

# High and Ultra High Vacuum Systems

Presented by:

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# Overview

- High Vacuum
  - Concerns
  - Pumps
  - Quick Solutions
- Ultra High Vacuum
  - Concerns
  - Gas Behavior
  - Cleaning and Handling
  - Pump Adjustments
  - Gauges
- Exercises

# High Vacuum Systems

- Pressure range
  - $10^{-6}$  to  $10^{-8}$  torr
- Applications
  - Electronic Devices
    - Depositions (sputtering, evaporation, pulsed laser)
    - Plasma Etching
    - Ion Implantations
  - Non-surface material characterization (EDS, SEM, etc...)

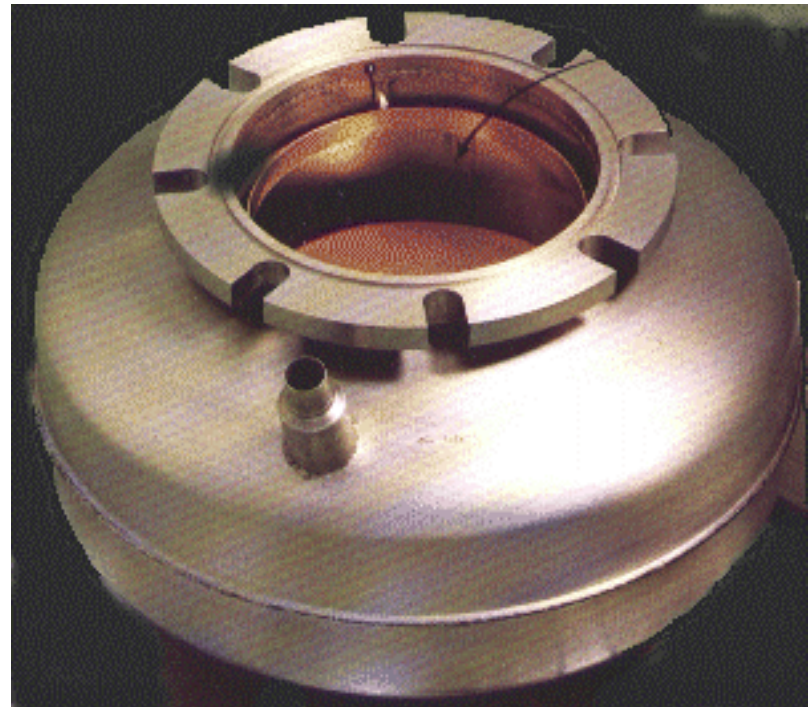
# High Vacuum Systems

- Pumping Options
  - Diffusion
  - Turbomolecular
  - Ion
  - Cryogenic
- Major Concerns
  - Outgassing (major concern water contamination)
  - Small leaks
- How can we solve these problems???
  - Combine and optimize various portions of the class
    - **Choose pump to meet desired needs**
    - **He leak detection**
    - **Minimize outgassing**

# Cold Traps

## Introduce a cold trap into your system:

- Prevents back streaming of oil from diffusion pump when cold traps run out of liquid nitrogen, with no loss in conductance.
- Fill the 2" cold trap with 3 liters of liquid nitrogen in the morning and run all day without refilling.



# High Vacuum Systems Wrap-Up

- Outgassing is primary source of gas load
  - Diffusion and permeation are a small part of the total gas load.
- Ionizing gauges can be used in HV effectively from  $10^{-6}$ - $10^{-10}$  Torr.
- LN cold traps, filters
- High grade oil where needed
- No single right answer
  - Use a system that meets your needs

# UHV - Systems

- Modern Applications
  - Fundamental Particle Studies
  - Fabrication of Magnetic Read/Write Heads
  - Numerous Semiconductor Processes
  - Opto-Electronic Device Processing
  - Material Surface Characterization

**Ultra High or Ultra Clean Vacuum Systems?**

# UHV - Systems

## Defining a Ultra High Vacuum

<b>Pressure (Torr)</b>	<b>Mean Free Path (cm)</b>	<b>Monolayer Formation time (sec)</b>
Atmosphere 760	3.9E-6	1.7E-9
High $10^{-3}$	5.1	2.2E-6
High $10^{-6}$	5.1E3 (160 feet)	2.2
Ultra $10^{-9}$	5.1E6 (31 miles)	2.2E3 (37 min)
Ultra $10^{-12}$	5.1E9 (very long)	2.2E6 (25.5 days)



# UHV - Systems

## Major Concerns:

- Outgassing, Permeation and Diffusion
- Ultra clean surfaces
- Incorrect data read out (gauges?)
- Pump type and usage
- Process feed throughs (electrical, translational, etc...)

