Washington State University Vancouver Cleanroom

Standard Operating Procedure

Equipment Name:	**Nano 36 Sputter**		
Scheduling Name:	Nano 36 Sputter	**Revision Number:**	0.64
Model:	Nano 36	**Revised by:**	Sam Judd (360)546-9201
Location:	VECS046, Bay 3	**Date:**	5/11/2023

The Nano 6 sputters dialectic and non-magnetic metals on wafers or other substrates.

Our KJ Lesker Nano36 system can sputter a wide range of materials using RF power. The chamber has a typical pump down pressure of 2.0x10-7 Torr. Our sample holder supports pieces up to 8" diameter.

BACKGROUND gleaned from Torus CD and training March 12, 2013

When the source has been properly installed, equipped with a target (see Target Installation) and connected to the power and water supplies, it is ready for operation.

New targets or those exposed to atmosphere for prolonged periods should first be cleaned by pre-sputtering (see Target Pre-Sputtering).

Sputtering

1) Pump down the vacuum chamber to high vacuum. The choice of base pressures is left to the user and determined by acceptable impurity levels.

2) Set the gas pressure by adjusting the argon gas inlet and throttle controllers.
Typical gas pressure should be between 1-15 mTorr.

3) Turn on the cooling-water supply. Nano 36 should always have cooling water on with chamber under vacuum (turbo pump running).

RF Operation

1) Turn output on to a nominal value of 50 watts forward power.

2) The matching network (tuner) should match the load (sputtering source) to the generated power and there should be low reflected power.

3) Check for the presence of a plasma, if there is no plasma, open and close the shutter or raise the pressure to 50mTorr of argon in the process chamber.

4) If still no plasma, briefly go to "manual" tuning on the matching network and return to "auto" tuning.

   1) If reflected power is high, press [Man] on both LOAD & TUNE in the MC2 keyboard.
   2) Press MIN or MAX to move TUN & LOAD to middle of range (~50%).
   3) Return to Auto mode for both TUNE & LOAD.

5) If there still is not plasma, turn off the power supply and contact a Kurt J. Lesker Company Applications Engineer for further advice.

6) If plasma has been established, slowly bring the pressure down to the working pressure.

7) Adjust power to the desired output level.

8) If the sputtering source is being powered through a cable connected from the matching network, monitor the temperature of this cable and the connectors for excessive heating.

CAUTION Do not touch cable while running source.

Target Pre-Sputtering

New targets or targets kept in oxygen environments should be pre-sputtered to remove oxides, contaminants, etc. from their surfaces.

During pre-sputter, the substrate should be shielded from the source. Close the shutter to avoid cross contamination when co-depositing targets.

Multiple arcs may be generated on the target surface during pre- sputtering. Generally, these are relatively low energy arcs that are random in nature and are not sustained. As the target surface is cleaned during pre-sputtering the arcs will decrease in number and the plasma will become very stable. During this time, the target voltage will generally slowly increase and eventually lock in at a value that was higher than the initial run voltage. The absence of arcs and the stability of the voltage (voltage will tend to slowly decrease from this stabilized value as the target is eroded away) is a good indication that the target has been sufficiently cleaned to start sputtering.

To pre-sputter a target:

1) Refer to the Sputtering section above for ignition of the source.

2) Adjust the source to the proper operating conditions for your specific application.

3) Let the target “burn-in” for 5-10 minutes, or until sputtering parameters have stabilized, then remove the shield from the substrate to start deposition.

Practical Sputtering Considerations

Vacuum Integrity during Deposition

A clean vacuum environment is a must for state of art thin film deposition. Sources of contamination of the chamber are various. HV and UHV compatible materials/structures will significantly enhance the vacuum integrity. For example, carbon steel and aluminum exhibits much higher vapor pressure than stainless steel and copper under vacuum. Outgassing of O-rings is also one of the main contamination sources. From the viewpoint of structures, any semi-enclosed volume within the vacuum environment, such as the bottom of tapped holes and contact space between the target and cathode, will form a virtual leak. The Kurt J. Lesker Company uses only stainless steel and copper for the cathodes and anodes of TORUS® sources. They also vent all tapped holes via either cross drill or vented screws. Only three Viton o-rings have been used to reduce the o-ring outgassing to a minimum. It is recommended to use the same practices for all fixtures, substrate holders/manipulators, etc. Call the Kurt J Lesker Company for assistance regarding HV and UHV components.

