

# DENTON VACUUM®

## PEM VERSION 1.0

PLASMA EMISSION MONITOR

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OPERATING MANUAL

**DENTON VACUUM**  
Enabling Innovation

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## SAFETY WARNINGS



1. The use of Denton Vacuum PEM involves the handling of precise mechanical and optical components that must be done carefully. Damage to any one of the components such as the PEM spectrometer, fiber optic cables, fiber optic vacuum feed through and the in-cathode plasma sensors can cause the PEM system to malfunction.
2. Always place the protective rubber end pieces on the fiber optic components when not in use to prevent damage.
3. Always ensure that all fiber optic components are fully connected. Any fiber optic endings exposed to deposition processes will become coated. Such coating will detriment the transmission of plasma emission species to the spectrometer and therefore results in a reduced feedback signal or worse, no signal at all. Such misuse will result in an inoperative optical component. Cleaning fiber optics of debris such as thin film coatings requires careful and precise procedures. This can be very difficult and requires precision. In the event that an optical component becomes coated, refer to the trouble shooting section in this manual.
4. A protective honeycomb fixture is provided at the end of the in-cathode fiber optic sensors to prevent the growth of thin film coating and prolong the tool set. The honeycomb fixture is the only component that may be cleaned and serviced by the operator provided the required tools are available. Refer to the trouble shooting section in this manual for instruction.

## INTRODUCTION

### **REACTIVE SPUTTERING & PROCESS CONTROL**

In the technical field of thin film deposition, magnetron sputtering is widely used to create compound thin film materials such as dielectrics, optical films and hard coatings. Varieties of ceramic compound targets such as silicon dioxide, titanium dioxide or aluminum oxide for example, have been used to deposit a variety of thin film materials. Several difficulties arise from operating processes with such ceramic targets. The difficulties range from insulating film materials causing cathodic arcing to low deposition rates and overall film quality. Selecting the right power supply to troubleshoot the arcing of ceramic targets generally involves RF power, which leads to very low deposition rates, therefore hindering the throughput of a process tool.

Reactive Sputtering techniques are used extensively in both research and production to overcome many of the difficulties and setbacks inherent with the sputtering processes of ceramics and other insulating materials. The reactive sputtering process involves the deposition of an elemental target (elemental titanium or silicon for example) in a gas mixture of a reactant (generally oxygen or nitrogen) with an inert gas (most often Argon) to form a chemical compound which is deposited onto a substrate. The inert gas, Argon in most cases, is used as the working gas while the reactive gas is varied in order to obtain a desired chemical compound, which is to be adhered to a substrate. Fixed gas flows, partial pressures or a slight permutation of each are then integrated into process to operate in a fully reacted compound state of the target, an elemental state or a transition state.

A fully reacted compound state of the target is often referred to as a fully poisoned target. An elemental target in transition mode is any state of the target in between the elemental state and the fully reacted (poisoned) state. A target in the poisoned state can provide ideal process conditions for desired films. Depending on the size and pumping speed of a system, which are crucial process parameters, a fully poisoned target may be the best situation to provide the necessary conditions of desired film characteristics. However, the deposition rate of a poisoned target is much less than that of the transition mode and even less than that of a pure elemental state of a target. Although the deposition rates of a fully

poisoned target are generally higher with a pulsed DC power supply as opposed to an RF power supply, a target in transition mode is the desired operating point for reactive sputtering. A sputter tool system configuration (i.e. size, ultimate pressure and pumping speed) can either enable or hinder the range of process window for transition mode sputtering to be more easily controlled by a variety of reactive gas control methods.

Transition mode reactive sputtering involves operating a process within an amount of reactive gas in which the target is in transition between the elemental state and the fully reacted compound state. Operating in the transition mode can enable higher deposition rates and target yield/utilization while still obtaining the desired chemical compound adhering to a substrate. In modern industrial thin film coating, Reactive Sputtering is a commonly used technique to deposit a wide variety of thin film coating materials for precision optics, hard coats, flat panel displays, semiconductors and decorative finishes among other applications. Therefore, operating in the transition mode of reactive sputtering is a highly valued technique, which is very sought after in both research and production.

## **REACTIVE GAS & PROCESS CONTROL**

It is often difficult to operate in transition mode for most reactive sputtering deposition chambers. Operating in this mode requires precise control of the reactive gas. The technique of reactive gas control for reactive sputtering processes has a strong influence on deposition rates, process yield and a complex variety of thin film properties. It is therefore a desirable approach in overcoming the process difficulties and setbacks inherent with the sputtering of ceramic compound targets or fully poisoned elemental targets. In both research and production environments, there are several techniques used for reactive gas control including partial pressure control, optical emission spectroscopy and plasma emission monitoring.

## **PLASMA EMISSION MONITORING**

Plasma Emission Monitoring is a technique used in reactive sputtering to improve both the control of a thin film process as well as the analysis of respective process parameters. An in-vacuum fiber optic sensor monitors the plasma species among a range of wavelengths of a specific sputtering process. The range of wavelengths (in nanometers) for plasma emission monitoring is generally from UV to near IR. Most process materials emit plasma species during reactive sputtering in this range of wavelengths. The feedback of a specific wavelength, associated with either the plasma emission of the elemental target or the reactive gas, is used to control the reactive gas input through a single closed loop PID.

Denton Vacuum offers an innovative solution to the difficulties of reactive magnetron sputtering with the integration of Denton Vacuum PEM 1.0. The details and purpose of this process tool are discussed in the following pages. It is encouraged that the user familiarizes them self with this manual for best use of the Denton Vacuum PEM 1.0 product.

## **DENTON VACUUM PEM 1.0**

### **Introduction**

Denton Vacuum has developed PEM as an innovation toward the control of reactive gas for reactive sputtering vacuum deposition equipment. As a budget friendly upgrade to any Denton sputter deposition vacuum chamber, Denton Vacuum PEM offers a more sophisticated process control system for reactive sputtering as well as insightful analysis for process application.

This manual details the components, hardware, installation, operation and instructional use of Denton Vacuum PEM 1.0. The manual will also detail the integration of Denton Vacuum PEM into automation recipes for fully automated process control of sputter deposition equipment as made possible by Denton Vacuum's software package. Denton Vacuum PEM can be operated for both process development and analysis or automated for precise production use and application.

Because Denton uses the finest available subsystems and components, the system is highly reliable and durable. The system offers you a range of thin film process options. However, it is important to note that with all of a system's potential, safety considerations exist. Safety protocols should be taken both for the well-being of the user and for the prolonged duration of the system's capability. Individuals who are to operate, service, or maintain this system should familiarize themselves with this manual.

If this equipment is use in a manner not specified by Denton Vacuum, the protection provided by the equipment may be impaired. A complete understanding of this control is recommended before operating the vacuum deposition system with Denton Vacuum PEM.

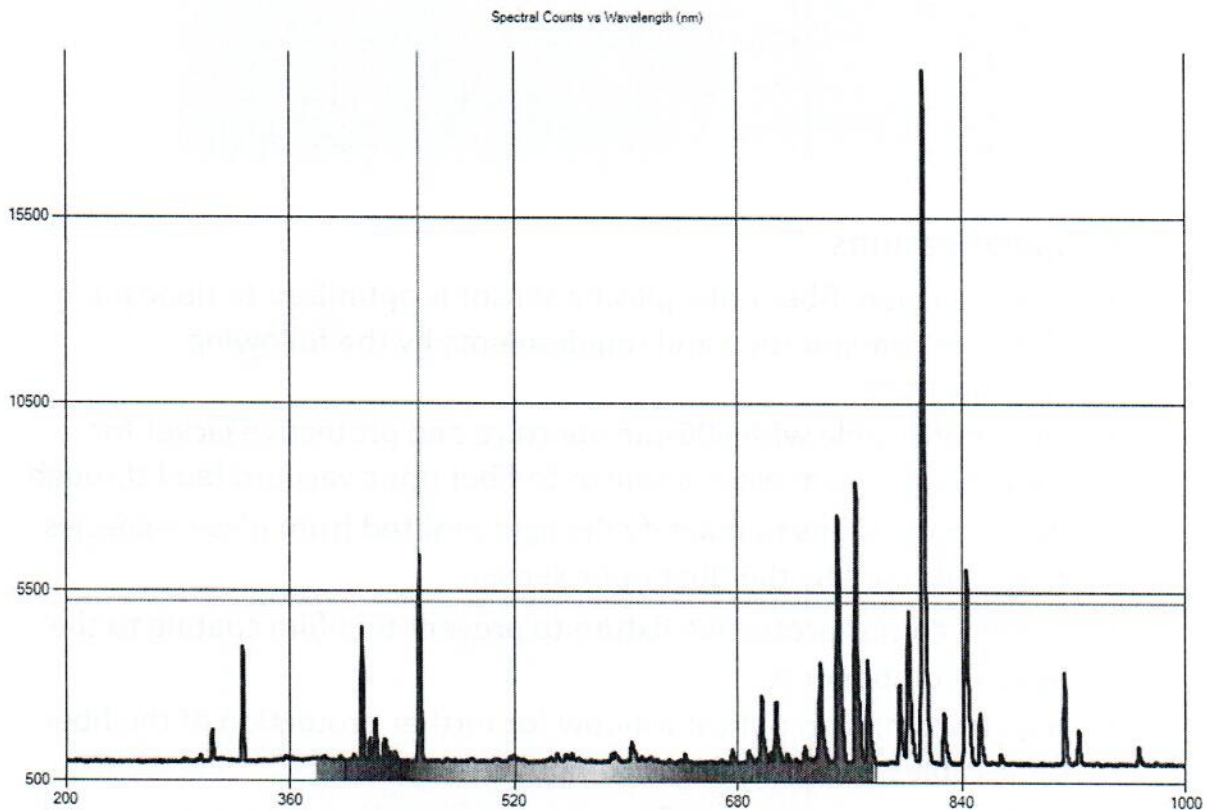


## Overview

Denton Vacuum has developed Denton Vacuum PEM as a plasma emission monitor available for full auto process integration to any of the Denton Vacuum chambers fitted for magnetron sputter deposition.

Denton Vacuum PEM enables the reactive sputtering process to operate in a desired, controlled deposition mode as determined by the user. The PEM system includes in vacuum fiber optic probes equipped with protective encasings, high precision in vacuum fiber optic cables, fiber optic vacuum feed through and a USB-UV-Vis spectrometer equipped for integrated process control with Denton Vacuum's automation software packages.

A typical feedback signal of the plasma emission species occurring during a reactive sputtering deposition process is displayed below. This specific emission display is for a Titanium Dioxide (TiO<sub>2</sub>) process. The display plots the relative intensity of elemental plasma emission species (spectral counts) with respect to wavelength (nm).

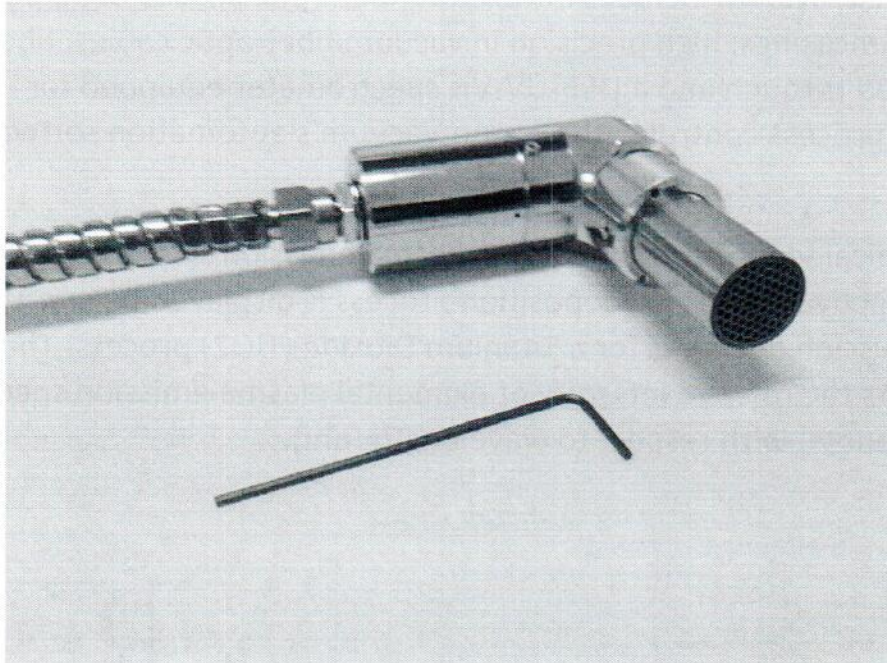


## SYSTEM COMPONENTS

### FIBER OPTIC HARDWARE

#### Plasma Sensor

- In vacuum fiber optic plasma sensor



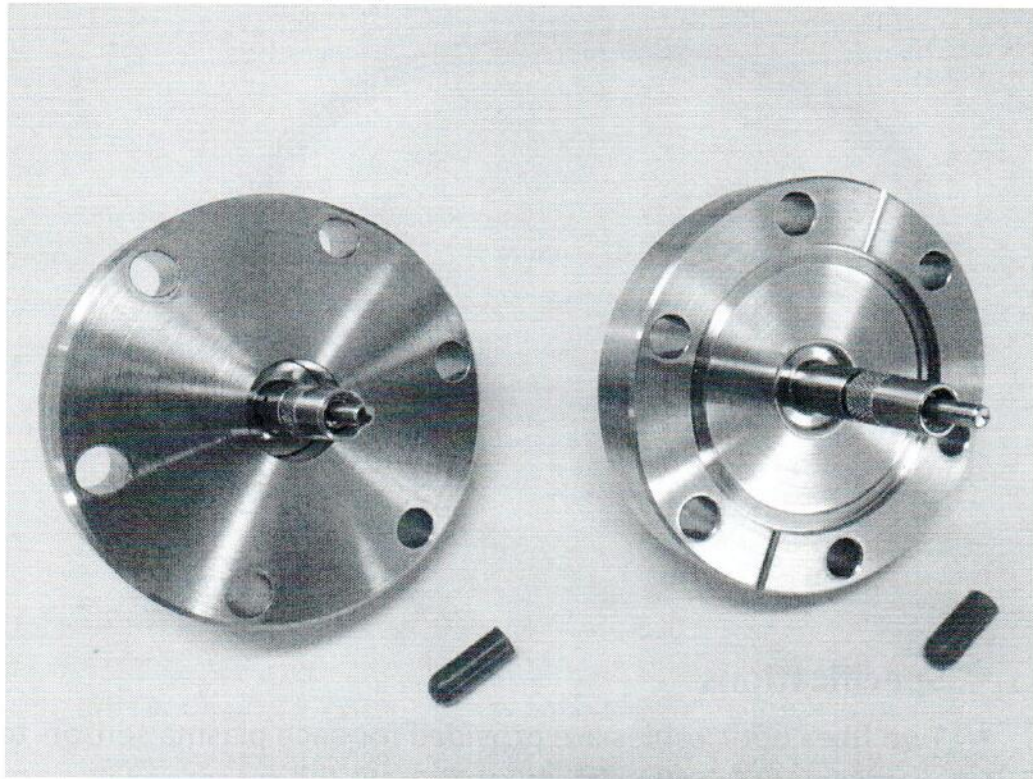
- **Specifications**
  - The in vacuum fiber optic plasma sensor is optimized to deposition chamber configuration and requirements by the following specifications
  - Fiber optic cable with 600- $\mu\text{m}$  aperture and protective jacket for transmission from plasma sensor to fiber optic vacuum feed through
  - Plano-convex lens to magnify the light emitted from plasma species to be collected by the fiber optic sensor
  - “Honey comb” protective fixture to prevent thin film coating to the optical components
  - High transmission optical window for further protection of the fiber optic cables and optical lens components
  - A 45° mirror to permit a 90° sensor configuration for more compact system and cathode configuration

## Fiber Optic Cables



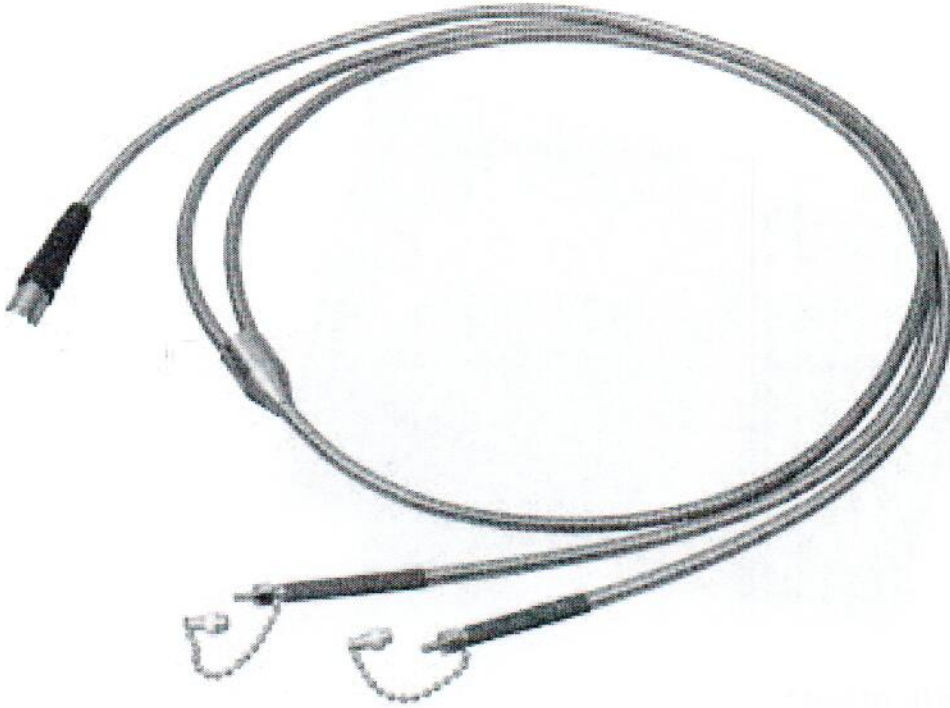
- **Specifications**
- Two fiber optic cables are provided for each plasma sensor- to - spectrometer configuration.
- One fiber optic cable with 600- $\mu\text{m}$  aperture and protective jacket for in vacuum use from the plasma sensor to fiber optic vacuum feed through
- One fiber optic cable with 600- $\mu\text{m}$  aperture and protective jacket for out of vacuum use from the fiber optic vacuum feed through to the USB spectrometer.

