

# Denton Evaporator Operation Manual

## University of Notre Dame



For training contact Dave Heemstra

# General Precautions

## Contacts

For problems, clarification of procedures, or general information pertaining to this machine please contact one of the following personnel.

<b>Mark Richmond</b>	<b>631-6478</b>	<a href="mailto:mrichmon@nd.edu"><u>mrichmon@nd.edu</u></a>
<b>Mike Thomas</b>	<b>631-7493</b>	<a href="mailto:mthomas8@nd.edu"><u>mthomas8@nd.edu</u></a>
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**In Case of Emergency, Please Contact Notre Dame Security at**

**911**

**Metal Verification is MANDATORY** – Failure to verify >50% metal in the crucible can result in serious equipment damage.

**Alignment and Sweeping of the Electron beam is MANDATORY.** Failure to sweep the beam can cause damage to the machine and cause you to lose your privileges to use this machine.

**Users must constantly be watching the metal source, deposition controller and emission current when the e-beam is on.**

If any step does not respond as it should, stop and put the machine into standby if you can and contact help. If unsure on how to get to standby mode safely, leave the machine as it is and contact above personnel.

YOU MUST VERIFY THERE IS ENOUGH METAL EVERYTIME YOU PERFORM A DEPOSITION. DO NOT PROCEED UNLESS THE CRUCIBLE IS FILLED MORE THAN 50%

### **Metal Verification:**

- 1 – Place System in “manual” mode
- 2 – Select “E-beam Status and Control” in upper right hand corner
- 3 – Select pocket number and press “Enter”
- 4 – Verify sufficient metal
- 5 – Place system back into “Automatic” mode

**!!!!!!!YOU MUST VERIFY ALL METALS – EVERY TIME!!!!!!!**

### **Standby Conditions:**

(These conditions allow for the safest and best conditions for the machine, your safety, and the best results for your research)

1. Telemark TT-8 Controller:
  - a. Key in OFF Position
  - b. Remote-Loc-Hand Switch in REMOTE Position
  - c. Bias switch in ON Position
2. Telemark XY Sweep Controller:
  - a. Control – Sweep Select switch in CONTROL position
  - b. Power switch in ON Position
  - c. Joystick in Midpoint
  - d. Spiral-Triangle-Manual switch in MANUAL
  - e. Manual – Auto switch in MANUAL
  - f. Sweep Select (1 2 3 4) switch set to position 1
  - g. All 4 Channels Mode (Spiral – Manual) switches in Manual
3. Software:
  - a. Left in Overview screen
  - b. System control in AUTO
  - c. Under E-Beam Control Screen (must be in manual operation mode to verify)
    - i. Control Mode #1 in XTC ON (green)
4. Chamber doors (upper and lower) closed and under High Vacuum.

## Software Overview:

There are 6 main software screens used by the ProcessPro Software package

1. Login:
  - a. ProcessPro logo in center
  - b. Startup screen upon computer power up
  - c. No Login required
2. Overview:
  - a. This screen comprises of the system layout and valve diagraming
  - b. Displays most process parameters
3. Recipe
  - a. Displays recipe tree/hierarchy
  - b. Used to create auto recipes with multiple process steps
    - i. Pressure
    - ii. Gas Flow
    - iii. Temperature
    - iv. Crystal Termination
4. Alarm View
  - a. Allows acknowledgement of system errors
5. Alarm Recover
  - a. Shows list of recovered alarms
6. Data Logger
  - a. Opens MS Access

