This educational seminar discusses creating, measuring, and troubleshooting **Ultra-High Vacuum** *(≈ $10^{-8} \rightarrow 10^{-12}$ Torr)* systems.

Specifically, today's talk will cover:

- Brief review of *High Vacuum* (Characteristics, Pumps & Gauges)
- Applications requiring Ultra-High Vacuum
- Ultra-High Vacuum Gauges
- Cryo-Pumps & UHV Pumps (detailed description of **Diode & Noble Diode Sputter Ion Pumps**)
- Challenges in achieving UHV Pressures

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Previous Webinars (***Vacuum Fundamentals, Rough Vacuum, High Vacuum***) are available for download at:

http://www.agilent.com/en-us/training-events/eseminars/vacuum

High Vacuum Review: Characteristics

Particles are moving in *Molecular Flow*

- *Long Mean Free Path (MFP)*
- *Few collisions with other molecules*
- *Large pump inlets (high conductance) key to achieving efficient pumping*
- *Gas Composition is constant through High Vacuum (80% H_2O, 10% N_2, 10% CO)*

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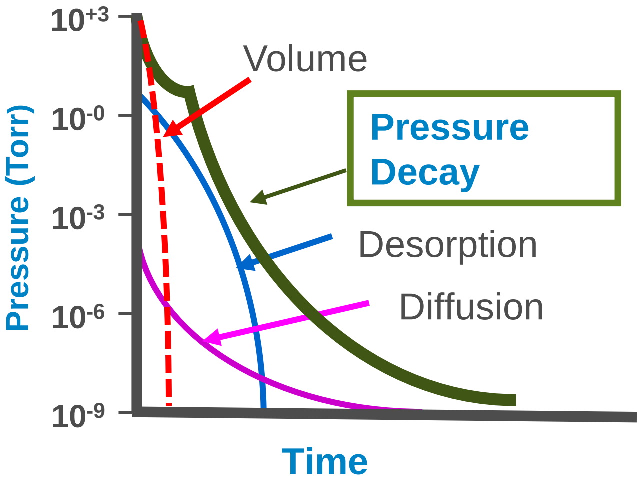
Chamber Volume is NOT the dominant factor governing pumpdown time

- *Surface Area, Material Type and Pump Speed determine ultimate pressure*



High Vacuum Review: Pumps and Gauges

Two types **High Vacuum** Pumps**:**

- ‘**Capture**’ pumps (Cryo-Pump)
- ‘**Displacemen**t’ pumps (Turbo-Pump & Diffusion Pump)
- No High Vacuum Pumps can compress gas to Atmosphere!

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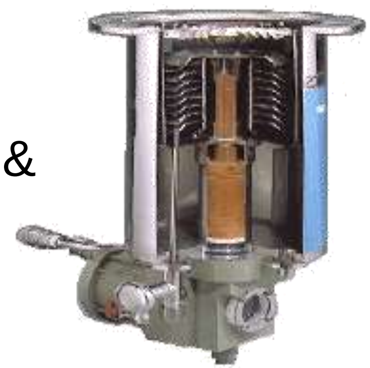
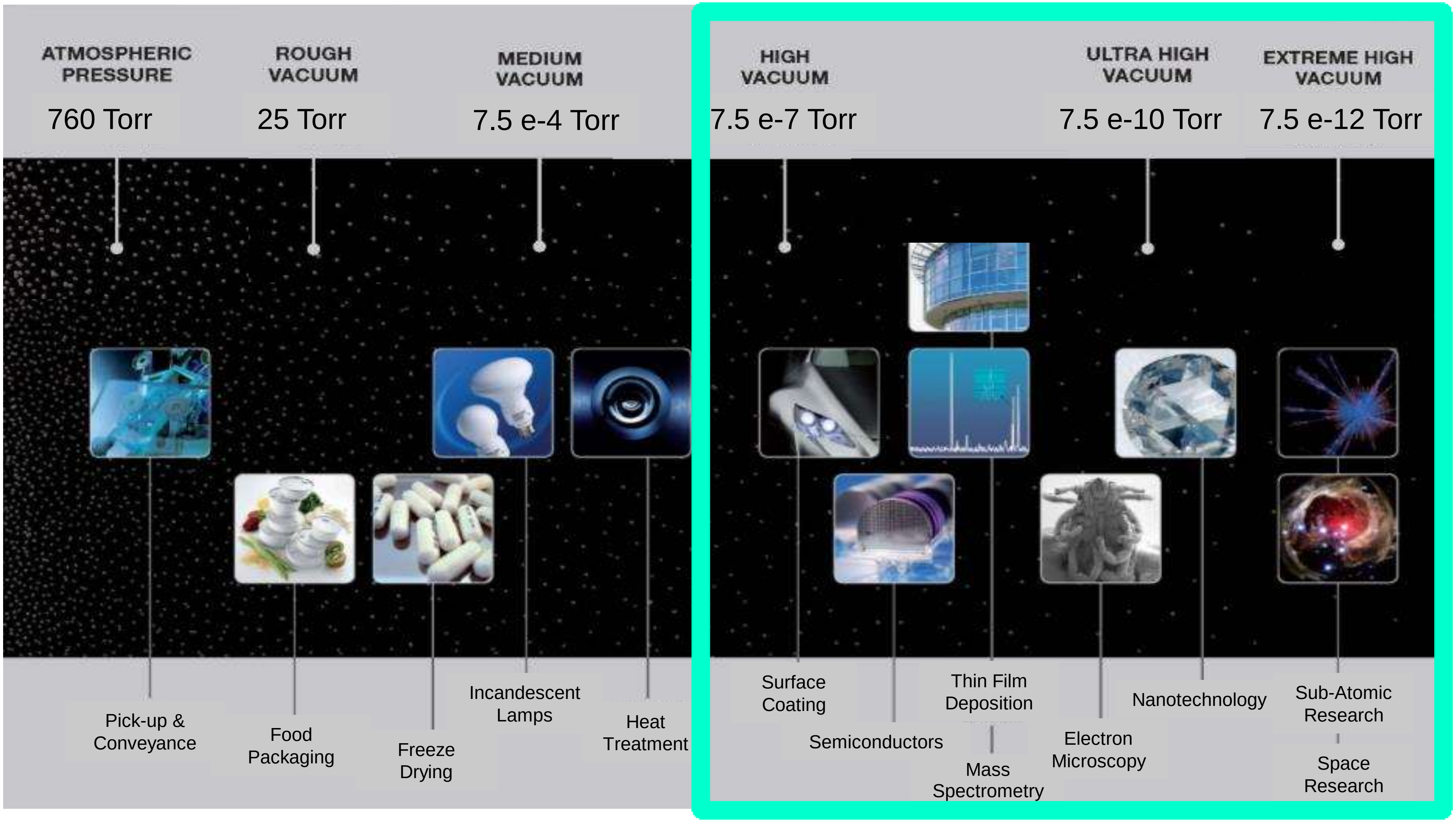


IONIZATION GAUGES used to measure High Vacuum pressures

- Hot Ionization Gauge (BA)
- Cold Cathode & IMG Gauges
- ‘Wide Range’ gauges use multiple technologies in a single housing (e.g. IMG & Pirani).

UHV Applications



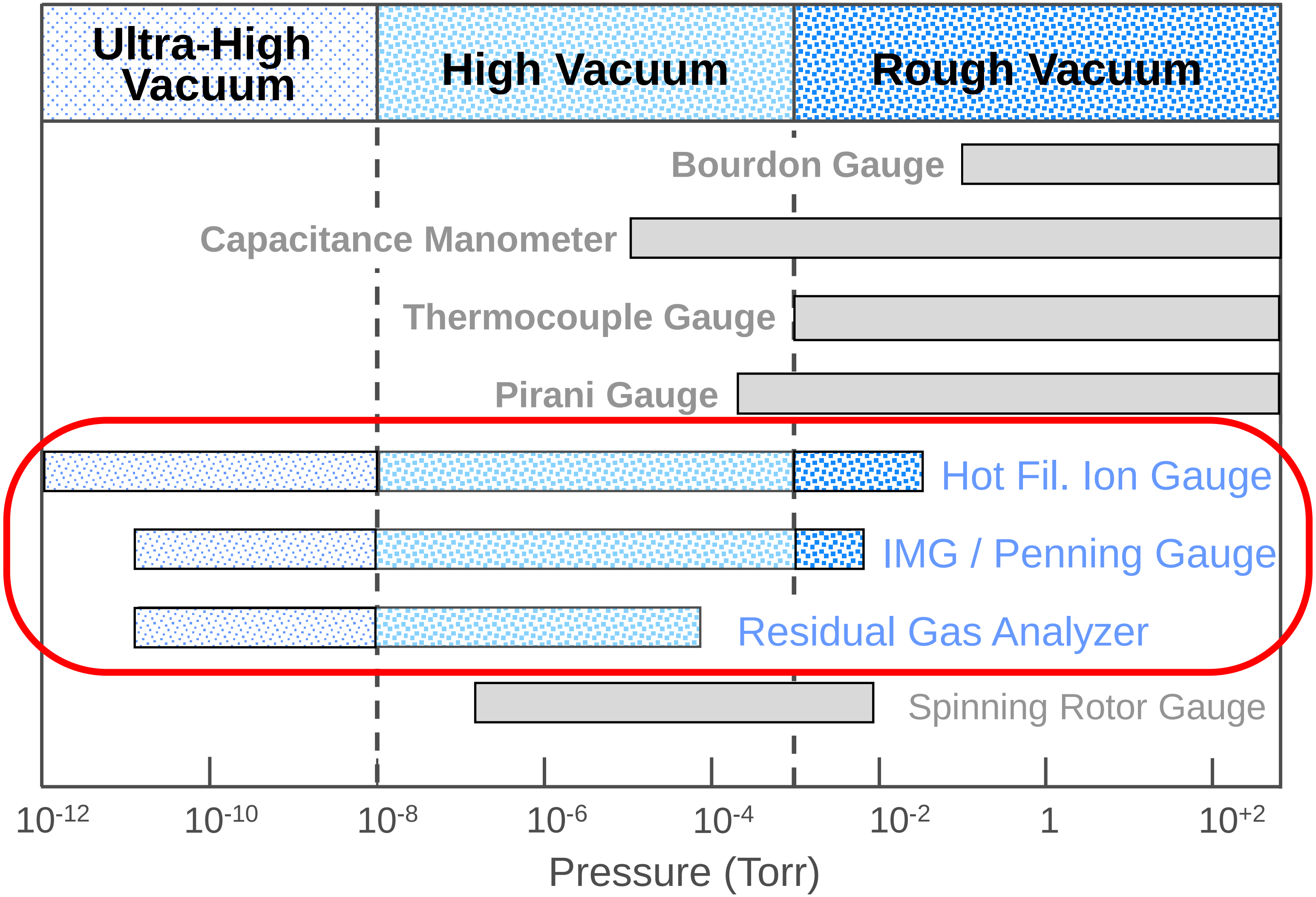
Vacuum Measurement Technologies

- Different technologies are required to measure the vacuum pressure in different vacuum regions

RANGE		GAUGE TYPE	EXAMPLES
Rough Vacuum	*Atm - 10^{-3}*	*Mechanical Deflection & Thermal Transfer Gauges*	*Bourdon Gauge Capacitance Manometer Thermocouple, Convection, Pirani*
High Vacuum	*10^{-3} - 10^{-9}*	*Mechanical Deflection & Ionization Gauges*	*Capacitance Manometer Hot Filament Gauge (BA) IMG / Penning Gauge*
Ultra High Vacuum	**< 10^{-10}**	**Ionization Gauges & Gas Analyzers**	**UHV Ionization Gauges Residual Gas Analyzer (RGA) Ion Pump Current**

https://cds.cern.ch/record/455555/files/p75.pdf

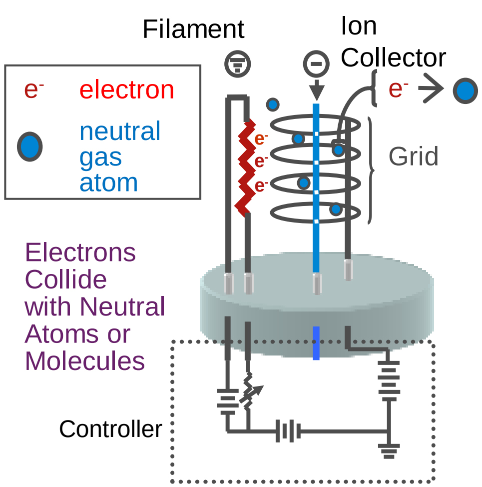
Ultra-High Vacuum Gauges



Modified Hot Filament Ion Gauge

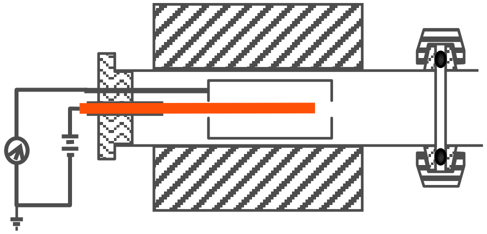
How it Works:

- Overcome Glass Permeation and 'X-ray Limit' (measurement inaccuracy caused by X-rays striking central filament)
  - *No Glass Envelope ⇒ eliminate permeation 'leak' through glass*
  - *Smaller Structures ⇒ reduce measurement error caused by X-rays*
  - *All-metal Seals ⇒ eliminate permeation through o-ring*
  - *Accuracy: ± 20% of full scale within a pressure decade*
- Can read to 5 x 10^{-12} Torr vs 3 x 10^{-10} Torr for Glass Envelope BA



Gas	Relative Sensitivity
Ar	1.2
CO	1.0 – 1.1
H_2	0.40 - 0.55
He	0.16
H_20	0.9 – 1.0
N_2	1.00
Ne	0.25
O_2	0.8 – 0.9

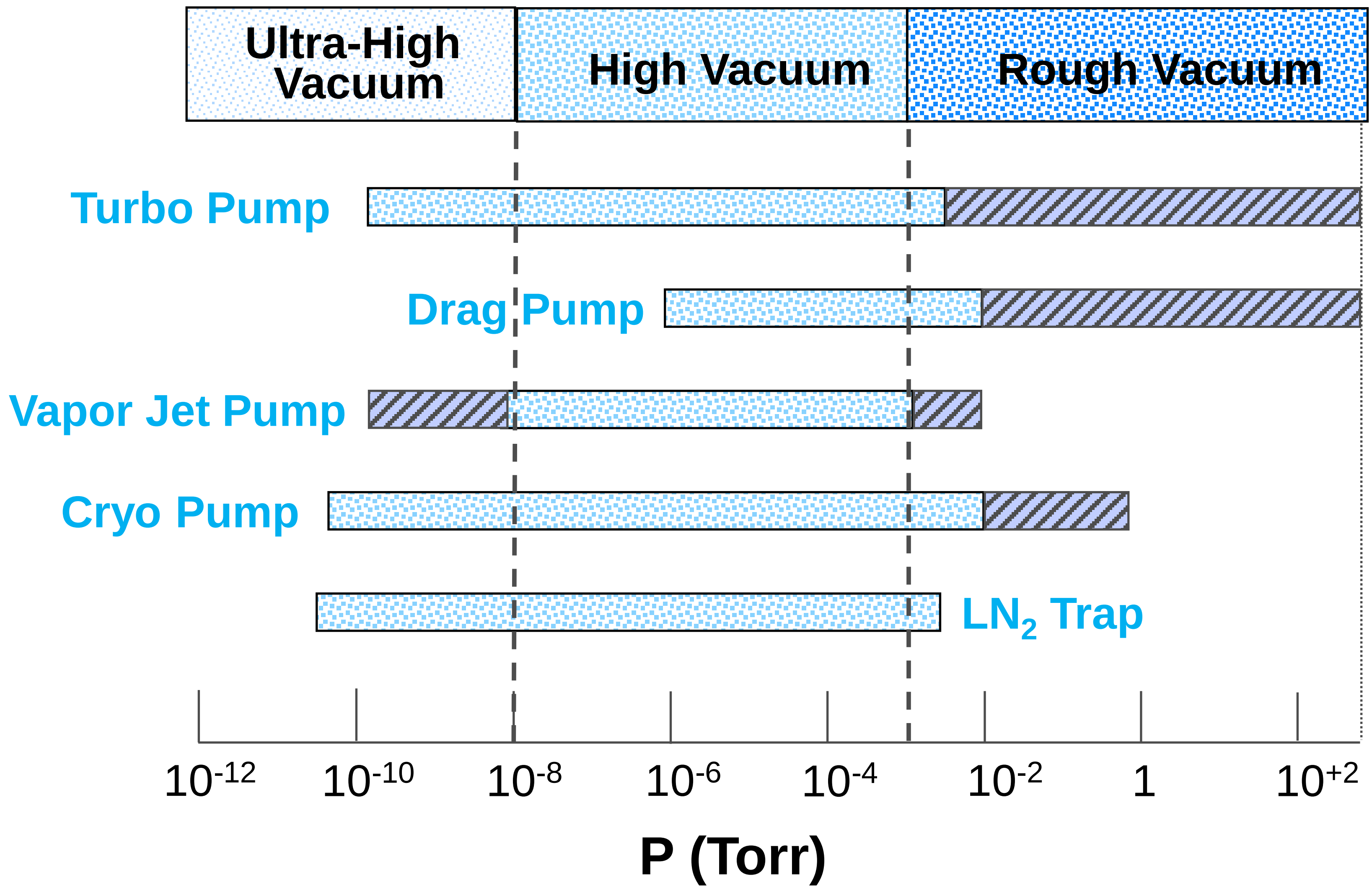
Inverted Magnetron Gauge



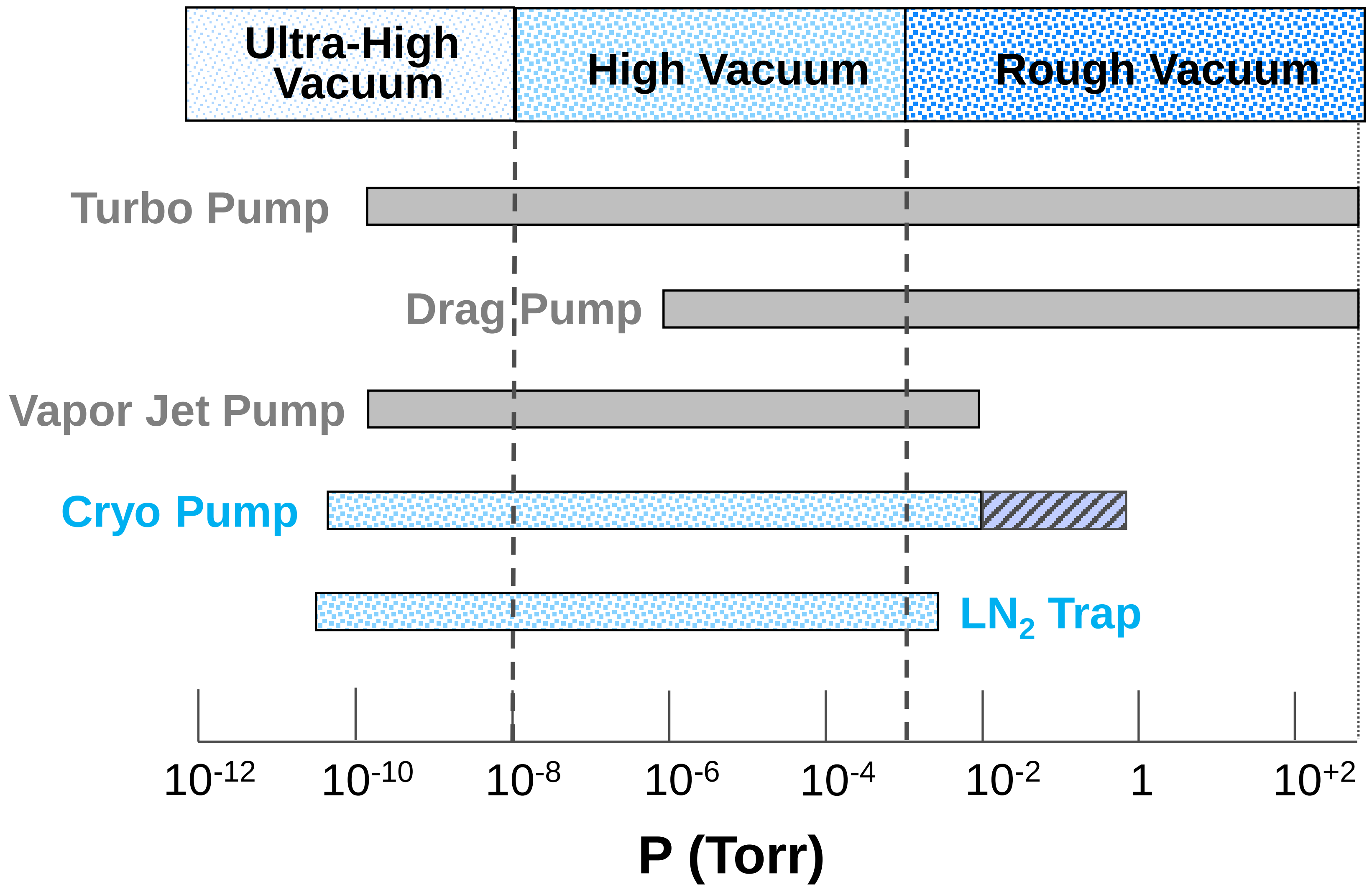
How it Works:

- Anode is a rod in the axis of an almost closed cylinder (Cathode)
- End discs of the of the cathode are shielded guard rings
  - *Prevents field emission affecting ion current measurement*
- Anode at ~4 kV vs -2 kV for Magnetron and is parallel to the gauge's magnetic field.
  - *Superior starting characteristics (vs Cold Cathode Gauge)*
  - *Stable Pressure reading and Fast Response*
  - *10^{-3} to 10^{-11} Torr Operating Range*
  - *Accuracy: ± 50% within a pressure decade*

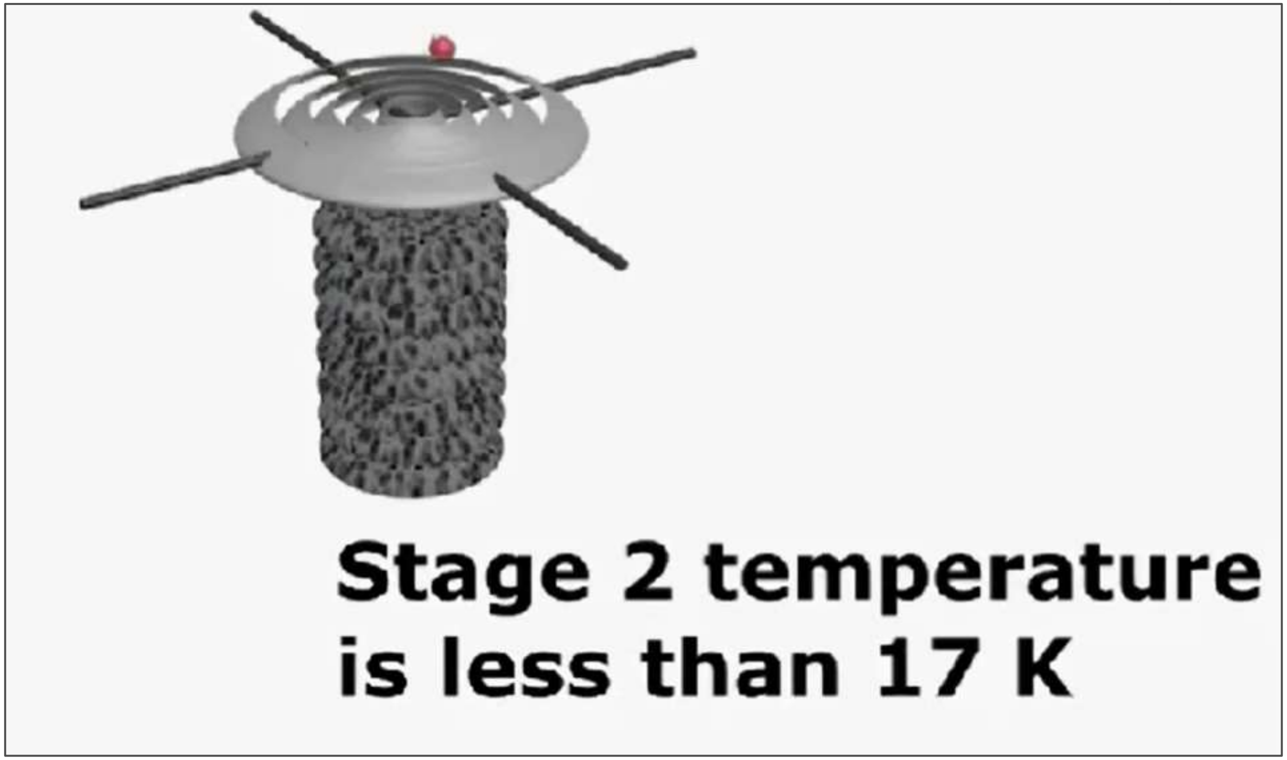
High Vacuum Pumps: Operating Range



High Vacuum Pumps: Operating Range

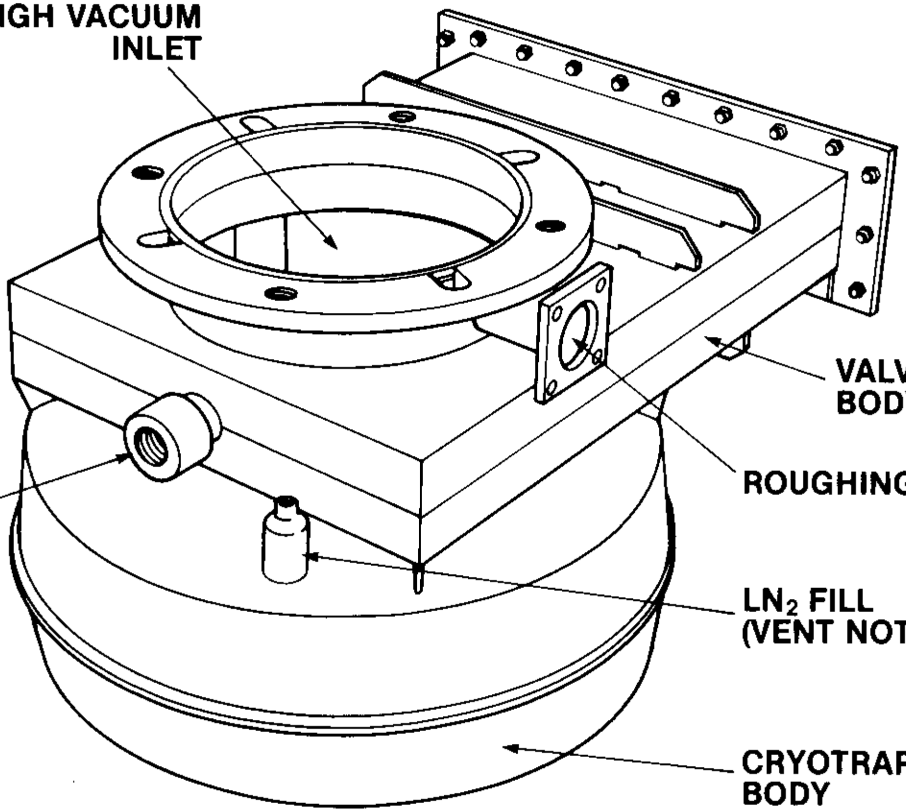


Cryogenic Pump Operation: UHV



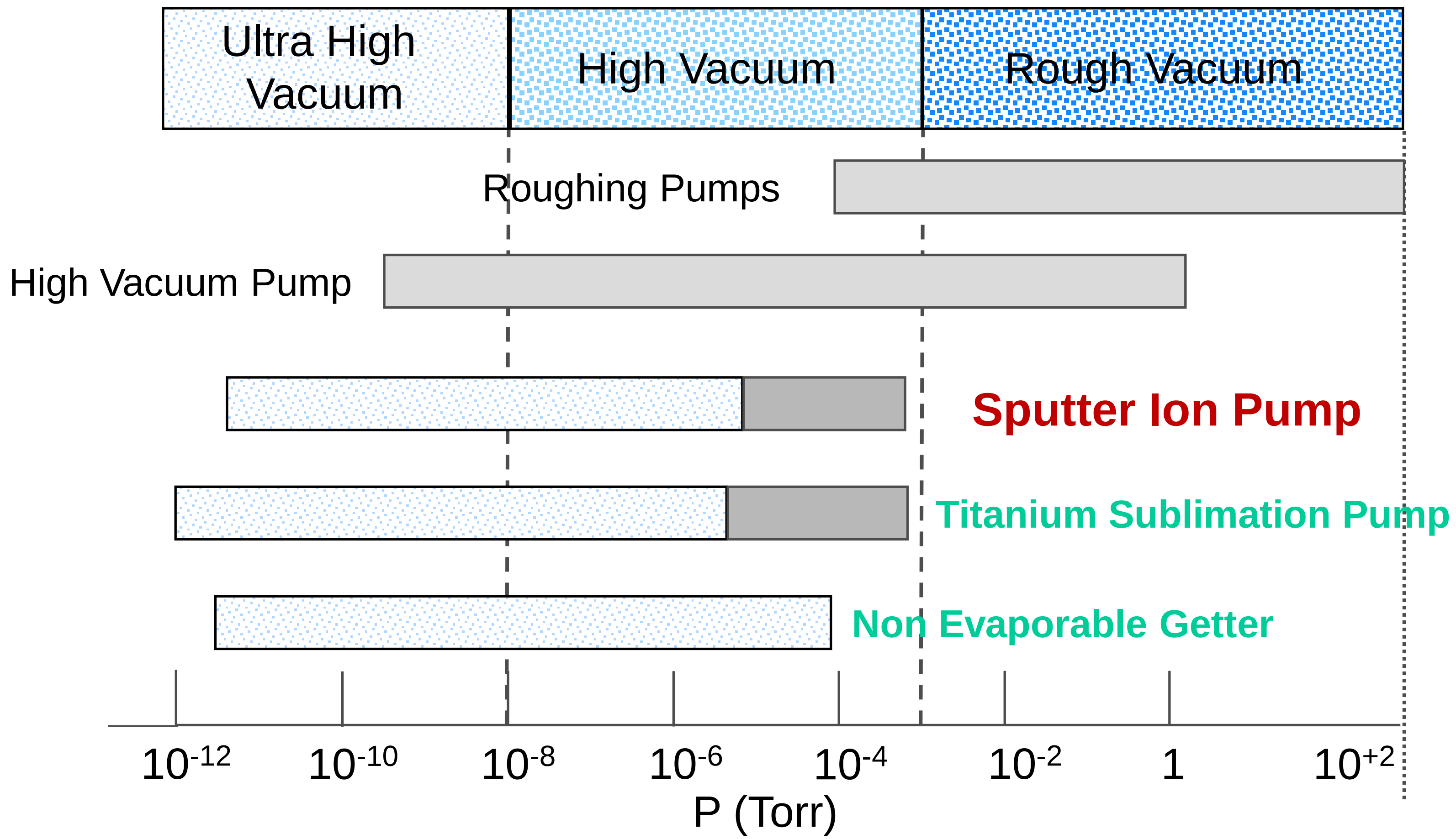
All CryoPumps require REGENERATION when Cold Heads become 'saturated' (drop in pumping speed/increase in 2nd stage temperature)

PASSIVE Cryogenic Trap (w/ Valve)



UHV Pumps

UHV Pump Operating Ranges



Sputter Ion Pump: Anatomy of a Diode Element

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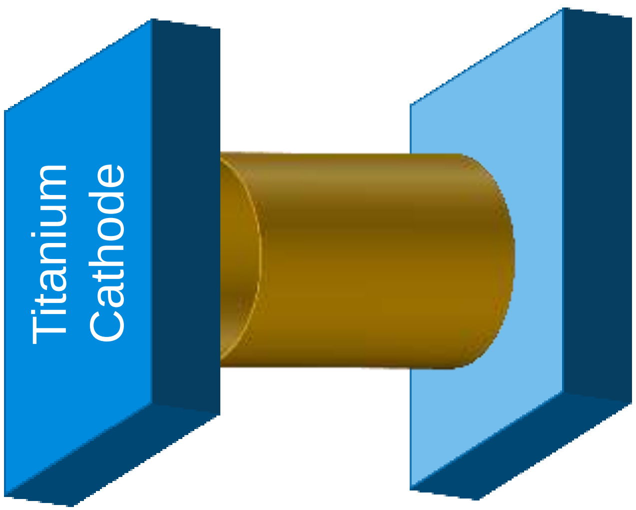
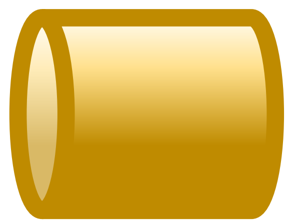


Single Cell Diode Pump Element

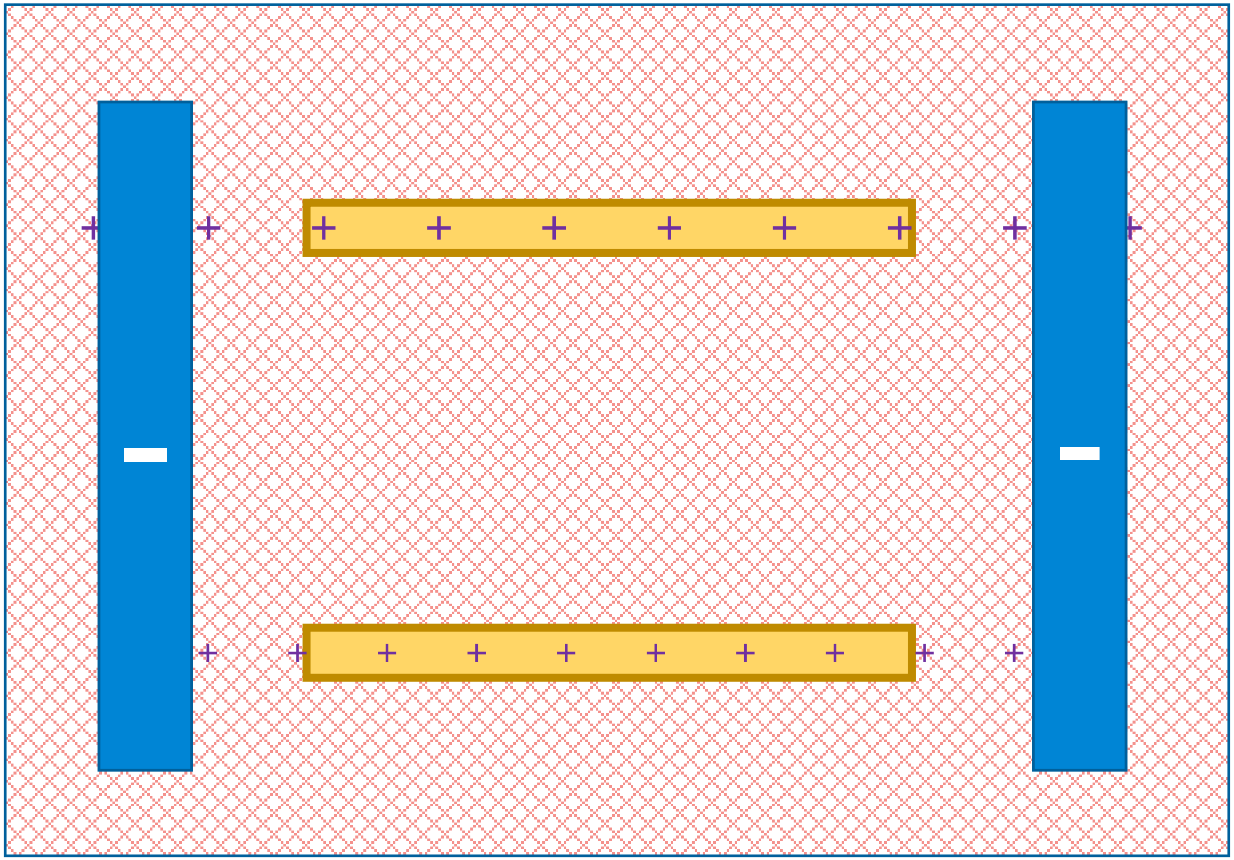


Stainless Steel Anode

Anode Cross Section

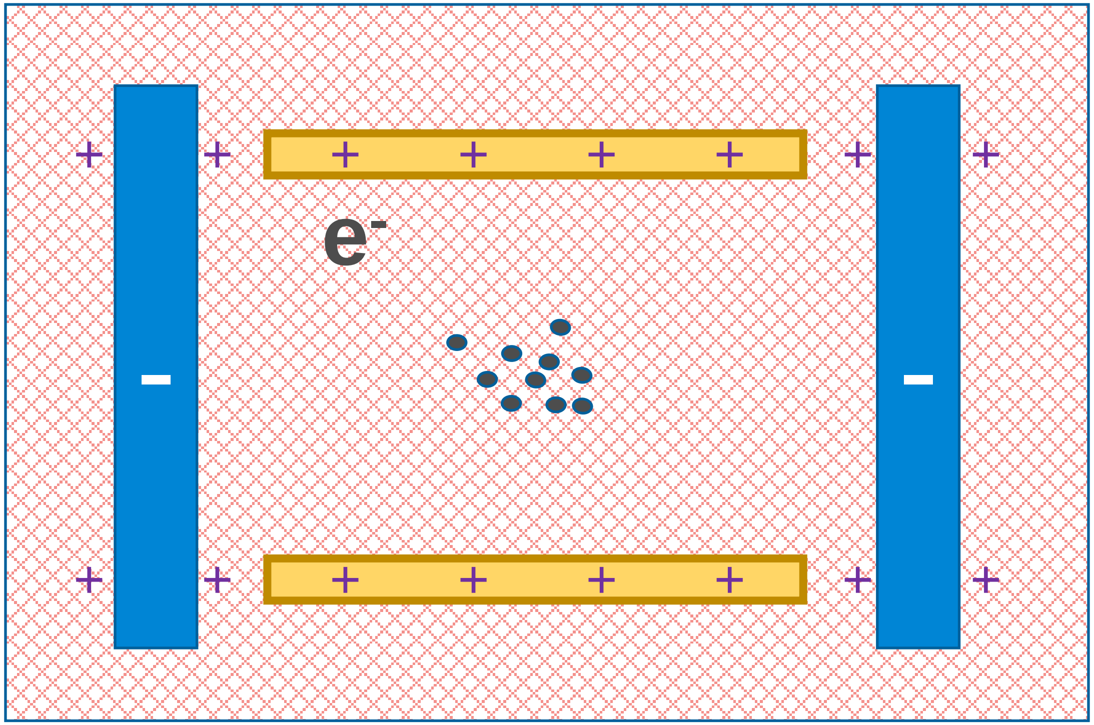
https://cds.cern.ch/record/454179/files/p37.pdf

Cross-section of a Diode Cell



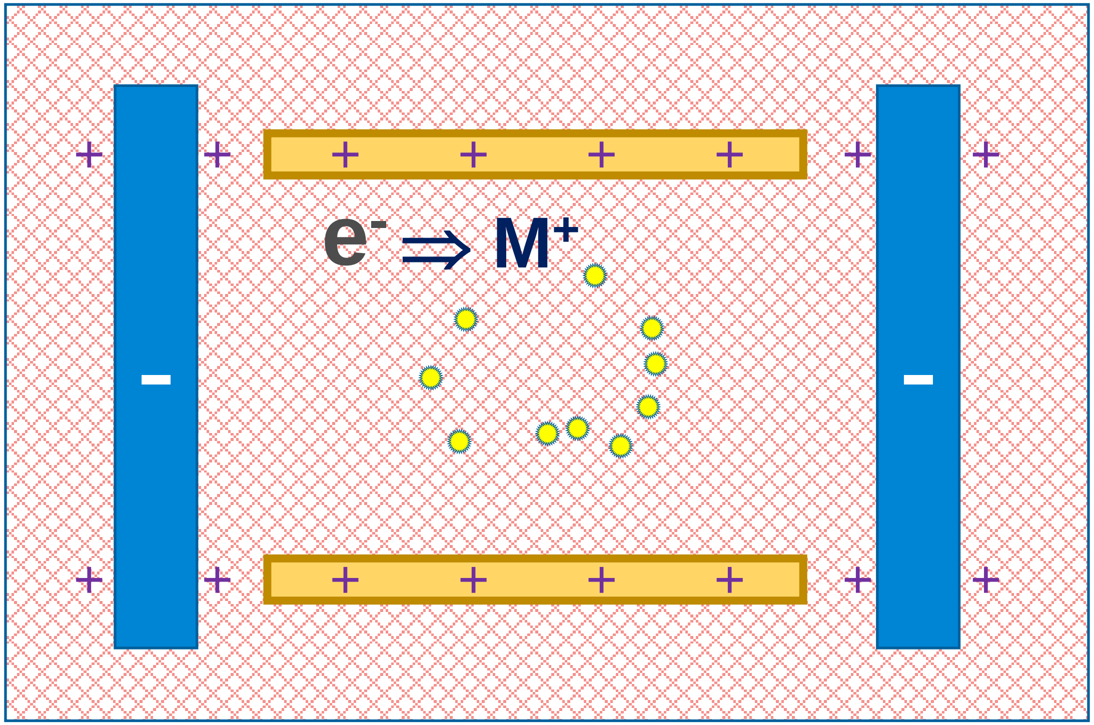
X = Magnetic Field INTO the Page

Voltage Potential Creates Plasma (Free Electrons!)



