Experiment 1

Preparation of a thin Bi film by vacuum evaporation

Basics of vacuum technology (pumps, valves, gauges) and vacuum evaporation techniques are explored in this experiment. Some of the equipment in use is dangerous and delicate; if unsure of how to proceed safely (for yourself or the equipment) be sure to consult the instructor first.

1.1 Introduction

Thin layers ("films") of solid materials, with thickness $\simeq 10^{-6}$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 vapor on a substrate until the desired thickness is reached. Modern techniques of molecular beam epitaxy are far more elaborate than our simple experimental setup, but the fundamental principles are the same.

Deposition is done in vacuum in order to:

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

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

Outline of the evaporator operation

To perform *vacuum* evaporation, the evaporator's bell jar needs to be *evacuated* first, using a two-stage pumping station. Schematically the setup is shown in Fig. 1.1, showing two different regimes of pumping: roughing and high-vacuum modes. Be sure to to read the following section very carefully. Operation of high-vacuum equipment has special safety considerations for the user, and failure to adhere to the proper procedure may result in catastrophic failures of expensive equipment.

The roughing regime is using a mechanical pump to evacuate the bell jar, via the bypass, to a pressure of about $\simeq 10$ mTorr. The evacuated gas is ejected into the room air, and since the back-flow is prevented by bubbling the gas through the pump oil, a fine oil mist is sometimes generated; the filter on the output of the mechanical "roughing" pump catches that oil. During this regime, a significant bubbling noise is heard, but it quickly diminishes as the pressure begins to decrease. If it does not, and the pump continues to be noisy, there is a leak: check that the bell jar vent valve is closed, that the bell jar itself is making a firm seal with the base plate, and that the transfer lines are all secured. If the turbo pump is already spinning, it must be completely isolated from the roughing pathway and maintained at low pressure. This initial evacuation of a bell jar to about 10 mTorr usually takes about 10 minutes. During the roughing regime, the gas pressure is too high for either ion gauge or the cold-cathode pressure gauges, and must be monitored only through the thermocouple (TC) gauge. The cold cathode gauge can be in stand-by mode

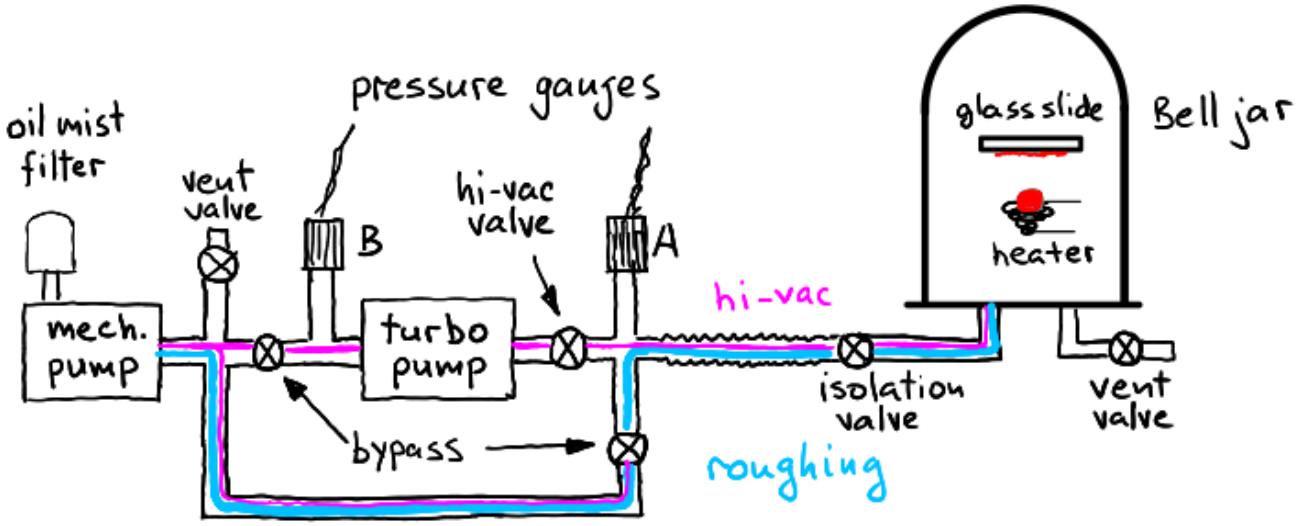

Figure 1.1: Schematic diagram of the evaporator setup, showing two different regimes of pumping: roughing and high-vacuum modes.

