



# **Progress in Vacuum Pressure Measurement**

**Dr. Martin Wüest**

**Balzers, Principality of Liechtenstein**

# Overview

- Historical Development, Drivers
- The most common vacuum gauge types
- Example of improving our product using rarefied gas dynamics
- Optical pressure sensing

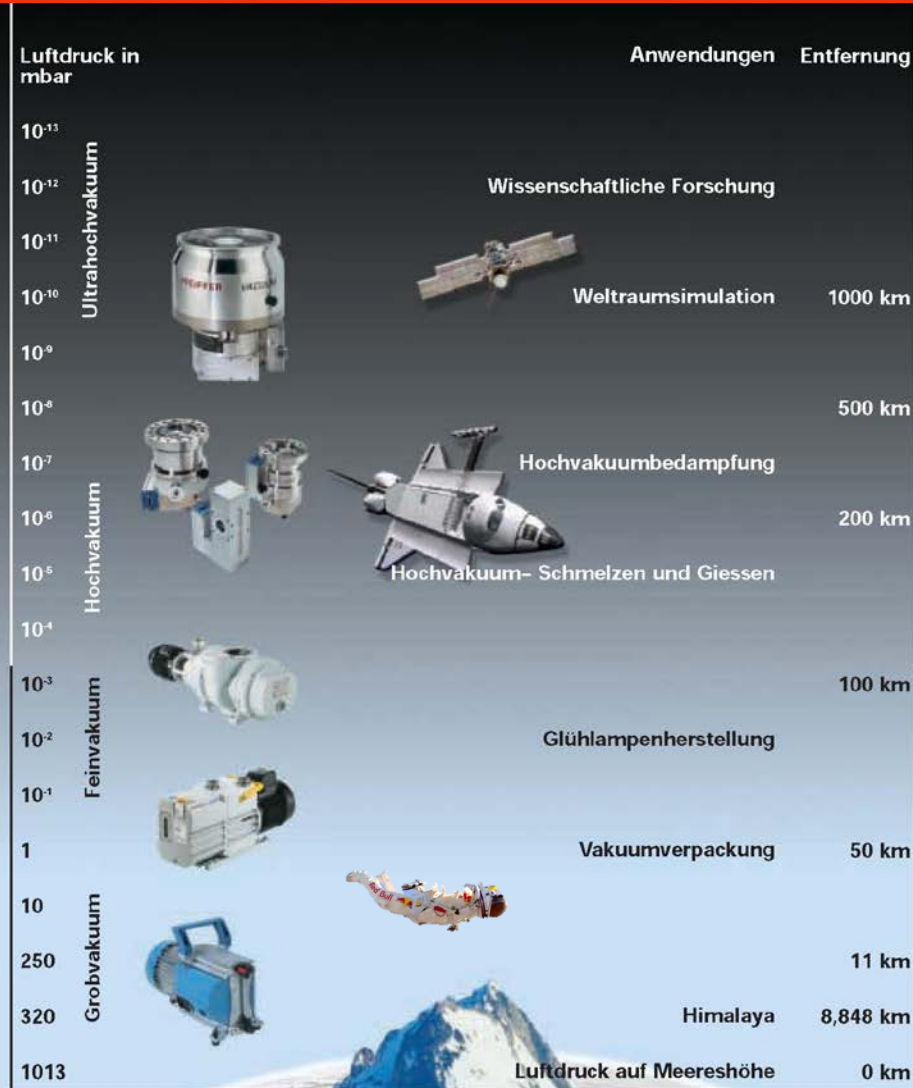
# What is Vacuum?

Particle density [# / cm<sup>3</sup>]

241

2.4 E22

2.4 E25

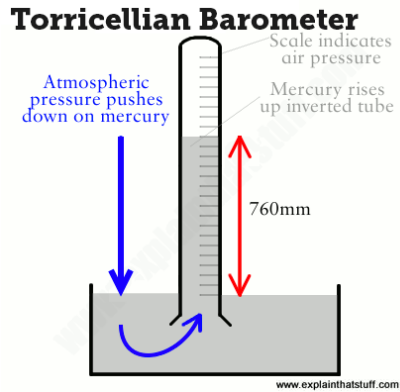


Mit Vakuum bezeichnet man den leeren Raum, das heißt ein nicht mit Luft oder einem anderen Gas gefülltes Volumen. Ideale Vakuumbedingungen findet man im interstellaren Raum. Hier herrscht eine Teilchendichte von einem Atom pro cm<sup>3</sup> vor. Im Labor oder in der Industrie erzeugt man Vakuum durch den

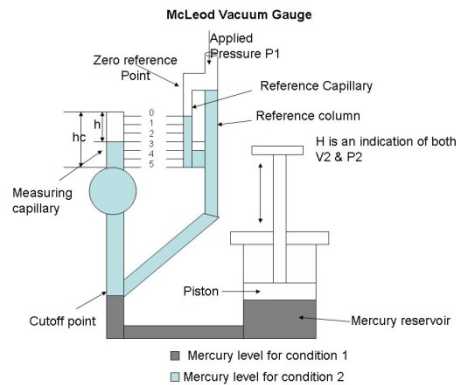
Einsatz von verschiedenen Vakuumpumpen. Je nach Anwendung wird an die Qualität des Vakuums eine unterschiedliche Anforderung gestellt. Man unterteilt daher die Vakuumanwendungen nach Grob-, Fein-, Hoch- und Ultrahochvakuum.



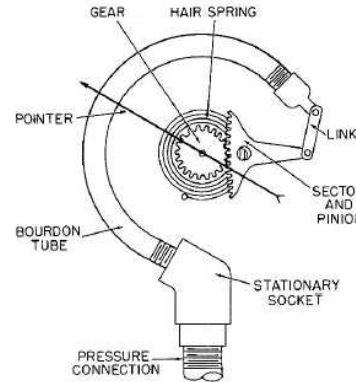
# Early History



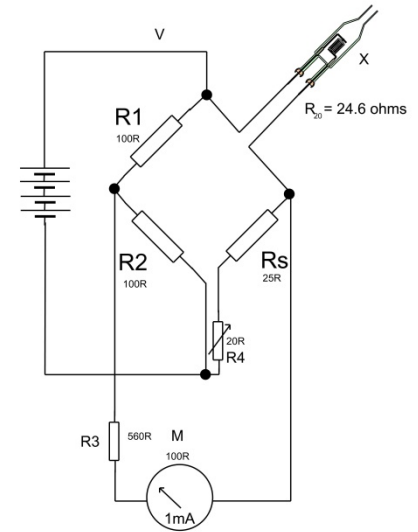
Barometer  
1643 – 1860+



McLeod  
1874 – 1916+



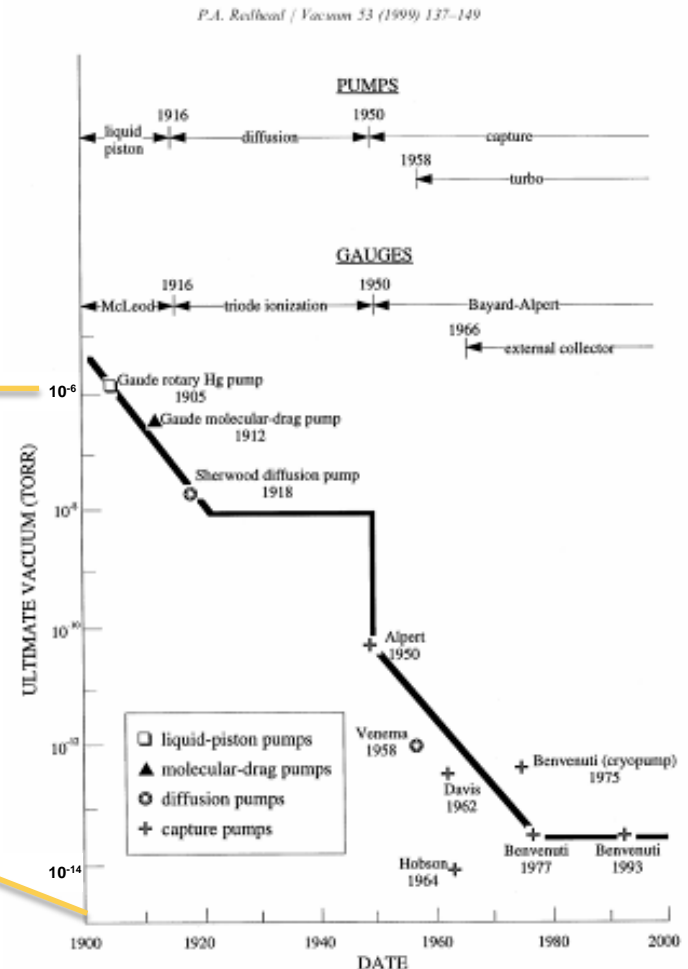
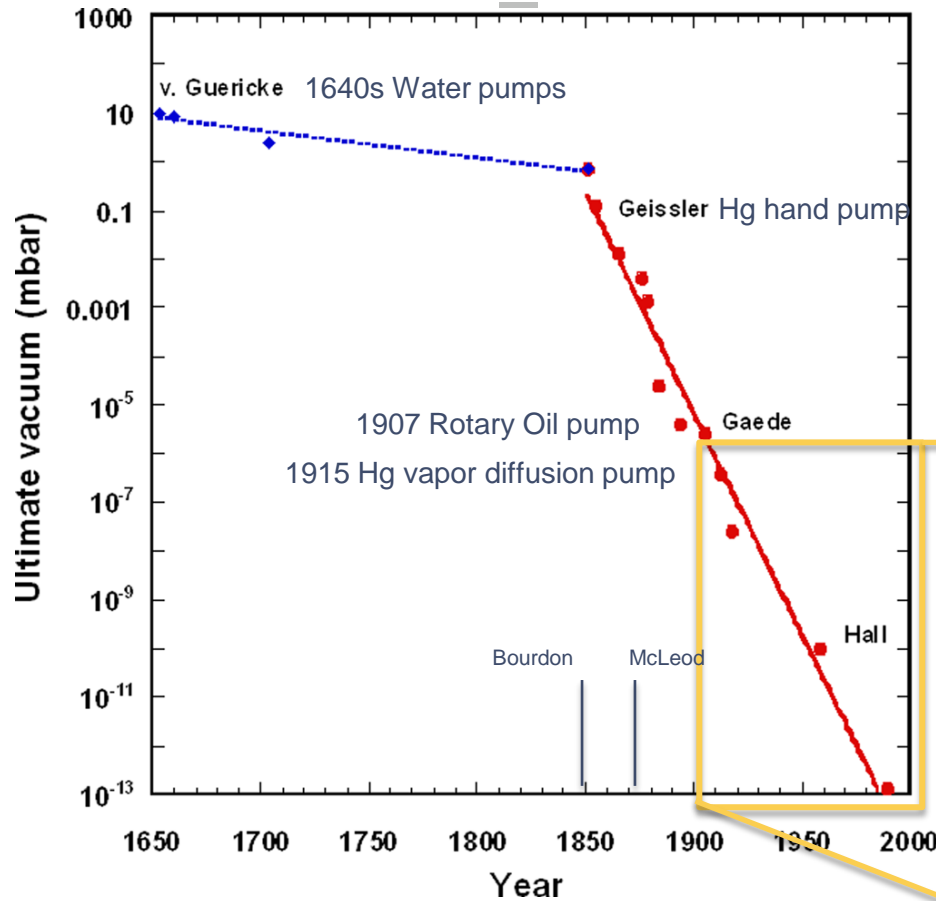
Bourdon  
1849 -  
mechanical



Pirani  
1906 -  
electrical



# The Quest for the Ultimate Vacuum



Georg Gaertner  
 J. Vac. Sci. Technol. B 30, 060801 (2012); doi: 10.1116/1.4747705

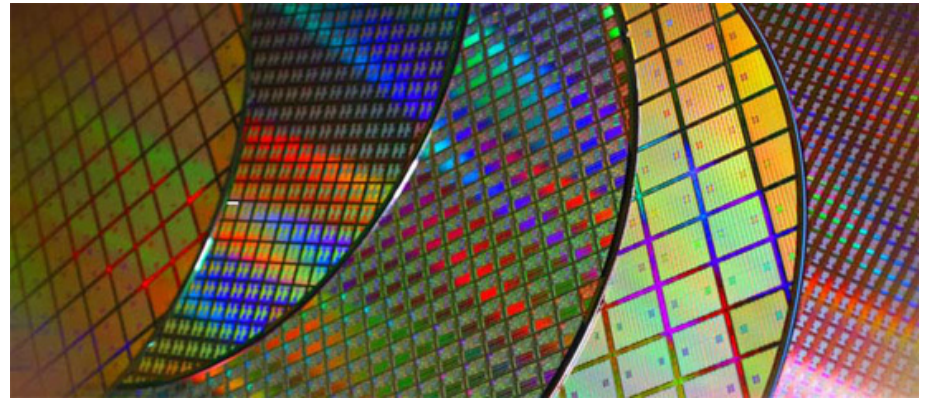
P.A. Redhead, Vacuum 53, 137 (1999)

