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## 1 Introduction

This is the laboratory script for the F-Praktikum Lab Course on “**Vacuum Science and the Influence of Vacuum Conditions on Thin Film Growth**”. During this two day course, the students will work with a high vacuum sputtering chamber and learn about how a vacuum is created and maintained as well as how the vacuum quality can be influenced. The script is organised as,

1. Notes about safety during this course
2. Some brief theory
3. Learning objectives and an overview of the two days
4. Details on the experiment to be performed over days 1 and 2
5. An appendix with additional background information on equipment and techniques used in the course

## 2 Safety

It is important to note that this experiment has two major dangers to the safety of the student.

The first is the high voltage (HV) cable to power the targets. It is FORBIDDEN for the student to touch this cable (indicated by a label saying “High Voltage”). The Student Assistant is the only person allowed to touch this cable, if needed, ask.

The second major danger is known as “Over-Pressure”. When venting the chamber (see section 5.1.1), take care to loosen the bolts on the main window. This is because, as the pressure inside the chamber rises, strain is put on the chamber. If the pressure inside the chambers is greater than atmosphere, this can cause the weakest parts of the chamber, the windows, to balance the pressure difference by exploding outwards. By loosening the bolts, as the pressures inside and out become equal, the main window opens.

## 3 Theory

Theory on thin films, basic gas theory and also sputtering can be found in this section however, the attached appendix contains more information about the workings of pumps, extending gas flow theory, pressure gauges etc.

### 3.1 Gas Theory

It is expected that the student has a knowledge of gas theory. Pages 29-66 of from Handbook of Vacuum Technology (Second Edition), edited by Karl Jousten, Wiley-VCH, 2016 (reprinted 2017), ISBN: 978-3-527-41338-6, give a good introduction. The key points of ideal gases are summarised here.

Unlike a solid or liquid, where the bonds between particles define the volume of the substance, a gas will fill the entirety of a container homogeneously. The larger the container, *volume V*, the thinner the gas will be. The gas in the container will exert a force, *F*, on the walls with area *A*. It is convenient to introduce the value of pressure *p* defined as the ratio of the perpendicular force exerted on a surface element of the container to the total surface area of the container,

$$p = \frac{F}{A} \tag{1}$$

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The units of pressure in the SI system is the pascal (Pa) where 1 Pa is the pressure at which a force of 1 N is exerted perpendicular to a surface of 1  $m^2$ . There are several units of pressure that are used around the world including bar (1 bar =  $10^5$  Pa), millibar (mbar), Torr and psi.

The pressure of a gas in a container changes when the temperature changes, meaning that the temperature is also an important quantity when talking about the state of a gas. The thermodynamic temperature scale uses the Kelvin (K) rather than Celsius ( $^\circ\text{C}$ ) to measure temperature, where 0 K is absolute zero and equal to  $-273.15\text{ }^\circ\text{C}$ . Kelvin and Celsius both have the same scale divisions so to convert between the two scales we use,

$$\text{Temperature in Kelvin} = \text{Temperature in Celsius} + 273.15 \quad (2)$$

We can therefore summarise that the three main variables to describe the state of a gas in a closed container are,

1. volume V,
2. pressure  $p$ ,
3. temperature T

These three variables are known as **State Variables**. There are also several other variables that can be used to describe the amount of a gas, the mass m, the number of particles, N, and the amount of substance,  $\nu$ . It is often inconvenient to measure gas as mass due to the small value as well as describing the gas in terms of the number of particles often provides more information as to how the gas behaves. We can scale the number of particles to the amount of substance that we have via,

$$\nu = \frac{N}{N_A} \quad (3)$$

where  $N_A = 6.022142 \times 10^{23} \text{ mol}^{-1}$  is known as Avogadro's constant and this equation relates the number of particles in a mole of substance ( $6.022142 \times 10^{23}$ ) to how many particles we have.

In the 17<sup>th</sup> and 18<sup>th</sup> centuries, experiments conducted by Boyle, Mariotte, Charles and Gay-Lussac led to the development of the equation of state for an ideal gas,

$$\frac{pV}{T} = \text{constant} \quad (4)$$

where the constant is proportional to the amount of gas in the system. This leads to three ideal gas laws depending on whether the amount of gas is measured in mass m, particle number N or amount of substance  $\nu$ ,

$$pV = mR_s T \quad (5)$$

$$pV = NkT \quad (6)$$

$$pV = \nu RT \quad (7)$$

where the above equations include the fundamental constants; the Boltzmann's constant ( $k = 1.380650 \times 10^{-23} \text{ J K}^{-1}$ ) and the molar gas constant ( $R = 8.314472 \text{ J mol}^{-1} \text{ K}^{-1}$ ). The constant  $R_s$  depends on the gas species ( $R_s = \frac{k}{m_p}$  where  $m_p$  is the mass of the particle species).

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### 3.1.1 Kinetic Theory of Gases

A gas will complete fill an available volume and display several macroscopic properties such as the temperature of the gas as well as the exertion of a temperature dependent pressure on the walls of the container. A gas additionally conducts frictional forces between surfaces in motion (viscosity), transfers thermal energy between surfaces with uneven temperatures (thermal conductivity) and influence the spreading of molecular particles (diffusion). These properties can be understood by considering the microscopic behaviour of individual particles by using the kinetic theory of gases. This theory is based on the assumption that a gas consists of a large number of particles moving thermally. The moving particles hit the walls of the container as well as each other where all collisions are assumed to be elastic and hence conserve energy. Using the model developed by Krönig in the 19<sup>th</sup> century, the pressure on the walls of a container can be calculated from the molecular impacts of many individual particles. This model also allows the transport properties of viscosity, thermal conductivity and diffusion to be calculated.

In the simplest form, the kinetic theory assumes that all gas particles are hard, incompressible spheres with a fixed diameter although it can be extended to include soft spheres that deform during collisions as well as mutually attract each other. For the hard-sphere model, we have N particles of mass  $m_p$  in a cube of edge length d and volume V. The particles are negligibly small and homogeneously distributed in the volume, moving randomly. The particle motion is distributed isotropically so that 1/3N particles are moving along (or reversely) each of the three axis (x-, y-, z-axis). This movement is described by the particle velocity  $\mathbf{c}$  with three components  $c_x$ ,  $c_y$  and  $c_z$ . We will consider a particle moving along the x-axis with constant velocity  $c_x$ .

Before a collision, the particle has momentum  $m_p c_x$  and afterwards momentum  $-m_p c_x$ , a momentum change of  $2m_p c_x$ . According to the laws of mechanics, the force exerted on the wall is a product of the change in momentum per collision and the collision frequency. To calculate the wall pressure, we divide this value by the wall's surface area,

$$\text{Wall pressure caused by a single particle} = \frac{m_p c_x^2}{d^3} \quad (8)$$

$d^3$  can be written as the volume V. The pressure applied to the wall by the total gas is then obtained by multiplying this equation by the number of particles hitting the wall (1/3N). We can then rearrange this to,

$$pV = N \frac{m_p c_x^2}{3} \quad (9)$$

Comparing to the ideal gas law in the previous section, we can see that these two equations are equal if the average velocity  $c_x$  is,

$$c_x = \sqrt{\frac{3kT}{m_p}} \quad (10)$$

This equation tells us that the speed of a particle increase with temperature and that heavier particles are slower than light ones.

Maxwell-Boltzmann statistics can be used to describe thermal ensembles of particles more completely as a probability distribution function in three dimensions,

$$f(v) = \frac{m}{2\pi kT}^{\frac{3}{2}} 4\pi v^2 e^{-\frac{mv^2}{2kT}} \quad (11)$$

which can result in three thermal speeds for a given particle, the most probable speed,

$$v_p = \sqrt{\frac{2kT}{m}} \quad (12)$$

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the mean speed which is the expected value of the speed distribution,

$$\langle v \rangle = \int_0^\infty vf(v)dv = \sqrt{\frac{8kT}{\pi m}} \quad (13)$$

and finally the root mean square speed which is the second-order moment of speed and what we derived above,

$$\sqrt{\langle v^2 \rangle} = \left( \int_0^\infty v^2 f(v)dv \right)^{1/2} = \sqrt{\frac{3kT}{m}} \quad (14)$$

### 3.2 Adsorption and Desorption

Please also see Chapter 6, pages 229-241 and 247-256 from Handbook of Vacuum Technology (Second Edition), edited by Karl Jousten, Wiley-VCH, 2016 (reprinted 2017), ISBN: 978-3-527-41338-6 for more information.

Atoms or molecules impinging on a surface have a probability of sticking to the surface or being reflected. These particles adhere to the surface via either physisorption (dipole or van der Waals forces) or covalent linkage (chemisorption/atomic bonds). The energy binding the molecule is known as the adsorption energy,  $E_{ad}$ . For these molecules to desorb (leave the surface), an expenditure of energy is required, called the desorption energy  $E_{des} = E_{ad}$ . The molecules can also absorb into the surface, they dissolve into the solid, requiring absorption energy  $E_{ab}$  to escape the surface, although we will not consider absorbed particles here.

When a vented, high-vacuum is evacuated such as when we open a chamber to load a sample or change targets, adsorbed particles can delay or prevent a certain, low pressure being reached. Gases such as  $O_2$ ,  $N_2$  and water vapour that are adsorbed or absorbed at higher pressures, such as when we vent a chamber, are released at different rates in a vacuum depending on the value of  $E_{des}$ .

Molecules adsorbed into the surface of a chamber have a kinetic energy  $E_{kin}$  related to their thermal vibrations. To desorb, they must have  $E_{kin} > E_{des}$ . From Boltzmann statistics, for an ensemble of  $\tilde{n}$  particles, only  $\Delta\tilde{n} = \tilde{n}\exp(-E_{des}/(RT_w))$  meet this requirement, where  $T_w$  is the temperature of our surface (chamber wall). We can then assume that the molecules oscillate as thermal oscillators, with a frequency  $\nu_0 = \frac{kT}{h}$  where  $h$  is Planck's constant, and define the desorption flux density,

$$j_{des} = \frac{d\tilde{n}}{dt} = -\nu_0 \tilde{n} \exp\left(-\frac{E_{des}}{RT_w}\right) \quad (15)$$

This equation is known as the Polanyi-Wigner equation. From this equation we can see that the rate of desorption is related to the temperature of our chamber.

### 3.3 Thin Films

Thin films are defined as two dimensionally confined layers of materials grown on a substrate material for mechanical support and strength. Due to the scaled down dimensions there arises many interesting properties which are not observable in the bulk form. Owing to these properties thin films therefore have many uses in various industry applications (Photovoltaic, Semiconductors, Optoelectronic, Data storage etc). Thin films can be grown of various different types of semiconductors, ferromagnets or any transition metal.

Different processes are used to deposit different kinds of materials depending on the ultimate thickness, and the quality desired from the growth process. There exist two broad categories of thin film deposition Physical Vapour Deposition (PVD) and Chemical Vapour Deposition (CVD). PVD processes rely on the ejection of atomic species from a target source either due to evaporation or sputtering, whereas CVD processes rely on changes in the physical properties during chemical reactions. There are basically two different

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kinds of CVD processes one which involves a film which is grown from a medium due to a certain chemical formations, typical methods of this kind of growth are electroplating, vapour phase deposition and chemical reduction plating. Another category of CVD processes involve the formation of a film by the use of precursor gases e.g. iodization, gaseous iodization, thermal growth.

For the purposes of this lab course, we will focus on PVD processes. Before starting this Praktikum, it is encouraged that the student research the processes that will be utilised. The basics are outlined in the appendix as well as chapters from the Handbook of Vacuum Technology edited by Karl Jousten which is available from the university library (Library reference: UX 1500 JOU2 (2))

Two key resources are

- Kiyotaka Wasa and Shigeru Hayakawa. *Handbook of Sputter Deposition Technology: Principles, Technology and Applications*. Noyes, 2012.
- Karl Jousten. *Handbook of Vacuum Technology 2<sup>nd</sup> Edition*. Wiley-VCH Verlag GmbH & Co. KGaA, 2016

### 3.3.1 Sputter Deposition

Sputtering is a mechanical deposition process and is defined as the physical bombardment by ionised atoms onto a target surface which leads to an erosion of the target species and a subsequent deposition on the substrate. This process is dominated by physical momentum exchange between the bombarding ion and the required material for deposition. The material atoms are mechanically ejected from the target surface and deposited onto the substrate. This process is very versatile and can be used to deposit a wide variety of materials. A gaseous species is used as a precursor to catalyse the process. In most cases, Argon is used as the sputtering gas, however Kr is also used at times. The purpose of carrying out sputtering in the presence of an inert gas environment is that the gaseous atoms are ionised and used to bombard the target species. An inert gas is used to prevent any modification of the chemistry of the target atoms.

Once the gas is ionised, the target atoms along with the gaseous ions form a plasma and by applying a bias voltage, the target species are adhered onto the substrate. The entire process with the physical mechanisms involved is outlined below: A DC voltage is applied to the diode. Free electrons in the chamber are accelerated due to the electric field towards the cathode and inelastically collide with the Ar atoms. This then leads to two processes. Excitation of the Ar leads to formation of Ar ions plus the release of secondary electrons. The secondary electrons then further the process of sputtering by ionising further Ar atoms.

Requirements for self-sustained discharge: In order to have a continued process of sputter deposition there are two primary restrictions which should be met.