## Gas Constituents

Low Vac.	High Vac.	Ultra High Vac.
Air (N <sub>2</sub> , O <sub>2</sub> )	80% H <sub>2</sub> O(g) 20% N <sub>2</sub> , H <sub>2</sub> , CO <sub>2</sub>	H <sub>2</sub>

# UHV – Outgassing, Permeation and Diffusion

## Outgassing Load:

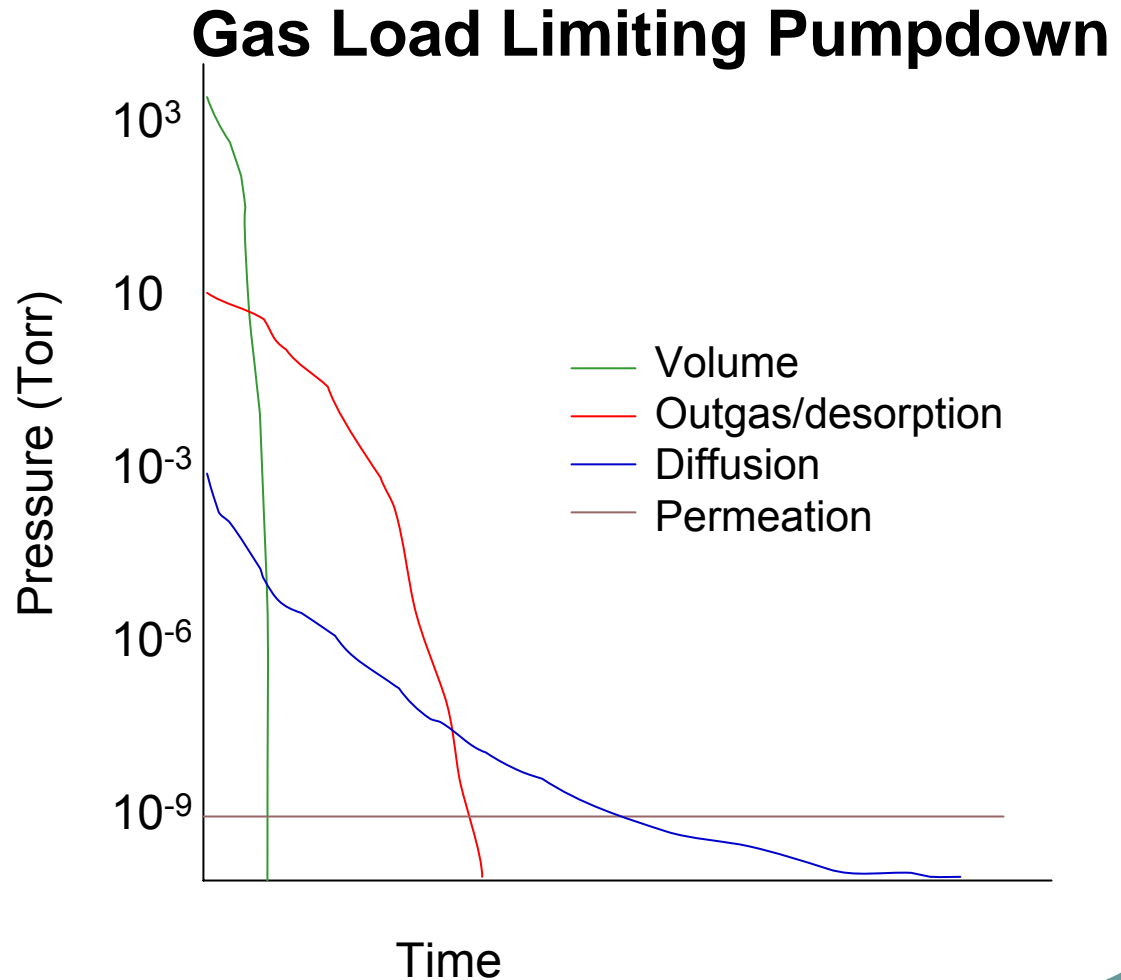
Gases originate at surfaces

## Permeation Load:

Gases originate outside of vessel

## Diffusion Load:

Gases originate inside of vessel walls



# UHV – Outgassing

## Outgassing Rate (q):

$$Q_{\text{outgas}} = q_{\text{outgas}} * \text{Area}$$

- Factors Include:
  - Material
  - Surface treatments
  - Temperature
  - Time.

Material	Outgas Rate (Torr L/s cm <sup>2</sup> )	UHV OK ?
Brass	10 <sup>-6</sup> -10 <sup>-7</sup>	No
Stainless Steel	10 <sup>-8</sup> – 10 <sup>-13</sup>	Yes
Copper	10 <sup>-9</sup> – 10 <sup>-12</sup>	Yes
Polymers	10 <sup>-5</sup> -10 <sup>-7</sup>	No
Aluminum	10 <sup>-8</sup> -10 <sup>-13</sup>	Yes

# UHV - Diffusion

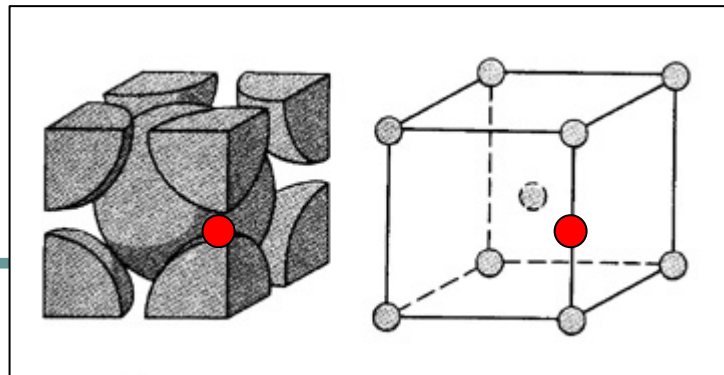
- Gas load contributions by diffusion are similar to permeation however the gaseous species are present intrinsically to the vacuum material.
- Diffusion coefficient is a function of temperature.

$$D = D_0 \exp(-E_a/k_B T)$$

## Case Study:

While processing steel, H<sub>2</sub> impurities sit on the interstitial sites in the Fe lattice. Baking above 400 C will drive H<sub>2</sub> towards the walls of the chamber.

$\alpha$ -Iron BCC Lattice  
with H<sub>2</sub> Interstitial



# UHV – Permeation

- Dependent on type of gas and material.

Examples:

- Stainless Steels - higher permeation rates with higher carbon content
- Copper - low permeation rates for all gases
- Polymers - permeable to all gases

## Case Study:

H<sub>2</sub> has a high solubility in most metals and is thought to permeate through the walls of the chamber.

**Decrease permeation by choosing different materials.**

# UHV – Ultra Clean Surfaces

## **Motivation:**

Assure low outgassing of material under vacuum.