# Changing Markets

- Incandescent Lights



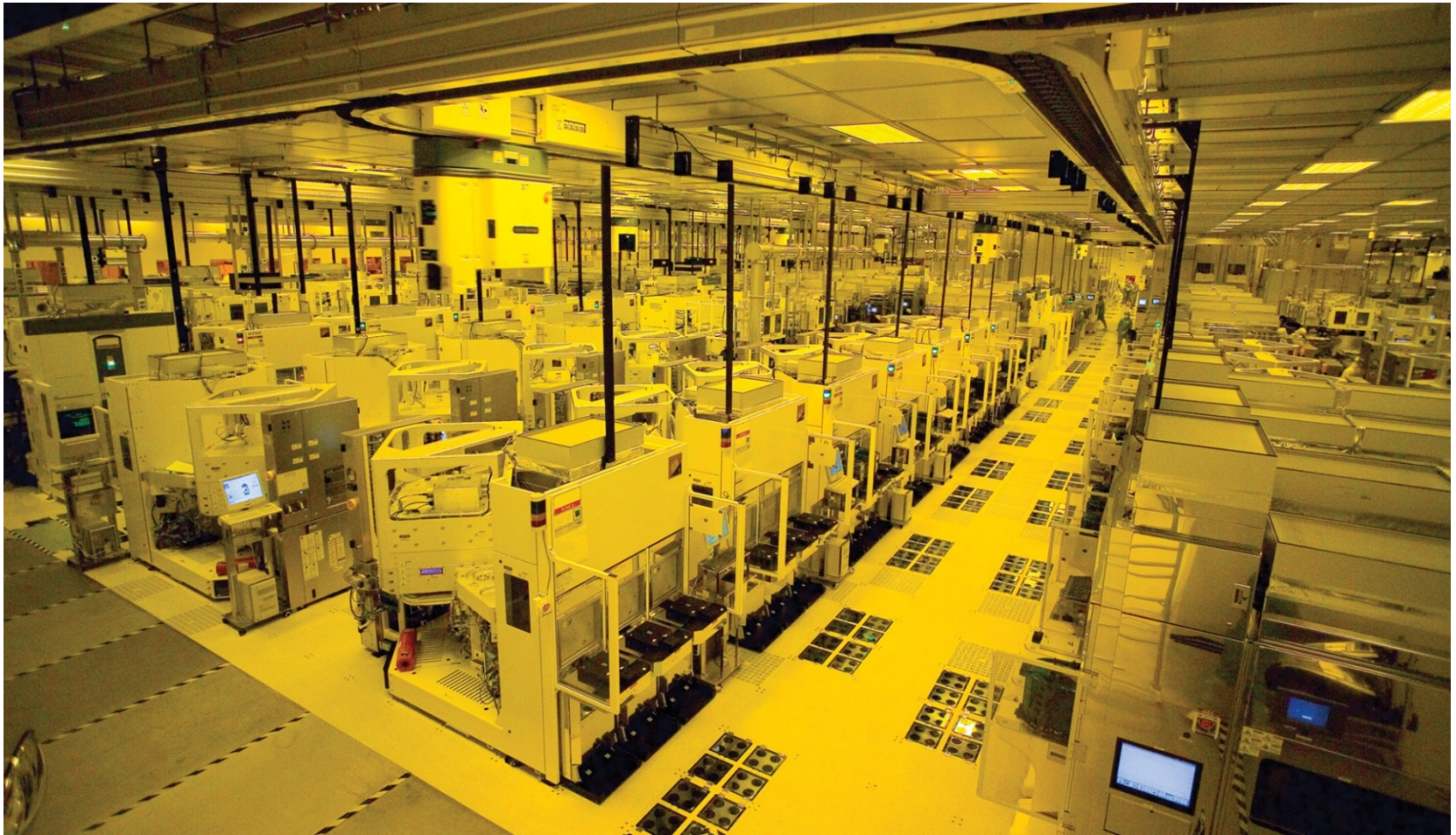
- Semiconductor



[http://www.chineselight.com/uploads/130107/1\\_114009\\_1.jpg](http://www.chineselight.com/uploads/130107/1_114009_1.jpg)  
[http://www.kitguru.net/wp-content/uploads/2014/07/semiconductor\\_umc\\_wafers.jpg](http://www.kitguru.net/wp-content/uploads/2014/07/semiconductor_umc_wafers.jpg)