1. The cathode-anode distance should be greater than the dark space length, if this is not the case there is very little Ar excitation and plasma cannot be sustained.
2. There should be sufficient Ar pressure in the chamber. If the number of Ar atoms is too low there would then be a large mean-free path and the secondary electrons may reach the anode before colliding with Ar atoms and hence the plasma will not be sustained.

It is therefore crucial in order to have a continued plasma glow discharge that there is a relatively high pressure in the chamber. However, in a high pressure environment the mean free path measured is relatively low which implies that the sputtered atoms have to undergo many collisions before reaching the substrate, this leads to a reduction in the deposition rate for high pressures. Furthermore, higher Ar flow leads to the

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possibility of Ar implantation within the substrate material.

It is therefore desirable to obtain low chamber pressures. One method to do this is to use Magnetron sputter deposition. As opposed to diode sputtering strong permanent magnets are located behind the target materials in the N-S-N configuration. These magnets confine the secondary electrons produced from the ionisation of Ar to near-the-surface of the target. The magnetic pole pieces are oriented such that the magnetic field lines are parallel to the cathode surface. Magnetron sputtering therefore allows for chamber pressures to be lowered while maintaining the process of sputtering.

An advantage of using sputtering as compared to evaporation is that due to its mechanical process of sputtering away target species, the stoichiometry of the metal compounds is preserved during sputtering since all components are sputtered with a similar rate. There is also better step coverage as compared to thermal evaporation. Sputtering is a very versatile PVD process as it is possible to control compositions by co-depositing two different materials at the same instance.

Using a DC voltage can allow a large number of metals to be sputtered. However, if one requires sputtering of oxides or other insulating materials, applying a DC voltage will lead to a charge build up on the target surface, this charge has to be removed otherwise the process of sputtering will stop. There are two methods which allow the removal of charge build up. One is by a natural process by removing the applied voltage and the charge will dissociate on its own. The second is to physically repel the charge away. This is done by using an RF (Radio Frequency) power supply. However, for the purpose of this F-Praktikum R.F. sputtering will not be used.

## 4 Learning Objectives

The lab course is designed to last over two days. During this period, the student is encouraged to keep a lab book detailing all of the necessary parameters in the experimental set-up. This is intended to instil good scientific practice in the student. It is required during the growth of thin film materials that extensive records of the procedures are kept so that the scientist is able to accurately reproduce the experiment. The student will learn about how vacuum systems work, calculating several key parameters of the system as well as learning about how the quality of the vacuum can be altered and how to deposit a thin film sample via sputtering. Tasks in this course will include:

- Measurement and calculation of pumping capacity
- Loading a substrate and introducing impurities to the chamber
- Activating a Turbo Pump and calculating the rate of desorption
- Measuring the quality of the vacuum using a mass spectrometer
- Baking out the chamber and observing the change in the vacuum quality as a function of temperature
- Generating a stable plasma and depositing material onto a substrate

## 5 Day 1

On the first day of the experiment, we will first compare how the pumping speed for our chamber is affected depending on the tubing that we choose. We will also observe the limit of using only the pre-pump to try and obtain a good base pressure in the chamber. After loading a substrate for the second day, we will pump the chamber using a turbo pump and take a spectrograph of the elemental composition of the atmosphere in the chamber and then bake the chamber.

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## 5.1 Pumping Speeds

The first part of this laboratory course will investigate the pumping speed of the pre-pump. To do this, the student will vent the chamber and measure how long it takes to pump the chamber using the pre-pump for a variety of tubing diameters.

### 5.1.1 Venting the Chamber

Venting the chamber is the act of bringing the chamber up to atmospheric pressure. To do so, start by ensuring the brass metal cap is securely fastened to the vent valve of the turbo pump. Use the PV turbo viewer computer program to turn off the turbo pump. Observe the speed of the turbo pump. The turbo should never be vented whilst running at full speed as it will cause damage to the internal mechanism of the pump.

Switch off the pre-pump by unplugging it from the power supply and wait for the turbo to slow to 30% (18 000 rpm) which will take around 20 minutes.

Once the turbo has slowed down enough to vent, open the vent valve by using the PV turbo viewer program. A yellow LED should light up on the vent valve at the base of the turbo pump. This LED tells you that the vent valve is now open. Check the pressure in the chamber. It should still remain in the high vacuum range of  $< 10^{-7}$  mbar. Gradually unscrew the brass valve on the side of the turbo and watch the pressure of the chamber increase. The full range pressure gauge will go out of range and display “OR” as the pressure reading. Check the pressure reading on the Agilent VEE program for the actual pressure reading.

Loosen the bolts on the main window to avoid creating an over-pressure and wait for the pressure inside the chamber to rise to atmosphere. The chamber is now vented. *Note: Take care when venting that the turbo is not running at full speed so as to not damage it. Please also make sure to loosen the bolts on the main window.*

### 5.1.2 Changing Tubings

By connecting tubing of different diameter between the pre-pump and the chamber, measure the pressure of the chamber as you pump with the 25 mm tube, the 40 mm tube and then both in series. Save each data file in the format ”YYYYMMDD\_YourNames\_TubeDiameter”.

To pump the chamber, close the brass venting valve and then use the PV turbo viewer program to close the electric vent valve. The yellow LED should now be off. Plug the pre-pump in at the mains and check the pressure decreases. Listen for any leaks as a high frequency noise. If you hear a leak, check the bolts on the main window and also your connection of the tube between the chamber and the pre-pump. After you have recorded the pressure as a function of time for each tube, follow the instructions in Section 5.1.1 to bring the chamber back to atmospheric pressure before changing the tube.

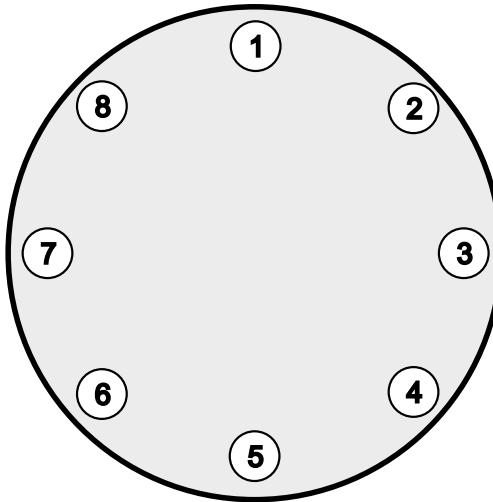
Using equation 17 in section A.1.3, plot your pressure data as a function of time and calculate the pumping speed of the pump for each diameter tube for the regime where the equation holds. What causes the equation to fail at lower pressures?

## 5.2 Loading a Sample

Before we pump down the chamber using the turbo pump, section A.2.2, we need to load a sample. Take an omicron holder and attach a silicon (Si) substrate using Kapton tape. Wear gloves when doing this. Follow the instructions in section 5.1.1 to vent the chamber, remove the nuts from the bolts of the front window,

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leaving the bolts in the holes. If you remove the bolts, when the pressure equalises, the window will fall off and break. Wait for the window to open (don't pull it, just wait), load the omicron holder with the sample face down into the slot inside the chamber. Hold the window and insert the bolts. Add the washers and nuts and tighten the bolts in pairs. When you tighten a bolt, always tighten the one directly opposite next. Don't over tighten the bolts, just to the point where to tighten further would require effort. Looking at the simple example in Figure 1, we would tighten bolt 1 gently, then bolt 5, then bolt 3 and 7, then 2 and 6 and finally 4 and 8. After we have gently tightened all these bolts, work back around in the same order and tighten again.



**Figure 1:** Simple example for bolt tightening. Tighten bolts in pairs so we would tighten bolt 1 gently, then bolt 5, then bolt 3 and 7, then 2 and 6 and finally 4 and 8. After we have gently tightened all these bolts, work back around in the same order and tighten again. No need to over tighten.

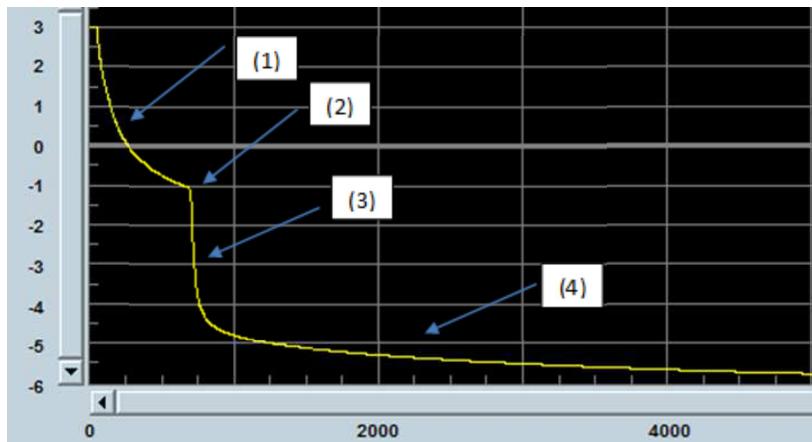
### 5.3 Turbo Pumping

Start measuring the pressure of the chamber utilising the VEE program on the computer, naming the file with the format "*YYYYMMDD\_YourNames\_PumpDownPressure*". Close the venting valve of the turbo and activate the pre-pump as detailed in section 5.1 and wait for the pressure to reach  $1 \times 10^{-1}$  mbar before activating the turbo. Wait for the turbo to reach full speed and make a note of the rpm and power. Once the turbo catches speed, the pressure will begin to drop in orders of magnitude. When it reaches full speed, the turbo should be in the  $1 \times 10^{-5}$  mbar range and dropping. The chamber is now being pumped down.

We will utilise the data taken in this section to calculate the desorption of gas in the chamber. From section A.2.2 we learnt that a turbo pump at full speed can remove 300 litres of gas a minute. Our chamber is 30 litres which means that the turbo has almost immediately removed all of the gas in the chamber once it is started. How then is it that we continue to lower the pressure as we pump longer?

### 5.4 Mass-Spectrometer

Once a pressure of below  $1 \times 10^{-6}$  mbar is reached, we can activate the mass spectrometer (QMS) attached to the chamber. Some brief details on operation can be found in the appendix. A word of warning, if the QMS is activated above this pressure, it will break. This is an expensive piece of equipment so please take



**Figure 2:** Change in pressure as a function of time. (1) shows the pressure drop on activating the pre-pump. (2) indicates the start of the Turbo pump. (3) Orders of magnitude of pressure drop which occurs within the time frame of the turbo speeding up. (4) Gradual decrease of pressure

care so that future students also have the opportunity to use it.

Every time a sputtering chamber is opened to the atmosphere, air enters the chamber and contaminates the targets. We maintain a vacuum and we sputter in an argon (Ar) atmosphere so the added oxygen ( $O_2$ ), carbon dioxide ( $CO_2$ ), nitrogen ( $N_2$ ), water ( $H_2O$ ) etc. are all unwanted compounds within our chamber.

In this section, we will assess the quality of our vacuum. First, make a note of the pressure we have in the chamber. Start the QMS and measure the spectrum of elements within the chamber. To do this, open the software on the computer for the QMS and select a Faraday bar graph. Click to connect the QMS to the software in the lower right window of the control program. Once the QMS is connected, you can turn the filament on, using the window of options in the lower left. Check through the view ports to see the filament. When the filament first ignites, the pressure in the chamber will increase, caused by desorption of molecules from the filament itself. In the first tab of the main window in the program, select to measure masses of 1 - 50 au with 10 seconds per au. Start measuring the spectra within the chamber. After 15 cycles have been completed, go to *File -> Export* and export your data. The resulting file will have data of atomic weight against intensity where the intensity is caused by a current with in the QMS. After you have exported your data, make sure to turn off the filament and disconnect the software from the QMS. Identify the peaks in your spectrum and label them according the elemental weight.

## 5.5 Baking Out a Chamber

Having assessed the quality of our vacuum, we will now do a process known as baking out, a process by which we will heat the chamber in a controlled manner for a given period of time.

Turn off the main water supply at the wall piping by turning the two red levers, Figure 3a. Disconnect the water lines supplying cooling water to the cathodes, there is a bucket in the sink to the left of the door to catch the excess water from the tubing, Figure 3b. Switch off the power supply for the cathode, Figure 3c. Check the pressure is below at least  $5 \times 10^{-6}$  mbar and record the current pressure in your log book. Remove the heat sensitive components from the chamber as they will melt during this process. These components include the QMS, loosen the collar with a hexagonal key and lift it up carefully. The wires to remove are labelled. Ask the student advisor to remove the HV cable if it is attached and to check that all components

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have been removed.



(a)



(b)



(c)

**Figure 3:** a) Main water lines(open) to supply cooling water to the cathodes during sputtering. They should be opened prior to sputtering. The two lines(Inflow and return lines) should be closed before baking out and once sputtering has stopped. This can be done by turning each lever  $90^\circ$ . b) Water lines for the cathodes, copper on the top, Py below. With the knob vertical, the flow is stopped, rotate the knob  $90^\circ$  to start the water. There are two valves for each cathode on the left and right of the rack. c) High voltage DC power supply for the cathode.