## Fiber Optic Vacuum Feedthrough



- **Specifications**
- A fiber optic vacuum feed through is provided for connecting the in vacuum fiber optic cable to the out of vacuum cable.
- The feed through consist of 2-3/4-inch con-flat connection fitted for a copper O-ring vacuum seal.
- Each end of the feed through is equipped with a 600- $\mu$ m aperture input port for connection of each fiber optic cable.

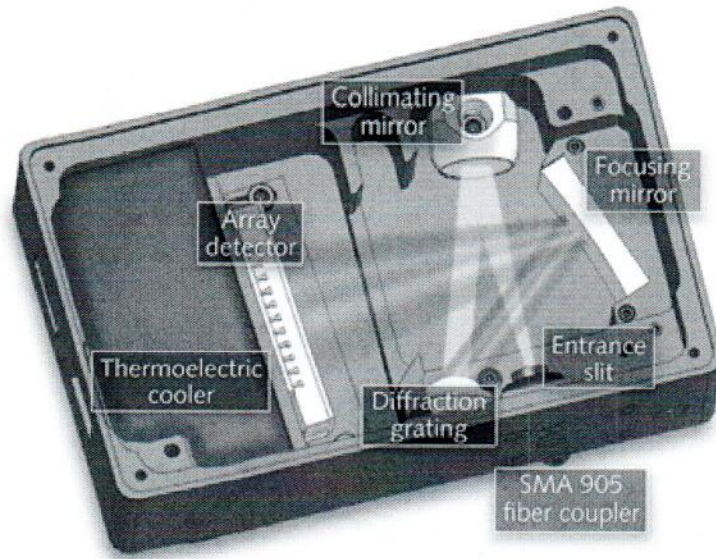
## Fiber Optic Splitter Adapter



### ▪ Specifications

- A fiber optic cable splitter is provided to couple the light from each fiber optic cable to enable the use of only one spectrometer.
- Without the splitter adapter, a separate spectrometer is needed for each plasma sensor to vacuum interface.
- Depending upon the number of plasma sensors, the splitter adapter will be different for each respective set up.

## USB UV-Vis Spectrometer

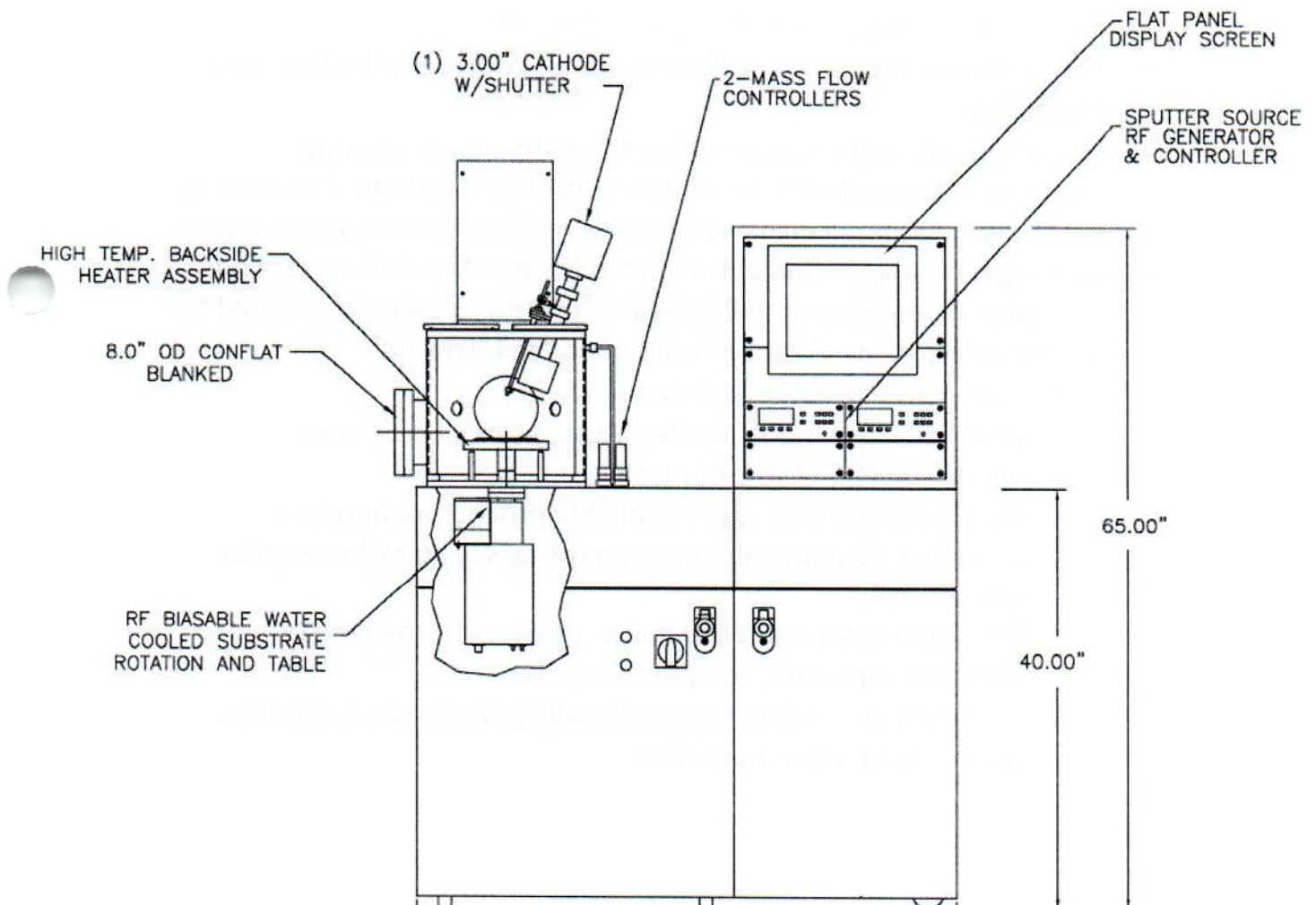


- **Specifications**

- USB-UV-Vis spectrometer can be used to integrate from one-to-four plasma sensor to fiber optic setups.
- The UV-Vis spectrometer is connected to the computer software via USB port, which also powers the device.
- USB-UV-Vis optical data acquisition system measures spectrum in the range of 200nm-1000nm, with wavelength resolution of <math><2\text{nm}</math> and integration time as low as .
- USB UV-Vis acquires data via the fiber optic connection port for fiber optic cables.

## VACUUM CHAMBER CONFIGURATION

- **Configuration:** The configuration of a sputter tool system (i.e. size, ultimate pressure and pumping speed) can either enable or hinder the range of a process window for transition mode sputtering to have more control stability by a reactive gas control method such as Plasma Emission Monitoring.
- **Reference:** An example schematic of a typical Denton sputter tool designed in a confocal array with rear pumping port is displayed below for reference.



## DENTON AUTOMATION SOFTWARE

- The control system for the vacuum system is GE Cimplicity® HMI (Human Machine Interface). Cimplicity® runs on a Windows operating system. This should make the interface between operator and machine familiar and easy to learn.
- This software links the operator to the PLC. It allows for data input and data display. Operators can use a mouse to select on-screen graphics by clicking on any active element on a screen. Data is input by pushing on-screen buttons or using the keyboard.
- Denton Vacuum PEM is fully integrated into all Denton software package capabilities including: visualization screens, separate PEM operating screens, auto recipe generation, etc.
- This software integration is done in conjunction with the following capabilities
- Single control cabinet mounted on the right side of vacuum chamber; Equipped with computer control by Windows 7 seven with a mounted flat panel display providing visualization of system layout.
- GE Programmable Automation Controller interfaced to system host computer; integrated with ProcessPro® control software configured for specific delivered hardware which include the following:
  - Computer control of subsystems
  - Multiple Operational modes: Auto, Manual & Service
  - Menu Driven Process Control
  - Recipe creation for all controllable process parameters including Plasma Emission Monitoring set point for reactive gas control
  - Full remote set point capability: power, Plasma Emission line set point, pressure, temperature, etc.
  - Comprehensive data logging for all operational parameters during Auto mode operation



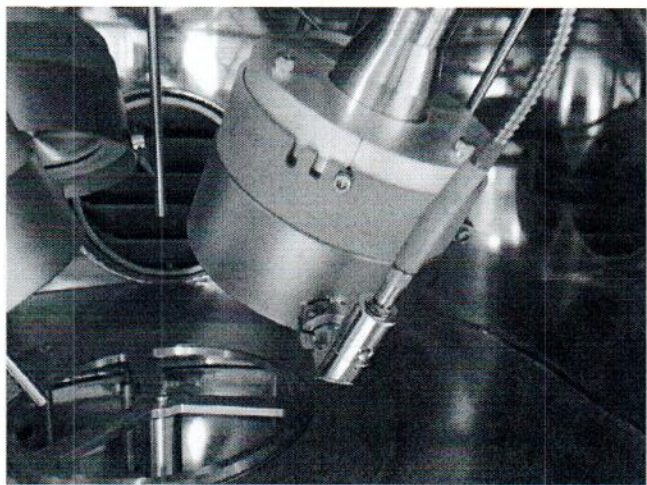
## SYSTEM SETUP

### INSTALLATION

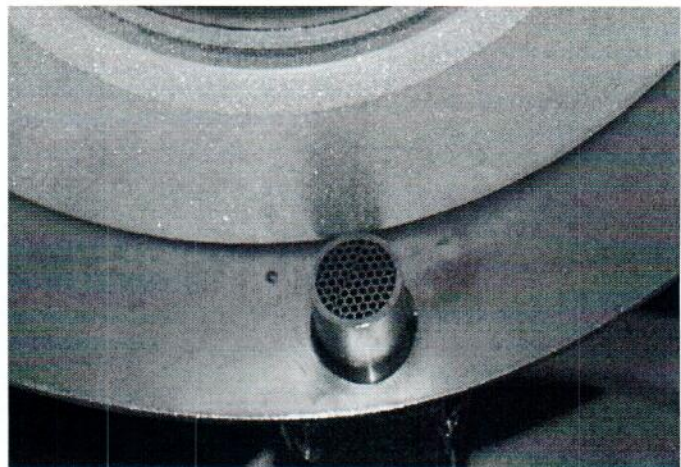
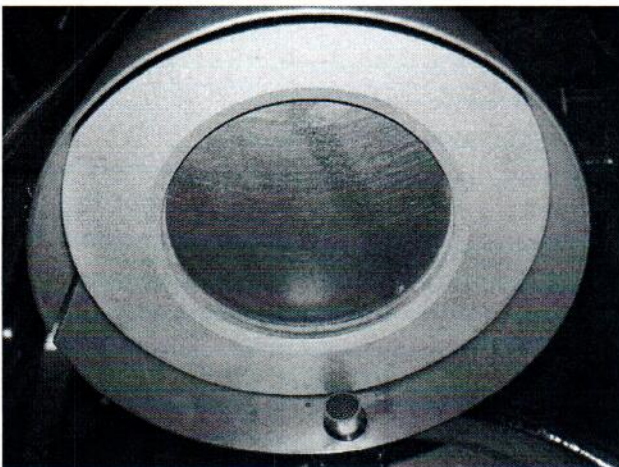
- **Purchased with System:** In the event that a Denton Sputter Deposition Chamber is purchased with the capability of Denton Vacuum PEM, all of the hardware and software will already be installed upon the completion and release of the final system.
- **Hardware Configuration:** A detailed display of the proper hardware configuration is provided in the next section as reference for the operator.

## HARDWARE CONFIGURATION

- **Plasma Sensor:** For optimal spectral signal with Plasma Emission Monitoring, it is important to assemble the Fiber Optic Plasma Sensor with precision and close proximity to target plasma interaction.
- **Plasma Sensor:** Below are several example displays to reference proper alignment and placement of the **Plasma Sensor**
- **Outer Cathode Shield:** A custom machined magna collar is use to fasten the **Plasma Sensor** to the cathode shield.

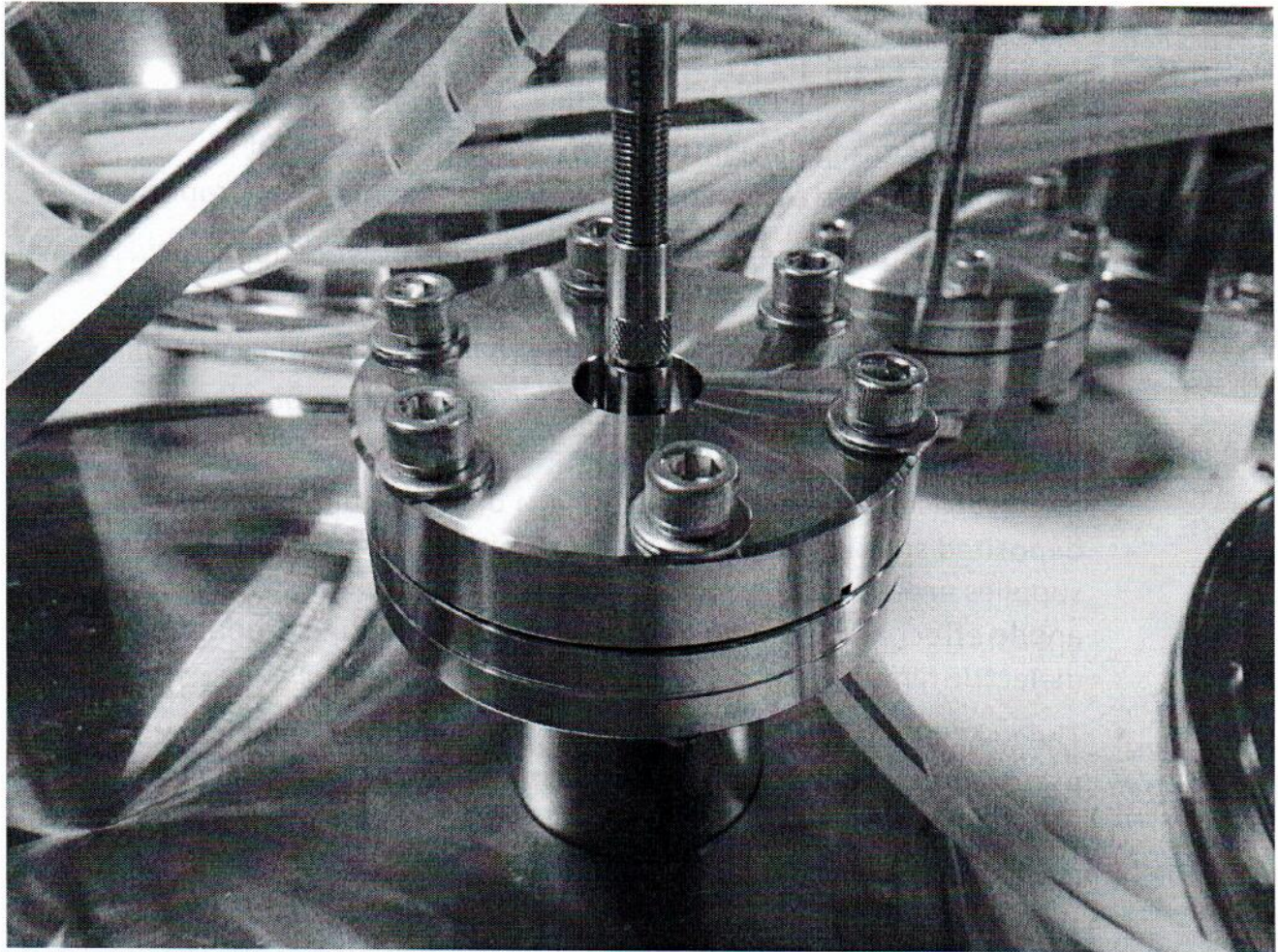


- **Inner Cathode Shield:** The Plasma Sensor is placed in close proximity to the anode but **not** touching. It is aligned to be perpendicular to the normal direction of the target. It is therefore in position to collect light in a direction which is across the target and through the plasma.