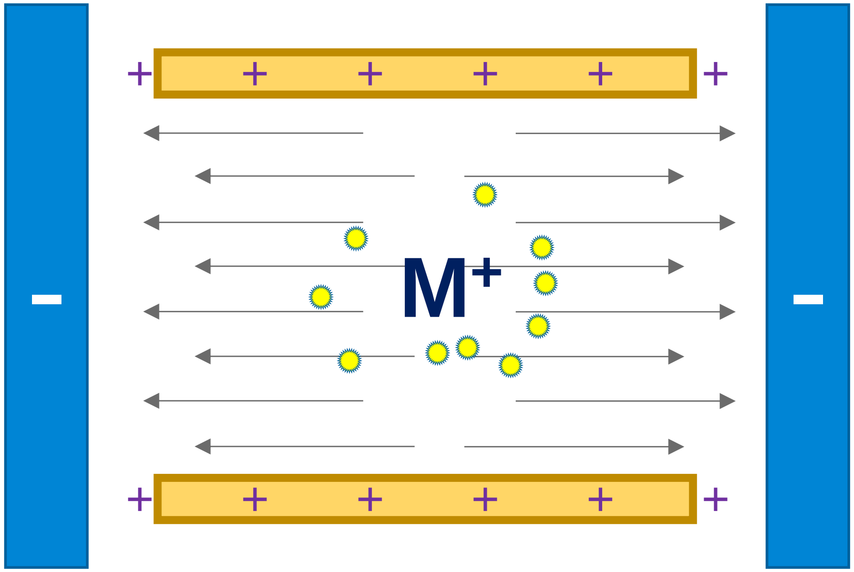
X = Magnetic Field INTO the Page

Electrons Accelerated by Magnetic Field



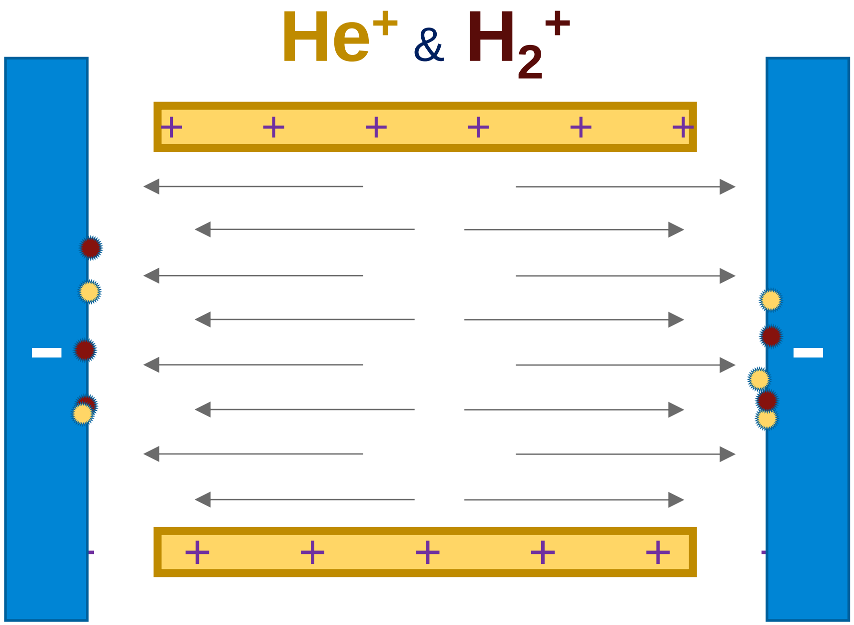
Resulting in Formation of POSITIVE Ions

Ions Accelerated by Electric Field

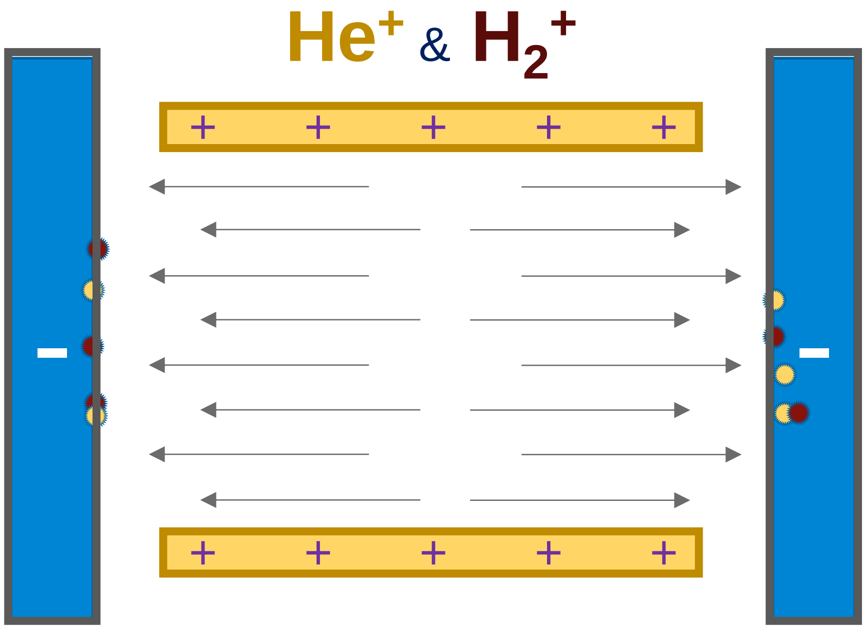


What Happens Next Depends on Which IONS are Formed

Lighter Ions are Accelerated Towards the Cathodes

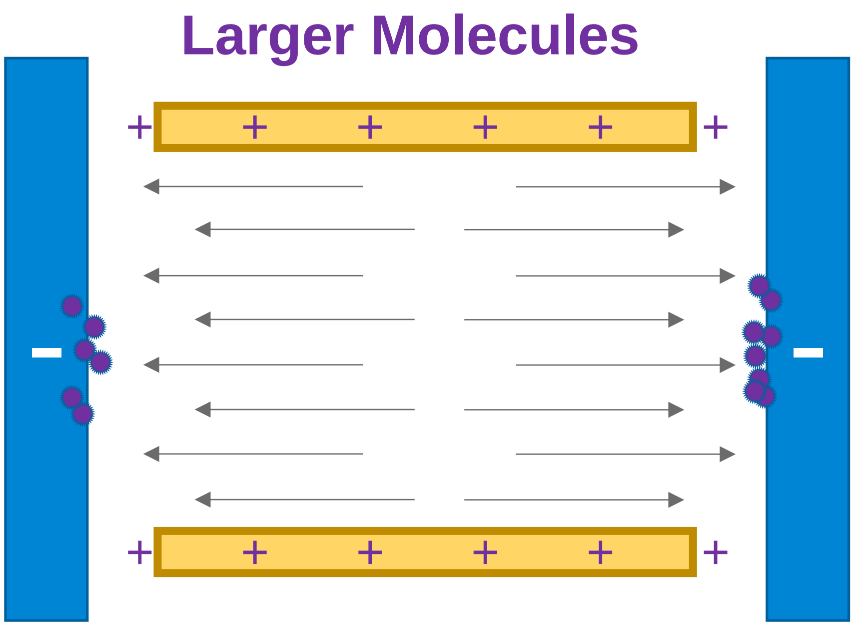


…. and Driven Into Cathode Material (Titanium)

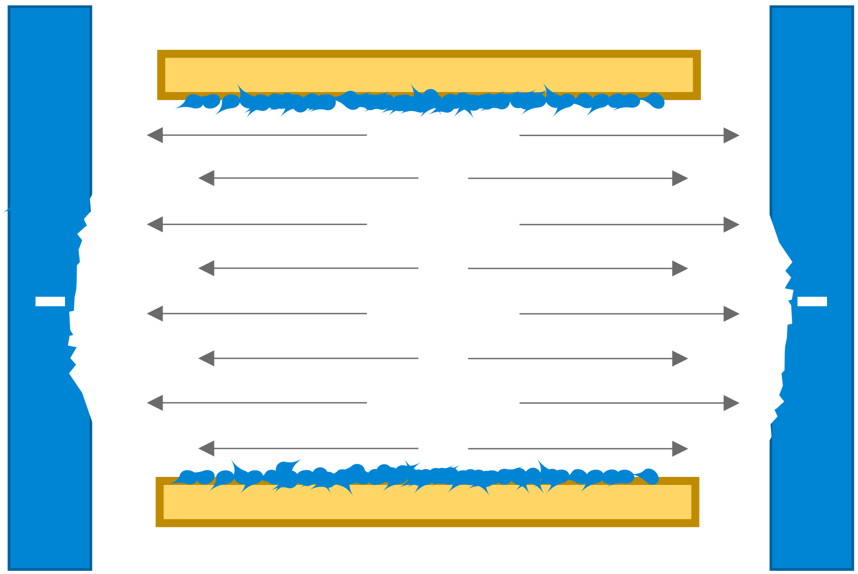


'Permanently' Trapping Them in the Cathode Structure

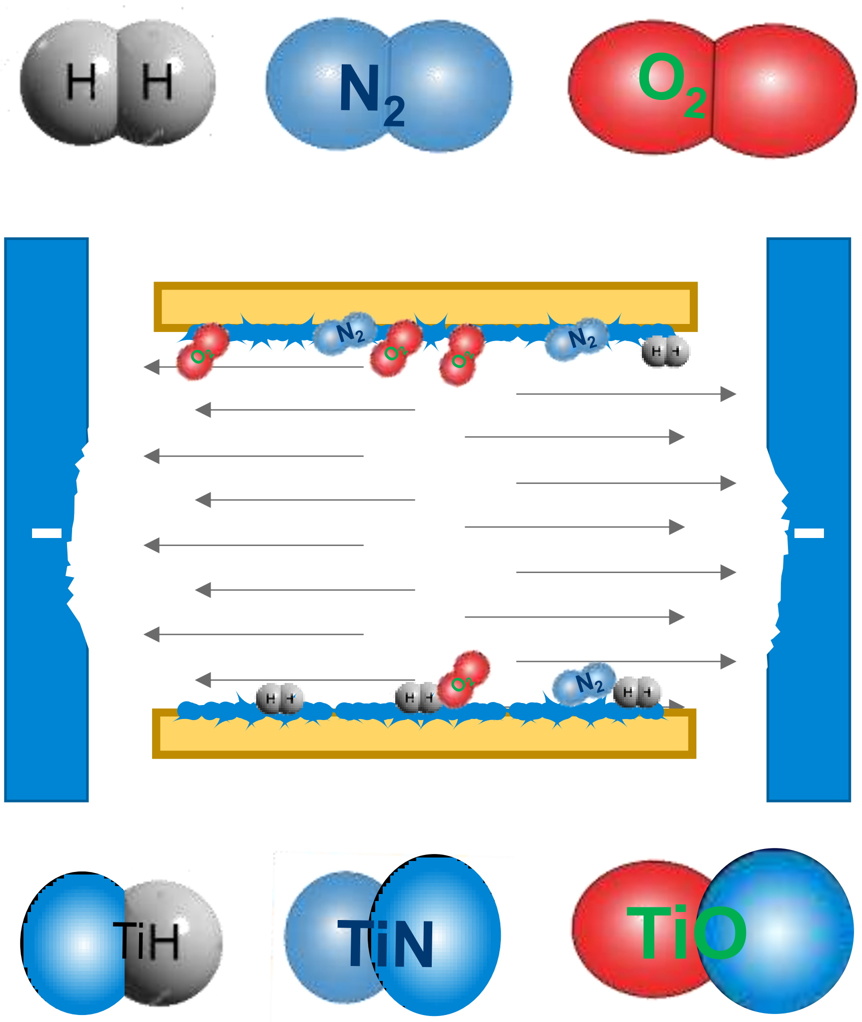
Larger Molecules Driven into Cathodes



… Sputtering **TITANIUM** Onto Anode Surface



‘Getterable’ Gas Species React with Sputtered Titanium



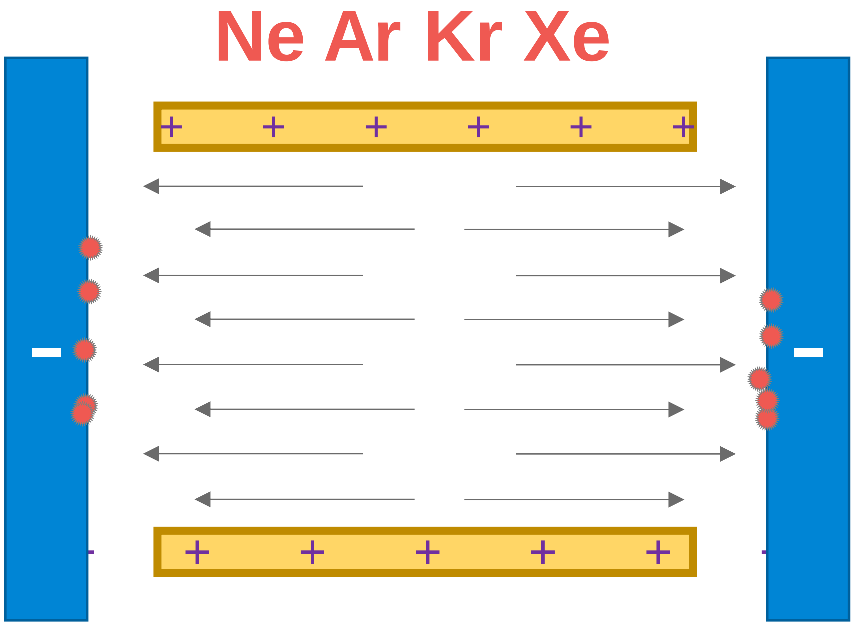
Forming Stable Compounds

PROBLEM: Noble Gases are Non-Reactive

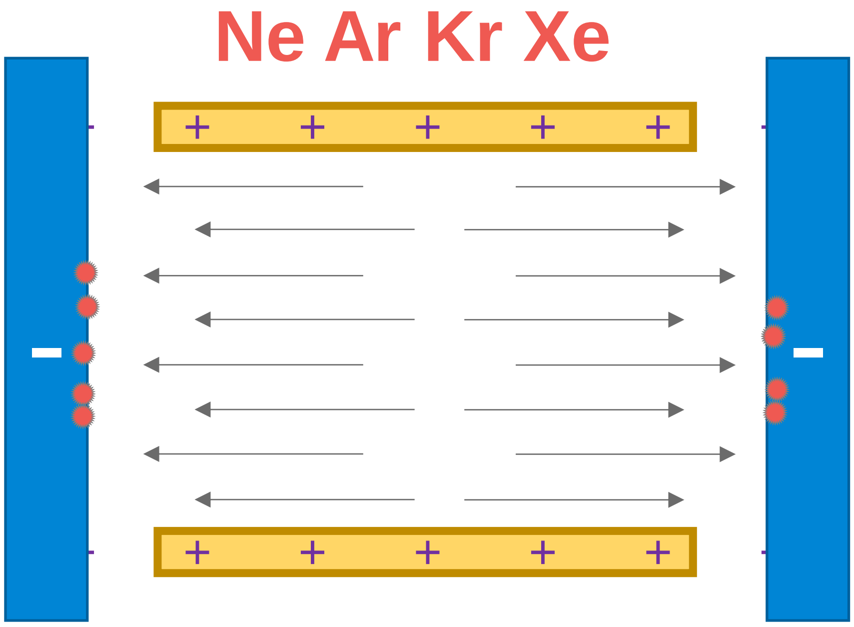
1 H 1.008																	2 He 4.0026
3 Li 6.94	4 Be 9.0122											5 B 10.81	6 C 12.11	7 N 14.007	8 O 15.999	9 F 18.998	10 Ne 20.17
11 Na 22.990	12 Mg 24.305											13 Al 26.982	14 Si 28.08	15 P 30.974	16 S 32.06	17 Cl 35.453	18 Ar 39.94
19 K 39.10	20 Ca 40.08	21 Sc 44.956	22 Ti 47.9	23 V 50.941	24 Cr 50.996	25 Mn 51.996	26 Fe 54.938	27 Co 55.84	28 Ni 58.7	29 Cu 63.54	30 Zn 65.3	31 Ga 69.72	32 Ge 72.5	33 As 74.922	34 Se 78.9	35 Br 79.904	36 Kr 83.8
37 Rb 85.467	38 Sr 87.62	39 Y 10.81	40 Zr 91.22	41 Nb 92.906	42 Mo 95.9	43 Tc 98.906	44 Ru 101.0	45 Rh 102.91	46 Pd 106.4	47 Ag 107.87	48 Cd 112.4	49 In 114.82	50 Sn 118.6	51 Sb 121.7	52 Te 127.6	53 I 126.90	54 Xe 131.30
55 Cs 132.91	56 Ba 137.3	57- 71 La SERIES	72 Hf 178.4	73 Ta 180.95	74 W 183.8	75 Re 186.2	76 Os 190.3	77 Ir 192.3	78 Pt 195.0	79 Au 196.97	80 Hg 200.5	81 Tl 204.3	82 Pb 207.2	83 Bi 208.98	84 Po	85 At	86 Rn
87 Fr	88 Ra 226.03	89-103 Ac SERIES															

La Series	57 La 138.91	58 Ce 140.12	59 Pr 140.91	60 Nd 144.2	61 Pm	62 Sm 150.4	63 Eu 151.96	64 Gd 157.2	65 Tb 158.92	66 Dy 162.5	67 Ho 164.93	68 Er 167.2	69 Tm 168.93	70 Yb 173.0	71 Lu 174.97
Ac Series	89 Ac	90 Th 232.04	91 Pa 231.04	92 U 238.03	93 Np 237.05	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

NOBLE Gas Ions Accelerate Towards the Cathodes



… but are NOT Permanently Trapped in Cathodes



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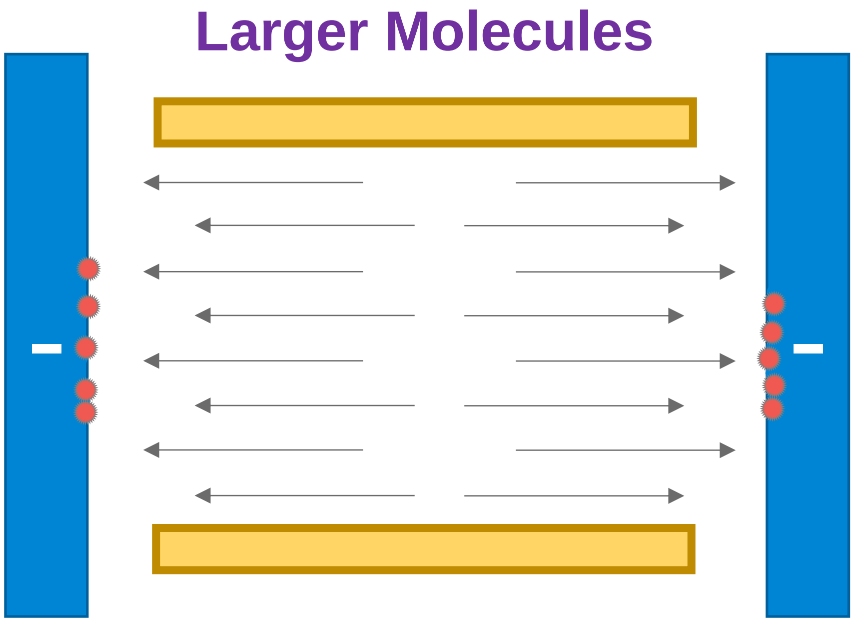