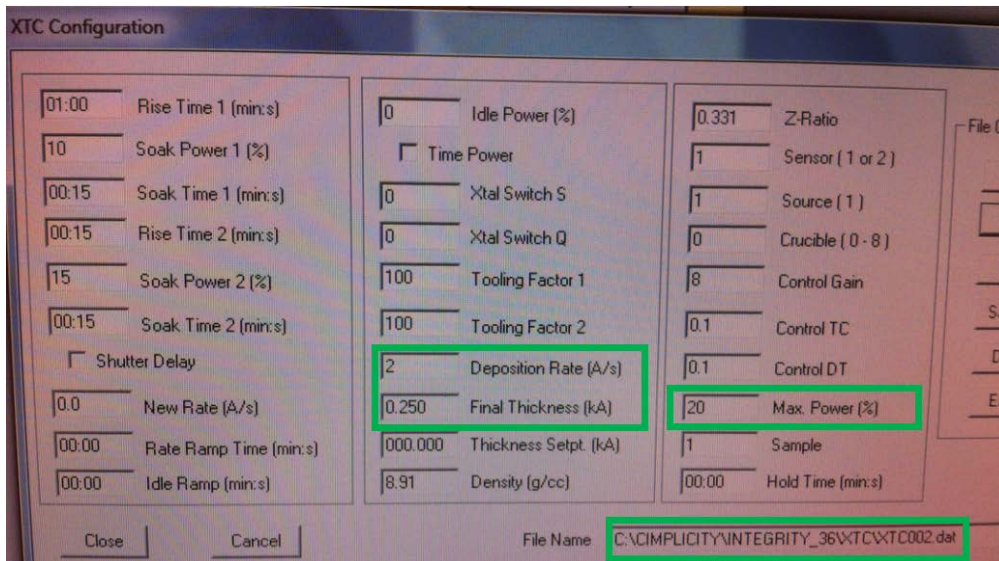
## Opening System and Loading a Sample:

1. Verify the system is in “Auto” mode on the Overview Screen
2. Select “recipe” button at the bottom of the software screen
3. Select “Edit/Create” under “Master Recipe”
4. Select “Open” under File Options
5. Select Recipe “AutoVent.dat” and click Open
  - a. File should be listed in the master sequence list
  - b. C:\CIMPLICITY\Integrity\_36\Master\_Recipe\AutoVent.dat should be listed in the Master recipe file name box
6. Select “Download”
  - a. Cryo pump Isolation valve should close at this time
7. Select “Close” to close the master recipe builder window
8. Select Overview screen
9. Select “Start” on the top left of the “Overview” screen
  - a. AutoVent Recipe will start to run
  - b. Chamber manometer should begin to rise
  - c. Door should open after 3 minutes
10. Remove Sample holder and mount sample to it
11. Install sample holder with sample back into the system
12. Close both doors and Latch shut
  - a. Top door needs to be latched
  - b. Bottom door needs to be latched
13. Select the “Recipe” button on the software menu

14. Select “Edit/Create” under Master Recipe Setting
15. Select “Open” under “File Options”
16. Select “AutoPump.dat” and click Open
  - a. AutoPump.dat should be listed in Master Sequence List
  - b. C:\CIMPLICITY\Integrity\_36\Master\_Recipe\AutoPump.dat should be listed in the Master recipe file name box
17. Select “Download”
18. Close the Master Recipe builder window and return to “Overview” screen
19. Select “Start” button on top left of “Overview” screen
  - a. AutoPump recipe should start to run
20. Verify Doors are closed and system pumps down
  - a. Vacuum system looks for <140 mT roughing pump
  - b. Cryo will not open if 140 mT is not achieved within 7 min
21. If Cryo pumping fails
  - a. Verify Top and Bottom Doors are closed
  - b. Restart Pump down routine
  - c. If second pumpdown fails – vent system and check door seals.

## **Editing a Standard Material Deposition Recipe:**

1. Pockets 1 – 6 correspond to XTC00#.dat crystal termination files (Where # equals 1 – 6)
2. Standard use requires the user to determine the pocket they wish to evaporate
  - a. Ex. Pocket 2 – Nickle
3. Go to Recipe Screen
  - a. Select XTC “Edit/Create” and Open the XTC00#.DAT File
  - b. Ex. For pocket 2 – open XTC002.DAT
4. Adjust/Verify the following 3 parameters
  - a. Deposition Rate (A/sec) – The Rate that you want
  - b. Final Thickness (kA) – How much material you want
  - c. Max Power (%)
    - i. Max power the system will put into the sample to achieve the deposition rate desired
    - ii. Currently setup for 2 A/Sec