## HARDWARE CONFIGURATION

- **Fiber Optic Cables:** It is important for the durability of these components to ensure that all fiber optic cables are connected carefully and securely. Fastening a cable too tight or abruptly may cause damage to the fiber optic ends and render it inoperable.
- **Vacuum Feedthrough:** The fiber optic ends of the feedthrough are just as delicate as they are on the cables. They should be handled with extreme care and caution to prevent damage. When connecting the feedthrough and making a seal to the copper o rings, torque should be applied in a traditional fashion to create an optimized seal. A display of proper vacuum feedthrough connection is provided below for reference.



## OPERATING THE TOOL SET

### GETTING STARTED

#### OVERVIEW

- The first thing that an operator will want to do when using Denton Vacuum PEM is make sure all hardware is properly installed as explained previously in chapter 3.
- The operator will also want to be sure that all targets, gas lines and power supplies be properly installed and ready to operate.
- As with any other vacuum deposition, it is important that all safety pre-cautions be set in place before operating process.

#### SELECTING THE POWER SUPPLY

- Before integrating PEM into a given deposition process it is important to have a good understanding of the material properties.
- This is particularly useful in selecting the best power supply for sputter deposition.
- Most if not all magnetron sputtering processes use either RF (radio frequency generally at 13.56MHz), Pulsed DC (either bipolar or unipolar) or traditional straight DC power generators.
- In most cases for reactive sputtering, a dielectric film is being deposited such as  $\text{TiO}_2$ ,  $\text{SiO}_2$ ,  $\text{Si}_3\text{N}_4$  etc. Either Pulsed DC or RF power supplies are typically used in these cases to avoid the disappearing anode effect and arc occurrences from the insulating properties of dielectric films.
- The use of Pulsed DC power supplies, both unipolar and bipolar, provide higher deposition rates than with RF power for the same given process.
- In the case of a reactively sputtered thin film that is conducting like TiN, there is no need to use RF or Pulsed DC. Therefore, conventional DC power generators are typically used for these films which yield the highest deposition rates.

## **OPTIMIZING SIGNAL & MATERIAL WAVELENGTH**

- As mentioned in chapter 3 - system set up, alignment of the plasma sensor is crucial for optimizing the plasma emission intensity for any material.
- For the same process parameters, one material (i.e. Titanium) may have a higher intensity plasma emission than another (i.e. Silicon). This is a representation of the inherent physics of reactive sputtering and does not mean that that plasma sensor alignment has changed from one target (i.e. Titanium) to another (i.e. Silicon).
- As evidence, the relative intensity of atomic spectra of the elements with respect to wavelength can be referenced in scientific literature or most any handbook of chemistry and physics. Via the internet, the National Institute of Standards and Technology (NIST) is also a useful reference of the relative intensity of atomic spectra.
- The material properties of different elemental targets are inherent to process application. It is important to reference scientific literature before transitioning the use of Denton Vacuum PEM from one elemental target (i.e. Titanium) to another (i.e. Silicon).
- It is however, still important to always check the alignment of the plasma sensor when transitioning materials as mentioned above.
- A list of some elemental atomic spectra with the most relatively intense wavelength for each is provided on the following page.

**ATOMIC SPECTRA REFERENCE**

Plasma Emission Species of a reactive sputtering process is a complex combination of elemental spectrum. Control of process using PEM is reliant upon the emission spectra of a selected wavelength and the concentration of that plasma species among the relative spectra. Since the plasma emission spectra can be very complicated, it is therefore important to be sure that a control wavelength is selected carefully. A table of reference wavelengths for commonly use elemental targets is provided.

Element	Wavelength (nm)
Si	253
Si	289
Al	310
Ta , Cu	330
N <sub>2</sub>	337
Zr , Co	340
Ni	360
Ti	365
Hf	370
Ni , Sn , Mo	380
Mo	390
N <sub>2</sub>	392
Al	397
Ti , W	400
Nb , In	410
Zr , Mg	420
In	450
Zr , Zn	470
Zn	480
Ti	502
W , Cu	510
Ti , Zn , Cr , Cu , Mg	520
Sn	560
C	600
H	655
H, N <sub>2</sub>	660
N <sub>2</sub>	670
Ar	750
O <sub>2</sub>	764
O <sub>2</sub>	777
Ar	812

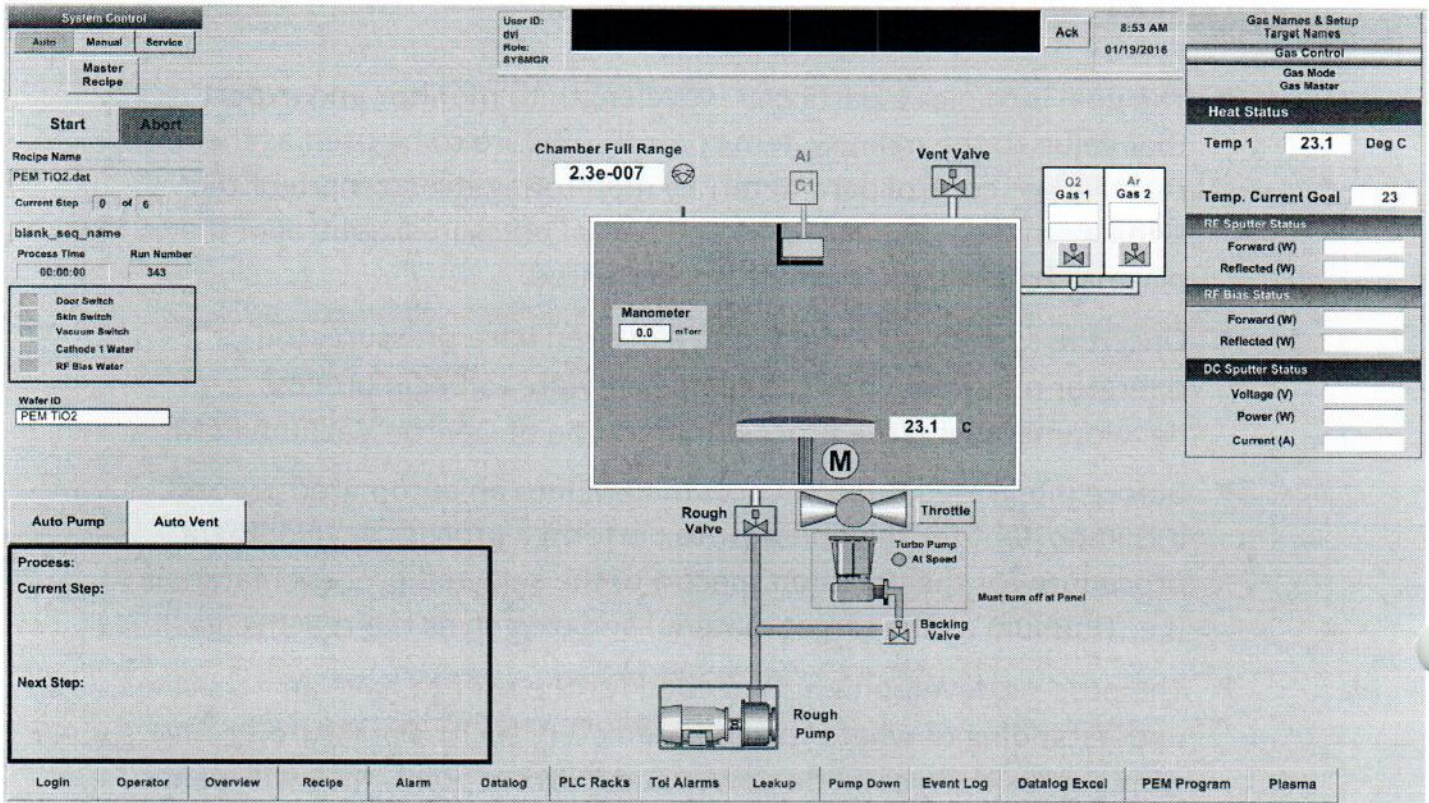
## MANUAL MODE OPERATION

### NAVIGATING THE SOFTWARE

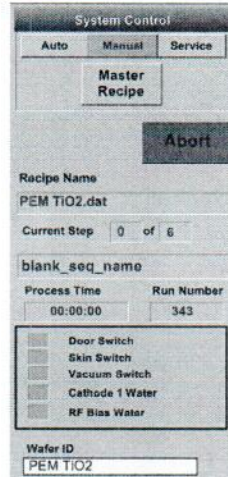
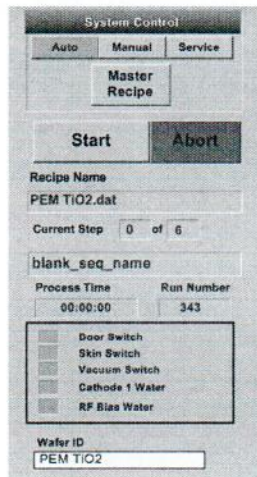
#### MANUAL MODE

- The goal is to pick a particular wavelength to monitor and export that value to the main system control software to be used as the reactive gas control parameter. By monitoring the strength of the signal with different gas flows and partial pressures, control of the plasma characteristics can be implemented.
- Once the system is evacuated to a desired base pressure, the operator may begin navigating the software to begin process development with the process integration of Denton Vacuum PEM.
- Before integrated Denton Vacuum PEM into an automated process, it is important for the operator to perform a process development procedure for the emission spectra of the selected process materials (i.e. Titanium as the target material and oxygen as the reactive gas).
- The process development is needed for the user to have an understanding of what process parameters such as pressure, working gas flow, cathode power and PEM set point, they will want to operate the reactive sputter process in.
- This will require the user to operate software in **manual mode** and generate a **hysteresis curve**.
- **Note:** A systematic procedure on how to do so using a reactively sputtered TiO<sub>2</sub> process for reference is provided on the following pages.

- **Manual Mode**
- **Overview Screen** - Once the system control software is open, start in the overview screen. Below is a typical overview screen with integrated PEM software.



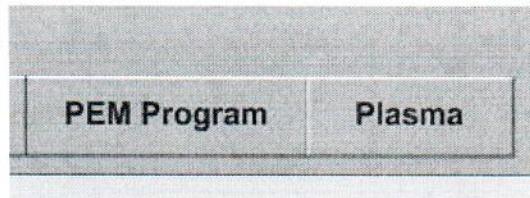
- **Manual Mode** - To begin PEM process development start by selecting the software from Auto mode into Manual Mode in the upper left corner of the overview screen.



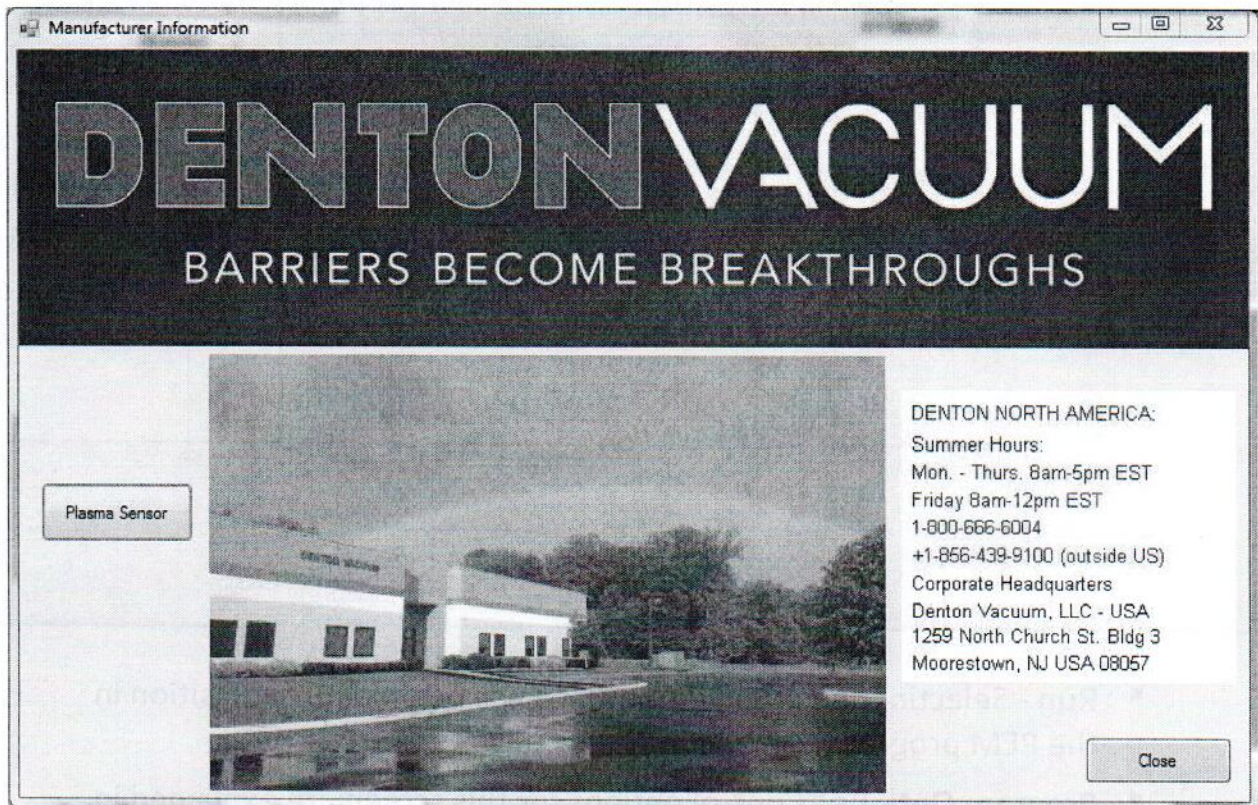


## PEM Program

- **PEM Program** - Next, the operator must select the **PEM Program** option from the bottom right hand corner of the tool bar in the overview screen

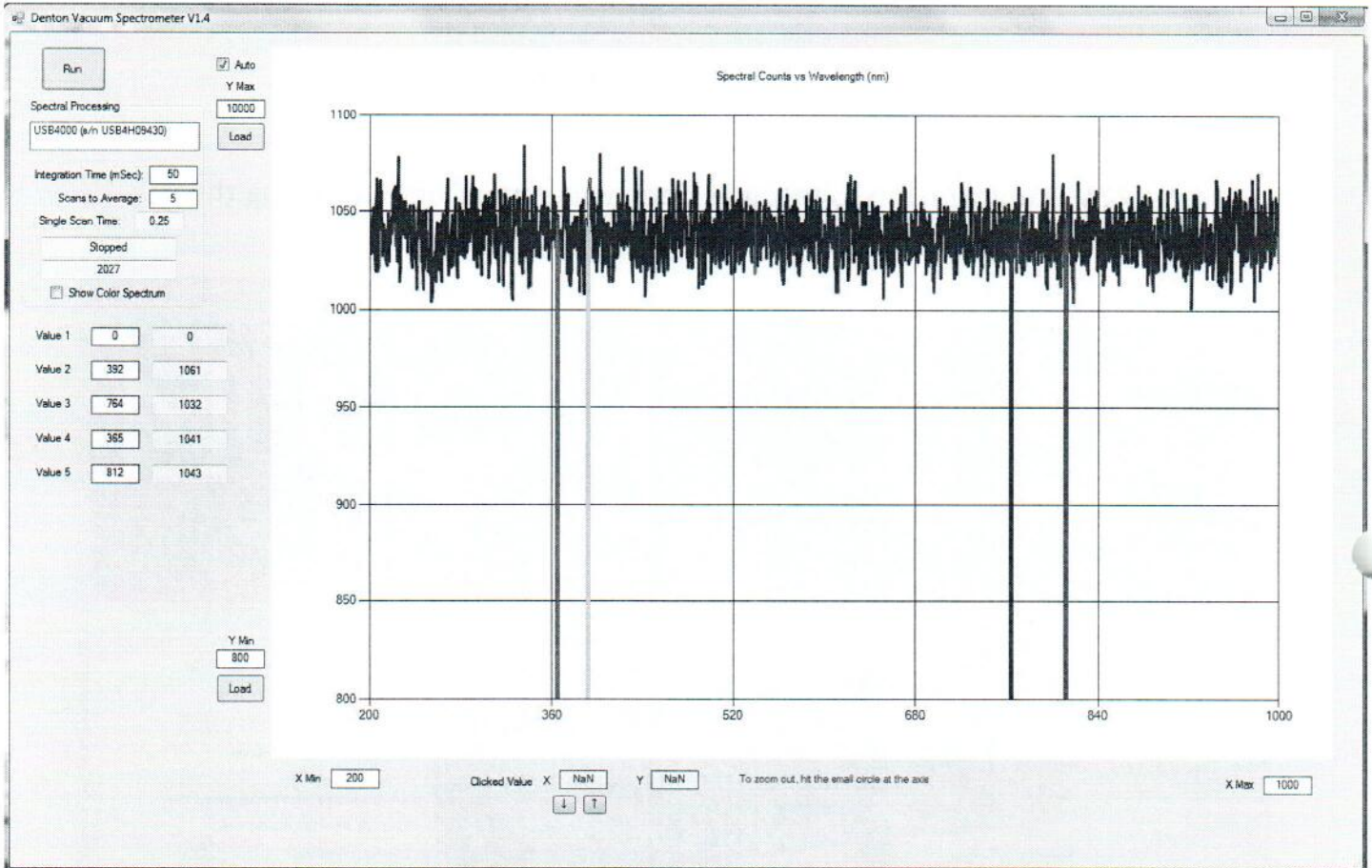


- **PEM Program** The following screen will appear upon opening the program



- **Plasma Sensor** - Select the plasma sensor option to fully open the **PEM program**. The above screen must stay open to operate the program; it will only need to be close by selecting close when the operator is finished with the PEM program.