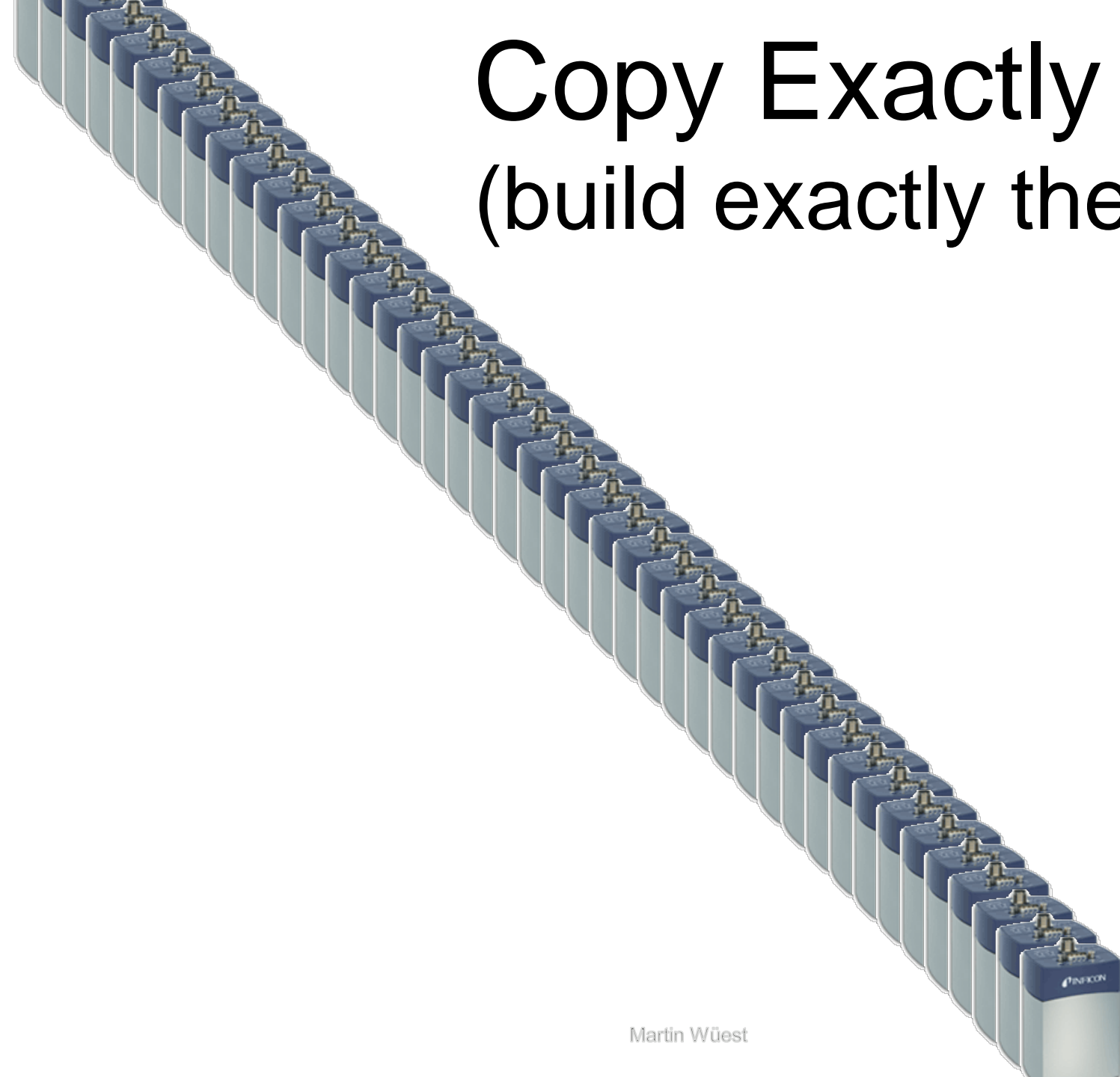
# The Quest for Reproducibility

# Semiconductor Manufacturing

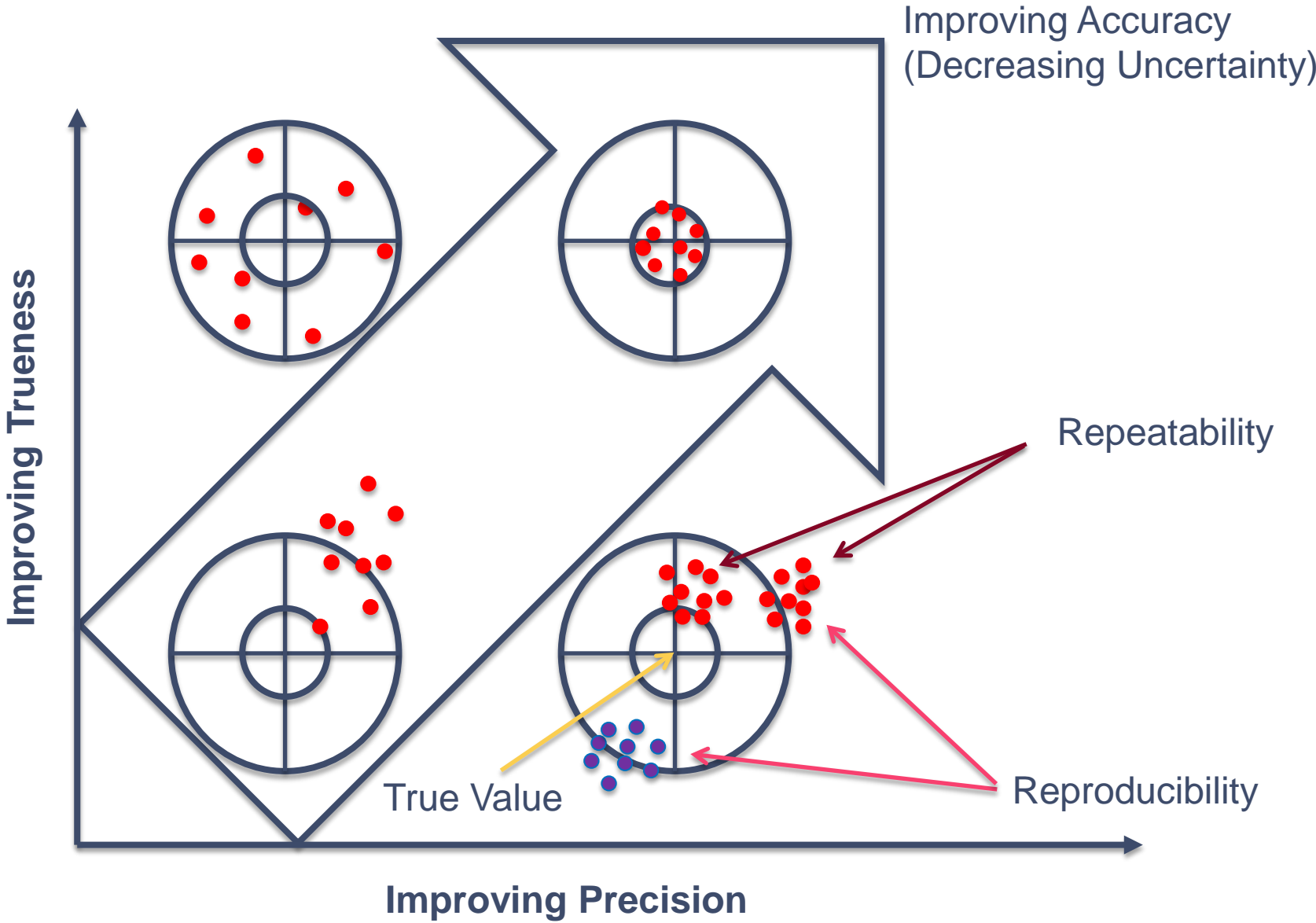




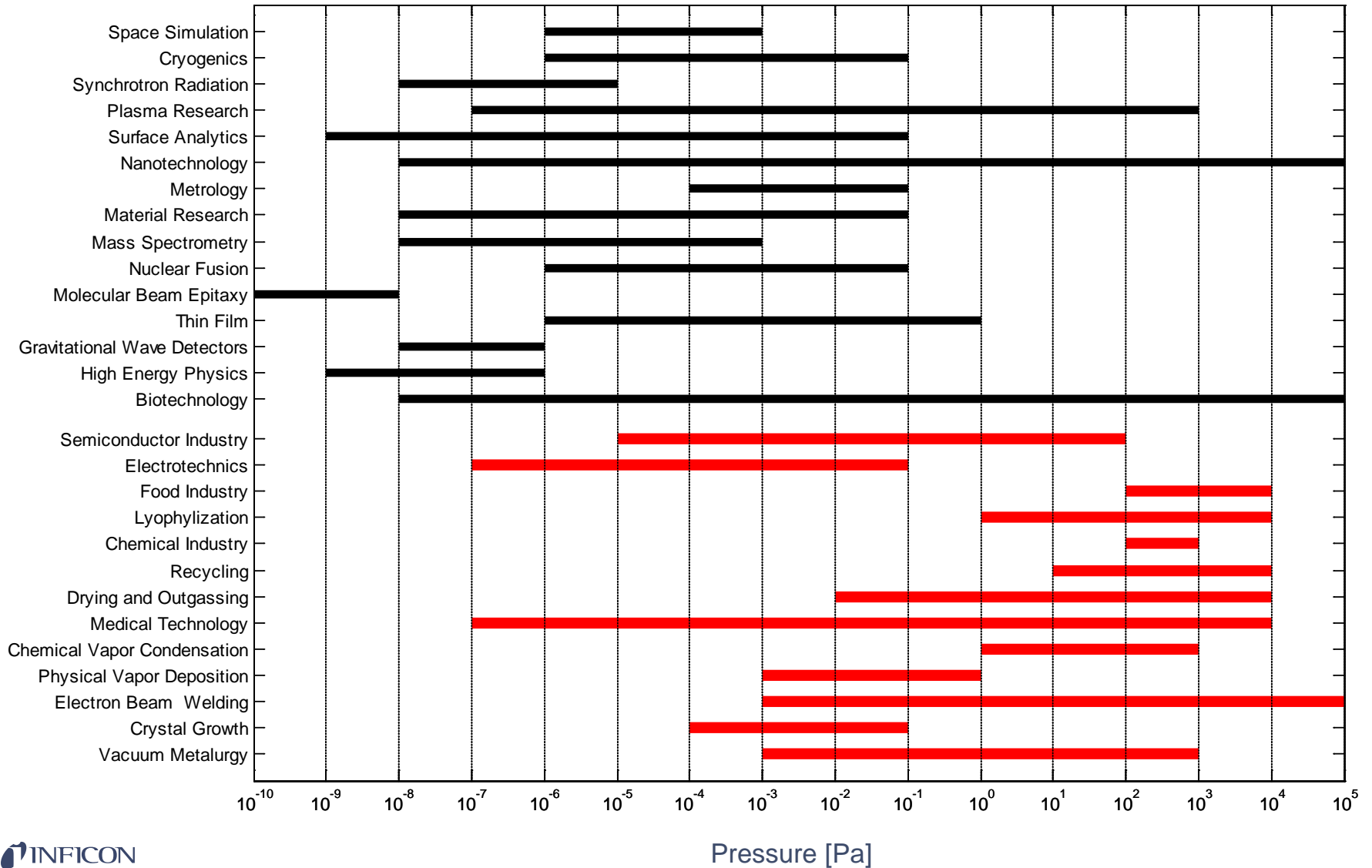
# Copy Exactly ! (build exactly the same)



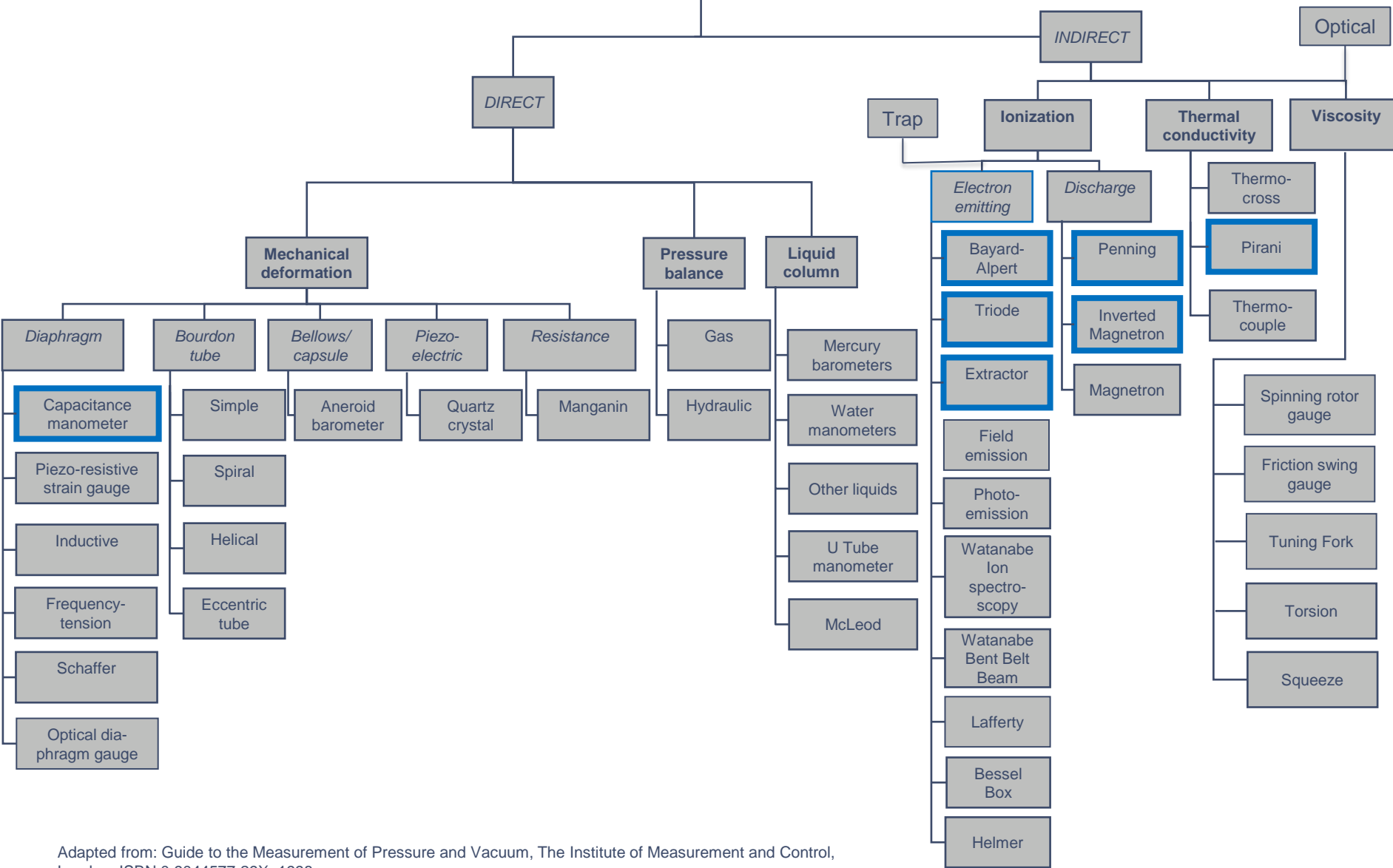
# Definitions



# Process Ranges



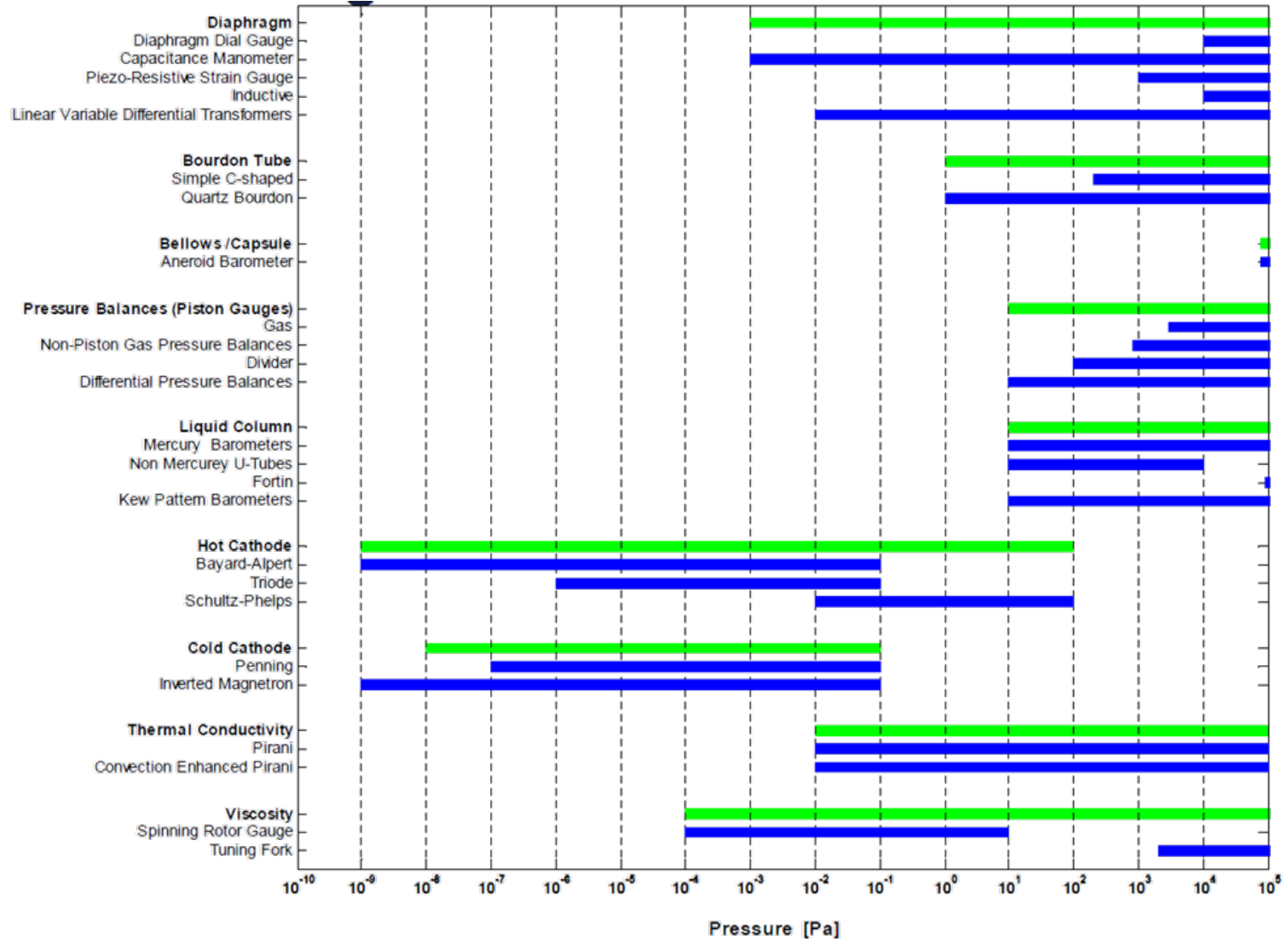
MEASUREMENT PRINCIPLES



Adapted from: Guide to the Measurement of Pressure and Vacuum, The Institute of Measurement and Control, London, ISBN 0 9044577 29X, 1998



