Creating, Measuring and Maintaining High and Ultra-high Vacuum: **Session 2** 

CAARI-SNEAP Conference: November 2022

John Screech Sr Applications Engineer Agilent Vacuum Products Division (VPD)





#### Ultra-High Vacuum Seminar: Part 2 of 2 Session Series

John Screech, Senior Applications Engineer, Agilent Technologies, Vacuum Products Division

This class provides further details into the challenges in achieving ultra-high vacuum (UHV) pressure. Session two will delve into the inter-relationship of pumping technologies used from primary vacuum to UHV, system troubleshooting techniques, and material selection. The curriculum for session two of this 90 minute class is excerpted from Agilent's one-day UHV Seminar and is intended to provide an introduction to ultra-high vacuum systems and practice for scientists, engineers and technicians. Attendees will receive a printed copy of the slide deck and are encouraged to add notes to this useful resource.

		<u>Appaloosa</u> (Hybrid)	Pioneer I	Pioneer II	Pioneer III	Pioneer IV	
Thursday, 11/3	9:00 AM	PS-PR-01					
	9:45 AM						
	10:15 AM	ISM-06	WKS-VAC2	AMP-01	IA-03	RE-05	
	11:45 PM						
	12:00 PM	Lunch & Closing Remarks - Quarter					
	1:30 PM		Lab Tour				

You Can Achieve Your Ideal Ultra or Extreme High Vacuum Pressure Easily with Agilent.

Join the comprehensive <u>2-part Ultra-High</u> <u>Vacuum Workshop</u>:

- Part 1: Nov. 2<sup>nd</sup> (Wed.) 2:30 4 pm
- Part 2: Nov. 3<sup>rd</sup> (Thurs.) 10:30 am 12 pm

And schedule a complimentary in-person consultation with our vacuum experts to discuss your application challenges. Limited availability, book your spot now by scanning the code or go to <u>https://calendly.com/agilent-vacuum/carri-sneap-</u> 2022





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## Topics: Session 2

#### Creating & Measuring High Vacuum

- Turbo-, Cryo-, and Vapor Jet ('Diffusion') Pumps
- Ionization Gauges & Wide Range Gauges

#### Creating & Measuring Ultra-high Vacuum

- Sputter Ion Pumps, TSP & NEG pumps
- UHV Ionization Gauges, RGAs & Ion Pump Current
- Troubleshooting High & Ultra-high Vacuum Systems



### Agilent Vacuum Technologies

For over 50 years, Agilent Vacuum Technologies (formerly Varian Vacuum) has developed cutting-edge *Vacuum Solutions* that have enabled scientific discoveries world-wide.



## Agilent Vacuum at a Glance



## Creating High Vacuum

High Vacuum (Atm 10<sup>-3</sup> to 10<sup>-8</sup> Torr) is characterized by fewer particles, behaving independently: Molecular flow.

We no longer have momentum transfer to help move gas into the vacuum pumps.

High Vacuum: 10° Torr - 10° Torr Measuring Vacuum Technologies

High Vacuum: 10<sup>-3</sup> Torr – 10<sup>-8</sup> Torr

# Creating High Vacuum



## Vacuum Technologies Sorted by Pressure





## From Viscous Flow to Molecular Flow

The *effective pump speed* of all our rough vacuum pumps falls off drastically below about 10<sup>-1</sup> Torr





## Vacuum Technologies Sorted by Pressure





## Turbo Pump: Principle of Operation

#### **Axial Compressor**

- Gas is compressed through MOMENTUM TRANSFER
- Gas molecules entering the pump inlet are struck by the first rotating Blade and are deflected downward
- Molecule 'reflects' off the first stationary element (Stator) which has the complimentary angle
- Multiple compression stages (Blade/Stator pairs) can compress gas to about 10<sup>-3</sup> Torr

Turbo pump of this design required **backing pumps** capable of maintaining around 10<sup>-3</sup> Torr at the turbo pump exhaust:

- Dual Stage RVPs
- Dual Stage Scroll Pumps





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### Molecular Drag Stage

• Drag stages compress gas beyond the limits of blades/stators





## Molecular Drag Stages

### Gaede Drag Stage

- Gas molecules 'land' on a surface and spend a finite residence time there
- IF the surface is in motion, molecules <u>leave</u> the surface with a component of momentum in the direction of rotation





# Turbo Pump: Principle of Operation

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### Molecular Drag Stage

- Drag stages compress gas beyond the limits of blades/stators
- Multiple drag stages allow modern turbos to exhaust around 1 - 10 Torr

Molecular Drag pumps require *smaller*, backing pumps with much *'poorer'* ultimate pressure specifications





# Turbo Pump: Principle of Operation

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- Gaede, Holweck and Siegbahn Designs

### MacroTorr (Gaede)





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## TwisTorr (Siegbahn)





## TwisTorr Drag Stage: Principle of Operation

Stators with **planar helical channels** replace the 'stripper' of the Gaede design.

