

# EE/Phys5383 – Plasma Technology Class and Lab – Spring 2002

(XX5383 will be the new listing in the upcoming catalog. This is being taught under the following labels: EE4v95, EE7v82, Phys5v49)

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Office Hours:  
Tuesday 1:30 to 3:30  
Wednesday 1:30 to 3:30 (Really ~8:30 to 5 M-F)  
Or by appointment

Prerequisites:  
EE 4301 or equivalent

Class: Room: EC 2.120  
Time: Monday, Wednesday 3:30-4:45  
Final: 2:00 PM Friday, May 10<sup>th</sup>.

Texts:

**Required:**

*A User's Guide to Vacuum Technology*, John F. O'Hanlon, Wiley-Interscience, New York, 1989, ISBN 0-471-81242-0

*Plasma Etching: an Introduction* Edited by D.M Manos and D.L. Flamm, Academic Press, 1989, ISBN 0-12-469370-9

**Recommended:**

*Introduction to Plasma Physics*, F.F. Chen, Plenum, New York, 1974, ISBN 0-306-41332-9

*Principles of Plasma Discharges and Materials Processing*, M.A. Lieberman and A.J. Lichtenberg, Wiley-Interscience, New York, 1994, ISBN 0-471-00577-0

NOTE YOU WILL BE EXPECTED TO LOOK AT OTHER BOOKS AS WELL AS  
THE TWO ASSIGNED

Grading: (dates subject to change)

Test 1	25%	Wed. April 3 <sup>rd</sup> .
Final or project	25%	2:00 PM Friday, May 10 <sup>th</sup> .
<b>Weekly notes</b>	<b>20%</b>	<b>(Rewrite of the week's reading/classnotes)</b>
<b>Homework</b>	<b>30%</b>	

*\*Homework and notes handed in after the due date will not be counted!*

*(They can be slid under my door anytime before 8 AM the next morning.)*

# Approximate course syllabus: (Includes Lab!)

MF-1 = Manos and Flamm chapter 1, JO-3 = J. O'Hanlon chapter 3

Week Number	Lecture Period	Lab Period	Lecture Date	Lab Date	Topic	Reading	Lab Manual Author	Plasma Tool
1	1		14-Jan		Class Overview: What is plasma, where is it found etc. Overview of the subset of plasma technologies to be studied.	MF-1, JO-1,2		
		1		15-Jan	<b>No Lab</b>			
	2		16-Jan		Basic Gas Kinetics/Properties	JO-3		
2			21-Jan		<b>Holiday</b>			
		2		22-Jan	<b>System Overview I: Device ID-ing, Venting, Pump-down, Safety (Includes CR Gowning and procedures overview)</b>		JM/KB/MJG	Indy
	3		23-Jan		Gas Flow	JO-4,5		
3	4		28-Jan		Gas Release and Pressure Measurement	JO-5,6		
		3		29-Jan	<b>Gas Systems I: Using Pressure Measurement Devices</b>		JM	Holly and Maggie
	5		30-Jan		Pressure and Flow Measurements	JO-7		
4	6		4-Feb		Pumping I: Pump Speed	JO-10,11,13		
		4		5-Feb	<b>Gas Systems II: Using Flow Control: Pressure vs. Flow, Leak Rate and Outgassing</b>		GE	Maggie
	7		6-Feb		Pumping II: Pump Operation	JO-19,(1-2), 21		
5	8		11-Feb		High Vacuum and High Flow Systems	JO-16,17		
		5		12-Feb	<b>Gas Systems III: Pumping systems (Turbo-Cryo-Roots-Rotary Vane)</b>		AJC	Indy
	9		13-Feb		Vacuum chamber materials	JO-22		
6	10		18-Feb		Powered materials (Including Wafer Chucks) in plasma chambers	MF-3		
		6		19-Feb	<b>Gas Systems VI: Pressure vs. Conductance</b>		LJO	Cappy
	11		20-Feb		RF Matching I: Power Matching Networks			
7	12		25-Feb		RF Matching II:			
		7		26-Feb	<b>Gas System Diagnostics I: Leak Detection</b>		JM	Indy
	13		27-Feb		Plasma Physics I: Bulk Plasma Properties	TBD		
8	14		4-Mar		Plasma Physics II: Collisions/Distributions	MF-4		
		8		5-Mar	<b>Gas System Diagnostics II/Basic Plasma Properties I: Residual Gas Analyzers and Plasma Chemistry</b>		LJO	Cappy
	15		6-Mar		Plasma Physics III: Sheaths	TBD		
			11-Mar		Spring Break			
			12-Mar		Spring Break			
			13-Mar		Spring Break			
9	16		18-Mar		Plasma Physics IV: Plasma Sources	TBD		
		9		19-Mar	<b>Basic Plasma Properties II: I-V-P Response of Magnetron Plasmas</b>		MJG	Maggie
	17		20-Mar		Plasma Diagnostics I: Langmuir Probes	MF-2		
10	18		25-Mar		Plasma Diagnostics II: Mass, Optical Spectrums			
		10		26-Mar	<b>Basic Plasma Properties III: Optical Emissions</b>		AJC	Holly
	19		27-Mar		Plasma Diagnostics III: RF Probes			
11	20		1-Apr		<b>Catch-up/Review day</b>			
		11		2-Apr	<b>Plasma Generation, Use and Diagnostics I: RF1: Capacitive coupled systems for deposition (Includes Power/Impedance Diagnostics)</b>		LJO	Cappy
	21		3-Apr		<b>*** Exam I ***</b> (Through plasma diagnostics III)			

12	22		8-Apr		Plasma Chemistry I: Etching Mechanisms			
		12		9-Apr	<b>Plasma Generation, Use and Diagnostics II: RF2: Inductively coupled systems for etch (Includes Density and Power Diagnostics)</b>		GE	Indy
	23		10-Apr		Plasma Chemistry II: Silicon and Compounds			
13	24		15-Apr		Plasma Chemistry III: Metals and Oxides			
		13		16-Apr	<b>Plasma Generation, Use and Diagnostics III: DC1: Hollow Cathode systems for propulsion (Includes Langmuir probe Diagnostics)</b>		GE	Holly
	25		17-Apr		Plasma Chemistry III: Metals and Oxides	MF-7		
14	26		22-Apr		Plasma Chemistry IV: Deposition	TBD		
		14		23-Apr	<b>Plasma Generation, Use and Diagnostics IV: DC2: Magnetrons for Sputter Deposition (Includes Raman spectra Diagnostics of films and film thickness)</b>		MJG/AJC	Maggie and Raman
	27		24-Apr		Particulate Contamination	TBD		
15	28		29-Apr		<b>Catch-up/Review day</b>			
		15		30-Apr	<b>Lab Practical?</b>			
	29		1-May		Student Choice or Class Review			
				6-May	Final exam week			
			10-May		<b>Final Exam or Reports due</b>			

## WHAT IS A PLASMA AND WHERE IS IT FOUND?

Often in grade school and high school students are told that there are three states of matter, solid, liquid and gas. These are in some sense differentiated by energy or temperature. Note that temperature has a very special meaning – that is temperature is a measure of random energy as opposed to directed energy. Random energy is where each individual particle is moving with a certain velocity but the average velocity of all particles is zero. Remember velocity is a vector and thus has both a magnitude and direction, while energy is a scalar and only has a magnitude. Directed energy is when the average velocity is not zero. We will discuss this in more detail later. Let's think about the chemical  $H_2O$ . When it is very cold, e.g. has low random energy we get ice or snow. (Note that one can throw a snowball – giving it directed energy but not random energy.) If we put an ice cube on top of a stove and turn on the stove, we turn the  $H_2O$  into water – a liquid. As we continue to heat the  $H_2O$  it boils and gives off steam – a gas. If we were able to continue to push energy into the  $H_2O$

A fourth state of matter, plasma, occurs when we continue to heat a gas. Let us do a gedanken (thought) experiment to see what might happen. First the original gas might be composed of molecules or free atoms or some combination of the two. As we heat the gas we are giving these particles more random energy. At some point some of this translational energy that the particles have gets transferred into internal energy for the particles. This will usually occur through a collision type process. At this point a number of things might occur. 1) The molecules can break up. (High energy usually does not allow the gas molecules to form larger molecules.) 2) An electron will be freed from the particle (atom or molecule). Having the molecules break up is not interesting – we still have a gas, albeit slightly different. For example  $H_2O$  might become  $HO$  and  $H$ . This however is still a gas. On the other hand having an electron escape its orbit can provide something very different. As the electron carries a charge, '-e', the left over part of the particle must also have a charge but of opposite sign, 'e', and it is now known as an 'ion'. If we have only a few of these charged particles in our gas, we do not see much charge in the properties of the system. However, if we continue to heat the gas and increase the number of charged particles eventually we will have enough of them that they interact in a 'collective' electromagnetic manner. At this point we have a plasma. (Note: A few years ago, ~1998, a fifth state of matter was observed in a high energy particle experiment. This state of matter is known as a "quark-gluon plasma". In this case even the standard elementary particles, neutrons and protons, have been shattered. It is thought that this was the state of the very early universe.) Where the precise division is between gas and plasma is an open debate. Often one can see some collective behaviors when as little as 0.0001% – or less! – of the gas particles have been turned into ions. For example candle flames have been shown to be movable with electric fields. This fact was used to design (and patent) an improved fire sprinkler system. Think about this the next time you have a birthday. On the other hand some people claim that 'true' plasmas occur only when all of the particles have been ionized.

Plasmas exist over a huge range of particle densities and particle temperatures (energies). At one extreme in particle density we have the sun, which has a density similar to typical solids or liquids. At the other extreme we have interstellar space, which is almost a perfect vacuum. Ion temperatures may run from room temperature to many Mega °C. Electron temperatures are observed over similar ranges. Note: Different species are not always in thermal equilibrium and hence have different temperatures. This is often true in the plasmas that we will consider here. Figure 1 shows just a few of the possible plasmas and where they fit in the density and energy range.

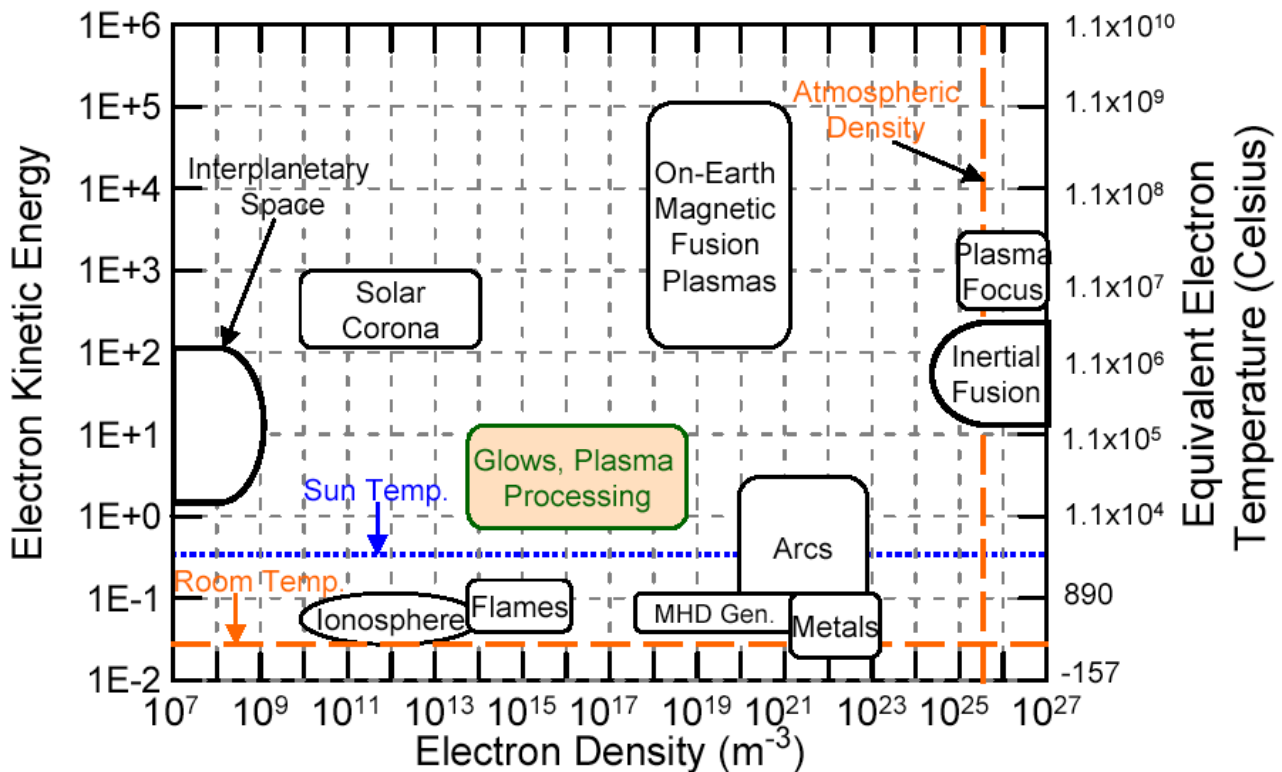


Figure 1: Where different plasmas exist in energy-density space. We will discuss mostly Glows as applied to plasma applications.

In this text we are mostly interested in useful plasmas that we can control and manipulate, which for simplicity we will call ‘laboratory’ or in some cases ‘process’ plasmas. This eliminates all of the extremely high-energy plasmas and most of the non-earth based plasmas. Note that we are still interested in some space plasmas. This is because understanding these plasmas is critical space exploration and satellite communications. (In the accompanying lab one of our plasma systems serves to mimic – as best possible – interplanetary space or to mimic a ‘plasma rocket’.) We will also deal indirectly with plasma-based lighting. However, because of a lack of time, we will limit our studies to plasmas that typically operate at sub-atmospheric pressures. Atmospheric pressure plasmas – also known as thermal plasmas – have additional complexities that are best studied after one has a firm understanding of sub-atmospheric plasmas. While we are only considering a small fraction of all plasma systems, we still have a huge number of plasmas and accompanying processes to study. In addition, understanding these plasmas is critical to continued economic growth. For example, process plasmas are used in approximately 30% of all steps in the production of computer chip. Plasma based lighting greatly reduces energy needs and understanding space plasmas is critical to the telecommunication industry.

To study even this greatly reduced set of plasmas requires knowledge in many fields. ‘Process’ plasmas are studied in multiple engineering (electrical, chemical) and scientific (physics, chemistry, material) departments. Process plasmas, as used in the semiconductor industry, can be further divided into plasma-enhanced chemical-vapor deposition (PECVD) systems, plasma-based physical-vapor deposition (PVD) systems, plasma-ash systems, plasma-etch systems and plasma-based ion implantation (PBII) systems.

While there are great differences between the various plasmas we are going to study, they do have a large number of common facets. First, all of our plasmas operate at low pressures and hence we need to understand vacuum technologies. Second, many of the plasmas have reactive gas or

surface species and hence we need to develop a basic understanding of the chemistry involved. We also need to understand how to flow replacement particles into the system as reactive species are used up. Third, many of our plasmas are used to process a material or surface and thus we need to understand how to hold these surfaces in contact with the plasma. Fourth, we need to understand efficient methods for creating and sustaining the plasmas that are also not detrimental to the material being processed. Finally we need to understand safety and environmental issues that are critical to production process.

## **SAFETY AND ENVIRONMENTAL ISSUES**

Because Laboratory plasmas use many chemistries that are very hazardous, one should learn to read Material Safety Data Sheets (MSDS). For example silane, a common feed gas for PECVD systems, is highly flammable and will easily explode in air if the density is too high. Likewise  $\text{BF}_3$ , which is used in PBII systems, creates gaseous HF when it is in contact with air. If the HF is breathed in, death can occur. An example of an MSDS from Air Liquide (on the gas Silane,  $\text{SiH}_4$ ) is given in below. You can find MSDSs from vendors and they are by law kept on file where they are used. Note that you can also often find them on the web. Before working in a new lab make sure that care has been taken to properly deal with hazardous materials. In addition, because of the dangerous nature of many of these chemistries, **ALL CHEMICAL (GAS, LIQUID OR SOLID) SPILLS SHOULD BE REPORTED IMMEDIATELY.**

In addition to the hazardous chemistries, laboratory plasmas also have dangerous current and voltage sources. It used to be a good rule of thumb that if a power supply was big enough to hurt you if you picked it up, it could kill you with its electrical output. However, because power supply technology has greatly improved, even light weight power supplies can deliver enough energy to kill. In addition an electrical shock may not kill you instantaneously. Instead, the shock disrupts your electrolyte balances in your body and you might die 24 hours later. Before working in a new lab make sure that care has been taken to properly deal with electrical systems. Hence, **ALL ELECTRICAL SHOCKS SHOULD BE REPORTED IMMEDIATELY.**



# MATERIAL SAFETY DATA SHEET

Prepared to U.S. OSHA, CMA, ANSI and Canadian WHMIS Standards

## 1. PRODUCT IDENTIFICATION

**CHEMICAL NAME; CLASS:** SILANE

**SYNONYMS:** Silicon Tetrachloride; Monosilane; Silicane  
**CHEMICAL FAMILY NAME:** Hydride  
**FORMULA:** SiH<sub>4</sub>

**PRODUCT USE:** Document Number: 20148  
 Production of amorphous silicon and silicon substrates; other analytical/synthetic chemical uses.

**SUPPLIER/MANUFACTURER'S NAME:** AIR LIQUIDE AMERICA CORPORATION  
**ADDRESS:** 2700 Post Oak Drive  
 Houston, TX 77056-8229

**EMERGENCY PHONE:** CHEMTREC: 1-800-424-9300

**BUSINESS PHONE:** General MSDS Information 1-713/896-2896  
 Fax on Demand: 1-800/231-1366

## 2. COMPOSITION and INFORMATION ON INGREDIENTS

CHEMICAL NAME	CAS #	mole %	EXPOSURE LIMITS IN AIR						
			ACGIH			OSHA			OTHER
			TLV ppm	STEL ppm	PEL ppm	STEL ppm	IDLH ppm		
Silane	7803-62-5	> 99%	5	NE	5 (Vocated 1989 PEL)	NE	NE	NIOSH REL: 5 ppm	
Maximum Impurities		< 1%	None of the trace impurities in this product contribute significantly to the hazards associated with the product. All hazard information pertinent to this product has been provided in this Material Safety Data Sheet, per the requirements of the OSHA Hazard Communication Standard (29 CFR 1910.1200) and State equivalent standards.						

NE = Not Established C = Ceiling Limit See Section 16 for Definitions of Terms Used.  
 NOTE: All WHMIS required information is included. It is located in appropriate sections based on the ANSI Z400.1-1993 format.

## 3. HAZARD IDENTIFICATION

**EMERGENCY OVERVIEW: PYROPHORIC GAS!** This product is a colorless, air-reactive gas, with a choking effect. This gas usually ignites upon contact with air, releasing a dense white cloud of amorphous silicon dioxide. Silane can react with water to form corrosive silicic acid. The primary health hazard associated with Silane is the potential for severe thermal burns from contact with flames resulting from the spontaneous ignition of this gas. Depending on the severity of the burns, such exposures can be fatal. Flame or high temperature impinging on a localized area of the cylinder of this product can cause the cylinder to burst without activating the cylinder's relief devices. If Silane is released at high pressure or high flow velocity, a delayed detonation may occur. Silane releases which have not spontaneously ignited must be considered extremely dangerous, and should not be approached. Emergency responders must have personal protective equipment and fire protection appropriate for the situation to which they are responding.

**SYMPTOMS OF OVER-EXPOSURE BY ROUTE OF EXPOSURE:** The gas is pyrophoric; over-exposures to the unignited gas would be unlikely under normal circumstances of use. The following symptoms may develop in the event of over-exposure to Silane and its decomposition products:

**INHALATION:** Inhalation of high concentrations of this gas can result in headache, nausea, dizziness, and irritation of the upper respiratory tract. Silane reacts with water to form silicic acid, which can be irritating to the mucous membranes and the respiratory system. Severe Silane over-exposures via inhalation may result in pulmonary edema. Decomposition of Silane will result in the production of amorphous silicon dioxide. Though inhalation of amorphous silicon dioxide can be irritating to the nose and throat, such exposure does not present the potential for adverse health effects as inhalation of crystalline silicon dioxide.

**SKIN and EYE CONTACT:** Silane reacts with water to form silicic acid, which can be irritating to the skin and eyes. Decomposition of Silane will result in the production of amorphous silicon dioxide. Skin or eye contact with particulates of amorphous silicon dioxide may be irritating.

**OTHER POTENTIAL HEALTH EFFECTS:** The chief health hazard presented by Silane is that it ignites so rapidly, personnel in the area of a release can receive severe thermal burns. Depending on the severity of the burns, such exposures can be fatal.

**HEALTH EFFECTS OR RISKS FROM EXPOSURE: An Explanation in Lay Terms.** Over-exposure to Silane may cause the following health effects:

**ACUTE:** The primary health hazard associated with Silane is the potential for severe thermal burns from contact with flames which result from the spontaneous ignition of this gas. Though unlikely to occur under normal circumstances, inhalation of high concentrations of Silane can cause headache, nausea, and irritation of the upper respiratory tract. Reaction of Silane with air or moisture can produce silicic acid and amorphous silicon dioxide; both decomposition products can irritate the eyes, mucous membranes and respiratory tract.

**CHRONIC:** No chronic health effect is currently associated with exposures to Silane.

**TARGET ORGANS:** Respiratory system, skin, eyes.

HAZARDOUS MATERIAL INFORMATION SYSTEM			
HEALTH	(BLUE)		1
FLAMMABILITY	(RED)		4
REACTIVITY	(YELLOW)		3
PROTECTIVE EQUIPMENT		X	
EYES	RESPIRATORY	HANDS	BODY
See Section 8			
For routine industrial applications			

## 4. FIRST-AID MEASURES

**RESCUERS SHOULD NOT ATTEMPT TO RETRIEVE VICTIMS OF EXPOSURE TO THIS PRODUCT WITHOUT ADEQUATE PERSONAL PROTECTIVE EQUIPMENT. At a minimum, Self-Contained Breathing Apparatus and Fire-Retardant equipment should be worn. Adequate fire protection must be provided during rescue situations.**

**NOTE:** Silane releases which have not spontaneously Ignited must be considered extremely dangerous, and should not be approached!