# Vacuum Pressure Sensors Ranges



# The Ubiquitous Four

- Capacitance Diaphragm Gauge



- Pirani



- Hot Ionization Gauge



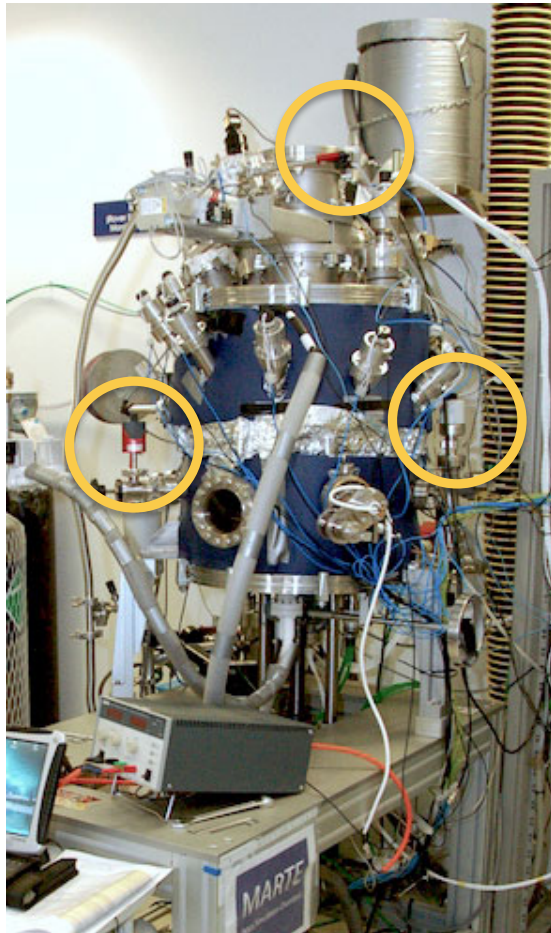
- Cold Cathode Gauge



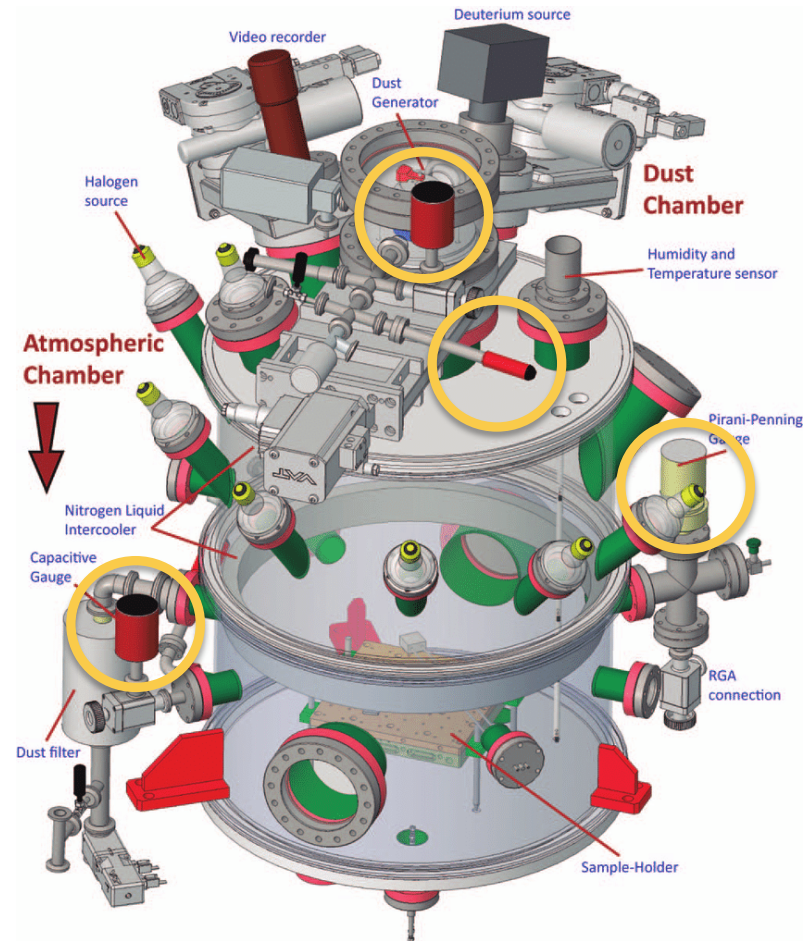
# Pirani in the Market Place



# MARTE, Mars Environment Simulation Chamber

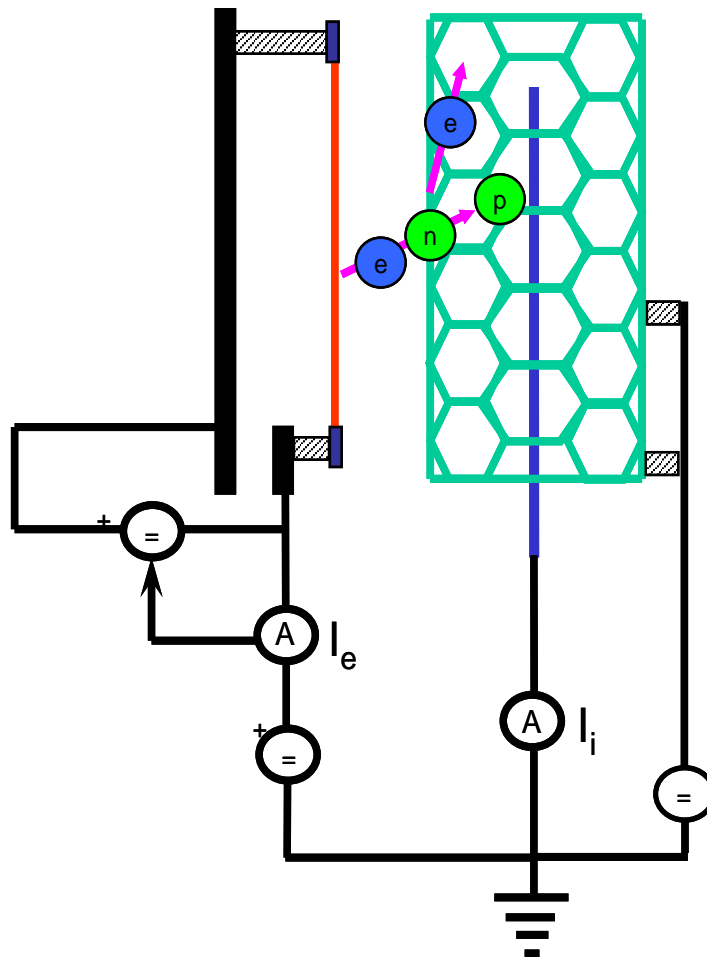


Jesus Sobrado, Juan Manuel Manchado and Jose Angel Martin-Gago  
Mimicking the Martian surface, Physics World, Aug 2015



J. M. Sobrado et al., Rev. Sci. Instrum, 85 035111, 2014

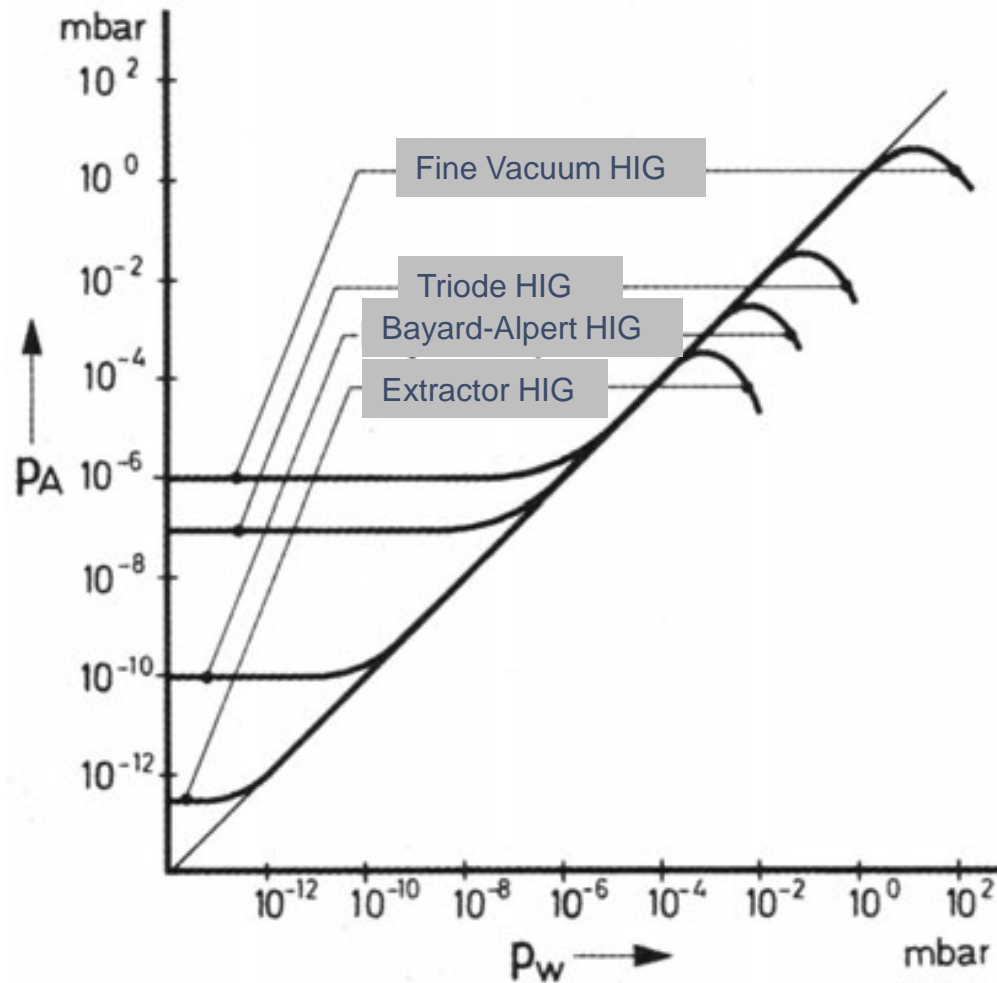
# Hot Ionization Gauge







# Hot Ionization Ranges



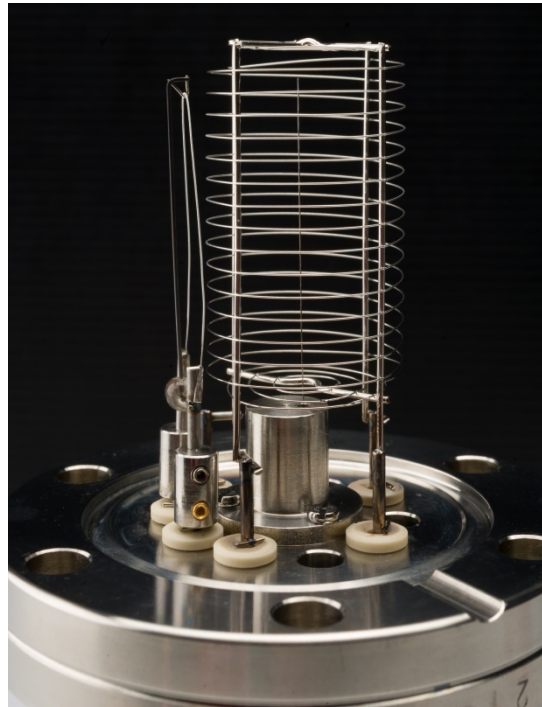
# INFICON Manufactured Bayard-Alpert Hot Ionization Gauges



BAG052



BAG051

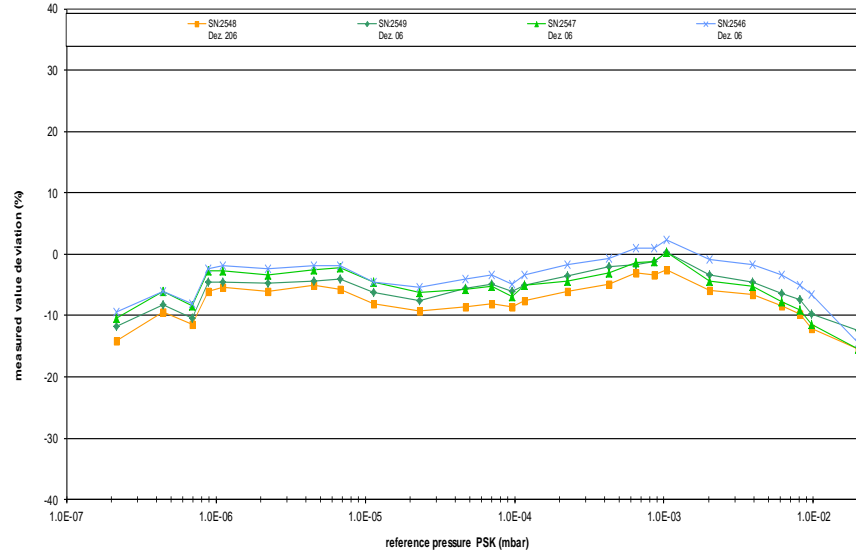
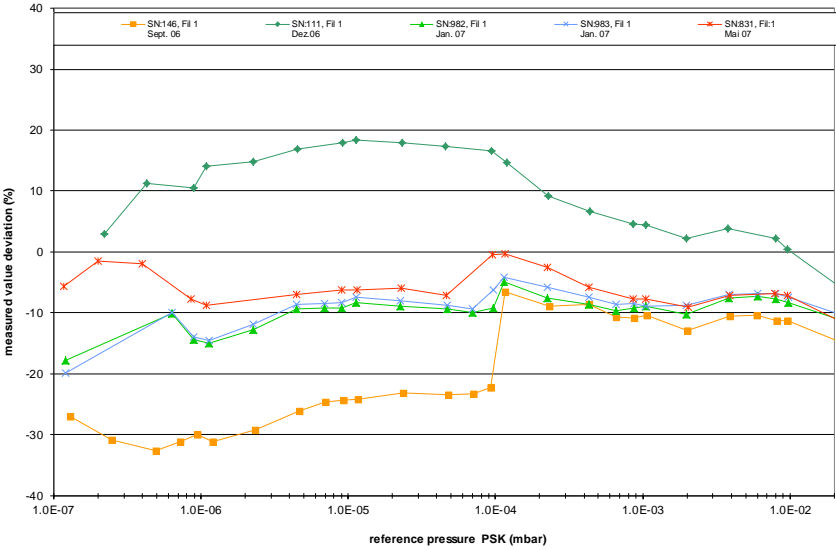