5. Save the XTC process
  - a. Ex – XTC002.DAT
6. To run the Process
  - a. Select the master recipe
  - b. Select Edit/Create
  - c. Select Open
7. Select the correct file that corresponds to the pocket you want to evaporate
  - a. Source0# where # equals 1 – 6
  - b. Ex. Source02 would evaporate pocket 2 – Nickel
8. Select Download
9. Return to the “Overview” Screen
  - a. Verify Recipe has downloaded
  - b. Verify System is in AUTO Mode
10. Turn the KEY for the HV module to the ON Position



11. Select Start
  - a. Beam will turn on after resetting the controller
  - b. Each source will process through 2 ramp/soak steps
  - c. **USERS MUST VERIFY BEAM POSITION**
  - d. **YOU MUST BE AT THE SYSTEM PAYING ATTENTION TO THE SYSTEM WHEN THE BEAM IS ON!!!!!!**
12. Once Deposition has completed – Wait 10 Minutes for source to cool before venting or depositing next layer. Sources are very large and take longer to cool

# MANUAL MODE OPERATION

## NOT RECOMMENDED

### Beam Positioning and Sweep Setup:

1. Ensure the Key on the Sweep Controller is in the OFF position
2. Enter the overview screen on the computer
3. Change the operation mode of the software from “Auto” to “manual”
  - a. Click on the manual button on the top left of the screen
  - b. Manual button will turn green and “Auto” button will turn gray
4. Click on the Gray “E-Beam Status and Control” Tool Bar
  - a. E-Beam Control Window will open up
5. In the “E-Beam Control” window:
  - a. Make certain the following conditions exist
    - i. All Four “System Enable” Buttons are off – grayed out
      1. If any are green, click on them to turn them off
    - ii. “Control Mode #1” in XTC is Green
  - b. Click on the number next in the “Crucible Control #1” box
    - i. Enter the desired crucible to select the proper material
    - ii. Press the Enter Button
6. On the Sweep Controller – turn power “ON”
7. Select the (Control – Sweep Select) switch to the control position
8. Set the Joystick to the midpoint position
9. Set the (Spiral – Triangle – Manual) switch to the manual position
10. Adjust the joystick so that the position indication LEDs are centered
11. Turn the Key to the ON position
12. On the software, in the E-Beam control Window
  - a. Enable system power by clicking the ON button
  - b. Button Should turn Green
13. Wait 30 seconds
  - a. This allows time for the air interlock switch to close
14. Click on the “Reset” button in the “E-beam Control” window
  - a. A series of LEDs will illuminate on the HV and Emission Control Module
    - i. Air Cab, VAC, Tank, Aux1 under the High Voltage Section
    - ii. HVAC, Water, Aux2 under the Source1 section
  - b. This clears and initializes all hardware interlocks
  - c. In any of these LEDs do not stay lit, the system will not allow the high voltage power supply to energize. Contact lab staff should this occur
15. Enable “High Voltage” power by clicking on the “ON” button next to “HV PWR” in the “E-Beam Control” window
  - a. Button should turn green

