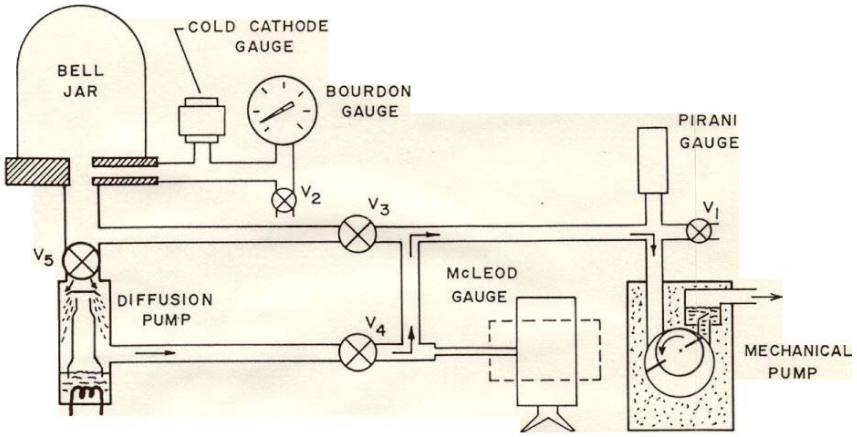
EVAPORATION OF A SILVER FILM

This experiment will give you some hands-on familiarity with the parts of a simple vacuum system. A thin film of silver will be evaporated in the vacuum and its thickness estimated by 3 methods. Such evaporation techniques have revolutionized modern electronics.

Vacuum System

The system consists of 2 pumps, 4 gauges and 5 valves for the control of gas flow. See Fig. 1.



V_1, V_2 - AIR ADMITTANCE VALVES.
V_3, V_4 - SHUT OFF (" SPEEDIVALVES")
V_5 - BAFFLE / ISOLATION VALVE.

FIG. 1

The mechanical (or roughing or backing) pump rotates an eccentrically mounted van inside a cylinder to compress and expel a volume of air once per revolution. It is used to "rough out" the system and "back" the diffusion pump. It becomes ineffective once the pressure is less than -10^{-2} mbar.

The first law of vacuum systems is: Do not leave the mechanical pump under vacuum when not running. Oil will be sucked back from the pump body. On shut down, (a) close all valves to the system, except v_5, (b) turn off pump, (c) open the bleeder valve v_1 to equalize pressure.

The diffusion pump is effective down to pressures of about 10^{-7}, but must have a backing pressure of -10^{-2} in order to operate. It works by boiling oil with a heater and back jetting the oil vapour with a system of jets, which seeps the air with the vapour stream. The upper walls of the pump are water cooled to recondense the oil, while the air finds its way to the exit. Diff pumps come with up to 14: throats, but your is a smaller 1" or 2 1/2" job.

The second law of vacuum systems is: Keep the backing pump and cooling water on while the diff pump is hot. Otherwise the oil will "backstream" into the bell jar and/or be oxidized.

Gauges: The thermal conductivity of a gas is proportional to the pressure. The Pirani Gauge has a hot wire in the vacuum space. Its resistance, measured with a Wheatstone bridge in the control unit, depends on the wire temperature and therefore p. It gives useful readings from -10^{-2} to 10^{-4} mbar. In the present case it gives the backing pressure.

The Penning Gauge monitors the current in the gas from a cold emission cathode to an anode. The electrodes are located in the field of a permanent magnet to give long trajectories. It overlaps the pirani gauge and goes to lower pressures - down to 10^{-7} mbar. It is your primary measurement of p in the bell jar.

Examine the system and locate all the parts. The various components are fully described in the detail book on this experiment obtainable from the wicket.

To run the system, you must (a) rough out the bell jar: V_1, V_2, V_5 closed, V_3 open. At the same time you can start the diff pump, water and open V_4; (b) When p – 10^{-1} torr[1] close V_3 open V_5. Note the rapid decrease in pressure as the diff pump "grabs" the bell jar. It will finally go to 10^{-4} or so.

To access the bell jar; close V_5 open V_2. When the pressure is equalized close V_2 again so you don't forget it.