IE414

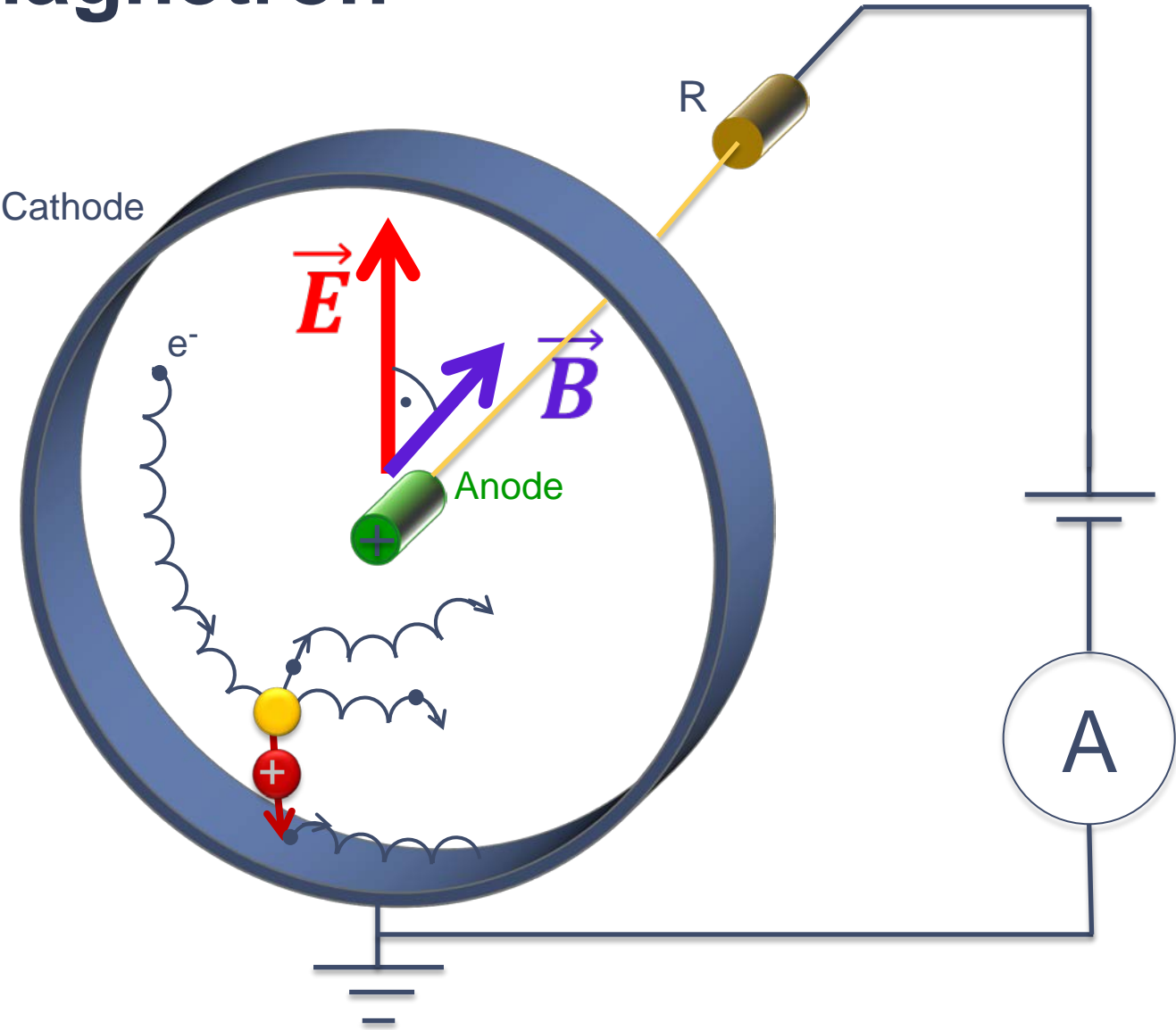


BAG402



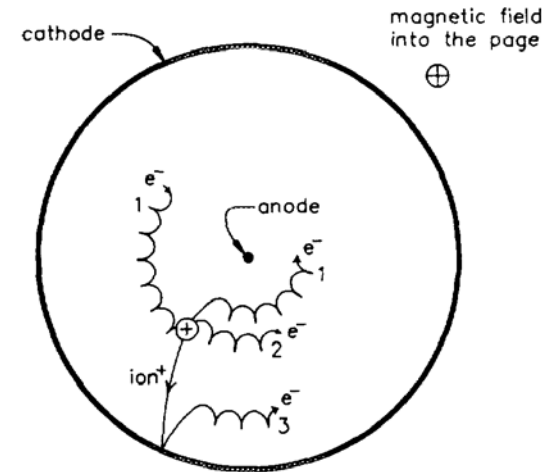
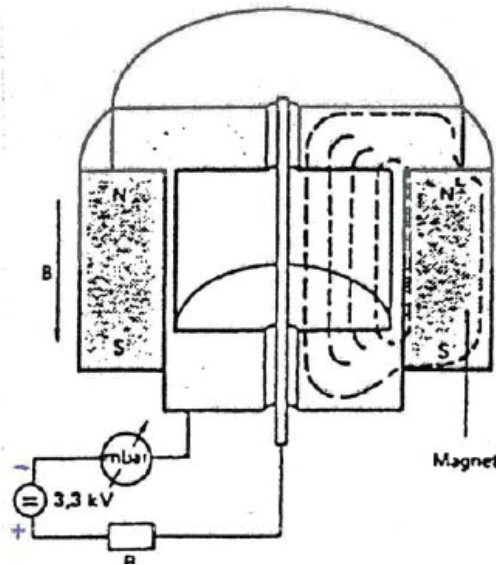


# Inverted Magnetron



$$I^+ = Cp^n$$

# Inverted Magnetron Ion Gauge



R.N. Peacock et al., JVST A9, 1977, 1991

# Inverted Magnetrons

passive

active



HV1

1960's



IKR013  
(CERN)