SIX individual compression stages provide longest path for light gas molecules for lowest ultimate pressure.



# Diffusion (Vapor Jet) Pump

Pump fluid (typically hydrocarbon or silicon based oil) is heated and ejected at high speed from a 'jet' forming a conical stream of high velocity particles.

Momentum transfer drives gas molecules outward from the center and down towards the exhaust of the pump.

Multiple jet stages provide a compression effect.



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## Cryogenic Pump

Cryo-condensation: Gas molecules are cooled below the condensation and are captured or trapped on cryoarrays.

Highest capacity for pumping water vapor.

Periodic 'regeneration' is required when pump becomes saturated (unable to maintain sufficiently low temperature to trap gases).



## Vapor Pressure of Some Gases



# Cryogenic Pump

# Pumping Mechanisms:

1st Stage (77 K)
Condensation (H<sub>2</sub>O, NO<sub>2</sub>)

• Cryo-Trapping (Ar<sub>2</sub>)

#### 2nd Stage (< 20 K )

• Condensation (NO, O<sub>2</sub>, N<sub>2</sub>, Ar<sub>2</sub>)

#### Charcoal (< 20 K) • Cryo-Sorption (Ne, H<sub>2</sub>, He)





# Cryogenic Pump

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**Charcoal (< 20 K )** • Cryo-Sorption (Ne, H<sub>2</sub>, He)





## High Vacuum Pump Comparison

Туре	Advantages	Disadvantages
	<ul> <li>Low Ultimate Pressure</li> <li>Clean, Continuous Pumping</li> <li>Smaller Forepump Required</li> </ul>	<ul> <li>Mechanical Bearing</li> <li>Some Vibration</li> </ul>
	<ul> <li>✓ Low Cost</li> <li>✓ No Moving Parts</li> <li>✓ Low Maintenance</li> </ul>	<ul> <li>Backstreaming</li> <li>No Foreline Tolerance</li> <li>May Require Cold Trap</li> </ul>
	<ul> <li>High H<sub>2</sub>O pumping speed</li> <li>Mounts Any Orientation</li> <li>Clean</li> </ul>	<ul> <li>Regeneration Required</li> <li>Affected by Heat</li> <li>Vibration</li> </ul>

## Measuring Vacuum Technologies



## Measuring Vacuum Technologies



## Additional Considerations: HV Gauges



- $\succ$  What gases will the gauge be measuring?
  - Correction Factors
  - Filament Type (if applicable)
- What are the environmental conditions the gauge will be exposed to?
  - Mechanical Shocks
  - Temperature
- Will the gauge interfere with the process I am trying to measure?



# Measuring High Vacuum



## Measuring High Vacuum

High Vacuum (Atm 10<sup>-3</sup> to 10<sup>-8</sup> Torr) is characterized by fewer particles, behaving independently: Molecular flow.

We no longer have momentum transfer to help move gas into the vacuum pumps.

Too few molecules to measure physical force or thermal transfer – transition to lonization Gauges.



## **Ionization Principle**

High speed electrons strike gas molecules in the gauge resulting in the formation of positive ions.

lons are attracted to a negatively charged collector.

Measuring the resulting current provides a measurement of the gas density or pressure.



## Hot Filament Ionization Gauge (BA)

Heated filament provides photo-electrons.

Grid accelerates the free electrons, resulting in the formation of ions.

M<sup>+</sup> lons are attracted to the negatively charged collector resulting in a measurable current.



## Hot Filament Ionization Gauge (BA)



#### **Heated Filament**

• Emits high energy photo-electrons

#### Grid

• Accelerates electrons promoting collisions with (neutral) gas molecules creating M+ ions

#### Collector

 Attracts M+ ions with high negative potential: Resulting current is proportional to gas density or pressure



## Cold Cathode Ionization Gauge



Russell and Sigurd Varian, 1953 Photograph by Ansel Adams © The Ansel Adams Publishing Rights Trust





## Cold Cathode Ionization Gauge

The attempt to produce a "cold cathode" ionization gauge led to the 'accidental' discovery of the sputter ion pump for UHV.




