HIGH VACUUM TECHNIQUES

GOAL

- To make a half silver mirror.
- To learn high vacuum techniques
- To learn vacuum deposition techniques
- To learn to write cookbook or check-list operating instructions.

INTRODUCTION

Vacuum techniques are important tools in a broad range scientific field and industrial settings, from accelerator for high energy physics, to epitaxial growth of semiconductors for the electronics industry. Usually the vacuum system is a tool, but in the case of a vacuum system it is a tool that if operated incorrectly is easily damaged. When using a diffusion pump the oil can be burnt or reduced to a tar-like substance. Because of this it is common to write a detailed cookbook procedure for using the system.

The production of a thin film is a fundamental procedure in the study of the properties of materials. It is important to use reproducible and documented procedures while making these films so that their characteristics can be changed in a controlled fashion. In this experiment, you are trying to produce a beam splitter by producing a half "silvered" or aluminized mirror. If the reflectivity is too high or low you need to be able to adjust the operating parameters to correct this on the next attempt.

Readings:

A.C.Melissinos, *Experiments in Modern Physics,* p. 126-137.

John H. Moore, Christopher C. Davis, and Michael A. Coplan, *Building Scientific Apparatus, A Practical Guide to Design and Construction* Second Addition, Addison-Wesley, Redwood City, CA, (1989) Q185.M66 1988, See Chapter 3, p 75-90.

J. Strong, *Procedures in Experimental Physics* Lindsey Publications, Bradley, Il. (1986) or Prentice Hall, Inc New York, (1938) QC41.S8. See Chapter III, High Vacuum Techniques; and Chapter IV, Evaporation and Sputtering. (This was probably the original source, from which this experiment was developed.)

S. Dushman, and J.M. Lafferty,*Scientific Foundations of Vacuum Technique,* John Wiley New York (1962) 533.5 D97 1949. See first few chapters and Chapter 10.

PROCEDURE

General Procedures

A. Make **detail diagrams** of the vacuum system, and the electrical power circuit for the filament.

B. Every time that the any part of the vacuum system is evacuated, you should **record a table of the pressure as a function of time.** One reason for doing this that a leak or other problem with the vacuum system will be apparent quickly. A second reason is that one can study pumping throughput.

C. Write a **detailed cookbook procedure** for the following operating conditions.

1. Starting up with everything cold.
2. Shutting down the system with the diffusion pump hot and LN_2 in the cold trap.
3. Opening the bell jar when the system is "hot."
4. Closing and pumping down the bell jar when the system is "hot."

Test your procedures using the simulator and on the actual equipment with the diffusion pump off..

Specific Procedures

If necessary replace the filament and the aluminum on it. Check the electrical connections. Clean the glass microscope sides that will have aluminum on them to make a mirror. Assemble the holder for the glass slide. First evacuate the entire system to about 50 mTorr using just the mechanical pump. Start the diffusion pump and evacuate the bell jar to high vacuum. Add liquid nitrogen after it is clear that there are no major leaks (that is leaks that will prevent the system from reaching 10^{-4} Torr). After the system has reached a low pressure, turn on the filament and deposit aluminum on the glass slides. Each time the bell jar is mounted its sealing edge, and the mating gasket must be cleaned and vacuum grease applied..

Plot the measured pressure in the bell jar as a function of time. Consider whether a linear, semi-log, or log-log plot is best. From this:

a. Estimate the ultimate pressure in the bell jar when pumped by the mechanical pump, the diffusion pump?

b. Determine what is the pumping speed of the system when evacuating the bell jar with the mechanical pump, with the diffusion pump?

WARNINGS

Do NOT drop the bell jar. Always put it down in a way that it can not roll off the table.

The bottom of the bell jar and its mating surface must be clean to form a vacuum seal.

WARNINGS:

Liquid nitrogen can cause severe frostbite. Be especially careful not to splash it in your eyes or anyone else's eyes. The other potential problem is get LN_2, into your shoes wear it can not flow away from your skin.

