PRINT FIRST NAME	PRINT LAST NAME	PRINT STUDENT ID	GRADE

Experiment 4

Preparation of a thin film by vacuum evaporation

Before starting the experiment, you should be familiar with the basic concepts of vacuum technique (pumps, gauges).

Introduction

Thin layers ("films") of solid materials, with thicknesses $\cong 10^{-6}$ m, are important components of many modern electronic and optical devices. They are often produced by evaporating the material in a vacuum, and condensing the vapour on a cold substrate until the desired thickness is reached. A vacuum is used in order:

- to prevent a chemical reaction of the material with air,
- to lower the boiling/sublimation temperature, and,
- to deposit the material in a smooth, even manner on the substrate.

In this experiment you will produce a thin film of the metal bismuth on a glass substrate, and measure its thickness with a Michelson interferometer. The electrical properties of the film will be measured in a subsequent experiment.

4.1 Vacuum Evaporator

The mechanical (rotary) pump evacuates the bell jar, via the bypass, to a pressure of $\cong$ (10→100) mTorr, and ejects the evacuated gas into the air. The Turbo pump can pump down to a limit of $\cong (10^{-5} \rightarrow 10^{-6})$ Torr .

Units of pressure (force per area):

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MKS unit	Pa (for Pascal)	1 $N{\cdot}m^{-2}$
Practical units	Torr (for Torricelli)	760 Torr (1 standard atmosphere)
	mm of Hg (mercury):	equal to 1 Torr
	atmospheric pressure:	760 Torr, or 1.013 $\times 10^5$ Pa.

It only operates properly when first evacuated by the rotary pump. The evaporation takes place in a glass bell jar, from a tungsten filament which acts as a boiling reservoir for a small

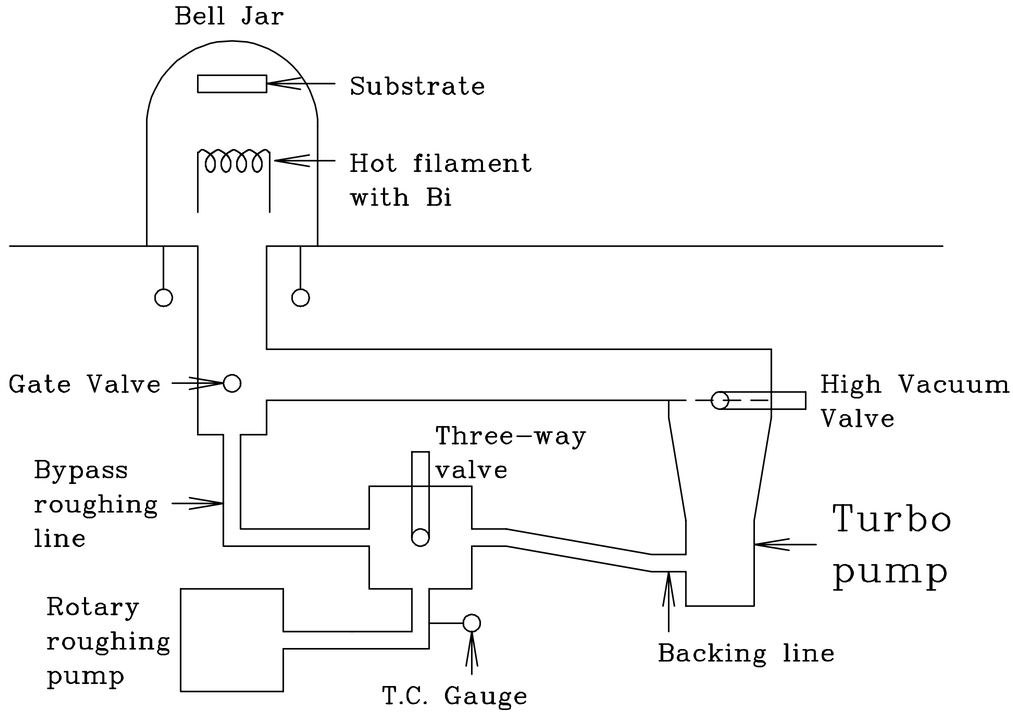

Figure 4.1: Diagram of a Vacuum Evaporator

quantity of Bi. The pressure in the bell jar is measured with either a thermocouple gauge (range $\cong (10^{-3} \to 10^{0})$ Torr), or with an ionization gauge (range $\cong (10^{-3} \to 10^{-6})$ Torr).

