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NJEngineering

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**Atomic Oxygen Text Facility**

The complete AO facility, as seen in Figure 1, consists of a stainless steel chamber pumped by a turbo pump (TURBOVAC 600C) down to a base pressure of of  $1 \times 10^{-6}T$ , the turbo pump is backed by a Fomblin oil filled mechanical pump.

To simulate the LEO environment in a laboratory setting, an Electron Cyclotron Resonance Source (ECR) was used. An ECR is a plasma device designed to generate a dense plasma. The ECR source was characterized by Kapton dosimetry and in-situ four point probe silver film resistivity measurements. The axial magnetic field ECR is attached to one of the ports of the AO system chamber and an 80 mesh copper screen is placed over the exit of the ECR to block any ion flux. The system is also equipped with an electronically controlled heater; a thermocouple is used to monitor the temperature accurately during non-heated runs.

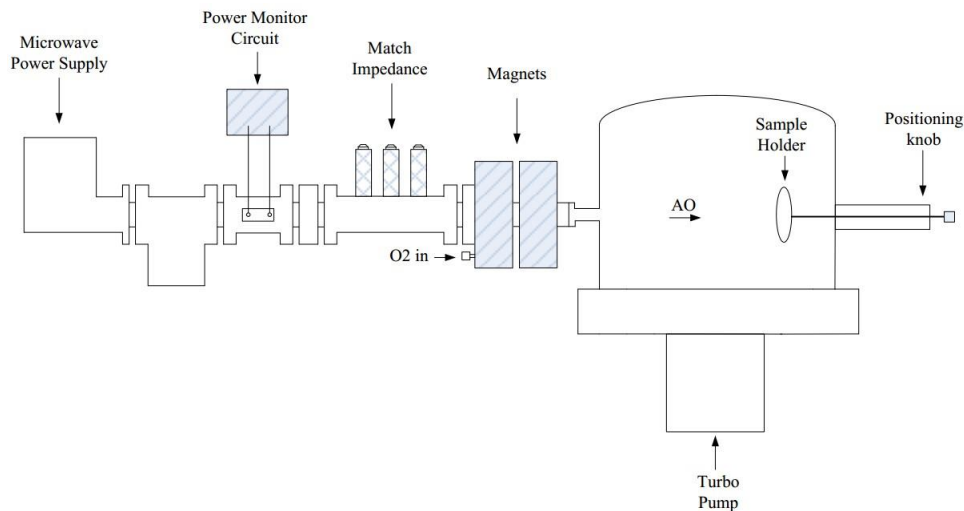


Figure 2. ECR setup for chamber

The typical operating condition for the chamber are 400W input power, gas flow 10sccm  $O_2$ , and pressure  $9 \times 10^{-4}$ T. This yield a flux density of  $1E16/cm^2$ -sec at 0.3eV. When the screen is removed a flux density of  $3E16/cm^2$ -sec at 5eV is obtained although an ionic component exists in the beam. The ER plasma produces radiation at 135nm, an atomic oxygen emission line. The substrate can be rotated opposite to the beam resulting in no VUV exposure, but a drop in flux density to  $3E15/cm^2$ -sec at an energy of about 0.03 eV which is essentially room temperature. Figure 2 is a photograph of the system.