Wrap the first heat belt around the top flange of the chamber, ensuring that the seal of the flange is at the centre of the belt. **Note: It is advised to wear gloves while handling the heating belts.** Wrap the second around the target cathodes under the chamber and as many flanges as possible. Make sure that all the windows are covered with aluminium foil as well as the gaps in the chamber to ensure uniform heat distribution and to prevent damage to the windows. Connect the power of the heating belts to the central heat control unit. Insert the heat probes within the folds of the heating belts, using aluminium foil to keep them in place, making sure that the probe for each power plug is inserted into the correct belt. Cover the chamber in blankets (if being used), making sure the **pressure gauges** and **turbo pump** are outside the blanket to protect them from the heat. Use clips to close any gaps in the blankets, including making sure a blanket goes under the chamber. Switch to the front panel on the sourcemeter to record the temperature (shown in volts) of the chamber during the bake-out. Set the first to a temperature just above room temperature and see if the belt turns on to check that the correct probe is wrapped in the correct belt. If both probes are in the correct belt, set the desired baking temperature to  $130^\circ\text{C}$ . Connect the control unit to the wall socket on the instrument rack via a programmable power control switch to set the desired baking time. Set the baking to end at 01:00 of the morning you wish to continue the second day of the Praktikum as the chamber needs to cool down.

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Start measuring the pressure of the chamber and start the bake out process, naming your save file with the format "YYYYMMDD\_YourNames\_BakeOutPressure". Once started, do not readjust the heating belts, they will become hot very quickly. The pressure will initially rise due to the desorption of water from the chamber walls. After this, the pressure will begin to decrease.

***WARNING:** Do not touch the chamber while baking. The walls and the periphery will become very hot. Ensure people in the proximity of the chamber are aware of the baking and put up warning signs.*

After baking, wait for the chamber to cool down to workable temperatures. Reduce the temperature knob on the control unit to a minimum, disconnect the power supply and remove the aluminium foil. Unwrap the heating belts and reconnect the electronics that were disconnected previously.



(a)



(b)

**Figure 4:** Photos of the top flange a) Unwrapped and b) Wrapped in the heating belts

This is the end of the first day. Please find below some questions to answer about what has been covered today.

## 6 Questions on the First Day

- What causes the limit on the pressure one can reach with the pre-pump alone?
- Calculate the mean free path of a nitrogen ( $N_2$ ) molecule in atmospheric pressure and at  $1 \times 10^{-9}$  mbar
- Based on the data from section 5.3, how much gas is desorbed from the walls after the turbo reaches full speed? *Hint: Consider the data over the time period after the turbo reaches speed. This is the pressure drop in a fixed time. The volume of the chamber is 30 litres and you can assume the chamber is at a constant temperature of 293K. The mass of air is approximately 29g/mol.*
- What is the purpose of baking out a chamber?
- What is the thermal energy at room temperature and the corresponding thermal speed of a water molecule ( $H_2O$ )?

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- What is the thermal energy at 130°C and the corresponding thermal speed of a water molecule ( $H_2O$ ) and a carbon dioxide ( $CO_2$ ) molecule? Why do they have different speeds?
  - What can you say about the mass of molecules that require higher baking temperatures? What factor influences whether a molecule will desorb?
  - If the desorption energy of a molecule with molecular weight 20 au is 54.7 meV, what baking temperature is required to draw the molecule from the walls? *Assume that there are 3 degrees of freedom.*
  - Calculate the conductance of the two tubes as well as the pumping speed of the pre-pump. For this, use equation 17 to fit to the linear part of the pump down, as measured by the high pressure gauge. Then we can relate the effective pumping speed,  $S_{eff}$ , to the true pumping speed and the conductance of the tubing. Using equations 4.28 and 4.34 from the textbook scans and sticking to the linear part of the  $\log(p)$  vs time plot, we can approximate a quasi-static solution. Having three measured values of  $S_{eff}$  for each tube combination, we can rearrange to obtain three equations where we have the unknowns of the true pumping speed and conductances of the two tubings, as well as the conductance of the combination. In this approximation, we are also assuming that the turbulence caused by the connections has minimal impact although in reality this is a false assumption.

## 7 Day 2

On this day, we will analyse how the baking out has helped our chamber before we grow a thin film on the substrate loaded yesterday.

### 7.1 Vacuum Comparison

Having baked out the chamber, make a note of the base pressure of the chamber. How does it compare to the pressure before baking? Why do you think this is?

Following the instructions of section 5.4, take another spectrum using the QMS. How have the elemental peaks changed from the spectra of the first day? What can you say about the baking process? How will the baking process affect the quality of the samples being grown?

### 7.2 Film Growth

#### 7.2.1 Pre-sputtering

Before continuing, make sure that the filament for the QMS is turned off so that the increase in pressure in this section doesn't break the spectrometer. Switch the voltmeter back to the rear-input to measure pressure from the high range gauge.

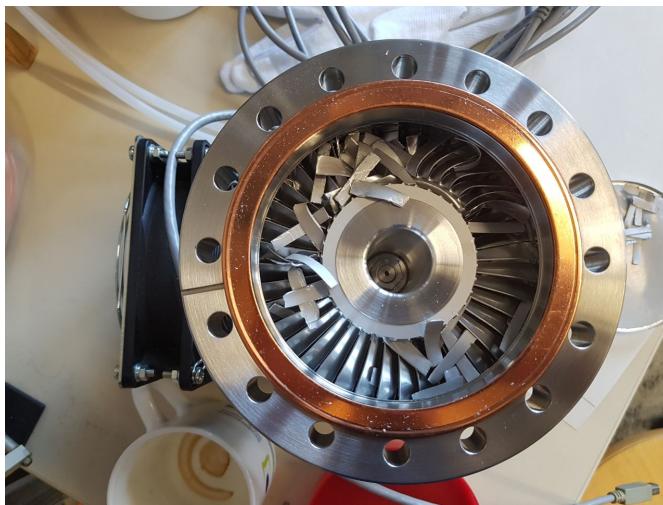
The opening of the chamber to load the sample introduced impurities to the atmosphere within the chamber. We have reduced the level of the impurities by baking the chamber but we also need to clean the target as the first few atomic layers will have oxidised. In order to achieve the correct given stoichiometry of a target, it is crucial to ignite the target and remove these layers.

Ask the student assistant to connect the HV cable. Do not try to do this alone.

Open the cooling water valves on the side of the chamber and the main supply on the wall. (Figure 3a and Figure 3b).

---

Rotate the sample stage using the rotator such that your substrate is away from the target we will be using. This is done to avoid any possible deposition on the surface of the sample. Using the turbo controller program, reduce the turbo speed to 50% by clicking the reduce speed icon, this should take around 10 minutes to complete. This is done to ensure there is no excessive load on the turbo during sputtering. Figure 5 shows a turbo pump from our group that has broken due to a high pressure (*Note that the TMP power will initially increase and then the rpm will gradually decrease.*)



**Figure 5:** A turbo pump with broken blades.

Turn on the power supply in the main rack, Figure 3c, and press & hold the “*Set Values*” button to choose a starting voltage and current (12.1 mA), release the button to set these values. We now need to introduce an Ar atmosphere. Open valve 1 on the Ar bottle - this releases the Ar gas from the bottle to the gas regulator. Open valve 2 to release the gas into the stainless steel piping. Do not touch the center valve on the regulator. Use the stainless steel needle valve to introduce the Ar to the chamber. Keep an eye on the pressure of the chamber as it raises to ensure the pressure doesn’t increase too quickly. You will need to regulate this until a stable pressure is reached at  $3 \times 10^{-2}$  mbar. Switch on the DC power supply and observe the plasma burning above the target. Pre-sputter the target for 5 minutes then turn off the DC power supply. Keep the pressure in the chamber below  $1 \times 10^{-1}$  mbar to reduce the strain on the turbo pump.

### 7.2.2 Power Variations

With the current off, the plasma should not be burning. By changing the current in steps from 1 mA to 30 mA, make notes of the voltage being output to create an I-V curve for the target. Don’t be concerned that the plasma doesn’t ignite at low currents, but sparks, this is normal. For these low currents, the voltage should be unstable and you should make a note of the different regimes for the plasma (stable/unstable).

Set the current to 12.1 mA and turn on the power supply. Check the pressure is still stable at  $3 \times 10^{-2}$  mbar and the plasma is burning & stable.

### 7.2.3 Sample Deposition

With a stable burning plasma under way, rotate the sample into the plasma for 15 minutes. After the 15 minutes is up, rotate your sample out of the plasma and turn off the DC power supply. Close the needle



(a)



(b)

**Figure 6:** a) Argon bottle with valves 1 and 2. The flow regulator (black) should not be touched. b) Needle valve to introduce Ar to the chamber and finely regulate the gas pressure inside the chamber during sputtering. The valve must be used with caution and should **not** be tightened with excessive force. Only finger tight. Excessive force can damage the needle valve. Gently turn the knob anticlockwise to begin Ar gas flow.

valve to shut off the gas flow to the chamber. This only needs to be finger tight, excessive force will damage the valve. Close valve 1 and 2 on the Ar gas bottle and watch the pressure drop to  $1 \times 10^{-7}$  mbar.

Follow the instructions in section 5.1.1 to vent the chamber. Undo the bolts on the main window as you did when loading the sample and wait for the chamber to vent. Wearing gloves, take out your sample, remove it from the omicron holder and place it in a sample box, labelling the sample. Now pump the chamber, as when you loaded the sample. Wait for the pre-pump to lower the pressure before starting the turbo. This is the end of the second day.

## 8 Questions on the Second Day

- Can you name some properties which might arise due to scaling down the thickness of a sample?
- How have the elemental peaks changed from the spectra of the first day? What can you say about the baking process? How will the baking process affect the quality of the samples being grown?
- Why does the pressure oscillate with clear periods of increasing and decreasing pressures during baking?
- What happens to the plasma as you increase the power being applied to the cathode?

- 
- Why do we pre-sputter the target before deposition?
  - Plot the Polanyi-Wigner equation as a function of desorption energy for an ensemble of  $5.35 \times 10^{13}$  particles for  $T_w = 300K, 500K, 800K, 1000K$  for  $E_{des}$  between 0 and 60 kJ/mol-1 (universal gas constant 8.31 J/mol-1). Scale the y-axis to  $10^{13}$  as a maximum.

This concludes the F-Praktikum on Vacuum Science. Please hand in your answers to the questions as well as labelled spectra from before and after baking and plots of the pressure for different tubings.

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## A Background

This section will cover basic background on the pumps and pressure gauges used in this experiment. The experimental technique of sputtering is also briefly explained. It is advised that the student reads about these techniques to familiarise themselves. Listed chapters are taken from *Handbook of Vacuum Technology*, Second Edition, Karl Jousten, Wiley-VCH, 2017.

### A.1 Gas Flow

Understanding the way a gas flows is critical in vacuum technology. For this reason, the following chapters from *Handbook of Vacuum Technology* (Second Edition), edited by Karl Jousten are of interest,

- 4.1
- 4.2.1, 4.2.5
- 4.3.1, 4.3.2, 4.3.4
- 4.4.1, 4.4.3, 4.4.4, 4.4.7, 4.4.8
- 4.5.3
- 4.7.1, 4.7.2, 4.7.3

although the main points of gas flow will be summarised here and in the following photocopied pages (pages 83-95, 114-119 and 145-148) from *Handbook of Vacuum Technology* (Second Edition), edited by Karl Jousten, Wiley-VCH, 2016 (reprinted 2017), ISBN: 978-3-527-41338-6.

There exist different types of flow regimes which are studied in hydrodynamics which apply here to the rate of flow and the mechanisms of the flow gases both within the chamber vessel being pumped as well as the vacuum tubing connecting the pumps and the chamber. These flow regimes are broadly classified into the following categories based on the ratio of the mean free path to the flow channel diameter. This ratio is known as the Knudsen number which is a dimensionless quantity. The movement of gas molecules in a chamber/ vacuum piping occurs in three defined flow regimes outlined below. Depending on the pressure and the conductivity (diameter dependent) of the vacuum tube, how the gas flows can vary significantly.

#### A.1.1 Laminar flow

The flow of gas molecules in this regime are classified as streamlined motion. The flow is laminar in nature, and can be visualised as layers of concentric cylinders such that laminar columns at the edge of the gas conducting pipe have the highest density and therefore moves slowest with gradually increasing speeds of movement towards the centre of the pipe. This kind of flow is dominated by gas-gas collisions. Such a regime is also referred to as continuum flow. In this regime the characteristic properties of the gas do not vary over several mean free path lengths such as temperature and density. Such a flow regime is experienced when the mean free path of the gas molecules is significantly shorter than the dimensions of the flow channel

#### A.1.2 Molecular flow

This flow regime is dominated by collisions between gas molecules and the chamber walls. In this regime gas molecules which hit the chamber wall are diffusely reflected, i.e. the direction of reflection of the gas molecules is independent of the incident angle. There are also no gas molecule collisions with each other.

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### A.1.3 Turbulent Flow

There exists a third kind of gas flow which is a transitional regime between the two above in which the gas movement is dominated by neither gas-gas nor gas-chamber collisions. Following this for much higher Knudsen numbers the regime experienced is the turbulent flow regime in which there are continuous collisions among different gas molecules.

