2.12 Recommended Readings

Glang, R. 1970. Chap. 1, "Vacuum Evaporation." In *Handbook of Thin Film Technology*, L. I. Maissel and R. Glang (eds.). New York: McGraw-Hill.
Sears, F.W. 1950. *An Introduction to Thermodynamics, the Kinetic Theory of Gases, and Statistical Mechanics*, Chap. 12–13. Cambridge, Mass.: Addison-Wesley.

Chapter

3

Vacuum Technology

Most of the film deposition processes to be discussed in this book operate under some degree of vacuum. Only atmospheric-pressure CVD does not, but the same vacuum techniques of contamination reduction and process control still apply to it. Vacuum technology is a large topic which is well treated in textbooks such as those in the recommended readings list at the end of this chapter. Our purposes here are more specific: first, to become oriented to the general topic and, second, to examine certain aspects of vacuum technology that are particularly relevant to film deposition and deserve special emphasis. As we know, "Nature abhors a vacuum," so good equipment and techniques are needed to create one.

Figure 3.1 is a schematic diagram of a typical vacuum system for thin-film deposition. The purpose and functioning of the components shown will be elaborated upon in the subheadings below. Sometimes not all of these components will be required for a particular process. As shown, the substrate is introduced through a "load-lock" chamber to allow the main process chamber to remain under vacuum, because this reduces contamination and shortens substrate turnaround time. The roughing pump evacuates the load-lock chamber from atmospheric pressure after the substrate has been loaded into it and before the valve is opened into the process chamber. Once the substrate is in the process chamber, it is heated and controlled at the film deposition temperature. Process gases and vapors are metered into the chamber through mass flow-controlled supply lines, which are discussed more in Sec. 7.1.2. Process pressure is measured by a vacuum gauge that can be coupled to a motor-driven throttle valve in the pump throat for pressure control. Sometimes, pressure is controlled instead by cou-

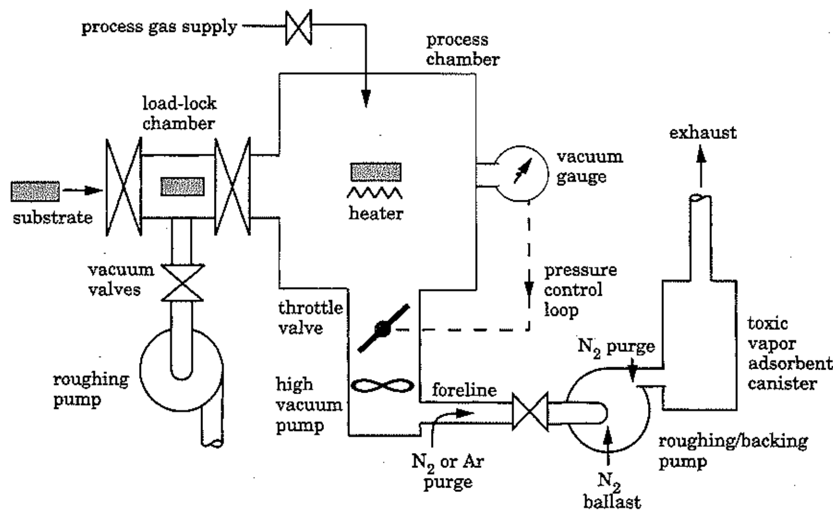

Figure 3.1 Typical vacuum-system components for thin-film deposition.

pling the vacuum gauge to the gas-supply metering valve, but that technique does not allow independent control of gas flow rate and pressure. Finally, process and impurity gases are evacuated through a high-vacuum pump followed by a "backing" pump which often serves to rough out the process chamber from atmosphere as well. For process vacuums above 10 Pa, only one stage of pumping is needed. The foreline and "ballast" nitrogen/argon purges which are shown are often required for reducing process and pump contamination, respectively. The exhaust nitrogen purge and the vapor-adsorbent canister provide safe disposal of flammable and toxic process vapors, respectively.

3.1 Pump Selection and Exhaust Handling

The principal types of vacuum pumps are listed in Table 3.1, along with their key characteristics. The choice of pumps will depend on the process vacuum level and on the properties of the vapors to be handled. Pumps fall into two categories by pumping principle: those that *displace* gas from the vacuum chamber and exhaust it to atmosphere, and those that *trap* it within the pump itself. Displacement pumps are often oil lubricated, which means that great care must be taken to avoid contaminating the process chamber with oil. On the other hand, they can pump large gas flows continuously without becoming saturated like trapping-type pumps do. Trapping pumps of the cryogenic variety are not recommended when pumping flammable vapors,

TABLE 3.1 Vacuum Pump Characteristics

Pressure ranges	Name	Category	Approx. $/(l/s)	Backing pump req'd?	Oil present?		Problematic gases and vapors	Other comments
					Inlet	Outlet		
	Dry rotary	Displacement	1000	No	No	Yes	Condensables require gas ballasting; see text	
	Oil-sealed rotary		300	No	Yes	Yes		Common for roughing/backing
	Roots blower		70	Yes	No	Yes		Oil contam. unless foreline purged
	Molecular drag		35	Yes	No	Yes*	Low compression ratio for H_2 and He	
	Turbo-molecular		40	Yes	No	Yes*		
	Oil diffusion		5	Yes	Yes	Yes		Greatest risk of oil contam.
	Cryosorption	Trapping	450	No	No	(No outlet)	Explosion danger with flammables	For dry roughing
	He-cycle cryopump		7	No	No	No†		Low capacity for He, H_2
	Sputter-ion		25	No	No	(No outlet)	Poor for inerts	
molecular / transition / fluid	Flow regime for 5 cm diameter tube							
−10, −5, 0, +5 Pa; −5, 0 Torr; 1 atm	log of pump inlet pressure							

Legend: ultimate limits (dotted); process operation (solid)

*except magnetically levitated bearing types, which use no lubrication

†Purge roughing pump line to avoid oil contamination during warmup regeneration cycle.

because usually air is pumped too. When the saturated pump is warmed up to regenerate it, the released gas can form an explosive mixture. Sputter-ion pumps are trapping pumps that do not release chemically active trapped gases, but they can periodically release bursts of inert gases during operation. Unlike the cryogenic trapping pumps, they cannot be regenerated when saturated but must have their internal parts replaced.

The next consideration is that the pump operate well at the desired process pressure. Notice in the table that the recommended pressure limits for process operation are much narrower than the ultimate pressure limits. The ultimate upper limit is the maximum pressure at which the pump can be started, whereas the process upper limit is the maximum pressure at which it will operate well on a continuous basis. Excessive operating pressure causes overheating or stalling, as well as rapid saturation in the case of trapping pumps. If the maximum starting pressure is less than 1 atm or if a turbomolecular or molecular drag pump is being used, the system must first be roughed out with another pump. The lower pressure limit of process operation is the pressure below which pumping speed drops off, except in the case of the oil-sealed rotary pump, which is widely used for both roughing and backing. There, the problem at < 10 Pa is contamination from pump oil back-diffusing (backstreaming) into the process chamber, as will be discussed in Sec. 3.4.1. The other two choices for roughing and backing avoid the oil problem but have other drawbacks. The dry rotary pumps are larger, noisier, and more expensive. The cryosorption pumps require frequent refills with liquid nitrogen and frequent regeneration due to saturation with pumped gas.

Another consideration is the cost factor. Approximate purchase costs per unit of pumping speed for medium-sized pumps are listed in Table 3.1. There is a large difference among pumps, but in many cases the higher cost is justified by improved performance. Moreover, there are other cost factors involved, such as maintenance, service life, and whether a backing or roughing pump is required. The pumps used for backing or roughing are the dry and oil-sealed rotary pumps and the cryosorption pump. These all have relatively high cost per unit of pumping *speed* in *l*/s. However, they require a much smaller speed than the high-vacuum pump on a given chamber, because of their higher operating pressure [see Eq. (3.3) below].