#### 4. FIRST-AID MEASURES (Continued)

Remove victim(s) to fresh air, as quickly as possible. Only trained personnel should administer supplemental oxygen and/or cardio-pulmonary resuscitation, if necessary.

**THERMAL BURNS:** In the event personnel are burned as a result of a Silane release, trained personnel should provide first aid treatment. Get medical attention immediately.

**SKIN EXPOSURE:** If silicic acid (from the reaction of Silane and water) contaminates the skin, immediately begin decontamination with running water. Minimum flushing is for 15 minutes. If amorphous silicon dioxide (from the reaction of Silane with oxygen) contaminates the skin, flush area with copious amounts of water. Remove exposed or contaminated clothing, taking care not to contaminate eyes. Victim must seek medical attention if irritation persists, or if there are other adverse health effects.

**EYE EXPOSURE:** If the decomposition products (silicic acid, amorphous silicon dioxide) contaminate the eyes, immediately begin decontamination with running water, open victim's eyes while under gently running water. Use sufficient force to open eyelids. Have victim "roll" eyes. Minimum flushing is for 15 minutes. Victim must seek medical attention.

Victim(s) must be taken for medical attention. Rescuers should be taken for medical attention, if necessary. Take copy of label and MSDS to physician or other health professional with victim(s).

#### 5. FIRE-FIGHTING MEASURES

**FLASH POINT:** Not applicable. Pyrophoric gas.

**AUTOIGNITION TEMPERATURE:** Not applicable. Pyrophoric gas.

**FLAMMABLE LIMITS (in air by volume, %):**

Lower (LEL): 1.4%  
Upper (UEL): 96%

**FIRE EXTINGUISHING MATERIALS:** Extinguish Silane fires by shutting-off the source of the gas. Use a fine water spray or fog to reduce combustion products formed in air. Do not use halocarbon-type fire extinguishing agents. Cool fire-exposed cylinders with water spray, from the maximum distance possible.

**UNUSUAL FIRE AND EXPLOSION HAZARDS: PYROPHORIC GAS!** This product is a colorless, air-reactive gas. This gas usually ignites upon contact with air, releasing a dense white cloud of amorphous silicon dioxide. The products of thermal decomposition of this material include amorphous silicon dioxide and hydrogen. Silane can react with water to form corrosive silicic acid. The decomposition products of Silane can be irritating to exposed tissue. **If Silane is released at high pressure or high flow velocity, a delayed detonation may occur. Silane releases which have not spontaneously ignited must be considered extremely dangerous, and should not be approached.**

**DANGER!** Fires impinging (direct flame) on the outside surface of unprotected cylinders of this product can be very dangerous. Exposure to fire could cause a catastrophic failure of the cylinder releasing the contents into a fireball and explosion of released gas. The resulting fire and explosion can result in severe equipment damage and personnel injury or death over a large area around the cylinder. For fires in large areas, use unmanned hose holder or monitor nozzles; if this is not possible, withdraw from area and allow fire to burn.

**Explosion Sensitivity to Mechanical Impact:** Not sensitive.

**Explosion Sensitivity to Static Discharge:** Static discharge may cause this product to ignite explosively.

**SPECIAL FIRE-FIGHTING PROCEDURES:** Structural fire-fighters must wear Self-Contained Breathing Apparatus and full protective equipment. If water is not available for cooling or protection of cylinder exposures, evacuate the area. Follow the evacuation guidelines of the North American Emergency Response Guidebook (Guide #116).

#### 6. ACCIDENTAL RELEASE MEASURES

**LEAK RESPONSE:** Evacuate immediate area. Silane is a pyrophoric gas and can ignite spontaneously upon contact with air. Uncontrolled releases should be responded to by trained personnel using pre-planned procedures. Normally, an accidental release of Silane will result in a fire. If Silane is released at high pressure or high flow velocity, a delayed detonation may occur. Silane releases which have not spontaneously ignited must be considered extremely dangerous, and should not be approached. This gas usually ignites upon contact with air, releasing a dense white cloud of amorphous silicon dioxide.

SILANE - SiH<sub>4</sub> MSDS

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#### 7. HANDLING AND USE (Continued)

- Use an alternate vacuum and inert gas purge cycles of the system to purge all air out of the system after it has been leak-tested or opened for any reason.
- Before disconnecting any system that has Silane in it, thoroughly purge the system of Silane with an inert gas. Any portion of a system that is dead-ended or allows "pocketing" of Silane should be treated with considerable vacuum-purge cycles.
- Vent Silane or Silane mixtures through small-diameter pipe or tubing ending under a shallow seal to prevent back diffusion of air. Venting should be to an area designed for Silane disposal, preferably by burning. Concentrations, even in the low percentage range, are dangerous and should not be exposed to air. Silane can also be vented by diluting with an inert gas to prevent ignition upon discharge to the atmosphere.

**SPECIAL PRECAUTIONS FOR HANDLING GAS CYLINDERS:** Compressed gases can present significant safety hazards. The following rules are applicable to work situations in which cylinders are being used:

**Before Use:** Move cylinders with a suitable hand-truck. Do not drag, slide or roll cylinders. Do not drop cylinders or permit them to strike each other. Secure cylinders firmly. Leave the valve protection cap (where provided) in-place until cylinder is ready for use.

**During Use:** Use designated CGA fittings and other support equipment. Do not use adapters. Use piping and equipment adequately designed to withstand pressures to be encountered. Do not heat cylinder by any means to increase the discharge rate of the product from the cylinder. Do not use oils or grease on gas-handling fittings or equipment. Do not "crack" valve open before connecting it, since self-ignition may occur. Leak check system with leak detection solution or other leak detection instruments suitable for Silane, never with flame. Immediately contact the supplier if there are any difficulties associated with operating cylinder valve. Never insert an object (e.g. wrench, screwdriver, pry bar, etc.) into valve cap openings. Doing so may damage valve, causing a leak to occur. Use an adjustable strap wrench to remove over-tight or rusted caps. Never strike an arc on a compressed gas cylinder or make a cylinder part of an electric circuit.

**After Use:** Close main cylinder valve. Valves should be closed tightly. Replace valve protection cap. Mark empty cylinders "EMPTY".

**NOTE:** Use only DOT or ASME code containers designed for flammable gas storage. Earth-ground and bond all lines and equipment associated with this product. Close valve after each use and when empty.

**STANDARD VALVE CONNECTIONS FOR U.S. AND CANADA:** Use the proper connections, **DO NOT USE ADAPTERS.**

**THREADED:** Up to 500 psig - CGA 510

Up to 3000 psig - CGA 350

**PIN-INDEXED YOKES:** Not Applicable.

**ULTRA HIGH INTEGRITY:** Up to 3000 psig - CGA 632

**PROTECTIVE PRACTICES DURING MAINTENANCE OF CONTAMINATED EQUIPMENT:** Follow practices indicated in Section 6 (Accidental Release Measures). Make certain application equipment is locked and tagged-out safely. Purge gas handling equipment with inert gas (i.e. nitrogen) before attempting repairs. Always use product in areas where adequate ventilation is provided.

#### 8. EXPOSURE CONTROLS - PERSONAL PROTECTION

**VENTILATION AND ENGINEERING CONTROLS:** Silane detectors should be installed in or near areas where this product is being used or stored. If appropriate, install automatic monitoring equipment to detect the level of oxygen and the presence of potentially explosive air-gas mixtures. Use with adequate ventilation. Provide natural or explosion-proof ventilation adequate to ensure Silane does reach its lower flammability limit of 1.4%. Local exhaust ventilation is preferred, because it prevents gas dispersion into the work place by eliminating it at its source.

**RESPIRATORY PROTECTION:** Maintain oxygen levels above 19.5% in the workplace. Use supplied air respiratory protection if oxygen levels are below 19.5% or during emergency response to a release of this product. If respiratory protection is required, follow the requirements of the Federal OSHA Respiratory Protection Standard (29 CFR 1910.134), or equivalent State standards.

**EYE PROTECTION:** Safety glasses.

**HAND PROTECTION:** Wear leather gloves when handling cylinders of this product.

**BODY PROTECTION:** Use body protection appropriate for task. Cotton clothing is recommended for use to prevent static electric build-up. Safety shoes are recommended when handling cylinders. Fire retardant clothing may be appropriate under some circumstances of use.

SILANE - SiH<sub>4</sub> MSDS

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#### 6. ACCIDENTAL RELEASE MEASURES

Proper protective equipment should be used. In case of a gas release, clear the affected area, protect people, and respond with trained personnel. Eliminate any possible sources of ignition, and provide maximum explosion-proof ventilation. If the gas is leaking from cylinder or valve, contact the supplier. Adequate fire protection must be provided. Use only non-sparking tools and equipment during the response.

Minimum Personal Protective Equipment should be **Level B: fire-retardant protective clothing, gloves and Self-Contained Breathing Apparatus.** Use only non-sparking tools and equipment. Locate and seal the source of the leaking gas. Protect personnel attempting the shut-off with water-spray. Allow the gas to dissipate.

Monitor the surrounding area for combustible gas levels. The level of Silane must be at acceptable levels (see Section 2, Composition on Information on Ingredients) before personnel can be allowed in the area without Self-Contained Breathing Apparatus. Combustible gas concentration must be below 10% of the LEL (1.4%) prior to enter. Attempt to close the main source valve prior to entering the area. If this does not stop the release (or if it is not possible to reach the valve), allow the gas to release in-place or remove it to a safe area and allow the gas to be released there.

**THIS IS A PYROPHORIC GAS.** Protection of all personnel and the area must be maintained.

#### 7. HANDLING and USE

**WORK PRACTICES AND HYGIENE PRACTICES:** Non-sparking tools should be used. Do not attempt to repair, adjust, or in any other way modify the cylinders containing Silane. If there is a malfunction, or another type of operational problem, contact nearest distributor immediately. Working alone with Silane should be avoided when possible. All work operations should be monitored in such a way that emergency personnel can be immediately contacted in the event of a release.

**STORAGE AND HANDLING PRACTICES:** Cylinders should be stored upright (with valve-protection cap in place) and firmly secured to prevent falling or being knocked over. Cylinders can be stored in the open, but in such cases, should be protected against extremes of weather and from the dampness of the ground to prevent rusting. Cylinders should be stored in dry, well-ventilated areas away from sources of heat, ignition and direct sunlight.

Keep storage area clear of materials which can burn. Do not allow area where cylinders are stored to exceed 52°C (125°F). Store containers away from heavily trafficked areas and emergency exits. Store away from process and production areas, away from elevators, building and room exits or main aisles leading to exits. Protect cylinders against physical damage. Because of the potential for delayed ignition of Silane, storage and use areas should be designed and constructed to protect workers from possible explosive detonation.

Cylinders should be separated from oxygen cylinders, or other oxidizers, by a minimum distance of 20 ft., or by a barrier of non-combustible material at least 5 ft. high, having a fire-resistance rating of at least 0.5 hours. Isolate from other incompatible chemicals, such as strong oxidizers, metals, and metal oxides (refer to Section 10, Stability and Reactivity, for more information).

Storage areas must meet national electrical codes for Class 1 Hazardous Areas. Post "No Smoking or Open Flames" signs in storage or use areas. Consider installation of leak detection and alarm for storage and use areas. Have appropriate extinguishing equipment in the storage area (i.e. sprinkler system, portable fire extinguishers).

Full and empty cylinders should be segregated. Use a first-in, first-out inventory system to prevent full containers from being stored for long periods of time.

Use non-sparking ventilation systems, approved explosion-proof equipment, and appropriate electrical systems. Electrical equipment used in gas-handling operations, or located in storage areas, should be non-sparking or explosion proof. Use a check valve in the discharge line to prevent hazardous backflow. Never tamper with pressure relief devices in valves and cylinders.

**SPECIAL HANDLING PROCEDURES FOR HANDLING SILANE:**

- Do not condense Silane by avoiding temperatures of -149°F (-100°C) or less. Condensation of Silane runs the risk of leaks developing and forming explosive mixtures (due to sucking-back of air into the system).
- Do not use Silane in conjunction with heavy-metal halides or free halogens, with which Silane will react violently. Care should be taken that all handling systems are purged free of halogens that might exist from degreasing agents, or chlorinated hydrocarbons.
- Evacuate and thoroughly pressure-check all systems, preferably with helium, for leaks at pressures two to three times the anticipated working pressure. In addition, regular leak-testing should be instituted and performed.

SILANE - SiH<sub>4</sub> MSDS

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EFFECTIVE DATE: JUNE 1, 1998

#### PHYSICAL and CHEMICAL PROPERTIES

**GAS DENSITY @ 0°C (32°F):** 0.0899 lb/ft<sup>3</sup> (1.44 kg/m<sup>3</sup>)

**BOILING POINT @ 1 atm:** -112°C (-169°F)

**FREEZING/MELTING POINT @ 1 atm:** -184.7°C (-300.5°F)

**SPECIFIC GRAVITY @ 21.1°C (70°F) and 14.7 psia (air = 1):** 1.2

**pH:** Not applicable.

**SOLUBILITY IN WATER @ 20°C (68°F):** Negligible.

**MOLECULAR WEIGHT:** 32.112

**EVAPORATION RATE (nBuAc = 1):** Not applicable.

**EXPANSION RATIO:** Not applicable.

**ODOR THRESHOLD:** Not determined.

**SPECIFIC VOLUME (ft<sup>3</sup>/lb):** 12.0

**VAPOR PRESSURE @ -122.2°C (-188°F) psig:** 7.73

**COEFFICIENT WATER/OIL DISTRIBUTION:** Not applicable.

**APPEARANCE AND COLOR:** Colorless gas, with a choking effect.

**HOW TO DETECT THIS SUBSTANCE (warning properties):** The odor cannot be considered a good warning property, because the odor threshold is above the level necessary for the ignition of Silane. This gas usually ignites upon contact with air, releasing a dense white cloud of amorphous silicon dioxide.

#### 10. STABILITY and REACTIVITY

**STABILITY:** Pyrophoric. Ignites spontaneously on exposure to air.

**DECOMPOSITION PRODUCTS:** Amorphous silicon dioxide and hydrogen.

**MATERIALS WITH WHICH SUBSTANCE IS INCOMPATIBLE:** Silane will react violently with heavy-metal halides and free halogens (i.e. bromine, chlorine, carbonyl chloride, antimony pentachloride, tin(IV) chloride). Silane ignites in oxygen and can react with other oxidizers. Silane is also incompatible with bases.

**HAZARDOUS POLYMERIZATION:** Will not occur.

**CONDITIONS TO AVOID:** Contact with air. Contact with incompatible materials and exposure to heat, sparks and other sources of ignition. If the cylinders are exposed to extremely high temperatures, these cylinders can rupture.

#### 11. TOXICOLOGICAL INFORMATION

**TOXICITY DATA:** The following toxicity data are available for Silane.

Inhalation - Rat: LC<sub>50</sub>: 9600 ppm/4 hours

Inhalation - Mouse: LC<sub>50</sub>: 9600 ppm/4 hours

**SUSPECTED CANCER AGENT:** Silane is not found on the following lists: FEDERAL OSHA Z LIST, NTP, CAL/OSHA, IARC and therefore is not considered to be, nor suspected to be a cancer-causing agent by these agencies.

**IRRITANCY OF PRODUCT:** This decomposition products of Silane are irritating to the eyes, skin, and tissues of the respiratory system.

**SENSITIZATION TO THE PRODUCT:** Silane is not known to be a sensitizer.

**REPRODUCTIVE TOXICITY INFORMATION:** Listed below is information concerning the effects of this product on the human reproductive system.

**Mutagenicity:** No mutagenicity effects have been described for Silane.

**Embryotoxicity:** No embryotoxic effects have been described for Silane.

**Teratogenicity:** No teratogenicity effects have been described for Silane.

**Reproductive Toxicity:** No reproductive toxicity effects have been described for Silane.

A **mutagen** is a chemical which causes permanent changes to genetic material (DNA) such that the changes will propagate through generation lines. An **embryotoxin** is a chemical which causes damage to a developing embryo (i.e. within the first eight weeks of pregnancy in humans), but the damage does not propagate across generational lines. A **teratogen** is a chemical which causes damage to a developing fetus, but the damage does not propagate across generational lines. A **reproductive toxin** is any substance which interferes in any way with the reproductive process.

**MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE:** Skin conditions and respiratory disorders may be aggravated by exposures to Silane and its decomposition products.

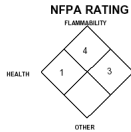
**RECOMMENDATIONS TO PHYSICIANS:** Administer oxygen, if necessary; treat symptoms; eliminate exposure. Be observant for initial signs of pulmonary edema.

**BIOLOGICAL EXPOSURE INDICES (BEIs):** Currently, Biological Exposure Indices (BEIs) are not applicable for Silane.

SILANE - SiH<sub>4</sub> MSDS

PAGE 6 OF 8

EFFECTIVE DATE: JUNE 1, 1998





<p align="center"><b>12. ECOLOGICAL INFORMATION</b></p> <p><b>ENVIRONMENTAL STABILITY:</b> Silane ignites spontaneously upon contact with air, generating amorphous silicon dioxide and hydrogen. Silane, upon contact with water or moisture, will generate silicic acid. All work practices must be directed at eliminating environmental contamination.</p> <p><b>EFFECT OF MATERIAL ON PLANTS or ANIMALS:</b> The primary health hazard associated with Silane is the potential for severe thermal burns to plants and animals from contact with flames which result from the spontaneous ignition of this gas. Depending on the severity of the burns, such exposures can be fatal.</p> <p><b>EFFECT OF CHEMICAL ON AQUATIC LIFE:</b> Silane, upon contact with water or moisture, will generate silicic acid. Silicic acid can lower the pH of water; subsequently, releases of Silane can have an adverse effect on aquatic life in contaminated bodies of water.</p>	<p align="center"><b>15. REGULATORY INFORMATION (Continued)</b></p> <p><b>OTHER CANADIAN REGULATIONS:</b> Silane is categorized as a Controlled Product, Hazard Classes A, B1, B6, and F as per the Controlled Product Regulations.</p> <p><b>STATE REGULATORY INFORMATION:</b> Silane covered under specific State regulations, as denoted below:</p> <table border="0"> <tr> <td>Alaska - Designated Toxic and Hazardous Substances: Silane.</td> <td>Minnesota - List of Hazardous Substances: Silane.</td> <td>Pennsylvania - Hazardous Substance List: Silane.</td> </tr> <tr> <td>California - Permissible Exposure Limits for Chemical Contaminants: Silane.</td> <td>Missouri - Employer Information/Toxic Substance List: Silane.</td> <td>Rhode Island - Hazardous Substance List: None.</td> </tr> <tr> <td>Florida - Substance List: Silane.</td> <td>New Jersey - Right to Know Hazardous Substance List: Silane.</td> <td>Texas - Hazardous Substance List: Silane.</td> </tr> <tr> <td>Illinois - Toxic Substance List: Silane.</td> <td>Kansas - Section 302013 List: None.</td> <td>West Virginia - Hazardous Substance List: None.</td> </tr> <tr> <td>Massachusetts - Substance List: Silane.</td> <td>North Dakota - List of Hazardous Chemicals, Reportable Quantities: None.</td> <td>Wisconsin - Toxic and Hazardous Substances: None.</td> </tr> </table> <p><b>CALIFORNIA PROPOSITION 65:</b> Silane is not on the California Proposition 65 lists.</p>	Alaska - Designated Toxic and Hazardous Substances: Silane.	Minnesota - List of Hazardous Substances: Silane.	Pennsylvania - Hazardous Substance List: Silane.	California - Permissible Exposure Limits for Chemical Contaminants: Silane.	Missouri - Employer Information/Toxic Substance List: Silane.	Rhode Island - Hazardous Substance List: None.	Florida - Substance List: Silane.	New Jersey - Right to Know Hazardous Substance List: Silane.	Texas - Hazardous Substance List: Silane.	Illinois - Toxic Substance List: Silane.	Kansas - Section 302013 List: None.	West Virginia - Hazardous Substance List: None.	Massachusetts - Substance List: Silane.	North Dakota - List of Hazardous Chemicals, Reportable Quantities: None.	Wisconsin - Toxic and Hazardous Substances: None.
Alaska - Designated Toxic and Hazardous Substances: Silane.	Minnesota - List of Hazardous Substances: Silane.	Pennsylvania - Hazardous Substance List: Silane.														
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Massachusetts - Substance List: Silane.	North Dakota - List of Hazardous Chemicals, Reportable Quantities: None.	Wisconsin - Toxic and Hazardous Substances: None.														
<p align="center"><b>13. DISPOSAL CONSIDERATIONS</b></p> <p><b>PREPARING WASTES FOR DISPOSAL:</b> Waste disposal must be in accordance with appropriate Federal, State, and local regulations. Return cylinders with any residual product to Air Liquide. Do not dispose of locally.</p>	<p align="center"><b>16. OTHER INFORMATION</b></p> <p><b>MIXTURES:</b> When two or more gases or liquefied gases are mixed, their hazardous properties may combine to create additional, unexpected hazards. Obtain and evaluate the safety information for each component before you produce the mixture. Consult an Industrial Hygienist or other trained person when you make your safety evaluation of the end product. Remember, gases and liquids have properties which can cause serious injury or death.</p> <p>Further information can be Silane found in the following pamphlets published by: Compressed Gas Association Inc. (CGA), 1725 Jefferson Davis Highway, Suite 1004, Arlington, VA 22202-4102. Telephone: (703) 412-0900.</p> <p>P-1 "Safe Handling of Compressed Gases in Containers" AV-1 "Safe Handling and Storage of Compressed Gases" "Handbook of Compressed Gases"</p>															
<p align="center"><b>14. TRANSPORTATION INFORMATION</b></p> <p><b>THIS MATERIAL IS HAZARDOUS AS DEFINED BY 49 CFR 172.101 BY THE U.S. DEPARTMENT OF TRANSPORTATION.</b></p> <p><b>PROPER SHIPPING NAME:</b> Silane <b>HAZARD CLASS NUMBER and DESCRIPTION:</b> 2.1 <b>UN IDENTIFICATION NUMBER:</b> UN 2203 <b>PACKING GROUP:</b> Not applicable. <b>DOT LABEL(S) REQUIRED:</b> Flammable Gas <b>NORTH AMERICAN EMERGENCY RESPONSE GUIDEBOOK NUMBER (1996):</b> 116</p> <p><b>MARINE POLLUTANT:</b> Silane is not classified by the DOT as a Marine Pollutant (as defined by 49 CFR 172.101, Appendix B).</p> <p><b>SPECIAL SHIPPING INFORMATION:</b> Cylinders should be transported in a secure position, in a well-ventilated vehicle. The transportation of compressed gas cylinders in automobiles or in closed-body vehicles present serious safety hazards and should be discouraged.</p> <p><b>NOTE:</b> Shipment of compressed gas cylinders which have not been filled with the owners consent is a violation of Federal law (49 CFR, Part 173.301 (b)).</p> <p><b>TRANSPORT CANADA TRANSPORTATION OF DANGEROUS GOODS REGULATIONS:</b> THIS MATERIAL IS CONSIDERED AS DANGEROUS GOODS. Use the above information for the preparation of Canadian Shipments.</p>	<p align="center"><b>15. REGULATORY INFORMATION</b></p> <p><b>SARA REPORTING REQUIREMENTS:</b> Silane is not subject to the reporting requirements of Sections 302, 304 and 313 of Title III of the Superfund Amendments and Reauthorization Act.</p> <p><b>SARA THRESHOLD PLANNING QUANTITY:</b> Not applicable.</p> <p><b>TSCA INVENTORY STATUS:</b> Silane is listed on the TSCA Inventory.</p> <p><b>CERCLA REPORTABLE QUANTITY (RQ):</b> Not applicable.</p> <p><b>OTHER U.S. FEDERAL REGULATIONS:</b></p> <ul style="list-style-type: none"> <li>Silane is not subject to the reporting requirements of CFR 29 1910.1000.</li> <li>Depending on specific operations involving the use of Silane, the regulations of the Process Safety Management of Highly Hazardous Chemicals may be applicable (29 CFR 1910.119). Silane is not listed in Appendix A of this regulation, however, any process that involves a flammable gas on-site, in one location, in quantities of 10,000 lbs (4,553 kg) or greater is covered under this regulation unless it is used as a fuel.</li> <li>Silane does not contain any Class I or Class II ozone depleting chemicals (40 CFR part 82).</li> <li>Silane is listed under Table 3 as a Regulated Substance, per 40 CFR, Part 68, of the Risk Management for Chemical Release Prevention, as a flammable substance. The threshold quantity for Silane under this regulation is 10,000 lbs.</li> <li>Silane is listed under Section 112(r) of the Clean Air Act. The threshold quantity for Silane under this regulation is 10,000 lbs.</li> </ul>															
<p>SILANE - SIH, MSDS</p> <p align="center">PAGE 7 OF 8</p> <p align="right">EFFECTIVE DATE: JUNE 1, 1998</p>	<p align="center"><b>AIR LIQUIDE</b></p> <p><small>This Material Safety Data Sheet is offered pursuant to OSHA's Hazard Communication Standard, 29 CFR, 1910.1200. Other government regulations must be reviewed for applicability to this product. To the best of Air Liquide America Corporation's knowledge, the information contained herein is reliable and accurate as of this date; however, accuracy, suitability or completeness are not guaranteed and no warranties of any type, either express or implied, are provided. The information contained herein relates only to this specific product. If this product is combined with other materials, all component properties must be considered. Data may be changed from time to time. Be sure to consult the latest edition.</small></p> <p>SILANE - SIH, MSDS</p> <p align="center">PAGE 8 OF 8</p> <p align="right">EFFECTIVE DATE: JUNE 1, 1998</p>															