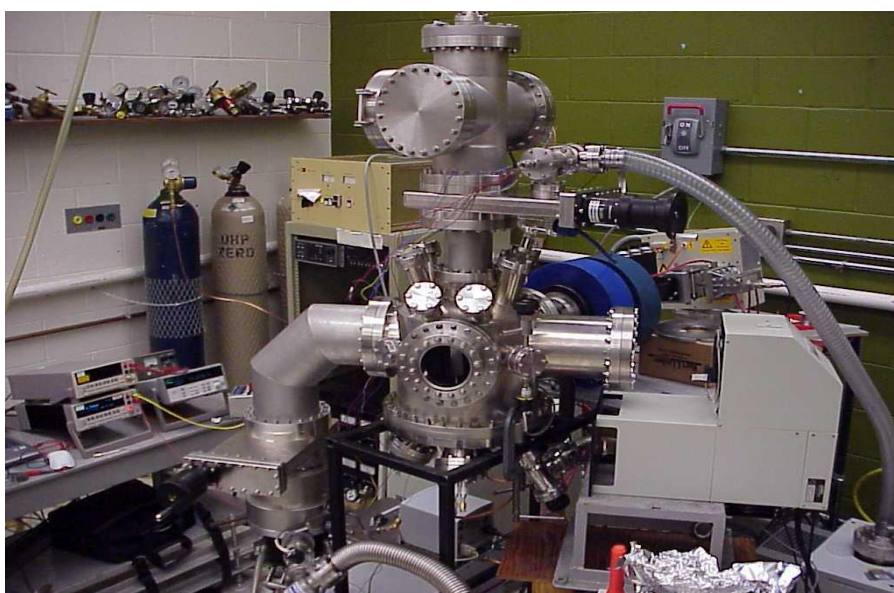


Figure 2. Photograph of AO System

## VUV Test Facilities

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The VUV test facility consists of a cryo-pumped stainless steel chamber with an LN2 shroud, base pressure  $< 2E-7T$ . The substrate plate is 8 inches in diameter and is temperature controlled. In general the plate is machined to hold a customer's specific samples. An OAI 1.1 kW AM0 solar simulator with intensity control provides the spectrum from about 350 nm to 1.8 microns while Hamamatsu Deuterium lamps calibrated via a McPherson VUV spectrometer and a calibrated Deuterium source provide an integrated intensity of 1 sun at the substrate plate between 110nm and 250 nm. The intensity can be measured in situ via a calibrated Si solar cell and VUV photodetector. A mass spectrometer with a range to 200 amu monitors background contamination levels. In addition a calibrated solar cell can be positioned above the substrate plate to provide an in-situ measure of the sun intensity. The cell can be retracted into a tube to shield from any photofixing that may occur. The system can perform the AIAA S-112A-2003 2000 esh test. A photograph of the system, is seen in Figure 3.

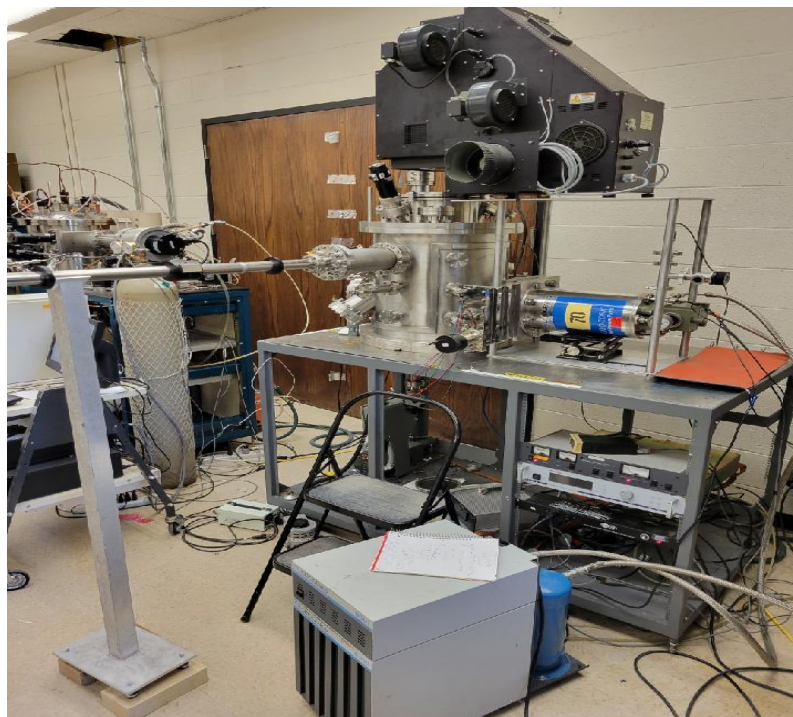


Figure 3. VV Test Chamber

## **Contamination Analysis Test Chamber (CATC)**

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The experimental chamber setup referred to as the Contamination Analysis Test Chamber (CATC), is shown in Fig. 4. The lower level of this chamber contains the contamination source for creating the flux of outgassing material for collection on a QCM surface or test sample surface. The source comprises a vapor effusion cell (VEC) made of copper with an electrical resistance heater silver soldered to the outside of the cell, and a type E thermocouple soldered to the outside of cell. The thermocouple is connected to a Micromega CN77300 thermal controller which drives a solid state relay that controls power to the heater. The heater surrounding the VEC is capable of 30 watts power output which can support the temperature of the VEC up to 200 C. The VEC itself is 45mm inner diameter, 47mm external diameter and 90mm tall. The orifice of the VEC lies in a plane with the QCM surface or test sample surface, and these two planes are coaxial, the QCM and VEC are exaggerated in this schematic, the actual distance between these two planes is around 13 inches. A shutter is installed in lower level of the chamber and can be adjusted by an air side knob. This shutter can used to cut off molecular flux to the QCM surface or test sample surface. The internal volume of the experimental chamber is surrounded by a LN2 shroud which can attract excess source molecular flux not directly incident on the collecting surface. The chamber is pumped by a cryo-pump to a base pressure in the 1E-7T range.