Deposition Rate

Deposition rates are affected primarily by the sputtering power, source-to-substrate distance, sputter gas pressure, and target material type. Secondarily, the sputtering rate is influenced by the thermal conductivity through the target system as well as the fragility of the target. All of these factors must be considered when trying to establish a rate for a given target material. In general, good thermally conducting, metal targets can run up to a power density of 150 watts/in^2 (target surface area) for indirectly cooled targets and 300 watts/in^2 for directly cooled targets. Poor thermally conducting targets and fragile targets may be limited to 20 to 50 watts/in^2. Our 2” targets have about 3.14 in^2.

Nano 36 Procedures Sam Judd (360)546-9201

Deposition rates are inversely proportional to the square of the source-to-substrate distances. The further the target is from the substrate, the lower the sputtering rates will be. So, rate can be increased by decreasing the throw distance.

Deposition rates are also inversely proportional to the sputtering gas pressure (usually). As the gas pressure goes up, the number of collisions between a gas atom and a target atom increases, this scatters the sputtered atoms more. This means that fewer target atoms arrive at the substrate at higher sputtering pressures.

Each material has its own sputter yield and therefore its own sputtering rate (see Deposition Rate Comparison Tables for some typical values). The higher the sputter yield for the given material, the higher the deposition rates obtainable for that material. Therefore, the sputtering rate is dependent on the intrinsic Sputter Yield value for that material. The sputtering rate will be different if you use the same target in the other Torus

Note: Press [ACK] to acknowledge any warning messages in the top of the touch panel display if system seems unresponsive.

WSUV Procedures:

Setup Procedure

1) Nano36 system is always left in high vacuum to keep sources dry and contamination free.
2) Vent vacuum chamber
   a. [Recipe] → [Vent]
   b. Venting takes about 30 minutes to reduce Turbo pump speed to 50% max RPMs.
   c. Can open chamber when venting recipe is complete. There are 10 steps to the VENT recipe.
   d. Press [Done] when recipe is complete.
3) Change Source Target if necessary
   a. Make sure to turn off power supplies. (R301 and MC2)
   b. Loosen the three screws around side of Torus cap.
   c. Open source shutter with LCD control panel.
   d. Loosen or remove four screws in source hold down ring.
   e. Remove source target and store in proper labeled container.
   f. Install new source target and record change in log.
   g. Use dark space spacer if target is 0.25" thick. Remove spacer if new target is 0.125" thick. (add photo)
   h. Tighten screws in hold down ring.
   i. Return Torus cap and tighten the three screws.
   j. Dark space should be 2mm. Change Torus cap slot to get close to 2mm as possible.
   k. Pre-sputter target to remove contamination after storage. See more information above.
4) Install new sample
   a. Remove sample plate from spindle at roof of vacuum chamber.
      i. Align plate spindle slot toward back of chamber.
      ii. Lift plate off spindle pins.
      iii. Move plate toward you and tip close edge down.
      iv. Flip plate over to load new sample.
      v. Hold sample with clips. Adjust clip placement as needed.
   b. Return sample plate to spindle.

Figure 1 Vacuum chamber with right Torus cap off. (Also w/o required chamber liner)

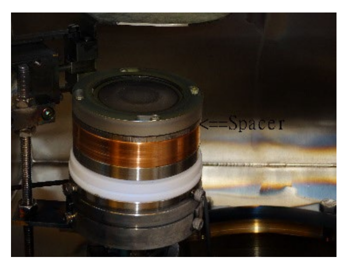


Figure 2 Torus with 0.25" target & spacer w/o cap.