Never seal a container of liquid nitrogen. If sealed, the pressure will increase as the nitrogen evaporate the pressure until either the lid or the vessel fails (explodes)!

Constraints

Below are some of the constraints for operating the vacuum system:

1. If the diffusion pump is on, warm or hot, the cooling water must be on.
2. If the diffusion pump is on, warm or hot, the pressure in the diffusion pump must be maintained at less than 100 mTorr.
3. The Penning gauge must not be operated at pressures above 10 mTorr.
4. If there is LN_2 in the cold trap, the pressure inside the trap must not exceed 100 mTorr.

EQUIPMENT

Vacuum system

- Mechanical Pump
- Diffusion Pump
- Cold trap
- Bell Jar
- Various values and plumbing

Pressure measurement system

- Mechanical Bourdon pressure gauges (2)
- Pressure gauge controller
- Cold cathode ionization gauge
- Thermocouple gauges (2)

Power supply 20 A
Tungsten wire
Aluminum wire

QUESTIONS

1. What are the relationships between the following units of pressure:
   - Torr
   - mTorr
   - Atmosphere
   - Pascal
   - mm of mercury
   - micron
   - psi

2. What is the average velocity of a gas molecule in terms of its molecular weight and its temperature ? What is this for a nitrogen molecule and 20° C ?

3. What is the average diameter of a gas molecule of nitrogen, of aluminum?

4. What is the mass of a gas molecule of nitrogen, of aluminum?

5. What is meant by the term ***mean free path***?

6. What is the relationship between pressure and the mean free path for a given molecule?

7. At 25° C for nitrogen, what is the number of molecule/cm^3, and the mean free path at the following pressures (in Torr) : 760, 1, 10^{-3}, and 10^{-6}

8. Make a table of containing pressure (maybe every power of 10), mean free path, appropriate pressure measuring device, appropriate vacuum pump.

9. What safety interlocks exist on this vacuum system and how do they help to protect the system and satisfy the operating constraint given above.

10. How do the following kinds of pumps work and over what range of pressures do they function:
    - Mechanical pump?
    - Diffusion pump?

11. How do the following kind of gauges work and over what range of pressures do they function:
    - Bourdon gauge?
    - Thermocouple gauge?
    - Cold cathode ionization (or Penning) gauge?

GOING FURTHER

1. There are three methods of estimating the temperature of the filament. A) The resistance of tungsten as changes with temperature. How can this change be used to monitor and quantify the temperature of the filament? B) The second method is to visually observe the color of the filament. An optical pyrometer is also available for estimating the filament temperature C). The third method is to calculate the power supplied to the filament and to assume the validity of the Stefan-Boltzmann law and work from there. Compare these three methods. What are the limitations of each method with the equipment at hand.

2 How do the following kinds of pumps work and over what range of pressures do they function:

- Cryo-pump?
- Ion pump?
- Sorption pump?

3. How do the following kind of gauges work and over what range of pressures do they function: gauge?

- Hot cathode ionization (Bayard-Alpert) gauge?
- Mercury manometer
- McLeod gauge?

