

Lab rotation on vacuum techniques and thermal imaging

Objectives

- Manually operate a high vacuum system through pump down and vent routines.
- Learn appropriate vacuum system terminology – “roughing, backing, foreline, gate valve etc.” by following instructions during the operations.
- Measure residual gas composition at different pressures during pump down.
- Measure the difference in pump rate of a chamber vented with air versus a chamber vented with N₂.
- Measure the effect on the composition of residual gases in a vacuum chamber by heating a tungsten filament until it glows red-hot.
- Use a thermal camera to image a heated crucible and measure its temperature.

Suggested Experiments

Record the partial pressure of H₂O, H₂, N₂ and O₂ during the vacuum conditions at the start of the session. Recording 5 minutes of data should be enough to give you a high vacuum baseline of the residual gases in the chamber. Start with both the pumping and process chambers under vacuum and the RGA and ion gauges switched off. You should leave the system in this condition at the end of the session.

Vent the chamber using liquid nitrogen and then pump it to vacuum again. Once you enter the high vacuum regime you should record the pump rates of the different gases in the chamber using the RGA (you should record

$$P_n = A_n e^{-\lambda_n t}$$

data for about 20-30 minutes). Fit exponential functions of the form to the partial pressure (P) of each gas (n) as a function of time (t). Determine A (relative initial partial pressure) and λ (rate constant) and composition of each of the different gasses in the chamber.

Vent the system using air instead of liquid nitrogen. Repeat the pump-down experiment again. Compare A and λ for each of the different gasses on this second pump down cycle. Are the parameters you record the same or different?

Heating the furnace With the Process Chamber a low pressure ($\sim 10^{-4}$ mbar) turn on the power source for the furnace system. Start at a low voltage (e.g. < 1 V), and observe (record) what happens to the gas levels within the chamber. Gradually increase the power by controlling the voltage. You will notice that as the filament gets hotter its resistance increases and power output drops.

You should continue to gradually (over a minute) increase the voltage across the filament until it glows red-hot. Record the current as a function of voltage. Plot a graph. Don't go above 35 A.

Finally, after recording a few minutes of data at different voltages select a fixed voltage and measure how long it takes for the current to stabilize. Continue to check the residual gases in the chamber and the total pressure in the chamber. Which gasses are released during the heating process?

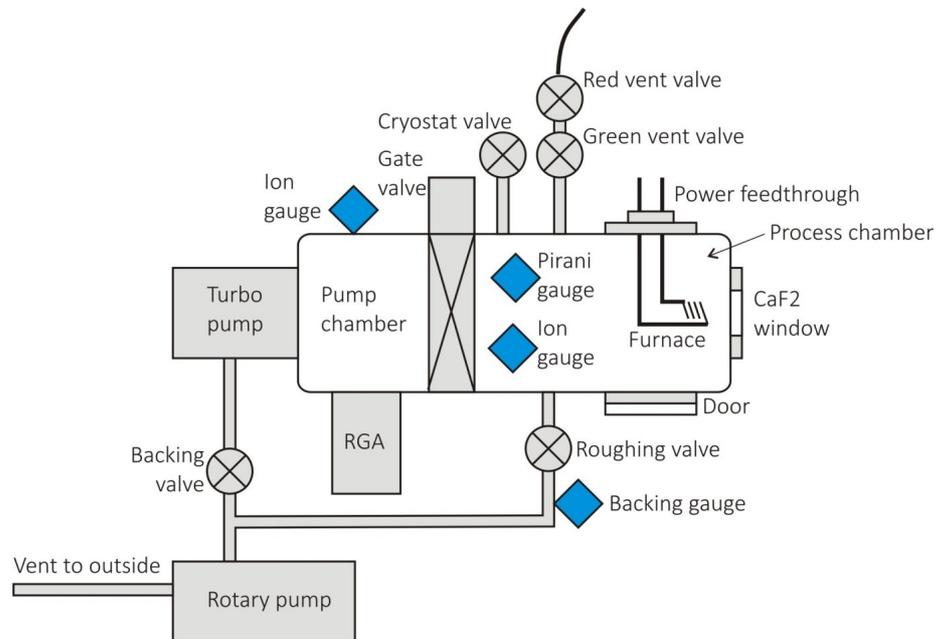
Following heating, ensure cool-down time before venting the chamber and exposing hot surfaces. Cool down depends on final temperature reached. Typically 10-20 minutes will suffice but at very high temperatures 30 minutes might be required.

Characterizing coil heating You should characterize the heating of the coil during current flow using a thermal camera Use the camera to measure the temperature of the coil as a function of current flow, and explore temperature uniformity across the coil. Explain your findings.

Vacuum system: hints and tips.

Study the drawing above and make sure you can correctly identify the different system components. You will need to discuss and confirm the components with your supervisor and you will be tested before you can continue.