The molecular-drag pump is the most recent addition to the selection of available pumps. Sometimes, it is integrated with a turbomolecular pump in a single unit, thus increasing the upper operating pressure of the latter. By itself, it still has a wide operating pressure range that spans the transition-flow regime. This range encompasses all of the glow-discharge plasma deposition processes as well as some CVD processes, so this pump is particularly useful for thin-film work. Moreover, its principle of operation is a classic illustration of gas kinetic behavior. As illustrated in Fig. 3.2, the pump incorporates a channel between the stationary surface of a "stator" and the surface of a "rotor," which is moving to the right at a supersonic velocity u_r. In construction, one of the two surfaces is a spiral channel, and the other is a cylinder surrounding it and almost touching the channel rim. In the molecular-flow regime, molecules bounce from surface to surface without encountering collisions along the way. They approach the rotor surface with mean thermal velocity $\bar{c}$ and bounce off of it having the sum of two velocities, namely: $\bar{c}$, which is randomly directed; and u_r, which is directed to the right. Thus, molecules are pumped to the right. This behavior is a consequence of the strong interaction between gas molecules and surfaces, which causes them to become trapped temporarily and thus lose memory of the direction from which they approached. (Helium may be an exception, at least on very smooth surfaces.)

The molecular-drag pump can also operate at the lower end of the fluid-flow regime. There, a velocity profile from u_r to zero develops across the channel as shown in Fig. 3.2*b*. In the absence of pressure gradients, this profile would be linear in accordance with a force balance using Eq. (2.28) and as discussed in fluid-mechanics texts. However, the pumping action causes pressure to decrease from right to left, which causes some back flow in that direction and somewhat "collapses" the velocity profile, as shown. Now, the mass pumping rate is proportional to molecular concentration, n, and thus to pressure, p,

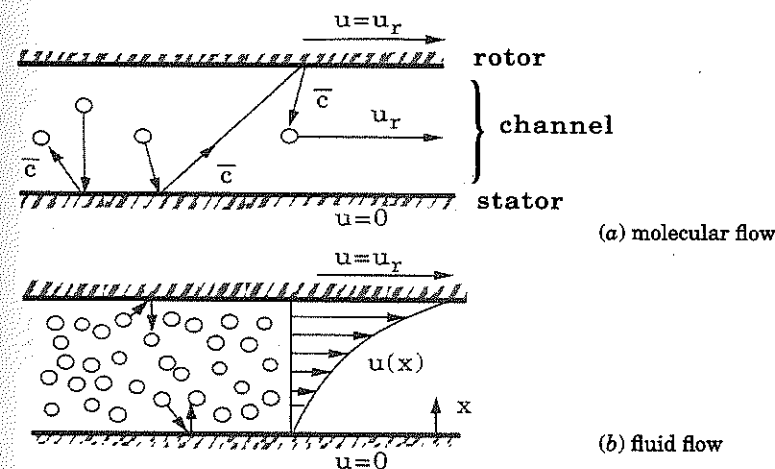

Figure 3.2 Molecular-drag pump operation in the two flow regimes.

whereas it will be seen in Sec. 3.3 that the mass flow rate induced by a p drop in the fluid regime is proportional to p^2. This means that as p is raised, back flow eventually becomes much larger than pumping rate, and this sets an upper limit to pump operating p.

3.2 Problem Gases

Some gases present special pumping problems. H_2 and He have high molecular speeds because of their low mass [Eq. (2.3)]. This limits their compression in molecular-drag, turbomolecular, or oil-diffusion pumps, all of which use supersonic velocity of the pumping medium to push molecules along. Reactive, condensable or toxic gases require special handling whatever pump is used, and we will discuss these three categories in turn below.

Reactive gases cause several problems. Acidic ones such as the chlorinated gases can decompose hydrocarbon pump oil and corrode metal pump parts. Oxidizing ones such as O_2 can explode the pump-oil vapor in the exhaust. Flammable ones such as methane can explode when mixed with air in the exhaust. Decomposition and explosion of the oil are avoided by using perfluorinated pump oils. These are organic molecules that have all of their H replaced by F, which makes them chemically inert due to the high strength of the C-F bond. Even with these, acidic gases dissolved in the oil can corrode the pump, and flammable gases can explode in the exhaust system. These two problems are avoided by N_2 purging at the pump ballast port and at the pump exhaust casing, respectively, as shown in Fig. 3.1. Sufficient and reliable N_2 purging can also prevent decomposition and explosion of hydrocarbon pump oil as an alternative to using the very expensive perfluorinated oil.

N_2 purging at the ballast port not only sweeps dissolved corrosives out of the pump oil, but also prevents **vapor condensation**. Condensates emulsify the oil and thus destroy its sealing and lubricating properties. The ballast port addresses this problem by injecting N_2 into the vapor as it is being compressed, which lowers the partial pressure of the vapor. If the dilution is sufficient, the vapor's partial pressure drops below its saturation vapor pressure, at which point no condensation can occur. In addition, the equilibrium concentration of corrosive gas dissolved in the oil drops roughly in proportion to the drop in its partial pressure over the oil. One example of a condensable and corrosive vapor situation is the $TiCl_4 + H_2O$ mixture sometimes used in the CVD of TiO_2. The saturation vapor pressures of these two reactants at room T are about 1400 and 3000 Pa, respectively. To prevent their condensation upon compression of the process stream to 1 atm (10^5 Pa), a minimum dilution of $10^5/1400 = 70/1$ is necessary. If the dilution provided in the process-gas supply and in the pump foreline is less than 70/1, the difference must be made up at the ballast port.

When **toxic gases** are involved, special procedures are necessary to protect personnel and the environment. For example, arsine (AsH_3), which is used in CVD of the important semiconductor GaAs, is one of the most toxic gases known. Oil removed from pumps for disposal or recycling should be assumed to contain some amount of any toxic gases which have been pumped, and it must be handled and labeled accordingly. The dissolved concentration can be reduced, though not necessarily to negligible levels, by a long purge with N_2 before the oil is removed. Regardless of whether any pumps are employed, the process exhaust stream must be tightly sealed and must be treated to reduce the concentration of toxics to negligible levels before release to the environment. Treatment methods include adsorption (usually on activated charcoal) as shown in Fig. 3.1, washing in a scrubber, and decomposition by burning or by catalysis. Different methods are appropriate for each vapor. The chemical manufacturer and local government authorities need to be consulted prior to selection of process equipment. In all cases, continuous environmental-monitoring sensors are advised at the gas supply, at the process chamber, and downstream of the exhaust treatment equipment.

3.3 Gas Throughput

The required sizes of the pump and its connecting line to the process chamber are determined by the gas load imposed by the film deposition process. The essential features of this situation are illustrated in Fig. 3.3. The mass flow of gas must satisfy the continuity equation, which is an expression of the law of mass (and also energy) conservation. This equation is fundamental to all flow and film-deposition pro-

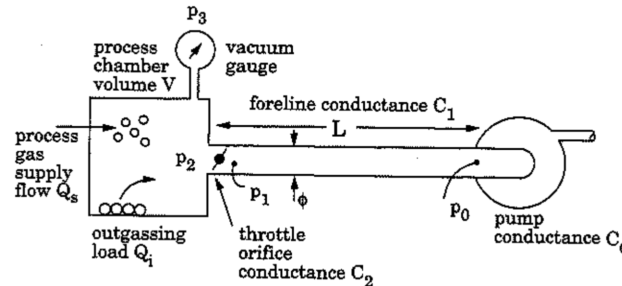

Figure 3.3 Geometry for gas throughput calculations.

cesses, and it will come up again and again throughout the book. It has the general form

$$\boxed{\text{input} + \text{generation} = \text{output} + \text{accumulation}} \tag{3.1}$$

That is, whatever mass or energy is introduced into a given space must either come out again or build up there. Here, the space is the process chamber's gas phase. The generation term typically arises with chemical reactions (mass) or heat production (energy), and it is zero here. We will first consider operation at a constant process-chamber pressure, p_2, so the accumulation term is also zero. There are two input terms: the mass flow rate of process gas supplied, Q_s, and "outgassing" from the chamber walls, Q_i (neglecting unwanted leaks, which should have been plugged). Outgassing is evolution of gaseous contaminants from chamber materials, and its minimization will be discussed in Sec. 3.4.2. The supply flow, Q_s, is often expressed in sccm, which is proportional to mc/s by the ideal-gas law, Eq. (2.10).