There is a change in the flow regime from the laminar flow to the turbulent when the velocity of the gas molecules increases the flow of gas molecules in adjoining layers are no longer parallel but break up into a disordered pattern. The region between the laminar flow and the turbulent flow i.e. the transition regime can be characterised by Reynolds number given by,

$$\text{Reynolds number} = \text{density of the gas} \times \text{mean velocity of the molecules} \times \frac{\text{characteristic length}}{\text{dynamic viscosity}} \quad (16)$$

- 5 Blanke, W. (ed.) (1989) *Thermophysikalische Stoffgrößen*, Springer, Berlin.
- 6 Lide, D.R. (1990) *CRC Handbook of Chemistry and Physics*, 71th edn, CRC Press, Boca Raton, FL.
- 7 Jäschin, W. (1999) *Vakuum-Lexikon*, Wiley-VCH Verlag GmbH, Weinheim.
- 8 Reid, R.C., Prausnitz, J.M., and Poling, B.E. (1987) *The Properties of Gases and Liquids*, McGraw-Hill, New York.
- 9 Mattland, G.C., Raby, M., Smith, E.B., and Wakeham, W.A. (1981) *Intermolecular Forces*, Clarendon Press, Oxford.

**Comprehensive general treatments of the subject**

- Edelmann, C. (1998) *Vakuumphysik*, Spektrum Akademischer Verlag, Heidelberg.
- Lafferty, J.M. (ed.) (1998) *Foundations of Vacuum Science and Technology*, John Wiley & Sons, Inc., New York.

## 4 Gas Flow

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This chapter covers various types of gas flows, each developing in specific geometry and in characteristic pressure range.

### 4.1 Types of Flows and Definitions

#### 4.1.1 Characterizing Flow, Knudsen Number, and Reynolds Number

Gas flow patterns play an important role in vacuum technology. When a vessel is evacuated, the gas that initially filled the vessel flows to the pump through tubes. During operation of the vessel, gas released by components (desorption) or supplied to the process flows from high-pressure to low-pressure regions. Knowledge of flow patterns is vital for designing vacuum systems intelligently and understanding their performance characteristics.

Flow (or flux) is a three-dimensional movement of substance. In a gas, the thermal motion of individual gas particles, as well as macroscopic forces due to local pressure deviations, causes flow. Pressure forces, inertial forces, and frictional forces determine flow behavior. Gravity, however, is usually negligible for gas flow. Usually, the total gas flow through a tube is of interest, but, in certain cases, knowledge of local flow densities in an apparatus is required.

Depending on the prevailing conditions, different types of flows arise. In order to understand flow patterns, it is favorable to consider the different types of flows individually in their pure form. Figure 4.1 shows the types of flows that occur in a tube of arbitrary length.

Depending on pressure and the cross dimensions of a tube, three types of flows can be differentiated:

- 1) For sufficiently low pressure, the mean free path of gas particles is high, compared with the cross dimensions of the tube. Any mutual particle collision

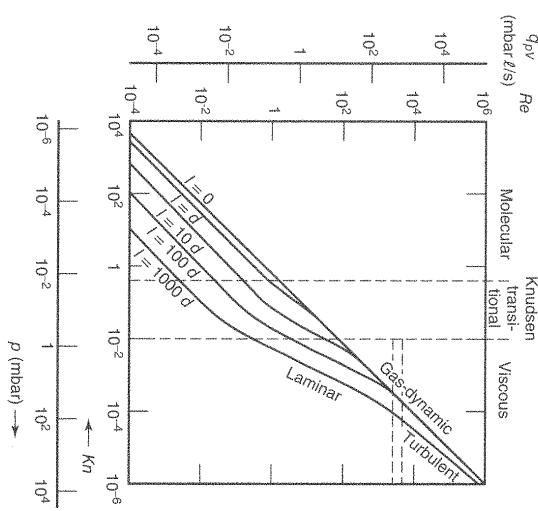


Figure 4.1 Flow types in tubes with circular cross section; diameter  $d = 1$  cm, and length  $l$  as indicated taken as an example. The gas is air at  $20^\circ\text{C}$ . Inlet pressure is taken as abscissa and the outlet pressure is assumed negligible.

hardly occurs. Each gas particle travels through the tube due to its thermal motion, independent of other particles. However, frequent collisions with the tube walls cause a zigzag route. On average, the paths of many individual particles combine to form the macroscopic flow behavior. This situation is referred to as single-particle motion or *molecular flow*.

2) Under high pressure, the mean free path of gas particles is much lower than the cross dimensions of the tube. The particles experience frequent mutual collisions, thereby exchanging momentum and energy continuously. Even a small volume contains many frequently colliding particles. Thus, the gas behaves as a continuum. A flow is the result of local pressure gradients. This situation is referred to as continuum flow or *viscous flow*.

3) The medium-pressure range is characterized by a transition between molecular and viscous flows. In this transition, collisions of gas particles with the wall occur just about as often as mutual collisions among gas particles. This situation is referred to as *transitional flow* or *Knudsen flow*.

Thus, for a particular type of flow to occur, two main criteria can be identified: one criterion is the mean free path of gas particles in relation to the cross dimensions of the tube (for circular cross sections, the diameter). The second

criterion is the velocity of flow for given cross dimensions of the tube and internal friction of the gas. Thus, two dimensionless characteristic numbers may be defined to describe these criteria quantitatively.

The *Knudsen number*  $Kn$  is the ratio of the mean free path  $\bar{l}$  of the gas particles between two particle-particle collisions and the characteristic geometrical dimension  $d$  of the tube's cross section (for circular tube cross sections, the diameter):

$$Kn = \frac{\bar{l}}{d}. \quad (4.1)$$

As shown in Chapter 3, the mean free path can be obtained from viscosity  $\eta$  (Eq. (3.72)). Thus, for practical reasons, Eq. (4.1) can be rewritten as

$$Kn = \frac{\pi}{4} \cdot \frac{\bar{v}\eta}{pd}, \quad (4.2)$$

denoting that the *Knudsen number* is inversely proportional to the pressure. A high *Knudsen number* indicates low pressure, and thus molecular flow, whereas a low value of the *Knudsen number* suggests viscous flow. Transition between the two types of flows is smooth and leads to a change in gas flow through the tube. The limiting cases of molecular or viscous flow are approximately reached when roughly 90% of this change in flow has established. The quantitative investigations described below show that this assumption leads to the following conditions:

$$\begin{aligned} Kn > 0.5, & \quad \text{molecular flow,} \\ 0.5 > Kn > 0.01, & \quad \text{transitional flow,} \\ Kn < 0.01, & \quad \text{viscous flow.} \end{aligned} \quad (4.3)$$

We will now investigate the second criterion for the type of flow: the velocity of flow. The velocity  $v$  of a gas flow is the mean velocity component of the gas particles in the direction of the tube. Usually, the velocity's mean value is given as an average across the tube's cross section.

In the case of molecular flow, the individual gas particles travel back and forth between the walls of the tube with thermal velocity. A particle's direction after hitting the wall is (nearly) independent of its direction prior to the collision. Thus, a zigzag route develops (Figure 4.2a). The geometry of the tube determines the resulting velocity of flow.

The situation is different in the case of viscous flow. Here, three types of flows in a tube are differentiated. The length of the tube determines the type of flow (Figure 4.2b–d).

- 1) Initially, the gas has to leave a reservoir (vessel) to reach the entrance of the tube. Subsequently, it streams into the tube (Figure 4.2b). Here, the gas accelerates from a quiescent state (velocity of flow equals zero) to a finite velocity of flow. This process requires acceleration energy that is taken from pressure energy (pressure drops) and thermal energy (temperature drops).

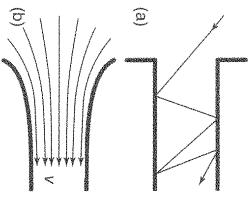


Figure 4.2 Different types of gas flows. (a) Molecular flow. (b-d) Different types of viscous flows: gas-dynamic (intake flow), laminar, and turbulent.

Thus, as a volume element of gas travels along a path, velocity rises, and simultaneously temperature and pressure drop. For short distances, wall friction is usually negligible. This so-called *intake flow* is a particular type of *gas-dynamic flow*.

- 2) Now, the gas flows through the tube. The velocity of flow at the inlet is approximately constant across the complete cross section. As the gas continues its way through the tube, the gas layers near the walls decelerate, and the velocity of flow drops to zero in the boundary layer at the wall. The thickness of the boundary layer increases along the way. The velocity of flow, the friction behavior of the gas, and the dimensions of the cross section determine the type of flow that develops after a certain intake stretch. For low velocities, all individual volume elements move in the direction of the tube. Now, the volume elements in the center of the tube move quicker than the volume elements at the boundary of the tube. Thus, a velocity profile develops across the cross section of the tube (Figure 4.2c). This type of flow is referred to as *laminar flow*.
- 3) If, however, flow velocity is high, frictional forces are high as well because they are determined by flow velocity. A volume element, traveling at higher velocity and some distance from the wall of the tube, is deflected toward the wall by the decelerating action of the slower moving layers near the wall. The deflecting effects increase with friction and thus velocity, whereas the inertia

of mass, which tends to preserve the direction of flow, remains unchanged by a change in velocity. Thus, for sufficiently high velocities, deflecting forces dominate and the flow shows turbulences and eddies (Figure 4.2d). The criterion for turbulences to develop is the ratio of frictional force (proportional to gas viscosity  $\eta$ ) and inertia of mass (proportional to gas density  $\rho$ ) for a specified velocity of flow  $v$  (cross-section average) and specified cross section. Typically, the *Reynolds number*  $Re$  is used to describe this criterion:

$$Re := \frac{\rho}{\eta} v d. \quad (4.4)$$

The quantity  $d$  characterizes the cross section of the tube. For a downpipe, this corresponds to the diameter  $d$ .

$$\begin{aligned} Re < 2300, & \text{ laminar flow,} \\ Re > 4000, & \text{ turbulent flow.} \end{aligned} \quad (4.5)$$

#### 4.1.2 Gas Flow, Throughput, and Pumping Speed

The flow rate  $q$  of a gas flowing through a duct is defined as transported gas per time. Several approaches of describing the amount of gas yield several different types of flow rates:

$$\text{Volume flow rate : } q_V = \frac{\Delta V}{\Delta t} = \dot{V}, \quad [q_V] = \text{m}^3 \text{ s}^{-1}. \quad (4.6)$$

$$\text{Mass flow rate : } q_m = \frac{\Delta m}{\Delta t} = \dot{m}, \quad [q_m] = \text{kg s}^{-1}. \quad (4.7)$$

$$\text{Molar flow rate : } q_v = \frac{\Delta \nu}{\Delta t} = \dot{i}, \quad [q_v] = \text{mol s}^{-1}. \quad (4.8)$$

$$\text{Particle flow rate : } q_N = \frac{\Delta N}{\Delta t} = \dot{N}, \quad [q_N] = \text{s}^{-1}. \quad (4.9)$$

At times, the terms *rate of flow* and *flux rate* are used synonymously when referring to *flow rate*. It should be considered that the flow rate can change along the length of the tube. For example, the volume flow rate at the end of a tube is higher than that at the beginning because the pressure drops along the tube and, correspondingly, the volume of the gas increases.

Furthermore, the concept of *pV flow* or *throughput* is used frequently:

$$pV_{\text{flow}} : \quad q_{pV} = p \dot{V}, \quad [q_{pV}] = \text{Pam}^3 \text{ s}^{-1} = 10 \text{ mbar} \cdot \text{m}^3 \text{ s}^{-1}. \quad (4.10)$$

Using the equation of state of an ideal gas (Eqs. (3.18)–(3.20)),

$$pV = NkT = mR_s T = \nu RT, \quad (4.11)$$

and the definition of the mean particle speed (Eq. (3.43)),

$$\bar{v} = \sqrt{\frac{8}{\pi} \frac{kT}{m_p}} = \sqrt{\frac{8}{\pi} R_s T} = \sqrt{\frac{8}{\pi} \frac{p}{\rho}} \quad (4.12)$$

the different types of flows can be converted:

$$q_V = \frac{q_{pV}}{p}, \quad (4.13)$$

$$q_m = \frac{q_{pV}}{R_s T} = \frac{8}{\pi} \frac{q_{pV}}{\bar{v}^2} = M q_{pV}, \quad (4.14)$$

$$q_{pV} = \frac{q_V}{RT}, \quad (4.15)$$

$$q_N = \frac{q_{pV}}{kT}. \quad (4.16)$$

Table 4.1 lists a number of common units for gas flow.

Table 4.1 Conversion of selected common units for gas flow.

| Unit                            | Conversion                  | Definition                                       |
|---------------------------------|-----------------------------|--|
| $\text{Pa m}^3 \text{s}^{-1}$   | $= 1$                       | $pV$ flow of 1 $\text{Pa m}^3 \text{s}^{-1}$     |
| $\text{mbar} \text{ l s}^{-1}$  | $= 0.1$                     | $pV$ flow of 1 $\text{mbar l s}^{-1}$            |
| Torr $\text{l s}^{-1}$          | $= 0.133322$                | $pV$ flow of 1 Torr $\text{l s}^{-1}$            |
| $\text{atm cm}^3 \text{s}^{-1}$ | $= 101325$                  | $pV$ flow of 1 $\text{atm cm}^3 \text{s}^{-1}$   |
| Jusec                           | $= 0.000133322$             | $pV$ flow of 1 $\mu\text{mHg s}^{-1}$            |
| sccm                            | $\Leftrightarrow 0.0018124$ | Flow of standard $\text{cm}^3 \text{min}^{-1}$   |
| slm                             | $\Leftrightarrow 1.8124$    | Flow of standard $\text{l min}^{-1} = 10^3$ sccm |
|                                 | $\Leftrightarrow 2437.4$    | Molar flow per second                            |
|                                 |                             | $\text{Pa m}^3 \text{s}^{-1}$ at 20 °C           |
|                                 |                             | Molar flow per second                            |

The prefix "standard" refers to gas volume under standard conditions (101325 Pa, 0 °C).