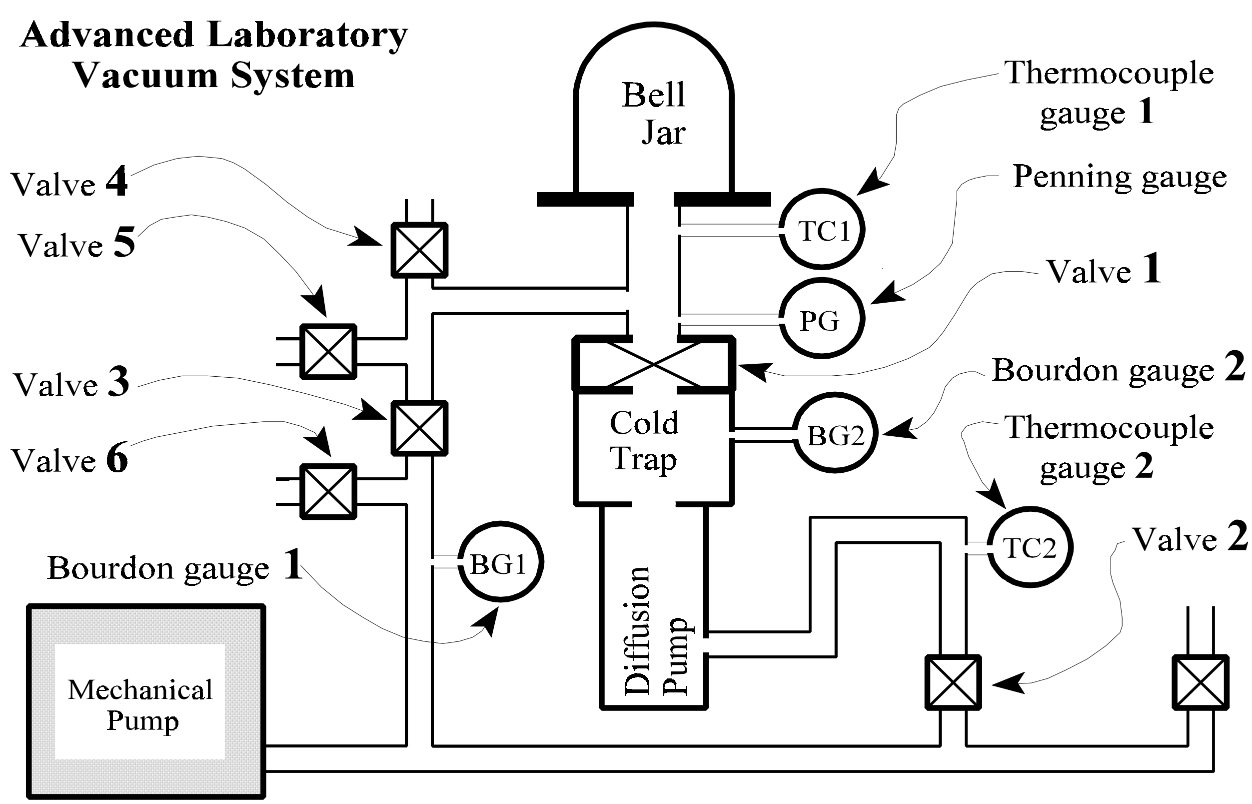
4 Describe how a ball valve and a butterfly valve work.

5 Describe how a cold trap works.

6 In what pressure range do molecular collisions dominate the movement of the gas, and in what pressure ranges do molecule-chamber-wall collision dominate?

7 What determines how quickly a system can be evacuated? How does the pumping capacity of the pump, the size of the chamber, the diameter and the length of the connecting pipes affect the pumping speed?

Consider a chamber at some initial pressure connected by way of a small hole to a second chamber with a large (approximately infinite) volume. If the pressure is the second chamber is initially zero describe the pressure in the first chamber as a function of time.? How is this pressure function related to the size of the opening connecting the two chambers? Hint: Consider how any one atom escapes the first chamber.