The output term in Eq. (3.1) is the flow of gas toward and through the pump(s). In Fig. 3.3, there are three elements in this flow path, each of which has a certain "conductance," C (liters/s, = *l*/s), for the gas. There is a throttle restriction (C_2), a pumping line restriction (C_1), and the pump itself (C_0). The throttle is needed only when p_2 is larger than the maximum operating p of the pump (see Table 3.1), as in the case of low-p CVD or glow-discharge processes run using a turbomolecular pump. The mass flow or "throughput," Q, past these elements is usually expressed in (Pa·*l*/s) or (torr·*l*/s), which is also proportional to mc/s by the ideal-gas law. For the two flow restrictions, C_1 and C_2, C is defined by

$$Q = C\Delta p \tag{3.2}$$

where Δp is the pressure difference across the element and is the driving force for the gas flow. For the vacuum pump, throughput is given by

$$Q_0 = C_0 p_0 \tag{3.3}$$

where the conductance C_0 is known as the pumping "speed" (*l*/s). Usually, C_0 varies little with p_o over the pump's operating range, meaning that pump throughput is proportional to its inlet pressure, p_0. Setting all of these Q values equal in accordance with Eq. (3.1), and taking the case where $Q_i << Q_s$ (it had better be!), we have

$$Q_s + Q_i \approx Q_s = C_2(p_2 - p_1) = C_1(p_1 - p_0) = C_0 p_0 \tag{3.4}$$

For a given Q_s, the three p values will adjust themselves to satisfy these equalities. Then, control of the process pressure, p_2, is accomplished by throttling down C_2 as shown in Fig. 3.1.

Conductances depend on geometry and flow regime. The simplest case is an **orifice in molecular flow,** meaning that the molecular mean free path is larger than the orifice diameter (Kn > 1). In this case, the flux through the orifice in each direction (downstream and upstream) is equal to the impingement flux at the plane of the orifice as given by Eq. (2.18). It is an important property of molecular flow that these two fluxes are independent of one another. This is because of the fact that the molecules cross paths without colliding. (We will see in Sec. 3.4.1 how this causes oil backstreaming.) The orifice throughput is the difference between the downstream and upstream fluxes through the orifice times its area, A; thus

$$Q_2 = (J_{i2} - J_{i1})A = \left[\frac{N_A}{\sqrt{2\pi MRT}}\right]A(p_2 - p_1) = C_2(p_2 - p_1) \tag{3.5}$$

The term in square brackets is the orifice conductance per unit area, C_A. For air at room temperature, conversion from SI units to liters and cm gives $C_A = 11.6$ *l*/s·cm^2, a very useful quantity to remember, since any restriction in the vacuum plumbing can be modeled approximately as an orifice. The conductance of an **orifice in fluid flow** is much more complicated to analyze, but it is also much higher. Therefore, it usually is not the throughput-limiting element in Fig. 3.3 unless it is made so deliberately, as in the case of the throttle valve. Appendix E gives the conductance of a fluid-flow orifice in the limit of sonic velocity in the orifice ("choked" flow).

For **long tubes in molecular flow,** the conductance in *l*/s for air at room temperature is

$$C_m = 12.3\phi^3/L \tag{3.6}$$

where ϕ = tube diameter, cm
L = tube length, cm; $L >> \phi$

Note that C_m is proportional to ϕ^3, versus ϕ^2 for the orifice in Eq. (3.5). Two powers of ϕ come from the area factor as in the case of the orifice, and the third power comes from the fact that the axial distance traversed by a molecule between bounces off the wall is proportional to ϕ. For gases other than air, C_m scales according to Eq. (2.18). For **long tubes in fluid flow,**

$$C_f = 1.41\phi^4 \bar{p}/L \qquad (3.7)$$

where $\bar{p}$ is the average pressure from end to end, in Pa. Note that C is independent of p in molecular flow but proportional to p in fluid flow. Thus, in fluid flow, Q is proportional to p^2. One power of p comes from the concentration of the fluid, and the second comes from the Δp driving force for flow. The third and fourth powers of ϕ here come from the viscous drag force, which is proportional to the radial gradient in axial flow velocity [Eq. (2.28)]. This velocity is inversely proportional to tube area for a given volumetric flow rate. For gases other than air, C_f scales inversely with viscosity. In the transition region ($0.01 < Kn < 1$), both of the above equations will estimate C somewhat low.

The minimum size of foreline tube and pump for a given process can be calculated from the anticipated gas load, Q_s. The tube should be sized so that its C is at least as large as that of the pump, since tubes are less expensive than pumps. However, depositing films often become contaminated by the outgassing load, Q_i. In such cases, a larger pump than the minimum is desirable. For processes operating without a throttle, faster pumping (larger C_0) reduces the partial p of these contaminant gases, p_i, in accordance with Eq. (3.3): that is,

$$p_i = Q_i/C_0 \qquad (3.8)$$

For processes operating at some fixed pressure p_2 with a throttle, faster pumping instead allows the supply gas flow, Q_s, of (presumably pure) process gas to be higher, which dilutes the flow of outgassing contaminant, Q_i, and thus reduces its p_i by

$$\frac{p_i}{p_2} = \frac{Q_i}{Q_i + Q_s} \approx \frac{Q_i}{Q_s} \qquad (3.9)$$

This equation assumes that the gases mix well in the chamber and that the ideal-gas law holds. Note here that p_i also scales with p_2, whereas in the unthrottled case of Eq. (3.8), p_2 does not appear. It does appear in the throttled case because it is presumed that Q_s is already set at the maximum which the pump can handle. Thus, p_2 cannot be increased by increasing Q_s, which would have diluted the contaminant. Instead, p_2 can only be increased by closing the throttle to reduce C_2, which also raises p_i. For this reason, film purity is improved by reducing process pressure, p_2, in cases where the pump must be throttled. Alternatively, a pump operable at higher p_o can be selected. The molecular-drag pump is attractive here, because it also minimizes oil contamination. Equation (3.9) also applies to atmospheric-pressure CVD, where Q_s *can* be increased to reduce p_i, because there is no pump throughput limitation.

The rate of pumpdown from 1 atm can also be calculated from the continuity equation, Eq. (3.1). Here we are considering only evacuation of the air in the process-chamber volume, V; we are not supplying any additional gas. Therefore, the input term is zero. The accumulation term is the evacuation rate. We also assume that the output term is limited by the pump throughput, since (1) the throttle valve will be open, and (2) the tube throughput is proportional to p^2 and will be relatively high at the high-p end. Thus, using the notation of Fig. 3.3, we have $p_2 = p_0$, and Eq. (3.1) becomes

$$0 = C_0 p_2 + V\frac{dp_2}{dt} \qquad (3.10)$$

where both terms have the units of throughput (Pa·*l*/s). Rearranging, integrating, and applying the initial condition that $p_2 = p_{2o}$ at time t = 0, we have

$$p_2 = p_{2o}\exp\left(\frac{-t}{V/C_0}\right) \qquad (3.11)$$

This is a classic exponential-decay situation and is analogous to discharging a capacitor through a resistor. V/C_o is the time constant for the process, and for a typical case of a 100 *l* chamber and a 10 *l*/s roughing pump, the time constant is 10 s. This means that evacuation to 10^{-5} Pa would take place in a mere 4 min. In practice, however, Eq. (3.11) is obeyed only down to 10 Pa or so. At lower p, evacuation slows down progressively as the added load from outgassing of the chamber walls now dominates the situation. Outgassing is one of the major contamination sources that we will discuss in the next section.