Figure 2: A typical MSDS as prescribed by US law. Note other countries have different forms. This MSDS was pulled from the US version of Air Liquide (a French company). Other gas and chemical commonly have these forms online. In addition, if you have a chemical in a factory/building you are required by law to have copies of the MSDS that available to everyone. (Note UTD has them in the gowning area of the cleanroom as well as elsewhere.)

The final issue in this area is related to 'safety' for the item being produced. Often these items are microscopic in nature and thus small particulate can damage or destroy the item. Because of this plasma systems are often found in 'cleanrooms.' The cleanliness of the cleanroom is given in "class 'some number'" or particulate per cubic meter. (UTD's cleanroom typically operates from class one in the lithography areas to class 1000 in the teaching lab areas.) These cleanrooms are rooms with special air filtration (Hepa-filters) systems. In addition special clothing and gloving, known as gowning, is often required to maintain this cleanliness around a person working in the cleanroom. (The human body and most clothing shed dust.) Before entering any cleanroom first learn and then practice the required gowning techniques. The gowning level is determined by the product, the number of filters, the air-flow speed and the number of workers and systems in the cleanroom. **Add figure of how a cleanroom runs.**

## CREATING PLASMAS

**Aspects of Plasma Processing:**  
**A brief overview of plasma science in industry**

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Plasma Laboratories

Matthew J. Goeckner

**Outline**

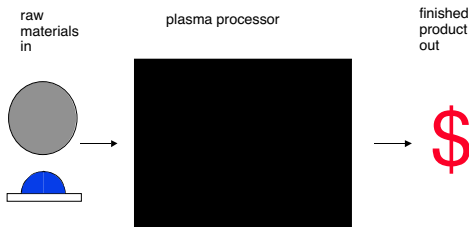
- 1) Why study plasma processing?
- 2) Diagnostic tools used to study processes
- 3) Overview of some plasma processes
- 4) Overview of some processing discharges
- 5) Opportunities in plasma processing

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Matthew J. Goeckner

**How some view plasma processing**

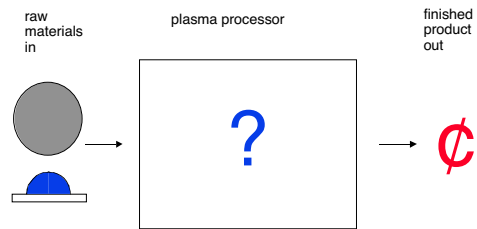


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**How plasma processing really works**



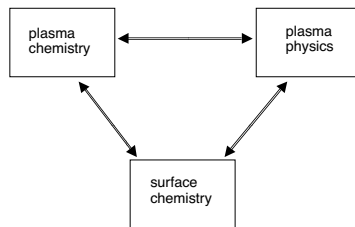
One desires to understand the process so as to improve the value of finished product

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**Plasma processing**



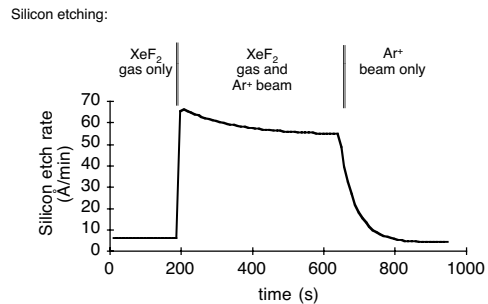
Every process is a complex interaction between  
 • gas phase chemistry  
 • plasma conditions  
 • surface phase chemistry/conditions

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**Classic example**



Work of Coburn and Winters in "Glow Discharge Processes" by Chapman (Wiley, 1980) p317

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**Plasma Physics**

The basic plasma physics can be examined using probes

Langmuir probes give:  
 •  $n_e$ ,  $T_e$ ,  $V_{pi}$  &  $V_{ti}$

**Probes are quick and simple and give general information**

Certain details can only be obtained with more exotic diagnostic techniques

- laser-induced fluorescence,
- e-beam
- $\mu$ wave interferometer, etc.

**These other techniques are more difficult**

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## Gas and Surface phase chemistry

To fully understand plasma processing one must understand the plasma and surface chemistry

The chemistry can be examined with the following

- FTIR spectroscopy
- absorption spectroscopy
- microwave spectroscopy
- optical emission spectroscopy
- laser-induced fluorescence
- mass spectrometry
- ellipsometry
- etc.

Each has advantages and disadvantages

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## Outline

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## General Types of Plasma Processes

- 1) Etching
- 2) Chemical Vapor Deposition (CVD)
- 3) Sputtering/Physical Vapor Deposition (PVD)
- 4) Implantation
- 5) Sprays
- 6) Chemical Production/Destruction
- 7) Medical Sterilization (Johnson & Johnson)
- 8) etc.

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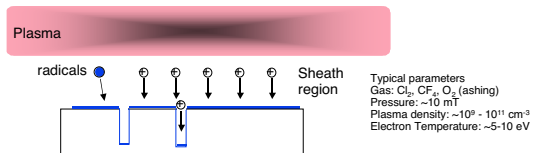
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## Plasma Assisted Etching

20 to 30 years ago most etching was "wet" chemistry

EPA and industrial requirements have almost reversed this



Radicals produced in the plasma will drift to the surface

Ions accelerated across the sheath deliver energy, driving the chemical reaction(s) between the radicals and the surface material

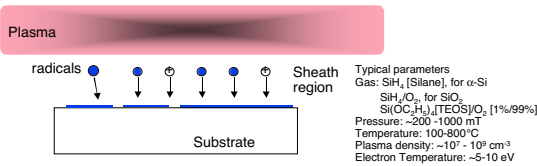
The resulting molecules leave in gaseous form

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## Plasma Assisted Chemical Vapor Deposition (PCVD)



Radicals produced in the plasma and the supplied feed gas drift to the surface

The radicals do not chemically react with the substrate

Instead the radicals combine to form stable chemicals (Solids!)

Ions accelerated across the sheath deliver energy that tends to "cross-link" these chemical bonds

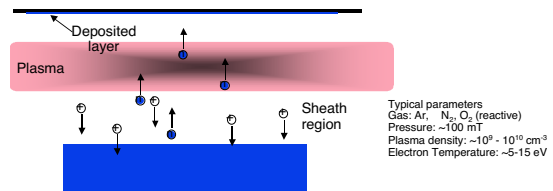
Growth pattern is very complex

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## Sputtering



Ions are accelerated into target

Some of the surface atoms are sputtered off of the target.

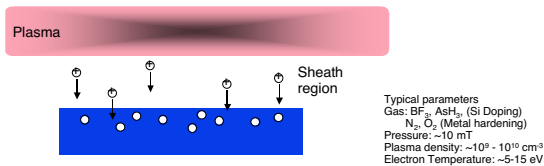
These sputtered atoms "flow" across the chamber to where they are deposited

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## Implantation



Ions are accelerated (Typically in a pulsed mode)

Upon impact, they drive deep into the cathode, where they are trapped

These implanted ions change the surface structure

This results in a change of the surface characteristics (Hardness, friction, wear resistance, etc)

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## Outline

- 1) Why study plasma processing?
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### General types of processing discharges

- 1) DC Glow
  - Cold Cathode
  - Hot Cathode ('Filament' discharge)
  - Magnetron (Magnetized cold cathode)
- 2) Radio Frequency (~0.1 - 100 MHz)
  - Capacitively Coupled (rf)
  - Inductively Coupled Plasma (ICP)
  - Helicon (Magnetically enhanced wave coupling)
- 3) Microwave (~1 - 20 GHz)
  - Microwave
  - Electron Cyclotron Resonance (ECR)
  - (Magnetically enhanced wave coupling)
- 7) Neutral Beams
- 8) Thermal Plasmas
  - Arcs
  - Torches
- 9) etc.

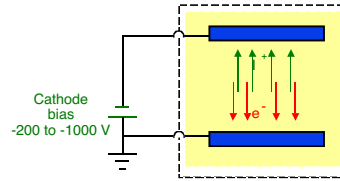
The choice of source depends on the desired process.  
We will look at some of the major sources for Si Processing (and a few others).

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### Cold Cathode (DC/ Pulsed DC discharge)



Ions are accelerated to the cathode

~10% of the impacts produce a secondary electron

These secondary electrons are accelerated back across the plasma  
Impact with neutrals produces additional ion/electron pairs which sustains the discharge

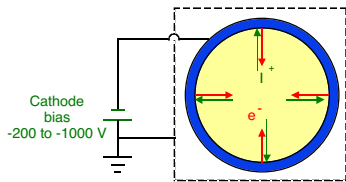
Pulsed version used for Plasma based ion implantation

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### Hollow Cathode (DC/ Pulsed)



Hollow cathode are a variant of the planar cold cathode

Major advantage is that the electrons are better confined

Result is a denser plasma

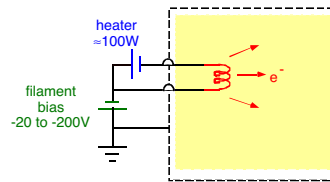
Pulsed version used for Plasma based ion implantation  
Patent pending (Goeckner et al.)

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### Hot DC Cathode (Filament discharge)



A heating current is drawn through a filament  
(Typically Thoriated Tungsten - Looks like a light bulb)

At about 1800°C the filament emits electrons

A second power supply is used to accelerate the electrons off of the filament

These energetic electrons ionize the local neutral gas

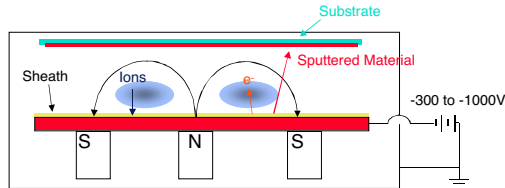
Used for standard ion implantation and Plasma based ion implantation

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### Sputtering Magnetrons (Magnetized cold cathodes)



The sputtering process  
Ions accelerated across sheath to surface  
material sputtered  
secondary electrons produced

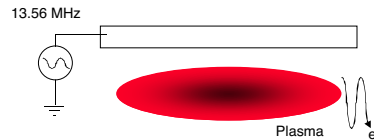
The plasma source  
Secondary, created by ion bombardment of the cathode  
are trapped between the sheath and B field and produce more ions

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### Radio Frequency (RF) Plasmas (Capacitively coupled)



The RF signal is used to setup a time varying electric field  
between the plasma and the electrode

This electric field accelerates the electrons in and out of the plasma

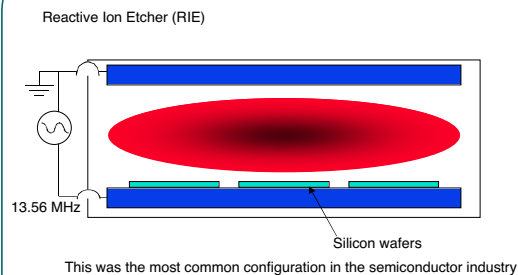
The electrons gain energy and ionize the local gas

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### Example configuration of an RF discharge



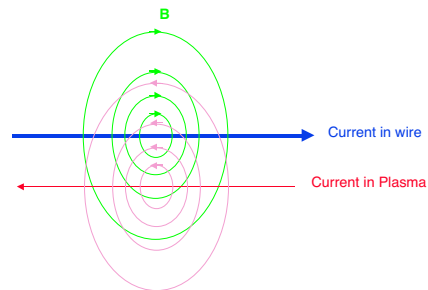
This was the most common configuration in the semiconductor industry

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### Inductively coupled plasmas (ICP)



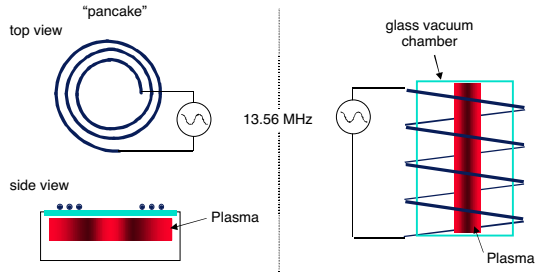
Other Names: Radio frequency inductive (RFI) &  
Transformer coupled plasmas (TCP)

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### Example antenna configurations for ICP discharges



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### Helicon Discharges



The antenna is used to launch Helicon waves.

Helicon waves can be excited over a range of frequencies  $f$

$$f_{ci} \ll f_1 \ll f \ll f_{ce} \ll f_{pe}$$

Typically  $f \approx 7$  to  $10$  MHz

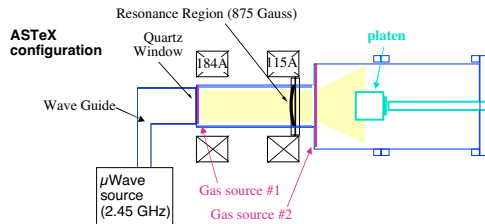
Currently Helicon discharges are being evaluated in basic physics experiments (This will change soon?)

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### Electron Cyclotron Resonance (ECR) discharges



In a magnetic field the electrons resonate at the cyclotron frequency

$$f = eB / 2\pi m_e c = 2.80 \times 10^6 \text{ B Hz}$$

When in resonance with the  $\mu$ Waves, the electrons absorb energy

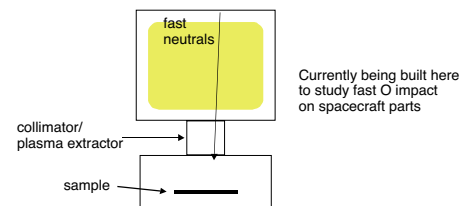
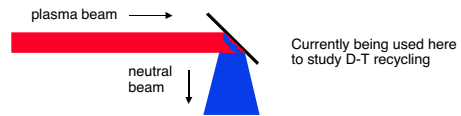
These energized electrons ionize the local neutral gas

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### Neutral beam sources



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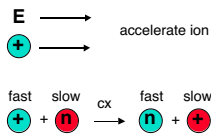
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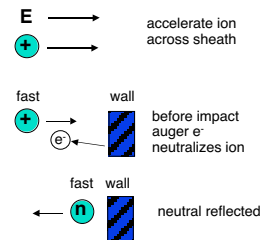
### Neutral beams

#### Methods of producing fast neutrals

#### Charge exchange



#### Wall neutralization

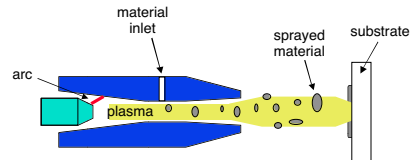


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### Thermal plasma sprays



Sprayed material is "melted" by plasma

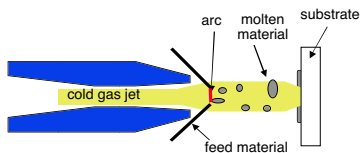
Thermals

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### Thermal arc sprays



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### Is Plasma Science Physics, Engineering or Chemistry?

The simple answer is: A lot of physics, engineering and chemistry.

The typical process plasma is not well understood.

Until recently the typical process plasma was "tweaked" to make it work.

Because of increasing demands on industry there is a push to understand why a process works, e.g. physics and chemistry.

This knowledge is then used to see how the process can be improved, e.g. engineering

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Outline	Opportunities in Plasma Processing
1) Why study plasma processing? 2) Diagnostic tools used to study processes 3) Overview of some plasma processes 4) Overview of some processing discharges 5) Opportunities in plasma processing	Are there opportunities in plasma processing? Yes. While not limitless there are opportunities. Academic: There appears to be a shift toward more applied physics. National Labs have built strong plasma processing groups Leaders are: Sandia & Los Alamos Research Universities are hiring 3 to 6 Profs/Year (Phys. & Eng.) Small Universities like Plasma Processing Relatively low \$ to run and easy to involve students Industrial: Over the last few years hiring has increased dramatically Some "fresh" PhDs are getting ~10 interviews and 3-4 job offers. (Note the market place does change!)

## CREATING AND SUSTAINING PLASMA

In each of the plasma sources discussed above, the plasma is sustained by heating electrons which in turn collide with neutrals to produce new ions and electrons.

In general electrons have larger velocities and are more energetic than ions. We can determine this from simple freshman physics. Let us first assume that the electron energy is the same as the neutrals and the ions. Thus we will assume that

$$\mathcal{E}_e = \mathcal{E}_i = \mathcal{E}_n$$

where e, i and n stand for electron, ion and neutral respectively. Rewriting this in terms of the velocity, we find that

$$\frac{1}{2} m_e v_e^2 = \frac{1}{2} m_i v_i^2 = \frac{1}{2} m_n v_n^2$$

but

$$m_e \ll m_i \approx m_n$$

so that

$$|v_e| \gg |v_i| \approx |v_n|$$

The exact energy balance is determined by the energy gains and losses for each of the species. Let us first consider the gains. Most of the heating is through an electric field – or in some cases an electromagnetic field. If we assume that the heating is just an electric field then

$$\mathbf{F} = q\mathbf{E} = m_e \frac{dv_e}{dt} = m_i \frac{dv_i}{dt}$$

Again because of the mass ratio, the electrons accelerate much faster than the ions. Thus the electrons gain energy at a much faster rate than the ions. (The neutrals are not accelerated and do not directly gain energy – they can however gain energy indirectly from energy transfer with ions.) Now we need to consider energy loss. Energy is lost mostly through collisions. Collisions can fall into three categories,

- 1) elastic
- 2) inelastic
  - a. excitation
  - b. ionization

Elastic collisions are those in which kinetic energy is maintained – sort of like pool balls colliding. Inelastic collisions transfer some of the initial kinetic energy into internal energy of the system – sort of like cars colliding. Often the most common type of collision for the charged species is an elastic collision with a neutral. In such a case we can calculate from conservation of energy

( $\frac{1}{2} m_{e/i} v_{e/i \text{ init}}^2 + \frac{1}{2} m_n v_n^2 \text{ init} = \frac{1}{2} m_{e/i} v_{e/i \text{ final}}^2 + \frac{1}{2} m_n v_n^2 \text{ final}$ ) and conservation of momentum

$(m_{e/i} \mathbf{v}_{e/i \text{ init}} + m_n \mathbf{v}_{n \text{ init}} = m_{e/i} \mathbf{v}_{e/i \text{ final}} + m_n \mathbf{v}_{n \text{ final}})$  that the energy transferred from an electron/ion is given by

$$\Delta \mathcal{E}_{e/i} = \frac{2m_{e/i}m_n}{(m_{e/i} + m_n)^2} \mathcal{E}_{e/i}$$

A quick inspection reveals that the electrons lose energy at a rate

$$\Delta \mathcal{E}_e \approx \frac{2m_e}{m_n} \mathcal{E}_e \approx \frac{1}{10000} \mathcal{E}_e$$

while ions lose energy at a rate

$$\Delta \mathcal{E}_i = \frac{2m_i m_n}{(m_i + m_n)^2} \mathcal{E}_i \approx \frac{2m_n m_n}{(2m_n)^2} \mathcal{E}_i \approx \frac{1}{2} \mathcal{E}_i.$$

The ion energy loss rate is obviously much larger than that for the electrons. Thus we have found that the electrons gain energy much faster and lose it much slower than the ions. This gives us

$$\mathcal{E}_e \gg \mathcal{E}_i \geq \mathcal{E}_n$$

Typical values are:

$$\mathcal{E}_e: 5-10 \text{ eV}$$

$$\mathcal{E}_i: 0.05 \text{ eV}$$

$$\mathcal{E}_n: 0.025 \text{ eV (Room temperature)}$$

## **VACUUM TECHNOLOGY OVERVIEW.**

The plasmas that we are studying operate under a variety of pressures ranging from ‘low’ vacuum to ‘ultra high.’ Note that low is closest to atmospheric pressure while ultra high is the closest to perfect vacuum that can be reached. This is, for some people, backwards to what they might originally guess. The typical definitions are given in the table below.