(high voltage off). At the completion of the roughing process, both TC gauges A and B should show the same pressure of about 10 mTorr.

After the pressure is lowered sufficiently for the safe operation of the turbo pump, the bypass pathway can be closed and the mechanical pump switched to the back-end support of the turbo pump. If the turbo pump was already spinning, isolated, the bypass valve connecting mechanical and turbo pump can now be slowly opened, followed by a slow opening of the hi-vac valve, completing the switchover to the hi-vac pumping pathway. At no time should the turbo pump be asked to pump at its front inlet without first establishing a back-end support from a mechanical pump.

Once in the hi-vac regime, the turbo pump can pump down to $\simeq 10^{-5}$–10^{-6} Torr. The ion or cold-cathode gauge can be safely operated below a few mTorr, and there should be some difference in pressure between the front and back ends of the turbo pump (TC A and TC B readings will differ); this indicates that the turbo pump is working. As the pumping continues, this difference will become smaller and will disappear when high vacuum is reached, as shown in Fig. 1.2. If the pressure fails to reach $\simeq 10^{-5}$ Torr after about 10-15 minutes of hi-vac operation, check for leaks: tighten the connectors and vent valves. If a rubber seal on the bell jar is suspected, immediately isolate the turbo pump, turn off the cold-cathode gauge, and switch to the roughing pathway, before trying to adjust the rubber seal. A sudden exposure to room pressure will cause a catastrophic failure of the turbo pump.

The evaporation takes place in a glass bell jar, when a tungsten filament coiled up into a small crucible is heated; it acts as a boiling reservoir for a small quantity of bismuth. Bismuth vapour then condenses on a nearby surface of a clean glass substrate (a microscope slide), held in a custom frame directly above the crucible. The film is deposited on the downward-facing side of the glass slide, usually through a mask of the desired shape sandwiched onto the slide. The best way to monitor the deposition process is to find a line of sight that passes through the glass slide and the opening in the mask to the crucible, then slowly increase the current to the crucible until it begins to glow red, and quickly turn off the crucible current when the view of the crucible through the opening in the mask grows dim, obscured by a bismuth film being formed on the glass. The arrangement can bee seen in Fig. 1.3. The length of time for the deposition process should be monitored and recorded, to ensure a correction can be made on the next attempt, if the film turns out to be too thin or too thick.

Bismuth vapour also condenses on the inside of the bell jar, necessitating a thorough cleaning of the