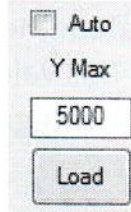
- **PEM Program**
- **Plasma Sensor** - After selecting the Plasma sensor option from the previous screen, the program will attempt to communicate with the spectrometer via the USB port connection on the PC. If a connection is established the following screen will open.



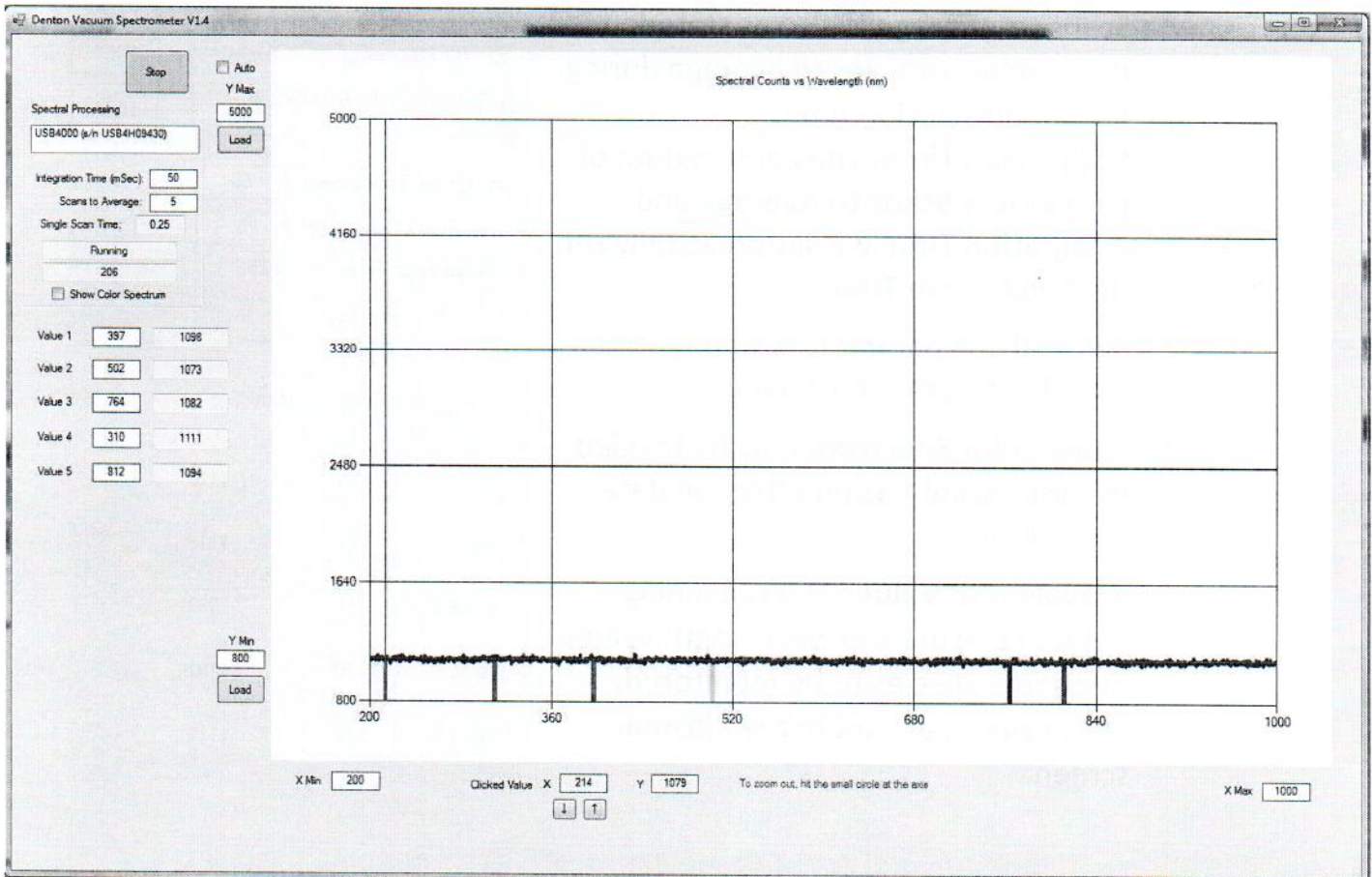
- **Run** - Selecting the green **Run** button will begin data acquisition in the PEM program.
- **Program Options** - Control options for this screen are explained in further detail on the following pages.

- **PEM Program**

- **Plot Axis** - Adjusting the axis from auto to fixed will change the amplitude of signal noise displayed on the plot of the spectral counts vs. wavelength. Once a fixed axis is selected, the **max** and **min** values can be set by entry of numerical value on the left hand side of the plot display.
- **Plot Axis** - Select the **load** button and the plot will be adjusted.



- **Fixed Plot Axis** - Fixed axis plot parameters will display a screen with less noise similarly to the display below. Since there is no plasma species available for data acquisition in this display, the spectrum is simply background noise and the spectrometer will not go below this value.



## ▪ PEM Program

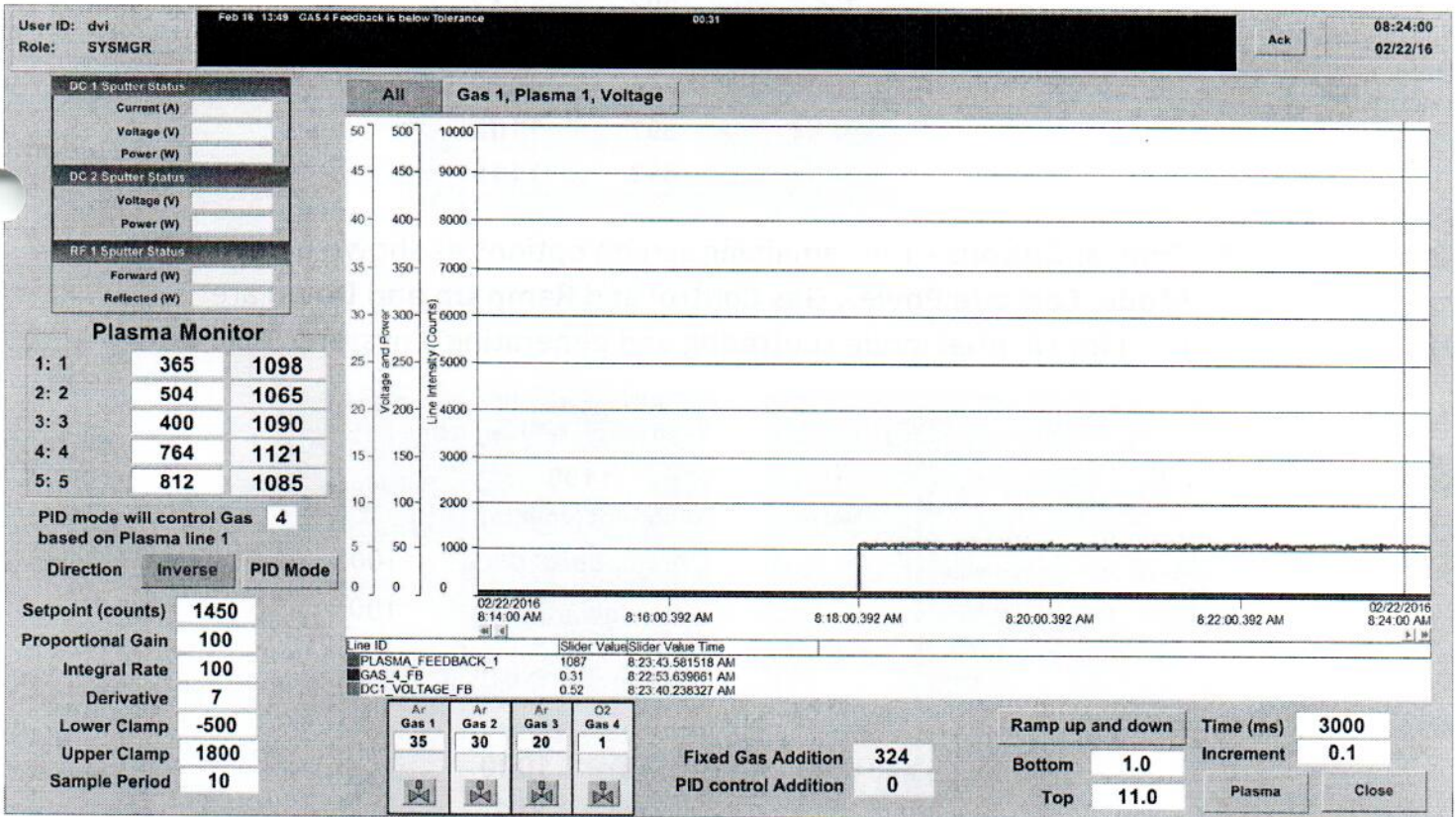
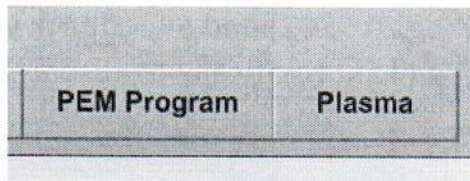
- **Control Options** The entire left hand side of the **PEM Program** screen displays all of the control options.
- **Spectral Processing** displays the spectrometer model # in which data acquisition is activated. A fault is displayed when no communication is occurring between the USB port and the **PEM Program** software.
- **Single Scan Time** is as stated: the time it takes to make a single scan of the plasma spectrum. This value is automatically set by the parameters entered in the **Integration Time** and **Scans to Average** options.
- **Integration Time (m-sec)** is essentially the amplification of the signal. The longer the integration time value is set to, the higher (or more intense) the feedback signal will be. Since the control of reactive gas needs to be fast, an integration time too long may be too slow for the reactive gas to respond in time for process stability.
- **Scans to Average** is also as stated. It is the number of scans to average during the set integration time.
- **Single Scan Time:** The combination of parameters **Scans to Average** and **Integration Time** will automatically set the **Single Scan Time**.
- Below these parameters simply states that the program is running.
- **Show Color Spectrum** may be toggled for display but has no effect on data acquisition.
- **Wavelength Values** The remaining Values (1-5) are the wavelength values of plasma species to be monitored. These values are set in the **Plasma** screen.

The screenshot shows the PEM Program control interface. At the top left is a 'Stop' button. To its right is an 'Auto' checkbox and a 'Y Max' field set to 5000. Below these is a 'Spectral Processing' section with a text box containing 'USB4000 (s/n USB4H09430)' and a 'Load' button. Further down are input fields for 'Integration Time (mSec): 50', 'Scans to Average: 5', and 'Single Scan Time: 0.25'. A 'Running' indicator shows the number '79'. A 'Show Color Spectrum' checkbox is checked. At the bottom, there is a table of five wavelength values:

Value 1	397	1118
Value 2	502	1116
Value 3	764	1099
Value 4	310	1101
Value 5	812	1117

## Plasma

- **Note:** An example of a reactively sputtered TiO<sub>2</sub> process will be used for tutorial use throughout the instruction of **Manual Mode Plasma** and **Hysteresis Curve Generation**.
- **Note:** The **monitoring wavelength** for Titanium Plasma Emission is set to 502 nm.
- **Note:** the **PEM Program** must be up and running in order for the Plasma Control Screen to display and run properly.
- **Plasma Screen:** Once the **PEM program** is running and the parameters are set to the desired values, open the **Plasma** screen from the bottom right corner of the main overview screen. The following screen will appear.



- **Note:** Since no deposition process is running, the screen will display ambient noise.
- **Control Options:** control options for the **Plasma** screen are provided in detail on the following pages.

- **Plasma**
- **Wavelength Values** - First, the operator should set the **Plasma Monitor** Wavelength values for each of the five Plasma Monitor feedbacks.
- **Control Wavelength** - While the reactive gas can only be controlled by one of the five plasma species, the **Plasma** Software can monitor all five simultaneously. Each element can be selected and a new prefix may be entered.
- **Control Wavelength** - Next to the prefix selection, a wavelength value is entered (i.e. Ti : 502) and a feedback value of intensity (spectral counts) will automatically be displayed on the right column.

Plasma Monitor		
1: Ti	502	1132
2: Ti	400	1113
3: O2	764	1092
4: N2	397	1108
5: Ar	812	1115

- **Control Options** - The remaining screen options as shown below: **PID Mode**, **Cathode Power**, **Gas Control** and **Ramp Up and Down** are used for **Manual** mode sputtering and generating a hysteresis curve.

Throttle

DC 1 Supply

Rate	100	Watts/min		Voltage (V)	
Start	100	0-1000 Watts		Power (W)	
Goal	500	0-1000 Watts		Current (A)	

Off

Shutter 1

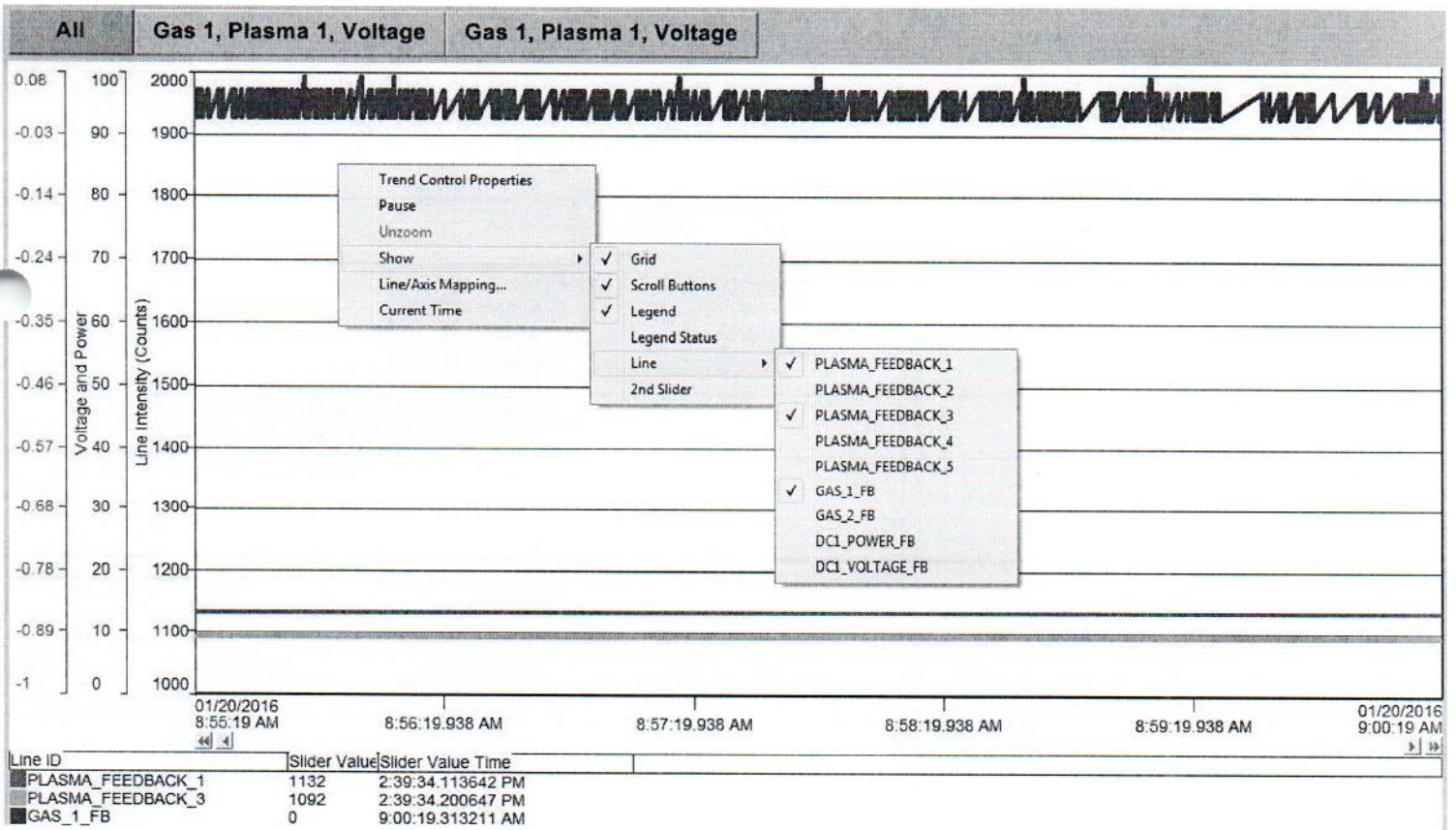
Closed

PID mode will control Gas 1 based on Plasma line 1

1100	<span style="border: 1px solid gray; padding: 2px;">PID Mode</span>
Setpoint (counts)	
Proportional Gain	100
Integral Rate	100
Direction	<span style="border: 1px solid gray; padding: 2px;">Inverse</span>