- b. LCD should read around -8.5 DC Kilovolts on the “High Voltage Control” module
16. Press the “Clear / Up / Reset” button on the Inficon XTC/2 controller
    - a. This will reset the film thickness monitor
  17. Press the “4 / MPWR” button on the Inficon XTC/2 controller
    - a. This will put the XTC/2 into manual power control
    - b. Ensure that the output power XTC/w reads 0.0%
      - i. If it does not – press the “Reset” button again
      - ii. Then the “MPWR” Button and verify the output reads 0/00%.
    - c. Emission control will be able to be increased by the hand controller at this time
  18. While looking through the chamber viewport – slowly start to increase the emission current until there is a very light beam visible in the crucible.
    - a. If the beam is visible with no increase of emission current power, then do not increase it at this time
      - i. You only need to be able to see the beam at this time
    - b. Keep the emission current very low
    - c. You only want to be able to see the beam at this time
    - d. Do not cause the source to start to get hot enough to evaporate
    - e. Do not exceed 30 mA as read on the “Source 1” LCD
  19. If only one sweep pattern is desired, then all position and beam patterning can be performed using the left control section
    - a. The recipe will disregard any channel selection and default to the settings on the control section
    - b. Typically a single sweep pattern will suffice for similar materials.
    - c. Center the beam on the source by moving the joystick around slowly
    - d. Switch the mode switch (Spiral – Triangle – Manual) to the desired pattern
      - i. Adjust the beam pattern using the lateral and longitudinal controls for the desired sweep pattern
      - ii. The “Mod Amp” is only used for the Spiral mode
        1. It is used to adjust the depth of the spirals collapse.
    - e. The control section is now programmed
  20. If more than one sweep pattern is desired for multiple deposition materials
    - a. Ensure the sweep select sections (Manual – Auto) switch, located on the far right bottom, is in the manual position
    - b. Rotate the (1 -2 -3 -4 Select) knob to the desired channel position
    - c. Switch the desired channels mode switch (Spiral - \_\_\_ - Manual) to “manual”
      - i. This is a 3-position switch
      - ii. The center position is the “Triangle” mode, there is no space for labeling
    - d. Change the (Control – Sweep Select) switch to “Sweep Select”
    - e. The beam position can now be adjusted by using the “LAT” and “LONG” knobs
      - i. Adjust the beam pattern using the lateral and longitudinal controls for the desired sweep pattern
    - f. Switch the mode switch (Spiral – Triangle – Manual) to the desired pattern
      - i. Adjust the beam pattern using the LAT and LONG controls for the desired sweep pattern
      - ii. The “Mod Amp” is only use for the “Spiral” mode
        1. It is used to adjust the depth of the spirals collapse
    - g. If a different material is to be used and a separate channels pattern is desired, the beam must be turned off prior to changing the crucible.
      - i. Press the XTC/2 Handheld button in to turn the beam off



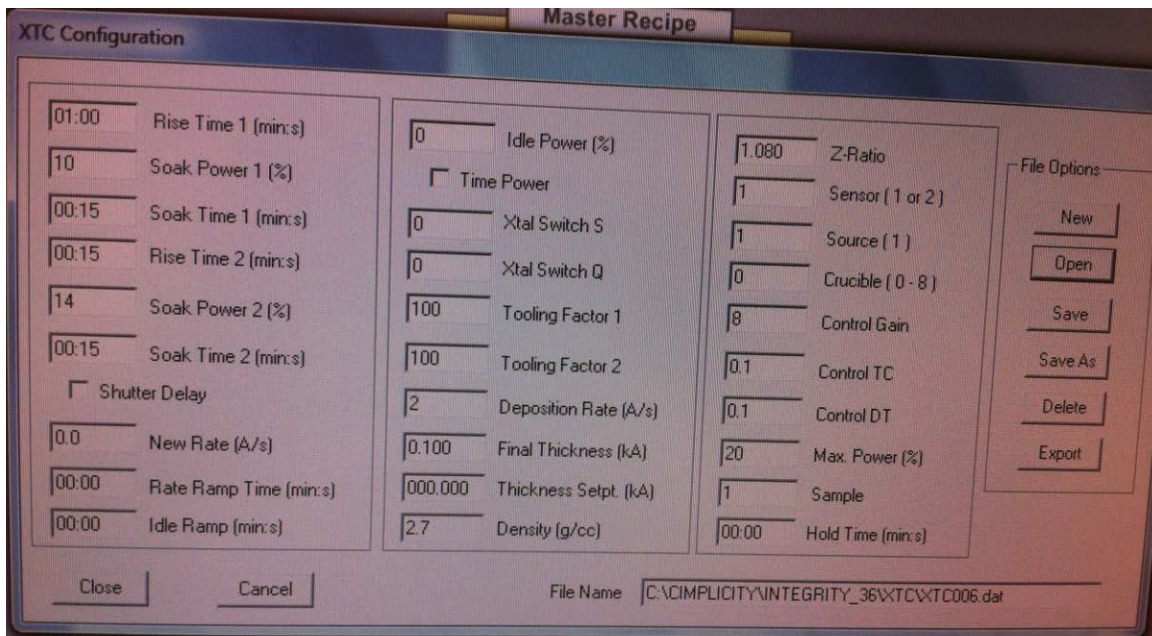
- ii. Press the “Clear/Up/Reset” button on the Inficon XTC/2 Controller
    1. This will reset the XTC/2
  - iii. Press the “4/MPWR” button on the Inficon XTC/2 controller
    1. This will put the XTC/2 into manual power control
    2. Ensure that the output power on the XTC/2 reads 0.0%
    3. If it does not press the “reset” button again followed by the “MPWR” button and verify the output power reads 0.0%
  - iv. In the “E-beam Control” Window
    1. Click on the number next to the “Crucible Control #1” box
    2. Enter the desired crucible to select the proper material
    3. Hit the “Enter” button
      - a. Crucible will rotate to the desired material pocket
  - v. Change the “1 – 2- -3 -4” Select knob to the desired channel and repeat the setup procedure for this channel
21. Turn gun current and high voltage off after beam position and sweep patterning setup is complete
- a. In the “E-Beam Control” Window toggle the “Emission”, “HV Pwr” and “System Power” buttons to OFF
22. Return the system to “Auto” in the “Overview” screen