3.4 Contamination Sources

The sensitivity of a deposition process to contamination varies greatly with the materials involved and depends on whether the depositing film incorporates or rejects the contaminants arriving at its surface. This, in turn, depends on the chemical reactivity of the surface with the contaminants. For example, gold is not very reactive and can be deposited with high purity in a poor vacuum, whereas aluminum reacts with and incorporates almost all arriving contaminants except the inert gases. The degree to which contamination needs to be controlled must be assessed separately for each material system by analysis of the resulting film. Whenever film properties are poorer than

desired, the question should be asked: to what degree are contaminants responsible for this result?

As discussed in Chap. 1, contaminants can be introduced in all four steps in the deposition process: source, transport, deposition, and analysis. The contaminants associated with the source and analysis steps will be addressed in Chaps. 4 and 10, respectively. Here, we are concerned with contaminants entering the vapor environment between the source and the substrate. These adsorb on the substrate before film deposition commences, and they mix with the transporting source material during deposition. Their reduction requires the application of good vacuum practice, whether the process is operating in ultra-high vacuum (UHV) or at atmospheric pressure. Substrates also can be contaminated before introduction into the process chamber, and this problem will be addressed in Chaps. 5 and 6. The principal sources of contamination entering the process vapor environment will be dealt with in turn below; they are: oil backstreaming from the pumps, gas evolution from chamber materials, and dust stirred up from surfaces.

3.4.1 Oil backstreaming

Oil backstreaming into the process chamber can occur whenever oil is used as the pump operating fluid or lubricant. The rate at which this occurs is much higher than one would predict from the relatively low room-temperature vapor pressures (p_v) of oils used in vacuum technology. This is because pumps usually run hot, and the p_v of any material rises very steeply (exponentially) with T. For example, a typical rotary pump oil has a p_v of 10^{-4} Pa at 25° C but a p_v of 10^{-1} Pa at a pump operating temperature of 85° C.

Backstreaming can occur in both the molecular-flow and fluid-flow regimes, but the mechanisms and remedies differ with regime. Molecular flow is illustrated in Fig. 3.4*a* for the tube between the process chamber and an oil diffusion pump. Despite the flow of gas toward the pump, oil molecules bounce freely backward toward the process chamber without encountering any resistance from the countercurrent gas flow, because the gas and oil molecules do not collide with each other when Kn > 1. Some but not all of the oil will condense on the room-temperature walls of the tube before reaching the chamber. The remedy is to place in the tube a baffle which is optically opaque, meaning that there is no line-of-sight path for an oil molecule through it without encountering at least one surface (two is better). Figure 3.4*a* shows the traditional "chevron" baffle or trap, named after the look-alike sergeant's stripes. The trap is cooled with chilled water or, better yet, liquid nitrogen (LN_2), so that the oil condenses on it and stays

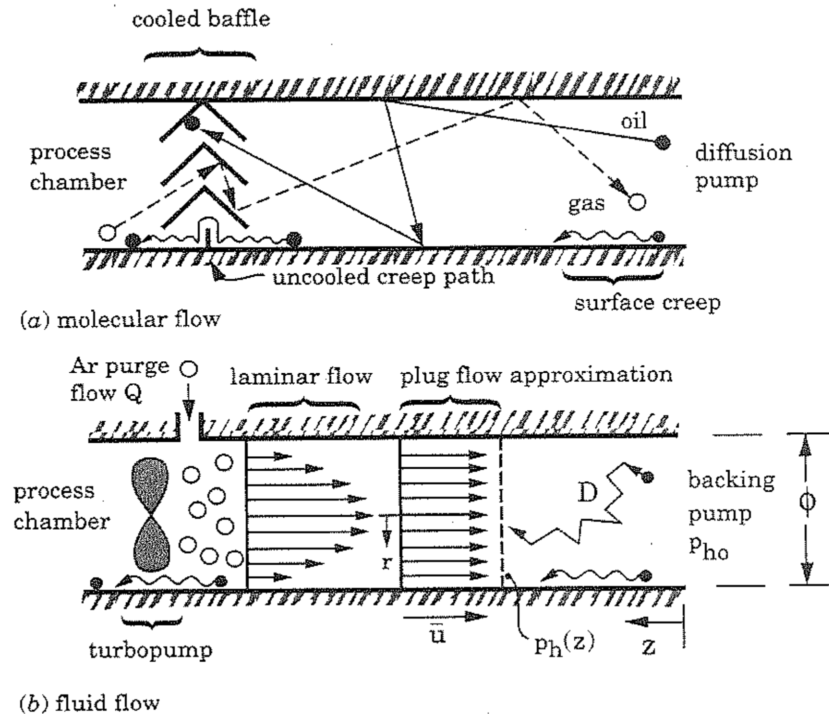

Figure 3.4 Oil backstreaming behavior in the two flow regimes, showing both gas-phase and surface paths

there. However, the trap will be warmed up on occasion, either for cleaning or due to coolant failure. A valve can be used to block off the process chamber on such occasions, but then oil still reaches the downstream face of the valve and is bound to work its way into the chamber eventually. Gas purging of the warm trap can stop the backstreaming, as we will see below, but this is getting complicated. It is clear why oil diffusion pumps are listed in Table 3.1 as having the highest risk of oil backstreaming.

A more insidious backstreaming mechanism which is independent of flow regime is surface diffusion (or "creep," or migration), as shown in Figs. 3.4*a* and *b*. It is possible to stop this with a trap having no surface pathway through it that does not encounter an LN_2-cooled surface, but most traps do not have this feature. Another possible but undocumented remedy is to heat the surface so that the oil evaporates from the surface and into the cold trap or, in fluid flow, into the gas stream. Even turbomolecular pumps, which are generally accepted as

being clean due to the very high compression ratio for heavy molecules such as oil, would seem to be susceptible to surface creep.

Turbopumps and molecular drag pumps lose their high compression ratio when stalled, such as during a power failure or following bearing seizure. Then, rapid backstreaming can occur through the gas phase if the pressure in the pump remains low enough, so these pumps should be interlocked to vent automatically on such occasions.

Unless one is careful, molecular-flow backstreaming also will occur in the foreline between the high-vacuum pump and the backing pump and in the roughing lines to all chambers and pumps. It will happen whenever the pressure in these lines is allowed to drop into the transition flow regime ($Kn > 0.01$). Thus, a 5 cm diameter tube should never be allowed to drop below 20 Pa. Oil roughing pumps should be valved off below this pressure, and backing-pump forelines should be held in the fluid-flow regime with a purge of N_2 or Ar unless oil already exists by design at the outlet of the high-vacuum pump. Alternatively, there are foreline traps available which use a canister of zeolite pellets to trap the oil. Zeolite, or "molecular sieve," is a ceramic with extremely fine porosity which gives the pellets an enormous internal surface area of about 1000 m^2/g. The chemically active surface holds onto oil that adsorbs on it until the trap becomes saturated and is regenerated by baking. Like the chevron baffles, these traps are risky. They can become saturated without anyone realizing it, or they may get baked without the required continuous purge.