	Torr	Pascals	Particle Density (#/cm <sup>3</sup> )
Low vacuum	760-23	10 <sup>5</sup> -3×10 <sup>3</sup>	2.5×10 <sup>19</sup> -7.5×10 <sup>17</sup>
Medium	23-10 <sup>-3</sup>	3×10 <sup>3</sup> -10 <sup>-1</sup>	7.5×10 <sup>17</sup> -2.5×10 <sup>13</sup>
High	10 <sup>-3</sup> -10 <sup>-6</sup>	10 <sup>-1</sup> -10 <sup>-4</sup>	2.5×10 <sup>13</sup> -2.5×10 <sup>10</sup>
Very high	10 <sup>-6</sup> -10 <sup>-9</sup>	10 <sup>-4</sup> -10 <sup>-7</sup>	2.5×10 <sup>10</sup> -2.5×10 <sup>7</sup>
Ultra high	10 <sup>-9</sup> -10 <sup>-12</sup>	10 <sup>-7</sup> -10 <sup>-10</sup>	2.5×10 <sup>7</sup> -2.5×10 <sup>4</sup>

$$1 \text{ atmosphere} = 760 \text{ Torr (mm Hg)} = 101323 \text{ Pa} = 2.5 \times 10^{19} \text{ at STP (Loshmidt's Number)}$$

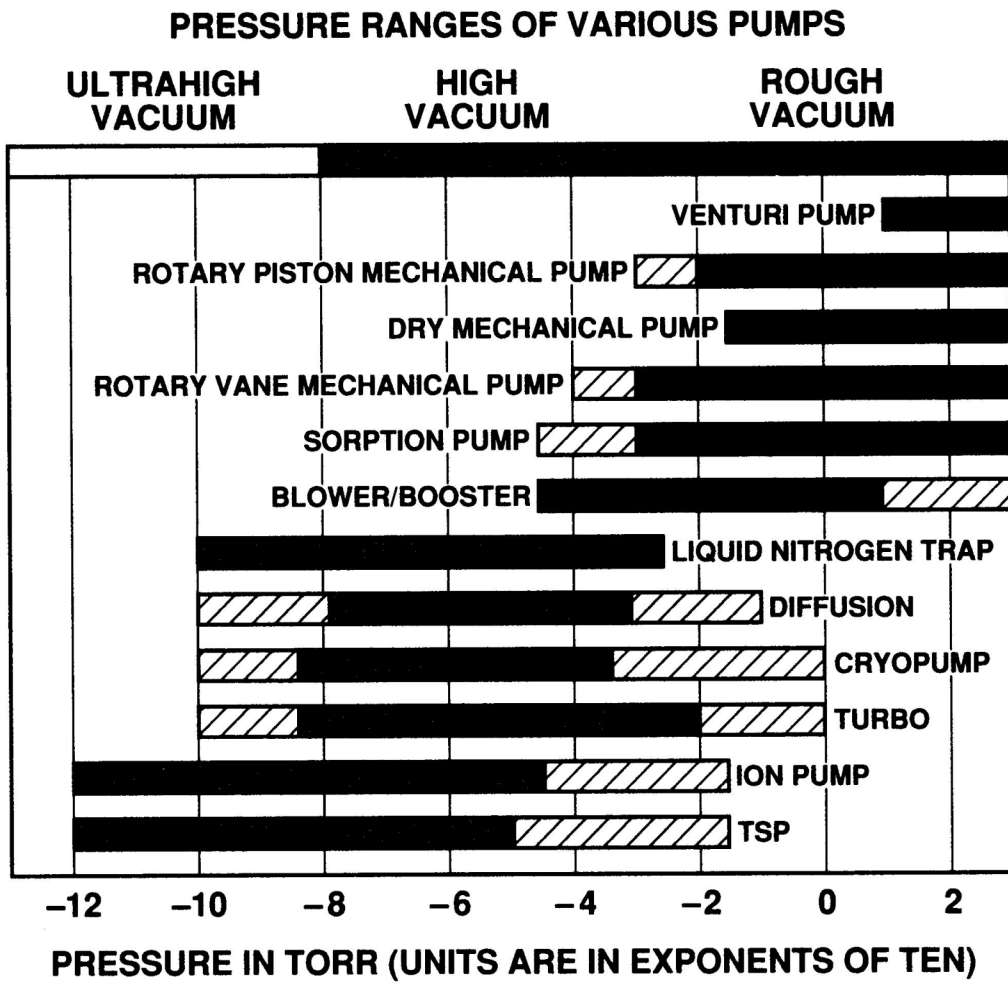
These numbers provide an effective method for describing two important issues in process plasmas, 1) how often a given particle collides with another and 2) how often a given particle collides with a surface or wall. This last item can be turned around to look at 3) how often a given spot on a surface gets hit by a particle (or how often the wall ‘collides’ with an atom). These three issues are important for the following reasons. 1) gives how often a particle interacts with other particles and can be used to determine how far it travels between collisions, the ‘mean free path’. In a similar manner 2) gives how often a particle interacts with the wall. For the more massive particles (ions and molecules) these collision cause energy transfer and hence help set the temperature of the particles. Thus comparing 1) and 2) will allow us to understand some of the energy transfer that occurs in plasmas. Typically particles in a low vacuum system will interact with

other particles much more than with the wall, while in an ultra high vacuum system, particles will interact mostly with the walls. Finally 3) gives how fast a monolayer might form on a surface.

We can achieve these various pressure regimes using an assortment of pumping and vacuum sealant systems.

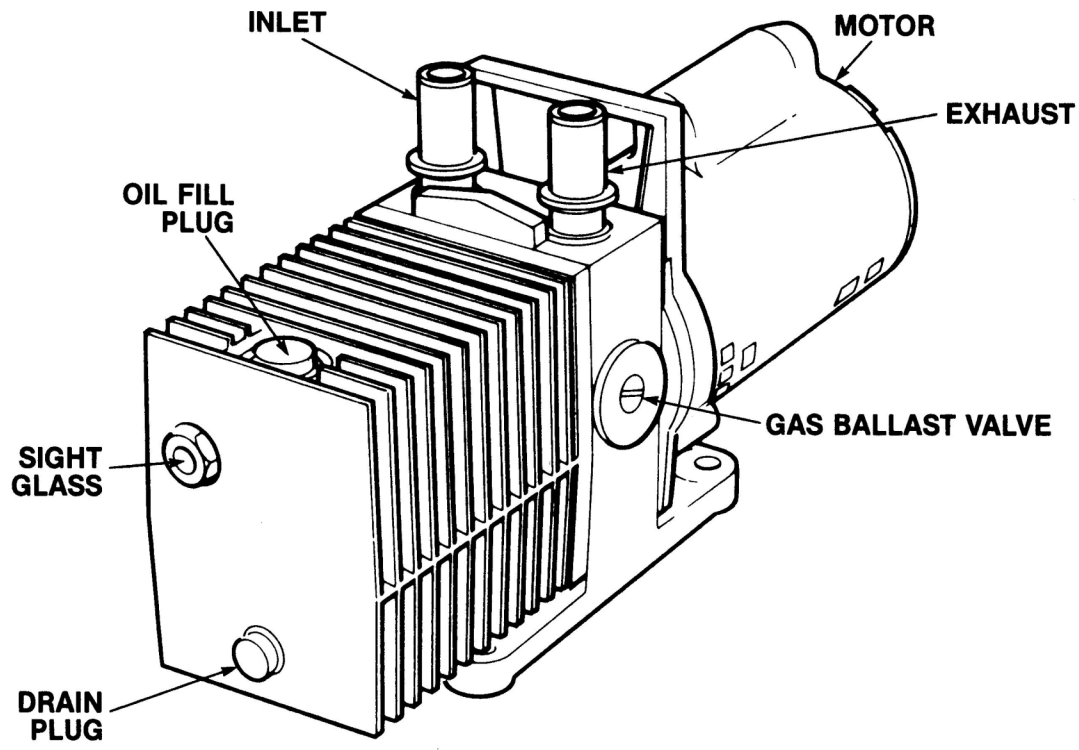
Pump types:

- 1) Rotary vane and piston pumps
- 2) Roots blowers
- 3) Ion pumps
- 4) Sorption pumps and cryo-pumps
- 5) Diffusion pumps
- 6) Turbomolecular pumps ('TMP' or 'turbo')

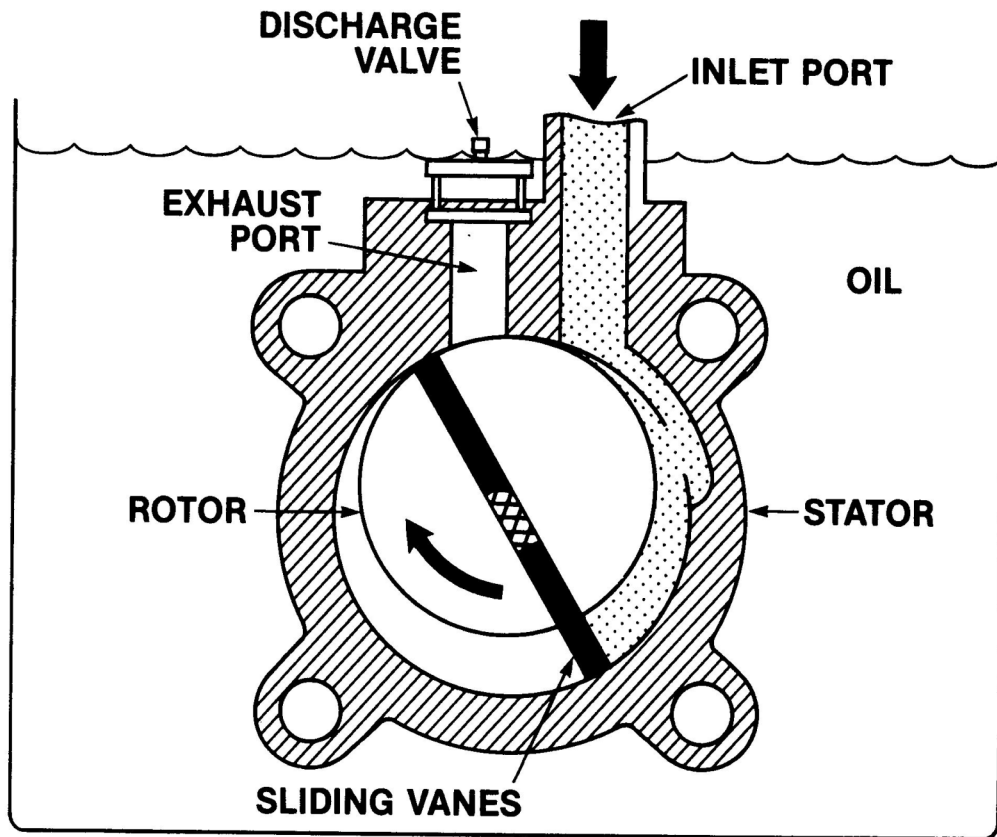


Typical pump combinations used for different pressure ranges. Ultrahigh runs from 1E-12 Torr to ~1E-8 Torr. High vacuum runs from ~1E-8 Torr to 1E-3 Torr. Rough vacuum runs from 1E-3 Torr to 760 Torr or ATM. (Figure from Basic Vacuum Practice, Varian Vacuum)



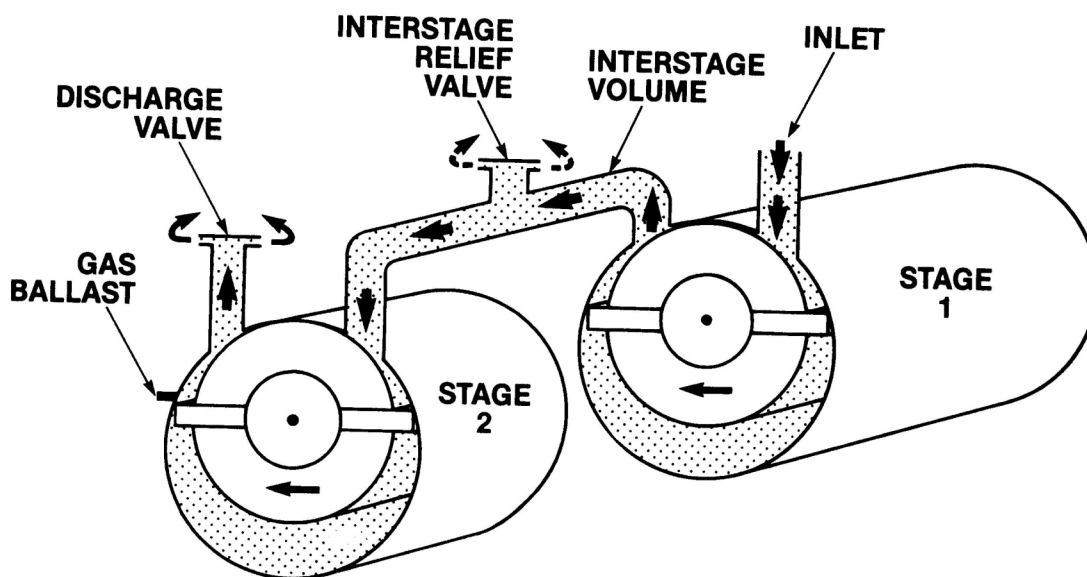


Typical housing for a 'wet' rough pump that is of the rotary mechanical pump type. (Figure from Basic Vacuum Practice, Varian Vacuum)



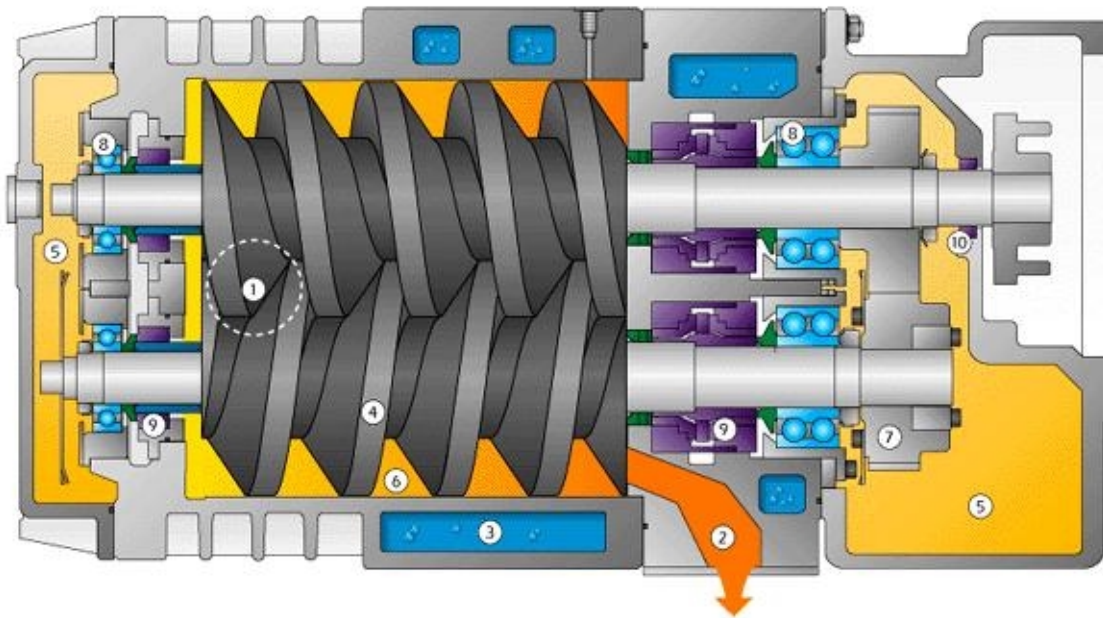
**ROTARY OIL-SEALED MECHANICAL PUMP MODULE**

Most 'wet' rough pumps are of the rotary mechanical pump type. Often there are several stages or modules to the pump which allows a typical base pressure of about 10 mTorr and an outlet pressure of atmosphere. (Figure from Basic Vacuum Practice, Varian Vacuum)



Multi stage 'wet' rough pumps are of the rotary mechanical pump type.

(Figure from Basic Vacuum Practice, Varian Vacuum)



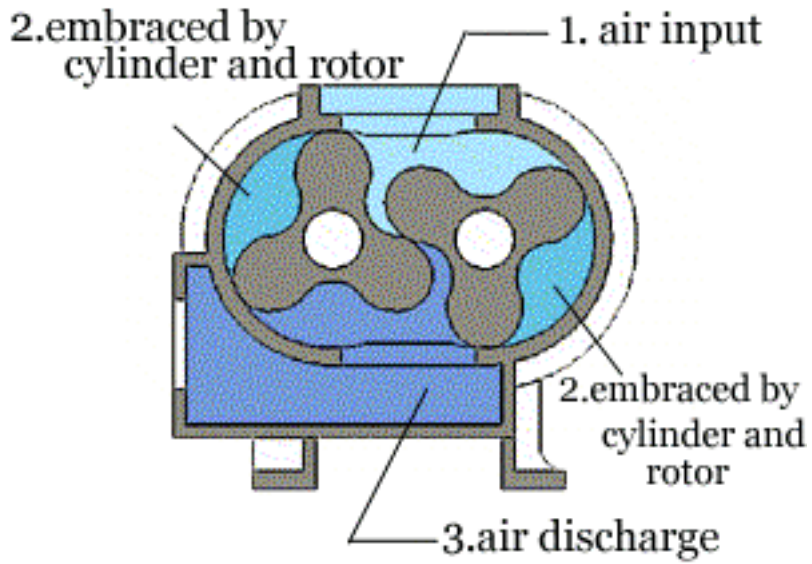
Wet pumps are now being phased out in favor of dry screw and dry rotary pumps.

This pump works by pushing gas from the inlet (1) along the gas path (6) to the exhaust (2).

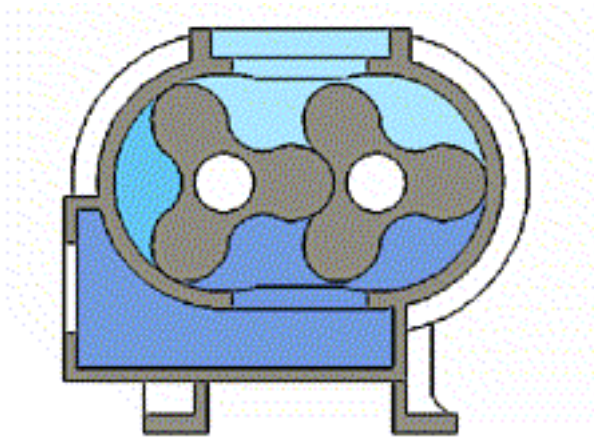
Because the screws are interleaved, the gas is trapped in pockets formed by the outer-wall and the screws.

