

UNIVERSITY OF WATERLOO

Physics 360/371 - Experiment 19

**VACUUM EVAPORATION OF DIELECTRIC INTERFERENCE FILTERS**

References: Practical Vacuum Techniques; Brunner & Batzer, Chapter 2  
Principles of Optics; Born & Wolf, Sect. 7.6.6  
Thin Film Technology; Berry, Hall & Harris, Sect. 2.3, 2.4, 2.6, 3.6  
Vacuum Deposition of Thin Films; L. Holland

Introduction: The purpose of the experiment is to familiarize students with vacuum systems and the techniques of vacuum evaporation, and simple optical measurements.

The basic principles of the Fabry-Perot interferometer can be easily extended to the case of an interference filter where a dielectric material is used to separate two semitransparent reflecting layers. These filters can be used to transmit a fairly narrow band of wavelengths in the visible spectrum. With vacuum evaporation techniques it is possible to prepare uniform and continuous dielectric films and metallic reflecting layers in the thickness range of hundreds of Angstroms. These relatively small thicknesses are required to attain semi-transparency of the reflecting coatings and low order interference to sufficiently separate the wavelengths transmitted.

1. Preparation:

Check the initial conditions of the system are as follows:

Main valve control	closed
Mains power	off
Leak valve	closed
Diffusion pump	off
Rotary pump	off
Air admittance	off

2. Turn on the cold water-cooling supply and turn the mains power to the coating unit on. Start the rotary pump by depressing the pump switch at the top of the control console. Now open the backing valve by turning the main valve control a quarter turn clockwise. Observe the Pirani gauge and allow the pressure to drop below 0.3 mbar, once this is attained you may start the diffusion pump by depressing the switch. The diffusion pump oil will reach operating temperature in approx. 25 mins.

- Carefully lift off the bell jar and place it in the holder. It is important that the rubber seal and sealing surfaces are kept clean. Turn on the frequency meter and check that the crystal oscillator used for sample thickness monitoring has a frequency within 200 KHz of 5 MHz. The relationship between deposition thickness (t), frequency change ( $\Delta f$ ) and density of evaporated material ( $\rho$ ) is

$$\Delta f = \frac{\rho t}{1.9} \quad \text{where } \Delta f \text{ is in Hz} \quad (i)$$

$\rho$  is in gm/cc  
t is in angstroms

	<u>Density</u>	<u>Melting Point</u>	<u>Refractive Index</u>
Al	2.7 gm/cc	660° C	
Na <sub>3</sub> AlF <sub>6</sub>	2.9 gm/cc	1000° C	1.33

- Prepare tungsten (W) and molybdenum (Mo) helix sources as described in appendix A and load as described with Al wire and Na<sub>3</sub>AlF<sub>6</sub> powder. Mount the sources (2 of each) on the support posts. Measure distances between sources, slides and oscillator; then weigh and mount clean glass slides in position. Refer to appendix B.
- Replace the bell jar, and the lucite implosion shield.
- Pumpdown. Close the backing valve and open the roughing valve by rotating the main valve control one half-turn counter clockwise. Allow the pressure (Pirani gauge) to drop to at least 0.8 mbar. Turn off the oscillator and turn on the glow discharge by selecting the H.T. control and increase the current to a maximum of 20 as read off the meter. Once the glow has diminished, zero the potentiometer and turn off the H.T. control then return the main valve control to the backing position. SLOWLY open the H.V. Valve by turning it one half-turn clockwise. Switch on the Penning gauge and set to range 1. Range 2 may be selected as the pressure in the bell jar is reduced. Using great care when handling, you may now fill the cold trap with liquid nitrogen.
- Using the source selector and LT control, evaporate 300 angstroms Al, 3000 angstroms of cryolite and 300 angstroms Al. Increase the potentiometer slowly at first for each source so it is allowed to outgas. During evaporation keep the pressure below  $1 \times 10^{-4}$  mbar with rates between 5 and 50 Hz per second as read on the oscillator frequency meter. Once you have the required thickness return the potentiometer to zero and select another source. For the sources listed in appendix A, depending on source load and condition, this corresponds to meter readings of:

Al	16-20 (10V)	40-50 (30V)
Cryolite	20-22 (10V)	50-55 (30V)

Once all evaporation is completed deselect the LT control and allow the sources and slides to cool for 5 minutes.