Foreline purging is the most reliable way to prevent backstreaming. Figure 3.4*b* shows an Ar purge flow, Q, entering just downstream of a turbomolecular pump. The same technique can be used for Roots blowers and molecular-drag pumps. N_2 can be used instead when the presence of its backpressure in the vacuum chamber is not a contamination issue for the deposition process at hand. Again, pressure in the line must be held high enough so that $Kn < 0.01$, either by raising Q or by throttling the pumping line at the backing-pump end. Sometimes, process-gas flow alone is enough to keep the pressure up. Even with the purge, backdiffusion of oil will occur to some extent against the downstream flow of gas. However, diffusion is driven by a concentration gradient in accordance with Eq. (2.27). Thus, the partial pressure of oil vapor, $p_h(z)$ (h denoting hydrocarbon oil) decreases with increasing distance z upstream from the backing pump and eventually becomes negligible. The profile of $p_h(z)$ in fact reaches a steady state at which the upstream diffusional mass flow due to the p_h gradient is exactly balanced by the downstream bulk flow of oil vapor diluted in purge gas. This balance applies to every cross-sectional plane along the z direction, one such plane being indicated by the vertical dashed line in Fig. 3.4*b*.

The solution to the above problem sets the criterion for sufficient purging and also illustrates a classic differential mass-balance situation which occurs elsewhere in film-deposition work. To solve it, we will make a simplifying assumption regarding the radial velocity profile, u(r), of the purge gas. The actual profile for laminar flow is parabolic, as shown in Fig. 3.4*b* and as derived later as Eq. (7.6). However, instead of this profile, we will assume "plug flow," in which u is constant across the tube diameter and is equal to the area-weighted average value of u(r), $\bar{u}$, as also shown in Fig. 3.4*b*. This assumption is valid for tube length $L >> \phi$ (diameter), because each backdiffusing oil molecule moves sideways in r as well as axially in z, so it soon samples all values of u(r). Now, setting the upstream diffusional flux equal to the downstream bulk flux for oil vapor of concentration $n_h(z)$, we have

$$-D\frac{dn_h}{dz} = |\bar{u}|n_h\left(\frac{mc}{cm^2 \cdot s}\right) \tag{3.12}$$

This integrates to

$$\frac{n_h}{n_{ho}} = \frac{p_h}{p_{ho}} = \exp\left(-\frac{|\bar{u}|z}{D}\right) \tag{3.13}$$

where n_{ho} and p_{ho} are the oil vapor concentration and partial pressure at the backing pump inlet. We can find $\bar{u}$ from

$$\bar{u} = \frac{W}{A} = \frac{Q/p}{\frac{\pi}{4}\phi^2} \tag{3.14}$$

where W = volume flow rate, cm^3/s
A = tube cross-sectional area, cm^2
Q = gas mass flow rate, $Pa{\cdot}cm^3/s$ at 25° C, = sccm × 1837
p = *total* pressure in the tube

As a conservatively high value of D, we take D (Ar-Ar) from Table 2.1. Scaling it for p, we have

$$D\ (cm^2/s) = 0.19\times10^5/p \tag{3.15}$$

Thus, the exponential factor in Eq. (3.13) becomes

$$\frac{\bar{u}z}{D} = 0.123\ Qy/\phi^2 \tag{3.16}$$

for units of sccm and cm. Note that there is no dependence on p, provided that the tube remains in fluid flow. For long, narrow tubes, very little flow is required to attenuate p_h. For example, with 5 sccm flowing through a 2 cm-diameter, 200 cm-long tube, $p_h/p_{ho} = 4\times10^{-14}$.

Thus, foreline and roughing-line purging can be very effective at preventing oil backstreaming through the gas phase. Surface creep still may occur, however, as shown in Fig. 3.4*b*. In critical applications, the only complete solution is to use an all-dry (that is, oil-free) pumping system.

3.4.2 Gas evolution

All materials constantly evolve gases and vapors, a behavior known as outgassing. Consequently, the materials used in thin-film deposition systems must be chosen and treated carefully to minimize contamination from this source. These contaminant gases come both from within the bulk of the material and from its surface, and they enter the process environment at a rate which is little affected by the pressure of the process gases. This means that the same problem exists and the same remedies apply whether one is dealing with UHV evaporation at 10^{-8} Pa or atmospheric-pressure CVD at 10^5 Pa. A few materials that evolve much less gas than others have become generally accepted for thin-film equipment, and these will be highlighted below, following some general comments about surface versus bulk sources of gas.

All surfaces that are exposed to ambient air or handled become covered with contaminants. These consist largely of oil and water, nature's two basic and ubiquitous solvents. The oil comes largely from machining and from fingerprints, and it should be removed with degreasing solvents such as acetone or trichloroethane. The water comes from adsorption of atmospheric moisture. The high polarity of the water molecule causes the first monolayer to bond quite strongly to chemically active surfaces such as metal, glass, and ceramic. After a few monolayers of adsorption, the chemical influence of the substrate dies out, and the water vapor behaves as it does over the liquid. That is, it does not condense if its partial pressure over the surface is lower than its saturation vapor pressure at that T (about 3×10^3 Pa at 25° C). In other words, it does not condense if the relative humidity is < 100 percent. But the first few monolayers do adsorb on the surface, and they stay there until the moisture-containing air is pumped away or purged out with a flow of dry process gas. Then, this water slowly "desorbs" into the process environment. ("Desorb" is the converse of adsorb.)

Although a monolayer does not seem like much, in a film-deposition environment it can be quite significant. A monolayer contains about 10^{15} mc/cm^2 (mc = molecule), so a chamber having 1 m^2 of internal surface area will need to dispose of at least 10^{19} mc of adsorbed water, which is 40 Pa·*l*. Suppose that the room-temperature desorption rate is such that a 100 *l*/s pump can maintain 10^{-4} Pa during the desorption, a typical situation. Since $V \propto 1/p$, the 40 Pa·*l* expands to 4×10^5 *l* at this pressure and will take 4000 s to pump away at 100 l/s. In practice, the desorption rate and water pressure decrease gradually over a longer period of time. Alternatively, consider the same 40 Pa·*l* desorbing into an atmospheric-pressure process-gas flow of 10 slm (= standard liters per minute or atm · l/m). If the desorption rate is such that it adds 1 ppm of water to this flow stream, the desorption rate is 10^{-2} cm^3/m, and it will take 40 m to purge out the monolayer of water. Again, in practice the desorption rate and the ppm level decrease gradually with time. Thus, background water levels on the order of 10^{-4} Pa in a high-vacuum process and 1 ppm in an atmospheric-pressure process are to be expected upon first startup, and in many cases these levels must be reduced considerably before good thin films can be deposited. In the above calculation, we have assumed a surface area equal to the macroscopic geometrical surface area. The microscopic surface area will be much larger if the surface is rough or porous, or if the native oxide on a metal surface is porous or dusty rather than impervious and adherent (see Exercise 3.8). Poorly finished or heavily oxidized metals will adsorb substantially more water.

The desorption rates of water and other surface contaminants can be speeded up by baking, which uses thermal energy to overcome the surface bonding. Desorption rates increase exponentially with T, so 150° C for a few hours is usually sufficient and still stays below the T tolerance limit of Viton, the usual elastomeric sealing material. The same degree of adsorbate removal can be achieved by desorption at room T over a longer period of time, typically a few days. Alternatively, exposure to UV light or to plasma can speed up desorption, but these methods only affect that part of the surface area which is directly exposed, whereas baking reaches into all the nooks and crannies, too. The amount of water introduced upon substrate loading from atmosphere can be reduced considerably by use of the load-lock of Fig. 3.1. An enormous pumping speed for water can be obtained at low cost by the use of a LN_2-cooled shroud within the process chamber (a Meissner trap). The vapor pressure of water at the 77 K temperature of boiling LN_2 is about 10^{-17} Pa (see discussion of Fig. 4.4), so all water that hits the shroud freezes out and stays there. This results in a water pumping speed of 11.6 *l*/s·cm^2, the same value as for orifice conductance [Eq. (3.5)]. Of course, if process gases are being used which also condense at this temperature, such as silane (SiH_4), this approach cannot be employed.