1972



IKR010

1977



IKR020

1980-1995



MPG400

1993

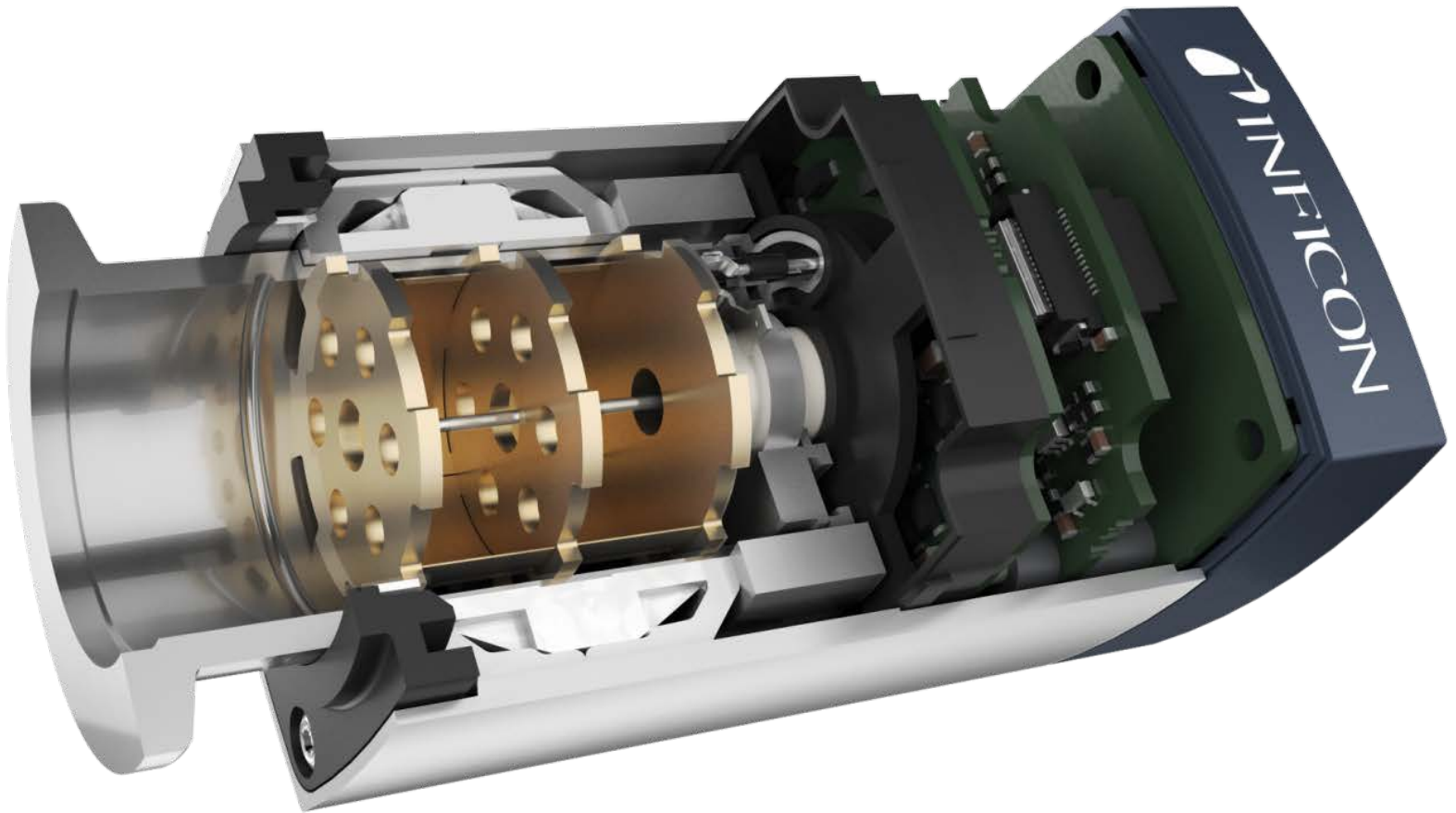


MPG500

2013

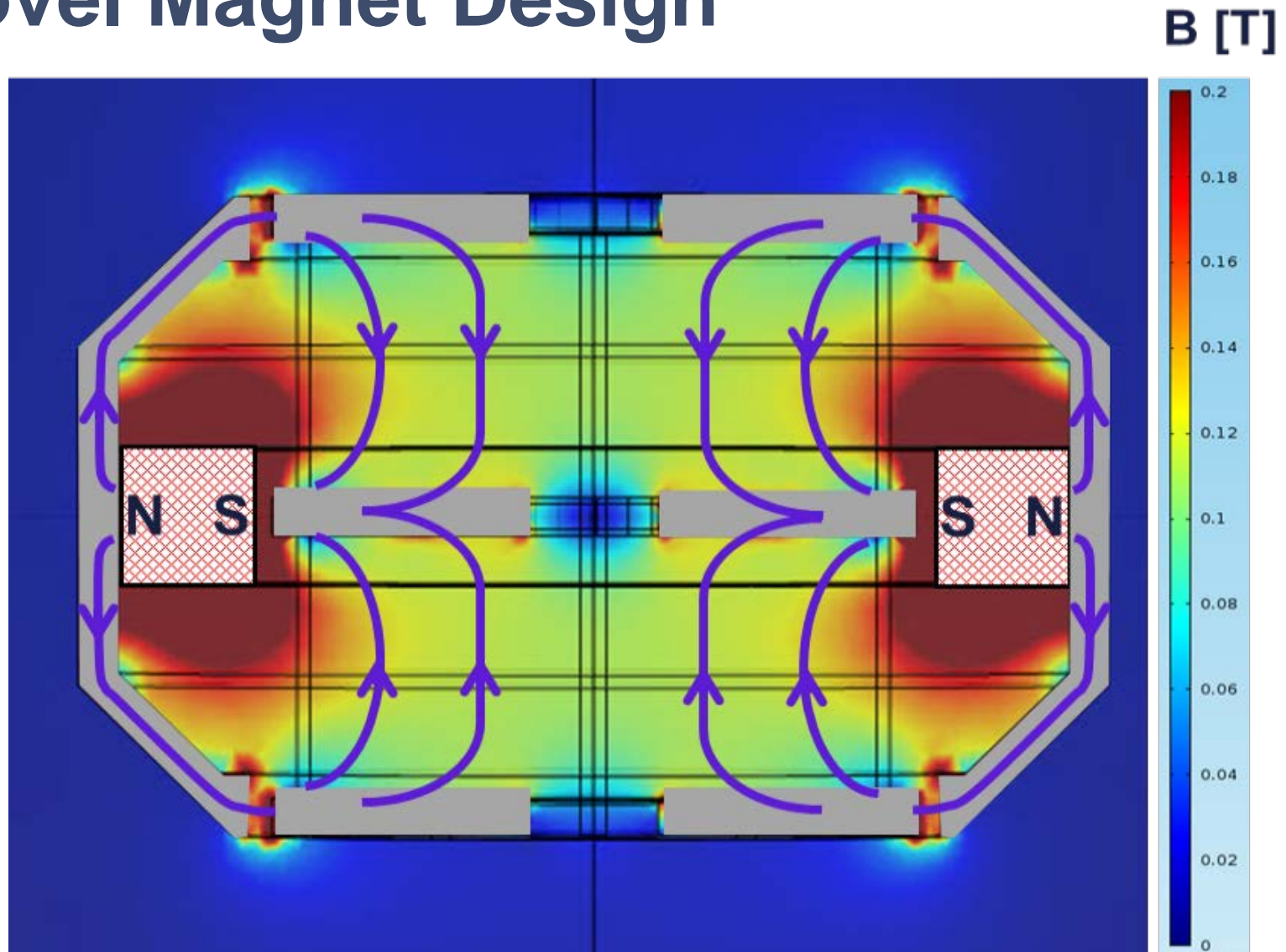


# Inverted Magnetron MPG550

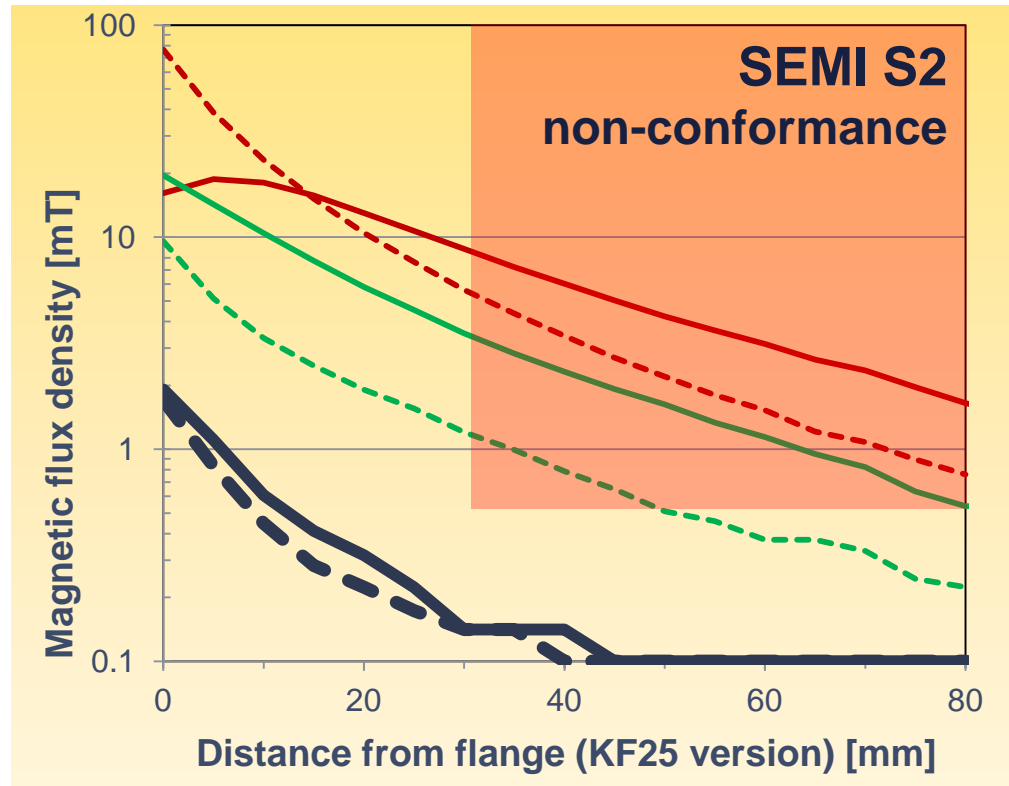




# Novel Magnet Design



# Low Magnetic Stray Field

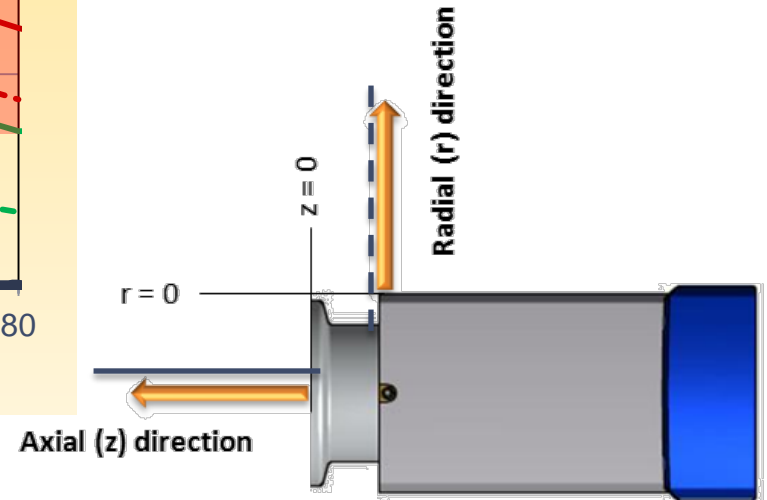


## Axial (z) direction

- Gemini MxG50x
- MPG40x
- state-of-art "low-field" design

## Radial (r) direction

- Gemini MxG50x
- - - MPG40x
- - - state-of-art "low-field" design









INFICON  
TCDG

# Vacuum Measurement

**direct**

$$p = \frac{F}{A}$$

**indirect**

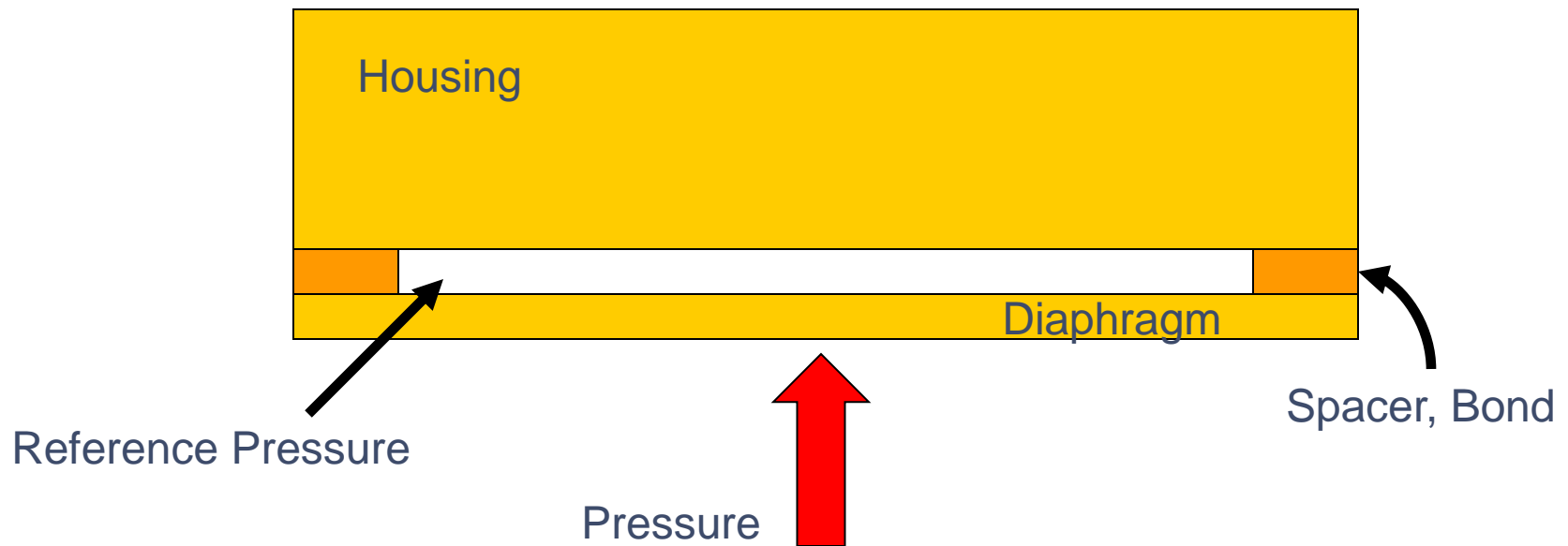
Ionisation

Viscosity

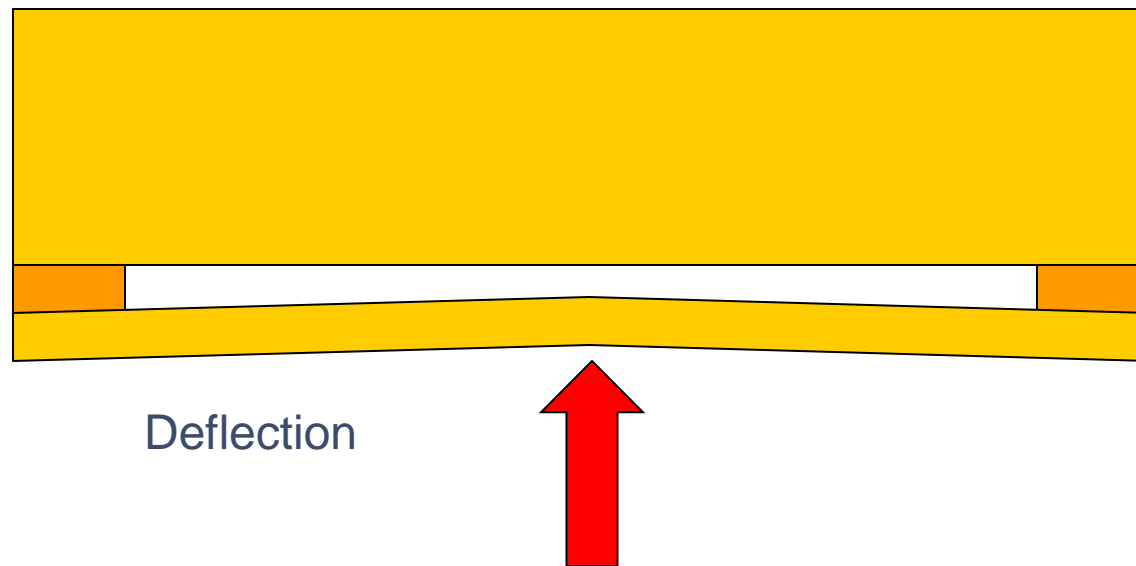
Convection

Thermal Conductivity

# Direct vacuum measurement (1)



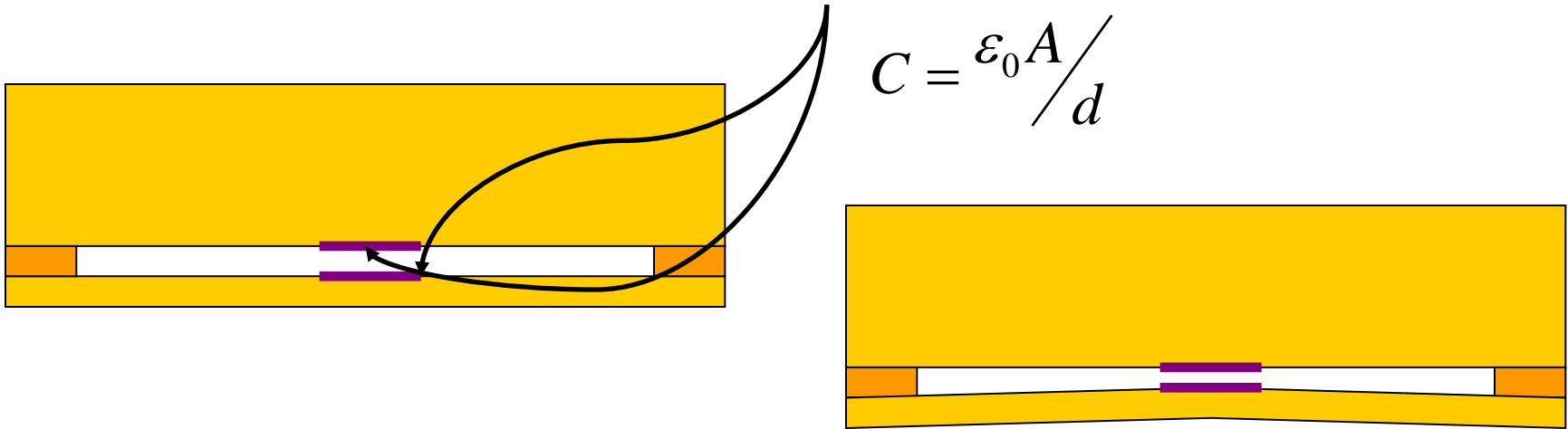
# Direct measurement of vacuum (2)



The deflection results from the net force of the pressure difference between the reference pressure (typical  $10^{-7}$  Torr) and the pressure around the cell (outside pressure - inside pressure).

# Direct measurement of vacuum

The capacitance  $C$  is given by the electrodes having a distance  $d$  and area  $A$



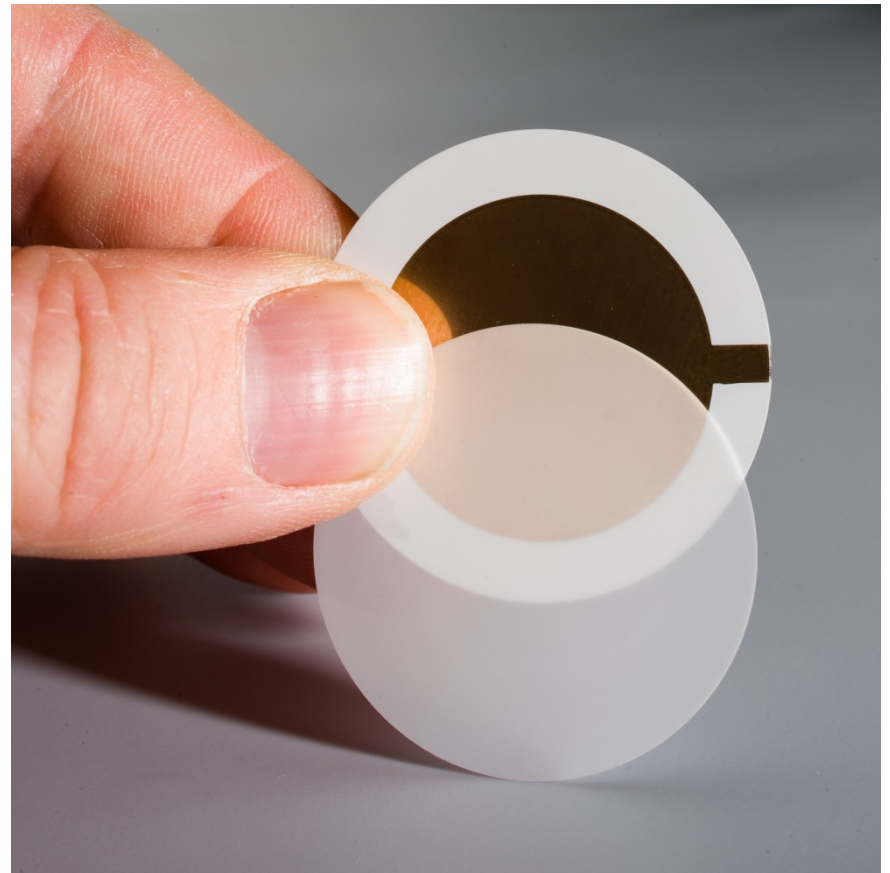
..and in case of deflection we see a change in capacitance, the distance  $d$  gets smaller:

$$\frac{\partial C}{\partial d} = -\frac{\epsilon_0 A}{d^2}$$

The electrical sensitivity of a CDG defined as the relative change of the capacitance depends strongly on the distance  $d$ !