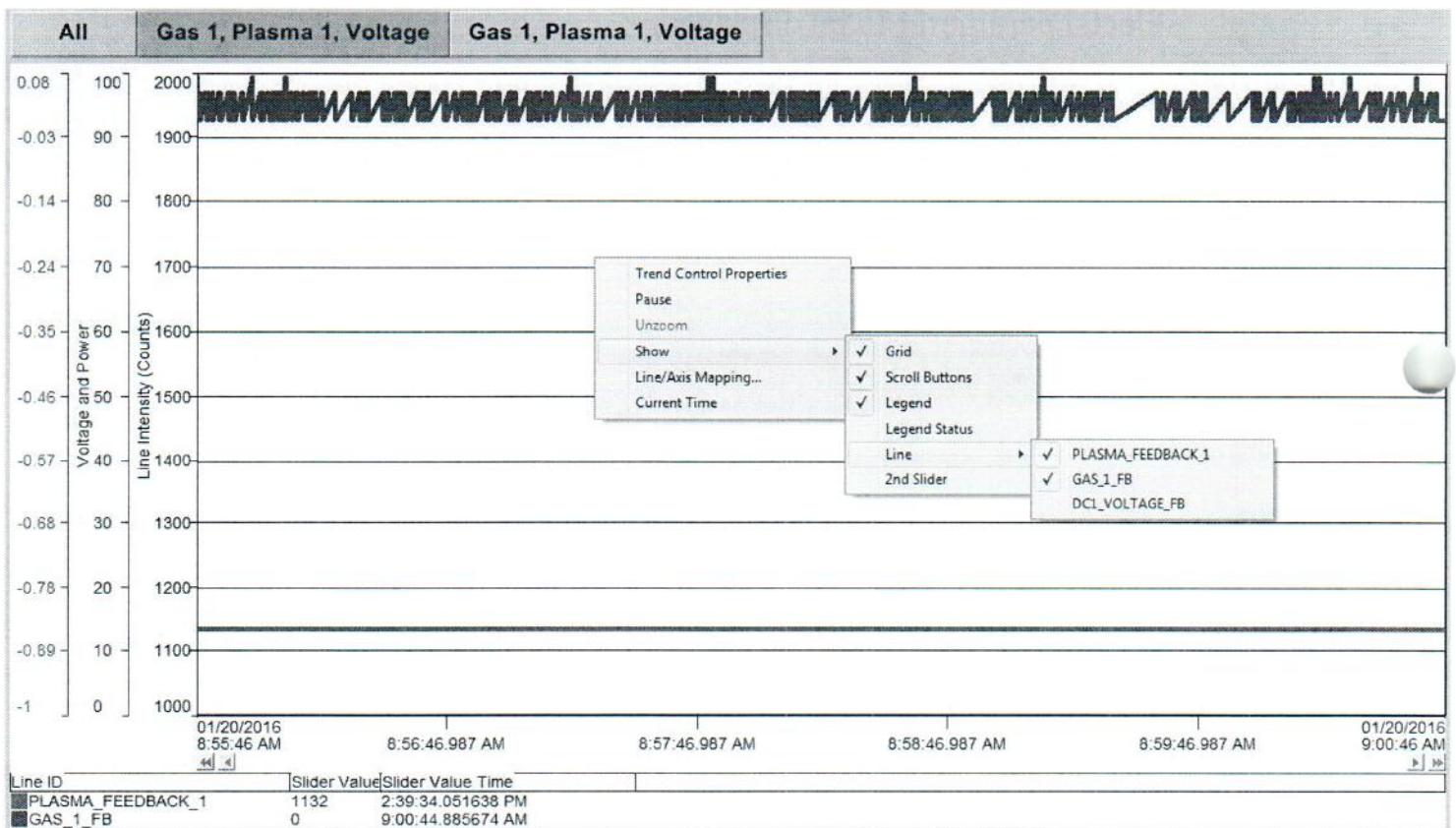
<b>Ramp up and down</b>	Time (ms)	1000	
Bottom	1.0	Increment	0.1
Top	10.0	<span style="border: 1px solid gray; padding: 2px;">Plasma</span>	<span style="border: 1px solid gray; padding: 2px;">Close</span>

- **Plasma**
- **Plot Options** - On the top of the plot display, the operator may toggle between two display modes which are labeled **All** and **Gas 1, Plasma 1, Voltage**.
- **All** - By selecting **All**, the operator may display the feedback of up to 5 Plasma, 2 gas, voltage and power.
- **Plot Parameters** - The operator can select any of these parameters to be displayed.
- **Plot Parameters** - Right click on the plot, select show, select line and select a parameter to be displayed.



- **Gas 1, Plasma 1, Voltage** mode is displayed on the next page.

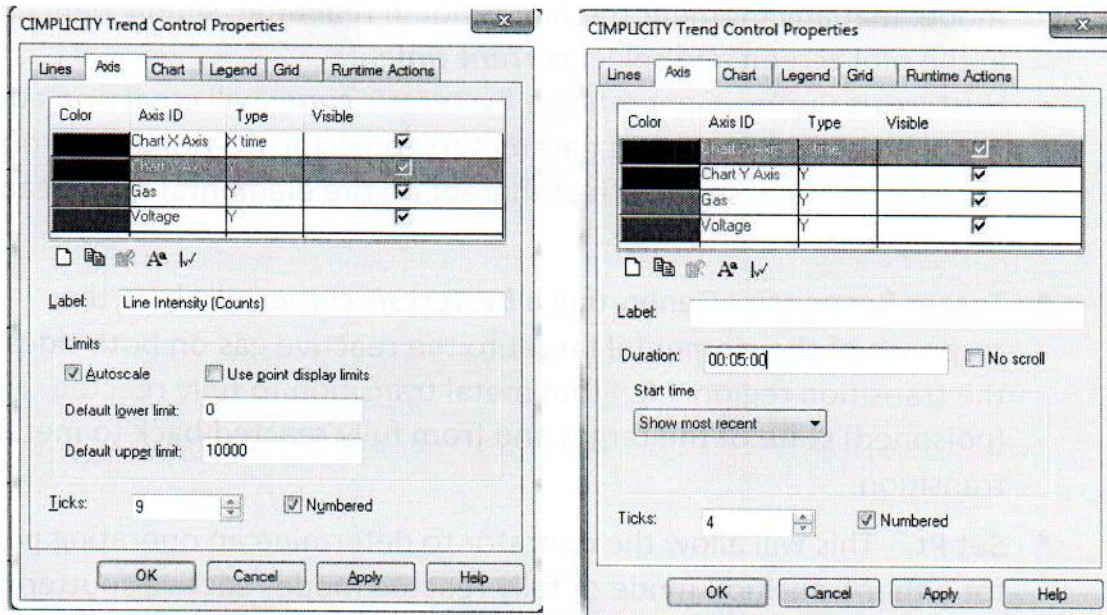
- **Plasma**
- **Gas 1, Plasma 1, Voltage** - By selecting *Gas 1, Plasma 1, Voltage*, the operator may only display the feedback of Plasma 1, Gas 1 and Voltage. This will generate a less complicated display mode.
- **Plot Parameters** - By right clicking, the operator can assign which of these parameters is displayed.
- **Plot Parameter Selection** - Right click on the plot, select show, select line and select a parameter to be displayed.



- **Display Options** - More display options are detailed on the following page.



- **Plasma**
- **Plot Axis** - While the x-axis is fixed to display real time, plasma intensity, gas and voltage are all displayed simultaneously on independent y-axes. Each of the y-axes may display in an auto or fixed setting.
- **Y - Axis** - An auto y-axis display will automatically scale the range of values to the amplitude of the feedback. A fixed y-axis display will keep the range of values constant regardless of the feedback amplitude.
- **Y - Axis** - To change between auto and fixed y-axis right click in the plot window and select **Trend Control Properties** and the following screen will appear.



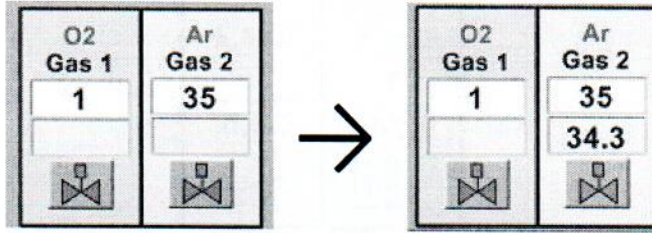
- **Control Options** - Select any of the Axis ID and control options will appear below.
- **Auto Scale** - For each of the **y-axes**, **auto scale** can be generated by selecting the check box. For a **fixed y-axis**, select the desired y-axis parameter, uncheck the auto scale box and select the **default lower** and **upper limits**.
- **X - Axis** - Regarding the **x-axis**, the duration or time scale can be changed by entering the desired value.
- **Apply** - After any trend control properties are changed, select apply and ok to generate a plot with the new parameters.
- **Note:** Once all of the plot parameters are set to desired values as determined by the operator, a hysteresis curve may be generated for process analysis and determination of the **PEM Plasma Set Point**.

## Hysteresis Curve Generation

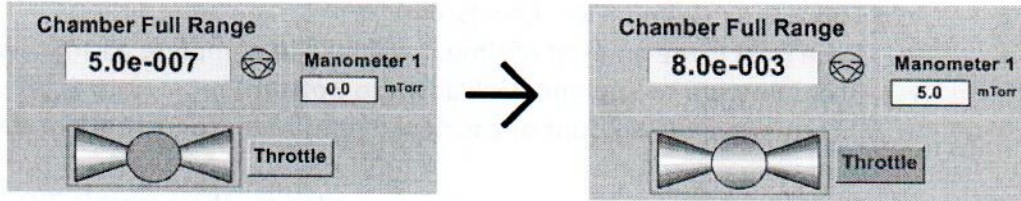
- **Overview**
- **Hysteresis Curve** - Once the system is evacuated to the desired base pressure (at least  $1 \times 10^{-5}$  Torr recommended) and the software parameters are set, the operator is ready to generate a **hysteresis curve**.
- **Note:** While operating sputter deposition and Plasma in manual mode; if at any moment the plot is not in real time, simply right click in the plot screen and select **current time**.
- **Hysteresis Curve** - Generating a **hysteresis curve** will create on auto process based on selected parameters to perform a ramp cycle of reactive gas flow so the effect thereof on the elemental target can be analyzed.
- **Target Response** - Generating a **hysteresis curve** will show the response of the elemental target to the reactive gas on both sides of the transition region. I.E. From metal transition to fully reacted (poisoned) state of the target and from fully reacted back to metal transition.
- **Set Pt.** - This will allow the operator to determine an operating point for either transition mode or fully reacted mode reactive sputtering.
- **Set Pt.** - The operator can the select examine the operating point and select a Plasma Emission set point (spectral counts) for which to use as the reactive gas control parameter in **auto sputter deposition**.
- **Note** - The goal is to pick a particular wavelength to monitor and export that value to the main system control software to be used as the reactive gas control parameter. By monitoring the strength of the signal with different gas flow and or partial pressure, we can control the plasma characteristics.
  
- **Procedure** - Instructional procedures on generating a hysteresis curve begin on the next page.

## Hysteresis Curve Procedure

- **Note:** An example Hysteresis curve for TiO<sub>2</sub> will be represented for step-by-step tutorial purposes. This will be done entirely in the **Plasma Screen**. The **monitoring wavelength** of Titanium Plasma Emission is set to **502 nm**.
1. Make sure the **Plasma screen** is still properly running as explained previously in this chapter.
  2. Start the substrate rotation at a recommended 25% to 50% rotation power.
  3. An operator must introduce a set amount of working gas (i.e. 35 sccm Ar).

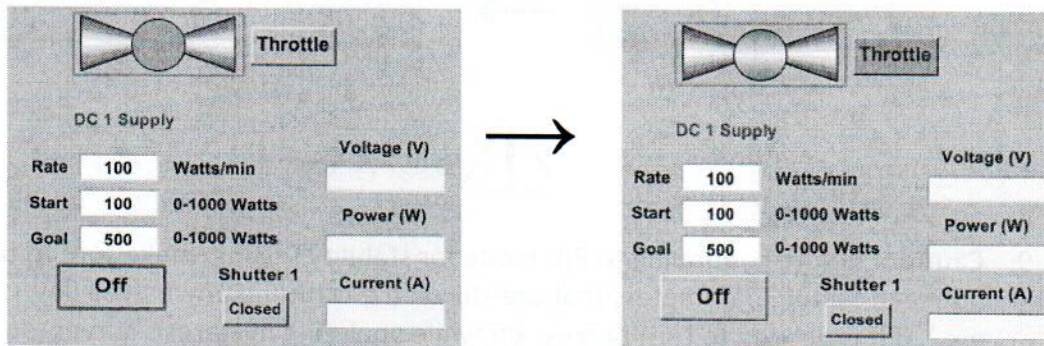


4. Once the working gas flow reaches the selected set point, enable the throttle mode to achieve a fixed pressure (i.e. 5mT). **Note:** the process pressure is read from the capacitance monometer.





5. When the pressure set point is stable, the chamber is ready for cathode ignition. First, select the desired cathode (i.e. Cathode 1-4). Enter in the desired **Set Point Goal Power**, **Start** (ignition) power and **Ramp Rate** and toggle the power switch from **Off** to **On**. The set parameters are example of the TiO<sub>2</sub> process as mentioned above.

**Note:** as a safety interlock, the cathode ignition power switch will only be enabled when the **gas flow** and **throttle** positions are active.



## Hysteresis Curve Procedure

6. As the cathode approaches the power set point, the Plasma Emission signal for **Plasma Feedback 1** will intensify. Once the cathode has reached the power set point and the Titanium target has been clean of oxidation, the Plasma emission signal should be stable.
7. Start the hysteresis procedure by introducing the smallest possible amount of reactive gas (i.e. 1 sccm O<sub>2</sub>). The Titanium Plasma signal will slightly decrease due target reaction: wait for the signal to re-stabilize.

O2 Gas 1	Ar Gas 2
1	35
0.749	34.5
	

8. To generate a hysteresis curve, the **Ramp Up and Down** control will be used.
  - a. Enter the desired parameters for the following set points:
  - b. **Bottom** is the starting gas flow (sccm)
  - c. **Top** is the maximum gas flow (sccm)
  - d. **Time (ms)** is the amount of time it will take to complete a ramp cycle (from **Bottom** to **Top** and back down to **Bottom**)
  - e. **Increment** is the amount of gas flow (sccm) for each increase or decrease step.
  - f. The following parameters are recommended for the example TiO<sub>2</sub> process.
  - g. Once the desired parameters are set, select the **Ramp Up and Down** button to generate a **hysteresis curve**.