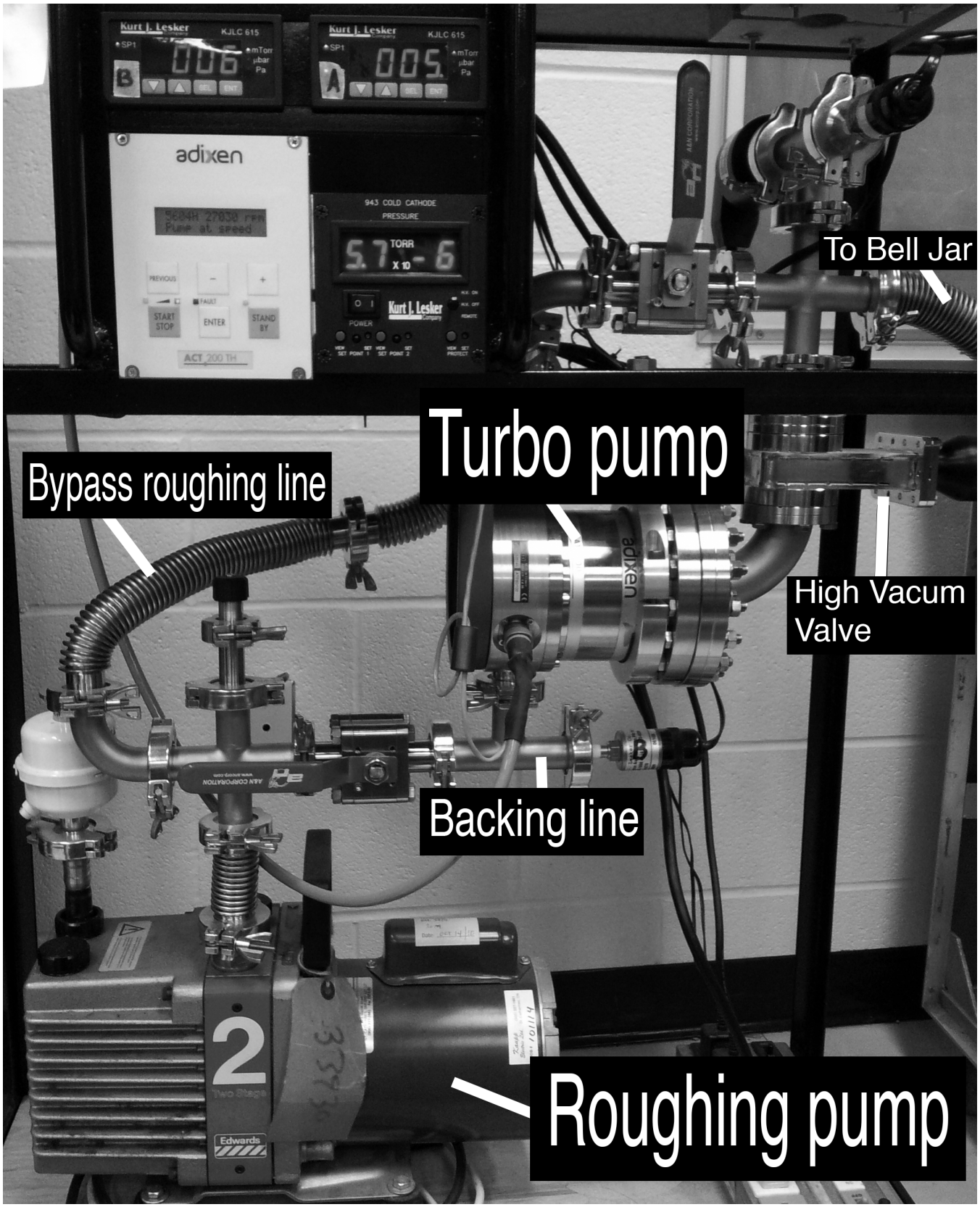

Figure 1.2: The pumping station, showing various TC and cold-cathod gauges, and the control panel of the turbo pump at the top left.

Figure 1.3: The tungsten crucible glows red, melting a small pellet of Bi metal placed into it. The glass slide with a mask plate underneath it is seen directly above the crucible. The score mark on the top surface of the glass slide is visible; the slide can be split into two separate samples along this mark.

inside of the bell jar between preparations.

Once the deposition has occurred, high-vac and the isolation valves can be closed and the bell jar slowly[1] filled with air through its vent valve, after which the bell jar can be lifted and placed sideways on foam supports for cleaning, and the sample extracted. Be sure to remove the deposition mask from the slide without sliding it sideways, to avoid scratching the newly made sample. During this time the turbo pump can continue to run, to maintain low pressure in the pumping system, but it must be isolated before the bell jar is reassembled, its vent valve closed, and the pumping station switched again to the roughing regime, to repeat the evaporation cycle.