Outgassing contaminants come not only from the surface but also from the bulk of process-containment materials, especially elastomers and other polymers. Elastomers are resilient synthetic rubbers used for gasket seals (O-rings). Viton is a perfluorinated elastomer which has a relatively low solubility for vapors as well as high chemical inertness, and it is the material generally used for vacuum seals. When particularly aggressive reactants are present, the much more expensive Kalrez may be needed. More rigid polymers such as Teflon and Delrin are used for insulators in fixturing. All polymers are particularly susceptible to *ab*sorption of water and solvent vapors into the bulk. This can involve much larger quantities than just *ad*sorption onto surfaces. Prolonged contact with common degreasing solvents should be avoided because of solvent absorption; instead, a quick swipe with a methanol-dampened lint-free cloth is recommended for the degreasing of polymer surfaces.

Because the outside of an O-ring seal is always exposed to atmosphere, it continually absorbs moisture from the air. This water diffuses through the O-ring and outgasses into the process chamber. Since the water is being replenished continually, it never goes away. Consequently, water is always the dominant background gas in a clean, elastomer-sealed vacuum chamber, and it limits the vacuum level to the 10^{-6} Pa range. Elastomers used only on internal seals not exposed to atmosphere eventually do lose their water, so if these are the only elastomer seals present, vacuum levels of $<10^{-8}$ Pa can be attained.

Metals outgas both high-vapor-pressure (p_v) alloying elements and dissolved gases. Common alloying elements to be avoided because of high p_v include Zn (in brass), Cd (in electroplated hardware), Pb and Sb (in soft solder), P (in phosphor-bronze bearings), S and Se (in type 303 stainless steel), and Te. Appendix B gives p_v data for the elements. Stainless steel types 304(L) and 316(L), which are Fe-Ni-Cr alloys, are the most common vacuum metals. They are free of S and Se, nonmagnetic, easily welded, and corrosion-resistant. They form a nonporous Cr oxide surface, and the L-designated versions have low carbon content, which makes them less susceptible to destruction of this protective layer by Cr carbide formation during welding. For parts which need to be heated above 450° C or so, stainless steels are not recommended because of metallurgical degradation and because of volatilization of their small Mn content. Mn evolution from hot stainless steel was a major problem in the early days of GaAs molecular-beam epitaxy, because it forms an electrical-charge-carrier trap in this semiconductor, so ppm concentrations of Mn can destroy the electrical conductivity of GaAs. The refractory (high-melting) metals W, Ta, and Mo are recommended for high-T applications. Among the three, Ta has the advantage of not becoming brittle after incandescent (glowing) heating. W has the highest melting point and lowest p_v of any metal, which is why it is used in light-bulb filaments. For solder-joining or sealing of vacuum equipment, the one low-T alloy which is suitable is the Sn-4 at.% Ag eutectic, which melts at 221° C. Sn has an extremely low p_v despite its low melting point.

Gas evolution from stainless steel consists mainly of CO, followed by H_2, CO_2, and CH_4. These gases come from reactions of dissolved O and H with the C in the steel. Outgassing rate can be reduced to the order of 10^{-10} Pa·*l*/cm·s by baking. Aluminum alloys can have even lower outgassing rates [1], but special surface treatment is required to prevent porous-oxide formation, welding is more difficult, seals are more easily damaged, and Al is subject to attack by elements often encountered in thin-film work, such as Ga and Cl.

Good choices for electrically insulating components in film-deposition systems include quartz, the alkali-free borosilicate glasses such as Pyrex, nonporous grades of silica-alumina ceramic, and pyrolytic boron nitride (PBN). Boron nitride has the valuable advantage of being easily machinable, and PBN is nonporous, unlike the less expensive hot-pressed variety of BN. These inorganic insulators are less gassy but more expensive to fabricate than the polymeric ones mentioned above.

Another source of gas evolution is the "virtual" leak. The one illustrated in Fig. 3.5 was produced by capturing a volume of gas between a substrate and its heater enclosure. The gas in this volume has only crevices through which to pump out, so evacuation will take a long time. Such situations often are misinterpreted as real leaks from atmosphere. The remedy is to provide a pumpout hole into the gas cavity. Another common place for virtual leaks is at the bottom of blind-tapped holes used for mounting internal hardware; here, one should use vented screws, which have holes drilled through them. Even vented cavities can be problematic when running processes in the fluid flow regime, because they act as "dead spots" in the gas flow

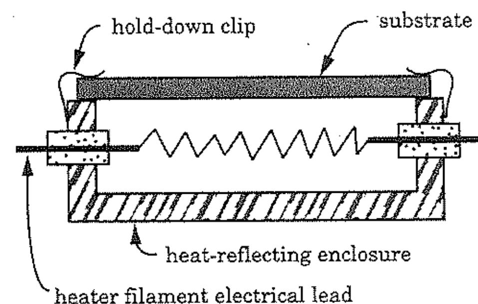

Figure 3.5 Virtual-leak problem caused by poor substrate heater design. Cross section through an enclosed cylindrical cavity is shown.

pattern. For example, if the gas composition in a CVD process is changed at one point to initiate the deposition of a second layer, the first gas will take a while to diffuse out of the cavity and get carried away. While doing so, it will contaminate the deposition of the second layer. If the dead spot cannot be avoided, pressure cycling of the second gas can be used to speed up the rate of gas exchange into the cavity by causing it to "breathe."

3.4.3 Dust

In many thin-film applications, contamination of the substrate surface with even submicron ($<10^{-4}$ cm) particles can destroy the process results. For example, it causes polycrystalline defects in epitaxial layers, electrical shorts between levels of integrated circuitry, and excessive light scattering in optical coatings. Ironically, thin-film deposition systems are copious generators of such particles. This is because films always deposit not only on the substrate of interest but also on other surfaces in the process chamber. The films on these other surfaces eventually begin to flake off (spall) due to a combination of poor adherence and stress buildup. They are also likely to deposit as loose particles because of gas-phase nucleation (more in Sec. 7.3.2). The resulting dust is easily transferred to the substrate before and during deposition. In high-vacuum processes, replaceable shielding around the source can be used to reduce deposition on chamber components. Minimizing T cycling of substrate heaters and of LN_2 shrouding reduces flaking caused by thermal stress. (Film stress is discussed in Sec. 5.6.)

Since pumpdown from atmosphere is very effective at stirring up dust, the load-lock of Fig. 3.1 is a helpful remedy; however, it is not a complete one. Even the load-lock will accumulate some particles by transfer both from atmosphere and from the process chamber. Moreover, while under vacuum, particles of previously deposited film can be *thrown* from surfaces by stress release, and these can reach the substrate even if it is facing downward. They will be held to the substrate by electrostatic forces, which are far stronger than the gravitational pull for such small particles, since they scale with area while the gravity force scales with volume. Also, the velocity of inrushing process gases can release particles and stir them up, even at process pressures of <100 Pa. Consequently, it is often necessary to periodically clean the process chamber of deposits by plasma etching, chemical etching, or mechanical scrubbing. The first technique has the advantage of not requiring disassembly, but it does require that the etching chemistry, reactant gases, and plasma equipment be available. Since larger particles are affected by gravity, it is preferable to deposit onto substrates held vertically whenever possible, so that these particles will fall neither onto the substrate nor into the source of depositing material.

Regardless of whether a load-lock is used, particle stirring can be minimized by "soft" (slow) pumpdown and venting. The conventional criterion for these operations is to avoid turbulence in the gas flow. However, the ease with which particles are lifted off of surfaces scales more closely with the gas *velocity* than with the particular gas flow pattern [2]. This velocity is kept to a minimum by using large-diameter inlet and outlet tubes and by keeping mass flow rates low. Low mass flow rate is especially important on the low-pressure ends of the cycles, where flow velocity is much higher for a given mass flow rate. It is not possible to quantify the maximum tolerable velocity, because this will vary with particle adherence and size as well as with chamber geometry. The best way to assess the situation is to examine the substrate by high-intensity light scattering after a pumpdown/venting cycle to determine the extent of particle accumulation.