High Vacuum: 10<sup>-3</sup> Torr – 10<sup>-8</sup> Torr

### Cold Cathode Ionization Gauge

Electric and Magnetic Fields create PLASMA within a partially closed cylinder.





High Vacuum: 10<sup>-3</sup> Torr – 10<sup>-8</sup> Torr

### Cold Cathode / Inverted Magnetron Gauge

Cold cathode gauges create a plasma with electrical and magnetic fields and use free electrons to create M<sup>+</sup> ions.



### Cold Cathode / Inverted Magnetron Gauge

Inverted Magnetron design uses HIGHER voltage (to initiate and sustain plasma) and INVERTS the voltages (ions created at High Voltage and attracted to Grounded cathode).



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#### High Vacuum: 10<sup>-3</sup> Torr – 10<sup>-8</sup> Torr

### Cold Cathode / Inverted Magnetron Gauge



#### **Electric and Magnetic Fields**

- Create plasma within a hollow stainless steel (typ.) cylinder
- Accelerate electrons to collide with (neutral) gas molecules creating M+ ions

#### Collector

- Attracts M+ ions with high negative potential
- Resulting current is proportional to gas density i.e. **pressure**



#### High Vacuum: 10<sup>-3</sup> Torr – 10<sup>-8</sup> Torr

# Ionization Gauge Comparison

		Advantages	Disadvantages	
	Hot Filament Ionization	<ul> <li>More accurate than CC / IMG</li> <li>Slightly wider range</li> </ul>	<ul> <li>X-Ray effect limits accuracy below 10<sup>-8</sup> Torr range</li> <li>Gas type dependent</li> </ul>	
Nrg-107	Cold Cathode / Inverted Magnetron	<ul><li>More rugged than Hot Ion gauge</li><li>Rebuildable</li></ul>	<ul><li>Limited accuracy, range</li><li>Gas type dependent</li></ul>	





# Wide Range Gauges

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Wide Range Gauges

#### **Combination Gauges**

Multiple pressure sensors can be combined in a single housing, to produce a gauge that accurately measures across rough, high, and ultra-high vacuum pressures.

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#### Wide Range

# Wide Range Gauges

Sensors with complimentary ranges can be combined to produce Wide-Range gauges.



Measuring Vacuum: Combination Gauges

## Agilent Wide Range Gauges

Sensors with complimentary ranges can be combined to produce Wide-Range gauges.



#### Measuring Vacuum: Combination Gauges

# Agilent Wide Range Gauges





# Creating Ultra-high Vacuum



## Targeted Pumping

UHV customers use *multiple* pumping technologies to optimize pumping speeds for individual gas species.



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# Anatomy of a Diode Pump

High Voltage between Anode and Cathode creates plasma (ions & free electrons).

Electrons are accelerated by 1-2 KGa Magnetic Field parallel to Anode axis.

Electrostatic attraction draws the M+ ions towards the Cathode (typically Titanium).



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## Ion Pump Selection: Gas Type

Choosing the best Ion Pump for your application requires careful consideration of the dominant gas species you are trying to remove.



#### Diode

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### Ion Pump Mechanisms



#### **Gettering at Anode Surface**

Chemically active gas ions react with the active Ti surface forming stable compounds (e.g. TiO and TiN)

### Ion Pump Mechanisms



#### Gettering at Anode Surface

Chemically active gas ions react with the active Ti surface forming stable compounds (e.g. TiO and TiN)

#### **Diffusion into Cathode**

Titanium's very high affinity for  $H_2$  can pump 'large' quantities of gas. He can be re-emitted by heating.



### Ion Pump Mechanisms



#### Gettering at Anode Surface

Chemically active gas ions react with the active Ti surface forming stable compounds (e.g. TiO and TiN)

#### **Diffusion into Cathode**

Titanium's very high affinity for  $H_2$  can pump 'large' quantities of gas. He can be re-emitted by heating.

#### Pumping Noble Gases

Ar, Xe, Kr are immune to active Ti surface: Diffusion produces unstable pumping as noble gases are only lightly buried in Ti Cathodes.



## Ion Pump Selection: Gas Type

Choosing the best Ion Pump for your application requires careful consideration of the dominant gas species you are trying to remove.





### A Better Solution: Triode Design





# Titanium Cathodes consist of louvred strips.

Cathodes held at high negative potential while circular Anodes are grounded.