## FULL RECIPE GENERATION PROCEDURE

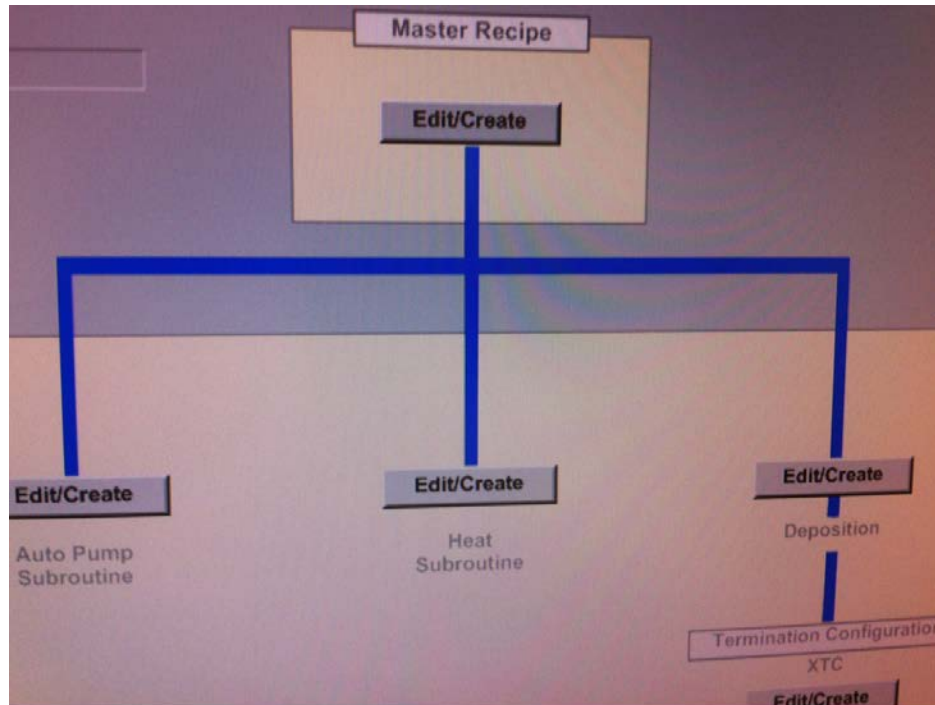
**XTC (Crystal Termination) Recipe Generation: This is ONLY to be used when creating CUSTOM RECIPES. Standard USE does not require users to adjust parameters.**

Note: It is possible to create multiple step depositions using the Denton Software. You may add or remove steps as needed for your research.