<b>Ramp up and down</b>	Time (ms)	3000		
Bottom	Increment	0.1		
Top		10.0	Plasma	Close

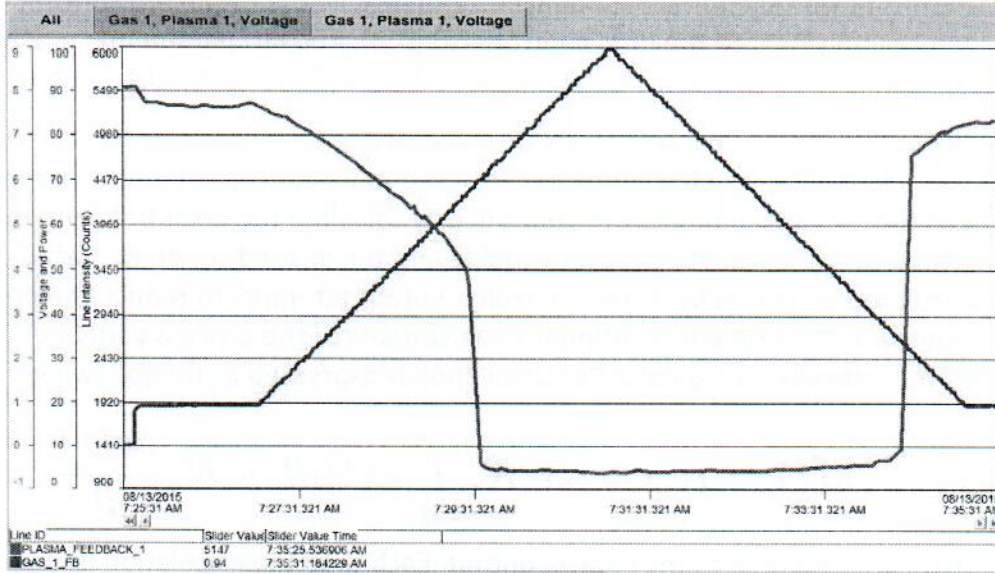


<b>Ramp up and down</b>	Time (ms)	3000		
Bottom	Increment	0.1		
Top		10.0	Plasma	Close

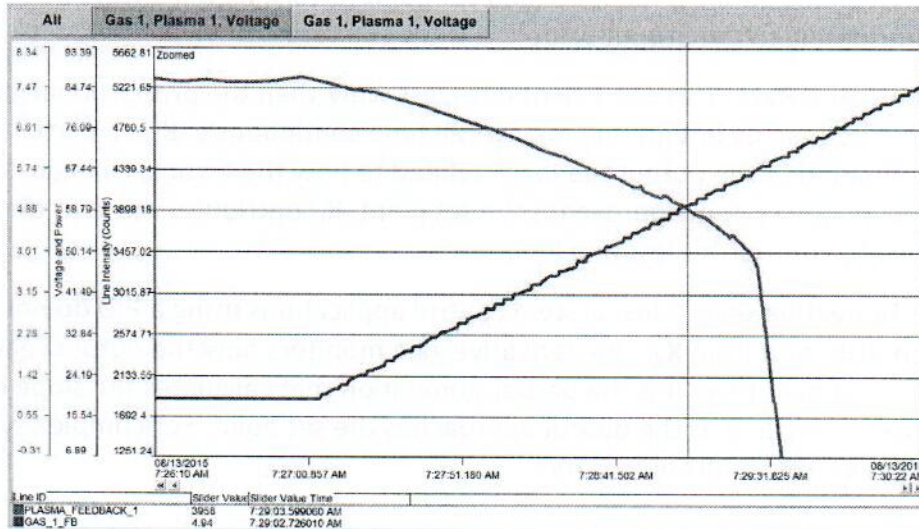


9. **Caution:** Before continuing to PID mode for stability testing, make sure to take notice of system parameters that are still on (i.e. cathode power, gas flows, etc.). If it is desired to turn process off while analyzing hysteresis curves, do so in reverse order of the above instructions.
10. **Note:** Display of the resultant hysteresis curve is provided on the following page.

- Hysteresis Curve** - The resultant hysteresis curve is displayed below. The purple line represents the ramp cycle of the reactive gas flow **Gas 1** ( $O_2$ ) from 1 sccm to 10 sccm. The red line represents the Plasma emission of **Plasma Feedback 1** as effected by the reactive gas.
- Target Response** - At the bottom of the Plasma Feedback it can be seen that the Titanium target is fully poisoned in this region.



- Set Pt.** - To select a Plasma emission **set point** it may be useful to zoom in on the **transition** region. To do so, left click and drag over the area of interest, unclick and it will automatically zoom. To un-zoom, simply right click and select unzoom.
- Cursor** - On the right side of the plot window, a cursor (represented by the vertical black line) can be dragged to display the selected values of the plot screen for which the cursor is placed. These values are displayed just below the plot. **See Example Below**



## PID MODE

### PID Overview

- **Note:** In the Plasma Screen control software, PID Mode is used to test the control stability of a set point as selected from the hysteresis curve. It is important for the operator to familiarize themselves with the PID concept before applying it to the PEM control system. Tested PID parameters are also to be used in auto recipe generation as described in the **Auto Mode** section.
- **PID: Proportional, Integral, Derivatives Control** is a process control method that uses a feedback loop to reduce the error around a desired set point. The three components are summated to continuously calculate the error between the measured feedback of a process variable and the desired set point. Based on the calculated error feedback, the controller output attempts to reduce the error repeatedly over time by continuous adjustments of the process variable. Mathematically, the general PID calculation is expressed as the following.

$$u(t) = K_p e(t) + K_i \int_0^t e(t) dt + K_d \frac{de(t)}{dt}$$

- **u(t):** This is defined as the control output. Each time the calculation is made; this new value is used as a control output to reduce error from the set point.
- **t – sample period:** time & instantaneous time dependent upon the associated coefficient.
- **e:** error: expressed as the absolute value of (set point – feedback value)
- **K<sub>p</sub>: Proportional Gain:** This coefficient does most of the work. It takes the error value and multiplies it by the K<sub>p</sub> value (i.e. 100) to become a partial summation to the output value. It does this in respect to instantons time. It is possible for a system to be run only by the K<sub>p</sub>. As long as there is an occurring difference between the variable feedback and set point (error), K<sub>p</sub> will always be contributing to the output value.
- **K<sub>i</sub>: Integral Rate:** This coefficient does less work than the proportional gain K<sub>p</sub> but it does so by integrating the overall time continuously. K<sub>i</sub> is a partial summation of the output feedback related to how the error is changing over time. As the system approaches the set point, K<sub>i</sub> contributes more to the output while K<sub>p</sub> contributes less and less.
- **K<sub>d</sub>: Derivative Gain:** Most system control applications using a PID do not require a contribution from K<sub>d</sub>. The derivative gain monitors how the output reaches the set point over time. It is the partial summation that calculates the slope of the output over time as the output approaches the set point. For complex systems, K<sub>d</sub> can be absolutely necessary.

- **PID Overview**
- **Additional PID Parameters:** The remaining PID parameters are related to Mass Flow Controller (MFC) properties and the reactive gas flow.
- **Mass Flow Controller:** The MFC of each system corresponds to the following PID set points and display values: Upper Clamp, Lower Clamp and PID Control Addition. Calculating values for these parameters is dependent upon the max flow of the MFC (sccm) and the initial reactive gas flow set point (i.e. 1 sccm) which is termed **Fixed Gas Addition**.
- **Fixed Gas Addition:** This value corresponds to the fixed reactive gas flow set point (i.e. 1 sccm) upon which PID control is initiated.
- **PID Control Addition:** The PID control addition display is an integer representing the calculated PID output to control the reactive gas. The integer is used to set the upper and lower clamps of the PID control if necessary.
- **Lower Clamp:** This values sets the minimum allowable value of the the PID output, it is based on the **fixed gas addition**.
- **Upper Clamp:** This values sets the maximum allowable value of the the PID output, it is based on the **fixed gas addition**.
- **Sample Period:** The sample period is set to adjust the time interval for which a calculation of set point error. I.E. a sample period of 10ms means that an error calculation from the set point is made every 10ms for the PID control output.

- **PID Overview**

- **Note:** An example Hysteresis curve for TiO<sub>2</sub> will be represented for step-by-step tutorial purposes. This will be done entirely in the **Plasma Screen**. The **monitoring wavelength** of **Titanium Plasma Emission** is set to **365 nm**.  
**Note: PID Mode** is controlled in the **Plasma Software Screen**.
- **Set Point:** Once the desired **set point** for the **Titanium Plasma Emission** is selected by analysis of the **hysteresis curve** as stated previously, it will be entered into the **PID Mode: Set point (counts)**.
- **PID:** Values are to be entered into the **Proportional Gain, Integral Rate & Derivative** selections. These values will be different for every material subsequent process. Values of 100 for both the Proportional Gain and Integral Rate are a good start. Depending upon process sensitivity, these values may each need to be increased or decreased.
- **Direction:** Regarding direction, this parameter is dependent upon the Plasma Emission selected for reactive gas control. There are two options for direction: **direct** and **inverse** as described below.
- **Direct:** Regarding the TiO<sub>2</sub> process, if the O<sub>2</sub> Plasma emission is selected for control, the direction should be set to **direct**. This is because the O<sub>2</sub> plasma emission is **directly** effected by the reactive O<sub>2</sub> gas flow (sccm).
- **Inverse:** Also regarding the TiO<sub>2</sub> process, if the Ti plasma emission is selected for control, the direction should be set for **inverse**. This is because the Ti plasma emission is **inversely** effected by the O<sub>2</sub> gas flow (sccm).
- **Example PID:** An example is below is provided below. Since values around 1,000 spectral counts are noise occurring when no plasma is being emitted, a set point above 1,000 is the lowest controllable value.

PID mode will control Gas <input type="text" value="4"/>	
based on Plasma line 1	
Direction	<input type="radio"/> Inverse <input type="radio"/> PID Mode
Setpoint (counts)	<input type="text" value="1450"/>
Proportional Gain	<input type="text" value="100"/>
Integral Rate	<input type="text" value="100"/>
Derivative	<input type="text" value="7"/>
Lower Clamp	<input type="text" value="-500"/>
Upper Clamp	<input type="text" value="1800"/>
Sample Period	<input type="text" value="10"/>

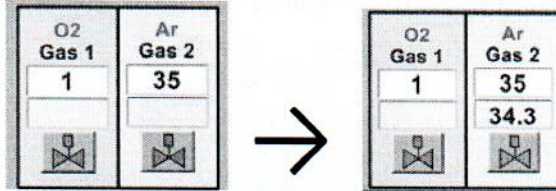
Fixed Gas Addition	<input type="text" value="324"/>
PID control Addition	<input type="text" value="0"/>

- **Note:** Systematic instruction procedure for operating in manual PID mode is detailed on the following page.

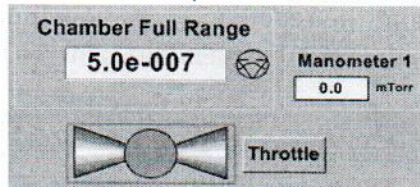


## PID Mode Procedure

1. Make sure the **PEM Program** screen is still properly running as explained previously in this chapter.
2. Start the substrate rotation at a recommended 25% to 50% rotation power.
3. An operator must introduce a set amount of working gas (i.e. 35 sccm Ar).

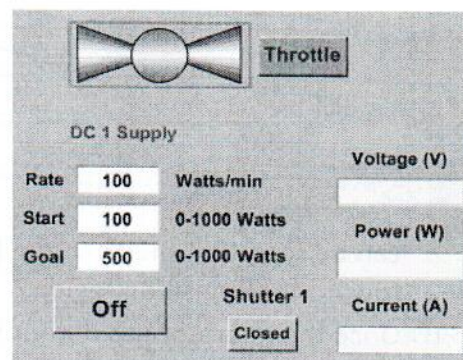
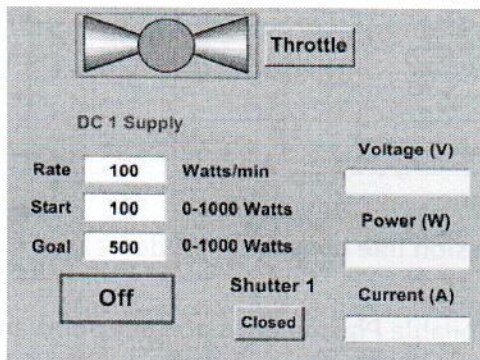


4. Once the working gas flow reaches the selected set point, enable the throttle mode to achieve a fixed pressure (i.e. 5mT). **Note:** the process pressure is read from the capacitance monometer.



5. When the pressure set point is stable, the chamber is ready for cathode ignition. First, select the desired cathode (i.e. Cathode 1-4). Enter in the desired Set Point **Goal Power**, **Start** (ignition) power and **Ramp Rate** and toggle the power switch from **Off** to **On**. The set parameters are example of the TiO<sub>2</sub> process as mentioned above.

Note: as a safety interlock, the cathode ignition power switch will only be enabled when the **gas flow** and **throttle** positions are active.



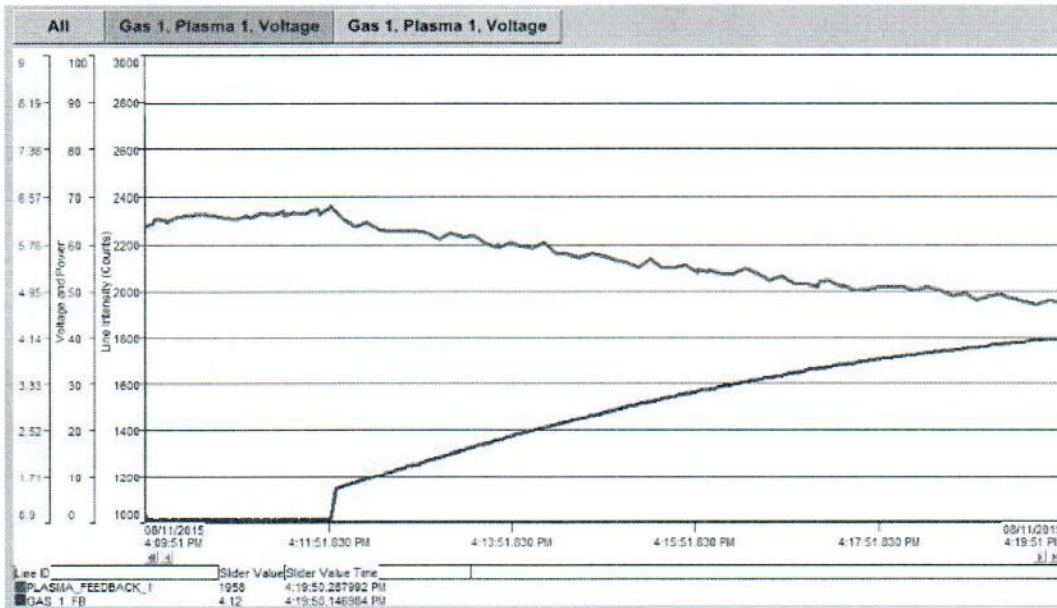
6. As the cathode approaches the power set point, the Plasma Emission signal for **Plasma Feedback 1** will intensify. Once the cathode has reached the power set point and the Titanium target has been clean of oxidation, the Plasma emission signal should be stable.

7. Select the desired PID parameters for stability testing as instructed in **Manual Mode**
8. To begin reactive gas control via PEM feedback, click on the **PID mode** button. For the given process example, the following parameters will be a good starting point. The **inverse** direction is chosen since the Titanium plasma emission is used as the reactive gas PID control parameter for the given TiO<sub>2</sub> process example.

PID mode will control Gas 1 based on Plasma line 1

4000	PID Mode
Setpoint (counts)	
Proportional Gain	100
Integral Rate	100
Direction	Inverse

9. If the PID parameters are selected right, a typical manual **PID Mode** will appear similar to the screen below. It can be seen that the reactive O<sub>2</sub> gas flow (purple display line) is approaching the set point. As the **reactive gas flow** is increased, it



can be seen that the Titanium **Plasma Emission line** consequently decreases toward the transition region.

10. Once the operator has determined a controllable **Plasma emission set point** and **PID** values for which to control the reactive gas, these parameters can be integrated into an automated process.
11. Instructional Procedure on the **Auto Deposition Recipe integration of Plasma Emission Monitoring** are provided in the next chapter.
12. **Caution:** Before continuing to **Auto mode** for recipe integration, turn off all process parameters in the proper order (i.e Power, gas flow, throttle valve etc).



## INTEGRATING TO PROCESS AUTOMATION

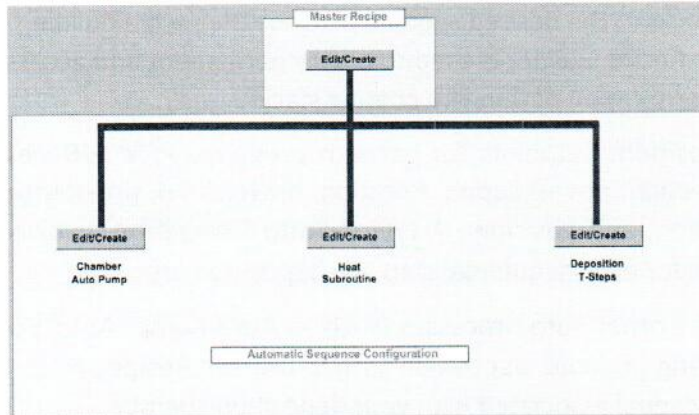
### AUTO MODE

### AUTO DEPOSITION OVERVIEW

- **Auto Recipe Generation:** Automatic processes are constructed by linking recipe Building Blocks in the desired sequence. Once the recipe building blocks are created, the Master Recipe Builder is used to create permanent and accessible recipe files for use in the generation of thin film coating stacks.
- **Auto Deposition:** Setpoints for vacuum pressure, *PEM PID parameters* heat, source selection, power supply selection, power level, pre-sputter time, sputter time, rotation, and gas flow. A typical **Auto Deposition** involves selecting the parameters for each sequential step in a deposition process.
- **Note:** For all other auto processes (such as **Auto Pump**, **Auto Vent** etc.) as well as integrating an auto deposition into a **Master Recipe**, refer to the Denton Operator Manual associated with your deposition chamber.
- **Typical PEM Auto Deposition Recipe Configuration:** Auto Deposition is configured in an excel spreadsheet with the following capabilities.
  - Step Time (sec)
  - Rotational Velocity (constant rot %)
  - Minimum Vacuum Set Point (Torr)
  - Chamber Heat Set Point (°C)
  - Heater Rate (°C/min)
  - Ignition Pressure (mTorr)
  - VAT Throttle Control
  - Gas 1 – Setpoint (sccm) , Gas 2 – Setpoint (sccm) etc.
  - DC Ramp Rate (Watts/min)
  - DC Starting Set Point (Watts)
  - DC Goal Set Point (Watts)
  - Cathode 1 Shutter (Open/Close)
  - **PEM Wavelength to Control (nm)**
  - **Gas 1 PID count Set Point (starting sccm)**
  - **Control Mode (PID direction → inverse/direct)**
  - **Proportional Gain, Integral Rate, Derivative etc.**
  - RF Bias – Set Point (Watts)
  - End Process (Yes) (If not leave blank)
- **Note: PEM Auto Deposition:** Instructional procedure for developing an Auto recipe with integrated PEM control is detailed in the following pages.