Good films can be produced when the atoms/molecules of the material, in our case Bi, travel from the hot filament to the substrate directly, without colliding with residual air molecules, and when the substrate is clean and has no layers of gas, grease, etc., absorbed on its surface. In this respect the following parameters are important and can be calculated from the kinetic theory of gases:

1. the number density of gas N_0; this is the number of gas molecules per unit volume. It can be calculated from the ideal gas law as
$$N_0 = \frac{P}{kT},$$
where P is pressure, T is temperature, k = Boltzmann's constant $= 1.38 \times 10^{-23}$ JK^{-1}.

2. the average speed $\bar{V}$ of the gas molecules:
$$\bar{V} = \sqrt{\left[\frac{8kT}{\pi m}\right]}$$
where m = mass of the gas molecule.

3. the mean free path $\bar{L}$ of the molecules:
$$\bar{L} = \frac{1}{\sqrt{2} \cdot \pi \cdot d^2 N_0}$$
where d = diameter of molecule

4. the rate at which gas molecules collide with a surface:

$$\nu = \frac{1}{4} N_0 \bar{V}$$

(collisions per unit area per unit time).

4.2 Exercise

Calculate, and plot versus P, the quantities $N_0, \bar{V}, \bar{L}$ and ν for $P = 760$ Torr, 76 Torr, etc., down to 7.6 $\times 10^{-6}$ Torr. Use nitrogen at room temperature as the gas: $d = 3.73 \times 10^{-10}$ m. Use double logarithmic scales for your graph.

At what values of P and T are:

1. $\bar{L}$ about equal to the distance from boiling reservoir to substrate.
2. the collision rate ($(\mathrm{m}^2 \cdot \mathrm{s})^{-1}$) about equal to the typical numbers of atoms per m^2 of substrate surface ($\cong 3 \times 10^{19}\ \mathrm{m}^{-2}$)?

It should be clear from your calculations that a vacuum of $< 10^{-4}$ Torr is required for proper vacuum evaporation.

Use the vacuum evaporator to produce a thin film of Bi, in the shape of a cross suitable for electrical measurements. Follow the procedure outlined below; the instructor will be present to assist you.

4.3 Procedure for Operating the Vacuum Evaporator

1. Preparation of substrate for film deposition:
   (a) Wash the glass slide with detergent and water and rinse.
   (b) Rinse with distilled water.
   (c) Rinse with ethanol.
   These steps remove water and grease from the slide. Ethanol may dissolve grease from your fingers and leave it on the glass when it evaporates. Use gloves.
   (d) Dry the slide in air. Check that it is clean.
2. Deposition of the bismuth film.
   (a) Check that the ion gauge is off.
   (b) Close the high vacuum valve.
   (c) Vent the vacuum chamber and lift the bell jar.
   (d) Put some bismuth into the filament basket.
   (e) Put the slide inside the mask and place it above the filament.
   (f) Put the bell jar back on, make sure that the vent valve is closed.
   (g) Use the roughing pump and pump down the bell jar to below 100 m Torr pressure via the by-pass.