Sputter Procedure

1) Put sample in baseline vacuum condition.
    a. Install new transparency sheet to door window. This protects the window from coating during sputtering process.
    b. Close door
    c. [Recipe] → [Pump Down]
    d. This will finish at about E-5 torr but it is necessary to wait for desired working pressure.
    e. This may take hours. This is necessary to remove moisture and contaminates from chamber. There are 20 steps to the Pump Down recipe. Press [DONE] when complete.
2) Start plasma
    a. Set vac turbo pump speed to 25% to start plasma. (Leave at this speed or use the speed used on Lesker form for selected target.)
        i. At about 90% turbo pump speed vent gas to slow pump.
            1. Turn Gas Line Selector to Ar gas (near top of black panel on right)
            2. [Gas inj] →Ar[CTRL] →Capman SETP→0.2mTorr
            3. This will slow the Turbo Pump down to speed much more quickly.
    b. Turn Gas switch above black power supplies to left to activate Ar gas MFC.
    c. Set Gas (Ar) to 15 mTorr after pump slows down to 25% speed. (This high pressure is used to start plasma.)
    d. [Gas] → Gas Inj On (square valve On) → Ar[CTRL] → Capman SETP →15 mTorr
    e. Turn on R301 and MC2 black box power supply controllers.
    f. Rotate sample. [SUBST] → [MOTOR ROTATE] → [SPEED] (Between 1 and 100).
    g. Open the crystal. [XTAL] button will show green when open.
    h. Set power to 50 Watts on Torus **2**. [DEP] →[SRC SW**2**] → [PWS1 ON]→ [SETPT] 50 [ENTER]
    i. MC2 Load and Tune should be in Auto mode.
    j. Toggle shutter if necessary to excite the plasma.
    k. Adjust plasma conditions for clean sputtering.
        i. Reduce Ar pressure to 3 mTorr (for Al, from Lesker form).
            1. [Gas] →Capman SETP→3mTorr
        ii. Set power ramp to 2. [Dep] screen
        iii. Increase power to 250 Watts for Al
            1. or up to max power listed on Lesker form or published tables for your target.
        iv. Run plasma with shutter closed until stable.
        v. Set Film Thickness on SQM-160 controller.
            1. Program 3 (use program that is set for the target density in use)
            2. Verify program density matches target density, adjust if necessary.
            3. Next to FINL THK
            4. Use knob to dial in thickness in kA.
    l. Open Source shutter to start coating wafer.
            1. [Shutter] on SQM-160 to open shutter and start deposition. (May need more revision)
            2. Note rate and calculate end time.
            3. Be prepared to end early.
    m. Close Source shutter when desired film thickness is reached if SQM-160 did not close it.
    n. Go to Exit Procedure or Setup Procedure to store or remove sample.
    o. Measure film thickness and adjust Tooling Factor if it does not match expected thickness.

Sample Removal Procedure

Nano 36 Procedures

Sam Judd (360)546-9201

1) Ramp power down to 50 watts to protect target from thermal shock. Then down to 0 watts.
2) Power off: [PWS1] to gray
3) Turn off R301 and MC2 controllers on the right.
4) Turn off gas: [Gas] →[Gas Inj] to gray
5) Turn off GAS LINE SELECTOR SWITCH above the R301 & MC2 controllers.

Exit Procedure

1) [DEP]
2) Switch off power [PWS1/off]
3) Deselect [SRC/SW1] source 1
4) Turn off both black box power buttons
5) Turn off platen motor. [SUBST]→ [MOTOR ROTATE]→ to gray off state.
6) Turn off gas line selector switch above power supplies.
7) [GAS], [CTRL] to turn off gas control
8) deselect Gas injection valve
9) 

to 
10) * Use these steps to leave chamber at ambient pressure *
11) [Recipe] → [VENT]

12) * The next steps are only required if leaving chamber under vacuum *
13) Set point to zero
14) [VAC] turbo full speed (100%)
15) This is the state for keeping targets dry

Note: Turn switch near EMO to START to start or restart the system after an EMO event.
An EMO event will show an **Emergency Stop** warning. Press [ACK] to acknowledge and continue.