#### Example 4.1

Flowmeters often measure in "sccm" (standard cubic centimeters per minute). 1 sccm means a gas flow of  $1 \text{ cm}^3 \text{ min}^{-1}$ , referring to standard conditions ( $p_0 = 101325 \text{ Pa}$ ,  $\theta_0 = 0^\circ \text{C}$ ). Conversion to  $pV$  flow at 20 °C is as follows:

$$1 \text{ sccm} \Leftrightarrow \frac{101325 \text{ Pa} \times 1 \text{ cm}^3 \text{ min}^{-1} \times 293.15 \text{ K}}{273.15 \text{ K}} = 1.8124 \times 10^{-3} \text{ Pa m}^3 \text{ s}^{-1} = 1.8124 \times 10^{-2} \text{ mbar l s}^{-1} \text{ at } 20^\circ \text{C.} \quad (4.17)$$

#### Example 4.2

The permissible leakage of an air conditioner is 3 g per year. What is the  $pV$  value (at 20 °C) for the leakage? Tetrafluoroethylene R134a ( $\text{CH}_2\text{F-CF}_3$ ), with a total mass number of 102, is the refrigerating medium. With Eq. (4.14), it follows that

$$q_{pV} = q_m \frac{R}{M} = \frac{0.003 \text{ kg}}{3.156 \times 10^{-5}} \times \frac{8.314 \text{ J mol}^{-1} \text{ K}^{-1}}{0.102 \text{ kg mol}^{-1}} \times 293 \text{ K} \\ = 2.27 \times 10^{-6} \text{ Pa m}^3 \text{ s}^{-1} = 2.27 \times 10^{-5} \text{ mbar l s}^{-1}. \quad (4.18)$$

When a vacuum vessel is evacuated by a vacuum pump, the gas volume flowing through the pump inlet per unit time (volume flow rate at the pump inlet) is the *pumping speed*  $S$  of the pump:

$$S := \dot{V}_{\text{inlet}} = q_{V,\text{inlet}}, \quad [S] = \text{m}^3 \text{ s}^{-1} = 1000 \text{ l s}^{-1} = 3600 \text{ m}^3 \text{ h}^{-1}. \quad (4.19)$$

The  $pV$  flow at the inlet of the pump is referred to as *throughput*  $\dot{Q}$  of the pump:

$$\dot{Q} = q_{pV,\text{inlet}}, \quad [\dot{Q}] = \text{Pa m}^3 \text{ s}^{-1} = \text{W} = 10 \text{ mbar l s}^{-1}. \quad (4.20)$$

The previous two equations indicate that pumping speed and throughput of a vacuum pump are related according to ( $p$  is the pressure at the inlet)

$$\dot{Q} = q_{pV,\text{inlet}} = pS \quad (4.21)$$

For many vacuum pumps, pumping speed  $S$  is (nearly) pressure independent. Then, throughput  $\dot{Q}$  is proportional to pressure as indicated in Eq. (4.21). Especially, throughput is low at low pressure. This is a comprehensible behavior because a volume element at low pressure, contains less gas particles (and thus less mass). Figure 4.3 shows the pressure-independent pumping speed  $S$  (top) and the pressure-dependent throughput  $\dot{Q}$  (bottom) of a pump as a function of inlet pressure.

#### Example 4.3

A mechanical displacement pump has a pumping speed  $S = 360 \text{ m}^3 \text{ h}^{-1} = 100 \text{ l s}^{-1}$  and pumps air with a temperature of 20 °C at an inlet pressure  $p = 10 \text{ mbar}$ . This calculates to

$$pV \text{ flow: } q_{pV} = pS = 10 \text{ mbar} \times 100 \text{ l s}^{-1} = 1000 \text{ mbar l s}^{-1}. \quad (4.22)$$

$$\text{Throughput: } \dot{Q} = q_{pV} = 1000 \text{ mbar l s}^{-1} = 100 \text{ Pa m}^3 \text{ s}^{-1} = 100 \text{ W}. \quad (4.23)$$

$$\text{Mass flow rate: } q_m = \frac{q_{pV}}{R_s T} = \frac{q_{pV} M}{RT} \\ = \frac{100 \text{ W} \times 0.029 \text{ kg mol}^{-1}}{8.3 \text{ J mol}^{-1} \text{ K}^{-1} \times 293 \text{ K}} = 0.0012 \text{ kg s}^{-1}. \quad (4.24)$$

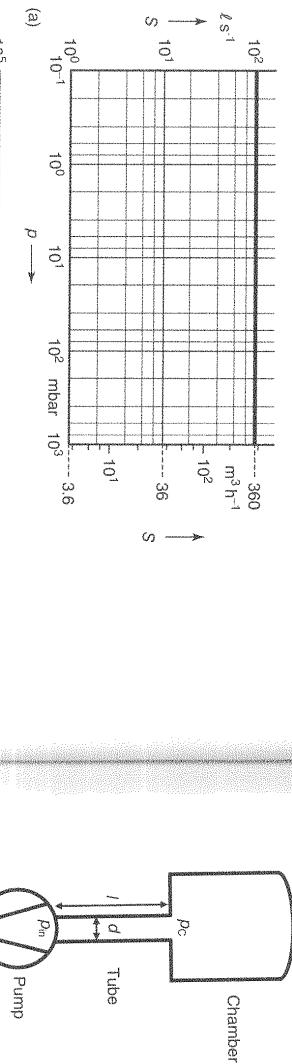


Figure 4.4 Vacuum system with pump line between vacuum chamber (pressure  $p_c$ ) and pump (pressure  $p_{in}$  at the pump inlet).

Section 18.2.2). As an analogy to the ohmic resistance of an electrical component, the flow resistance  $R$  and conductance  $C$  of a tube are defined as

$$R := \frac{\text{pressure difference}}{\text{flux}} = \frac{\Delta p}{q}, \quad (4.26)$$

$$C := \frac{1}{R} = \frac{q}{\Delta p}. \quad (4.27)$$

Depending on the unit used for the flow, the quantities flow resistance and conductance are obtained with the corresponding units. Typically,  $pV$  flow is used, which leads to  $[R] = \text{s m}^{-3}$  or  $\text{s} \cdot \text{l}^{-1}$  and  $[C] = \text{m}^3 \text{s}^{-1}$  or  $\text{l} \cdot \text{s}^{-1}$ . If the particle flow rate is given, then  $[R] = \text{Pa s}$  and  $[C] = \text{Pa}^{-1} \text{s}^{-1}$ . Unless otherwise stated,  $pV$  flow will be used here.

For *electrical* conductors connected in series and parallel, the individual resistances and conductances add up to the total resistance  $R$  and total conductance  $C$ , respectively:

Series connection :

$$R = R_1 + R_2 + R_3 + \dots \quad \text{and} \quad \frac{1}{C} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3} + \dots \quad (4.28)$$

Parallel connection :

$$C = C_1 + C_2 + C_3 + \dots \quad \text{and} \quad \frac{1}{R} = \frac{1}{R_1} + \frac{1}{R_2} + \frac{1}{R_3} + \dots \quad (4.29)$$

The validity of Eqs. (4.28) and (4.29) for gases is quite limited: an inlet flow develops at the inlet of a two-tube-series connection or in regions where a change occurs in the cross section. An additional flow resistance accompanies this type of flow. Thus, the overall assembly determines the flow resistance of a tube section. Flow resistance is higher if the tube is mounted

#### 4.1.3 Flow Resistance and Flow Conductance

The terms flow resistance and flow conductance are exemplified by considering a tube, connecting a vacuum chamber with a vacuum pump, as shown in Figure 4.4.

During evacuation, gas flows from the chamber, through the tube, and to the pump. This requires the pressure difference

$$\Delta p = p_{\text{chamber}} - p_{\text{pump inlet}} = p_c - p_{\text{in}}. \quad (4.25)$$

The behavior of a gas flow  $q$  is analogous to that of an electrical current, and the pressure of a gas corresponds to an electrical potential (see also

directly at the vessel, as opposed to being mounted in a position beyond an additional tube. Series connections of components will be discussed in detail for molecular flow (Sections 4.4.7 and 4.4.8). Equation (4.29) is only applicable for parallel connections if the tube inlets are separated far enough, so that the inlet flows do not interfere.

Practical calculations of multicomponent tube assemblies subdivide the systems into individual segments, according to geometrical dimensions and types of flow. Nonstationary gas flow can be treated analogously to an electrical current as well; the tube volume corresponds to the capacity of a capacitor in the same way as the inertia of the flowing gas mass corresponds to the inductance of a coil.

#### 4.1.4 Effective Pumping Speed of a Vacuum Pump

We will consider a vacuum pump, attached to a vessel via a long tube (Figure 4.4), and will investigate the influence of the connecting tube on the pumping action.

For the (quasi-)stationary case, gas flow (given as, e.g., particle flow) is constant; that is, particle flows at the inlet and outlet of the pump are the same. The temperature of the gas can change due to the flow in the connecting tube (expansion), and due to the subsequent pumping process (compression). However, due to heat exchange with the environment, the change in temperature remains moderate. As an approximation, gas temperature is assumed to be constant, and thus,  $pV$  flow  $q_{pV}$  remains constant as well.

At the inlet of the connecting tube (chamber flange CF), the pressure is  $p_c$ , and at the outlet of the tube (inlet flange PF of the pump), the pressure is  $p_{in}$ . Assuming constant  $pV$  flow, it follows that

$$q_{pV} = p_c \dot{V}_c = p_{in} \dot{V}_{in}. \quad (4.30)$$

The volume flow rate at the inlet flange of the pump is just the pumping speed of the pump; thus,  $\dot{V}_{in} = S$ . The volume flow rate at the vessel flange is the pumping speed available for evacuating the vessel; thus,  $\dot{V}_c = S_{eff}$  and is referred to as effective pumping speed (net pumping speed). Using these quantities, Eq. (4.30) can be rewritten as

$$S_{eff} = \frac{p_{in}}{p_c} S < S. \quad (4.31)$$

The effective pumping speed  $S_{eff}$  is lower than the pumping speed  $S$  because  $p_c > p_{in}$  in order to maintain flow. However, due to the condition of continuity, the throughputs are the same at the inlet and the outlet of the tube.

By rewriting Eq. (4.27), the conductance  $C$  of the tube

$$C = \frac{q_{pV}}{p_c - p_{in}} = \frac{p_{in}S}{p_c - p_{in}} = \frac{p_c S_{eff}}{p_c - p_{in}} \quad (4.32)$$

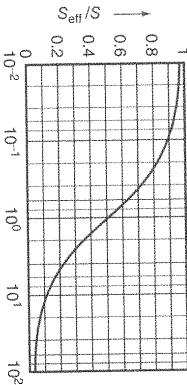


Figure 4.5 Dependence of pump efficiency  $S_{eff}/S$  on the ratio of pumping speed  $S$  of the pump and the conductance  $C$  of the tube.

yields the pressure ratio  $p_c/p_{in}$ :

$$\frac{p_c}{p_{in}} = 1 + \frac{S}{C}. \quad (4.33)$$

The series connection of pump (pumping speed  $S$ ) and tube (conductance  $C$ ) determines the effective pumping speed  $S_{eff}$  available at the vessel:

$$\frac{1}{S_{eff}} = \frac{1}{S} + \frac{1}{C} \quad \text{and} \quad S_{eff} = \frac{S}{1 + S/C}. \quad (4.34)$$

Figure 4.5 shows a plot of Eq. (4.34). Obviously, a pumping efficiency  $S_{eff}/S = 0.9 = 90\%$  requires at least a 10-fold line conductance, compared with the pumping speed of the pump. If the conductance is just equal to the pumping speed, the effective pumping speed is only 50% of the pump's pumping speed. If the conductance is considerably below the pumping speed, the effective pumping speed is determined largely by the conductance and hardly by the pumping speed of the pump. Thus, any larger pump would not increase the effective pumping speed significantly. Consequentially, when installations are planned, tubes with maximum possible conductance should be selected (short tubes with large cross sections).

## 4.2 Inviscid Viscous Flow and Gas Dynamics

### 4.2.1 Conservation Laws

We will now consider viscous flow of gas through a tube. To simplify, flow shall be stationary (constant in time). In the stationary case, mass flow, that is, the mass flowing through a cross section of the tube, remains constant along the

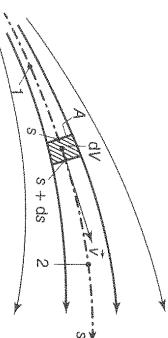


Figure 4.6 Flow field with flow filament.

line. Thus, the product of cross-sectional area  $A$ , density  $\rho$ , and velocity  $v$  of gas flow remains constant as well:

$$\dot{m} = \frac{dm}{dt} = \rho v A = \text{constant} \quad ; \quad \text{conservation of mass.} \quad (4.35)$$

For a short tube, interactions of the gas with the walls are often negligible; that is, momentum exchange due to friction as well as energy transfer due to heat exchange does not occur. Assuming this, additional conservation laws apply.