When the dust from deposits is toxic, opened chambers should be dealt with cautiously to avoid breathing the dust and contaminating the surroundings. Some materials, such as As, produce a toxic reaction when they are absorbed into the bloodstream through the lung tissue or digestive tract. Others, such as Be and SiO_2, remain in the lungs and over a period of years become encased in scar tissue. Since the scar tissue does not exchange O_2, suffocation eventually results. Wearing of respirators and use of "HEPA"-filtered vacuum cleaners and wet wiping are among the precautions that should be taken in working around toxic dust.

3.5 Pressure Measurement

The above techniques have permitted us to achieve an appropriate level of vacuum to operate a deposition process. It is now necessary to measure the process pressure. In flow processes, it is usually necessary to *control* the pressure as well, by the throttle feedback technique illustrated in Fig. 3.1. Many different vacuum gauges have been developed, but we will discuss only the few most widely used in thin-film work.

The **thermocouple gauge** and the **Pirani gauge** are simple, inexpensive instruments whose operation is based on heat removal by the gas. The thermocouple gauge is illustrated in Fig. 3.6*a*. A constant current, I, is passed through a resistive wire, which heats up. The wire temperature, T, is monitored as a millivolt (mV) reading on a thermocouple attached to it. When no gas is present, the wire reaches a

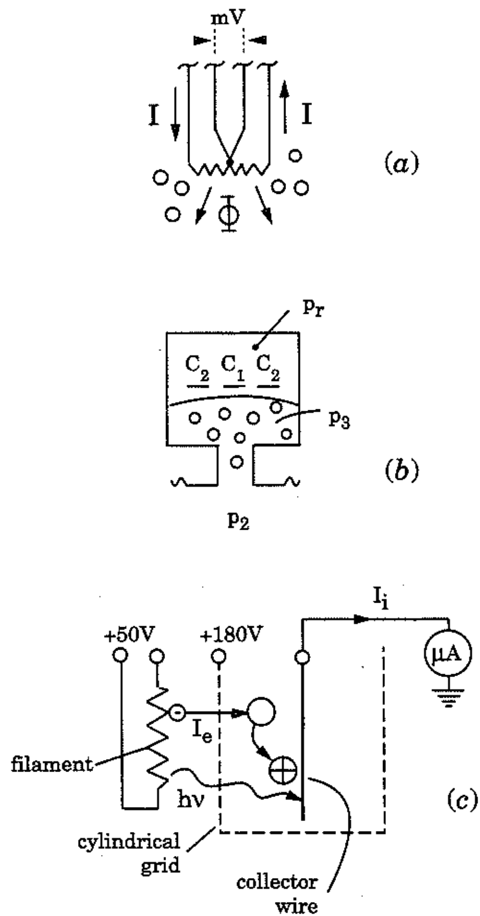

Figure 3.6 Vacuum-gauge operating principles: (*a*) thermocouple gauge, (*b*) capacitance diaphragm gauge, and (*c*) ion gauge.

steady-state T which is determined by radiative heat loss and by heat conduction along the four wires. In the molecular-flow regime, background gas removes additional heat in proportion to its heat-transfer coefficient, h_c, as defined in Eq. (2.32). Since $h_c \propto p$, the wire T drops with increasing p. However, when p rises into the fluid-flow regime, gas heat removal is governed instead by the bulk thermal conductivity, K_T, which is independent of p. From Fig. 2.9, it can be seen that gauge sensitivity is going to die out above 100 Pa or so for reasonable heat-transfer gap lengths. For $p < 0.1$ Pa, sensitivity is again lost because heat transfer through the gas becomes negligible compared to radiative and conductive losses from the wire. The thermocouple gauge also has the disadvantages of nonlinearity, calibration dependence on gas composition, and drift in the zero reading, which is the wire T at zero p.

The best gauge for the above p range is the **capacitance diaphragm gauge** or capacitance "manometer," which is illustrated in Fig. 3.6*b*. Here, a thin metal diaphragm separates a region at pressure p_3 from a sealed-off reference vacuum at $p_r << p_3$. Here, p_3 is the gauge p, which is also referred to in Fig. 3.3. The mechanical force exerted on the diaphragm by the p difference across it deflects it as shown in Fig. 3.6*b* and thus increases the capacitance, C_1, between the diaphragm and a fixed metal disc. The capacitance, C_2, to a fixed metal annulus increases less, and this C difference is converted to p. The use of such a differential measurement is a very effective way of decreasing drift and noise in many instruments. Here, it cancels much of the drift resulting from thermal expansion of the components. This drift can be reduced further by T control of the gauge body. Operating the gauge at an elevated T has the additional advantage of avoiding condensation of process gases which are operating near their saturation vapor pressures.

Residual T drift limits the sensitivity of the capacitance diaphragm gauge to about 10^{-3} Pa, which is actually quite remarkable when one considers that this p corresponds to a diaphragm deflection of less than one atomic diameter! The upper p limit is determined by the strength of the diaphragm or by collapse of the capacitance gap. For a given diaphragm, the dynamic range can be as high as 10^5. The gauge is usually linear to better than 1 percent, and its calibration is independent of gas composition. It is the best choice for reactive gases, because only corrosion-resistant alloys are exposed to the process and because there are no hot wires to decompose these gases. The manufacturer's calibration is usually reliable, but it can be checked against atmospheric (barometric) pressure for high-range gauges. Lower-range gauges then can be calibrated against the high-range one. The one problem with capacitance gauges is zero offset. This offset must be balanced electronically under conditions where p_3 is known to be less than the uncertainty desired in the process p reading. To do this, a pump must be available which can achieve this low a p_3, and when there is any uncertainty about the p achieved, p_3 needs to be verified using a gauge that does not have a significant zero offset, namely the ionization gauge discussed below. When oil is present at the inlet of the pump being used, time spent at low p_3 must be minimized to avoid oil backstreaming.

Still lower pressures are measured by ionizing the gas and measuring the collected ion current I_i, as shown in Fig. 3.6*c*. This is called the Bayard-Alpert or **ion gauge**. Ionization is accomplished by acceleration of electrons emitted from a heated filament. Electron-impact ion-

ization is used in many areas of thin-film technology and is discussed further in Sec. 8.1. The filament is biased at about +50 V so that the electrons will be repelled from the chamber wall, which is at ground potential. The electrons are accelerated toward the cylindrical grid, which is biased at about +180 V. I_i is proportional to the electron emission current, I_e, the ionization cross section, σ_i, and the gas *concentration*, n. I_e is regulated at a specified value, typically 4 mA. The value of σ_i varies with gas composition and electron energy. Small molecules with high ionization potentials have lower σ_i. Helium has the lowest σ_i, at one-sixth that of N_2. The lower p limit of the ionization gauge is determined by the flux of high-energy photons (shown as hν in Fig. 3.6*c*) irradiating the collector wire from the filament; this is called the "x-ray limit." The resulting photoelectrons emitted from the collector produce a residual collector current which is equivalent to about 10^{-9} Pa. The upper p limit of this gauge is reached at about 1 Pa, where the electron mean free path, l_e, becomes too small to allow the electrons to reach the space between the grid and the collector. Between these limits, the gauge is very linear. The hot filament causes considerable outgassing from itself and surrounding materials, and this can make the local pressure, p_3, higher than the chamber pressure, p_2, by as much as 10^{-4} Pa. Most gauge controllers have a provision for degassing the gauge at a higher T than the measuring conditions. If the chamber is to be baked, degassing is most effective if carried out during bakeout.