Appendix A

Nano 36 2.00" diameter Targets

Material	Purity	Part#	Density	Z-Ratio	Ramp (W/sec)	Power (Watts)
Aluminum, 1/8"	99.9900	EJTALXX402A2	2.70	1.080		250
Aluminum, ¼"	99.9900	EJTALXX402A4	2.70	1.080		250
Cobalt, 1/8" (Dr. Sekhar)	99.95	EJTCOXX352A2	8.9	0.343		240
Copper	99.99	EJTCUXX402A4	8.92	0.44		150
Nickel, 1/8" (Dr. Zhao)	99.99	EJTNIXX402A2	8.91	0.331		250
Silicon Dioxide, ¼"	99.995	EJTSIO2452A4	2.65	1.000		250
Silicon Dioxide, 1/8"	99.995	EJTSIO2452A2	2.65	1.000		250
Silicon Nitride, bonded	99.90	EJTSINX302A2	3.44	*1.000	0.2	60
Silicon, un-doped	99.999	EJTSIXX502A2	2.32	0.712		60
Silver, bonded			10.5	0.529		60
Titanium	99.995	EJTTIXX452A4	4.5	0.628		250
Vanadium (Dr. Sekhar)	99.500	EJTVXXX252A4	5.69	0.530		60
Yttrium Oxide, bonded	99.9900	EJTYOXX402A2	5.01	*1.000	0.2	72
Zinc	99.99	EJTZNXX402A4	7.14	0.514	1	100
Zirconium Oxide/Ytia		EJT???XX402A2	5.6?	*1.000	0.2	60

An * is used to indicate that the Z-Ratio has not been established for the material. Default value is 1.0.

See Kurt J. Lesker Company | Sputtering Targets | Enabling Technology for a Better World for target information.

Deposition Rate Comparison

Material	Name	Rate	Material	Name	Rate
Al2O3	Alumina	0.05	Ag	Silver	2.88
SiC	Silicon Carbide	0.22	Al	**Aluminum**	**1.00**
SiO2	Silicon Dioxide	0.21	Au	Gold	1.74
TaC	Tantalum Carbide	0.09	Be	Beryllium	0.21
Ta2O5	Tantalum Pentoxide	0.39	C	Carbon	0.23
Ti	Titanium	0.53	Cu	Copper	1.42
Zn	Zinc	0.3	Co	Cobalt	

Appendix B

Ramp Procedure for Ceramic Target Conditioning

The ceramic target may have a low thermal conductivity making it susceptible to thermal shock and high operating temperature. Use ramp-up and ramp-down procedures when applying or reducing power.

1. Attempting to ignite plasma at low power may require a high argon gas pressure.
   a) Typically, 5E-2 Torr is required for low power ignition.
   b) Once the plasma has stabilized, the pressure can be reduced to that normally used for sputtering the particular target material.
2. Power to the target should be ramped up slowly through the break-in period.
   a) The ramp should be limited to 10 - 20 watts per minute.
   b) If the target material's properties and its response to the application of power are in doubt, choose the lower power ramp value.
   c) If arcing, voltage spikes, or other plasma irregularities occur during ramp up, **stop ramping power** and allow the plasma to stabilize before starting the ramp again.
3. Since the deposition rate increases as the power density increases, the natural tendency is to use high power to speed up deposition. However, each particular target mounted in a particular sputter source will have its own heat transfer characteristics. Applying a higher power density than is acceptable results in the target overheating and subsequently fracturing and/or bond failure (if the target is bonded).
4. When the deposition run is complete, it is just as important to ramp-down power at the same rate as the ramp-up, allowing the target to cool slowly to avoid thermal shock and the potential for target fracture.
   a) As an added precaution, allow the chamber to cool slowly to ambient temperature before venting.
5. If you have questions you can talk to the technician or technical support at techinfo@lesker.com.