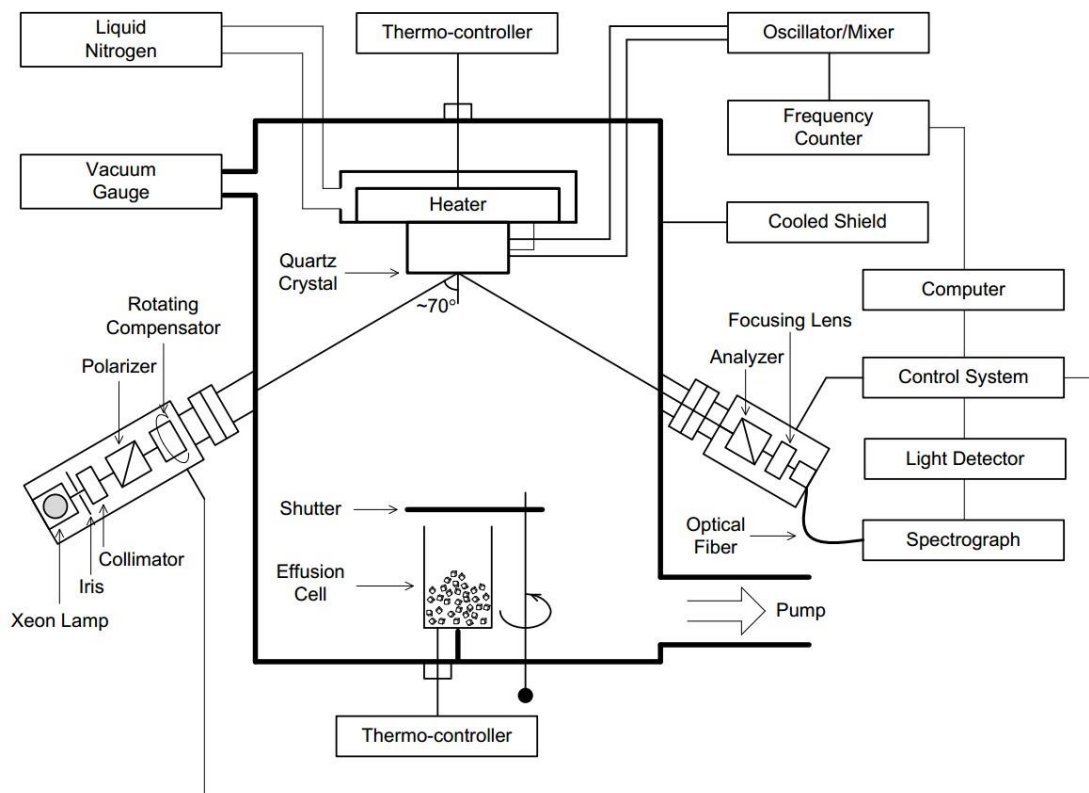


Figure 4: Schematic of Contamination Analysis Test Chamber

At a distance of 13 inches from the opening of the VEC is the test sample surface and QCM. The QCM is bolted by its heat sink through the hole in the liquid nitrogen filled shroud. The copper case of the QCM can be held at cryogenic temperature while the crystals are heated to the desired temperature via the in-situ heater. The crystal temperature can range from 84K to 400K. The QCM views the VEC orifice through a shuttered hole permitting the impinging outgassing flux to be interrupted when needed. In this Mark 18 model QCM, two crystals are used (sense and reference) in a crystal pack. Although not shown in the figure, a 1 sun solar simulator with spectral range between 300 and 1200nm, as well as a Xe microwave cavity light source can be made incident in the substrate plane.



Figure 5. Photo of VCM chamber

One of the strengths of the facilities is spectroscopic ellipsometry is incorporated in the test chambers. In spectroscopic ellipsometry, optical constants and film thicknesses of samples are determined from the change in the polarization state by light reflection over a wide range of wavelengths and at one or multiple incident angles in real time.

Initially the optical interference can be utilized as a simple way to understand how phase difference is a function of incident angle, thickness, light wavelength, etc..