8. Close the H.V. valve by turning the valve control 180 degrees counter-clockwise to return it to the backing position. ONLY when this is completed should the air admittance switch be depressed. Carefully remove the bell jar and the prepared films.
9. Weigh the glass slides and verify the constant in the frequency equation, using

$$\text{constant} = \frac{W}{A\Delta f}$$

where: W is total weight evaporated

$\Delta f$  is total frequency change

A is area of the slide.

10. System off. Replace bell jar and turn off diffusion pump. Wait 30 mins, and then return main valve control to the closed position. Turn off the rotary pump and the mains power and close the valve on the cooling water supply.
11. Using the spectrophotometer - computer set-up, you may obtain transmission versus wavelength plots for your prepared filters as well as plots for several commercially prepared filters. (These plots can be prepared during the period you are pumping down and evaporating.) The control program "SPECT" is used. This is a comprehensive program which allows maximum flexibility in tailoring your output. A program flowchart is attached to illustrate the various features of this program.
12. Since the metallic reflecting layer absorbs some of the light, an effective phase shift occurs at the interface such that for 1st order

$$\frac{\lambda_0}{2} = \frac{nh}{1 - \frac{f}{p}}$$

where  $\lambda_0$  is wavelength of transmitted peak intensity

h is thickness of dielectric

$\phi$  is effective phase shift

n is refractive index of dielectric

For 1st order reflection from thin metallic films at normal incidence

$$d = \frac{l}{F \left( 1 - \frac{f}{p} \right)} \quad (\text{Born and Wolf, Sect. 7.6.6}) \quad (\text{iv})$$

and  $F = \frac{p\sqrt{R}}{1-R}$  (Born and Wolf, Sect. 7.6.2) (v)

where  $d$  is the width of the transmission peak at half intensity

$R$  is reflectivity of the dielectric metal interface.

From your plot of optical transmission versus wavelength, determine the reflectivity of the dielectric metal interface.

## Appendix A: Computer - Spectrometer usage

To operate the setup, you must first turn on the computer, printer, DVM's and the spectrometer. The control program will load automatically. When the CAL message on the spectrometer goes out, set the control switch (located on the bottom front of the unit) to the HOLD position. Use the MODE button to select %T.

To obtain:

### i) Background:

- Ensure an **empty** sample holder is properly in place.
- Use the wavelength +/- keys to set the wavelength to 690 nm.
- Depress the Set Ref key (display should read 100%).
- Use the +/- keys to set wavelength to 325 nm (**wait** for a measurement to be displayed)
- Click on the read background button.
- Move the control switch to sweep (SWP). The background is now being recorded.

Note that the background only needs to be recorded once for the experiment.

### ii) Filter Spectra:

- Set the control switch to HOLD
- Insert the filter in the appropriate holder and insert into the spectrometer.
- Set the wavelength (+/- keys) to the position of maximum intensity (as displayed on the MEASUREMENT readout). For the commercial filters this will be near the wavelength on the side of the filter, for your prepared filter you will have to "hunt" for the wavelength of maximum intensity.
- Depress the Set Ref key (display will read 100%)
- Set wavelength to 325 nm (**wait** until a reading is displayed).
- Click on read spectra, then switch to sweep (SWP)
- When wavelength reaches 900 nm the program will prompt you for a target filename. The path should be C:\DATA\filespec.

There are several other very intuitive options available to you in the program;

- Display Spectra (to screen). A good idea before you print.
- Print Spectra i.e. hard copy.
- Data from file: shows a list of all files in C:\DATA\ click on a file you wish to retrieve.

# The Vacuum System