These are derived by considering a small mass element (face area  $A$ , length  $ds$ ), and by investigating its motion along the direction  $s$  of flow (Figure 4.6).

If the static pressure changes along the path, the forces on the two face areas of the volume element are not balanced, and thus, a decelerating or accelerating force develops that affects the mass element. The inertial force of the mass element opposes this force. Thus, when frictional force is neglected,

$$\frac{dm}{dt} \frac{dv}{ds} + dpA = 0. \quad (4.36)$$

Since  $dm = \rho A ds$ , we obtain

$$\rho A \frac{v}{t} + pA = 0. \quad (4.37)$$

Dividing by  $\rho A$  and integrating across the path from location 1 to location 2 returns

$$\int_1^2 \frac{dv}{dt} ds + \int_1^2 \frac{1}{\rho} dp = 0. \quad (4.38)$$

Solving the integrals finally gives

$$\frac{1}{2}(v_2^2 - v_1^2) + \int_1^2 \frac{dp}{\rho} = 0, \quad \text{conservation of momentum.} \quad (4.39)$$

This is referred to as *Bernoulli's equation* for gases. It describes the relationship between static pressure and the velocity of flow along the path. If, for example, the pressure drops, then the gas accelerates and thus velocity increases.

If energy transfer with the wall is neglected, the total energy of a flowing mass element remains constant. This is made up of three components: pressure energy ( $p dV$ ), kinetic energy (motion with velocity of flow), and thermal energy (random particle movement):

$$\frac{\rho m}{\rho} + \frac{1}{2} mv^2 + cvmT = \text{constant.} \quad (4.40)$$

Because mass is constant,  $dm$  can be canceled out, yielding

$$\frac{p}{\rho} + \frac{1}{2} v^2 + cvT = \text{constant} \quad ; \quad \text{conservation of energy.} \quad (4.41)$$

The three conservation laws for mass, momentum, and energy do not yet define flow behavior clearly. Now, two cases are discussed:

*Case 1:* The cross-sectional area changes gradually along the line. The flowing gas adjusts to this change. Density, velocity of flow, pressure, and temperature alter gradually. Furthermore, no energy transfer between a mass element and its environment shall occur, giving an *isentropic* change of state. This case is discussed in Section 4.2.2.

*Case 2:* For supersonic flow, the flow can change abruptly at a certain area (shock surface). Density, velocity of flow, pressure, and temperature change abruptly. Because the cross-sectional area is unchanged at the shock surface, mass flow density  $j_m = \rho v$  must be constant due to conservation of mass (Eq. (4.35)). This case is discussed in Sections 4.2.6 and 4.2.7.

#### 4.2.2 Gradual Change of Cross-Sectional Area: Isentropic Change of State

We will examine the flow of a mass element in a tube, with negligible friction and energy transfer at the wall. The cross-sectional area  $A$  of the mass element  $dm = \rho A ds$  is equal to the cross section of the tube. If the cross-sectional area gradually decreases or increases along the direction of flow, the gas volume increases (expansion) or decreases (compression) accordingly (Figure 4.7).

A volume change in a gas is accompanied by a change in temperature. Without heat exchange between the mass element and its environment, its entropy will remain constant along the path. This behavior is referred to as *isentropic* or

The critical velocity (Eq. (4.53)) is

$$v^* = \sqrt{\frac{8.314 \text{ J mol}^{-1} \text{ K}^{-1} \times 520 \text{ K}}{0.435 \text{ kg mol}^{-1}} \cdot \frac{2 \times 1}{1.1 + 1}} = 102 \text{ m s}^{-1}, \quad (4.76)$$

and the critical flow density (Eq. (4.54)) is

$$j_m^* = \frac{1333 \text{ Pa}}{159 \text{ m s}^{-1}} \left( \frac{2}{1.1 + 1} \right)^{1/(1.1-1)} \sqrt{\frac{16}{\pi} \cdot \frac{1}{1.1 + 1}} = 8.4 \text{ kg m}^{-2} \text{ s}^{-1}. \quad (4.77)$$

### 4.3

#### Frictional-Viscous Flow through a Tube

##### 4.3.1

##### Laminar and Turbulent Flows through a Tube

This section deals with viscous flow in long tubes and covers its fundamental phenomena. Figure 4.21 shows the various phenomena of tube flow. The phenomena are investigated in the order in which they appear along the course of the gas flow.

The inflow of the fluid into the tube with flow contraction has already been treated in Section 4.2. In the subsequent region of the tube, boundary layers near the wall are slowed down by friction. In nozzles and short tubes, the boundary layer is so thin that the total flow is hardly influenced by friction.

The thickness of the boundary layer increases gradually along the tube and eventually reaches considerable size. Thus, it affects the flow increasingly and, ultimately, determines the flow considerably. Due to wall friction, a velocity distribution develops across the cross section of the tube. This velocity profile changes gradually within the intake range until it eventually reaches its final shape (Figure 4.22).

Wall friction decelerates the flowing fluid, and thus, momentum is no longer a conserved quantity. Furthermore, the moving fluid's wall interaction leads to an exchange of thermal energy, which results in (nearly) isothermal flow. Consequently, energy also is no longer a conserved quantity.

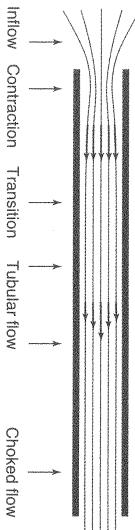


Figure 4.21 Gas flow through a long tube.

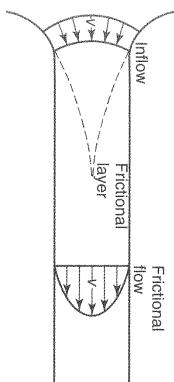


Figure 4.22 Flow pattern developing across the cross section of a tube behind inflow into the tube.

The following investigations consider flow through a tube. We will assume a long tube and will examine a section of the tube, far beyond the inlet of the tube, where the final velocity profile has established.

Equations given here are true for stationary isothermal flow. In the case of pulsating flow, flow resistance is usually higher (compare electrical analogy: additional resistance caused by impedances).

As described in Section 4.1, flow in long tubes can be laminar as well as turbulent. The *Reynolds* number *Re* is the criterion used to differentiate between the two types of flows:

$$Re = \frac{\rho v d}{\eta}, \quad (4.4)$$

where  $\rho$  is the density of gas flow,  $v$  is the velocity of gas flow (mean value across tube's cross section),  $d$  is the cross section (for circular tubes, diameter) and  $\eta$  is the dynamic viscosity of the gas.

For a tube with circular cross section (*circular tube*),

$$Re \left\{ \begin{array}{l} < 2300 \text{ characterizes laminar flow,} \\ > 4000 \text{ characterizes turbulent flow.} \end{array} \right. \quad (4.5)$$

The *Reynolds* number is not appropriate for precisely characterizing the accurate transition between the two types of flows. This is because the flow behavior is determined by the condition of flow at the inlet into the considered stretch, and is very sensitive to wall roughness.

Usually, flow velocity  $v$  is unknown but it can be calculated from  $pV$  flow  $q_{pV}$ :

$$v = \frac{1}{A} \cdot \frac{dV}{dt} = \frac{4}{\pi d^2} \cdot \frac{q_{pV}}{p}. \quad (4.6)$$

Putting in the velocity of flow into the term for the *Reynolds* number (Eq. (4.4)) yields

$$Re = \frac{4}{\pi} \frac{q_{pV}}{\eta d} = \frac{4}{\pi} \frac{\rho q_{pV}}{\eta p d} = \frac{32}{\pi^2} \frac{q_{pV}}{\eta p d^2}. \quad (4.7)$$

Thus, the condition given by Eq. (4.5) for the type of flow can be rewritten as

$$\frac{q_{pV}}{d} \left\{ \begin{array}{l} < 709 \eta c^2 \text{ for laminar flow,} \\ > 1234 \eta c^2 \text{ for turbulent flow.} \end{array} \right. \quad (4.80)$$

In *laminar flow*, the individual layers slide along one another. The frictional force is proportional to the velocity and viscosity of the flowing liquid (Newtonian approach). As derived elementally in textbooks on fluid mechanics, this type of arrangement yields the following *Hagen-Poiseuille equation* for volumetric flow:

$$\frac{dV}{dt} = \frac{\pi}{128} \frac{1}{\eta} \frac{dp}{dl} d^4. \quad (4.81)$$

This equation applies to fluid elements with constant volume. In gas flow, the pressure drops along the tube, and thus, the volume increases accordingly. If the temperature remains constant along the flow path due to heat exchange with the walls of the tube,  $pV$  flow  $q_{pV}$ , however, is constant. In this case, Eq. (4.81) that applies to a short piece of tube can be integrated over the length  $l$  of the tube, from point 1 to point 2. Thus,  $pV$  flow

$$q_{pV} = \frac{\pi}{256} \frac{1}{\eta} \frac{d^4}{l} (p_1^2 - p_2^2), \quad \text{circular tube; laminar flow,} \quad (4.82)$$

and conductance

$$C = \frac{q_{pV}}{p_1 - p_2} = \frac{\pi}{256} \frac{1}{\eta} \frac{d^4}{l} (p_1 + p_2), \quad \text{circular tube; laminar flow.} \quad (4.83)$$

For *turbulent flow*, a semi-empirical method is used to calculate throughput and conductance. As above, the temperature is assumed to be constant. The following semi-empirical formulation describes the pressure loss  $dp$  in the tube per length  $dl$ :

$$\frac{dp}{dl} = \lambda \frac{1}{2d} \rho v^2. \quad (4.84)$$

Here,  $\lambda$  is the so-called dimensionless friction coefficient of the tube and  $\rho$  is the gas density. The mean longitudinal flow velocity  $v$  is simply the ratio of volume flow and cross-sectional area (Eq. (4.78)).

The *Blausius* equation is commonly used to calculate the coefficient of friction  $\lambda$  in a circular tube with smooth inner surface:

$$\lambda = \frac{0.3164}{\sqrt[4]{Re}} = 0.3164 \sqrt[4]{\frac{l}{\rho d^2}}. \quad (4.85)$$

Equations (4.84) and (4.85) yield the pressure loss per distance as a function of flow velocity. The velocity of flow can be substituted by the flow (Eq. (4.78)), and density may be eliminated using pressure (Eq. (3.43)):

$$\frac{dp}{dl} = \frac{0.3164}{2} \frac{1}{p} \sqrt{\frac{8^3 \times 4^7}{\pi^{10}} \frac{1}{d^{19}} \frac{\eta}{c^2} q_{pV}}. \quad (4.86)$$

Integrating the pressure loss over the length of the tube and rearranging yields the  $pV$  flow through a circular tube with diameter  $d$  and length  $l$ :

$$q_{pV} = 1.015 d^{19/7} \left( \frac{c^6}{\eta} \right)^{1/7} \left( \frac{p_1 + p_2}{l} \right)^{4/7} (p_1 - p_2)^{-3/7} \quad \text{for turbulent flow,} \quad (4.87)$$

and conductance

$$C = 1.015 d^{19/7} \left( \frac{c^6}{\eta} \right)^{1/7} \left( \frac{p_1 + p_2}{l} \right)^{4/7} (p_1 - p_2)^{-3/7} \quad \text{for turbulent flow.} \quad (4.88)$$

A tube is considered smooth if wall roughness is less than 1% of the diameter, if the *Reynolds* number is not too high. In rough tubes and tubes with sharp bends, gas flow and conductance are below the values calculated with the preceding equations.

Careful attention should be paid to the outlet pressure  $p_2$  of the tube. In laminar as well as turbulent flow, the pressure drops along the tube due to wall friction. Thus, at constant temperature, the volume, volume flow, and velocity of flow increase. If the outlet pressure  $p_2$  of the tube is sufficiently low, the velocity of flow approaches a maximum, namely, the speed of sound  $a$ . At this critical pressure  $p_*$ , flow becomes choked and the throughput attains its maximum value  $q_{pV}^*$ . The pressure at the exit of the tube cannot drop below the critical pressure. If, however, the vessel pressure is reduced further, the emanating gas suffers an abrupt pressure drop when escaping from the tube into the vessel. Reducing vessel pressure below the critical pressure does not increase gas flow. We will now calculate the critical outlet pressure  $p_*$  at which choked flow occurs.

The maximum  $pV$  flow at a cross section  $A$  ( $a$  = speed of sound, see Eq. (4.55)) is

$$q_{pV}^* = p^* A a. \quad (4.89)$$

Equation (4.82) specifies the  $pV$  flow through a circular tube for laminar flow, and Eq. (4.87) for turbulent flow. For the approximation  $p_1^2 - p_2^2 \approx p_1^2$ , the critical

pressure is obtained by putting in the  $pV$  flow into Eq. (4.89) and rearranging:

$$p^* = \frac{d^2 p_1^2}{64 \eta l a}, \quad \text{critical pressure for laminar flow,} \quad (4.90)$$

$$p^* = 1.92 \frac{1}{ad} \left( \frac{\rho_0}{\eta} \right)^{1/7} \left( \frac{d^3 p_1^2}{2l} \right)^{4/7}, \quad \text{critical pressure for turbulent flow.} \quad (4.91)$$

Maximum gas flows  $q_{pV}^*$  for laminar and turbulent flows through a circular tube are calculated from Eqs. (4.82) and (4.87), respectively; if the critical pressure  $p^*$  is used as exit pressure  $p_2$ , according to Eqs. (4.90) and (4.91), respectively:

### 4.3.2 Airflow through a Tube

#### Airflow through a Tube

We will now study airflow through a circular tube (diameter  $d$ ) at 20 °C (Figure 4.23) and will derive matched numeric-value equations. Numeric-value equations will be marked with a bar to the left, in order to differentiate them from primarily used physical-quantity equations. A tube with pressures  $p_1$  at the inlet and  $p_2$  at the outlet is examined. To obtain numeric-value equations, the following properties of air will be used:

- Mean thermal particle speed,  $\bar{v} = 463 \text{ m s}^{-1}$ .
- Viscosity,  $\eta = 18.2 \times 10^{-6} \text{ Pa s}$ .
- Flow function at critical point,  $\Psi(p^*/p_1) = 0.484$ .
- Speed of sound,  $a = 343 \text{ m s}^{-1}$ .