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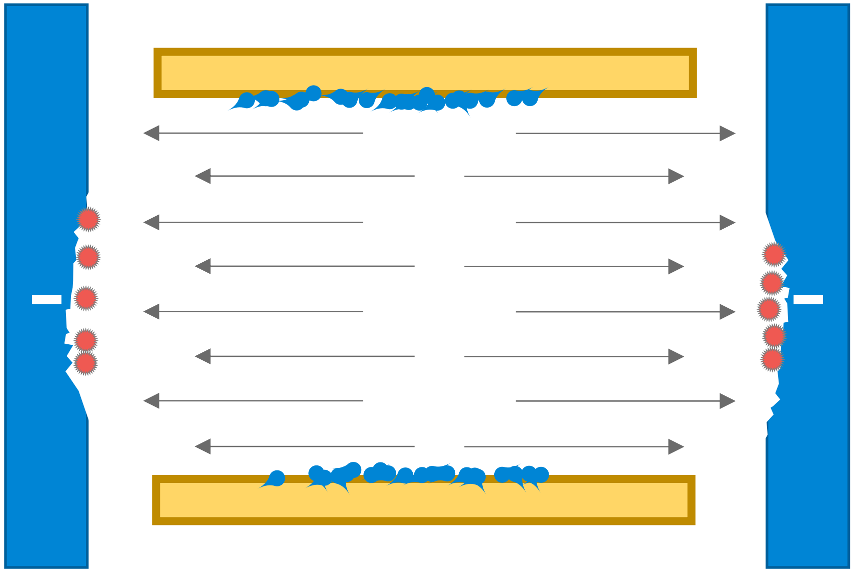
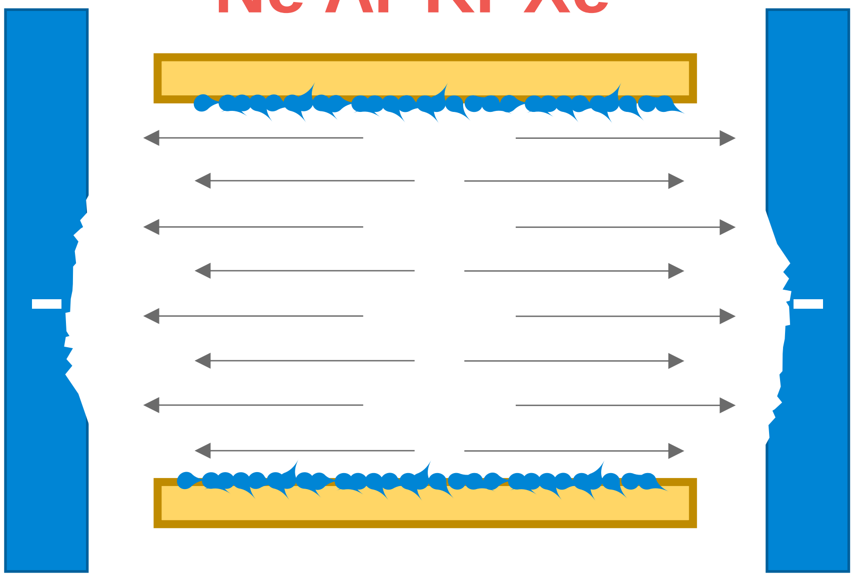


… and are Liberated as TITANIUM is Removed



***NOBLE* Gases are *NOT* Permanently Trapped in the DIODE PUMP's *Titanium* Cathode Structure**

Diode Pump: Best 'All-Round' UHV Pump

DIODE (2x Ti Cathodes)

- Highest Pumping Speed & Capacity for Hydrogen (2x Flat Titanium Cathodes)
- Highest Pumping Speed for Chemically Reactive ('Getterable') Gases (CO, CO_2, H_2, N_2, O_2 etc)
- Not Recommended for pumping noble gases
  - Argon instability after ~ 20 h at 10^{-6} Torr

Diode Cathode & Anode

22
Ti
(47.9)

NOBLE DIODE: Solving ‘Argon Instability’ Problem

NOBLE DIODE (1x Ti, 1x Ta Cathodes)

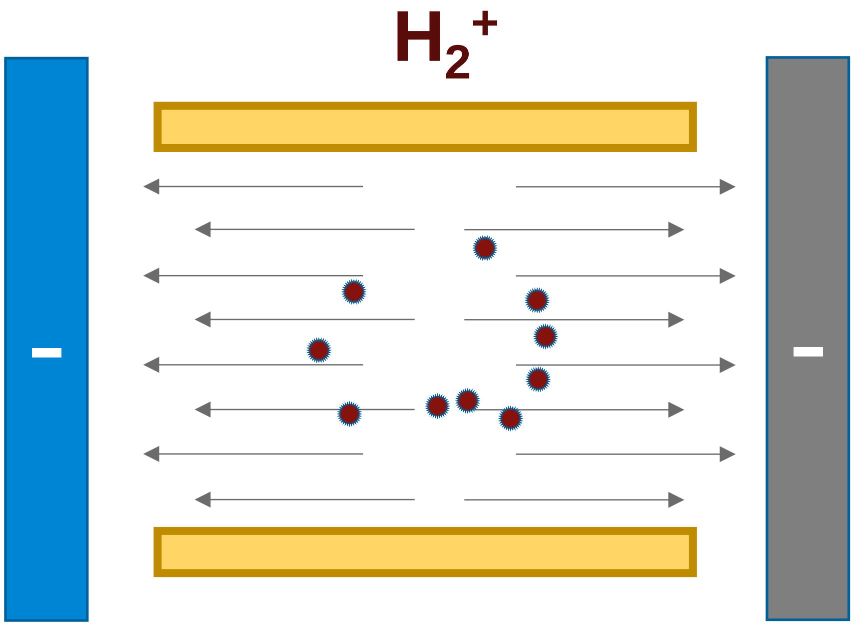
- **Tantalum** cathode reflects NEUTRALS with High Energy (ie. have greater ability to penetrate Titanium on Anode Surface)
- Reduced H_2 Pumping Speed (1x **Titanium** Cathode & 1x **Tantalum** Cathodes) through entrapment
- Reduced Pumping Speed for ALL Getterable Gases (less Titanium available to be sputtered)

22 Ti (47.9)	73 Ta (180.95)

Noble Diode Cathode (Ti Plate) & Anode

How it Works: Noble Diode Pump

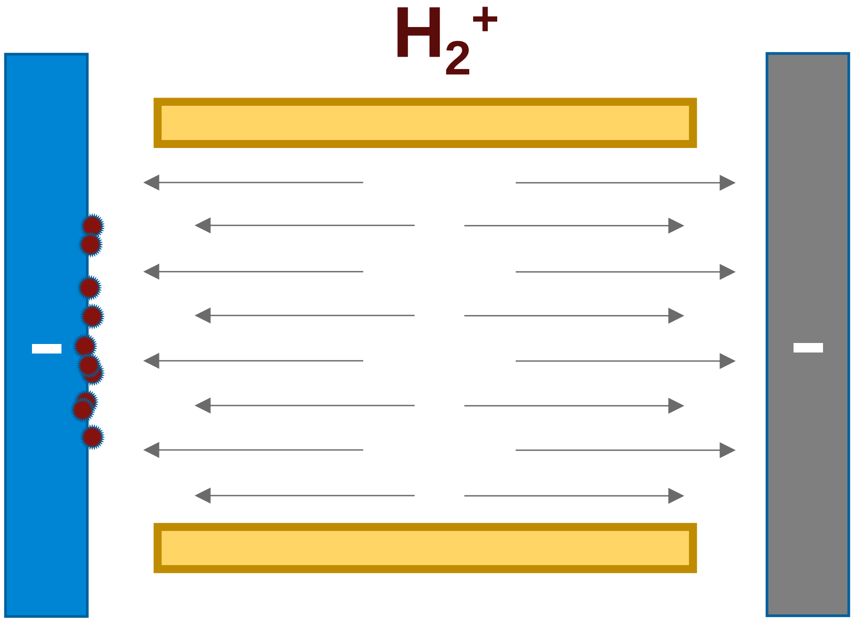
***TANTALUM* is more dense than Ti**



How it Works: Noble Diode Pump

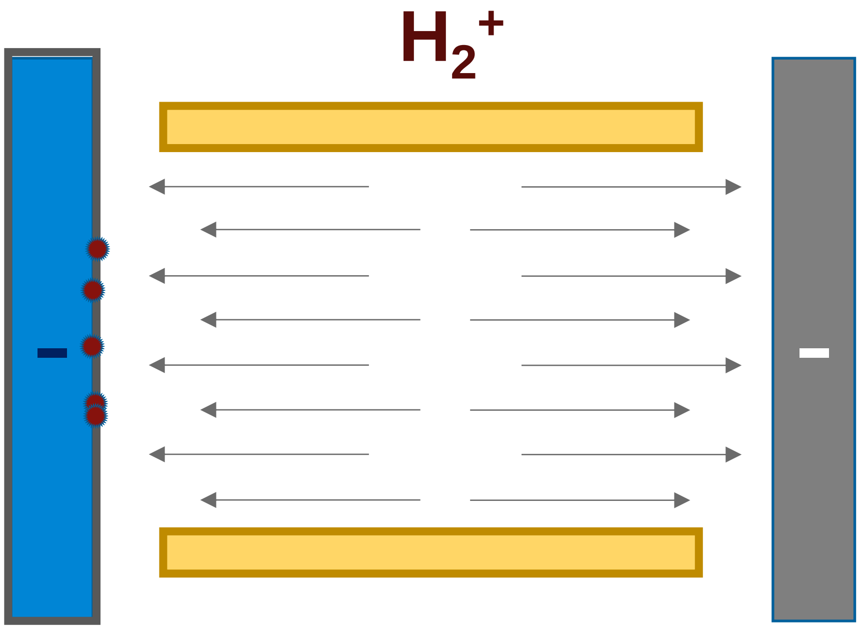
***TANTALUM* is more dense than Ti**

- ***H_2^+ Ions are Neutralized and Reflected by the Cathode with HIGH ENERGY***



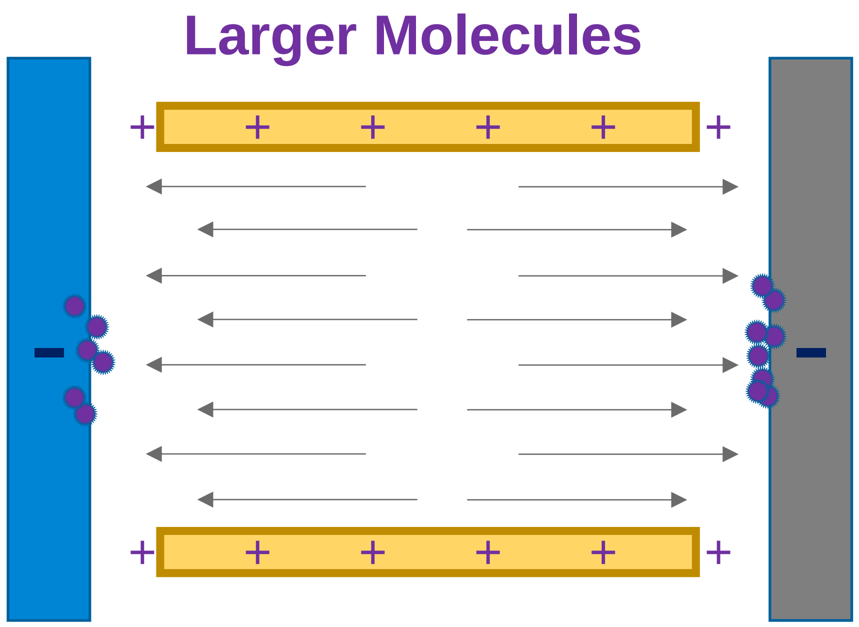
- ***They become re-ionized in the Electron Cloud (<u>not shown</u>) and are driven towards the TITANIUM Cathode***

SOLUTION: Noble Diode Pump Uses 1x Tantalum Cathode

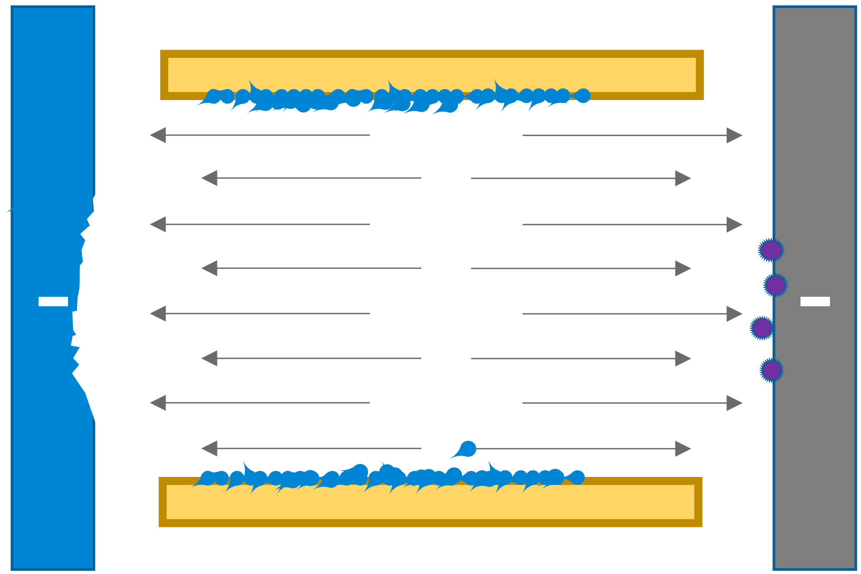


Lighter Gases are Again 'Permanently' Trapped in the Titanium Cathode Structure

Larger Molecules Driven into **TITANIUM** Cathode

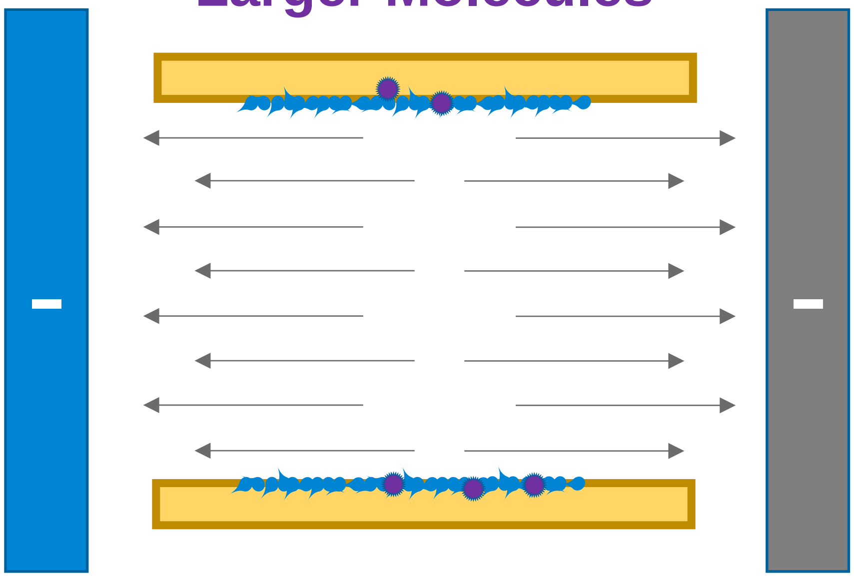


… Sputtering (LESS!) TITANIUM Onto Anode Surface



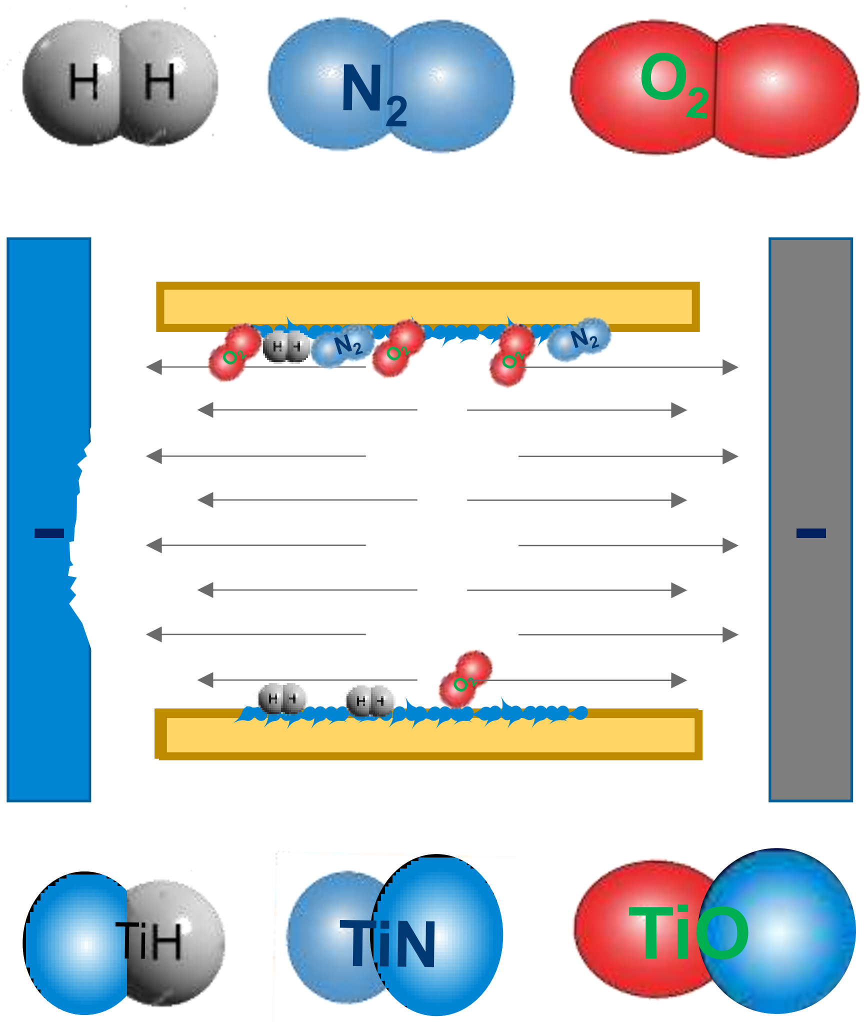
Ions Striking Ta Cathode Reflected as High Engergy Neutrals

Larger Molecules



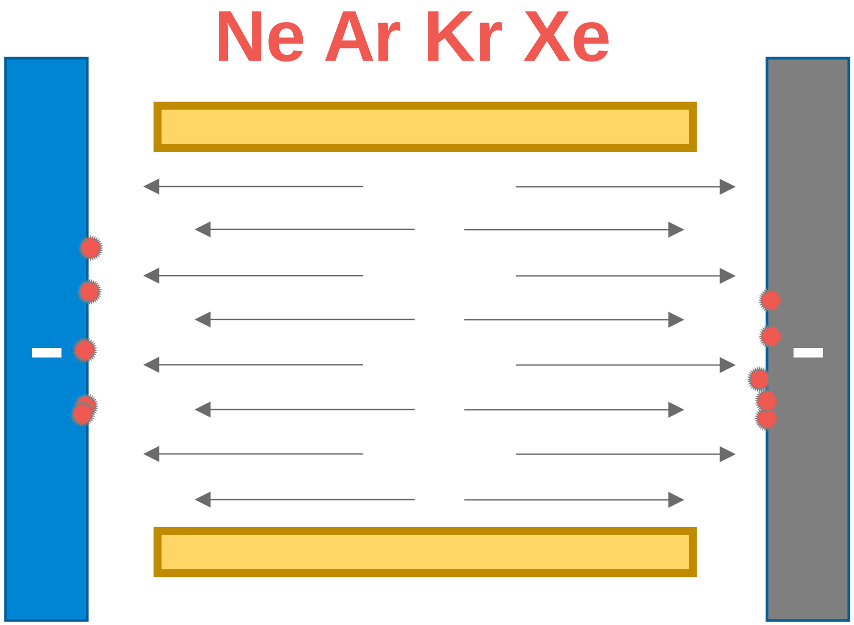
…With enough energy to penetrate the TITANIUM coating on the Anode: Since no sputtering occurs at the Anode, they are permanently trapped (and 'coated' with fresh Ti after)