To repump: close V_4 briefly and rough out with V_3 open. This admittedly is a transient violation of the 2nd Law, but it protects the diff pump from the major surge of air.

Evaporation

We want a film of –50 nm thickness. Weigh out enough silver to give this thickness at the sample position, assuming isotropic evaporation over a solid angle of 2π. A good "rule of thumb" is for every 1 nm thickness you need 1 mg of silver (explain). Load the boat.

Set a blank begin the mask and attach the electrodes - the resistance is to be monitored during the deposition with an ohmmeter.

Put on the bell jar and evacuate. Practise opening the shutter with the magnet.

When the pressure is as low as it will go, get set to turn on the boat current, then at t = 0 open the shutter and time some resistance readings after t = 0.

1. Not SI unit of pressure, 1 Torr = 1 mm Hg = 1.333 mbar.

The boat current is very critical. Increase the variac setting slowly until you notice the silver

evaporating. This will take place at a variac setting of between 65 V (– 25A) and 75 V (– 35A). When the silver starts evaporating hold the variac setting constant.

At a resistance of from 25 to 50 Ω or when you run out of silver close the shutter. Use a Hewlett Packard 3476A autoranging digital meter available from the wicket to monitor the resistance change. Turn off boat current. Turn off the Penning gauge, close V_2 open V_2. A good film will have a fairly stable resistance - probably falling a bit as the air comes in. Remove the sample.

Estimate the film thickness by finding the % transmission of blue light through it. (See Fig. 2). An optical bench with a phototube is set up somewhere. This measurement will not be feasible if the film is so thick you can't see the room lights when looking through the sample.

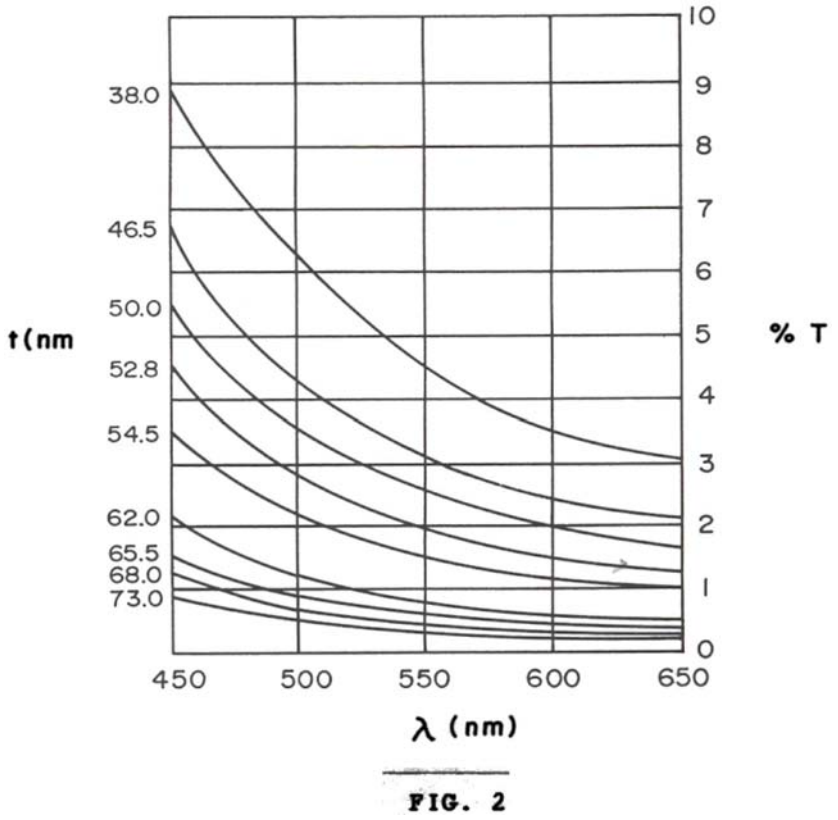


FIG. 2

Compare the result with that calculated from the final resistance value. You can take the resistivity as –4X the value for bulk silver (1.6 μΩ-cm) due to the structure present in the film. See Figs. 3 and 4. An evaporation at lower pressure and higher temperature (i.e., faster) would allow much higher quality.