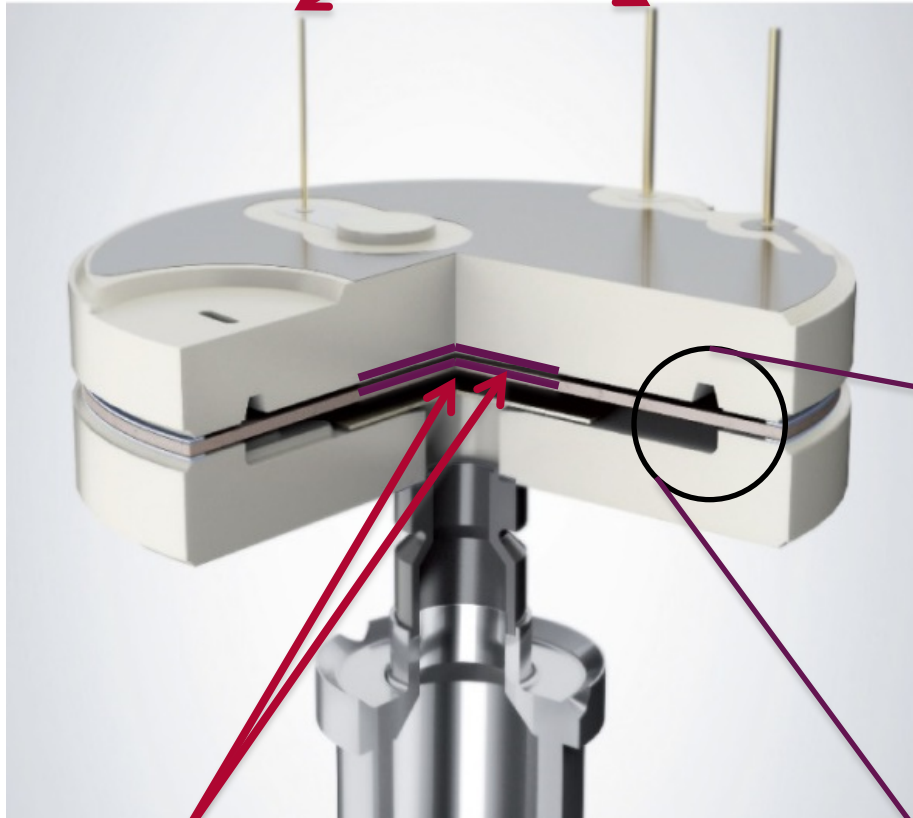
# Membrane

- Ultrapure  $\text{Al}_2\text{O}_3$  ceramic
- Polycrystalline
- High corrosion resistance
- Thicknesses down to  $30\mu\text{m}$
- Thinfilm gold electrode  
(reference cavity side)



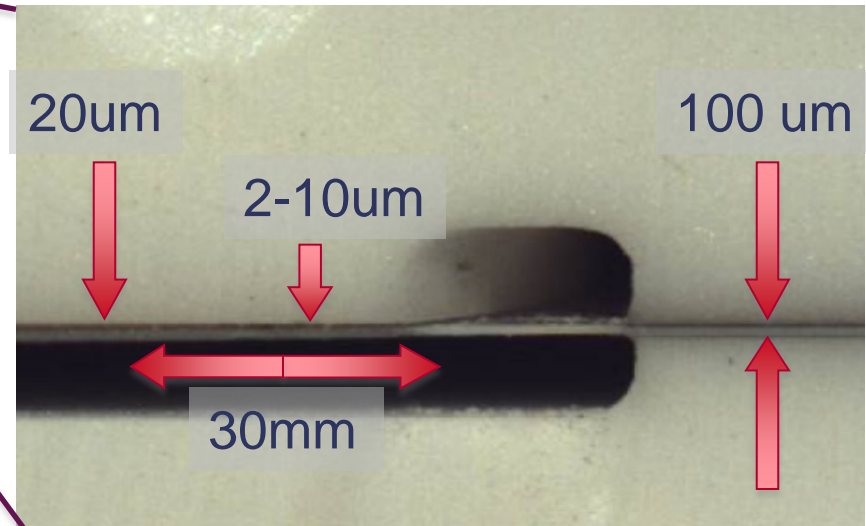
# CDG Cell

Capacitance



Electrodes

Ceramic CDG cell



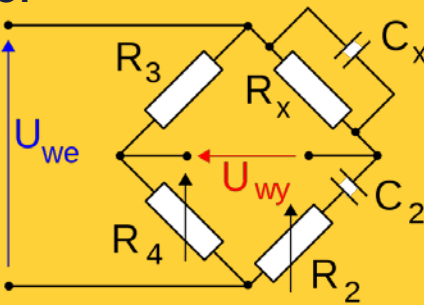
From  $\Delta C$   
to a readout

Change of capacity



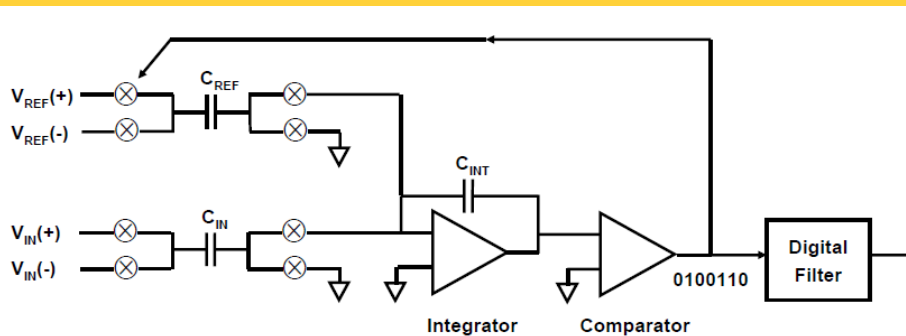
Change in  
pressure

### Oszillator



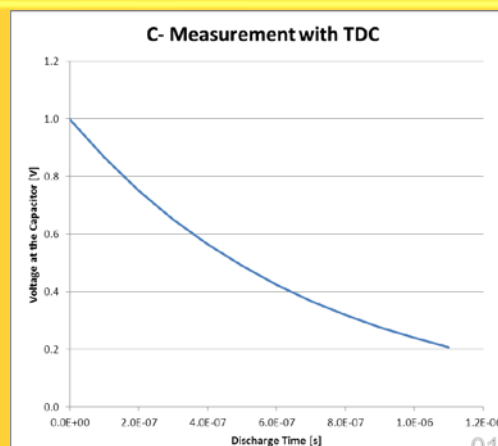
V

### Charge Based: Sigma Delta



V  
bit

### Time based: TDC



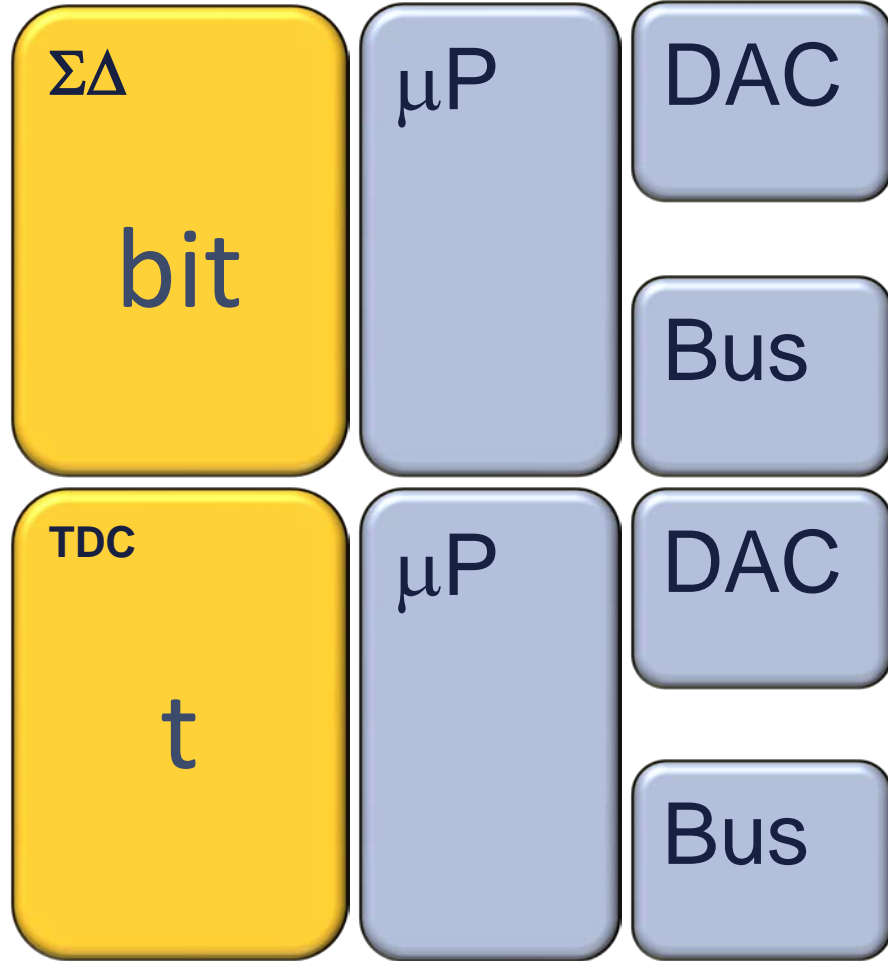
t



# From $\Delta C$ to a readout

Change of capacity

Change in pressure



# Capacitive Diaphragm Gauges

INCONEL  
analog



Late 90 s

$\text{Al}_2\text{O}_3$   
analog



2000

$\text{Al}_2\text{O}_3$   
digital



2006

1ms  
10mTorr  
 $\text{Al}_2\text{O}_3$   
digital



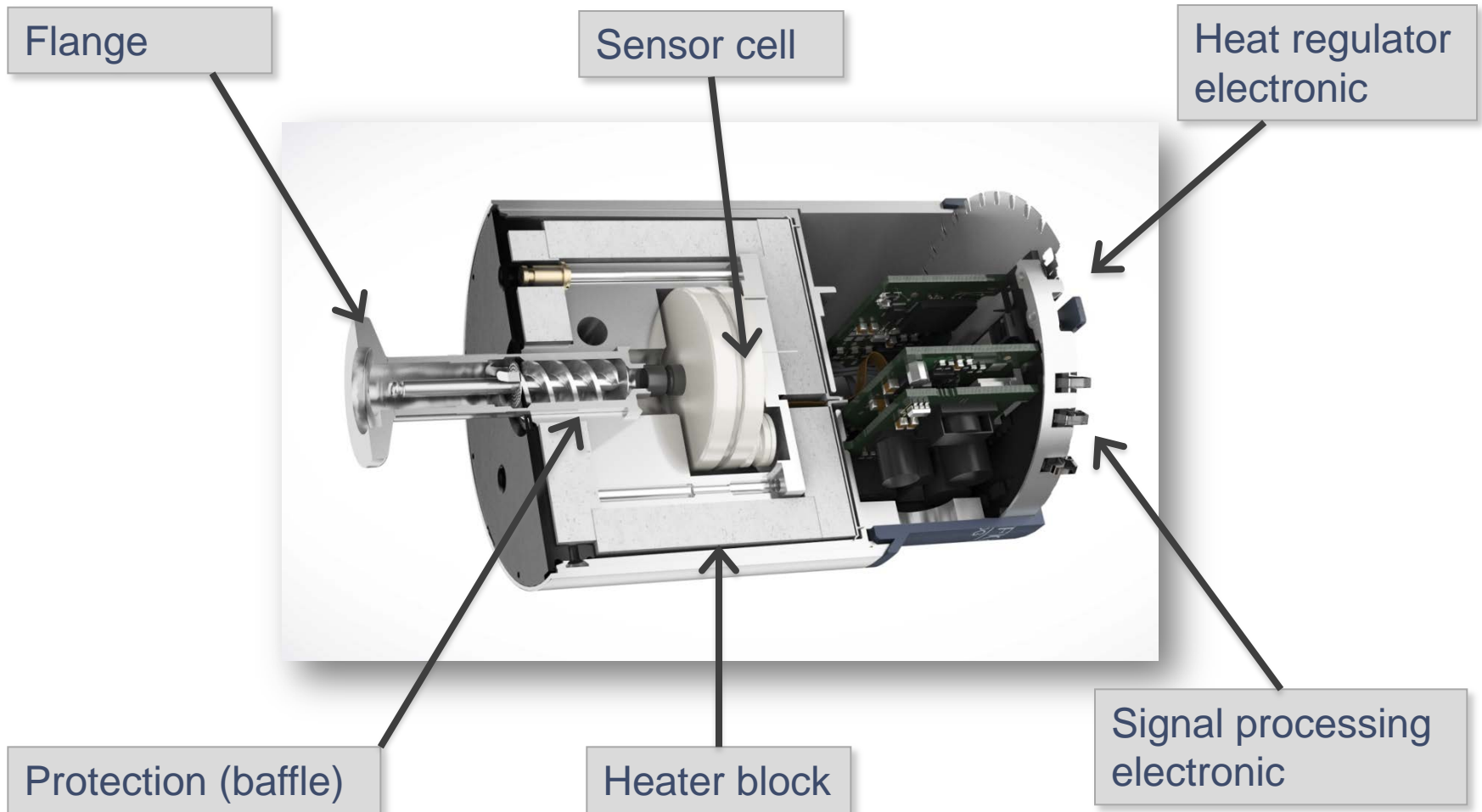
2014

# Innovative Portfolio



Spot Porter 025D X3 XXXD FIRRG Edge Stripe Kola-X Remote Cube

# Functional Blocks of a CDG



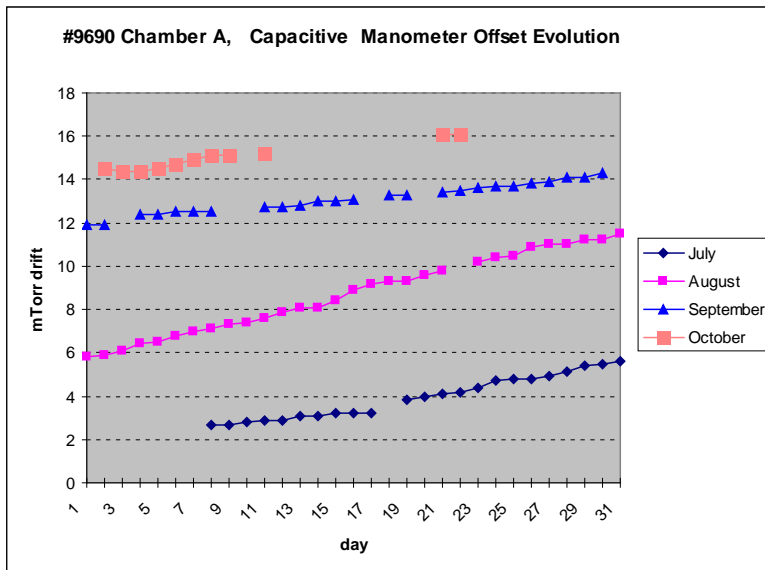
# Effect of Process Contamination on CDG's