Since the x-ray limit of an ion gauge is much lower than the sensitivity of a capacitance gauge, the ion gauge can be used to verify the low p_3 that is needed to correct the zero offset error, p_e, of the capacitance gauge. Having made this correction, the capacitance gauge's indicated pressure, p_c, will be the true p. Then, the ion gauge's indicated p, p_i, can be calibrated against p_c for each gas of interest. These procedures are illustrated in Fig. 3.7. A sufficiently sensitive capacitance gauge will be accurate at a low enough p so that the ion gauge

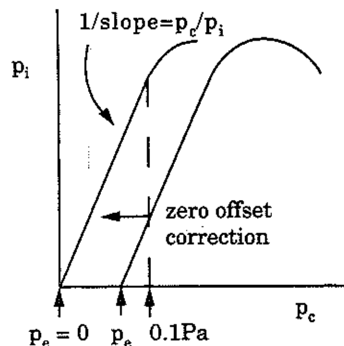

Figure 3.7 Calibration of an ion gauge (p_i) against a capacitance diaphragm gauge (p_c); p_e = zero offset error.

will be linear over some finite overlapping range of the two gauges. Once this range of overlapping linearity has been verified by the Fig. 3.7 plot, the resulting calibration factor, $f = p_c/p_i$, can be used to extrapolate p readings accurately down to the x-ray limit of the ion gauge. Sometimes, ion-gauge calibration factors are provided by the manufacturer, but it is best to verify them by the above procedure when accuracy of better than 50 percent is desired.

A valuable extension of the ion gauge involves separating the ions by mass-to-charge ratio, m/q, in a mass spectrometer. The type of mass spectrometer which uses a quadrupole electrostatic rf field rather than a magnetic field to accomplish the separation is the most convenient and is known as a **residual gas analyzer** (RGA). Examination of the gas spectrum distinguishes readily between water outgassing, pump oil, air leaks, and leaks through faulty process gas valves. Leaks from atmosphere can be located by setting the RGA to He and then blowing He gas around the outside; this is known as a He leak detector. If an *extremely low* flow of He through a fine tube is used, the leak point can be located within 1 mm. The RGA can also be used to monitor process-gas purity. However, for those impurities which also outgas from the RGA itself, such as water and CO, sensitivity is limited. The maximum operating p of the RGA is 10^{-2} Pa, because the mean free path, l_i, of the ions must be long enough for them to pass through the 20 cm-long quadrupole mass filter. Gases at higher p levels can be analyzed by separately (differentially) pumping the RGA and metering the gas into it through an orifice or adjustable leak valve. However, much better sensitivity to impurities in process gases is obtained by first ionizing the gas at the higher p and then extracting the ions into the differentially-pumped mass filter through an orifice, thus avoiding ionization of the impurities which are outgassing from the mass filter and its vacuum chamber. This technique can be extended to **atmospheric-pressure ionization** by using a corona plasma discharge from a high-voltage tip for ionization. Here, *preferential* ionization of the impurities is also obtained when the primary gas has a higher "ionization potential" [defined after Eq. (8.13)] than do the impurities, and then sub-ppb (parts per billion, or one in 10^9) sensitivity is often achieved [3]. This is a powerful technique for monitoring process-gas purity.

With any of the above vacuum gauges, T differences between the gauge (at p_3, T_3) and the process chamber (at p_2, T_2) produce an error in p measurement due to "thermal transpiration" when the gauge and chamber are separated by a restriction or tube as shown in Fig. 3.3 and when Kn > 1. In steady state, the molecular fluxes must be the same going both ways through the tube, and they will be proportional

to the fluxes impinging upon each end of the tube, which are given by Eq. (2.19). This means that

$$\frac{p_3}{\sqrt{MT_3}} = \frac{p_2}{\sqrt{MT_2}} \tag{3.17}$$

or

$$\frac{p_2}{p_3} = \sqrt{\frac{T_2}{T_3}} \tag{3.18}$$

Thus, a gauge operating at 100° C will read 11 percent high in measuring the p of a 25° C high-vacuum chamber. Conversely, a 25° C gauge connected to a 600° C hot-wall CVD reactor will read only 58 percent of the reactor p if the connecting tube is operating at $Kn > 1$. The thermal-transpiration error dies out for $Kn << 1$, because then the p levels equilibrate by the fluid flow which develops in any p gradient. A solution to thermal transpiration has also been derived for the more complicated transition-flow regime [4]. T-stabilized capacitance gauges sometimes come with calibrations already corrected for thermal transpiration (these assume a room-T chamber).

3.6 Conclusion

The choice of vacuum equipment for film deposition depends on the level of vacuum and process cleanliness required and on the type and quantity of gas being pumped. Different deposition processes have widely different operating-pressure ranges, as we will see in subsequent chapters. Whatever the pressure, a good deal of care may be required to achieve a sufficiently clean process environment, and much of vacuum technology addresses this issue. The *tolerable* level of impurities depends on the composition of both the impurities and the material being deposited. Susceptibility of a film to contamination depends largely on its chemical reactivity, as pointed out in Sec. 3.4 for Au versus Al. It also depends on the composition of the contaminant compared to that of the film. For example, water will not contaminate the CVD of SiO_2 from SiH_4 (silane) and O_2, because water is a product of the film-forming reaction anyway. It is best to assess at the outset which contaminants are likely to be especially troublesome and which are of little concern, and then to design the system accordingly. Then, the system must be *operated* carefully to avoid introducing contamination from the atmosphere, backstreaming, outgassing, and dust. One can further reduce film contamination by predepositing chemically-active film constituents on vacuum-chamber surfaces or by purging a CVD system with reactive gases. These procedures serve to "getter" impurities out of the system just prior to deposition. Impurities in the process gases and vacuum chamber can be monitored by mass spectrometry, and impurities in the film can be analyzed by the techniques described later in Sec. 10.2.

3.7 Exercises

3.1 Show that the conductance per unit area of a molecular-flow orifice is 11.6 $l/s \cdot cm^2$ for air at 25° C.

3.2 It is desired to operate a plasma CVD process at 100 Pa with a supply flow of 200 sccm SiH_4. The chamber is being pumped through a 2 m-long, 3 cm-diameter tube. (a) What is the maximum allowable pressure at the pump inlet? (b) What is the minimum speed of the pump?

3.3 The molecular-flow and fluid-flow expressions for tube conductance should give the same value of C somewhere around the middle of the flow transition region. For what value of Kn do Eqs. (3.6) and (3.7) give the same C, using ϕ as the characteristic dimension in Kn?

3.4 A process chamber of 1 m^2 surface area is outgassing at a rate of 10^{13} $mc/cm^2 \cdot s$. For a process-gas flow of 200 sccm and a total pressure of 100 Pa, what is the partial pressure of the outgassing impurities?

3.5 List the advantages of N_2 purging of pumping systems.

3.6 What are the pump choices for a process operating at 10^{-2} Pa, and what are the relative advantages and disadvantages of each?

3.7 A pump foreline 5 cm in diameter and 300 cm long is being purged with Ar. (a) How many sccm of Ar flow are required to attenuate the pump-oil partial pressure by 10^{10} over the length of the tube? (b) What will be the total pressure, p, at the upstream end if the pumping speed is such that p at the downstream end is 50 Pa?

3.8 Suppose that the inside surface of an Al vacuum chamber is coated with 100 nm of poorly formed anodic oxide having 20 percent porosity consisting of 2-nm-diameter cylindrical pores. (a) What is the ratio of the total internal surface area of the pores to the macroscopic area of the Al surface? (b) If two monolayers of water are adsorbed on all of the internal surface area, how many scc are adsorbed per m^2 of macroscopic area?

3.9 How many seconds will it take for a 1 cm^3 virtual leak to evacuate from 10^2 to 10^{-6} Pa through a 10^{-2} cm-diameter, 1 cm-long capillary?