At the end of operations, pumps need to be turned off. Consult the instructor: if another group still needs to perform their experiment, the instructor may recommend that the pumping continues overnight. To shut down the pumping system safely, the turbo pump can be isolated with bypass and hi-vac valves, and switch into spin-down mode on the control panel. This will take several minutes to complete. The turbo pump can be left under whatever high-vacuum remains inside it when it is not operating, but once the mechanical pump is turned off, the space in front of it must be vented to the atmosphere through its own vent valve. If this is not done, the vacuum there will suck the pump oil into the piping of the vacuum system, requiring a long and laborious cleanup.

Film thickness

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 impurities 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; which is the number of gas molecules per unit volume, can be calculated from the ideal gas law as
$$N_0 = \frac{P}{kT} \tag{1.1}$$
where P is pressure, T is temperature, and $k = 1.38 \times 10^{-23}\text{JK}^{-1}$ is the Boltzmann constant.

2. The average speed $\overline{V}$ of the gas molecules can be found from
$$\overline{V} = \sqrt{\frac{8kT}{\pi m}} \tag{1.2}$$
where m = mass of the gas molecule.

3. The mean free path $\overline{L}$ of the molecules is given by
$$\overline{L} = \frac{1}{\sqrt{2}\pi d^2 N_0} \tag{1.3}$$
where d is the diameter of molecule.

4. The rate at which gas molecules collide with a surface (collisions per unit per unit time) can be found from
$$\mu = \frac{1}{4} N_0 \overline{V}. \tag{1.4}$$

[1] Why slowly? be sure to understand the answer to this question before proceeding.

Exercise

Calculate and plot versus as a function of pressure P, the quantities N_0, $\overline{V}$, $\overline{L}$ and μ for $P = 760$ Torr, 76 Torr, *etc.*, down to 7.6×10^{-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 the following conditions met:

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

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

1.2 Sample preparation checklists

Use the vacuum evaporator to produce a thin film of Bi, circular in shape, with four contact pads suitable for making electrical measurements. If in doubt, contact the instructor first.

Preparation of substrate for film deposition

These steps remove water and grease from the microscope slide. Ethanol may dissolve grease from your fingers and leave it on the glass when it evaporates. Use gloves.

1. Score the slide down the middle so that the slide may be split in two.
2. Wash the glass slide with detergent and water.
3. Rinse with distilled water.
4. Rinse with ethanol.
5. Dry the slide in air. Check that it is clean.

Deposition of the bismuth film

1. Check that the ion or cold-cathode gauge is off.
2. Close the high vacuum valve, the by-pass roughing line, and the isolation valve which connects the bell jar to the pumps.
3. Vent the vacuum chamber by opening the vent valve, and lift the bell jar.
4. Put some bismuth into the filament basket.
5. Place the mask on top of the clean slide (on the opposite side of the score mark), and place it, mask side down, into the holder frame. Center the opening in the mask 5-10 cm above the crucible.
6. Clean the inside of the bell jar, especially near the top so that you can look inside. Put the bell jar back on; make sure that the vent valve is closed.
7. Switch the pumping station into the roughing regime. If the turbo pump is spinning, it can remain spinning provided it is isolated on both sides by the hi-vac and roughing valves. Open the valves in-between the roughing pump and the bell jar. Use the roughing pump and pump down the bell jar to about 10-20 mTorr via the roughing bypass.

8. Close the bypass valve on the high-vacuum side, and open the valve connecting the mechanical pump and the turbo pump. Slowly open the 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 pressure should fall below 10^{-4} Torr and the cold-cathode gauge can be switched on. Wait until the pressure is $\leq 5 \times 10^{-5}$ Torr before starting the evaporation. This may take up to 20 minutes.

9. Turn on the filament supply switch, increase the current slowly from zero until the filament just starts glowing. Keep it like that for a minute to outgas it. Increase filament current, until bismuth melts and starts evaporating. Hold filament current until you can no longer see the glow from the top of the bell jar through the opening in the mask and much of Bi has evaporated, as seen from the side.