1. Enter the Recipe Screen
  - a. Select the button at the bottom labeled "Recipe"
2. To create a XTC Controlled process recipe:
  - a. Click on "Edit/Create" under "Termination Configuration"
    - i. The XTC configuration window will open
  - b. Enter the appropriate process parameters
    - i. Rise Time 1
    - ii. Soak Power 1
    - iii. Rise Time 2
    - iv. Soak Power 2
    - v. Tooling Factor
      1. Set to 100
    - vi. Final Thickness
    - vii. Density (g/cc) – Material Specific
    - viii. Z-Ratio – Material Specific
    - ix. Sensor = 1
    - x. Source = 1
    - xi. Crucible = 0
      1. Does not need to be set here – It will be defined in the "Deposition Configuration" recipe
    - xii. Control Gain = 0.001
    - xiii. Control TC = 0.1
    - xiv. Control DR = 0.1
    - xv. Max Power = 50%
    - xvi. Sample = 1
    - xvii. Hold Time = 00:00
    - xviii. New Rate, Rate Ramp Time and Idle Ramp – No need to define



3. Once all parameters have been set – Select “Save As”
  - a. A dialog box will open where the file name can be entered
  - b. File name must take the form:
    - i. XTC###.DAT
      1. Where ### are three numbers (007 – 999)
      2. Do Not use 001 – 006
        - a. These have been setup for the specific metals in the pockets
      3. Select a number set that is not in use unless adjusting an existing recipe
      4. Remember the number (###) as it will be needed in the Deposition recipe
    - ii. Dialog box will open stating:
      1. File will be saved as ....
      2. Click OK



## Deposition Configuration Recipe Generation:

1. Click on “Edit/Create” above “Deposition”
  - a. The “Deposition Configuration” window will open
  - b. You will be able to set the following process parameters:
    - i. Process Heat
    - ii. Minimum Vacuum Required
      1. “Y” and a value less then “1E-4”
      2. or “N”
    - iii. XTC File (3 digit XTC file)
    - iv. Crucible Number (1-6)
    - v. Sweep Pattern (1 – 4)
      1. Default is 1 if manual mode
    - vi. Process gas flow
      1. Fixed “SCCM” (0 – 100 sccm)
      2. Or PID Pressure in Torr
    - vii. Nanopositioner XYZ (um)
      1. X and Y axis: 0 – 100 um
      2. Z axis: 0 – 10 um
    - viii. Select Save As
      1. Dialog window opens
      2. Name must be 7 characters or less
      3. No special characters
      4. States “File will be saved as”
      5. Select OK

## Heat Subroutine Recipe Generation:

1. On “recipe” main screen – select “Edit/Create”
2. Opens Auto Heat Configuration dialog box
  - a. Enter heat set point in “Heat Setpt” box
    - i. Values in Celsius
    - ii. Max Temp 130 C
    - iii. Crystal damage can occur
  - b. Enter Soak Time
    - i. Heat subroutine will run for this time after it reaches temp set point
  - c. Minimum Vacuum required
    - i. Yes or No
    - ii. If yes – min vacuum in Torr
    - iii. PID Settings are only available in manual mode
  - d. Save As
    - i. Dialog box opens with heat routine name
    - ii. 7 characters or less
    - iii. No special characters
    - iv. “File will be saved as” – select OK