Marked items are 1) inlet, 2) Exhaust, 3) water jacket 4) screw, 5) oil, 6) gas path, 7) timing gears, 8) bearings 9) shaft seals, 10) oil seal

(Picture from [www.buschpump.com/cobra\\_cutaway.html](http://www.buschpump.com/cobra_cutaway.html))

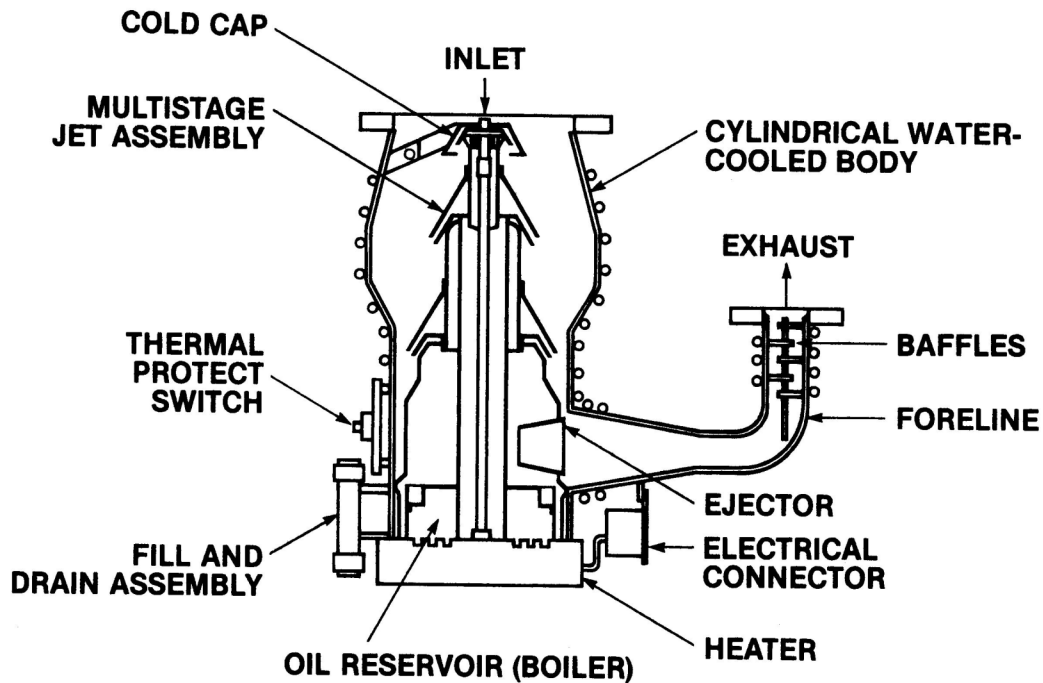


Typical setup of a roots blower (From [www.rootsblower.com.tw/](http://www.rootsblower.com.tw/))

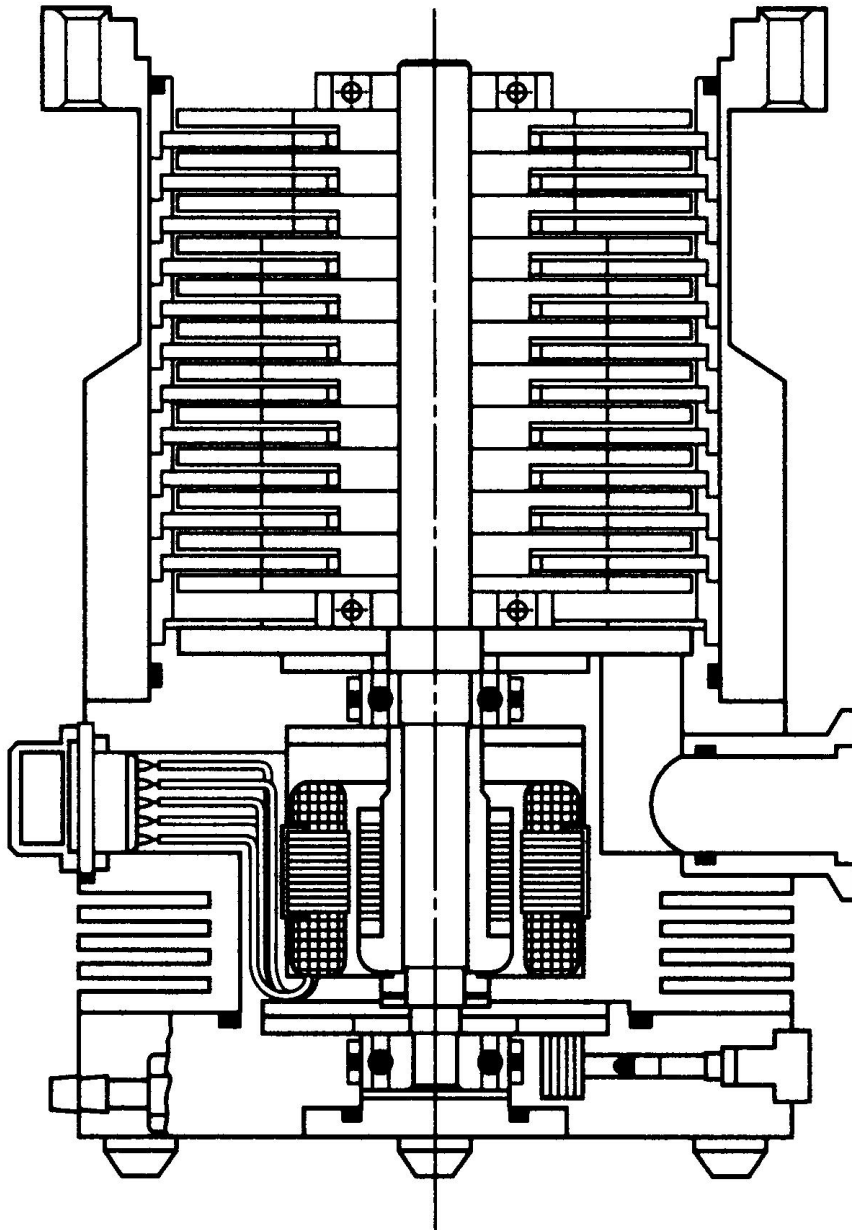


Movie of a roots blower (From [www.rootsblower.com.tw/](http://www.rootsblower.com.tw/))

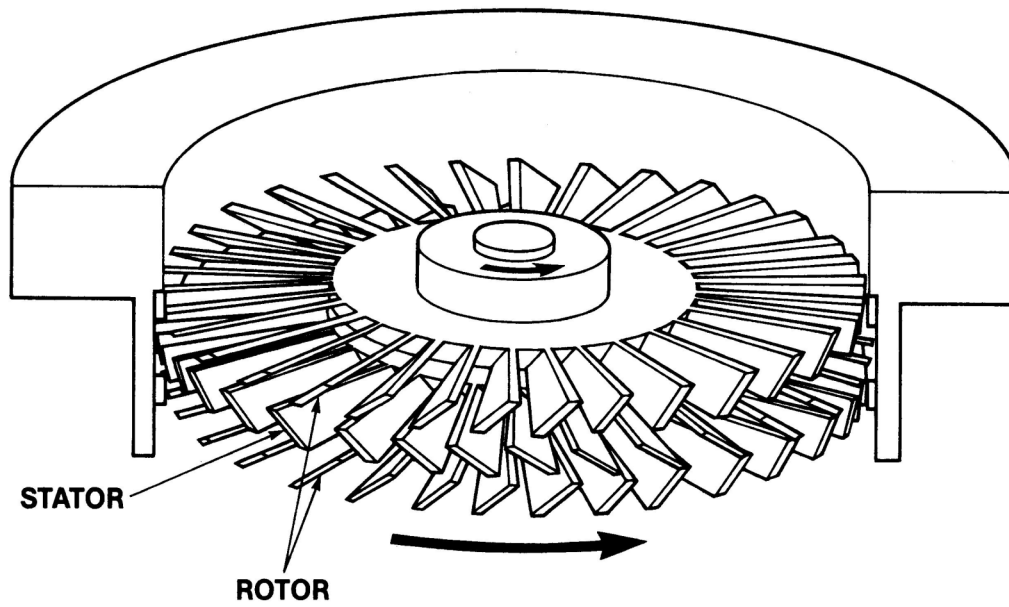
Roots blowers have limited use to only those systems needing ~10's to 100's mTorr operating pressures.



Set up for a diffusion pump. Diffusion pumps used to be the most common high vacuum pump. However in the last two decades they have been replaced with Turbomolecular pump. Super heated oil vapor in the base of the pump is forced up the central tubes to the central jet assembly. The oil vapor, which is now traveling at supersonic speeds, is reflected downward (and outward) off of the jet toward the cooled walls. During the trip to the walls, this oil entrains the gas particles and draws them down into the oil reservoir, where they are pumped out the exhaust port. (Figure from Basic Vacuum Practice, Varian Vacuum)

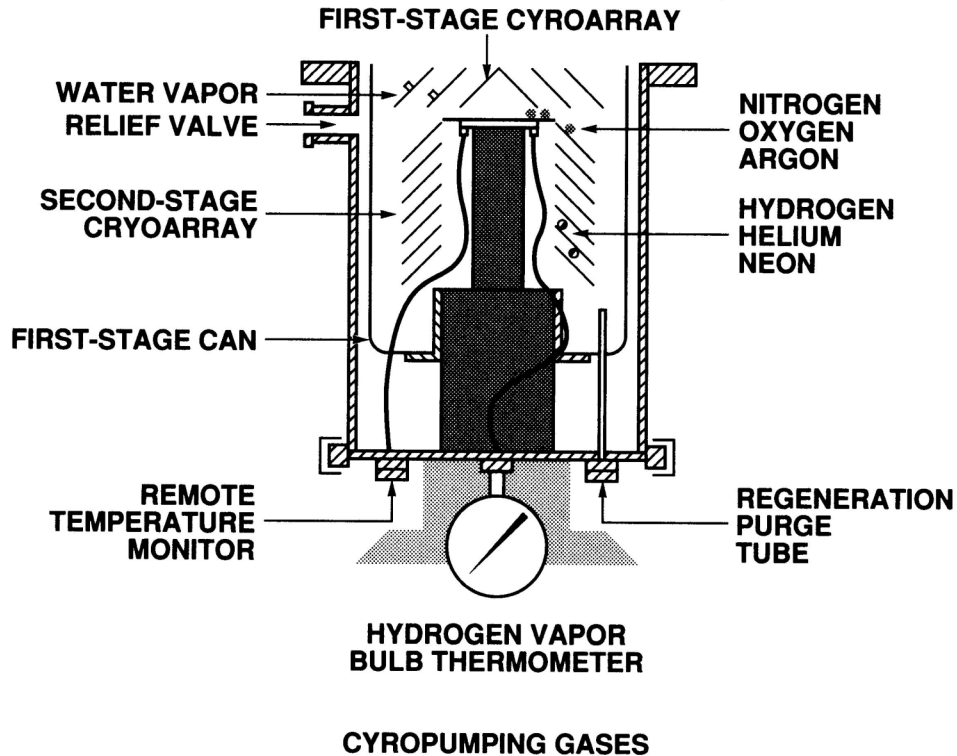


Turbomolecular pump (TMP). This is the most common type of pump for high vacuum processes. The pump consists of a series of rotors (rotating fans) and stators (fixed fans) with opposed blades. (The TMP is like a jet engine!) (Figure from Basic Vacuum Practice, Varian Vacuum)



The rotors and stators for a turbo pump. Typically the rotors turn at  $\sim 25,000$  RPM. The rotors push the gas downward and around. The blades of the rotors and stators are aligned such that the gas hits the underside of the stator blades, which cause the gas to move further downward and in the opposite rotation. Compression on a turbo pump might be as high as  $1E9$ , with a base pressure near  $1E-8$  Torr and outlet pressure near 10 mTorr. (Figure from Basic Vacuum Practice, Varian Vacuum)





Cryopumps are the second most common high vacuum pump system. They are typically only used in systems that do not have a significant use of feed gas. (An example would be e-beam systems.)

They usually have two pumping mechanisms. First, they have highly cooled vanes that freeze out most gas types. Secondly, for He, H/H<sub>2</sub> and Ne, they have adsorption traps, typically C, that confine the more mobile gases. (Figure from Basic Vacuum Practice, Varian Vacuum)

Sealant types: **(NEED TO ADD PICTURES)**

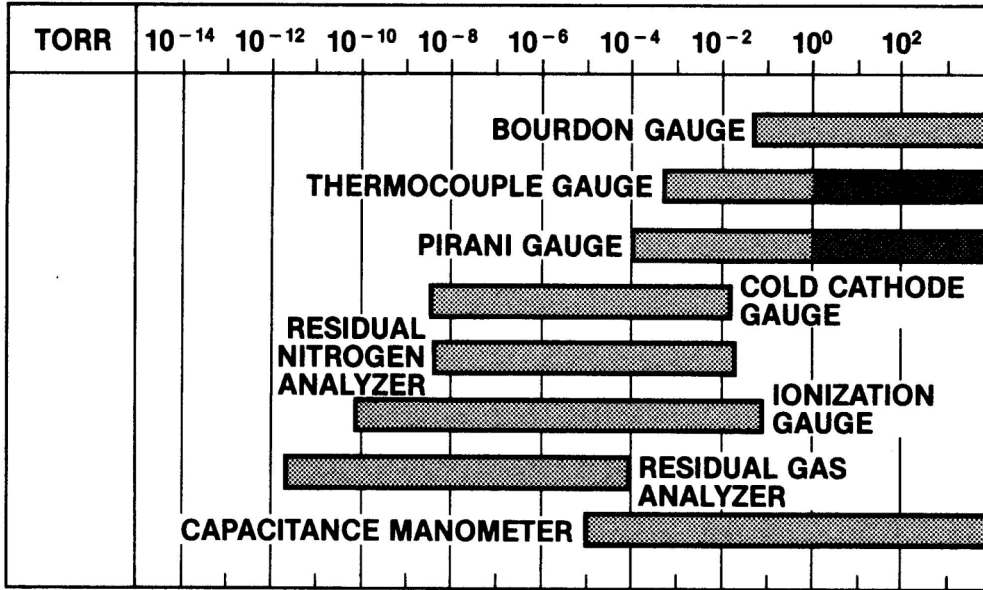
- 1) O-ring
  - a) ISO
  - b) KF (Kwik flange)
  - c) Custom o-ring
  
- 2) Metal
  - d) CF – copper also known as Conflat (Varian Vacuum Brand name)
  - e) Gold

In addition we need to have methods to measure the pressure. Typical pressure measurement tools include:

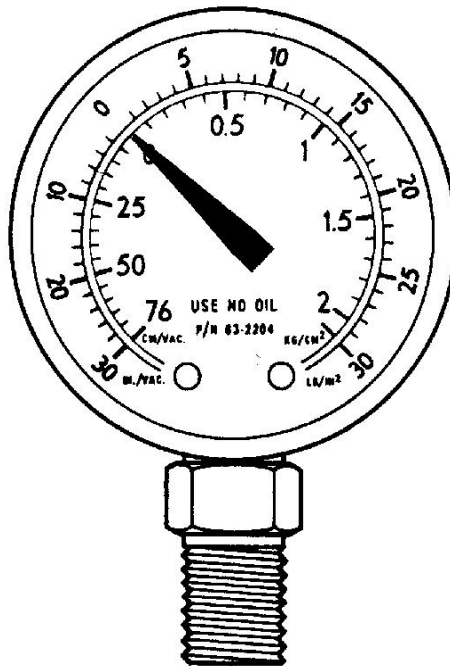
- 1) Bourdon Gauge
- 2) Therocouple
- 3) Pirnai
- 4) Cold Cathode

- 5) Ionization gauge
- 6) Capacitance Manometer – also known as a Baratron (MKS brand name)

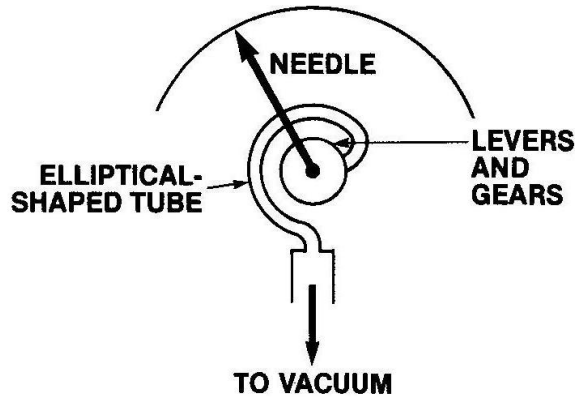
### TYPICAL GAUGE PRESSURE RANGES



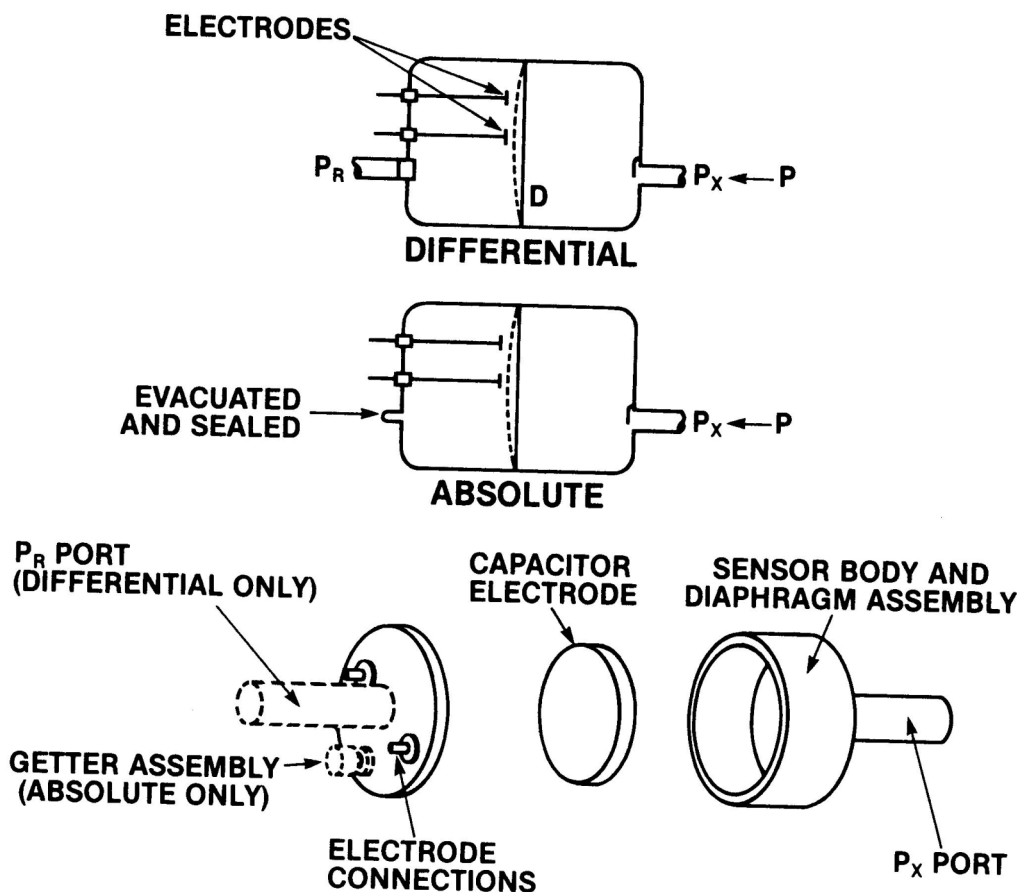
Typical pressure gauges. Only two of these, Bourdon and Capacitance Manometer, are direct measurements of the pressure. The others rely on secondary measurements to deduce the pressure. (Figure from Basic Vacuum Practice, Varian Vacuum)



Bourdon Gauge. (Figure from Basic Vacuum Practice, Varian Vacuum)

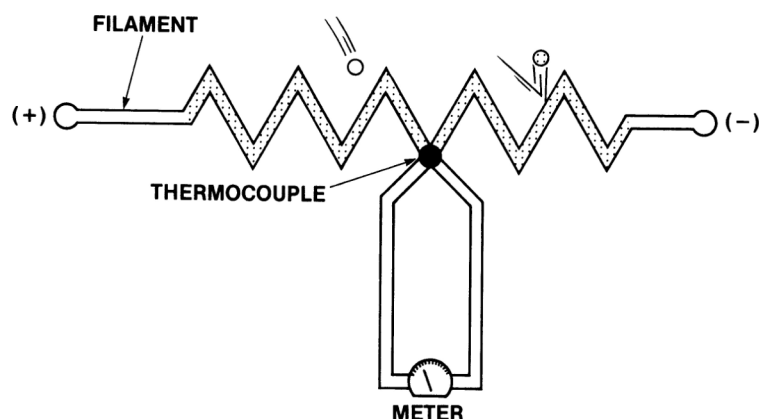


Bourdon Gauge insides. The elliptically shaped tube bends as more external pressure is applied, or it has lower pressure inside. This bend vs pressure is determined, allowing one to determine the inside/outside pressure differential. (Figure from Basic Vacuum Practice, Varian Vacuum)



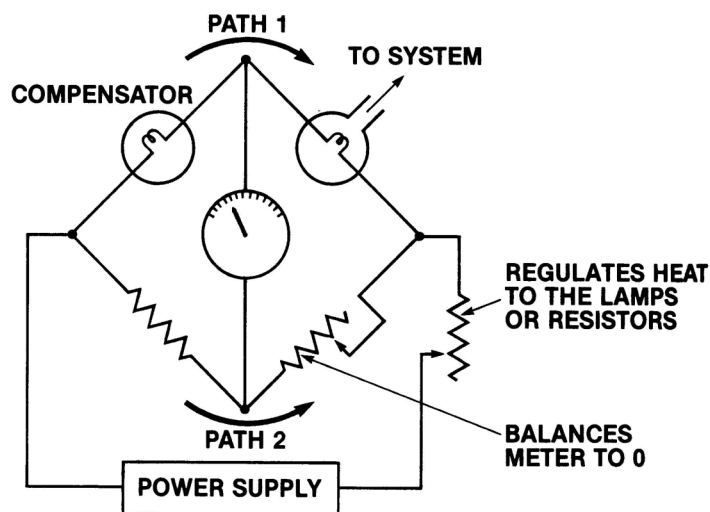
Capacitance manometer or Baratron gauge. ('Baratron' is a trademarked name, of MKS Instruments.) The gauge operates measuring the position of a diaphragm. This measurement is really a measure of the change in capacitance between two electrodes. As the diaphragm changes shape, due to the pressure, the capacitance changes. Thus the pressure can be measured. (Figure from Basic Vacuum Practice, Varian Vacuum)

### THERMOCOUPLE GAUGE PRINCIPLE



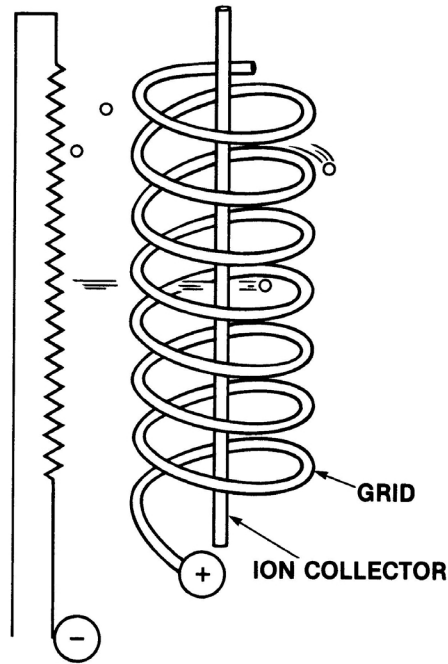
Thermocouple gauge operates by driving a current through a resistor of resistive wire. The temperature of the wire is determined by this heating as well as the cooling due to radiative cooling thermal cooling out the ends at via gas molecules carrying away heat. Under certain regimes, pressures greater than 1 mTorr, the dominate cooling mechanism is loss of energy to the gas molecules. (Note that the cooling gas dependent!) By measuring the temperature of the wire, and knowing the gas type, one can determine the pressure.