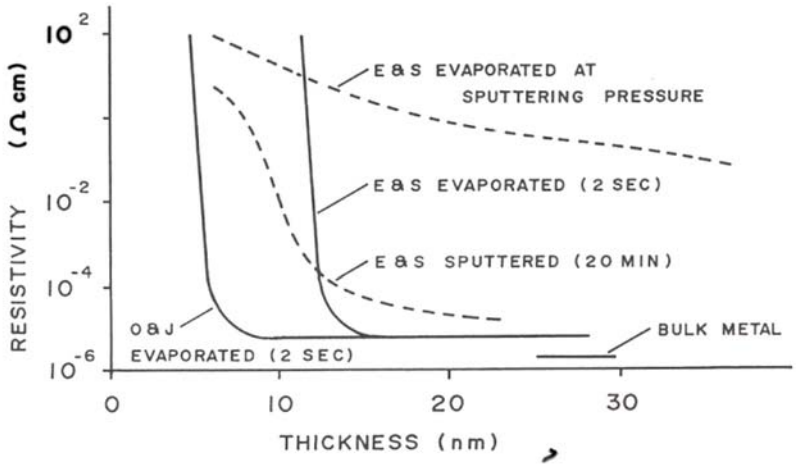
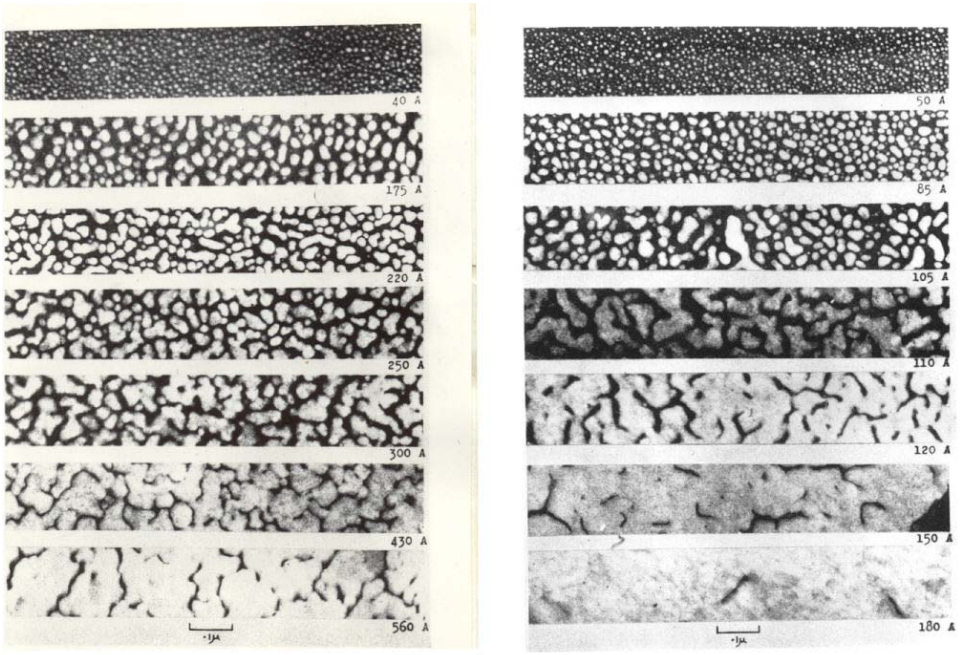


FIG 3

Comparison of the resistivity of slowly sputtered and rapidly evaporated silver films as a function of thickness. Curves taken from Ells and Scott and Oppenheim and Jaffe.



(*a*) Silver films evaporated slowly (20 min.)

(*b*) Silver films evaporated rapidly (2 sec.)

Electron photomicrographs of (a) slowly and (b) rapidly deposited silver films, after Sennett and Scott.[2] Micrographs show that silver films of up to 50 mm or more in thickness have not developed a fully connected structure when the evaporation time is prolonged (20 min.). Granulation due to slow deposition is responsible for increasing the optical absorption of silver films.

2. The Structure of Evaporated Metal Films on Their Optical Properties, R.S. Sennett and G.D Scott. A copy is available at the wicket.