‘Getterable’ Gas Species React with Sputtered Titanium

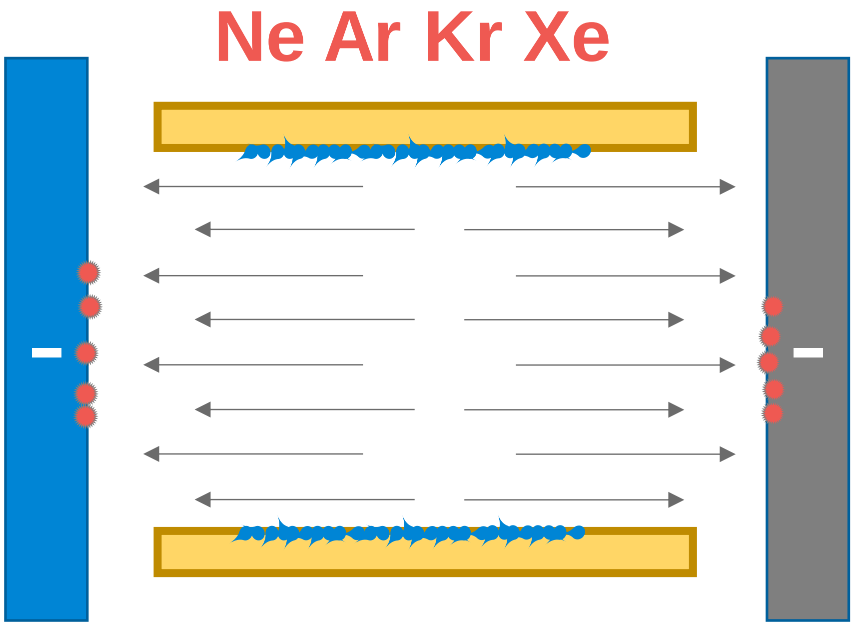


Forming Stable Compounds

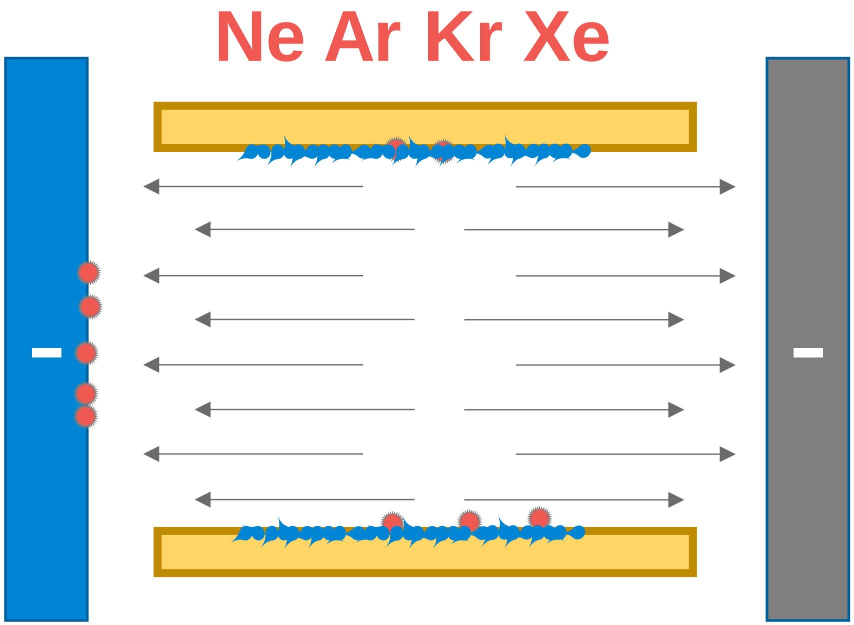
NOBLE Gas Ions Accelerate Towards the Cathodes



… and are again 'lightly buried' in the ***TITANIUM*** *Cathode*

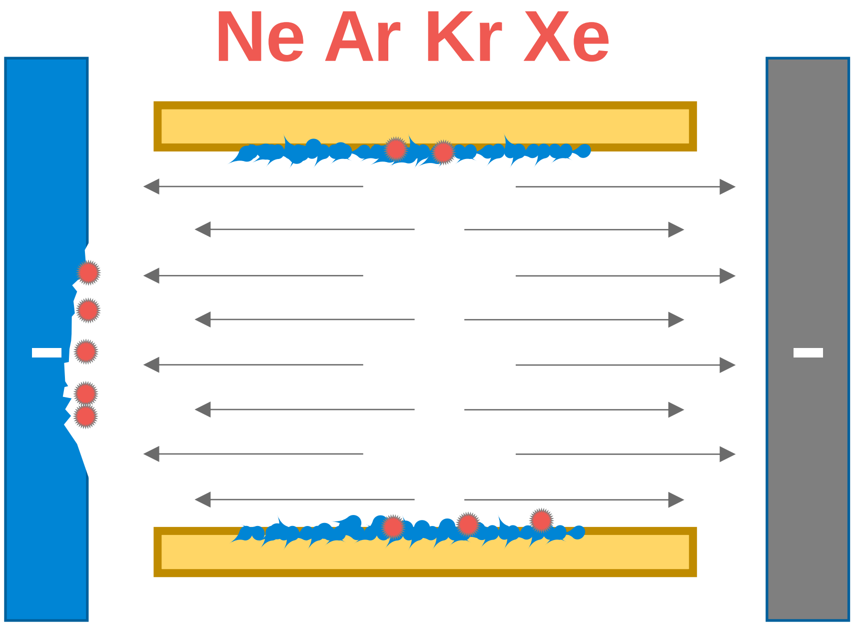


… and are again 'lightly buried' in the *TITANIUM* Cathode



… but they are REFLECTED as <u>High Energy Neutrals</u> off the TANTALUM Cathode (Can penetrate the Titanium layer on the Anode Surface)

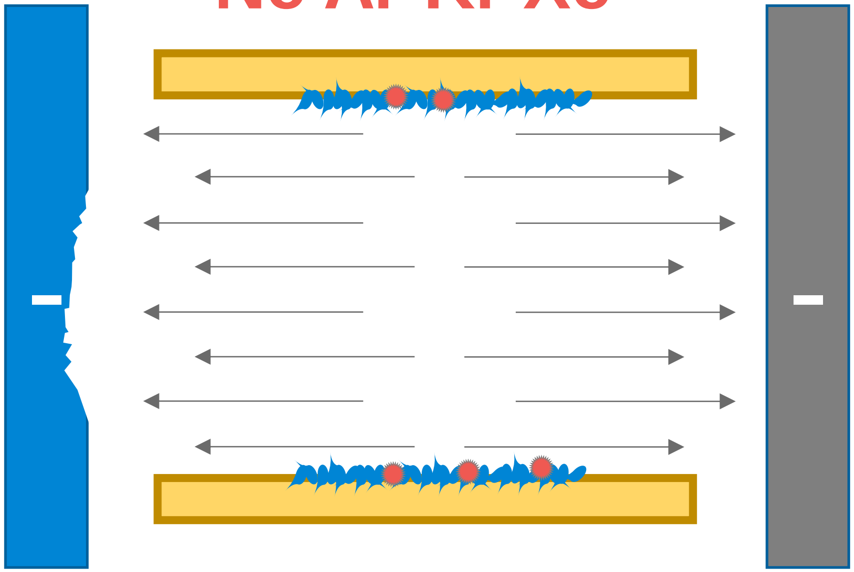
NOBLE Gas Molecules Can Be Covered by Sputtered Titanium



Effectively Trapping Them on the Anode Surface

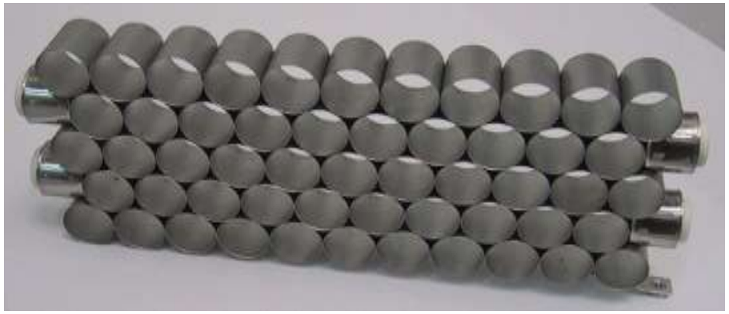
NOBLE Gas Molecules Can Be Covered by Sputtered Titanium

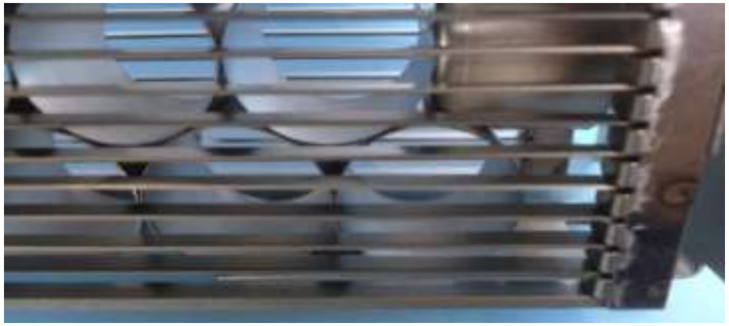
Ne Ar Kr Xe

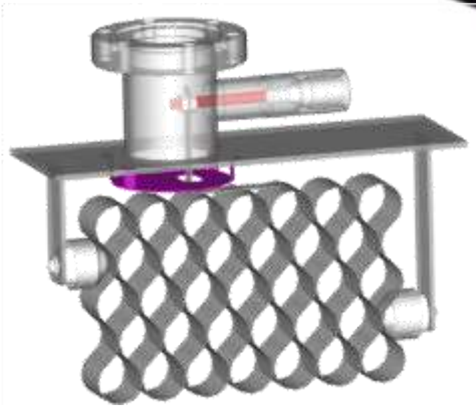


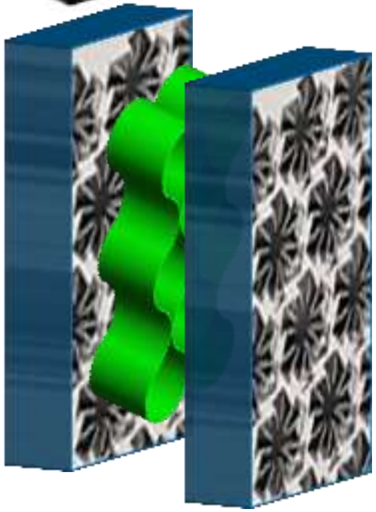
Unlike NOBLE Gas Ions on the TITANIUM Cathode (that are again released by subsequent bombardments)

Triode Pumps (inc. StarCell) Use Modified Cathode Geometry





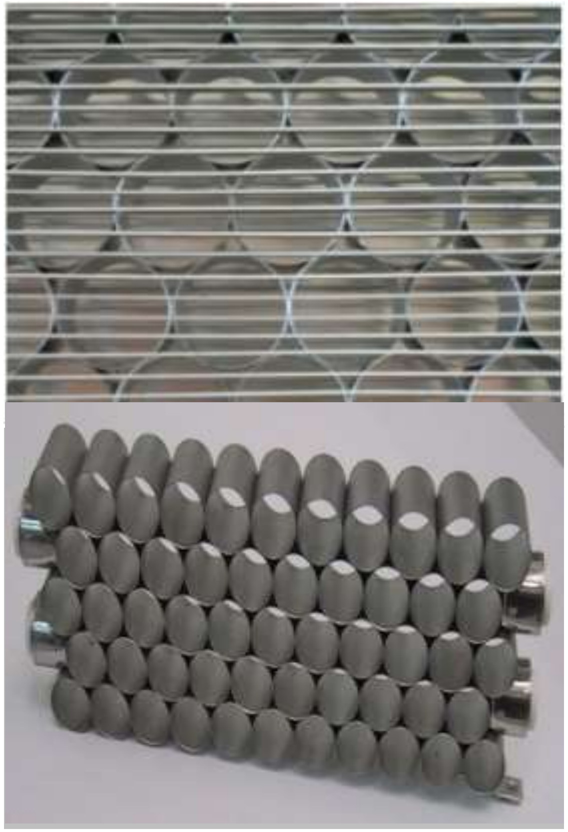




Alternate Cathode Designs Fore Specific Gases

TRIODE

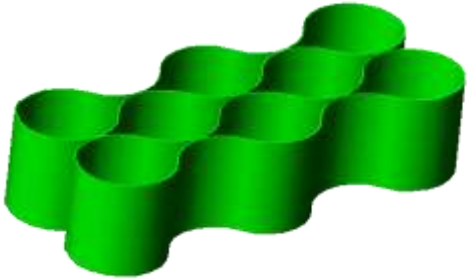
- Anode is grounded; Cathode consists of STRIPS of **TITANIUM** at Negative voltage
- Noble gas ions have glancing collisions with Cathodes and then become high energy neutrals
- Neutrals resting (even briefly) on the Anode and Pump wall can be ‘covered up’ by freshly sputtered Titanium
    - No sputtering occurs on the pump walls or at the Anode so Noble gas pumping is stable
- Chemically active gases are also pumped on the walls and Anode (Hydrogen is ‘Gettered’ and diffused into cathode structure)



StarCell Design: The Ultimate Triode Pump!

STAR CELL

- Optimized Triode Design: Star shaped cut-outs in **TITANIUM** Cathode provide
    - maximum pumping speed for Noble Gases
    - optimized lifetime vs Diode Pump (80K hours vs. 50K hours)
    - ~20 times longer Argon stability than Noble Diode
- Pumping speed for Getterable gases and Hydrogen comparable to Noble Diode Pump
- Higher Hydrogen capacity than Noble Diode



Ion Pump Controller

ION PUMP CONTROLLER

Precision power supplies (80 – 200 W) required for each Ion Pump

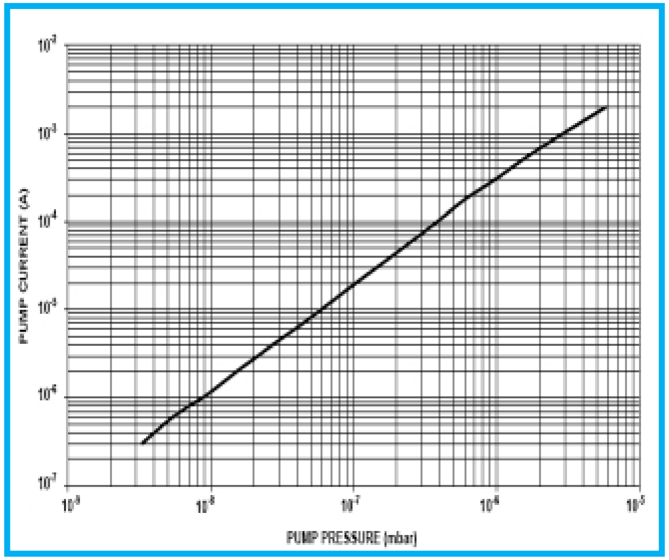
- Agilent Ion Pump controller software ramps voltage to 'ignite' plasma
- Current (between Cathode and Anode) can be measured to indicate the Vacuum Pressure ('Cold Cathode')
- Step Voltage feature optimizes sputtering of Titanium
  - Voltage <u>reduced</u> after ignition (reducing Leakage Current) which allows pressures in 10^{-10} Torr range to be measured

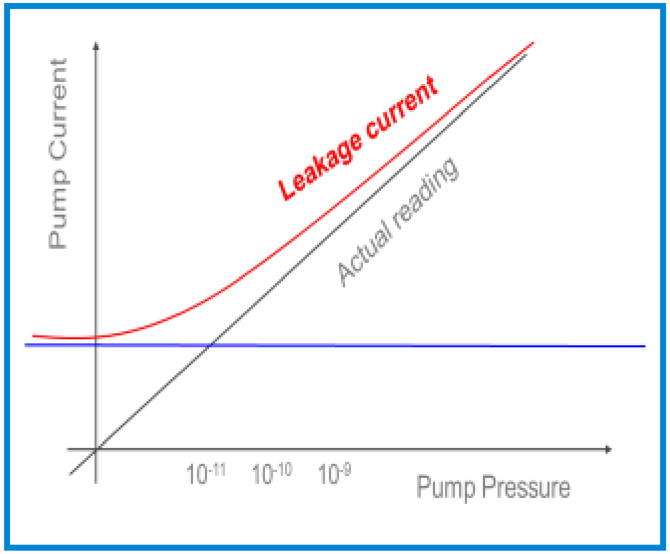
Using ION PUMP Current as UHV Gauge

ION PUMP CURRENT

Current in an ion pump is linearly proportional to the pressure so the ion pump can be used to roughly read the pressure

- Limitation at low pressure is given by the leakage current (roughly α Ion Current Potential)





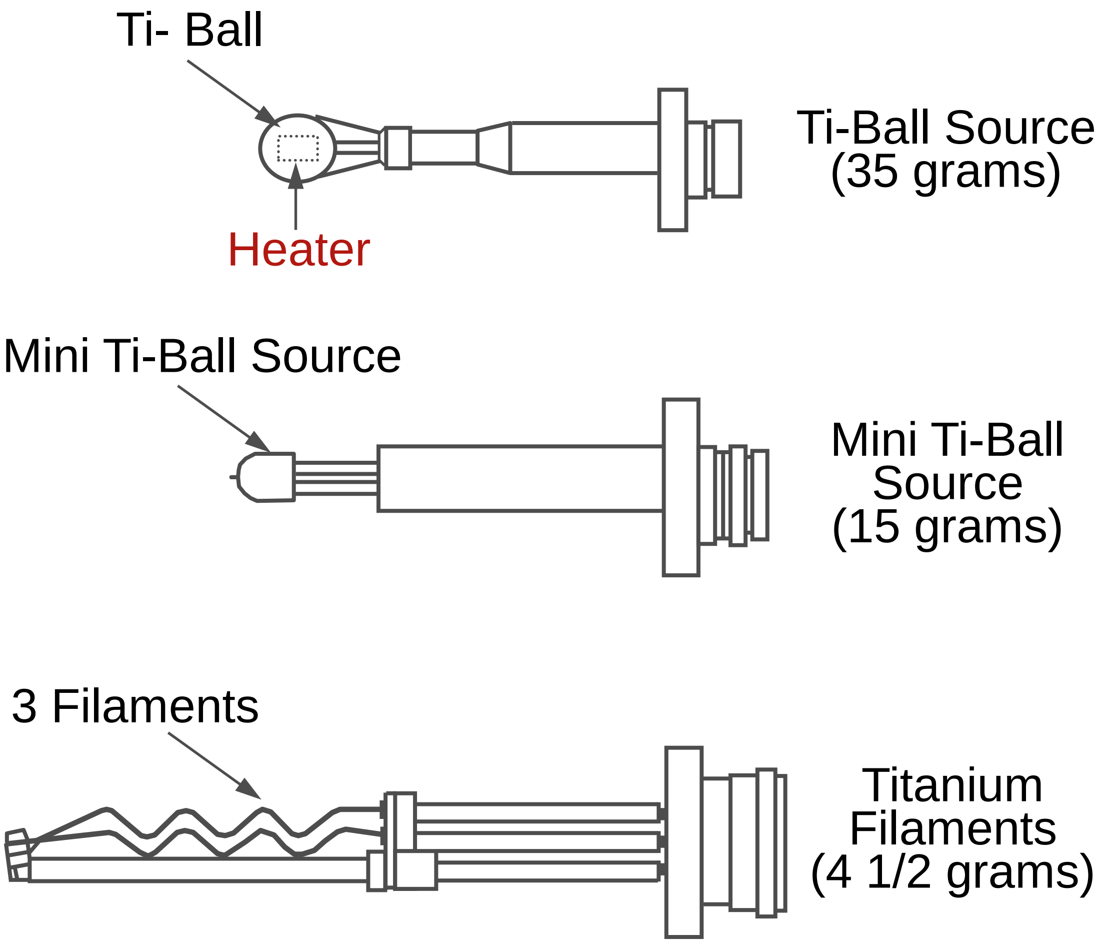
Getter Pumps: Pumping without Sputtering

There Are Two Ways of Producing a Clean Gettering Surface:

Non-Evaporable Getters - NEG: by heating an oxidized getter (i.e. powder coated metallic strip) to a temperature high enough to diffuse oxygen from the surface into the getter bulk.