The vacuum system is shown schematically in the accompanying figure. The system is



composed of two chambers; a pump-chamber connected to a turbo pump, and a process chamber connected to the pump chamber via a gate valve. The process chamber is also pumped to coarse vacuum using a roughing pump.

Within the process chamber, there is a heated filament that can be used to deposit thin films via thermal evaporation. Optical access to the process is available via a door with a glass window.

A residual gas analyzer (RGA) is also connected to the pump-chamber. This is used to sample the different gasses in the chamber through measuring their atomic weights and determine their relative partial pressures. There are also two ion gauges, a Pirani-gauge and a backing gauge that are used to record the vacuum pressure in different parts of the system.

Caution - important information:

At no point should you ever open the cryostat valve

You should ensure the vent to outside line is intact and functioning!

You should not operate the RGA or ion gauge unless the chamber pressure is $< 10^{-4}$ torr (10^{-4} mbar)

The backing valve should not be closed for more than a few minutes otherwise the turbo pump will stall

The rotary pump should always provide a low backing pressure for the turbo unless it is being used to "rough" (reduce pressure from atmosphere) the process chamber

RGA SOFTWARE

Start the RGA software by searching on computer for 'Easy View' software. Then run graph (trend to follow pressure). Make sure you switch the filament 'on' by clicking on the "^^^" symbol.

Select masses: 2 (H₂), 18 (water), 28 (CO and N₂), 32 (O₂)

Save data using the Disc icon - you should check that you can save and read the data and plot it in excel before continuing.

Note that when the ion gauge is on, H₂ levels will rise in the chamber as the filaments outgas.

PUMP-DOWN PROCESS IF ENTIRE SYSTEM IS AT ATMOSPHERIC PRESSURE (You shouldn't need to do this)

- 1 Make sure door is closed.
- 2 Close red and green valves.
- 3 Open Gate valve.
- 4 Close Roughing valve.
- 5 Open backing valve.
- 6 Switch on roughing pump (rotary pump).
- 7 Switch on turbo pump.
- 8 Wait until Edwards ADD unit (backing gauge) starts to record a pressure (about one minute). This measures the backing pressure in mTorr.
- 9 When Pirani gauge pressure is below 1 mTorr ($\sim 10^{-3}$ mbar), switch on the ion gauge.
- 10 System is now under vacuum and ready.

VENT PROCESS CHAMBER TO ATMOSPHERIC PRESSURE

- 1 Close Gate valve and Roughing valve.
- 2 Put hose leading from Red / Green valves into a bucket of liquid nitrogen (or leave in air if vacuum base pressure not critical).
- 3 Open Green Valve.
- 4 Open Red Valve.
- 5 N₂ (or air) will be sucked into the chamber.
- 6 Door can be opened when chamber is at atmosphere.
- 7 Close red valve.

PUMP-DOWN PROCESS IF PUMP CHAMBER IS UNDER VACUUM AND PROCESS CHAMBER IS AT ATMOSPHERIC PRESSURE

- 1 Make sure door is closed (but not too tightly).

- 2 Ensure red valve is closed.
- 3 Close Backing valve.
- 4 Open Roughing valve.
- 5 Watch Backing gauge pressure drop (takes about 1 minute to get a reading). Also watch the Pirani gauge drop too.
- 6 When pressure of Pirani and backing reaches <100 mTorr (this is the same as $\sim 10^{-1}$ mbar), close the Green valve, then close the roughing valve, then open the Backing valve. This should be done relatively rapidly.
- 7 Slowly (over 20 seconds or so) open the Gate valve. Start slowly and get faster.
- 8 Switch on the RGA and ion gauges (if needed) and record the pressure of the different gasses in the chamber.

FLIR A6750sc Thermal Imaging Camera: Hints and tips

Check that the camera is connected to the PC via the GigE cable.

Connect the FLIR power supply.

Switch camera on and wait for the 'cold' light to come on (~ 5 -6 mins).

Open the ResearchIR software.

Go to camera/select to connect to the camera (software may take a couple of minutes to find the camera).

Choose the appropriate temperature range:

- o NB: 'empty' means no filter - for temperatures lower than 350 °C.
- o For higher temperatures the ND2 filter must be added to the lens.

Select the appropriate acquisition frame rate.

Set the parameters (NB: can also be done after recordings are taken):

- o Emissivity,
- o Distance,
- o Ambient temperature,

Focus the camera using the manual focus.

Set the recording settings using the tool box in the options along the top of the displayed image.

Record video or snapshots.

Open saved file.

Go to tools/'make self viewing file' to save the file for further analysis on any PC.

Use the analysis tools along the left/top of the displayed image to extract temperature data for spots, lines or areas of the images.

Switch camera off after use.