## Master Recipe Generation:

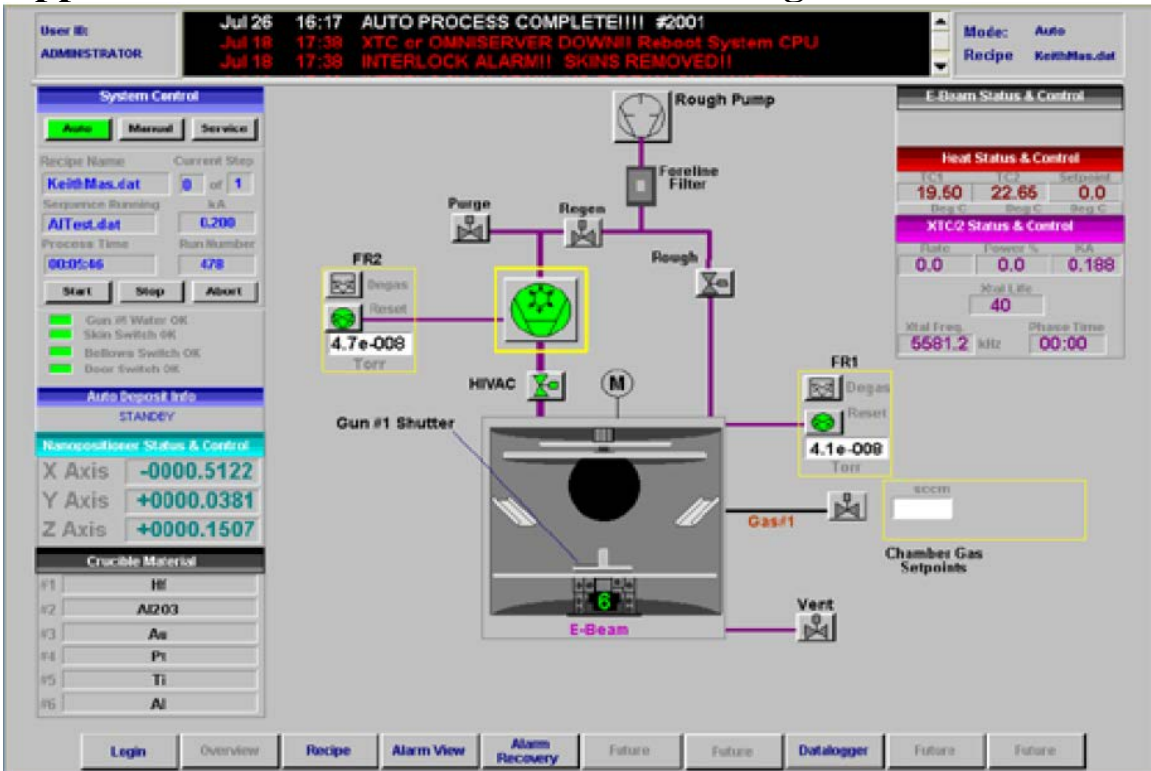
1. On main “Recipe” Screen – select “Edit/Create” under Master Recipe
2. Master Recipe builder dialog box will open
  - a. Single or multiple sequence can be built
  - b. A master recipe can include
    - i. Auto Pump
    - ii. Auto Heat
    - iii. Auto Vent
    - iv. Auto Regen – Not recommended
  - c. If you want to build a master
    - i. Click on the pull down next to “Sequence Type”
    - ii. Click on the desired sequence name from the pull down
    - iii. Add recipes from Master Sequence list or User Defined recipes
  - d. Save As when completed

## **Programming the Film Thickness Monitor (SQM 160):**

This monitor is capable of retaining the characteristics of 9 different films. However due to the many different films we use here at ND, the number of users this machine has, and the amount of use it sees I suggest that you verify your information every time you use this machine.

1. If you start using this monitor and it is displaying CRYSTAL 1, press “Xtal life” button to bring it back to its normal view. This reading shows the amount of life the Crystal has left. Change if  $< 60\%$
2. Press “Zero” button to zero the monitor readings (Thickness and Time)
3. On the film thickness monitor. Press the “program” button.