Evaporable Getters -TSP: the active surface is obtained by "in situ" deposition under vacuum of a fresh clean metallic film

Titanium Sublimation Pumps



Ion Pump Selection Criteria

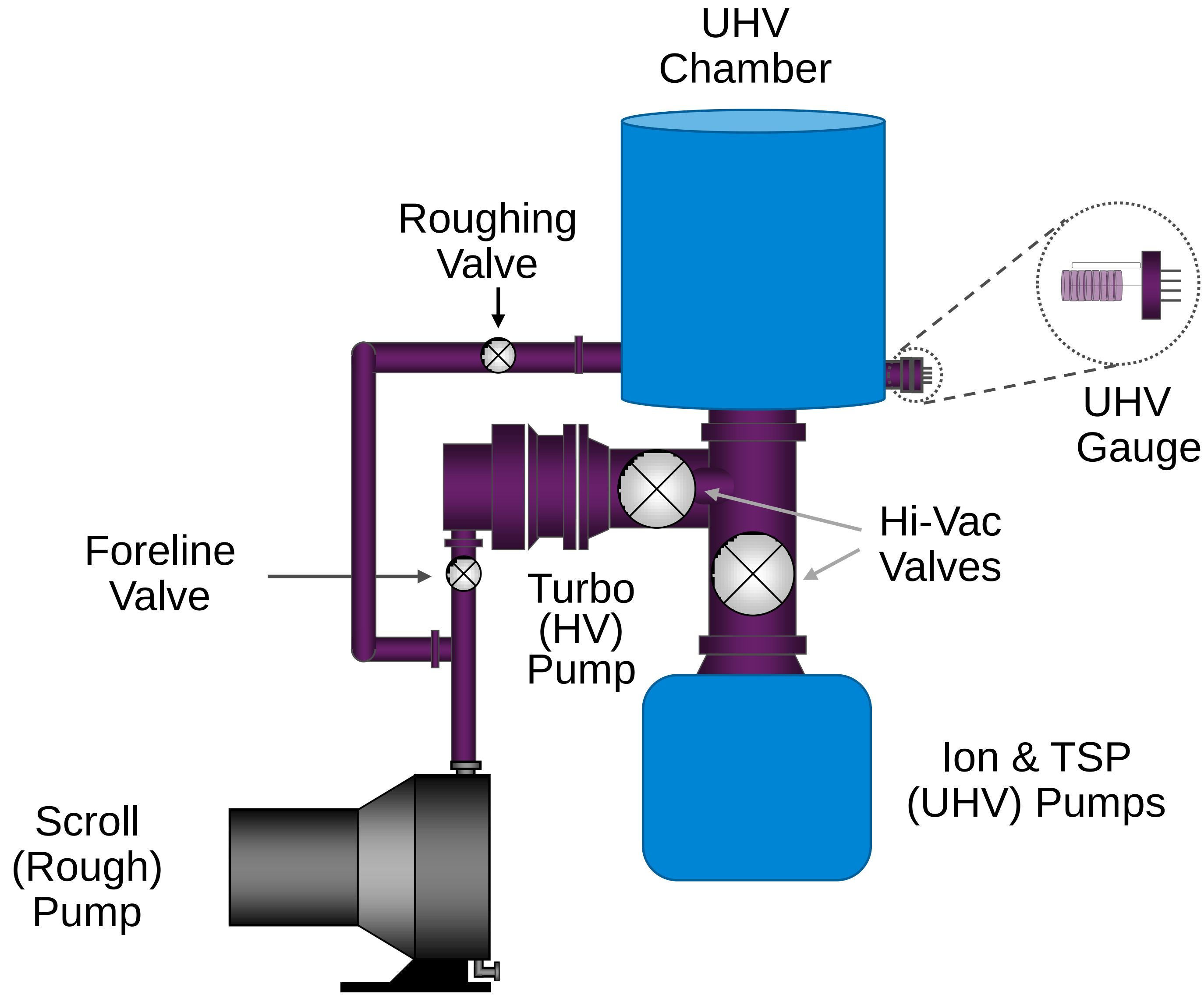
	TSP	Triode	Star Cell	Diode	Noble Diode
Hydrogen	3	1	2	3	1
Helium	0	3	4	1	3
Water	3	2	2	3	2
Methane	0	3	3	2	3
Nitrogen	3	2	2	3	3
O_2, CO, CO_2	4	2	2	3	3
Argon	0	3	4	1	3

Null	0
Poor	1
Good	2
Excellent	3
Outstanding	4

Combination Star Cell + TSP is best combination for a wide variety of gases

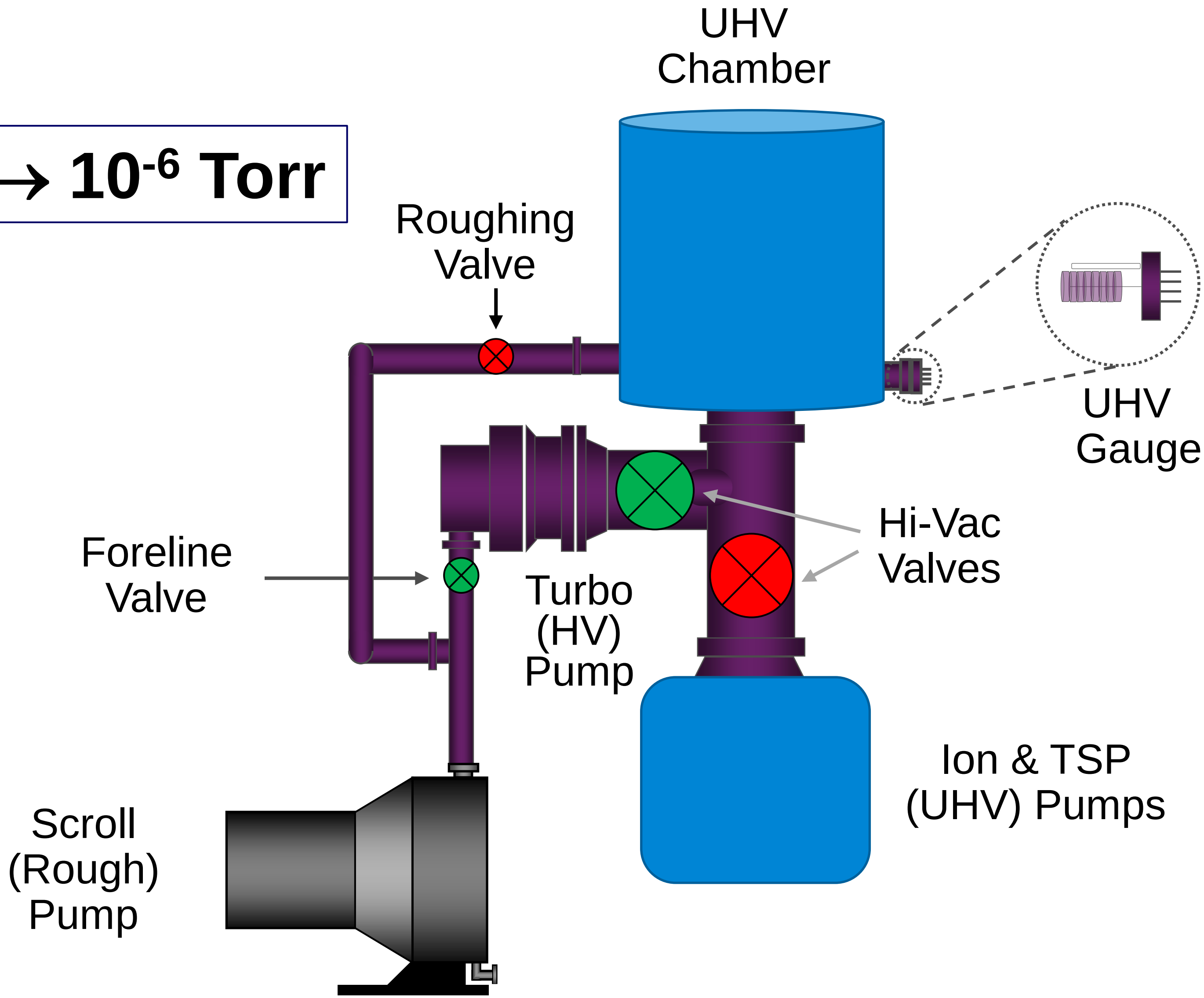
Diode pump has optimum pumping speed for Hydrogen

UHV System Schematic



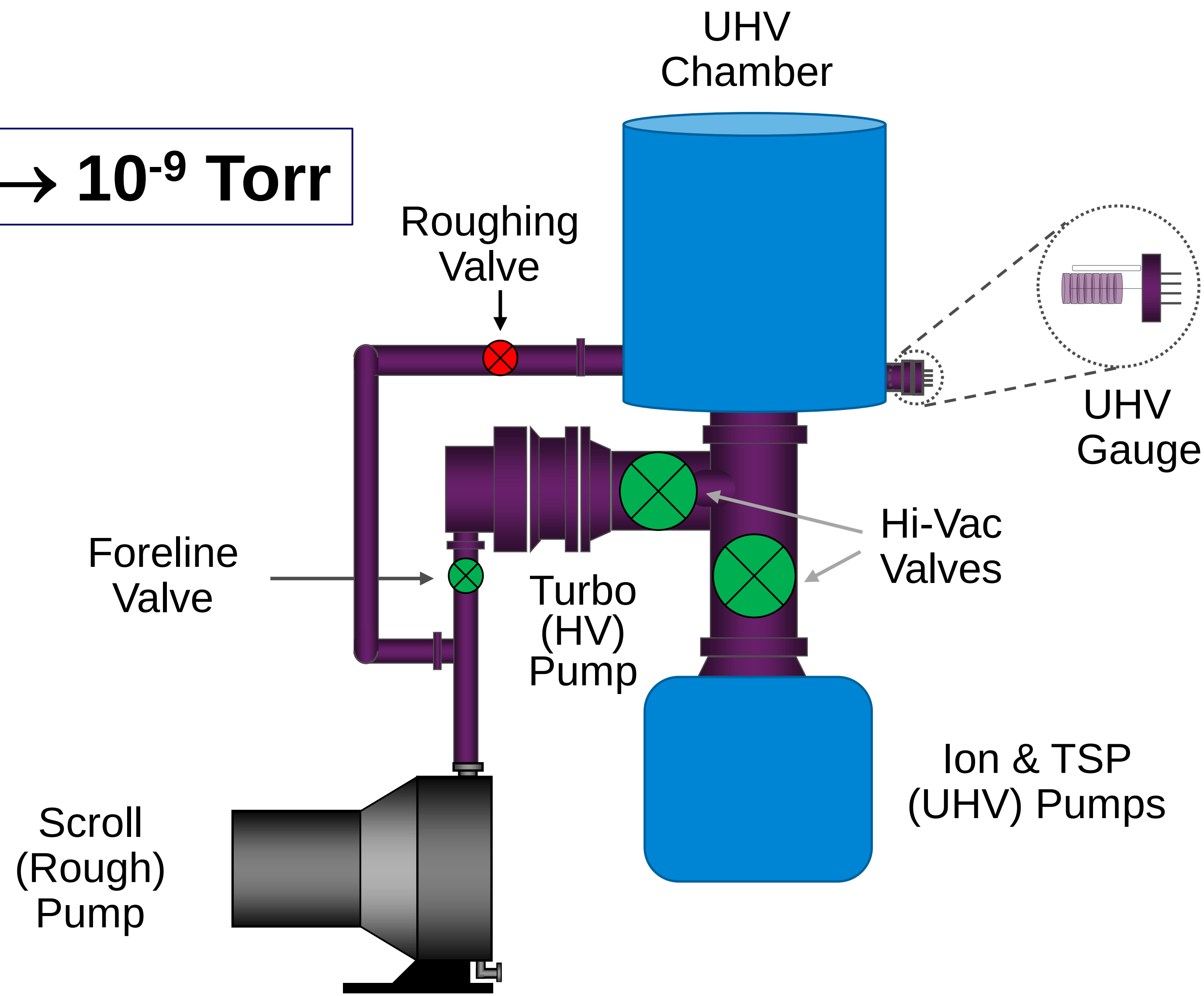
UHV System Operation

$10^{-2} \rightarrow 10^{-6}$ Torr



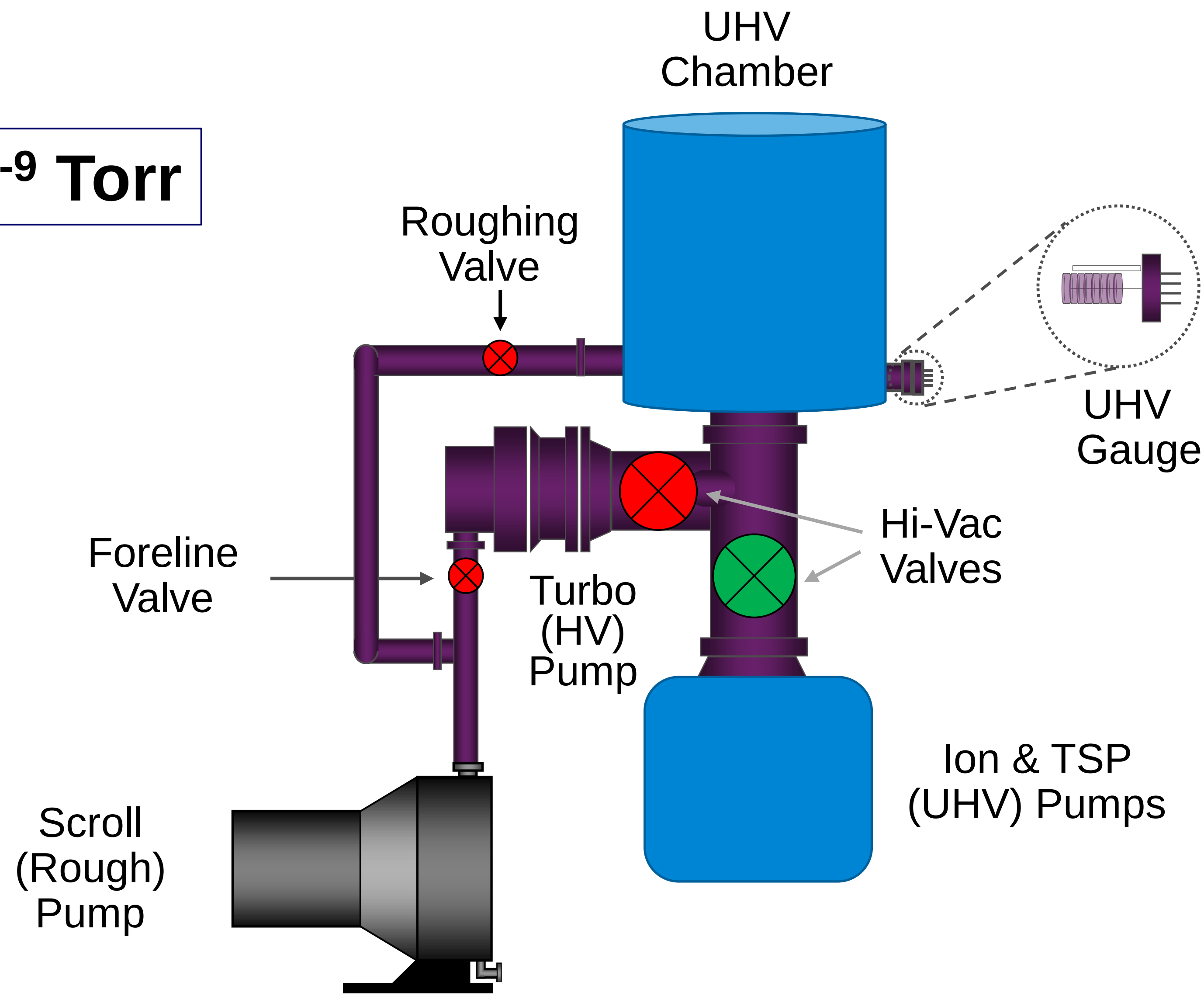
UHV System Operation

$10^{-6} \rightarrow 10^{-9}$ Torr

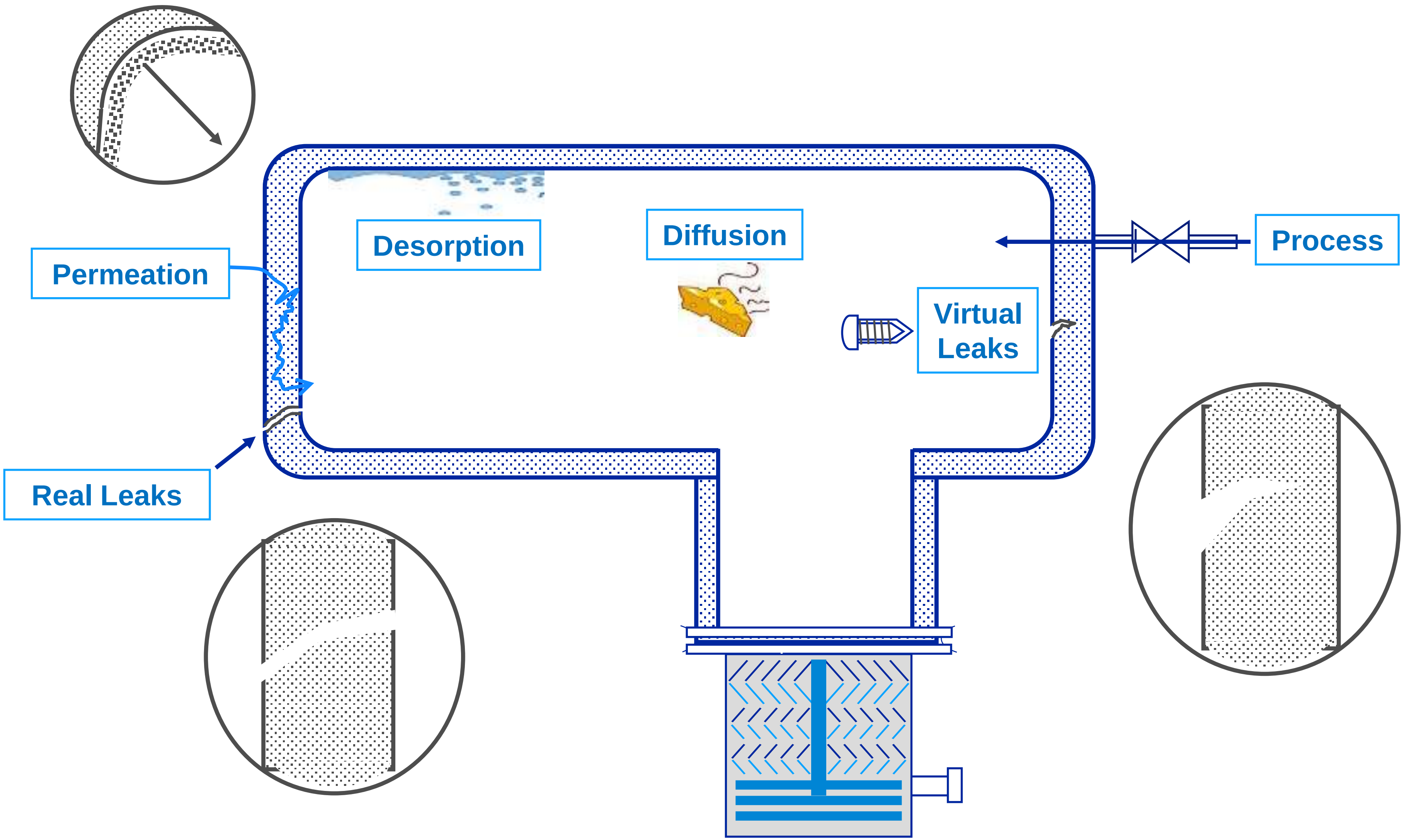


UHV System Operation

< 10^{-9} Torr



Troubleshooting Ultra-High Vacuum Systems



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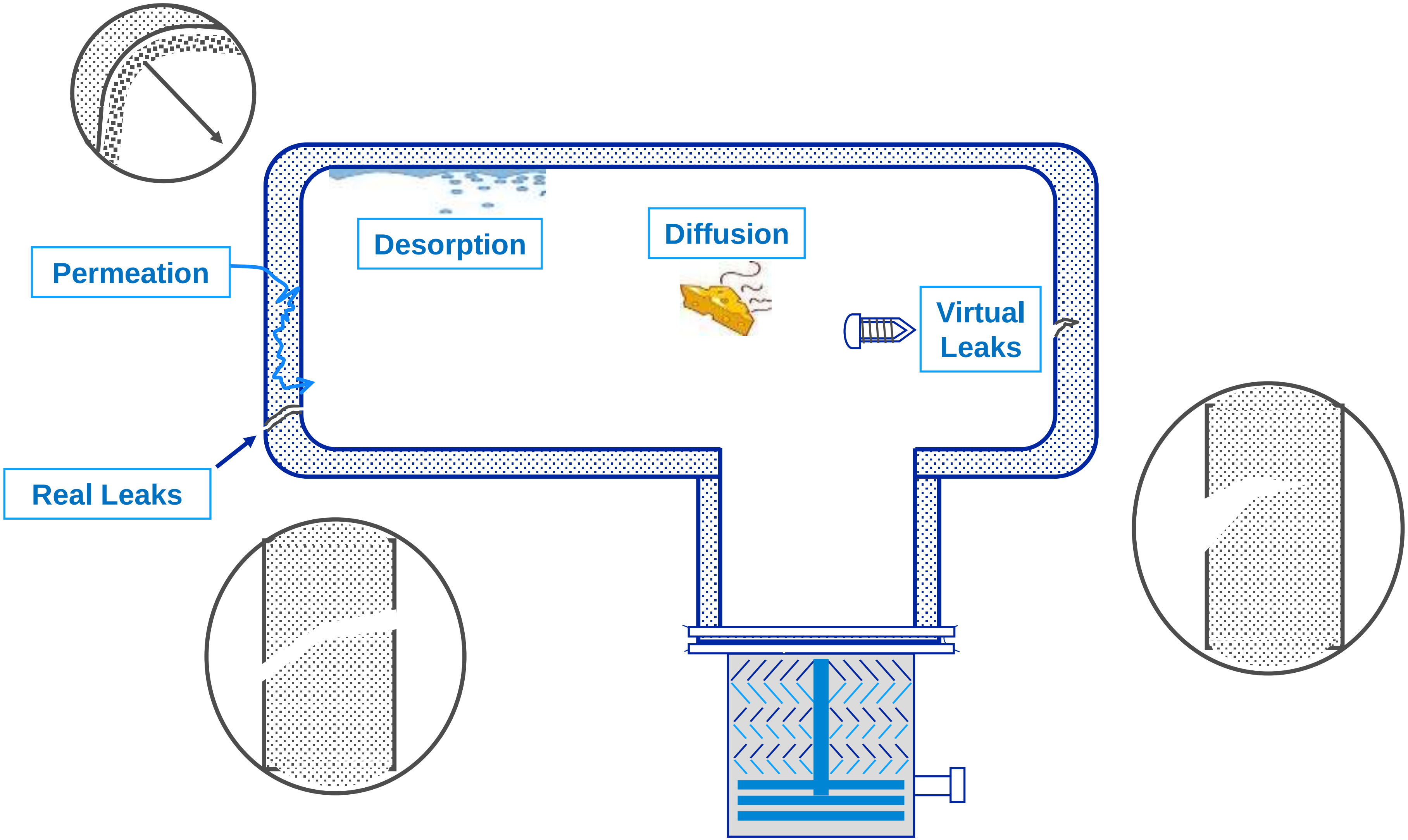