(Figure from Basic Vacuum Practice, Varian Vacuum)



Pirani Gauge. Pirani gauges are very similar to thermocouple gauges. Here, however, one measures the resistance in the heated wire. Resistance is a function of temperature. Typically, the higher the temperature, the higher the resistance. In the balance bridge circuit, the current flow in path 1 and 2 are set to be the same. The variable resistor in path 2 is used to balance the voltage across the central meter. When this is done the ratio of the resistors is the same as the ratio of the resistances in the two heated filaments. (The second filament is the 'compensator' and it is located in a area with well characterized pressure and temperature.) By determining the relative resistance, one can deduce the

temperature of the filament and hence the pressure – after one has assumed what is the gas type.  
 (Figure from Basic Vacuum Practice, Varian Vacuum)



Ionization gauge. Ionization gauges work in the following manner. Electrons are boiled off of a heated filament. (Shown on the left on side of the figure.) These electrons are accelerated toward the collector grid by a bias between the filament and grid. Many of the electrons do not hit the grid directly but rather miss it and pass in to the area between the grid and the ion collector. (The electrons make several pass past the grid before hitting it, resulting in an electron cloud around the grid.) While inside the grid, some of the electron will ionize the local gas. These ions are collected by the collector. The amount of current is determined by the gas type and the gas density. (Figure from Basic Vacuum Practice, Varian Vacuum)

## **BASIC GAS PRINCIPLES**

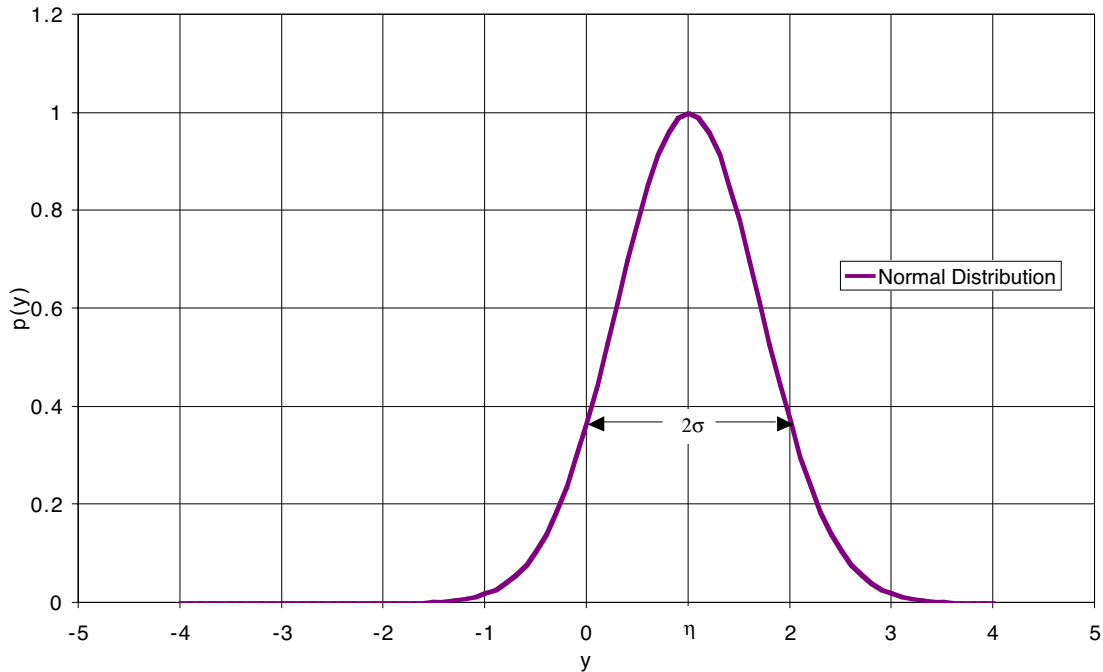
We now have tools to pump, hold and measure vacuums. However, to fully understand what is going on in our vacuum system we need to first understand some basic issues about the gas.

### **Velocity Distribution**

As gas atoms/molecules (or particles) move through out a volume they collide and randomly distribute their energy. A basic tenant of statistics is that random processes result in Normally distributed results. This is know as the central limit theorem, see for example Box Hunter and Hunter, “Statistics for experimenters” (This title might not be quite correct.) The normal distribution is given by

$$p(y) = const \frac{1}{\sigma} \exp\left[\frac{-(y - \eta)^2}{\sigma^2}\right].$$

Here  $\sigma^2$  is the population variance,  $\sigma$  is the population standard deviation, and  $\eta$  is the central value. The constant is usually set such that the total probability is 1. The distribution looks like



## Concept of Temperature

Maxwell and Boltzman proposed that this same distribution can be used to model the velocity distribution of particles in thermal equilibrium. (This is known as the Maxwell Distribution, Boltzman Distribution and the Maxwell-Boltzman Distribution.) Using this assumption we have that the velocity distribution is

$$f(\bar{\mathbf{v}}) = \text{const} \exp\left[\frac{-m(\bar{\mathbf{v}})^2}{2kT}\right],$$

where  $\mathbf{v}$  is the velocity,  $\mathbf{v}_0$  is the average or drift velocity (which is often zero)),  $k$  is Boltzman's constant,  $T$  is the gas temperature in Kelvin and  $m$  is a particle mass. Notice that the temperature is often stated in units of energy (typically eV). When this is done you are using  $kT$  not just  $T$ . Note also that the temperature is a measure of the standard deviation of the velocity distribution – hence it is a measure of the variation in the velocity. We can also rewrite this as an energy distribution

$$f(\mathcal{E}) = \text{const} \exp\left[\frac{-\mathcal{E}}{kT}\right],$$

where the two constants are different.

To get the constant for the velocity distribution, we set the constant such that

$$n = \iiint f(\mathbf{v}) d\mathbf{v}$$

where  $n$  is the particle density then we find that for our gas particles we get a velocity distribution of

$$f(\mathbf{v}) = n \left( \frac{m}{2\pi kT} \right)^{3/2} \exp\left[ \frac{-\frac{1}{2} m(\mathbf{v} - \mathbf{v}_0)^2}{kT} \right]$$

The velocity distribution is such that the peak is at the average velocity and is bell shaped.

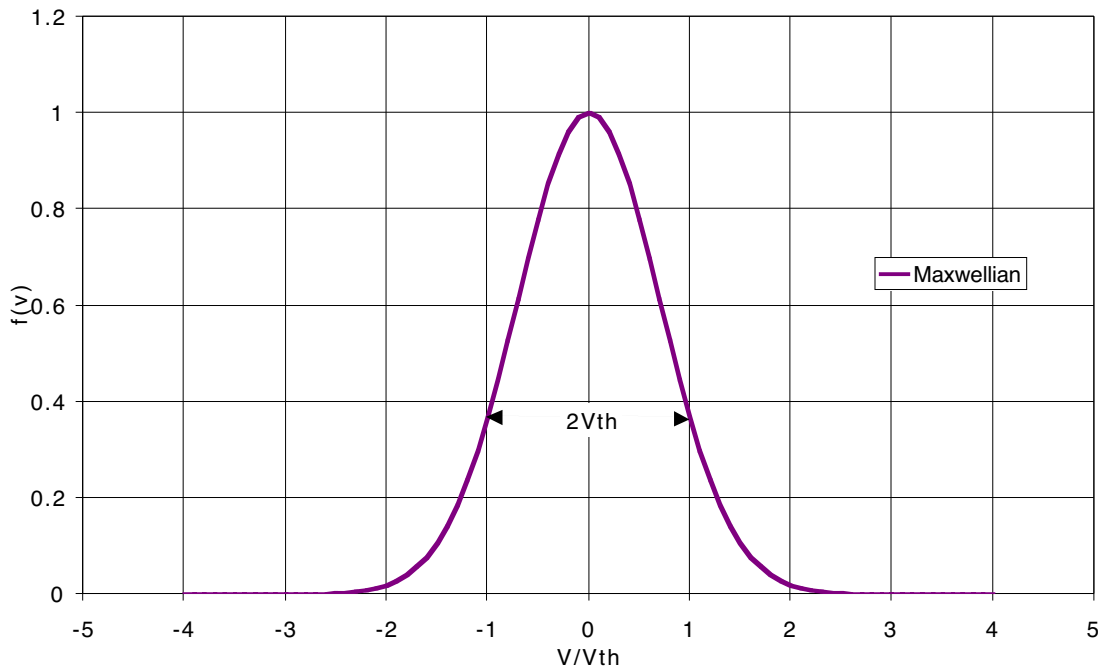
### Problem 1

Show that if in one dimension,  $\int f(v)dv = n$ , then  $const = n\left(\frac{m}{2\pi kT}\right)^{1/2}$ .

Also show that if in three dimensions,  $\int f(\vec{v})dv^3 = n$ , then  $const = n\left(\frac{m}{2\pi kT}\right)^{3/2}$ .

Typically, the velocity distributions are well behaved and we have distributions as such:

#### The Maxwellian Distribution

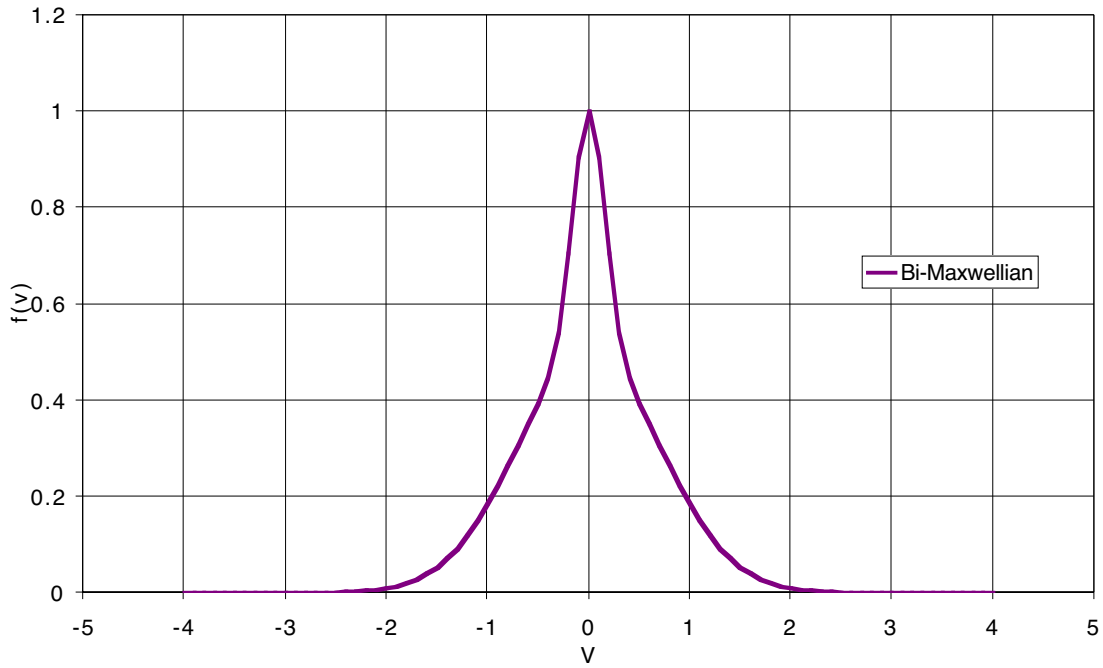


Where

$$f(\mathbf{v}) = n\left(\frac{m}{2\pi kT}\right)^{1/2} \exp\left[\frac{-m(\mathbf{v})^2}{2kT}\right].$$

This is what happens if there are enough collisions to evenly distribute the energy. In other cases, however, we may be working in with a system in which the rate of collisions is not large enough to evenly distribute the energy. Some typical examples are:

#### The bi-Maxwellian Distribution

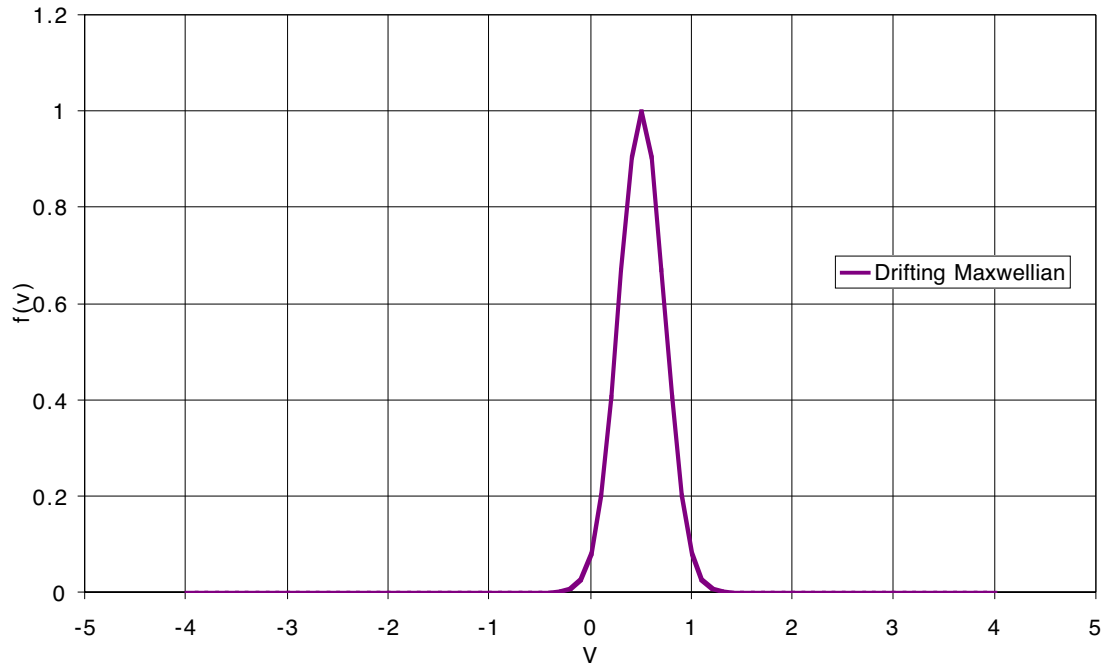


Where

$$f(\mathbf{v}) = (1 - \alpha)n \left( \frac{m}{2\pi kT_1} \right)^{1/2} \exp \left[ \frac{-m(\mathbf{v})^2}{2kT_1} \right] + (\alpha)n \left( \frac{m}{2\pi kT_2} \right)^{1/2} \exp \left[ \frac{-m(\mathbf{v})^2}{2kT_2} \right]$$

The drifting Maxwellian Distribution

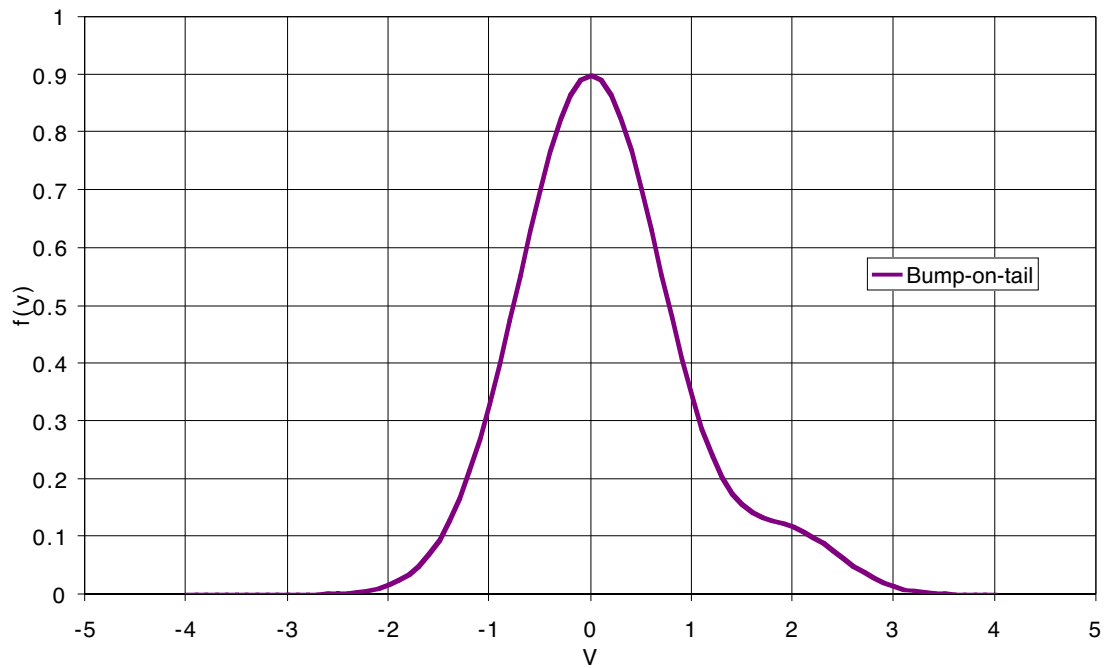




Where

$$f(\mathbf{v}) = n \left( \frac{m}{2\pi kT} \right)^{1/2} \exp \left[ \frac{-m(\mathbf{v} - \mathbf{v}_0)^2}{2kT} \right].$$

and the 'bump-on-tail'



Where

$$f(v) = f_{Maxwell} + f_{drift}.$$

While other distributions are certainly possible, these four comprise most of what is observed in laboratory plasmas.

At this point we can examine the velocity integral in more detail.

$$\begin{aligned} n &= \iiint f(\mathbf{v}) d\mathbf{v} \\ &= \iiint f(\mathbf{v}) dv_x dv_y dv_z \quad (\text{Cartesian}) \\ &= \iiint f(\mathbf{v}) v^2 dv \sin \theta d\theta d\phi \quad (\text{Spherical}) \\ &= 4\pi \int_0^\infty f(\mathbf{v}) v^2 dv \\ &= 4\pi \int_0^\infty n \left( \frac{m}{2\pi kT} \right)^{3/2} \exp \left( \frac{-\frac{1}{2} m \left( \mathbf{v} - \underbrace{\mathbf{v}_0}_{=0} \right)^2}{kT} \right) v^2 dv \\ &= \int_0^\infty \underbrace{4\pi n \left( \frac{m}{2\pi kT} \right)^{3/2} \exp \left( \frac{-\frac{1}{2} m v^2}{kT} \right)}_{\text{speed distribution}} v^2 dv \\ &\Rightarrow \frac{dn}{dv} = f_{\text{speed}}(v) = 4\pi n v^2 \left( \frac{m}{2\pi kT} \right)^{3/2} \exp \left( \frac{-\frac{1}{2} m v^2}{kT} \right) \end{aligned}$$

Likewise, we can convert this into energy space to get the energy distribution

$$\begin{aligned} n &= \int_0^\infty \underbrace{4\pi n \left( \frac{m}{2\pi kT} \right)^{3/2} \exp \left( \frac{-\frac{1}{2} m v^2}{kT} \right) v^2 dv}_{\text{speed distribution}} \quad \text{noting } \mathcal{E} = \frac{1}{2} m v^2, \quad d\mathcal{E} = m v dv \\ &= \int_0^\infty \underbrace{2\pi n (\pi kT)^{-3/2} \sqrt{\mathcal{E}} \exp \left( \frac{-\mathcal{E}}{kT} \right) d\mathcal{E}}_{\text{energy distribution}} \\ &= \\ &\Rightarrow \frac{dn}{d\mathcal{E}} = g(\mathcal{E}) = 2\pi n (\pi kT)^{-3/2} \sqrt{\mathcal{E}} \exp \left( \frac{-\mathcal{E}}{kT} \right) \end{aligned}$$

which of course gives us our energy distribution constant from above

We can further use these equations to determine the most probable speed,

$$\frac{d}{dv} f_{\text{speed}}(v) = 0$$

⇓

$$v_p = \left( \frac{2kT}{m} \right)^{1/2}$$

Most probable energy

$$\frac{d}{d\mathcal{E}} g(\mathcal{E}) = 0$$

⇓

$$\mathcal{E}_p = \frac{1}{2} kT$$

Mean (average) velocity

$$\int \mathbf{v} f_{\text{speed}}(\mathbf{v}) d\mathbf{v} = \mathbf{v}_{\text{ave}} = 0$$

Mean (average) speed

$$\int_0^{\infty} v f_{\text{speed}}(v) dv = v_{\text{ave}} = \left( \frac{8kT}{\pi m} \right)^{1/2}$$

Mean (average) energy

$$\int_0^{\infty} \mathcal{E} g(\mathcal{E}) d\mathcal{E} = \mathcal{E}_{\text{ave}} = \frac{3}{2} kT$$

RMS velocity

$$\int |\mathbf{v}| f_{\text{speed}}(\mathbf{v}) d\mathbf{v} = v_{\text{RMS}} = \sqrt{\frac{3kT}{m}}$$

Gas pressure

Lets assume that we have a particle in a box of sides  $l$ . Assume that the particle is reflected with no

loss of energy (why?) when it hits wall A1. Thus  $\Delta E = 0$  but

$$\Delta M = \underbrace{-mv_x}_{\text{final momentum}} - \underbrace{mv_x}_{\text{initial momentum}} = -2mv_x$$

Where we have assumed that  $v_y = 0 = v_z$ . Now let us assume that the particle travels across the volume without striking anything else and reflects off side A2. The round trip time is  $\Delta t = \frac{2l}{v_x}$

The force applied to side A1 is

$$F = -\frac{\Delta M}{\Delta t} = \frac{2mv_x}{2l/v_x} = \frac{mv_x^2}{l}$$

and the pressure is

$$P = \frac{F}{l^2} = \frac{mv_x^2}{l^3}$$

Putting in more particles we find

$$\begin{aligned}
P &= \sum_{\text{all particles}} \frac{mv_x^2}{l^3} = m \frac{N}{l^3} \sum_{\text{all particles}} \frac{mv_x^2}{N} \\
&= mn \langle v_x^2 \rangle \\
&= 2n \frac{1}{2} n \langle v_x^2 \rangle = 2n \langle E_x \rangle \\
&= nkT - \text{the ideal gas law!}
\end{aligned}$$

(Note that this is done in 1-D. If we were to do it in 3-D we of course get the same result.)  
Now if we add van der Waals' Force and account for the size of the particles, we get the more complex version of the gas law

$$\left( P + \frac{a}{V^2} \right) \left( V - \frac{b}{\text{particle size}} \right) = NkT$$

van der Waals attraction      particle size

For the most part, the ideal gas law is sufficient to accurately describe our systems.

### **ENERGY LOSS AND GAIN**

The exact energy distribution that a gas, ions or electrons might have is dependent on energy gain and loss. Typically understanding energy gain is somewhat difficult and will be explored in the next class in the plasma sequence, EE/Phys 6383. Energy loss is somewhat more manageable.

For most individual particles energy loss will be through a number of possible collision processes. These collisions can either be with particles of other species or collisions with walls. (We do not consider collisions between like particles as they typically do not result in energy loss from the total energy of that species.) While some individual collision process may be very complex, we only consider a small set of the most common collision types.