Appendix C

Troubleshooting Plasma

Try these steps if the plasma does not ignite right away:

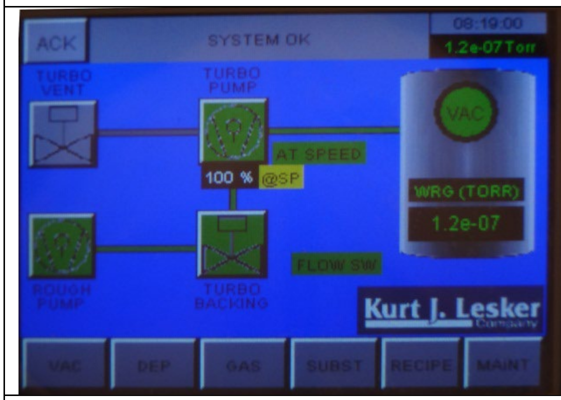
4) Make sure LOAD & TUNE are in Auto mode.
5) Make sure the TURBO PUMP speed is at 25% in the VACuum screen.
6) If reflected power is high press [Man] on both LOAD & TUNE in the MC2 keyboard.
7) Press MIN or MAX to move TUN & LOAD to middle of range (~50%).
8) Return to Auto mode for both TUNE & LOAD.
9) Toggle the shutter
10) Should have a plasma
11) If still no plasma, try increasing Ar gas pressure step wise up to 50 m Torr repeating the steps above with each pressure increase.
12) If still no plasma, try increasing the power up to 50% more and repeat steps 1 to 6.

Figure 3 Controls including R301 & MC2 power control and SQM-160 film thickness monitor.

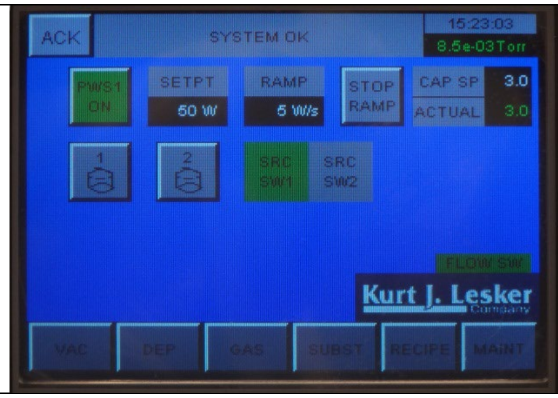
13) If still no plasma, check the dark space for 2 mm gap. Gap can be a little more but not much less.
14) Last, call tech to check for shorts in Torus gun system.