## AUTO MODE PROCEDURE

1. **Note: Important:** The operator must verify that the **PEM Program** and **Plasma Screen** from manual mode are left open and running during auto deposition.
2. On the top left hand corner of the Overview Screen, select the system control software into **Auto Mode**.
3. On the bottom of the **overview** screen in the toolbar section, select **recipe**. The following screen will appear.



4. From this screen, select **Edit/Create Deposition T-Steps** auto deposition on the right hand side and the following blank excel spreadsheet will appear.

Step Number	T000	T001	T002	T003	T004	T005	T006	T007	T008	T009	T010	T011
0 Step Time (sec)												
1 Rotation Velocity (constant rot %)												
2 Min Vacuum Setpoint (Torr)												
3 Chamber Heat Setpoint (°Celsius)												
4 Heater Rate (Degrees/min)												
5 Ignition Pressure (mTorr)												
6 VAT Throttle Control												
7 Gas 1 - Setpoint (sccm)												
8 Gas 2 - Setpoint (sccm)												
9 DC Ramp Rate (Watts/min)												
10 DC Starting Setpoint (Watts)												
11 DC Goal Setpoint (Watts)												
12 Cathode 1 Shutter												
13 Wavelength to Control												
14 Gas 1 - PID count setpoint												
15 Control Mode												
16 Proportional Gain												
17 Integral Rate												
18 RF Bias - Setpoint (Watts)												
19 End Process (Yes)												
20 Check Logic Validity												
21 File Directory												

## Auto Mode Procedure

5. The **Automatic deposition** process follows a programmable timeline. The programming of the automatic process is organized into a spreadsheet format. Each column is a programmed step. The data in the column is active for the time programmed into the Step Time row.
6. Enter the desired process parameters. An example TiO<sub>2</sub> process **auto deposition** t-step spreadsheet is given below.

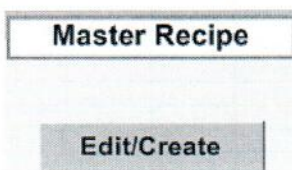
Step Number	T000	T001	T002	T003	T004	T005	T006	T007
0 Step Time (sec)	5	10	300	600	1200	5		
1 Rotation Velocity (constant rot %)	25	25	25	25	25			
3 Min Vacuum Setpoint (Torr)	5.0E-06							
4 Chamber Heat Setpoint (°Celsius)								
5 Heater Rate (Degrees/min)								
7 Ignition Pressure (mTorr)		25						
8 VAT Throttle Control		YES	YES	YES	YES			
11 Gas 1 - Setpoint (sccm)				1	1			
12 Gas 2 - Setpoint (sccm)		5	35	35	35			
16 DC Ramp Rate (Watts/min)			300					
17 DC Starting Setpoint (Watts)		150	150					
18 DC Goal Setpoint (Watts)		150	500	500	500			
19 Cathode 1 Shutter						Open		
21 Wavelength to Control		502	502	502	502			
22 Gas 1 - PID count setpoint				2700	2700			
23 Control Mode				Inverse	Inverse			
24 Proportional Gain			300	300	300	300		
25 Integral Rate			250	250	250	250		
27 RF Bias - Setpoint (Watts)								
33 End Process (Yes)						YES		
Check Logic Validity						YES		
<b>File Directory</b> C:\Cimplicity\Discovery\Deposition\								

## Auto Mode Procedure

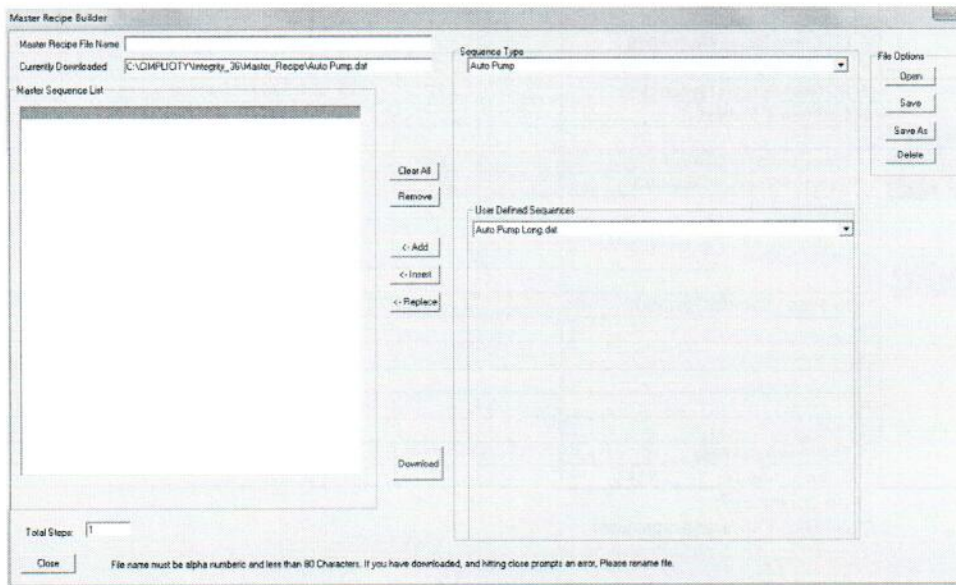
- From here, an emphasis on the **PEM PID** parameters is given for t-step generation. The following parameters should be taken from Manual mode.

21	<b>Wavelength to Control</b>	502
22	<b>Gas 1 - PID count setpoint</b>	4000
23	<b>Control Mode</b>	Inverse
24	<b>Proportional Gain</b>	100
25	<b>Integral Rate</b>	100

- Once all of the t-step parameters are entered and the formation of the desired **auto deposition** is completed, save the file by selecting save as. Enter a file name and save. (i.e. **PEM TiO<sub>2</sub>**)
- After the **auto deposition recipe** has been saved on file, exit the excel spreadsheet and return to the **recipe** screen.
- To start an auto deposition, it must first be downloaded from **recipe** to **overview**. To do so, select **Master Recipe** at the top of the **Recipe** screen.

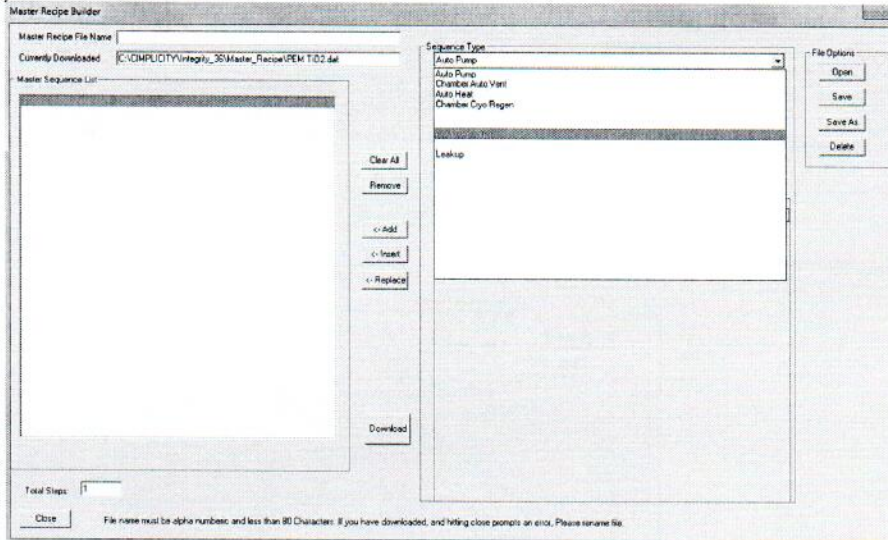


- Clicking on the **Edit/Create** button opens the blank **Master Recipe Builder** screen as displayed below.

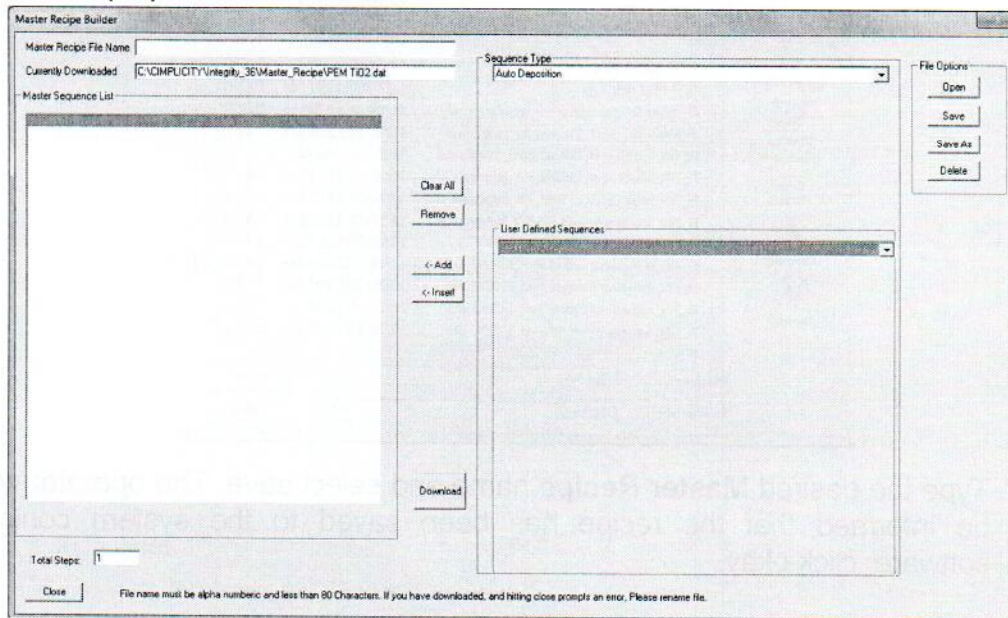


## Auto Mode Procedure

12. From the blank **Master Recipe Builder** screen, open the dropdown box under the **Sequence Type** option.

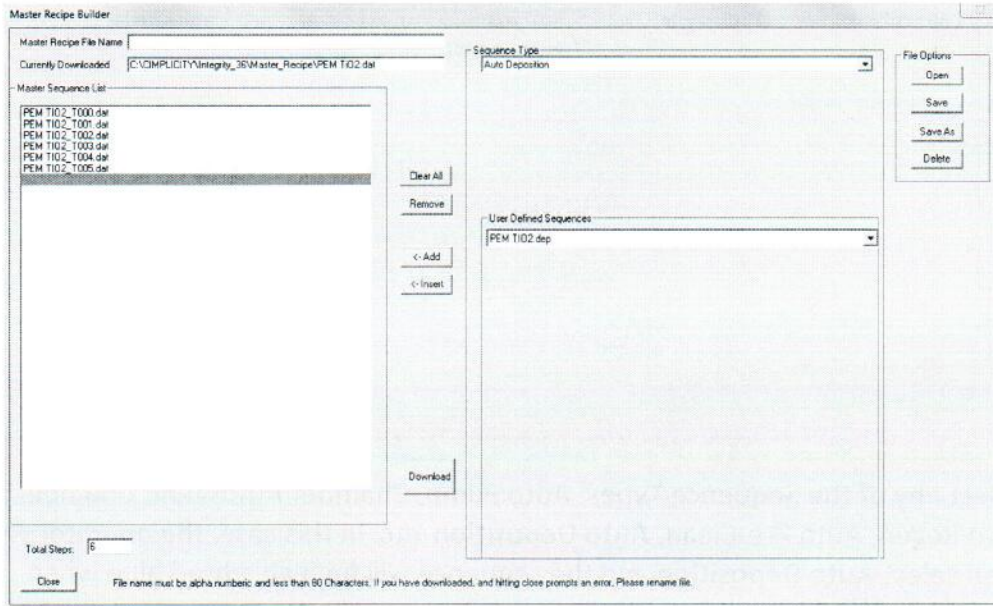


13. Select any of the **Sequence Types**: Auto Pump, Chamber Auto Vent, Chamber Cryo Regen, Auto Pre-Clean, **Auto Deposition** etc. In this case, the operator will then select **Auto Deposition** and the sequence will be highlighted blue when properly selected.
14. After selecting the **Sequence type** (i.e. **Auto Deposition**), open the dropdown box under the **User Defined Sequences** as displayed below. This is where the operator will find the **Auto Deposition Recipe with integrated PEM control** that was previously created. (i.e. **PEM TiO<sub>2</sub>**). Select the **Recipe** by highlighting in blue as displayed below.

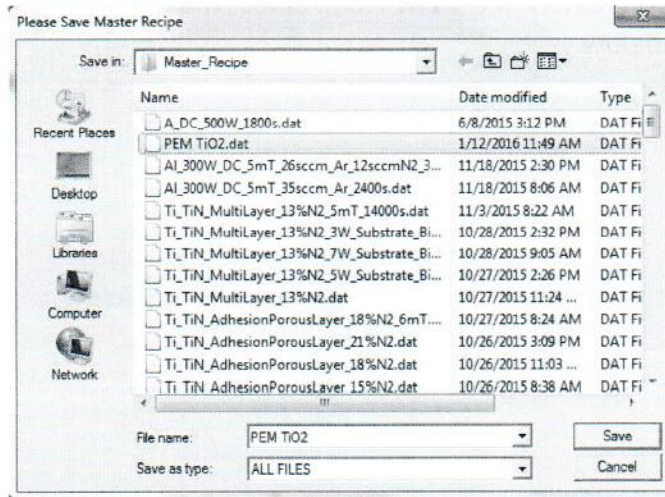


## Auto Mode Procedure

15. Once the **Auto Deposition Recipe** is selected (**PEM TiO<sub>2</sub>**), move that recipe to the **Master Sequence List** box by clicking **Add** or **Insert**. The **t-steps** will then appear in the **Master Sequence List** box on the left hand side similar to the display below.



16. Next, select **Download** to add the **New Master Recipe** to the **Overview** screen for process initiation. The **Download** command will prompt the operator to save the **New Master Recipe** as follows.



17. Type the desired **Master Recipe** name and select save. The operator will be informed that the recipe has been saved to the system control software: click okay.