10. Dial down the current, and turn off the current supply.

11. Repeat steps 1. – 3.

12. Remove slide with bismuth film on it, lift the mask straight up without dragging sideways.

13. Reassemble a fresh glass slide-and-mask "sandwich", place it in the holding frame, mask side down, replenish Bi in the crucible, replace the bell jar, and start evacuating the evaporator chamber to high vacuum again, in preparation of the next sample, or for the next group of students.

1.3 Measuring film thickness

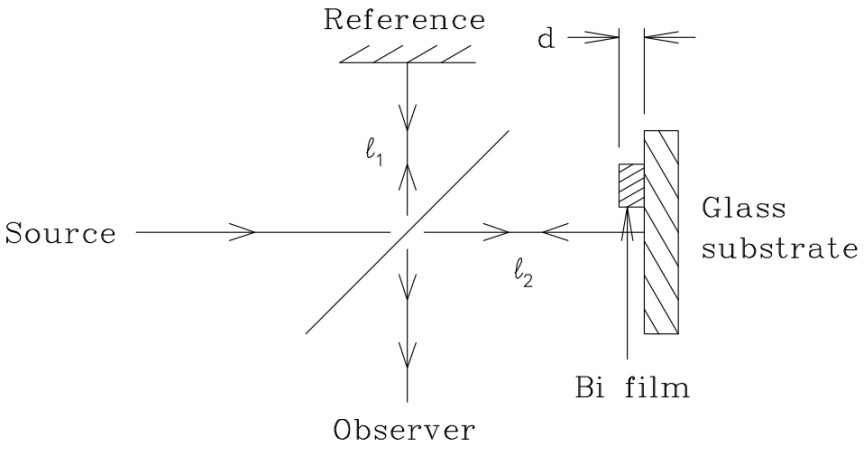

Figure 1.4: A schematic diagram of a Michelson Interferometer.

Since the film thickness d is of the same order of magnitude as the wavelength of visible light, we will use a device that uses interference of light waves to measure the thickness of the film, the Michelson Interferometer, as illustrated in the schematic diagram of Fig. 1.4. When the optical paths ℓ_1 and ℓ_2 differ by $k\lambda$ ($k = 0, 1, 2, \ldots$) the monochromatic waves of wavelength λ are reflected back to the observer out of phase, and destructive interference produces a pattern of fringes in the field of view. The spacing between fringes varies, and depends on the angle of slight misalignment of the reflecting plane from being perpendicular to the optical axis. minute adjustments can create a convenient regular grid of fringe lines of certain spacing.

The glass substrate with a thin metal film on it 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 shifts with respect to the pattern over the glass slide. From the amount of shift, in units of the fringe spacing, the thickness d can be easily calculated.