### A Better Solution: Triode Design



Titanium is sputtered from <u>both</u> Cathodes by M+ ions.

## Optimized Triode: Agilent StarCell<sup>™</sup>

Star shaped cut-outs in Titanium Cathodes promote 'glancing' collisions. Reflection of Neutrals improves pumping speed for Noble Gases.

- Dual Titanium cathodes provide maximum capacity for H<sub>2</sub> pumping (diffusion)
- 'Controlled Erosion' of Ti material eliminates weakness of 'classic triode' design





## Ion Pump Selection: Gas Type

Choosing the best Ion Pump for your application requires careful consideration of the dominant gas species you are trying to remove.

	<b>—</b>
Hydrogen	
Helium	
Water	
Methane	
Nitrogen	
O <sub>2</sub> , CO, CO <sub>2</sub>	,
Argon	,





# **Complimentary Ion Pumps**

#### Titanium Sublimation Pump (TSP)

High current applied to filament creates Active Titanium surface creating stable compounds (Oxides and Nitrides) with getterable gases.





#### Non-Evaporative Getter (NEG) Pump

Substrate coated with thin layer of reactive material (e.g. Zr, V, Fe alloy) that adsorbs gas through Diffusion and the formation of stable compounds (Oxides and Nitrides)



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## Ion Pump Performance Rating

Combining a <u>Complementary</u> <u>Technology</u> (TSP or NEG Pump) with one of the Sputter Ion Pumps results in best <u>overall pumping ability</u> for most applications.

	Diode	Noble Diode	(Optimized Triode)	or NEG
Hydrogen	****	****	****	****
Helium	****	****	****	****
Water	****	****	****	****
Methane	****	****	****	****
Nitrogen	****	****	****	****
O <sub>2</sub> , CO, CO <sub>2</sub>	****	$\star \star \star \star$	****	****
Argon	****	****	****	****

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### Best Overall UHV Pump Performance

Combining a <u>Complementary</u> <u>Technology</u> (TSP or NEG Pump) with one of the Sputter Ion Pumps results in best <u>overall pumping ability</u> for most applications.

Hydrogen
Helium
Water
Methane
Nitrogen
O <sub>2</sub> , CO, CO <sub>2</sub>
Argon



# Measuring Ultra-high Vacuum



# Measuring Vacuum Technologies



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### Challenges when Measuring UHV Pressure

Ultra-High Vacuum (10<sup>-8</sup> to 10<sup>-11</sup> Torr) presents challenges when measuring pressure. Modifications to High Vacuum ionization gauges made to address or mitigate the factors which compromise their performance at very low pressures



# Measuring Vacuum Technologies



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# Hot Filament Ionization Gauge (for UHV)



#### Eliminate Glass Envelope

• Pyrex glass is a permeation leak

#### No O-Ring Seals

• All-metal seals appropriate for UHV

#### No Elbow Connection

• Measure the pressure closer to point of interest

#### Reduce the Size of Grid and Standoffs

• Lessens impact of x-rays on measurement accuracy

# Hot Filament Ionization Gauge (for UHV)



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#### Reduce the Size of Grid and Standoffs

• Lessens impact of x-rays on measurement accuracy



### X-Ray Limit for Hot Ionization Gauges

At pressures  $< 10^{-8}$  Torr electrons striking the grid create low-energy x-rays. When the x-rays strike the collector, they cause the emission of photoelectrons that cause an offset current, distorting the pressure reading.





## Inverted Magnetron Gauge (for UHV)

Higher 'strike' voltage improves ability to ignite and sustain plasma (esp. at UHV pressure).

All metal seal better suited to UHV applications.





## Inverted Magnetron Gauge (for UHV)



**Better Start Capability at UHV Pressure** Starts more reliably at pressures below 10<sup>-8</sup> Torr

All Metal Seals Appropriate for UHV systems



### Residual Gas Analyzer

Residual gases at HV/UHV pressures can be ionized and separated using a quadrupole mass filter. Individual ions can be counted by a Faraday Cup or electron multiplier.



SOURCE: SRS RGA300 Residual Gas Analyzer
### **Residual Gas Analyzer**

Partial pressures of gas species can be added to estimate total vacuum pressure in a chamber.



SOURCE: SRS RGA300 Residual Gas Analyzer



## Sputter Ion Pump Current





#### As UHV Gauge

Ion pump current is proportional to pressure so the ion pump can be used as a vacuum gauge.

• Low pressure limit (10<sup>-10</sup> Torr range) driven by leakage current (roughly  $\alpha$  ion current potential)



### Ion Pump Current Measures UHV Pressure

Low pressure limit driven by leakage current.