A *short tube* behaves just like a nozzle. Here,  $pV$  flow is given by Eq. (4.59). Supposing outlet pressure is less than, approximately, half of the inlet pressure ( $p_2 < \frac{1}{2} p_1$ ), choked flow develops. For this, the numeric-value equation ( $q_{pV}$  in  $\text{mbar} \text{ s}^{-1}$ ,  $d$  in cm,  $p_1$  in mbar) reads

$$q_{pV} = 15.6 d^2 p_1, \quad \text{choked nozzle airflow.} \quad (4.92)$$

For *long tubes*, it is necessary to verify whether flow is laminar or turbulent. The criterion for this is expressed in Eq. (4.80). With the values, the numeric-

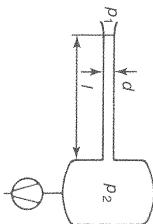


Figure 4.23 Flow inside a tube. Flow at the inlet is nearly inviscid, whereas the flow is viscid inside the long tube.

value equation is obtained ( $q_{pV}$  in  $\text{mbar} \text{ s}^{-1}$ ,  $d$  in cm):

$$\frac{q_{pV}}{d} \begin{cases} < 277, & \text{laminar flow,} \\ > 481, & \text{turbulent flow.} \end{cases}$$

Equation (4.82) is used for *laminar flow*. With the values for air, the following numeric-value equation is found ( $q_{pV}$  in  $\text{mbar} \text{ s}^{-1}$ ,  $p_1$  and  $p_2$  in mbar,  $d$  and  $l$  in cm):

$$\frac{q_{pV}}{d} = 135 \frac{d^3}{l} \cdot \frac{p_1^2 - p_2^2}{2}, \quad \text{laminar airflow.} \quad (4.94)$$

Equation (4.87) is applicable to *turbulent flow*. With the values for air, the following numeric-value equation is found ( $q_{pV}$  in  $\text{mbar} \text{ s}^{-1}$ ,  $p_1$  and  $p_2$  in mbar,  $d$  and  $l$  in cm):

$$\frac{q_{pV}}{d} = 136 \left( \frac{d^3}{l} \frac{p_1^2 - p_2^2}{2} \right)^{4/7}, \quad \text{turbulent airflow.} \quad (4.95)$$

Finally, we will consider the critical pressure by introducing the values to Eqs. (4.90) and (4.91) ( $p^*$  and  $p_1$  in mbar,  $d$  and  $l$  in cm):

$$p^* = 2.5 \frac{d^2 p_1^2}{l}, \quad \text{critical pressure for laminar airflow,} \quad (4.96)$$

$$p^* = 5.1 \frac{1}{d} \left( \frac{d^3 p_1^2}{2l} \right)^{4/7}, \quad \text{critical pressure for turbulent airflow.} \quad (4.97)$$

For convenient practical use, the previous equations are plotted in Figures 4.24 and 4.25.

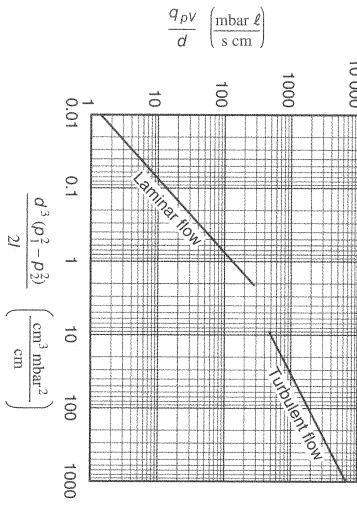


Figure 4.24 Nomogram for obtaining the airflow (20 °C) according to the equations for laminar airflow, Eq. (4.94), and turbulent airflow, Eq. (4.95), through a long tube with circular cross section for fixed inlet and outlet pressures.



Figure 4.40 Axis length in a tube bend and a tube elbow.

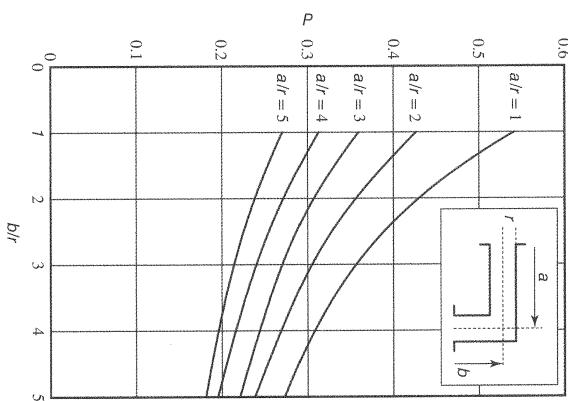


Figure 4.41 Transmission probabilities of 90° tube elbows with circular cross sections and selected dimensions. As in the previous figures, the transmission probabilities apply to a mounting position between two chambers but not to a position between tubes.

**Example 4.12**

Two vacuum vessels are connected via a 90° elbow consisting of a tube with nominal diameter DN 25 ( $d = 25 \text{ mm}$ ). The mounting dimensions of the elbow are the common metric ones, that is, 50 mm displacement in both x- and y-directions.

**Case 1:** A radius elbow is used. The effective length is  $\frac{1}{4}$  of a full circle with 50 mm radius of curvature, and thus,  $l = \frac{1}{4}\pi r c = 79 \text{ mm}$ . The  $l/d$  ratio is  $79/25 = 3.16$ . Using Eq. (4.158) or Figure 4.37, the transmission probability  $P = 26.5\%$ .

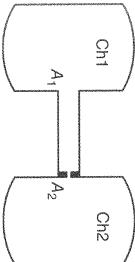
**Case 2:** A mitered elbow is used. The effective length of the tube is the sum of the side lengths, 100 mm. The  $l/d$  ratio is  $100/25 = 4$ . As calculated with Eq. (4.158) or obtained from Figure 4.37, the transmission probability  $P = 22.4\%$ . The precise result of Monte Carlo simulation (Figure 4.41) in the considered case ( $a/r = b/l = 50/12.5 = 4$ ) is  $P = 24 \pm 1\%$  (reading error of figure).

Considering conductance, the radius elbow is superior to the mitered elbow. Better than both of these components is a large sphere with two attached short tubes for the connections. It should be noted that the transmission probabilities given in the example correspond to the intrinsic conductance, that is, are valid for a component mounted directly between the two vessels where the gas particles approach isotropically. If the components are attached via stubs, as commonly used, other transmission probabilities result due to the developing tubular flow at the inlet to the components. This type of arrangement is covered in Section 4.4.8.

**4.4.7 Series Connection of Tube and Aperture**

In practice, tubes with different cross sections are often combined in a serial arrangement. A simple example is a series connection between two chambers, made up of a tube (cross-sectional area  $A_1$ ) and an aperture (cross-sectional area  $A_2$ ) (Figure 4.42). First, we will investigate molecular flow from the left-hand chamber Ch1 into the right-hand chamber Ch2.

The particle flow hitting the entrance plane  $A_1$  from chamber Ch1 is  $q_{N1}$ . The transmission probability for the tube shall be  $P$ . Then, a particle flow  $q_{N1}P$  reaches the area  $A_2$  at the aperture. Here, a portion  $A_2/A_1$  passes through the aperture and enters into chamber Ch2. The other portion,  $1 - A_2/A_1$ , is reflected backward, in the direction opposite to the inflow. Again, from the reflected gas particles, a portion  $P$  reaches  $A_1$  and thus finds its way back into chamber Ch1 while the remaining portion  $1 - P$  reaches  $A_2$ . From this portion, a fraction (corresponding to the surface-area ratio  $A_2/A_1$ ) enters into chamber Ch2 while the rest is reflected backward just as in the first reflection. The total particle flow leaving the aperture toward the right-hand side is the sum of the contributing particle flows, traveling the tube once, with an extra double passage, with two

Figure 4.42 Two chambers connected by a series connection of a tube (cross-sectional area  $A_1$ ) and an aperture (cross-sectional area  $A_2$ ).

extra double passages, and so on. Adding up the geometric series yields the transmission probability  $P_{12}$  of the arrangement according to Figure 4.42 in the direction oriented from surface  $A_1$  to surface  $A_2$ :

$$\frac{1}{P_{12}} = \frac{1}{P} + \frac{A_1}{A_2} - 1. \quad (4.161)$$

Analogous treatment of a flow from chamber Ch2 to chamber Ch1 yields the transmission probability in the opposite direction, from 2 to 1:

$$\frac{1}{P_{21}} = \frac{A_2}{A_1} \cdot \frac{1}{P} - \frac{A_2}{A_1} + 1. \quad (4.162)$$

By eliminating the quantity  $P$  from the previous two equations, it follows that

$$A_2 P_{21} = A_1 P_{12}. \quad (4.163)$$

The product of the area of the exit plane and the transmission probability is just proportional to the conductance. Thus, we find that the conductance from chamber Ch1 to chamber Ch2 is identical to the conductance in the opposite direction. This relationship is universally valid for any arrangement of passive line elements. It indicates that, under stationary conditions, the pressure among connected chambers with the same temperature is balanced.

#### 4.4.8 Series Connection of Components

We will consider a series connection of several components with dissimilar cross sections and lengths. The components include tubes, apertures (tube of length zero), inserted chambers (tube with very large diameter), and other types of components such as bent tubes (Figure 4.43).

The net conductance cannot be obtained by treating the arrangement as a simple series connection of the individual components. The intrinsic conductance of a component applies to the case where the component lies between two large chambers. This quantity has two constituents: the inflow to the component and the tubular flow through the component. If the cross section of the line at the transition between two subsequent components remains unchanged or even

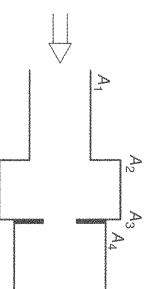


Figure 4.43 Series connection of four tubes with different cross-sectional areas  $A$ .

increases, the inflow loss disappears for the succeeding component. However, if the cross-sectional area of the line decreases at the transition from one component to the next, inflow loss occurs to an extent determined by the drop in the cross-sectional area. In the previous section, a descriptive example showed that, in a series connection of components, the transmission probability depends on the direction.

The sum of resistances due to inflow and tubular flow is a good approximation for the intrinsic flow resistance of an individual component. In a series connection of components, the total resistance is then given by the sum of individual resistances. However, if the cross-sectional area of the line at the intake of the component does not decrease, the resistance due to inflow is set to zero (Oatley's approach). Thus, the following addition theorem is obtained for a series connection of components:

$$\frac{1}{A_1} \left( \frac{1}{P_{in}} - 1 \right) = \sum_{i=1}^n \frac{1}{A_i} \left( \frac{1}{P_i} - 1 \right) + \sum_{i=1}^{n-1} \left( \frac{1}{A_{i+1}} - \frac{1}{A_i} \right) \delta_{i,i+1}. \quad (4.164)$$

where  $i$  denotes the component number;  $A_i$  is the cross-sectional area of intake to the arrangement;  $A_i$  is the cross-sectional area of component  $i$ ;  $P_{in}$  is the total intrinsic transmission probability for the arrangement;  $P_i$  is the intrinsic transmission probability for component  $i$ ;  $\delta_{i,i+1} = 1$  if  $A_{i+1} < A_i$  (decreasing cross-sectional area), and  $\delta_{i,i+1} = 0$  if  $A_{i+1} \geq A_i$  (without reduction in cross-sectional area).

*Special case:* Series connection of two components with cross-sectional areas  $A_1$  and  $A_2$ , and transmission probabilities  $P_1$  and  $P_2$ . From Eq. (4.164), it follows for the general case with arbitrary cross sections:

$$\frac{1}{P_{12}} = \frac{1}{P_1} + \frac{A_1}{A_2} \left( \frac{1}{P_2} - 1 \right) + \left( \frac{A_1}{A_2} - 1 \right) \delta_{1,2}. \quad (4.165)$$

*Case 1:* The cross-sectional areas of both tubes are the same,  $A_1 = A_2$ . Thus,  $\delta_{1,2} = 0$  and

$$\frac{1}{P_{12}} = \frac{1}{P_1} + \frac{1}{P_2} - 1. \quad (4.166)$$

*Case 2:* The cross-sectional area increases,  $A_2 > A_1$ . Thus,  $\delta_{1,2} = 1$  and

$$\frac{1}{P_{12}} = \frac{1}{P_1} + \frac{A_1}{A_2} \left( \frac{1}{P_2} - 1 \right). \quad (4.167)$$

*Case 3:* The cross-sectional area decreases,  $A_2 < A_1$ . Thus,  $\delta_{1,2} = 1$  and

$$\frac{1}{P_{12}} = \frac{1}{P_1} + \frac{A_1}{A_2} \cdot \frac{1}{P_2} - 1. \quad (4.168)$$

**Example 4.13**

Two tubes with circular cross sections, equal diameter  $d$ , and lengths  $l_1 = 2d$  and  $l_2 = 3d$  are connected and thereby form a tube with length  $l_{12} = 5d$ . With Eq. (4.158), the transmission probabilities of the two tubes are  $P_1 = 0.355$  and  $P_2 = 0.274$ . If the transmission probability for the assembled tube is calculated by considering a simple series connection of conductances, as in electrical engineering, with the equation  $1/P_{12} = 1/P_1 + 1/P_2$ , the result is  $P_{12} = 0.155$ . If the transmission probability is calculated with the approximation formula for vacuum ducts in Eq. (4.166),  $1/P_{12} = 1/P_1 + 1/P_2 - 1$ , then  $P_{12} = 0.183$ .