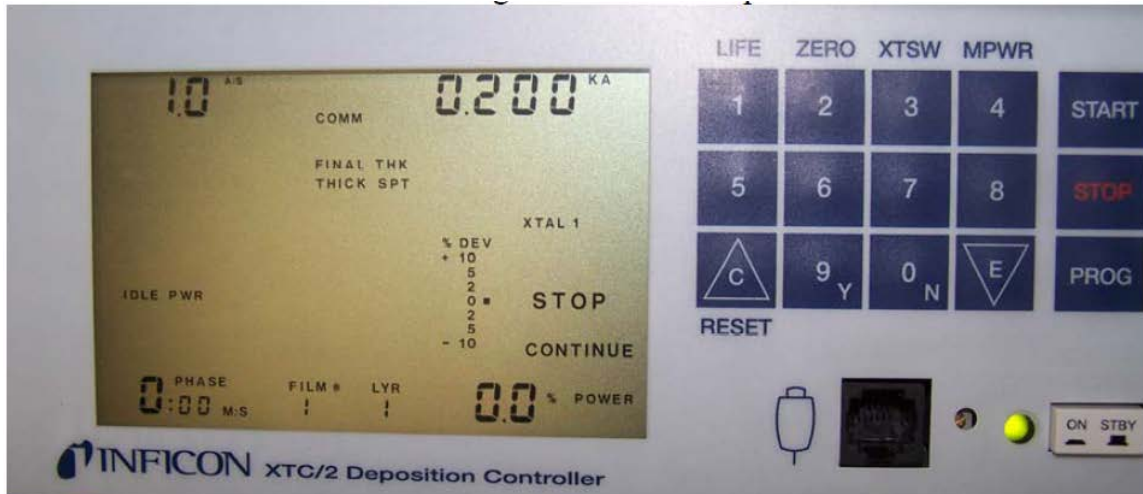
# Appendix A: Software and Hardware Images



Software Screen



Telemark TT-8/10-15 High Voltage and Current Controller containing XY Sweep Controller (with Memory Module) and Beam Positioning controls



Inficon XTC/2 Deposition Controller

- LIFE – Displays % crystal life used
- ZERO – Clears the displayed thickness
- XTSW – Changes the active crystal
- MPWR – Places the unit in manual power control or rate control mode
- START – Initiates the sequencing
- STOP – Halts the operation
- PROG – Toggles the display between the program and operate screen
- ON/STBY – Switches power of the instrument between on and standby
- Green LED – Indicates that the unit is set to ON
- ENTER/DOWN – Cursor control and decreases power in MPWR
- 0/N – Zero or NO – Also enters communication set up if held during power up
- 9/Y – Nine or Yes – used when in program mode
- CLEAR/UP/RESET – Cursor control, raises power in MPWR and resets unit
- DIGITS 0-9 – If the nine key is held during power up – LCD segment test



## Appendix B: Density and Z-Ratio Chart

<b>Material</b>	<b>Formula</b>	<b>Ratios</b>	<b>Density</b>	<b>Z-Ratio</b>	<b>Notes</b>
Aluminum	Al		2.700	1.080	Slowly heat – crucibles crack easily
Aluminum Oxide	Al <sub>2</sub> O <sub>3</sub>		3.97	0.336	
Aluminum Boron Silicon	Al/B/Si	98-1-1	2.693	1.169	
Aluminum Silicon	AlSi	98-2	2.692	1.073	
Chromium	Cr		7.200	0.305	Sublimes
Cobalt	Co		8.710	0.343	
Copper	Cu		8.930	0.437	
Germanium	Ge		5.35	0.516	Permission Required
Gold	Au		19.300	0.381	
Gold Germanium	AuGe	88/12	17.626	0.397	
Gold Palladium	AuPd	60/40	16.380	0.371	
Hafnium	Hf		13.09	0.34	
Molybdenum	Mo		10.200	0.257	
Nickel	Ni		8.910	0.331	
Palladium	Pd		12.038	0.357	
Platinum	Pt		21.400	0.245	
Platinum Iridium	PtIr				Permission Required
Silicon	Si		2.320	0.712	Permission Required
Silicon – p-doped	Si (boron)		2.320	0.712	Permission Required
Silicon Dioxide	SiO <sub>2</sub>		2.200	1.070	Permission Required
Tantalum	Ta		16.6	0.262	
Titanium	Ti		4.500	0.628	
Tungsten	W		19.300	0.163	
Vanadium	V		5.96	0.53	