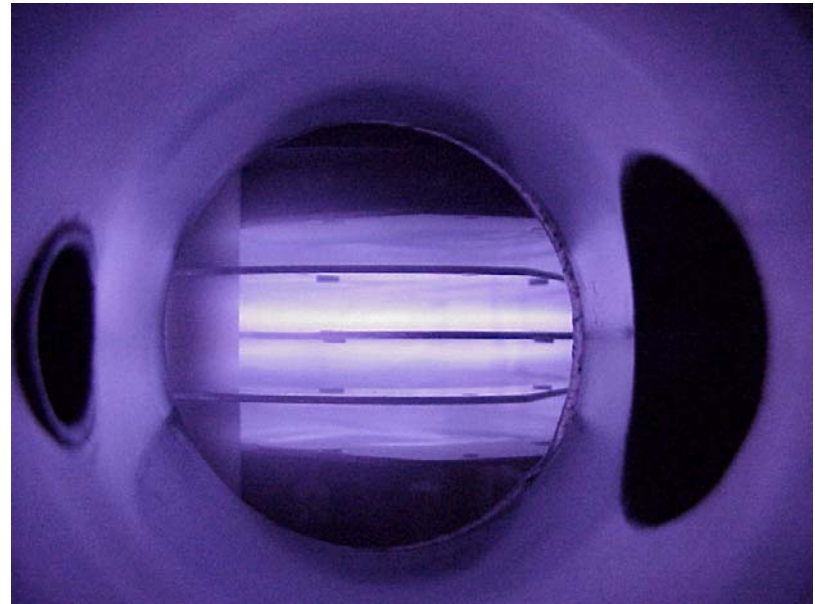
## **Methods:**

Bead Blasting

Glow Discharge (top right)

Reactive Gas Clean

Rough Abrasives



Ultrasonic

Electropolishing

Chemical (Acid and Alkaline)

# UHV – Ultra Clean Surfaces

- **Baking Options**
  - Temporary Heat Enclosure
    - Temperatures up to or exceeding 400 °C
  - Vacuum Oven (right)
    - Temperatures of up to 900 °C



# UHV – Ultra Clean Surfaces

## **Cleaning method for 300 series stainless**

**(Brookhaven National Laboratory)**

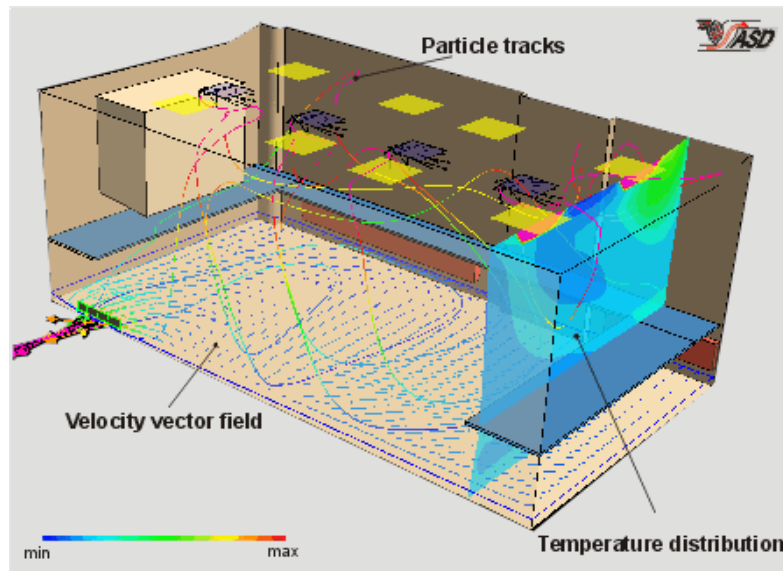
1. Degrease with ultrasonic agitation
2. Hot alkaline pH 11 wash with air agitation
3. Rinse cold tap water
4. Rinse DI water with air agitation
5. Dry in hot air oven
6. Vacuum bake at 950 C for 2h
7. Glow discharge clean
8. Reactive gas clean



# UHV – Material Handling

## Cleaned Parts can be Recontaminated

- Unpowdered latex gloves
- Wrap in lint free-paper or wax-free aluminum foil
- Certification for clean room use



Example WCAM: Class  
100

<100 Particles/ft<sup>3</sup>  
Particle: Dust, Air Borne  
Moisture,  
“Killer Particles”

# UHV - Pumps

## Turbo Pump Adjustments:

- Must bake a minimum of 100 °C to get rid of water vapor
- Must be magnetically levitated
- Two in series will increase the compression ratio of H<sub>2</sub>
- Oil free scroll pump should be used for backing
- Lower limit of 10<sup>-9</sup> torr at best, will need a capture and/or cryopump to get lower