For the assembled tube, the effective transmission probability, according to Eq. (4.158), is  $P_{12} = 0.190$ .

The given approximation formula for a series connection of flow conductances thus yields a considerably more accurate result compared with the simple equation for serially connected electrical conductances. The reason is that the flow at the outlet of the first tube is no longer isotropic, as it is when it enters the first tube. In fact, it is directed forward in the tube (*beam-effect*) and thus passes through the second tube more easily.

**Example 4.14****Series connection of elements in a line.**

Certain components such as valves feature a more complex geometry and their transmission probabilities are usually unknown. Manufacturers list the intrinsic conductance  $C$  in technical specifications from which the transmission probability can be calculated using  $C = \frac{1}{4}\bar{c}AP$ . If, furthermore, isothermal conditions are assumed (constant thermal velocity  $\bar{c}$  within the line), the conductance of the series connection can be calculated by rewriting Eq. (4.164):

$$\left(\frac{1}{C_{in}} - \frac{4}{\bar{c}A_i}\right) = \sum_{i=1}^n \left(\frac{1}{C_i} - \frac{4}{\bar{c}A_i}\right) + 4\bar{c} \sum_{i=1}^{n-1} \left(\frac{1}{A_{i+1}} - \frac{1}{A_i}\right) \delta_{i,i+1}. \quad (4.169)$$

**Molecular Flow through Conical Tube with Circular Cross Section (Funnel)**

Figure 4.44 shows a conical tube (circular cross section) as is used, for example, for reducing adaptors between pump flanges and vacuum lines.

If the transition in cross section is smooth, the cone can be thought of as a series connection of short circular-cylindrical tube elements with changing

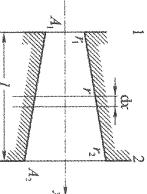


Figure 4.44 Conical tube.

diameters. According to Eq. (4.156), the inverse transmission probability  $1/P_{dx}$  of a short element of length  $dx$  is given by

$$\frac{1}{P_{dx}} = \frac{1}{1 - (dx/2r)} \approx 1 + \frac{dx}{2r}. \quad (4.170)$$

The latter approximation is correct, since  $dx/2r \ll 1$ . Analogous to Eq. (4.164), the transmission probability of the conical tube piece can be calculated by treating it as a series connection of tube elements and by integrating over the length of the tube (rather than summing up the contributions of the tube elements).

This yields the transmission probability in the direction of increasing diameter:

$$\frac{1}{P_{12}} = 1 + A_2 \int_{x_1}^{x_2} \frac{1}{A(x)} \cdot \frac{dx}{2r} = 1 + \frac{r_1 + r_2}{4r_2^2} l, \quad (4.171)$$

and facing toward the direction of decreasing diameter,

$$\frac{1}{P_{21}} = 1 + A_2 \int_{x_2}^{x_1} \left( \frac{1}{A(x)} \cdot \frac{1}{2r} - \frac{1}{A^2(x)} \cdot \frac{dA}{dx} \right) dx = \frac{r_2^2}{r_1^2} + \frac{r_1 + r_2}{4r_1^2} l. \quad (4.172)$$

By calculation, it is easy to verify Eq. (4.163) for a funnel; therefore, conductances for both directions of flow are the same as it has to be.

The given equations can be tested by equating the two radii of the conical tube, which then leads to the known case of a cylindrical tube with circular cross section. The obtained equation corresponds to the approximation formula in Eq. (4.154). This is because both equations are based on the approach that the transmission probability for an arbitrarily long tube can be calculated with the approximation of inversely summing up the probabilities of passage for an entry aperture and a very long tube. This approximation is correct for the special cases of very short and very long tubes. However, for  $l/d \approx 2$ , it produces a positive error of 13%.

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## A.2 Pumps

The system utilises two pumps, a pre-pump and a turbo pump.

### A.2.1 Pre-Pump

The pre-pump is located under the chamber. This pump is used to pump the chamber from atmospheric pressure into a pressure range where the turbo pump can be effective. This pump can evacuate around 8 litres of air a minute and is known as a “Positive-Displacement Pump”. The most basic explanation of this type of pump is that a partial vacuum is generated within the pump by increasing the volume of a cavity, reducing the pressure inside the pump below that of atmosphere. Because of the pressure differential, gas from the chamber flows into this cavity. This cavity is then closed off, opened to the atmosphere, compressed and then closed to the atmosphere before being expanded again.

This pump moves the same volume of gas with each cycle, so its pumping speed is constant unless it is overcome by back-streaming, leakage and outgassing when we will reach the base pressure for the pump.

### A.2.2 Turbo Pump

For more information on turbo molecular pumps, one can read Chapter 10 (specifically pages 419-421,430-432 and 450-455) from Handbook of Vacuum Technology (Second Edition), edited by Karl Jousten, Wiley-VCH, 2016 (reprinted 2017), ISBN: 978-3-527-41338-6.

The turbomolecular or ”turbo” pump is controlled through a program on the system computer. This pump is an example of a momentum transfer pump which are used to obtain and maintain high vacuum environments. It can be understood as a series of spinning blades which impart momentum to gas particles, deflecting them through the pump to the exhaust. A turbo can only work when the mean free path of the gas being pumped is greater than the separation between blades, or else it will stall and there will be no net pumping. This means that we need the chamber and the turbo pump exhaust to both be below atmospheric pressure to operate, which we obtain by using the pre-pump. A turbo pump can evacuate hundreds of litres of gas a minute when running at full speed, the one attached to the chamber here is around 300 litres a minutes.

In order to calculate the pumping speed of different pumps and to try to optimise the time for pumping down a chamber of a given volume  $V$ , one can relate the pumping speed of a pump as follows

$$S_{eff} = \frac{V}{\Delta t} \ln \frac{p_0}{p_1} \quad (17)$$

where  $\Delta t$  is the time to pump between pressures  $p_0$  and  $p_1$

## A.3 Pressure Gauges

The chamber has several pressure gauges. Each one works on a different principle and operates in a particular range. At some pressures, more than one gauge will display a reading however, only one gauge should be trusted in each range due to the accuracy of the method employed. Pressure is sensed by mechanical elements such as plates, shells, and tubes that are designed and constructed to deflect when pressure is applied. This is the basic mechanism converting pressure to physical movement. Next, this movement must be transduced to obtain an electrical or other output.

### A.3.1 Capacitative Pressure Gauge

In capacitive pressure gauge the sensing element is a taut metal diaphragm located equidistant between two stationary metal surfaces, comprising three plates for a complementary pair of capacitors. An electrically

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insulating fill fluid (usually a liquid silicone compound) transfers motion from the isolating diaphragms to the sensing plates, and also doubles as an effective dielectric for the two capacitors. Any difference of pressure across the cell causes the diaphragm to flex in the direction of least pressure. Since capacitance between conductors is inversely proportional to the distance separating them, capacitance on the low-pressure side will increase while capacitance on the high-pressure side will decrease. A capacitance detector circuit connected to this cell uses a high-frequency AC excitation signal to measure the different in capacitance between the two halves, translating that into a DC signal which ultimately becomes the signal output by the instrument representing pressure. This gauge can be used for higher pressures.

### A.3.2 Pirani Pressure Gauge

The Pirani gauge consists of a metal filament (usually platinum) suspended in a tube. The filament is connected to an electrical circuit from which, after calibration, a pressure reading may be taken. The filament is heated when electric current flows through it. This wire suspended in a gas will lose heat to the gas as its molecules collide with the wire and remove heat. As the gas pressure is reduced the number of molecules present will fall proportionately, the conductivity of the surrounding media will fall and the wire will lose heat more slowly. Measuring the heat loss is an indirect indication of pressure. The electrical resistance of the wire varies with its temperature, so the measurement of resistance also indicates the temperature of wire. The gauge may be used for pressures between 0.5 mbar to  $10^3$  mbar.

### A.3.3 Penning Pressure Gauge

The Penning gauge is a sensor consisting of two electrodes, an anode and cathode. The outer cylinder of the gauge is the cathode and is at room temperature. The anode consists of a tungsten wire mounted in the centre of the tube and a potential difference is applied between anode and cathode. A magnetic field is introduced at right angles to the plane of the electrodes by a permanent magnet. The electrons emitted from the cathode are deflected by means of the magnetic field are made to take a long helical path before reaching the anode loop. The electrons ionise the gas between the anode and cathode by collision, even at low pressures. This creates a cascade of electrons that flows towards the anode, producing a current through the gas. This small current is calibrated to give a measure of the pressure of the gas and hence the chamber to which it is attached. The Penning gauge can detect pressures from  $10^2$  to  $10^7$  mbar.

### A.3.4 How the Mass Spectrometer Works

The quadrupole mass analyser (QMS) filters sample ions, based on their mass-to-charge ratio ( $m/z$ ). Ions are separated based on the stability of their trajectories in oscillating electric fields that are applied within the instrument.

The QMS consists of four parallel cylindrical metal rods. Each opposing rod pair is connected together electrically, and a radio frequency (RF) voltage with a DC offset voltage is applied between one pair of rods and the other. For a given applied voltage, only ions of a certain mass-to-charge ratio will reach the detector after travelling through the QMS. Other ions have unstable trajectories and will collide with the rods. This permits selection of an ion with a particular  $m/z$ .

Further reading:

- Chapter 14.2.4 (pages 655-661) from *Handbook of Vacuum Technology* (Second Edition), edited by Karl Jousten, Wiley-VCH, 2016 (reprinted 2017), ISBN: 978-3-527-41338-6.
- de Hoffmann, Edmond; Vincent Stroobant (2003). *Mass Spectrometry: Principles and Applications* (Second ed.). Toronto: John Wiley & Sons, Ltd. p. 65. ISBN 0-471-48566-7.

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- Chernushevich, Igor V. (2001). "An introduction to quadrupole time-of-flight mass spectrometry". Journal of Mass Spectrometry. **36** (8): 849–865

## A.4 Thin Film Growth

The film morphology is defined as the total film microstructure which it obtains and the structures which result due to the specific orientations of atomic species over the process of thin film growth. This may be modified by the following ways.

1. Substrate temperature
2. Deposition rate
3. Geometry of chamber/orientation of cathodes
4. Bulk and surface diffusion of atomic species
5. Deposition method

These factors can be tuned using various experimental apparatus at the disposal of the student. The substrate temperature can be increased using the substrate heater which can influence the surface diffusion of the incoming atomic species thereby affecting thin film morphology. The deposition rate can be tuned by changing the power provided to the cathodes so as to change the sputter yield and this again will affect the growth of the thin film. The growth pressure can be tuned using the needle valve to inject Ar gas into the chamber, this in turn affects the mean free path of the ejected atomic species which changes its incoming kinetic energy and therefore the surface diffusion on the substrate.

### A.4.1 Growth Modes

During the different thin film deposition processes described above. The incoming material species can adhere on the substrate via different modes. The three typical growth modes namely are Island growth (Volmer-Weber), Layer by-layer growth (Frank van der Merwe) and mixed growth (Stranski Krastanov). Thin film growth processes are not always perfect in which one gets perfect homo-epitaxial surfaces between two neighbouring materials. Nucleation/aggregation of atomic species usually occurs at defects. Thin film defect and dislocations study is an entire branch of condensed matter research. There are a range of different defects which may occur during growth processes. The more inclined readers of this manuscript are urged to seek references [Niklas Nilius, Surface Science Reports 64 (2009) 595] It should be noted that in certain instances, defects have been proven to be an asset for industrial applications of various thin films.

#### A.4.1.1 Volmer-Weber

Such a film growth mode is characterised by 3-D island type growth in which the material atoms are strongly bonded to each other as compared to the substrate surface. Moreover there is slow diffusion of the atoms on the surface of the substrate. The lowest energy state is obtained when as much of the substrate surface is left uncovered. The total surface energy of the film interfaces is larger than the substrate-vapour interface. The material therefore reduces its surface area to minimise interface with the substrate.

#### A.4.1.2 Frank-van der Merwe

When individual atoms form a layer-by-layer growth due to the fact that the substrate-vapour surface energy is larger than the two combined. The thin film wets the surface of the substrate to lower the surface energy.

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#### A.4.1.3 Stranski-Krastanov (mixed)

This growth mode initially begins as a layered growth, however due to changes in the strain energy between the substrate and the incoming atomic species the growth mode changes to island like growth after a few monolayers. The initial atoms adhering to the substrate are strained to match the lattice of the substrate however after a few monolayers the strain is relaxed.