## Auto Mode Procedure

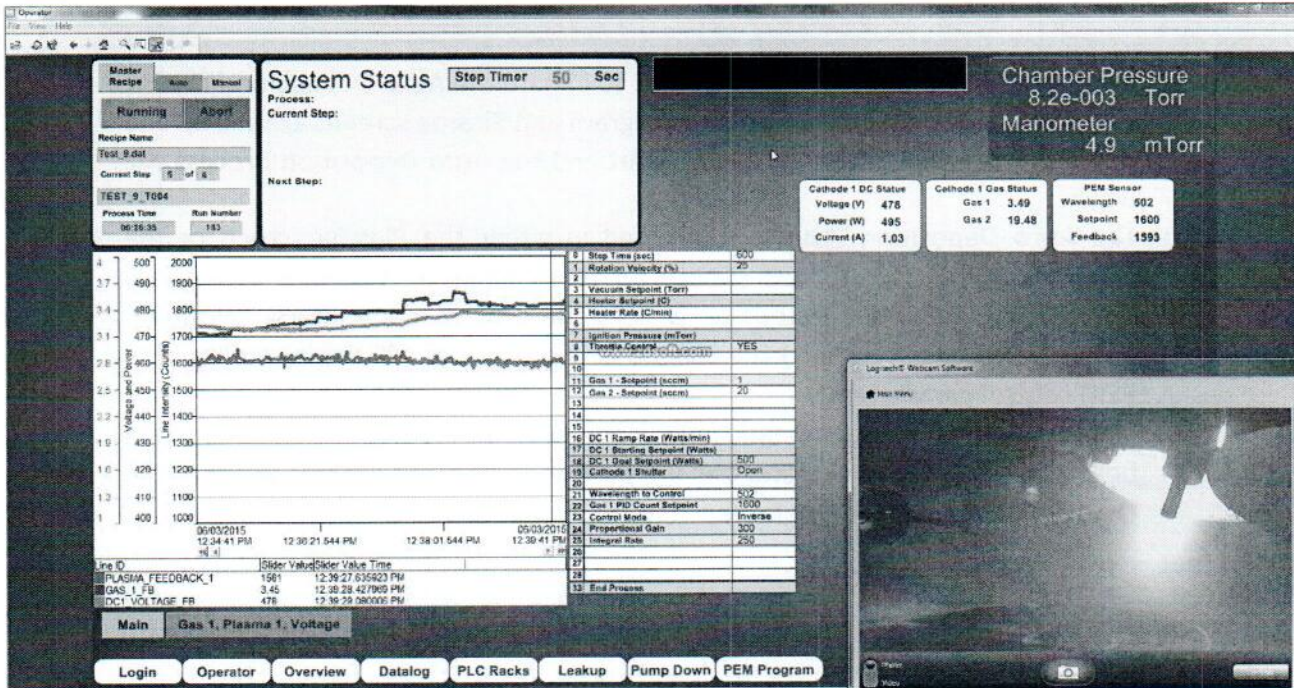
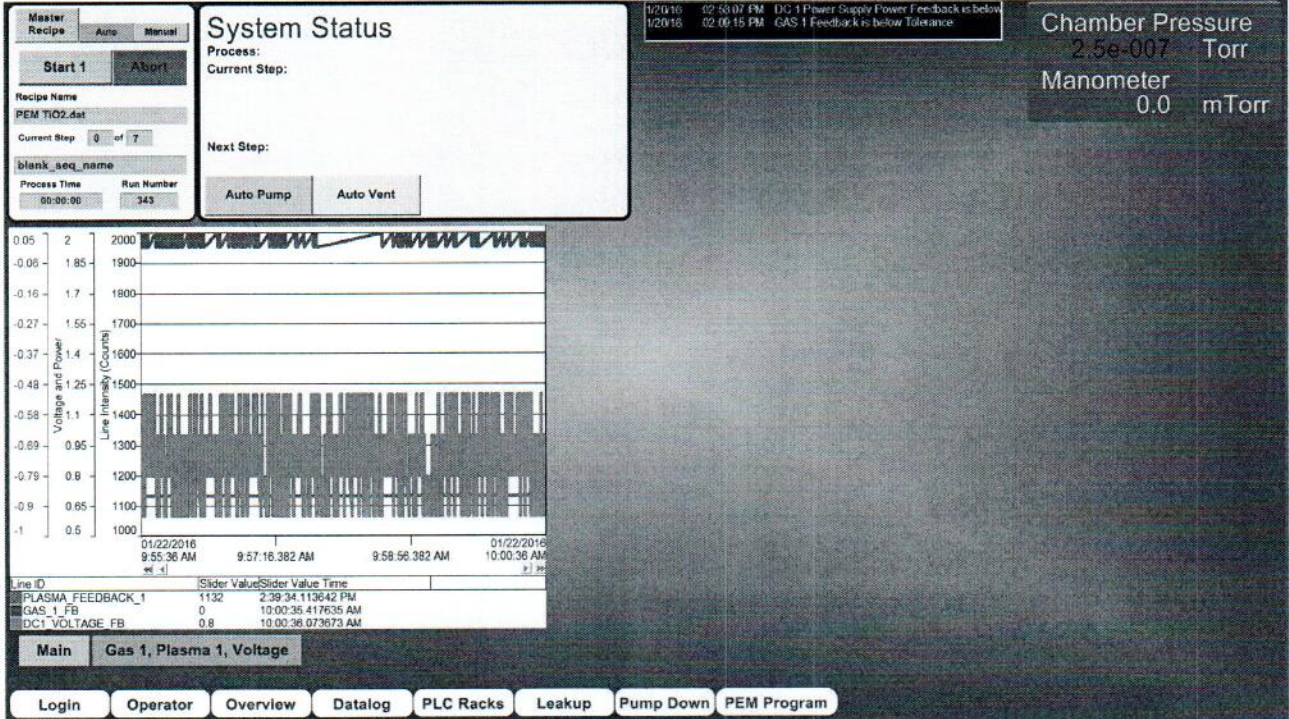
18. Now that the recipe is saved, the operator can exit the recipe screens and return to the main **Overview** screen. The recipe should now be displayed in the top left of the screen under the **Recipe Name** section as displayed below.

The screenshot shows the 'System Control' interface. At the top, there are three tabs: 'Auto', 'Manual', and 'Service'. Below them is a 'Master Recipe' button. A 'Start' button is highlighted with a white border, and an 'Abort' button is to its right. The 'Recipe Name' is 'PEM TiO2.dat'. The 'Current Step' is '0 of 6'. The 'blank\_seq\_name' field is empty. The 'Process Time' is '00:00:00' and the 'Run Number' is '343'. Below this is a list of status indicators with checkboxes: 'Door Switch', 'Skin Switch', 'Vacuum Switch', 'Cathode 1 Water', and 'RF Bias Water'. At the bottom, the 'Wafer ID' is 'PEM TiO2'.

19. **Note: Important:** Before selecting **Start** to initiate the **Auto Deposition Process**, double check to make sure the **PEM Program** and **Plasma** screens are still up and running. Once that is verified, select **Start** and the **Auto Deposition Process** will begin.
20. The **Auto Deposition Process** monitored in either the **Plasma** screen or the **Operator** screen (see following page for reference). The **Operator** screen will display the **sequential T-steps** as well as the process parameters (gas flows, power, voltage, current etc.) The **Plasma** screen however will display more of the **PEM** control parameters.
21. In the event that any systematic faults occur during **auto deposition**, alarms will be presented and the operator has the ability to **Abort** the process in either the **Overview** screen or the **Operator** screen.
22. **Note: Important:** for more auto deposition recipe options regarding **editing recipes**, incorporating other recipes etc.; refer to the Denton manual associated with your vacuum deposition chamber.

## OPERATOR SCREEN

- Operator: Typical operator screens are provided below for reference.



## PROCESS APPLICATION

### PROCESS CONTROL

#### Reactive Gas Control

It is often difficult to operate a reactive sputtering process in transition mode with most deposition chambers. Operating in this mode requires precise control of the reactive gas. The technique of reactive gas control for reactive sputtering has a strong influence on deposition rates, process yield and a complex variety of thin film properties. It is therefore a desirable approach in overcoming the process difficulties and setbacks inherent with sputtering elemental targets in a fully reacted compound state. Plasma Emission Monitoring is a technique used for control of the reactive gas in reactive sputtering processes.

#### Plasma Emission Monitoring

Plasma Emission Monitoring is a technique used in reactive sputtering to improve both the control of a thin film process as well as the analysis of process parameters. An in-vacuum fiber optic sensor monitors the plasma species among a range of wavelengths of a specific sputtering process. The range of wavelengths (nm) for plasma emissions monitoring is generally from the UV to the near IR. Most process materials emit plasma species during reactive sputtering in this range of wavelengths. The feedback of a specific wavelength associated with; either the plasma emission of the elemental target or the reactive gas used to control the reactive gas input through a single closed loop PID.

#### Denton Vacuum PEM

Denton Vacuum offers an innovative solution to the difficulties of reactive magnetron sputtering with the integration of Denton Vacuum PEM 1.0. Denton Vacuum has developed PEM as an innovation toward the control of reactive gas for reactive sputtering vacuum deposition equipment. As a budget friendly, upgrade to any Denton sputter deposition vacuum chamber, Denton PEM offers a more sophisticated process control system for reactive sputtering. Denton Vacuum PEM can be operated for both process development and analysis or automated for production use and application.

## **Denton Vacuum PEM**

Denton Vacuum has developed Denton Vacuum PEM a plasma emission monitor available for full auto process integration. Denton Vacuum PEM enables the reactive sputtering process to operate in a desired, controlled deposition mode as determined by the user. Denton Vacuum PEM includes in vacuum fiber optic probes equipped with protective encasings, high precision in vacuum fiber optic cables, fiber optic vacuum feed through and a USB UV-Vis spectrometer equipped for integrated process control with Denton Vacuum's automation software packages.

## **Material Application**

The integration of Denton Vacuum PEM for reactive sputtering has a broad range of process applications. It can be used as a reactive gas control system for the following reactively sputtered coatings: Metal-oxides such as  $\text{TiO}_2$  and  $\text{SiO}_2$  for decorative, dielectric and optical coatings. Nitrides like TiN and ZrN for protective hard coatings. Conductive clear coatings and medical coatings are other applications for PEM as well. As previously mentioned, the PID single loop control method used by plasma emission monitoring will be different for every process material application. The configuration of each vacuum deposition chamber will also strongly influence the process parameters of PEM for each material application and therefore, it is up to the operator to optimize the respective process control parameters.

## **ANALYTICAL USE**

### **Process Analysis**

In addition to the control of reactive gas in-situ of process automation, Denton Vacuum PEM may also be used for process analysis. Because Denton Vacuum's software control enables the user to data log automated process runs, analysis of each data log may be applied. With minor similarities to the use of a residual gas analyzer, Denton Vacuum PEM enables the operator for process chamber analysis by use of the plasma emission spectra.

### **Data Log**

Process data logs can record the intensity of plasma emission for up to five wavelengths in units of nm. Although PEM may only control the reactive gas by the feedback of one plasma emission wavelength, an additional four wavelengths can be also be data logged in-situ of process automation. By viewing the trend of each plasma emission wavelength value for a given process run data log, correlations can be made and applied to process analysis. For example, a reactive sputtering process of elemental Titanium with reactive gas Nitrogen, PEM can be used to analyze any residual chamber Oxygen present in the reactive plasma emission in situ of a deposition process.

### **Target Poisoning**

Another analytical application of Denton Vacuum PEM involves **target poisoning**. As described in chapter 4, generating a hysteresis curve from a reactive gas ramp cycle in manual mode provides insight to elemental target modes for reactive sputtering. By analyzing a plasma emission trend line in of an elemental target as effected by the reactive gas, the operator can identify a flow rate limit of that reactive gas in which the elemental target enters a state of a fully reacted compound. This characteristic of an elemental sputter target is also referred to as the shift from a transition state to a fully **poisoned** state. Inherent with reactive sputtering, the amount of reactive gas needed to fully poison a given elemental target varies dependent upon process parameters such as system pumping speed, base vacuum pressure, process pressure and cathode voltage, current etc.

## TROUBLESHOOTING & MAINTENANCE

### HARDWARE



- **Damaged Fiber Optic Cables**
  - In the event that any of the Fiber Optic Components are damaged or coated, contact Denton Vacuum LLC for details on replacement or repair components and procedures.
- **Fiber Optic Vacuum Feed Through**
  - If a leak is detected at the connection of the **VFT**, replace the 2  $\frac{3}{4}$  " copper O-ring.
  - In the event that the Fiber Optic Vacuum Feedthrough is damaged or coated, contact Denton Vacuum for details on replacement or repair VFT's and procedures.
- **Coated Plasma Sensor**
  - In the event that a Plasma Sensor has been coated and is therefore weakening the Plasma Emission Signal, it can be cleaned.
  - Cleaning procedure of the Plasma Sensor must be done with **extreme caution** as these parts are costly.
  - The honeycomb fixture at the Plasma Sensor end is in place to protect all of the optical components that cannot be cleaned.
  - To clean the honey comb fixture, carefully remove it from the sensor. To remove coating from the fixture, it can be bead blasted. Although the fixture is relatively robust, bead blasting must be done with **extreme caution**. It is recommended to hold the fixture in a secure fashion while bead blasting.
  - For any questions or concerns regarding the Plasma Sensor or replacement parts, please contact **Denton Vacuum**.



## Hardware

### ▪ **Low Plasma Emission Signal**

- First, check all fiber optic connections and fiber optic ends to ensure that no parts are coated or damaged.
- The **Plasma Sensor** contains a protective honey comb fixture that builds up coating over time. This will cause a weaker **Plasma Emission Signal** and eventually the Plasma Sensor will need to be cleaned. See the above section ***Coated Plasma Sensor*** for details.
- In the **PEM Program** Screen, the **integration time** and **scans to average** can be adjusted to produce a better signal.  
Note: increasing the scan time to produce a stronger signal can reduce the speed of the feedback loop to control the reactive gas. This may cause undesired process control.
- Tuning the **integration time** and **scans to average** options is a balance between optimizing **Plasma Emission Signal** and the speed of the feedback loop for reactive gas control.
- Other process parameters such as an increase in cathode Power, process pressure and Ar gas flow can all strengthen the **Plasma Emission signal**.

### ▪ **No Plasma Emission Signal Feedback**

- Again, check all fiber optic components, ends and connections to ensure that no parts are coated or damaged.
- Check the condition of all data acquiring components.
- Make sure the necessary software screens are opened in the proper sequence as described by chapter 4.
- Check to make sure the spectrometer is on and connected to the PC via the USB port.
- Verify that communication is established between the spectrometer and the PEM Program software screen.

## Hardware

- **USB Spectrometer**

- Check to make sure the spectrometer is on and connected to the PC via the USB port.
- If there is no communication feedback from the spectrometer in the PEM Program software, close the program. Then, disconnect and reconnect the spectrometer.
- Ensure that the software screens are opened in proper sequence as detailed in chapter 4.
- If the USB is malfunctioned beyond repair, contact **Denton Vacuum** for replacement.

- **Replacement Parts**

- In the event that any PEM hardware components are in need of repair or replacement, please contact **Denton Vacuum** for details and procedures.



## SOFTWARE

### ▪ Auto Mode PEM

- In the event that PEM control is not initiating in auto mode, diagnose the following.
- Check that all of the proper software screens are opened in proper sequence as mentioned in chapter 4.
- Make sure the PEM parameter for the selected **recipe** are properly entered and selected.
- In the recipe **T-Step** labeled **Control Mode**, make sure the entry box is either selected to **Inverse** or **Direct**; starting at the **T-Step** in which the operator desires to begin **PEM control**.
- Check to make sure the desired recipe is properly downloaded to the **Master Recipe** builder.
- Check to make sure there is no damage or coating on any fiber optical components and that they are all connected appropriately.

### ▪ Manual Mode PEM

- In the event that PEM control is not initiating in manual mode, diagnose the following.
- First, the operator should familiarize themselves with the **Manual Mode** section chapter 4.
- Check that all of the proper software screens are opened in proper sequence as mentioned in chapter 4.
- Make sure that a signal is established in the **PEM Program** software for the desired control wavelength.
- Make sure that a minimum reactive gas set point is initiated before selecting **PEM Control** via **PID Mode**.
- For further assistance in trouble shooting with the software applications, please contact **Denton Vacuum**.

## Software

- **Plasma & PEM Program Screens**
  - In the event that PEM software screens are not responsive, make sure the main overview control screen is set to manual mode.
  - In Auto mode, the screens must be left open for operation.
  - If further problems arise, please contact **Denton Vacuum** for assistance.
  
- **Lacking Spectrometer Detection**
  - Ensure that the software screens are opened in proper sequence as detailed in chapter 4.
  - In the event the **PEM Program** software screen does not detect the **USB Spectrometer**, check to make sure it is full connected to the PC's USB port.
  - If the Spectrometer is already properly connected, close the program. Then, disconnect and reconnect the spectrometer. Re-open the PEM Program to establish communication and feedback from the Spectrometer.
  
- **No Spectral Data Acquisition**
  - Make sure software programs are opened in the proper sequence as mention in both the auto and manual operating chapters.
  - Ensure that proper connection and feedback is made between the **PEM Program** and the **Spectrometer**.
  - Check hardware connections as mention in the *hardware trouble shooting* section.

## Software



### ▪ **Process Stability**

- By far, the most complicated trouble shooting parameter is process stability.
- To assist in process optimization, it is highly recommended that the user should fully familiarize themselves with this Manual as well as any available references regarding Reactive Sputtering processes.
- Optimizing process stability will be different for every process application and material.
- Every process parameter plays a role in the stability of a reactive sputtering process.
- PID parameters are to be optimized for each different process application and deposition material. Ultimately, the operator should adjust the PID control loop to do as little “work” as possible but enough to effectively control the process.
- As a starting point for PID parameters, reference chapter 4 on PID control loops.
- Another key process parameter affecting the stability of reactive sputter deposition is pumping speed. Every system and pump will have a different pumping speed. In addition, different process gases also have different pumping speeds.
- Optimizing pumping speed will help with process stability and hysteresis effects inherent with reactive sputtering processes.

### ▪ **Low Plasma Transmission**

- Refer to section 3.2 regarding Hardware troubleshooting.

### ▪ **Additional**

- For any additional information regarding troubleshooting, please contact **Denton Vacuum** Technical Support. They will direct you to qualified assistance.