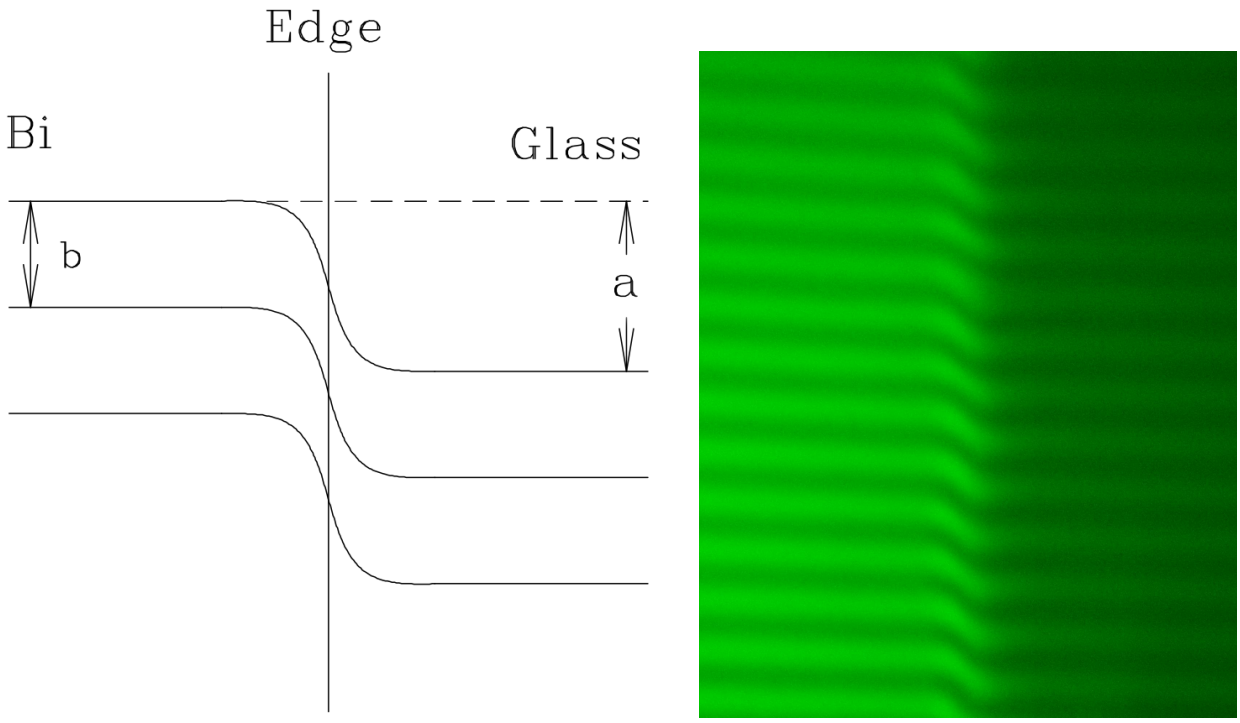

Figure 1.5: A shift in the fringe pattern can be used to find film thickness as $d = \frac{a}{b} \times \frac{\lambda}{2}$. A typical image is on the right, a schematic illustration of how to measure a and b is on the left.

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.

Thickness measurement checklist

Put the sample under a microscope, Bi-film-side up, and check the uniformity of the film, by measuring its thickness at several points all around the periphery of the sample.

1. The instructor will have put the interferometer over the microscope objective, and adjust ℓ_1 until a clear fring pattern is seen. Be careful not to disturb the alignment.

2. Place your slide (film up) under the microscope; turn the backlight illumination 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. Turn off the backlight, and switch to monochromatic top illumination by putting a Fabry-Perot interferometer filter (green) in the light path.

5. Gently adjust the vertical position of the film, until you see interference fringes (be patient). The fringes are easily destroyed by the vibrations of the apparatus: do not lean on the microscope table, make small adjustments to the vertical position and then move your hand away from the knob and let the image settle.

6. For best results, gently adjust the tilt screws of the reference mirror until the fringes are approximately perpendicular to the film edge. You should see a fringe pattern similar to the one on the right-hand-side of Fig. 1.5.

7. Use the camera to take several snapshots of your fringe pattern. Try to time your snapshots when the building vibrations are at a minimum. You may need to adjust the contrast and exposure for best results. If vibrations prove excessive, make a short video recording, from which a few good frames may be selected later.

8. Estimate b (the fringe spacing) and a (the fringe shift) from your images. You may use any software that allows you to measure distances on screen, or print the images on paper and use a ruler to make your measurements. You should measure a distance over several fringe spacings and divide by the number of fringes to receive an average value of b. Make several measurements of the shift a and calculate the average value. The film thickness is then:

$$d = \frac{a}{b} \times \frac{\lambda}{2} \tag{1.5}$$

9. Repeat at different positions along the edge of the film. You want to be ale to know both the average thickness of the film, and also have a sense of how uniform your deposition was.

References

For vacuum techniques, use books such as:

- J. Yarwood, *High Vacuum Techniques.*
- J. F. OHanlon, *A Users Guide to Vacuum Technology.*
- H. Mark and N. T. Olson, *Experiments in Modern Physics*, Ch. 4.

For a description of the Michelson Interferometer:

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