, no	50mm, 3mm, 0°, 3	2.0E-5, 1.0E-2, no, 1x
Cobalt	R	Co	4.0nm, 100mA, no	50mm, 3mm, 0°, 3	2.0E-5, 1.0E-2, no, 1x

Start

11. Click only after the Pressure reaches to 5E-5 mbar.
 Warning: You have to wait until the desired vacuum level, otherwise the error message "Quartz unstable" may show.

Please see trouble shoot if you see any error.

Pressure:	Sputtering		C	Nickel				E1303	
6.7E-7 mbar						GUERCERN	Process	E1303	
Pump	Characteristic	s		Source	right	process	Process terminated Quartz unstable.		
Standby	Material	Current [mA]	Ar [mbar]	WD (mm)	Tilt m	Full speed	Change quartz to guarantee valid measurement		
Vent	Ni	70	2.0E-2	99	19	Torspers			
								Close	
	Thickn	ess:	1.0nm	+		-	Stotus Falled Time alapsed 00.04	Rate: n/a Thoras n/a	

12. Watch for sputtering. Make sure the Ar gas flow is around 7.5 psi.

• There are several steps before sputtering, including Wait for pre-proc. Vacuum, Wait for control-speed.

	E600			Leica EM AC	E600		
Pressure: 1.2E-4 mbar Vent after process	Gold	Wait for pre-proc. vac (< 5.0E-5 mBar)	uum	Pressure: 9.8E-3 mbar Vent after process	Gold	Wait for control-speed (78600rpm)	
Full speed	Frepare Second	Gold Sputter (left)		Full speed	Prepare Vacuum	Gold Sputter (left)	
	Status: Idle Time elapsed: 06:22	Rate: n/a Thickness: n/a			Status: Idle Time elapsed: 12:24	Rate; n/a Thickness: n/a	
Main 6	Menu 🔳 Light	Stop	17:28 2019-07-02	Main (A)	Menu 📄 Light	Stop	17:34 2019-07-02

- 13. **Sputtering**. You can check the rate and thickness.
 - The final thickness will be shown when it is done, as shown in the following figure.
 - If the thickness is less than your expectation and sputtering is terminated, the sputtering rate may be small, increase **Current** and change thickness to sputter again.
- 14. Click **Init** when it is done to recover the stage. Then click **Close**.

Leica EM AC	CE600		Lei	EM ACE600				
5.5E-2 mbar Vent after process	Gold	Sputtering	Pres 1.5E Ve	Gold - Result Status: Finished Total time: 00:16:02		Pump:	Pumping	
Full speed	Préciara Vacuara	Gold Spotter (Wr)	Fu	Material: Film thickness: Sputter rate: Source temp.:	Gold 3.11nm 0.03nm/s 31 °C / 31 °C			
-	Proces Timesous 14:08 (United	00.21) Thickness: 0.3nm (3.0nm	17:36 2019-07-02	Log file: Export Menu		nit St	Close	× 17:38 2019-07-02

- 15. Click Vent.
- 16. Open the chamber until the pressure reaches 8E+2 mbar.
- 17. Take your samples out. Clean the stages.
- 18. Lock the window and Click **Pump**.

The chamber must be left in vacuum state when you are done.

- 19. Close N2 valve.
- 20. Logout FOM.
- 21. Sign in Log book.

Please write down the final target information of each head in logbook **Comments**. This is very important, because a lot of targets have similar color.

Contact Lei Wang at (203) 745 8460 if needed.

Trouble shoot:

1. If you see the **motor error**, please click the error to remove it.

2. Sputter head **over-temperature**. Please wait 30 min to cool down the head. If you are working on thick sputtering (> 50nm), please

(1) Turn on the small fan to cool the head.

(2) Change Working distance to $80 \sim 50$ mm to increase the sputtering rate.



3. **Failed**. The film thickness is less than setup.

(1) Check the **sputtering rate**, if sputtering rate is too low, ~0.01 A/Sec or 0, the quartz may not detect the metal sputtering and will stop after a while.

(2) Increase the **Current** and sputter again.

-	Silver - Result				_
4.0E Vs pr	Status: Failed Total time: 00:17:54 Material: Film thickness: Sputter rate: Source temp.:	Silver 5.39nm 0.02nm/s 31 °C / 33 °C	Pump:	Pumpin'g	2
	Log file: Export	Stage:	Init	Close	×

4. Quartz missing error.



- (1) Click Menu.
- (2) Click Quartz.
- (3) Click Start.
- (4) Check whether showing Status: OK.
- (5) If it shows Status: Change, Click Stop and then Start to test again.

Note: if test results have several **Status: Change**, it may need new quartz replacement, please let manager know. Sometime the quartz shows

- (6) Click **Stop**.
- (7) Start sputtering again.



- 5. Process terminated. Ignition fault.
- (1) Check agron flow **pressure**, it should be ~7.5 psi.

(2) Check the sputter head and connection cable. The pin must be straight. Never force push cable when reconecting the cable.