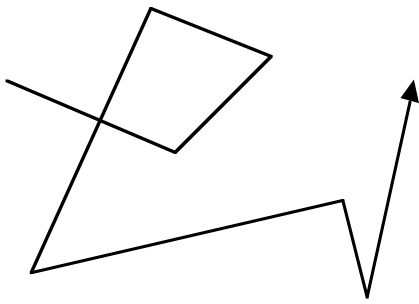
Electron-Neutral

Electron-Ion

Ion-Neutral

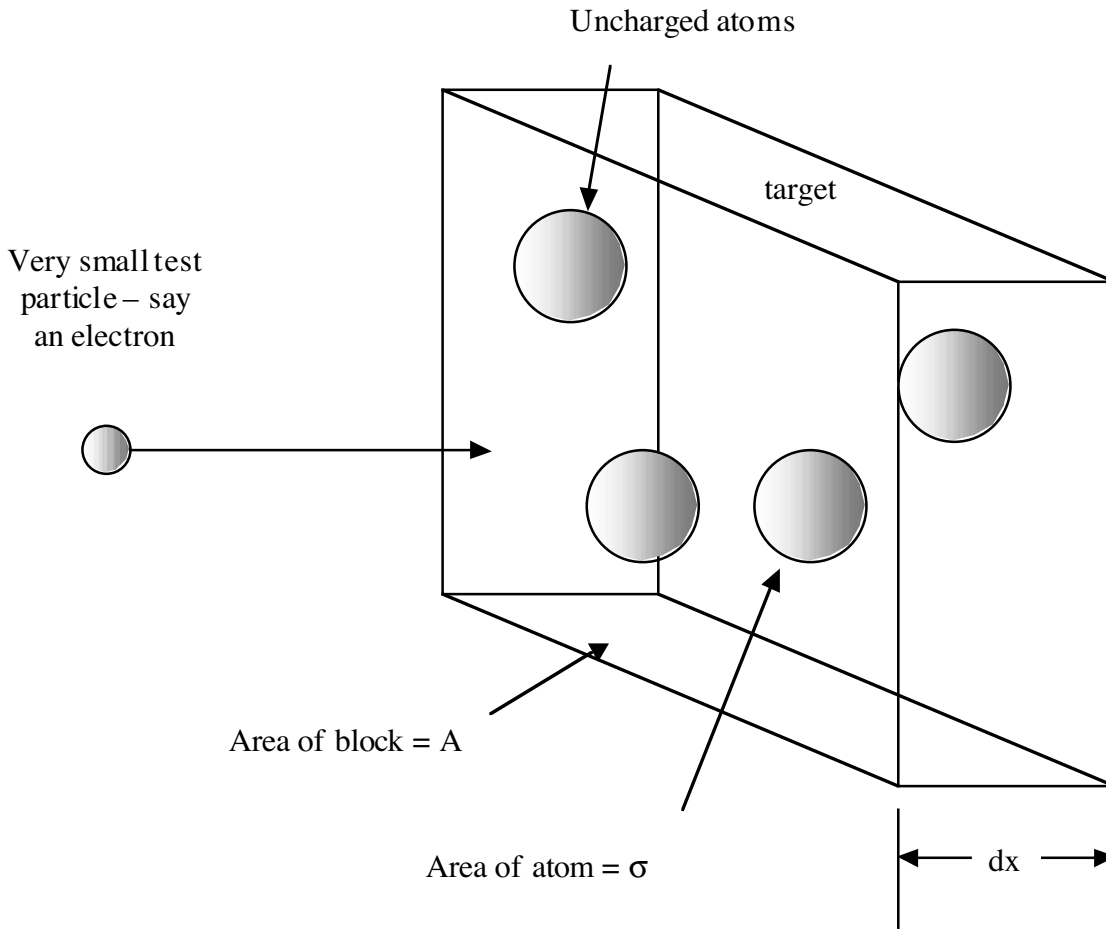
### Concept of Mean-free path

As atoms/molecules pass through a volume, they collide with other atoms/molecules. A prime example of this is Brownian motion – the motion of a dust particle in air that moves in disjointed fashion.



also known as  
a random walk

We can calculate the mean-free path, mfp, if we consider the following picture.



If we were to repeatedly fire test particles at the target, a fraction of the test particles will be scattered by collisions with atoms in the target. The fraction scattered is simply the area ratio.

$$\text{Fraction scattered} = \frac{N\sigma}{A}$$

where N is the number of atoms in the target. Assuming that our target is a part of a larger piece of material, in which we know the density, n, of the material then

$$\text{Fraction scattered} = \frac{nAx\sigma}{A} = n\sigma dx.$$

Now let us send a continuous flux,  $\Gamma$ , of test particle – or a current density  $\mathbf{J}$ , at the target then the change in  $\mathbf{J}$  across the target is

$$\begin{aligned}\Delta \mathbf{J} &= \mathbf{J}_{after} - \mathbf{J}_{before} \\ &= (1 - n\sigma dx)\mathbf{J}_{before} - \mathbf{J}_{before} \\ &= -\mathbf{J}n\sigma dx\end{aligned}$$

or

$$\frac{d\mathbf{J}}{dx} = -\mathbf{J}n\sigma$$

⇓

$$\mathbf{J} = \mathbf{J}_0 \exp(-x / \lambda_{mfp})$$

where  $\lambda_{mfp} = \frac{1}{n\sigma}$ .

We can make approximations of the cross section of the target atom, based on the radius of the target atom. This is known as the hard-sphere approximation, which is at best imprecise. The reality is that the interaction, and hence collisions, is related to the target atom electron orbitals and particle energies, i.e. it is quantum mechanical in nature. From our above discussion, we can calculate the Mean frequency of collisions

$$v = \frac{v}{\lambda_{mfp}} = vn\sigma$$

And the mean period of collisions

$$\tau = \frac{1}{v}$$

Of course the average collision frequency for a distribution of velocities is

$$\langle v \rangle = n \langle v\sigma \rangle \text{ noting } \sigma = \sigma(v).$$

Collisions with walls

We are not really interested in the collision rate of a particle with a wall but rather we interested in how many particles are hitting the wall. (This may seem like it is just semantics but it is not.) To determine this we must first consider the flux of the particles in one given direction.

$$\Gamma = \langle n v_z \rangle = \int v_z f(\mathbf{v}) d\mathbf{v}$$

$$= \frac{1}{4} n v_{ave}$$

$$= \frac{1}{4} n \sqrt{\frac{8kT}{\pi m}}$$

(The 1/4<sup>th</sup> arises from integrating over all the angles.)

Now we can consider how long it might take to completely cover the wall with a monolayer of particles. This is simply

$$\tau_{monolayer} = \frac{1}{\Gamma A_{particle}}$$

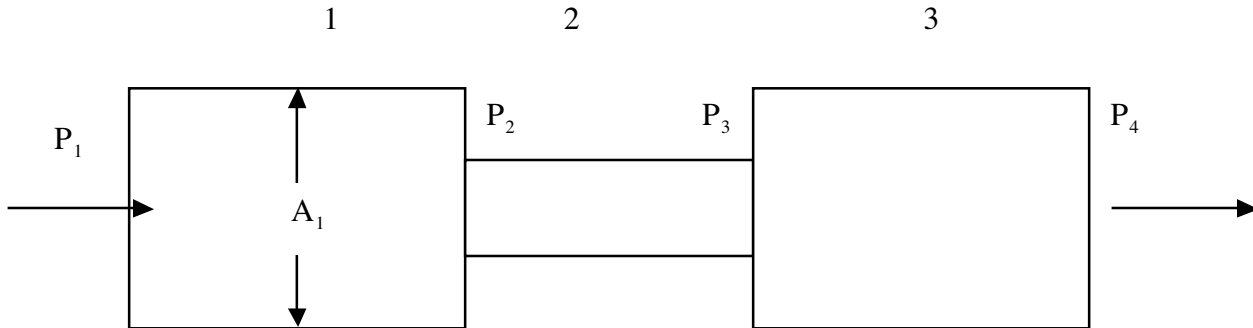
where  $A_{particle}$  is the area of a particle.

[Note that when the mean-free path is on the order of the size of the chamber or longer, the collision rate of the particles with walls can be given by the effective chamber size divided by the average speed.]

## Gas Flow

Conservation of Flux (which we may prove later)

If a gas passes through a series of pipes with various cross sectional areas, then the mass flux is conserved. Physically this should be obvious. Think of this as the same as saying that we do not have rarefaction/compression of the gas, nor do we have a source/sink for the gas.



$$A\Gamma = A_1 n_1 \langle v_1 \rangle = A_2 n_2 \langle v_2 \rangle = A_3 n_3 \langle v_3 \rangle \dots$$

Flow resistance/conductance

If a gas flow through a pipe, we expect some pressure differential to cause the flow. The ratio of the pressure to the flow is known as the resistance,  $R$ .

$$R = \frac{\Delta P}{\Gamma}$$

The conductance is simply the inverse of the  $R$ ,

$$C = \frac{1}{R} = \frac{\Gamma}{\Delta P} \quad \frac{\# / s / \text{area}}{\text{Force} / \text{area}}$$

These equations are very similar to the resistance in a circuit, where we replace voltage with pressure. Thus for a series of pipes

$$\Delta P_{total} = \frac{\Gamma}{C_{total}} \quad \text{but}$$

$$\Delta P_{total} = \sum_i \Delta P_i \quad \text{and so}$$

$$\Delta P_{total} = \frac{\Gamma}{C_{total}} = \sum_i \frac{\Gamma}{C_i} = \Gamma \sum_i \frac{1}{C_i}$$

⇓

$$\frac{1}{C_{total}} = \sum_i \frac{1}{C_i}$$

or in a similar vane,

$$R_{total} = \sum_i R_i.$$

For Parallel pipes we find

$$C_{total} = \sum_i C_i \quad \text{and}$$

$$\frac{1}{R_{total}} = \sum_i \frac{1}{R_i}$$

## Pumps

We can think of pumps as simply another conductance,  $S$ . After all we are trying to have a certain  $\Gamma$  go through the pump and the pump acts as if the back side has a pressure of zero. Here however, the pump conductance is given a special name,  $S$ , for 'speed'.

If a pump has a pipe connecting it to the chamber, we get an effective pump speed from

$$\frac{1}{S_{eff}} = \frac{1}{S} + \frac{1}{C_{pipe}}$$

Example:

Given a pipe with a conductance  $C = 100$  L/s and a pump speed of  $S = 200$  L/s we find that the effective pump speed is 66.6 L/s. This means that we using only 33% of our possible pumping ability. This can become critical in the design of a process system.

Types of gas flow.

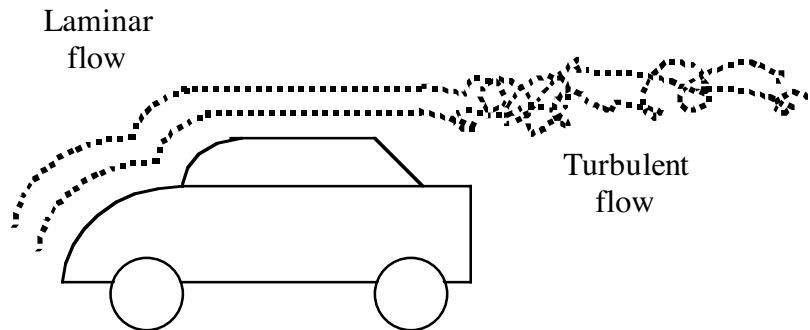
There are three radically different types of gas flow. They are:

Laminar fluid (viscous) flow

Turbulent fluid flow

Molecular flow

The distinction between Laminar ('layer') and turbulent is often seen in wind tunnel experiments on new cars.



One can determine if one is operating in fluid flow or molecular flow by determining the Knudsen number,  $K_n$ .

$$K_n = \frac{\overbrace{\lambda_{mfp}}^{\text{mean-free path for all collisions}}}{\underbrace{d}_{\text{characteristic distance across system}}} = \begin{cases} < 1 & \text{Molecular (most interactions with walls)} \\ \sim 1 & \text{Transition or 'slip' region} \\ >> 1 & \text{Fluid (most interactions with other particles)} \end{cases}$$



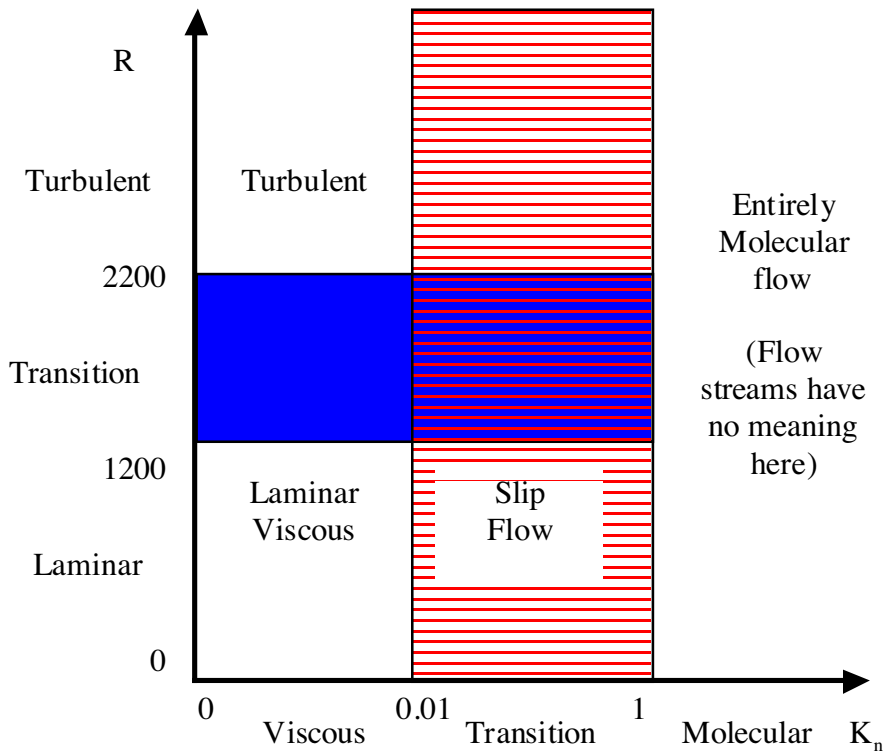
In addition we use Reynold's number to describe the smooth or turbulent nature of the flow

$$R = \frac{\text{Stream speed } \vec{u} \text{ (m/s)} \cdot \text{mass density } \vec{\rho} \text{ (kg/m}^3\text{)} \cdot d}{\text{gas viscosity } \vec{\eta} \text{ (Pa s)}} = \begin{cases} < 1200 & \text{Laminar (smooth flow streams)} \\ \text{else} & \text{Transition} \\ > 2200 & \text{Turbulent (chaotic flow streams)} \end{cases}$$

Kinetic theory gives

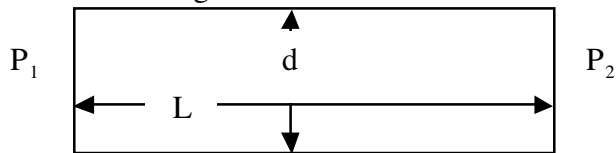
$$\eta \approx \frac{1}{2} nmv_{ave}\lambda_{mfp}$$

This gives rise to the following diagram:



We can calculate the conductance in molecular and fluid flow regimes from the following equations. (Note these equations are at least partly experimental approximations.)

Fluid Flow through a round tube



with the requirements  $P_1 > P_2$  and  $L \gg d$ .

Laminar Flow (the most common for us)

$$C = \frac{\pi d^4}{128\eta L} \frac{P_1 + P_2}{2}; \quad \eta = \text{gas viscosity}$$

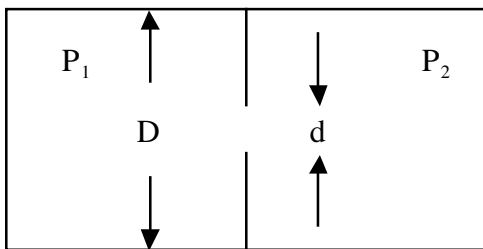
Here  $P_1$  and  $P_2$  are in mbar and L and d are in cm, giving C in L/s.

Turbulent Flow

$$C = \frac{d}{P_1 - P_2} \left( \frac{4}{\pi\eta} \right)^{1/7} \left[ \frac{\pi^2}{3.2} \frac{5}{4} d^3 \frac{P_1^2 - P_2^2}{2L} \right]^{4/7} \left( \frac{\overset{\text{gas constant}}{\tilde{R}} T}{\underbrace{M_{\text{molar}}}_{\text{Molar mass}}} \right)^{3/7}$$

Here,  $M_{\text{molar}}$  is in g/mole, R is 83.14 mBar L/Mole/K and T is in K.

Fluid Flow through an orifice



with the requirements  $P_1 > P_2$ .

$$C = \frac{P_1}{P_1 - P_2} \frac{\pi d^2}{4} \left( \frac{2RT}{M_{\text{molar}}} \right)^{1/2} \left( \frac{P_2}{P_1} \right)^{1/\chi} \left[ \left( \frac{\chi}{\chi - 1} \right) \left( 1 - \frac{P_2}{P_1} \right)^{(1-\chi)/\chi} \right]^{1/2}$$

where  $\chi = \frac{C_p}{C_v}$  is the adiabatic constant

Molecular flow through a round tube

$$C = \frac{\pi d^2}{4} \frac{\left[ \frac{RT}{2\pi M_{\text{molar}}} \right]^{1/2}}{\left( 1 + \frac{3L}{4d} \right) \zeta}; \quad \text{where } 1 \leq \zeta \leq 1.12 \text{ is a fudge factor}$$

Momentum flow

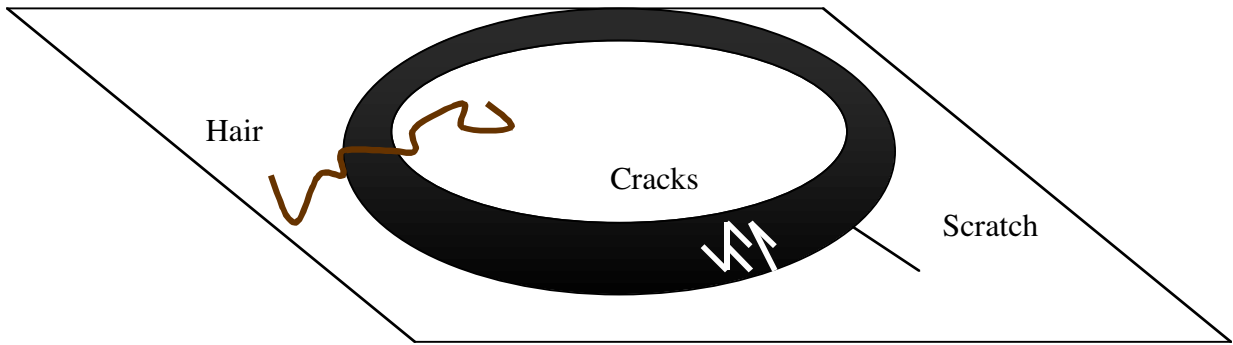
Heat flow

## Leaks

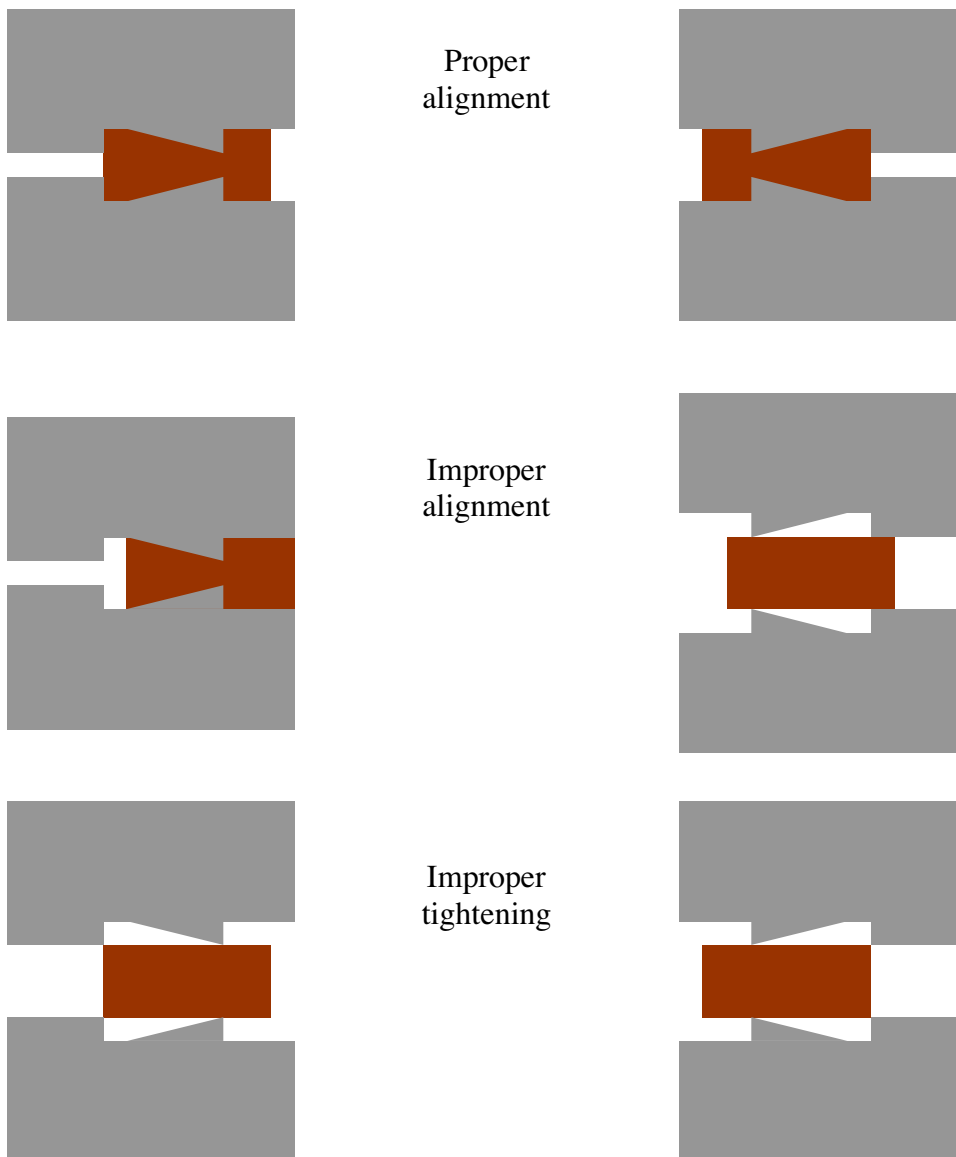
External leaks

Obviously leaks can come from a variety of sources. The most common place that one might envision a leak is at a weld or on a gasket between two flanges. These are known as 'external' leaks. Such leaks can be the result of hair across the oring, a scratch on the oring surface or a nick in the

copper gasket or a scratch on the knife-edge of the conflat. Such vacuum leaks are common but they are also usually easily found and fixed.