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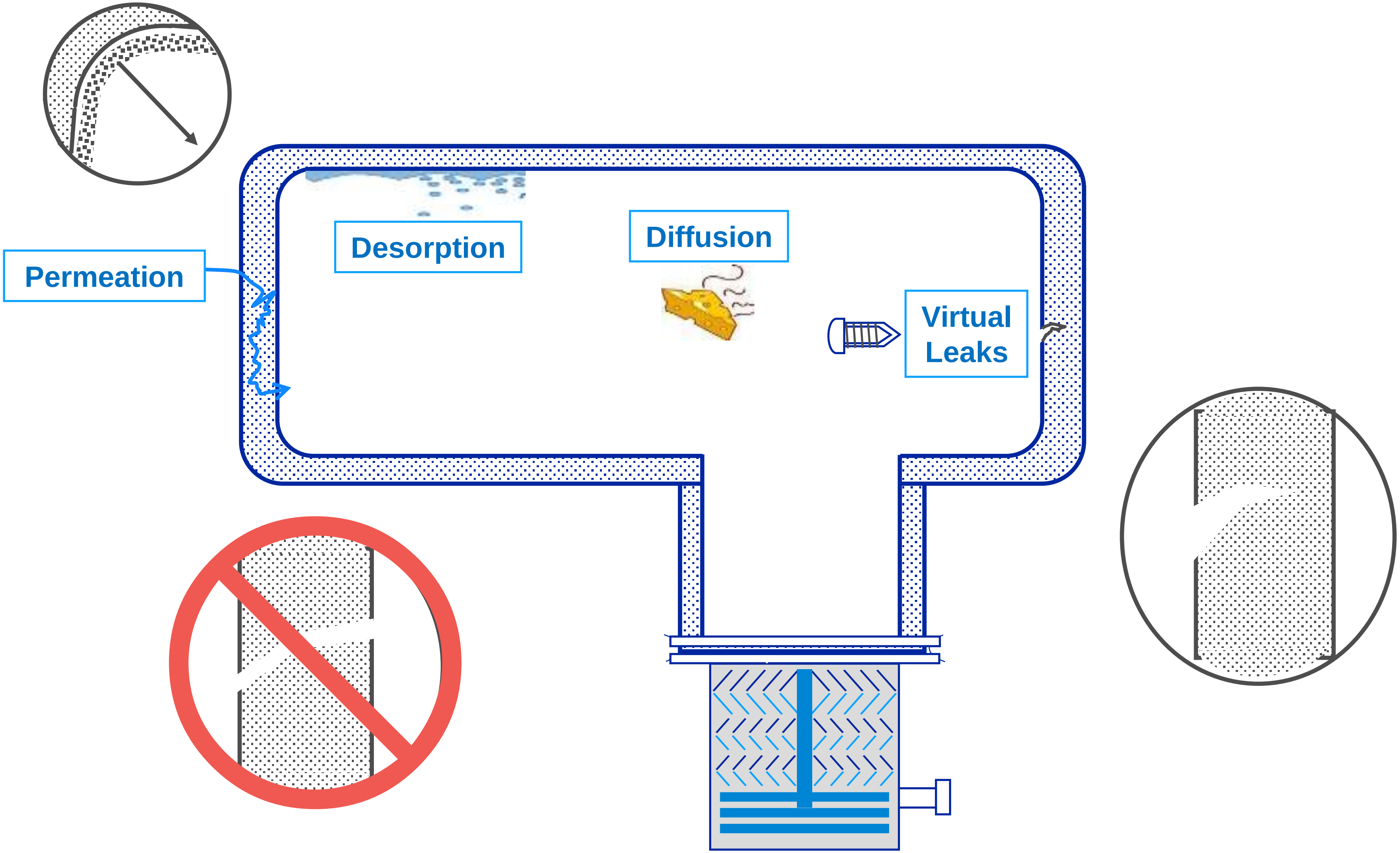

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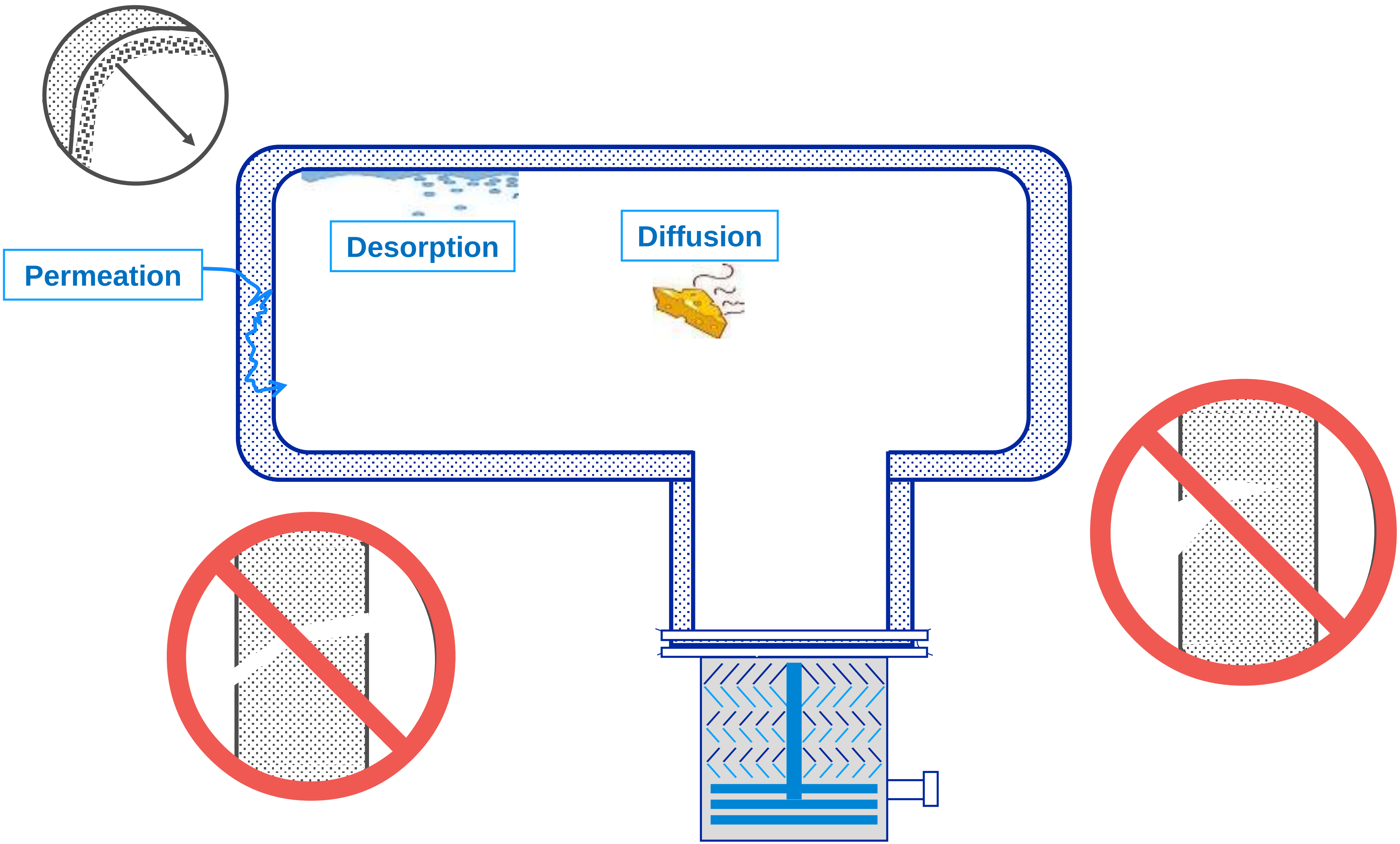

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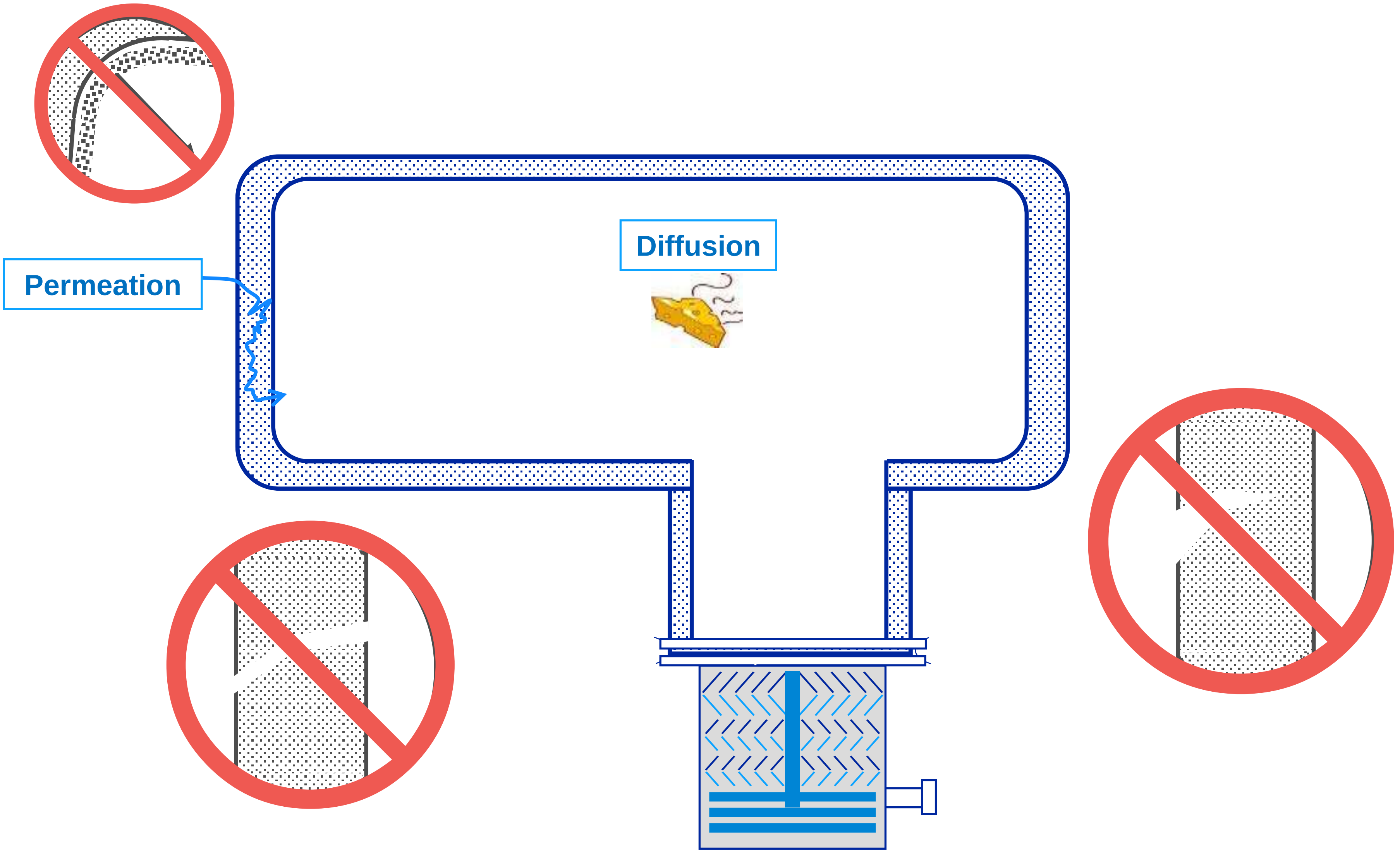

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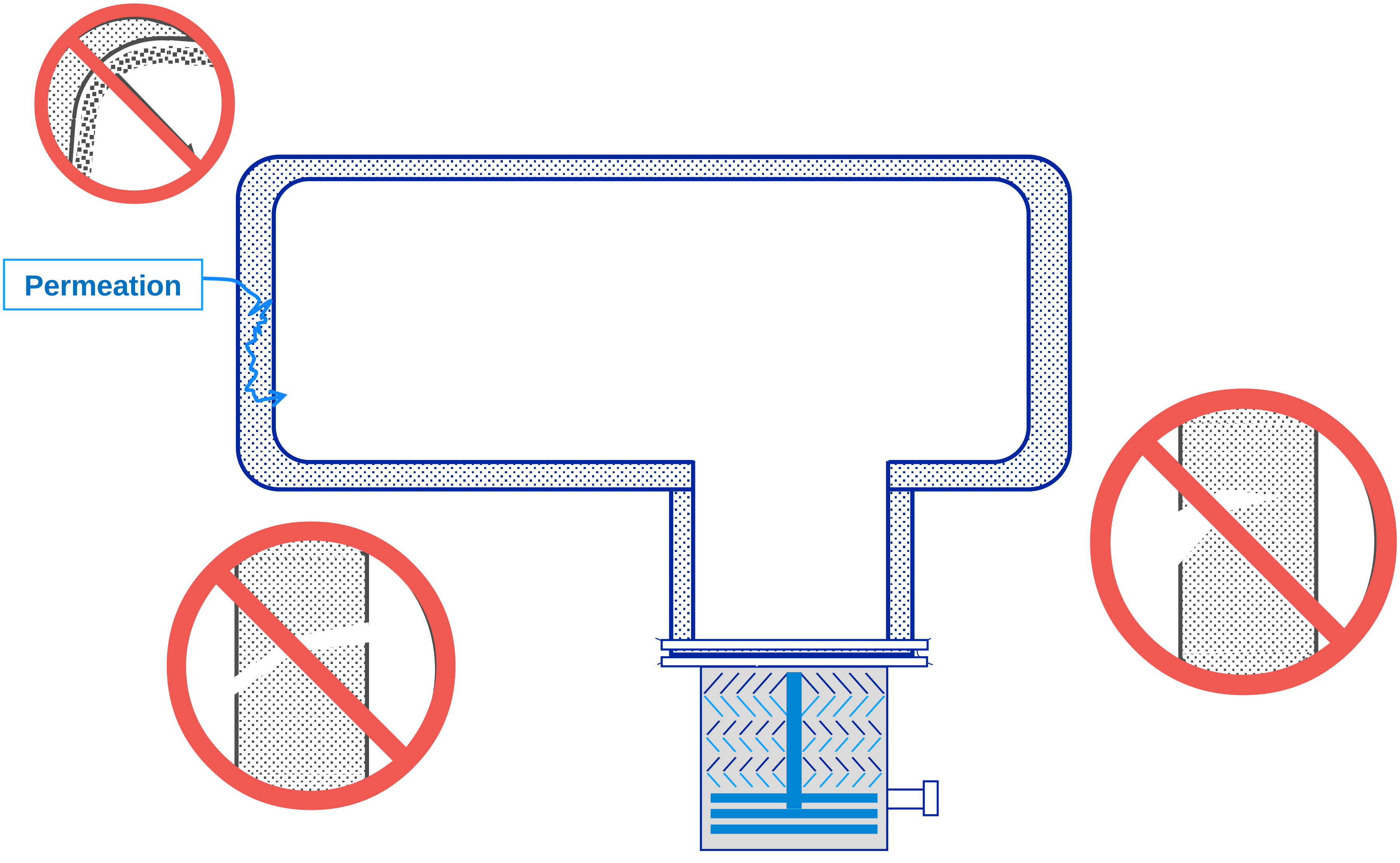

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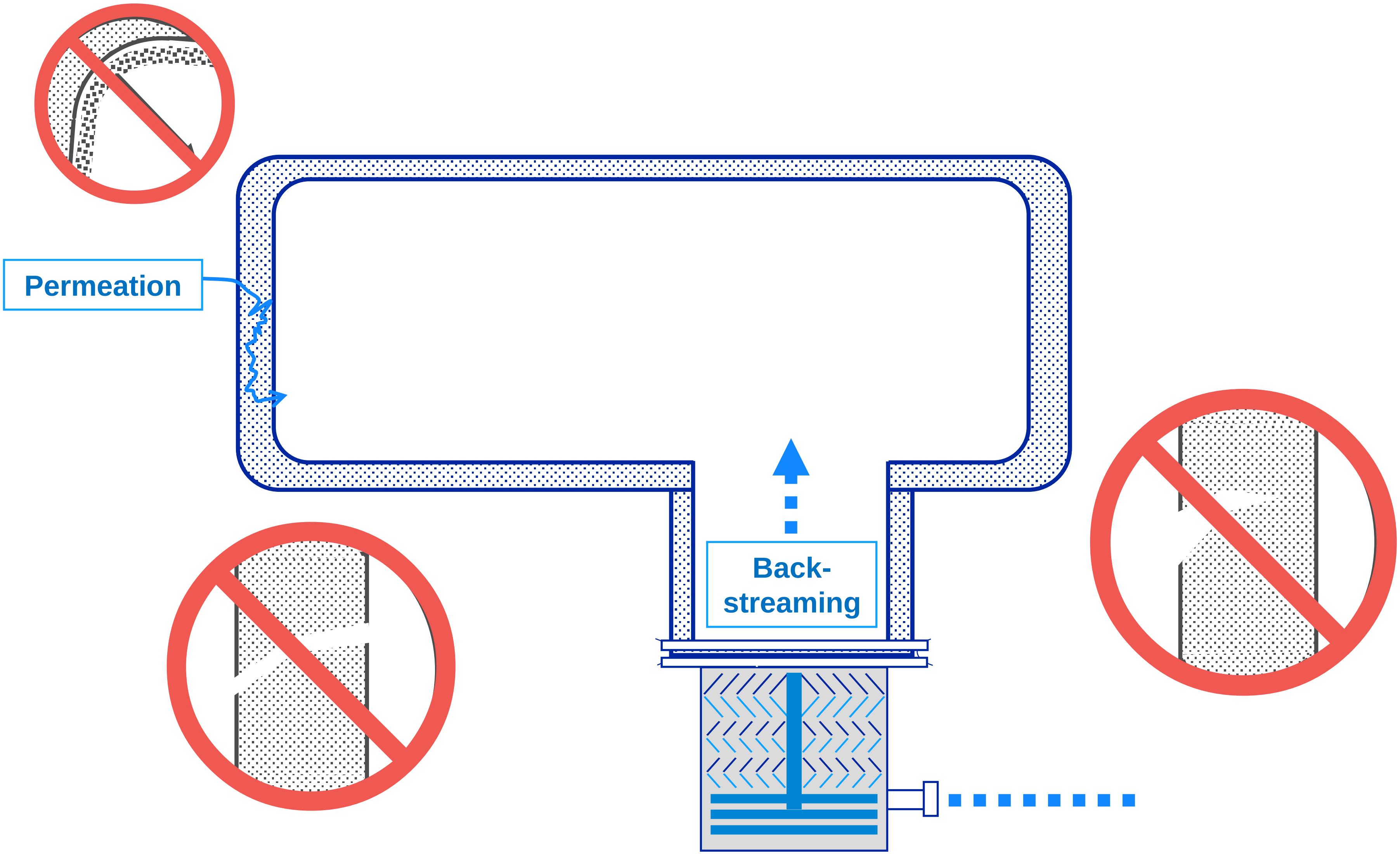

Locating REAL Leaks

Helium Mass Spec Leak Detection (HMSLD) is a very sensitive technique typically used to locate REAL ('outside-in') leaks in UHV Systems.