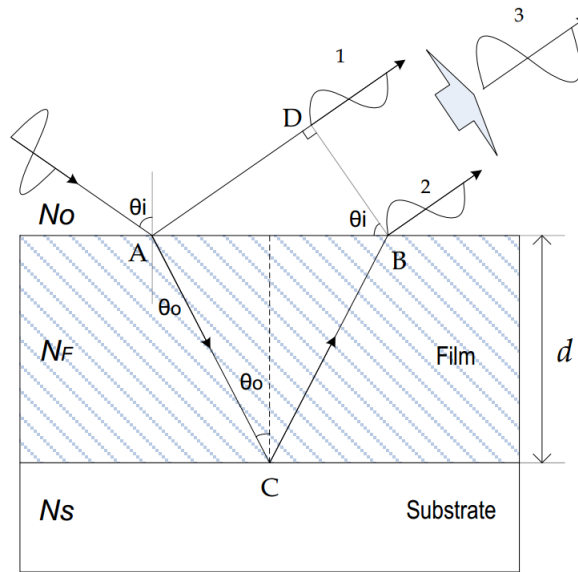


Figure 6. Optical interference in a thin film

Measuring the thickness of a thin film on a substrate can be deduced from the optical interference illustrated in the Fig 6. The reflected beam (*primary1*) and the transmitted beam (*secondary2*) overlap into form of beam 3 which has a larger electric field amplitude (if the two beams have the same phase) or smaller electric field amplitude (if the two beams are out of phase). The phase difference can be explained by the optical path difference between  $(AC + CB)$  and  $AD$  shown in the equation below

$$\alpha_{1,2} = K_F(\overline{AC} + \overline{CB}) - K_O\overline{AD} \quad (1)$$

$$K_i = \frac{2\pi N_i}{\lambda}$$

Where  $K_i$  is the propagation number for beam 1 and 2.

By using Snell's law, the secondary beam (2) shows a total phase difference of the form

$$\alpha = \frac{4\pi d N_F}{\lambda} \cos\theta_o \quad (2)$$

As seen from equation 2, in a thin film/substrate structure, if assuming that the phase difference  $\alpha$  induced by this optical interference corresponds to  $\Delta$  observed in ellipsometry, knowing the thickness of the film, the optical constant  $N_F$  can be derived, or vice versa.

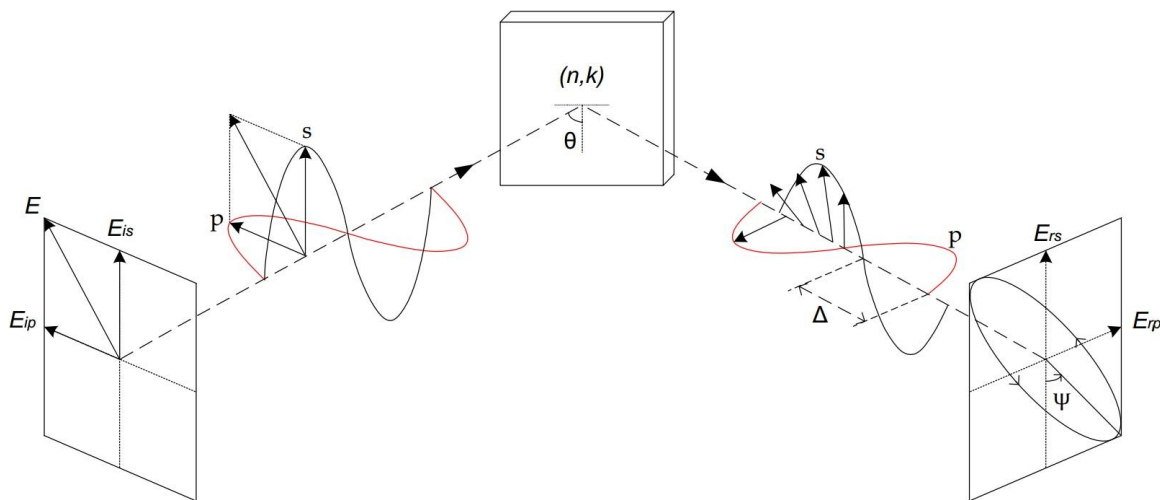


Figure 7. Measuring principle of ellipsometry

Figure 7. Illustrates the measurement principle of ellipsometry, the polarization states of<sup>9</sup> incident and reflected light waves are described by  $p$ - and  $s$ - polarizations (parallel to and perpendicular to the plane of incidence). In this figure, the incident light is linear polarized with an orientation of  $+45^\circ$  to  $E_{ip}$  axis. The amplitudes of  $p$ - and  $s$ - polarizations are the same ( $E_{is} = E_{ip}$ ) and the phase difference between them is zero.