Backstreaming not acceptable  
for pressure below 10<sup>-9</sup> Torr



# UHV - Pumps

## Cryopump Adjustments:

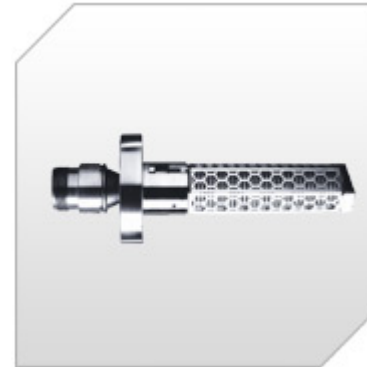
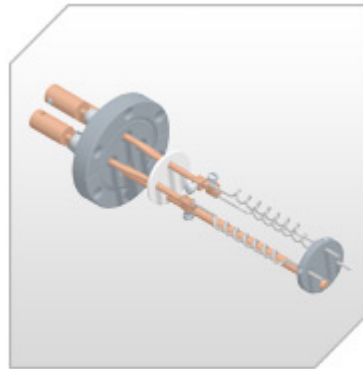
- Cannot bake over 100 C (indium gaskets)
- Must change Viton sealed overpressure relief valves to knife edge.
- Displacer generates some vibration
  - Might cause carbon adsorber to release dust
- Cool 2<sup>nd</sup> stage with a Er<sub>2</sub>Ni heat exchanger
  - The stage can then reach a temperature of 3.6K



# UHV – Pumps

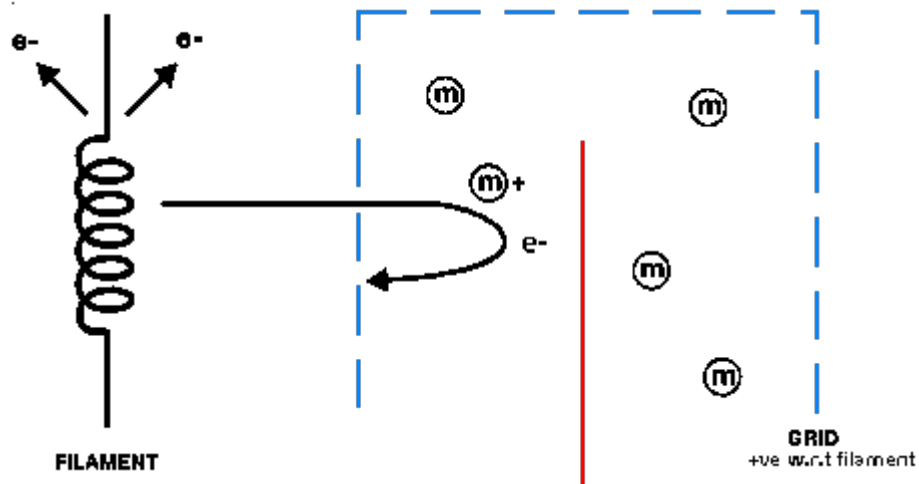
## Capture Pumps (NEG, TSP, SIP):

- Use multiple types of pumps
  - SIP can effectively pump noble gases
  - NEGs have relatively high pumping speeds for  $H_2$
  - $H_2$  pumping is improved in TSP if the surface was LN cooled instead of water cooled



# UHV - Gauges

- **Below  $10^{-6}$  Torr**
  - RGA and Ionization gauges must be used
- **Below  $10^{-10}$  Torr**
  - Gauges fight issues of ESD and electrode outgassing
  - Limited number of commercially available UHV gauges.