<u>Theory of Operation</u>

- Mass Spectrometer 'tuned' to detect only Helium
- Why we use Helium?
  - Highly mobile, inert, (relatively) available & inexpensive, low surface absorption (easy to pump away) present in air in low quantity (≈ 5 ppm)

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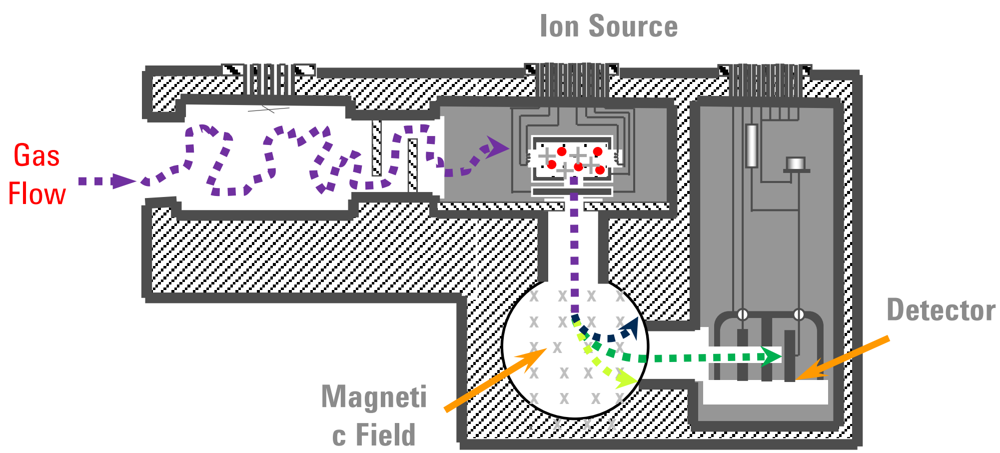
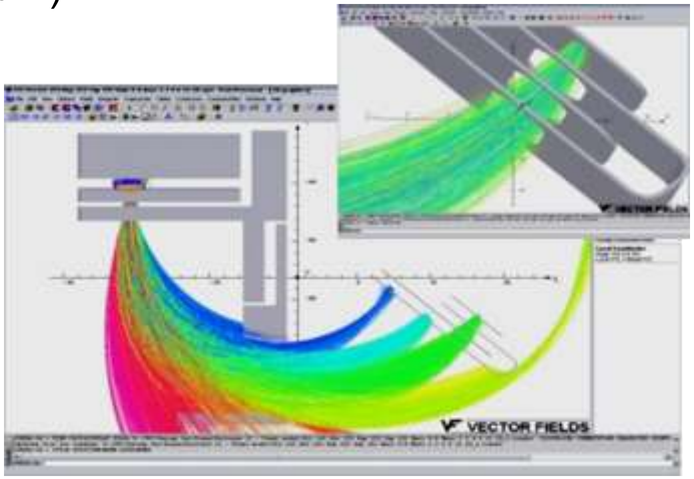


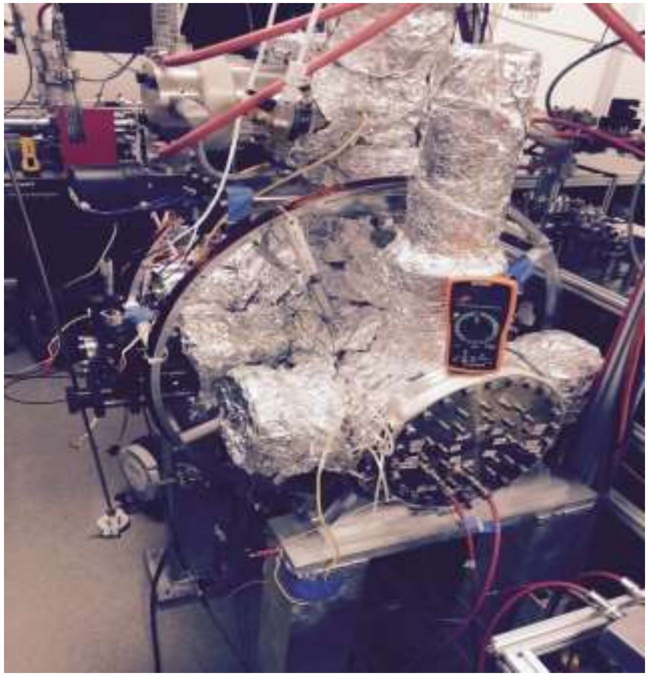


Eliminating VIRTUAL Leaks: Desorption & Diffusion

Desorption & Diffusion

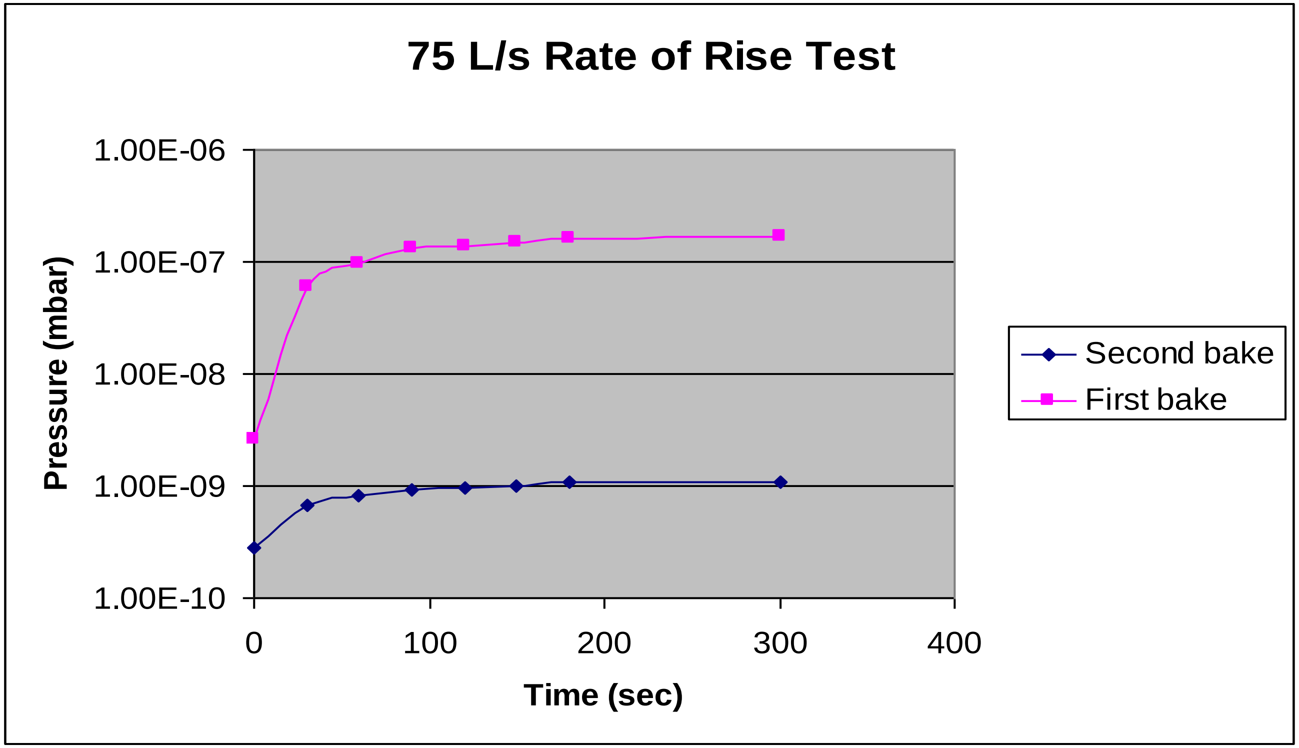
Minimize the amount of moisture entering the vacuum system, and accelerate the rate of Desorption and Diffusion of gases that ARE in the system

- Keep vacuum system interior CLEAN and free of moisture
- When necessary, vent chamber with inert gas
- Minimize exposure of clean parts to air
- Choose materials with high bakeout temperatures
  - **EFFECTIVENESS OF BAKEOUT IS LINEAR WITH TIME BUT EXPONENTIAL WITH TEMPERATURE**



Bake Out Example

- First Bake Was Done With No Insulation and Uneven Heating
- Second Bake Used Insulation to Improve Heating



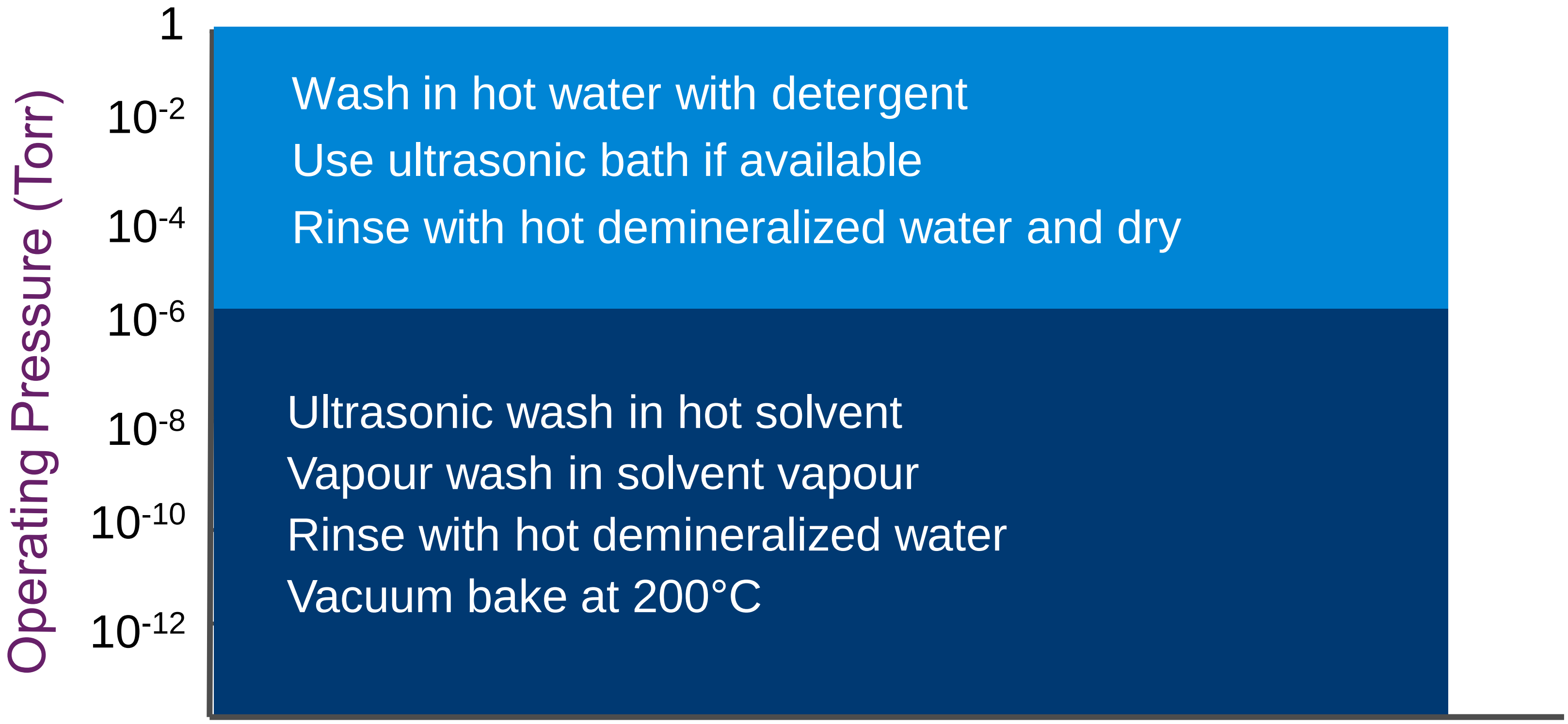
Materials Selection for UHV

Choose UHV Vacuum System materials based on:

1. Vacuum Compatibility
   - Outgassing Rates (available online; temp & surface finish specific)
   - Permeation Rates (available online for metals and elastomers)
   - Bake-Out Temperature (critical in achieving UHV pressures!)

2. Mechanical Properties
   - Physical Strength
   - Conductivity
   - Radiation

Typical Cleaning Material Procedure



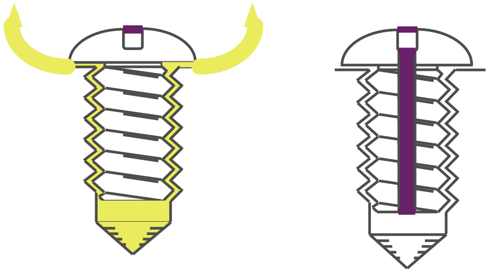
Some Typical Chemical Cleaning Agents (CERN)

Agent	Examples	Advantages	Disadvantages	Disposal
Water		Cheap; readily available	Need to use demineralised for cleanliness. Not a strong solvent	To foul drain.
Alcohols	Ethanol, methanol, iso-propanol	Relatively cheap, readily available, quite good solvents.	Need control – affect workers, some poisonous, some flammable, stringent safety precautions.	Evaporate or controlled disposal.
Organic solvents	Acetone, ether, benzene	Good solvents, evaporate easily with low residue.	Either highly flammable or carcinogenic.	Usually evaporate!
Detergents		Aqueous solutions, non toxic, cheap and readily available, moderate solvents.	Require careful washing and drying of components. Can leave residues.	To foul drain after dilution.
Alkaline degreasers	Almeco™, sodium hydroxide	Aqueous solutions, non toxic, moderate solvents.	Can leave residues and may deposit particulate precipitates.	Requires neutralisation, then dilution to foul drain.
Citric acid	Citrinox™	Cheap and readily available, quite good solvents.	Require careful washing and drying of components. Can leave residues. Unpleasant smell.	To foul drain after dilution

Possible Sources of Leaks in UHV

Virtual Leaks

- Residual solvents (following maintenance cleaning)
- Liquid leaks such as cooling fluids
- Trapped volumes of Gas or Liquid
- Trapped space under non-vented hardware



- Gasses or solvents in spaces With Poor Conductance
- High Vapor Pressure materials
- Porous materials exposed to liquid or atmosphere

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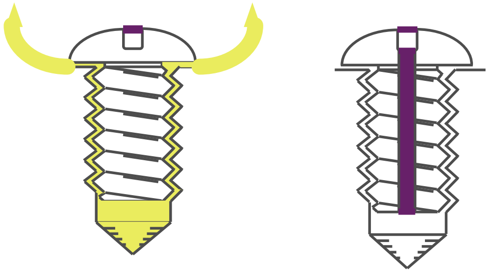

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- All metal seals (minimize use of O-ring seals where practical)
- Use less porous materials
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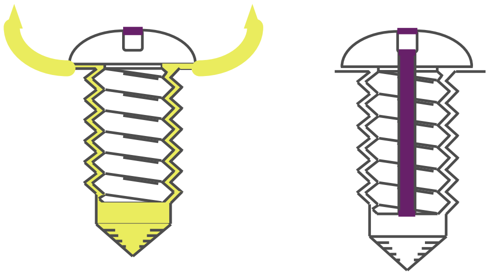

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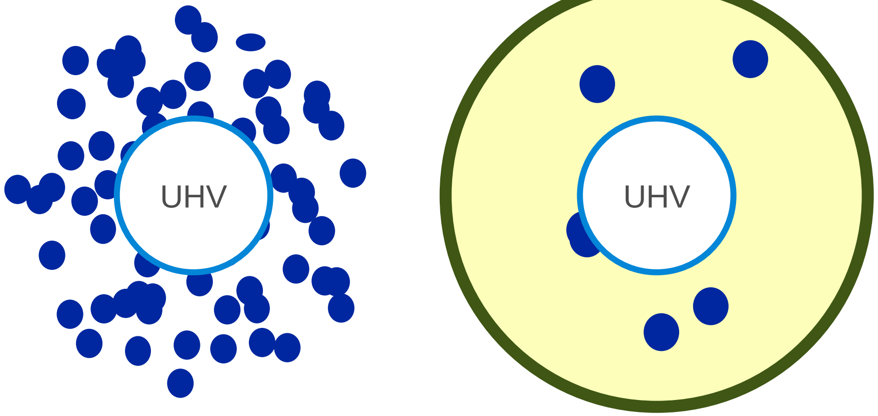
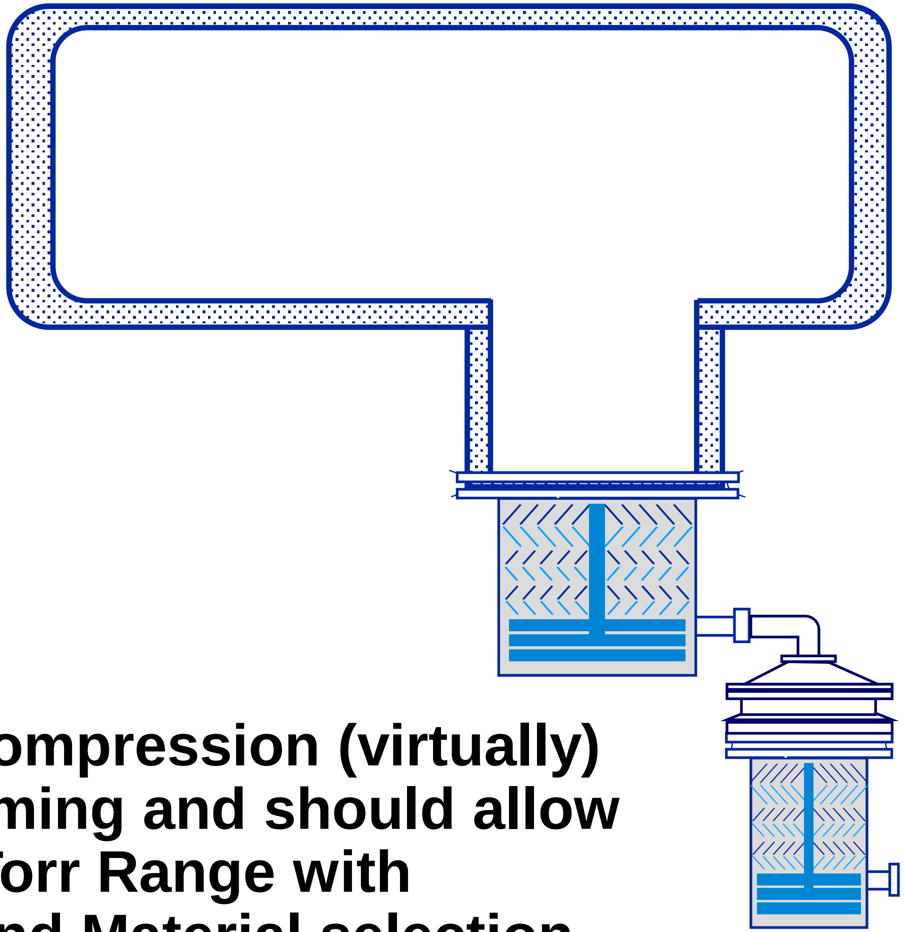


Reducing Back-streaming Through Compression: XHV



Extra DECADES of Compression (virtually) Eliminate Back-streaming and should allow pressures into 10^{-10} Torr Range with appropriate Baking and Material selection

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- ✓ Materials Selection (permeation & 'bake-ability') and Back-streaming must be dealt with to achieve lowest ultimate pressure
- ✓ Techniques for troubleshooting HIGH VACUUM applications include Pumpdown Curves, Rate-of-Rise Tests and Helium Mass Spec Leak Detection

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- Custom multi-day classes at your site
- Other custom training classes to fit your needs

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