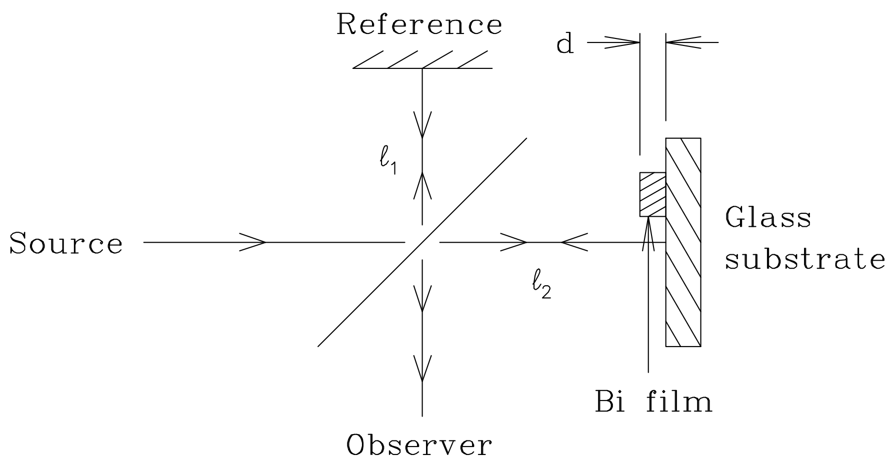

Figure 4.2: Diagram of a Michelson Interferometer

(h) Turn on the turbo pump open high vacuum gate valve. The turbo pump will now start removing the "low pressure" residual air from the bell jar. After a few minutes the thermo-couple gauge will read 10^{-3} and the cold cathode gauge switches on automatically at the 10^{-4} Torr range. Wait until the pressure is $\leq 5 \times 10^{-5}$ Torr.

(i) Turn on the filament supply switch, increase the current until the filament starts glowing. Keep it like that for a minute to outgass it. Increase filament current, until bismuth starts evaporating. Hold filament current until all Bi has evaporated.

(j) Shut down the filament current; turn the filament supply off.

(k) Repeat steps (2a)–(2c).

(l) Remove slide with bismuth film on it, replace bell jar, evacuate chamber to high vacuum as in steps (2g) and (2h).

4.4 Thickness Measurement

Since the thickness is of the same order of magnitude as the wavelength of visible light, we will use a method based on wave interference, the Michelson Interferometer.

When the optical paths ℓ_1 and ℓ_2 differ by $k\lambda$ ($k = 0, 1, 2, \ldots$) the monochromatic waves (wavelength λ) on their way to the observer are out of phase, and destructive interference produces a dark fringe in the field of view. The glass substrate plus film will form one mirror of the interferometer. The light reflecting from the Bi film travels a distance $2d$ less than the light reflected from the glass slide, and therefore the fringe pattern over the Bi film is shifted with respect to the pattern over the glass slide. From the shift the thickness d can be easily calculated.

The actual instrument is a Michelson interferometer optically "folded-up" so that it fits over a microscope ($\times 10$) objective. It contains the beam splitter and reference mirror (which can be tilted) and a means of controlling the distance ℓ_1.

4.5 Procedure for measuring d.

1. The instructor will put the interferometer over the microscope objective, and adjust ℓ_1.

2. Place your slide (film up) under the microscope; turn the illuminator on; use white light.
3. Focus on a film edge by adjusting the vertical position of the microscope stage. This sets the distance ℓ_2.
4. Switch to monochromatic illumination by putting a Fabry-Perot interferometer filter in the light path.
5. Gently adjust the vertical position of the film, until you see interference fringes. (Be patient).
6. Adjust the tilt screws of the reference mirror until the fringes are perpendicular to the film edge. You should see a fringe pattern as shown in figure 4.3.

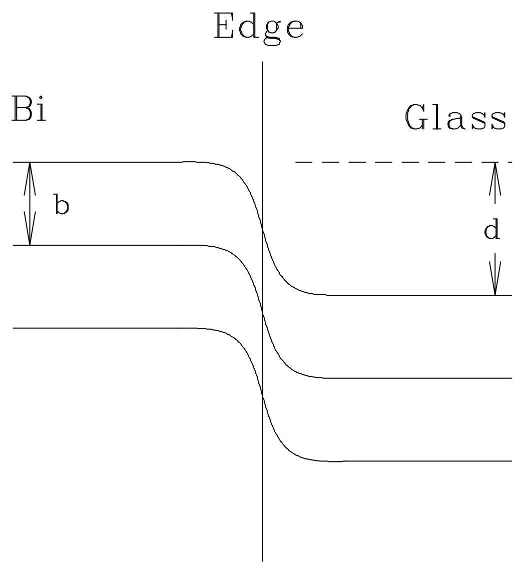

Figure 4.3: Fringe pattern

7. Estimate b (the fringe spacing) and a (the fringe shift) using a measuring eyepiece with a scale in it. The film thickness is then:

$$d = \frac{a}{b} \times \frac{\lambda}{2}.$$

8. Repeat at different positions along the edge of the film.

References

For vacuum techniques, books such as:

- J. Yarwood, *High Vacuum Technique.*
- J. F. O'Hanlon, *A User's Guide to Vacuum Technology.*
- H. Mark and N. T. Olson, *Experiments in Modern Physics*, Ch. 4.

For the Michelson Interferometer:

- E. Hecht and A. Zajac, *Optics*, Chapter 9.10.2