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LEAKAGE CURRENT CAUSES ERRONEOUS READINGS

# Sputter Ion Pump Current

Agilent **4UHV Controller** can operate up to 4 sputter ion pumps.

**STEP VOLTAGE** function reduces applied voltage after ignition, reducing leakage current and enabling accurate pressure measurement in the 10<sup>-10</sup> Torr range.



# UHV Gauge Comparison

		Advantages	Disadvantages
	Hot Filament Ionization	<ul><li>Wide pressure range</li><li>Better accuracy</li></ul>	<ul> <li>'Hot' Filament produces photo- electrons</li> <li>Additional wire required for collector</li> </ul>
	Inverted Magnetron	<ul><li>Rugged &amp; rebuildable</li><li>No 'Hot' filament</li></ul>	<ul> <li>Limited accuracy, range</li> </ul>
	Residual Gas Analyzer	<ul> <li>Identifies WHICH gas species are present</li> </ul>	<ul> <li>Limited accuracy when measuring total pressure</li> </ul>
e Menange Bandaria 6.6e-11 Mar e an e an e an e an e an e an	Ion Pump Current	• 'Free' Vacuum Gauge	<ul> <li>Leakage current can compromise accuracy</li> <li>Location of pressure measurement</li> </ul>



Introduction to UHV

# Troubleshooting HV & UHV Systems



# Identifying the Dominant Gas Source

Understanding the dominant gas source in a vacuum system is *critical* when troubleshooting High Vacuum and Ultra-high Vacuum systems



# Desorption







# Desorption

Removing ALL the water from a system is a requirement for achieving UHV pressure in a chamber or beam line:

- Bake-out effectively removes water vapor & slowly reduces Hydrogen and other gases (CH<sub>4</sub>, CO, CO<sub>2</sub>)
- Chambers must be heated AND cooled evenly
- Effectiveness of bake-out is LINEAR with Time, but EXPONENTIAL with Temperature
- User TURBO pump to REMOVE desorbed gases from the system
- Vent with dry gas



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### Permeation







# Permeation

Reducing permeation leaks (gas loads) begins at the design phase for HV & UHV systems

- Choose materials with lowest permeability for gases of interest
- All-metal seals are preferred for HV but essential for UHV systems
- Avoid materials with high permeability for He as these will interfere with Leak Testing



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# Diffusion







# Diffusion

Diffusion of gas molecules from bulk materials inside the vacuum chamber is typically the dominant gas source between 10<sup>-7</sup> and 10<sup>-9</sup> Torr

- Avoid using materials that will absorb water inside a vacuum chamber
- Pre-bake system components and install under 'clean-room' conditions
- Vent system infrequently and use dry gas during vent-up





# Virtual Leak







# Virtual Leaks

Virtual Leaks that are NOT associated with Outgassing (Desorption & Diffusion) are typically related to pockets of gas in areas of *poor conductance* within a vacuum system

- Eliminate 'blind' screw-threads at design stage (use vented or slotted screws where necessary)
- Cross-drill chamber bores where appropriate
- Avoid narrow tubulations (eg Vacuum Gauges) that can result in trapped gases
- Solvents used for cleaning must be evacuated before their benefit is realized



Vacuum

Chamber



Real Leak





## Real Leaks

Gas molecules at Atmosphere (and even at Rough Vacuum pressure) are EXTREMELY motivated to enter a High or Ultra-high Vacuum system through compromised chamber seals or chamber defects:

- Seals where motion occurs
- Sliding Seals
- Rotating Seals
- Seals on chamber doors
- Bellows seals on valves
- Welds and brazed joints





# Backstreaming







# Backstreaming

When operating at or near its compression limit, a turbo-pump can become a source of gas molecules

- Light gases are (relatively) poorly compressed by turbo-pumps and can travel backwards through the pump (Helium Leak Detection depends on this!)
- Desorbed gases can condense inside the body of a turbo-pump and release after baking cycle is complete
- Ability to isolate the turbo-pump from the chamber or beamline is essential for achieving UHV pressure

#### Maximize Performance of UHV System Pumps



# Agilent Resources



https://www.agilent.com/en/solutions/vacuumsolutions/particle-plasma-physics

Book a Meeting with Agilent at CAARI-SNEAP 2022:



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#### Vacuum Technology Webinar Series

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# QUESTIONS?

Performance, innovation and attention to detail

# Thank you



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