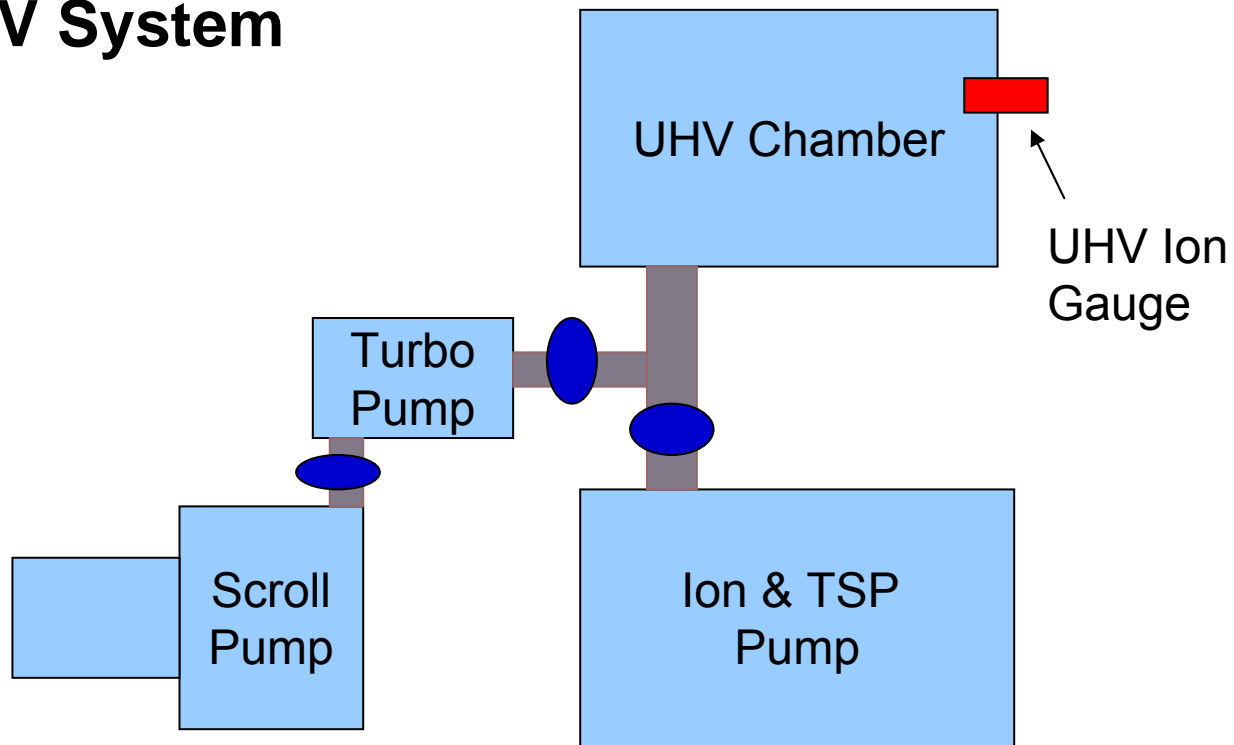
Principle of Ion Gauge



Residual Gas Analyzer

# UHV – Example System

## A Basic Simplified UHV System



# UHV – Example System

## System Parameters:

- **Scroll Pump connected to chamber through a KF-40, 3 feet (91 cm) long line. Diameter KF-40: 4.0 cm**
- **Turbopump and Ion Pump connected to chamber through a 6-inch (15.2 cm) diameter, 18-inch (45.7 cm) long line.**
- **Rough systems to  $5 \times 10^{-2}$  torr with the scroll pump; pump with turbo till  $1 \times 10^{-6}$ , then valve in ion pump. Shut off turbo at  $1 \times 10^{-9}$  torr?**
- **Chamber: Stainless Steel; Length: 3 feet, diameter 2 feet volume: 267 liters: surface area  $2.33 \times 10^4$  (cm<sup>2</sup>)**
- **Roughing Pump: Speed: 10l/s (atm. to 1 torr); 8.5l/s at 100 mtorr**
- **High Vacuum Pumps: Turbopump with 500 l/sec speed Ion Pump with 400 l/sec speed**

# UHV – Example System

**Scroll Pumping time:**

$$t := c \cdot \frac{V}{S} \cdot \ln\left(\frac{P_i}{P_f}\right) \quad S_T := S_{\text{pump}}$$

**From Atmosphere to  $5 \times 10^{-2}$  torr:**

$$t = 278 \text{ s}$$

**Turbo Pumping Time:**

$$t := c \cdot \frac{V}{S} \cdot \ln\left(\frac{P_i}{P_f}\right) \quad \frac{1}{S_T} := \frac{1}{S_{\text{pump}}} + \frac{1}{C_T}$$
$$S_T := 285 \frac{\text{L}}{\text{s}}$$

**From  $5 \times 10^{-2}$  torr to  $1 \times 10^{-6}$  torr:  $t = 8.6 \text{ s}$**



# UHV – Example System

What is the maximum outgassing rate allowed to achieve  $1 \times 10^{-9}$  torr?