Upon reflection from a sample, polarizations are changed in amplitude and phase in the directions of both  $p$ - and  $s$ -. As show in Fig 3, the reflected light wave converts into elliptical polarization. The two values ( $\Psi$ ,  $\Delta$ ) measured present the amplitude ratio of the Fresnel reflection coefficients  $r_p$  and  $r_s$  and phase difference between  $p$ - and  $s$ - polarizations, respectively.

The raw ellipsometric data is related to the ratio of complex reflection coefficients, which defined as:

$$\rho = \frac{r_p}{r_s} = \tan\Psi \exp(i\Delta) \quad (3)$$

The optical constants and thickness cannot be solved in analytical fashion. Given initial standard deviation estimates ( $\delta\Psi$  and  $\delta\Delta$ ) for the measured  $\Psi$  and  $\Delta$ , pairs of  $\Psi$ ,  $\Delta$  values are calculated for the measured wavelengths and angles of incidence. By varying the model parameters, the mean-square error can be reduced then minimized using Levenberg-Marquardt algorithm, which in the form of weighted test function below,

$$\zeta^2 = \frac{1}{2n - m} \sum_{j=1}^n \left\{ \left( \frac{\Psi_j^{ca}(\mathbf{x}) - \Psi_j^{me}}{\delta_{\Psi,j}} \right)^2 + \left( \frac{\Delta_j^{ca}(\mathbf{x}) - \Delta_j^{me}}{\delta_{\Delta,j}} \right)^2 \right\} \quad (4)$$

where  $n$  is the measured  $\Psi - \Delta$  pairs,  $m$  is the number of fitting parameters,  $\mathbf{x}$  is a vector with  $m$  components including thickness, Sellmeier coefficients,  $\Psi^{me}$ ,  $\Delta^{me}$  and  $\Psi^{ca}$ ,  $\Delta^{ca}$  are measured and calculated data, respectively. Then the optical constants and thickness can be obtained as a result of regression fit from the figure of merit.

## Ex-situ Ellipsometry Facilities

### Visible-Near IR Spectral Range

The M2000VI Ellipsometer consists of a base M2000 with near infrared (NIR) extension. It is one kind of Rotating Compensator Ellipsometer (RCE) with high-speed charge coupled device (CCD) detection to collect the entire spectrum in a fraction of a second. The wavelengths range is between 370 nm and 1690 nm.

### VUV Spectral Range

The VUV-VASE (vacuum ultraviolet- variable angle spectroscopic ellipsometer) employs a  $MgF_2$  polarizer and analyzer rather than the more common calcite optics. It measures wavelengths from VUV to NIR, about 147 nm to 1700 nm. The spectrometer upgrades from single beam to dual beam for more accurate transmission measurements and better instrument stability. The instrument assures high accuracy utilizing an AutoRetarder and is fully nitrogen purged to avoid absorption of VUV light by ambient oxygen and water vapor which is important at wavelengths below 200 nm. Light from both the deuterium and xenon lamps passes through a double-chamber to provide wavelength selection and stray-light rejection. The spot diameter of light source on the surface of the sample is 2 mm. Computer-controlled slit widths can adjust the bandwidth to insure adequate spectral resolution of optical features in the data such as the closely spaced interference oscillations, which arise in very thick films. One of its advantages is that it could be applied to measure liquid samples and determine the optical constant of liquid materials.

### Infrared Spectral Range

The infrared (IR) VASE ellipsometer from J. A. Woollam Company is composed of a Fourier-transform infrared spectrometer and a rotating-compensator variable-angle ellipsometer. It

covers a spectral range from 333 to 5900  $\text{cm}^{-1}$  (from 1700 nm to 30  $\mu\text{m}$ ). Spectroscopic ellipsometric data contain both phase and amplitude information from the reflected or transmitted light, and the phase information from IR ellipsometry offers higher sensitivity to ultrathin films than FTIR reflection or absorbance, while retaining the sensitivity to chemical composition.

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The spectra information in the infrared range is very attractive. In the near infrared (NIR) range, there are still contributions of electronic transitions in the mid-infrared (MIR) range there are contributions of free carriers and molecular rotation and vibration transitions in the far infrared (FIR) range, phonons play an important role. Most materials exhibit some properties according to these mechanisms, which makes infrared absorption techniques a valuable tool in the analysis of materials.