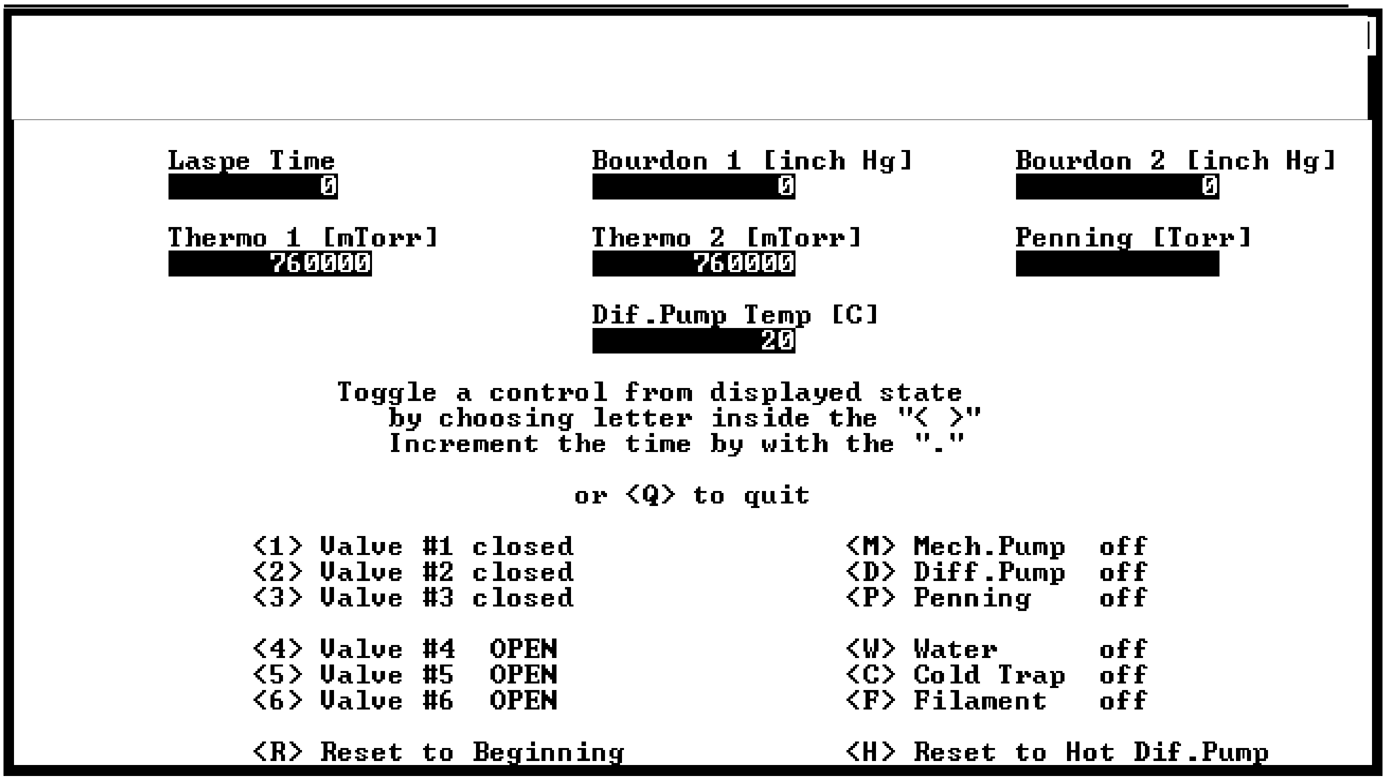
VACUUM SYSTEM SIMULATOR

The Vacuum System Simulator is a program written in Pascal that does a crude simulation of the vacum system used in the University of Arizona's Advanced Physics Labs. The program was written as a text only program for increased portability and ease of programing. The program was written locally so you may take a copy home with you, or download it from the web.

The program name is

`vacuum.exe`

and it is usually in a directory named `vacuum` but this may vary from system to system. Below is the screen appears when the program is started.



The top half of the screen consists of mostly pressure indicators while the lower portion of the screen shows the controls for the valves, pumps, etc. One toggles a given control device by pressing the letter or number within the preceding "< >". For example, to open Valve # 1 one simply presses the key labeled: **1**.

Unlike the real system where time moves at its own pace, in this simulation the operator controls time. To progress a unit of time press the period **.** key.

The purpose of this simulation is for you to make your mistakes on it rather than one the real vacuum system where things can be damaged. The simulator will merrily announce any simulated damage you produce. One can reset the system to startup with the **R** key or to a hot state with the **H** key.

Exit the program with the **Q** key