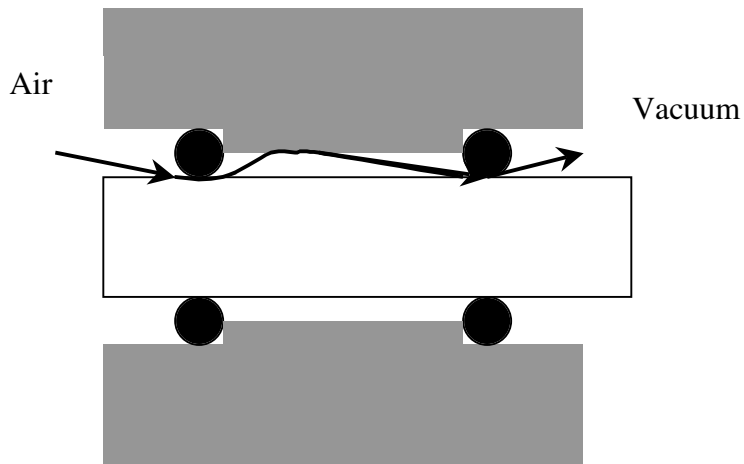


To fix leaks at oring seals, one usually does the following. Hair or lint is removed by carefully cleaning the surface with alcohol and a lint-free towel. Acetone is never used as it degrades most oring materials. Then a light coating of oring grease is applied to fill small imperfections. (Note the use of oring grease is not required and in some cases – when using certain chemicals – not warranted.) If cracks are present in the oring the oring is replaced with a new one of similar material. (Note orings come in a wide variety of materials with a huge variation – several orders of magnitude – in prices. The specific material that is used depends on the application. Typically one picks the material such that it is reasonably priced and can withstand the environment.) Scratch in the oring surface can be repaired one of two ways. If the scratch is deep, the surface may have to be remachined. If the scratch is small, it can be taken out with fine emory cloth (a fine type of sand paper). To sand out the scratch one typically uses a broad fairly flexible surface to hold the emory cloth against the oring surface. (A thumb is usually ideal for this.) By sanding the oring surface in this manner, one replaces that scratch with a long shallow depression in the surface which the oring can easily conform to.



Typically conflat gaskets only leak when the gasket is improperly aligned or the gasket is not tight enough for the knife edge to properly cut into the gasket. These leaks can be repaired by replacing the gasket, if it is improperly aligned, or simply tightening the seal. (The force due to air pressure is strong enough to seal most oring seals if they are properly aligned but this is not true for metal-metal seals such as conflats.) On rare occasions nicks will occur in the knife edge of the conflat. Typically this means that the flange must be replaced, although sometimes the knife edge can be resharpened with a file.

Lastly we will consider external virtual leaks. These occur when someone has placed a double seal, or a double weld, on the system think that this will result in a better seal. Usually this does not improve the system but it can lead to leaks that are very hard to detect. (Detection is usually done with a secondary gas such as He. We will get to this later in the course.) An example of an internal leak is shown below.



### Internal leaks

While the above external leaks are typically what one thinks of when one thinks of leaks, they are not the most common. The most common are internal leaks. Typically these leaks are smaller than external leaks and they are such that the amount of gas introduced into the chamber decreases with time.

Internal virtual leaks

Backstreaming

Gas release from solids

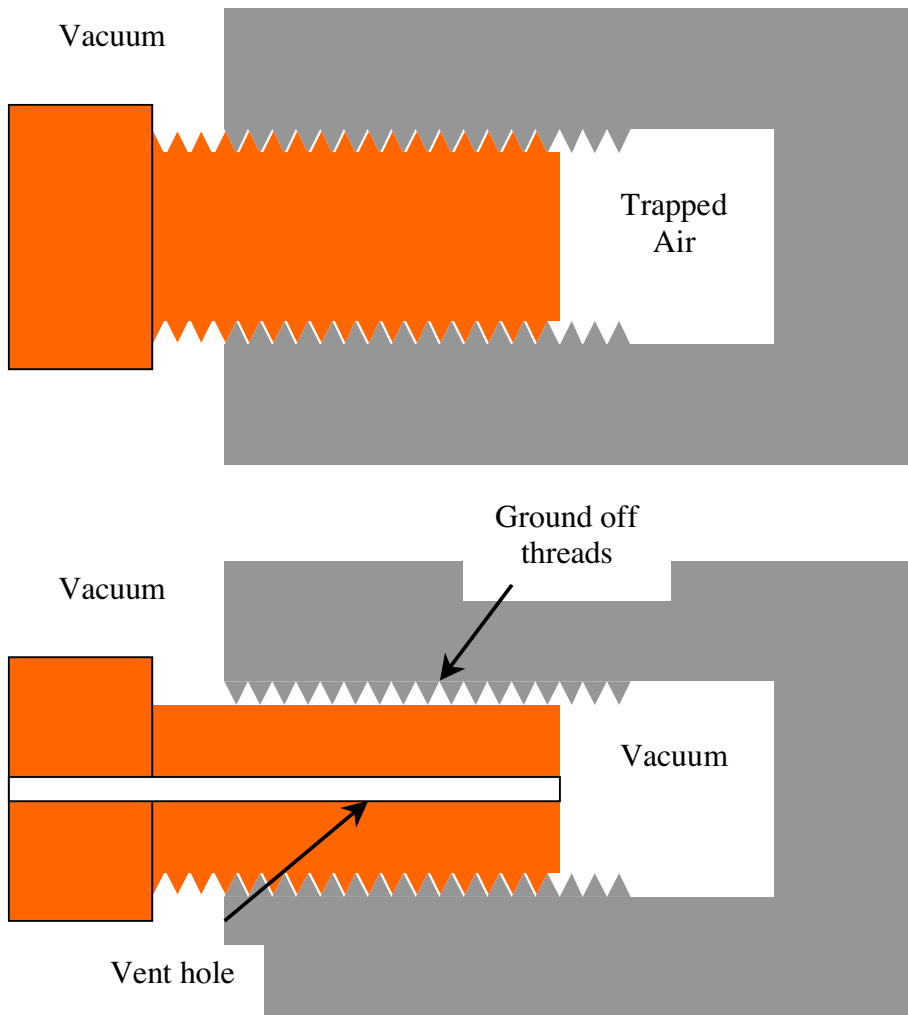
a) Vaporization

b) Diffusion

c) Permeation

d) Desorption

Internal virtual leaks are not unlike external virtual leaks. However, instead of having an infinite source of gas, the gas source is limited. Typically the source of the leak is trapped air inside a screw hole. The air leaks out slowly past the screw threads until all of the gas has escaped. To prevent such leaks one often drills hole through the screw or grinds off one side of the screw.



Vaporization occurs when a liquid or solid with a low vapor pressure is left in the chamber. Typically this is not an issue, and it can be used as a way of producing a useful process gas out of a material that is typically solid or liquid at standard pressure and temperature.

Backstreaming is due to oils and/or gases that the pump allows to flow backward into the process chamber. While most gases can be pumped efficiently with most pump systems, extremely light gases – such as He – can travel in the opposite direction. Often this is not a problem as He is fairly rare in the atmosphere. Backstreaming due to pump oil can however be a real issue. Such backstreaming occurs because the molecules are acted on randomly and some very small fraction just happen to migrate in the ‘wrong’ direction. In pumps that make use of oil for pumping backstreaming becomes an issue because of the large amount of oil present. This is why many plasma process systems now use ‘dry’ pumps that do not require oil.

Typically the most important ‘leak’ after large external leaks is desorption from surfaces. There are typically two types, first and second order, which are closely related. In order for the material to leave the surface it must have enough energy to overcome the binding energy. (Binding energy can be either physical – via the weak van der Waals force – or a strong chemical bond. This gives rise to physisorbed and chemisorbed.) The fraction of the material trapped on the surface that has enough energy is

$$C = C_0 \int_{E_d}^{\infty} e^{-E/kT} dE$$

where  $C_0$  is the total concentration,  $E_d$  is the desorption energy and  $T$  is the material temperature. Assuming particles with sufficient energy leave the surface immediately, we can determine an equation describing the lose rate from the surface

$$C = C_0 \int_{E_d}^{\infty} e^{-E/kT} dE \Rightarrow C_0 e^{-E_d/kT} \text{ (particles with larger energy have already left)}$$

⇓

$$\frac{dC}{dt} = C(t) e^{-E_d/kT}$$

⇓

$$C(t) = C_0 e^{-E_d/kT} e^{-t/\tau_r}$$

where  $\tau_r$  is the residence time of the particles. (Note that J.O.'s equations are not 'correct' in terms of units. Unfortunately, I have not found a better book with which to replace this discussion.)

Second order desorption occurs when the particle splits into two pieces on the surface. In this case, the two halves must first run into each other AND have sufficient energy to escape. The rate at which the matching parts meet each other is proportional to the square of the concentration of complete particles. (This comes directly from the collision rate of a test particle with a set of background particles,

$$\tau = \frac{\lambda_{mfp}}{v}$$

⇓

$$f = n\sigma v$$

Now accounting for all of the like test particles we find that we have a collision density rate of

$$f = n_{test} n_{fixed} \sigma v.$$

We can now consider method that will speed up the desorption process. All of them are related to increasing the temperature of the surface of the chamber.

Heating

Ion bombardment

Electron bombardment

Photon bombardment

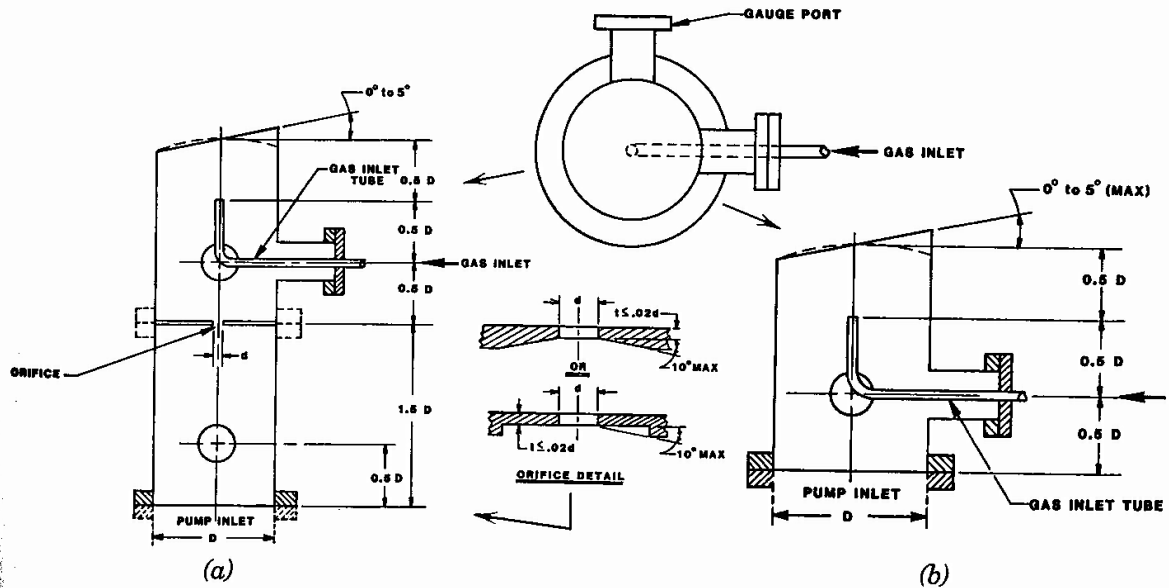
Mass Flow measurements

There are three main methods for mass flow measurements, of which only thermal mass flow is commonly used in low pressure plasma processing.

The two less common flow measurement techniques are Rotameters and differential pressure measurements. Rotameters rely on a ball in a tapered tube that the flow pushes upward. (ADD PICTURE OF ROTAMETER and picture from JO) The gas or liquid then flows around the sides of the ball. Rotameters are only used for very flow rate SLM (Standard Liters per Minute) and are only accurate to approximately 10-20%.

Differential Pressure measurements rely on well characterized long capillary tube(s). Under proper conditions, the flow is directly proportional to the pressure difference across the capillary. Thus the flow rate can be adjusted by raising or lowering the pressure difference. While these are reasonably accurate, they are somewhat difficult to use. (ADD picture from JO)

The most common method of measuring the flow relies on thermal conduction. (ADD paper by Aaron and Sachin)



**Fig. 7.2** AVS high vacuum test domes: (a) orifice method; (b) flowmeter method. Reprinted with permission from *J. Vac. Sci. Technol. A*, **5**, p. 2552, M. Hablanian, Copyright 1987, The American Vacuum Society.

Volumetric pumping speed can be calculated from

$$S = \frac{Q}{P}$$

There are number of ways to get at this. We can use an orifice, such is shown in the AVS test dome, to determine the flow based on the conductance through the orifice. When this is done,  $Q$  is replaced by  $C(P_1 - P_2)$ , where 1 and 2 represent the top and bottom chambers.  $P$  in the above equation is the same as  $P_2$ , giving

$$\begin{aligned} S &= \frac{Q}{P} \\ &= \frac{C(P_1 - P_2)}{P_2} \\ &= C \left( \frac{P_1}{P_2} - 1 \right) \end{aligned}$$

To correct for the baseline pressure  $P_0$  (from outgassing etc.) we subtract it from the pressure in each chamber. This gives

$$S = C \left( \frac{P_1 - P_{10}}{P_2 - P_{20}} - 1 \right)$$

We can also 'measure' the pumping speed if we know two different flows and corresponding pressures. This of course assumes that the speed is reasonably independent of the flow/pressure. (This is an ok estimate if we are not loading the pump significantly.) Then we find that

$$S = \left( \frac{Q_2 - Q_1}{P_2 - P_1} \right)$$



We can use the above equation along with the inward flow, e.g. shutting down the pumping and noting that

$$Q = \frac{d}{dt} PV$$

$$= V \frac{d}{dt} P$$

to arrive at

$$S = V \left( \frac{\frac{d}{dt} P_2 - \frac{d}{dt} P_1}{P_2 - P_1} \right)$$

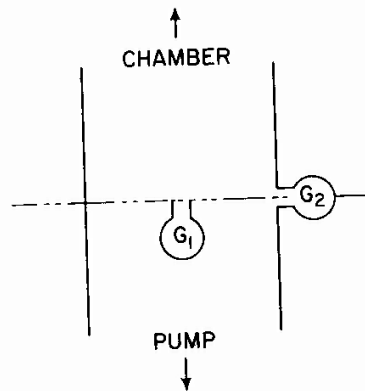
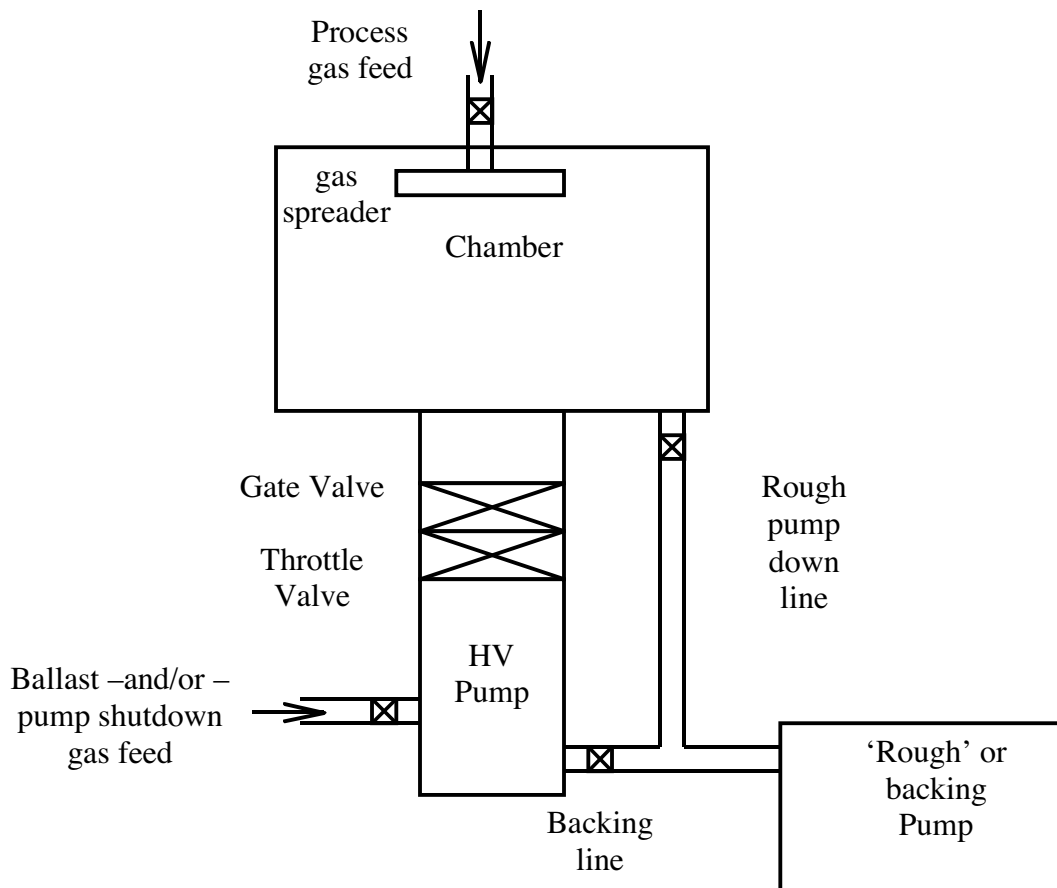


Fig. 7.3 The effect of orientation on pressure gauge readings.

As a final note the direction that the gauge faces, influences the pressure measurement. This is because of the directionality of the molecular flow.

**Building a pump stack**

Building a pumping system that is appropriate for a plasma process chamber is part art part science. In general your pump stack will look like



Chamber materials – must be compatible with process. This is why a lot of new systems are steering away for SS. SS has heavy metals which contaminate Si. The push is toward Al or SiO<sub>2</sub> chambers. Also one must look at how the process effects interacts with the materials and the material costs. For example Cl reacts with Cu – as in Cu gaskets in metal seals. These gaskets can be coated with Nickel and then things are ok. Often corrosive series pumps are coated internally with Nickel.

### Pump sizing

Lower limit on size set by required process gas flow or desired base pressure which is in turn set by chamber size.

Upper limit on pump size is set by cost or by the desire to further reduce backstreaming of oil/gas, e.g. compression ratio of species, at the highest flow rates. At very high flow rates the pumping speed can be reduced by loading of the pump. (Max throughput) If it is than a larger pump is necessary.

Throttle and gas flow can be used to set the pressure in the chamber – somewhat independently.

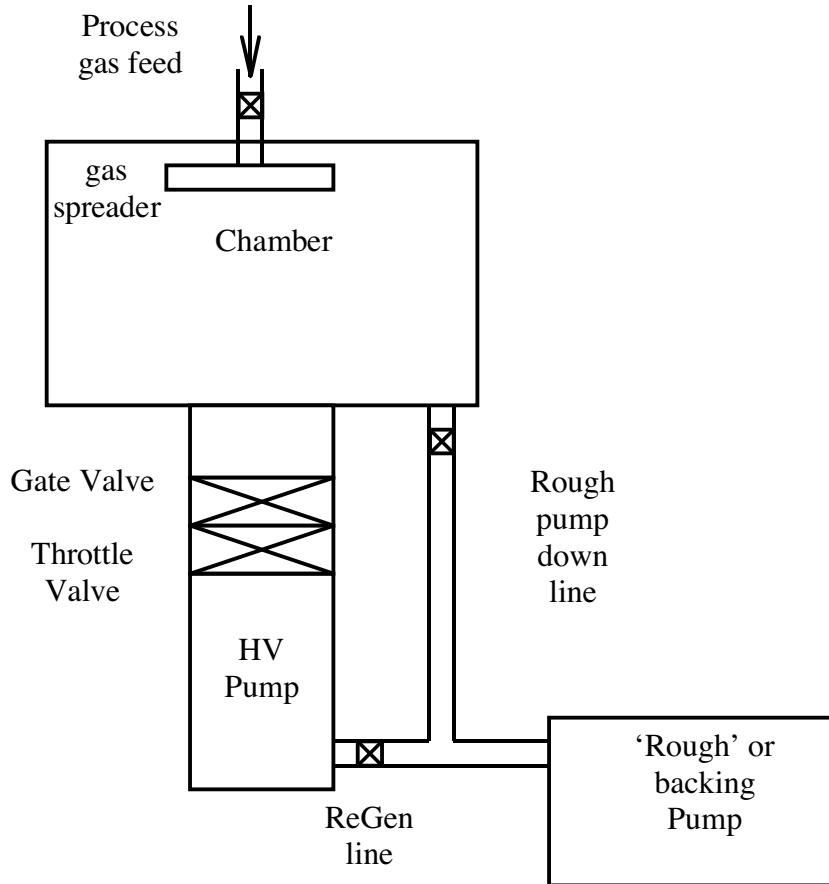
Order of gate /throttle valves to keep throttle valves clean and safe.

Ballast gas to help shut down pumps (Very important for stopping turbo pumps and saving bearings) as well as to reduce backstreaming and reduce problems associated with corrosive gases..

Rough pump line to allow initial pumpdown of main chamber with out going through the HV pump. Rough pump down is never done below a pressure such that you are in molecular flow. (It does not have to be fluid flow.) This is so that the overall gas motion serves to trap – through collisions – any wayward gas molecules.

Gas 'spreader' – to help make the distribution of the fresh process gas more uniform and hence the process more uniform.

The pumping system looks somewhat different for cryopump based systems



The backing line is replaced by a reGen line which is not usually open to the roughing pump. The only time that it is opened is when the Cryo pump is full and the adsorbed gases must be boiled off to reuse the pump.