Appendix D

VACuum screen



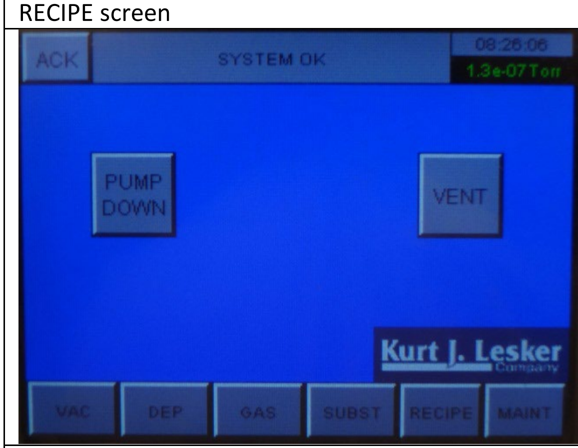
DEP screen



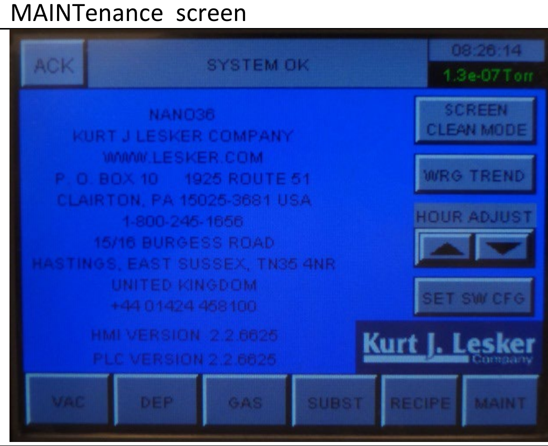
GAS screen

SUBSTrate screen

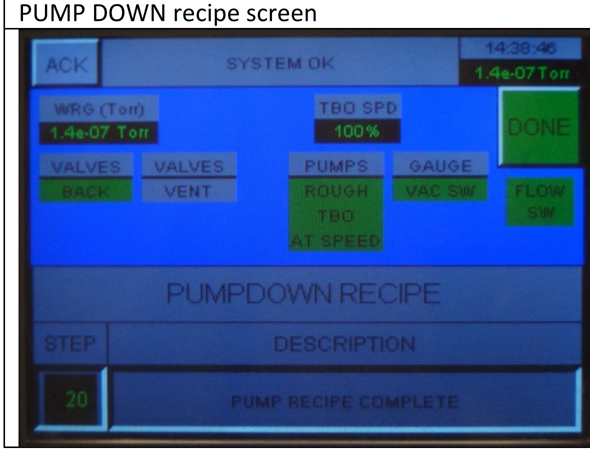
RECIPE screen



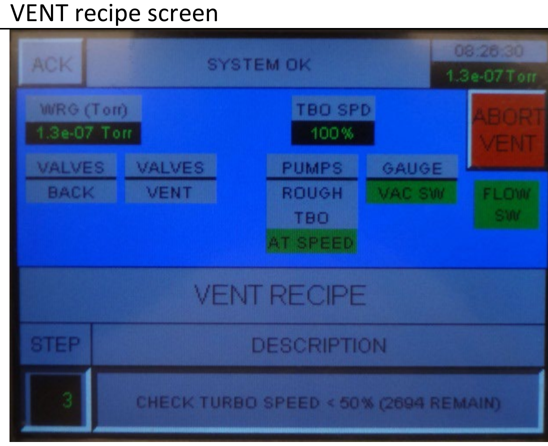
MAINTenance screen



PUMP DOWN recipe screen



VENT recipe screen



Appendix E

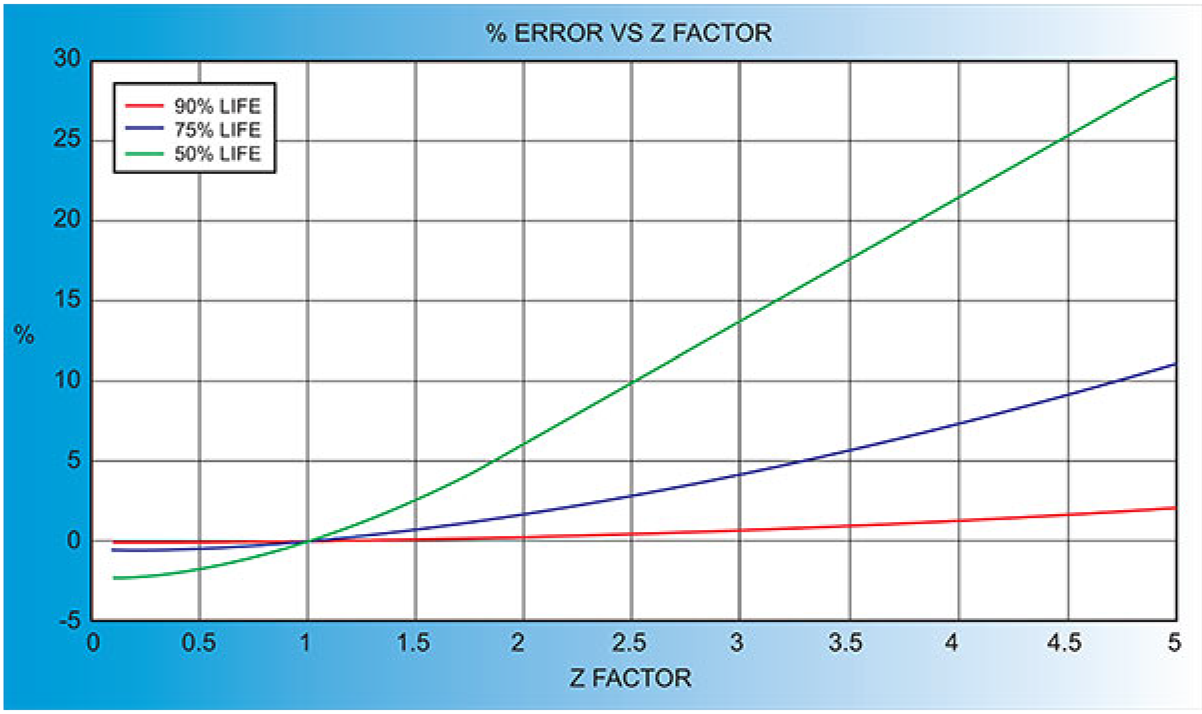
Z-Factors

Empirical Determination of Z-Factor

Unfortunately, Z Factor and Shear Modulus are not readily available for many materials. In this case, the Z-Factor can also be determined empirically using the following method:

- Deposit material until Crystal Life is near 50%, or near the end of life, whichever is sooner.
- Place a new substrate adjacent to the used quartz sensor.
- Set QCM Density to the calibrated value; Tooling to 100%
- Zero thickness
- Deposit approximately 1000 to 5000 A of material on the substrate.
- Use a profilometer or interferometer to measure the actual substrate film thickness.
- Adjust the Z Factor of the instrument until the correct thickness reading is shown.

Another alternative is to change crystals frequently and ignore the error. The graph below shows the % Error in Rate/Thickness from using the wrong Z Factor. For a crystal with 90% life, the error is negligible for even large errors in the programmed versus actual Z Factor.



From Kurt J. Lesker:
https://www.lesker.com/newweb/deposition_materials/depositionmaterials_sputtertargets_1.cfm?pgid=zr5#

Nano 36 Sputtering

Washington State University Vancouver

User Name:	**Last 4 of WSU ID#:**

Item No.	**Item Description**	**Trainer Initial**	**Trainee Initial**	**Date**
1	Know location of operating procedure, manual and logbook			