$$Q_{\text{outgas}} := q_{\text{rate}} \cdot A$$

$$Q_{\text{outgas}} := S_T \cdot P$$

$$q_{\text{rate}} := \frac{S_T \cdot P}{A}$$

$$q_{\text{rate}} := 1.223 \cdot 10^{-11} \frac{\text{torr} \cdot \text{L}}{\text{s} \cdot \text{cm}^2}$$

# UHV Example Data – Stainless Steel Outgassing

Method	Outgassing Rate (torr*L/sec*cm <sup>2</sup> )				
	1 hour	5 hours	10 hours	20 hours	40 hours
<b>Sand Blasted</b>	$9.0 \times 10^{-9}$	$3.0 \times 10^{-9}$	$1.0 \times 10^{-9}$		
<b>Mechanically Polished</b>	$4.0 \times 10^{-9}$	$1.5 \times 10^{-9}$	$4.0 \times 10^{-10}$	$3.0 \times 10^{-10}$	$1.2 \times 10^{-10}$
<b>Electropolished</b>	$1.7 \times 10^{-9}$	$9.0 \times 10^{-10}$	$4.0 \times 10^{-10}$	$2.0 \times 10^{-10}$	$8.0 \times 10^{-11}$
<b>H2 Fired pipe</b>	$2.5 \times 10^{-10}$	$1.5 \times 10^{-10}$	$1.0 \times 10^{-10}$	$8.0 \times 10^{-11}$	
<b>Chem Cleaned pipe</b>	$2.0 \times 10^{-9}$	$3.5 \times 10^{-10}$	$4.0 \times 10^{-11}$	$1.2 \times 10^{-11}$	
<b>Glass Bead Honed Pipe</b>	$1.5 \times 10^{-10}$	$2.5 \times 10^{-11}$	$1.1 \times 10^{-11}$	$8.0 \times 10^{-12}$	$3.0 \times 10^{-12}$

# UHV Example Data – Pumpdown Pressure

Method	Pressure in Torr			
	H <sub>2</sub>	H <sub>2</sub> O	CO	Total
<b>Pumped Under Vacuum for 75 hours</b>	<b>6.2 x 10<sup>-9</sup></b>	<b>4.0 x 10<sup>-9</sup></b>	<b>6.0 x 10<sup>-10</sup></b>	<b>1.1 x 10<sup>-8</sup></b>
<b>.....then baked out for 50 hours (150°C)</b>	<b>2.7 x 10<sup>-9</sup></b>	<b>1.2 x 10<sup>-10</sup></b>	<b>4.2 x 10<sup>-11</sup></b>	<b>2.9 x 10<sup>-9</sup></b>
<b>.....then baked out for 40 hours (300°C)</b>	<b>5.7 x 10<sup>-10</sup></b>	<b>4.8 x 10<sup>-12</sup></b>	<b>1.6 x 10<sup>-11</sup></b>	<b>5.9 x 10<sup>-10</sup></b>
<b>.....then baked out for 40 hours (400°C)</b>	<b>1.3 x 10<sup>-10</sup></b>	<b>2.0 x 10<sup>-12</sup></b>	<b>3.1 x 10<sup>-12</sup></b>	<b>1.4 x 10<sup>-10</sup></b>

# Questions

**1. What types of pumps can you use to reach vacuums of  $10^{-10}$  torr or less?**

Answer: SIP, TSP, NEG, Cryopump (Most be capture)

**2. Where can oil be used in an UHV system?**

Answer: No where

**3. What is the most effective method of removing hydrogen from the stainless steel walls?**

Answer: Bake at high temperatures (above 400 °C) Diffusion coefficient is exponentially proportional to temperature

# Questions

**4. If someone went through the trouble of fully cleaning a vacuum chamber component and then had lunch (McDonalds). On return he brushed the component with his/hers fingers before putting gloves on. Would that system ever reach a UHV pressure?**

Answer: Yes, the gas load from outgassing will decrease with pumping time. Remember the gas load from permeation is the only constant.

**Additional Questions?????**