2	Identify components of the instrument a) Touch panel controller b) On/Off switches for power supplies and gas c) Targets and Torus guns d) Vacuum control and logging e) Shutdown EMO			
3	Understand safety aspects (temperature, vacuum, RF)			
4	Demonstrate ability start up the Nano36			
5	Demonstrate ability to properly load samples			
6	Demonstrate ability to load/replenish source material using the correct gun locations			
7	Demonstrate the ability to create or revise a desired process including use of Film Thickness Monitor (Remember density for each target material)			
8	Demonstrate ability to run a deposition process, including adjusting ramps and power limits			
9	Demonstrate ability to enter run information on the sputtering run sheet in the logbook			
10	Demonstrate ability to unload samples and source material (target)			
11	Demonstrate ability to properly clean the system after a deposition run (new window cover and foil)			
12	Demonstrate ability to shut down the Nano36 and leave it in the appropriate safe state for the next user			

Certification completed:

I have received the above training.	
Signature of trainee	**Date**
I have instructed the lab user on the items listed above.	
Signature of trainer	**Date**

Nano 36 Sputtering Machine Log Sheet

Master User: Praveen Sekhar, 6-9186 **System Type: Kurt J. Lesker Nano36 /**Source Type: Torus 2 (Std Magnets) **/ Power Supply: R301 (RF)**

Technician in Charge: Sam Judd, 6-9201 **Primary Gas: Argon (note if O_2)** For best results, record Final & Measured thickness to calculate Tooling Factor.

Username	User Phone Number	Date 2023	Base Press (Torr)	Source (1 or 2)	Material/ Density	Power (W)	Working Pressure (mTorr)	Did you change the target? (Y/N)	Deposition Start time