Process byproducts that do not bond to the plasma shield (baffle) will deposit heavily around the center of the diaphragm

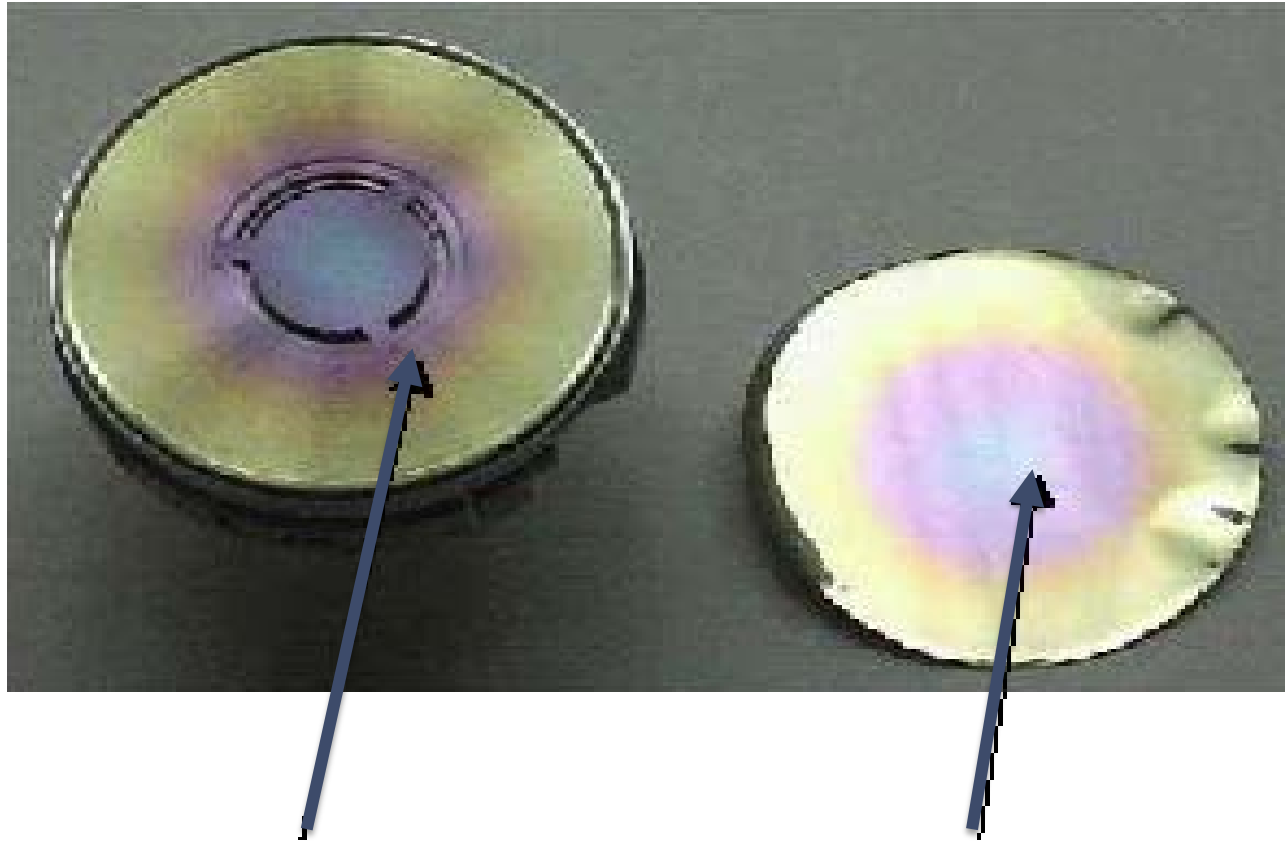
This results in uneven tensioning and weighting of the diaphragm causing changes which are typically seen as zero drift

As the deposition continues, the gauge will rapidly run out of zero adjustment and will need to be replaced.



Intentional Cu coating on CTR90, 10 Torr open sensor

# Process build-up on CDG's



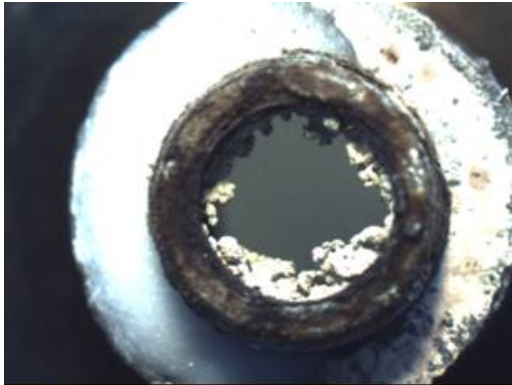
Heavy process build-up on plasma shield

Process build-up around the center of the metallic diaphragm

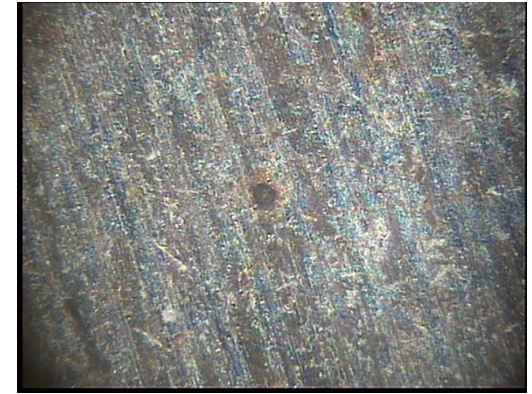
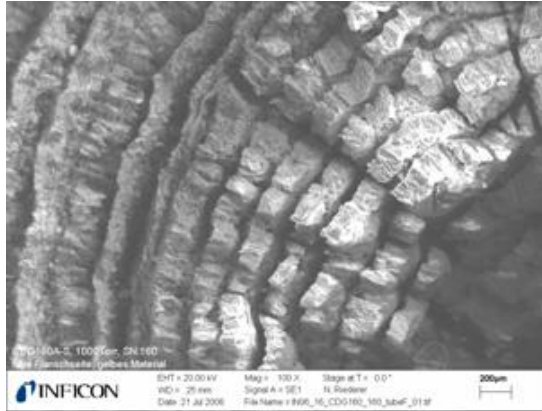
S.A. Tison, Transition Flow, Applications in Semiconductor Fabrication, Gas Dynamics Workshop, 30. June 2003, Avila, Spain.



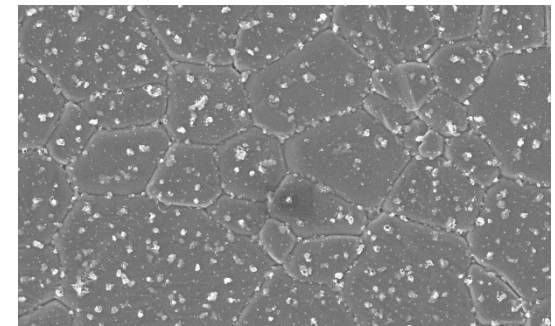
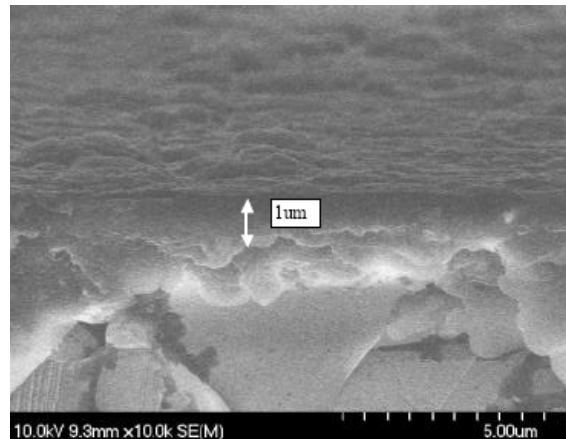
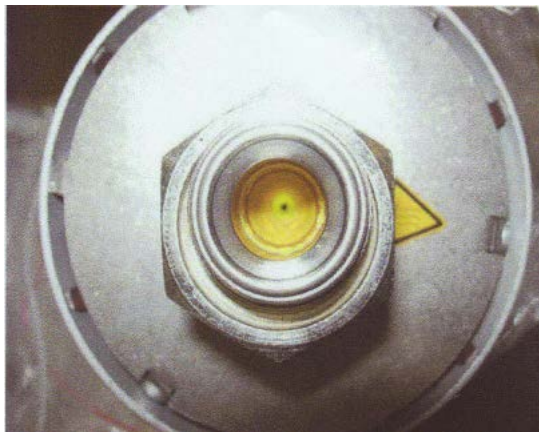
# Observed Contaminations



Vacon  
CR096 10T\_SN333  
Ti/TiN



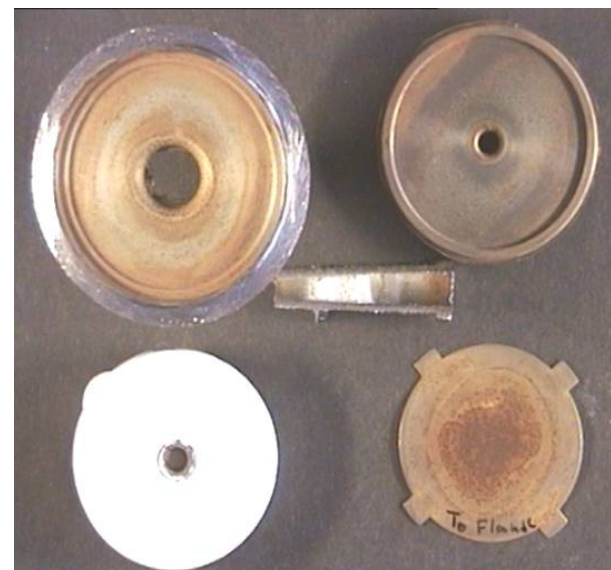
Plasma shield pitting corrosion  
CR095-S 1330Pa SN 013 or 105  
TiN



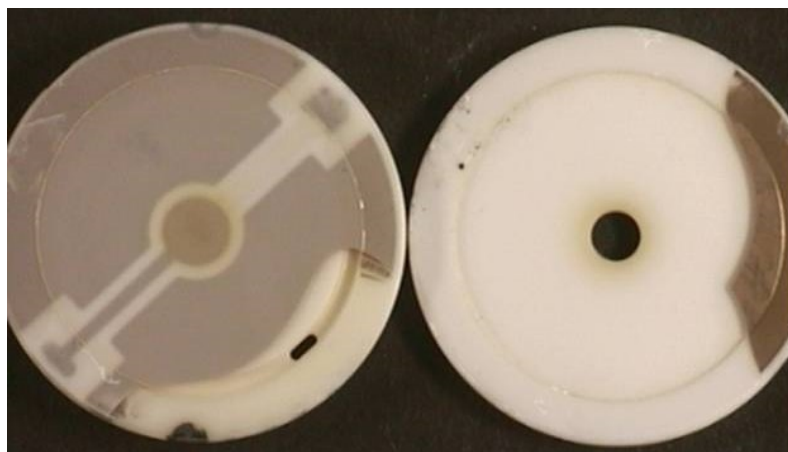
Diaphragm  
CR096-S\_133Pa SN104  
TiN

# CR096-S 10T SN225/SN333

Application	Ti/TiN	Ti/TiN
Product	CR096-S	CR096-S
Range	10 Torr	10 Torr
Serial number	225	333
Production / start warranty	31.5.2002 / ca.Okt.2002	12.11.2003 / 11.11.2003
Return	27.04.2006	27.04.2006
Precursors	TiCl <sub>4</sub> , NH <sub>3</sub> H <sub>2</sub> , N <sub>2</sub> , Ar ClF <sub>3</sub>	TiCl <sub>4</sub> , NH <sub>3</sub> H <sub>2</sub> , N <sub>2</sub> , Ar ClF <sub>3</sub>
Customer remarks	long time running	



SN 225



SN 333

# Design Improvements

**Gauge inherent**

**Process related**

# Issues

- A vacuum pressure gauge should accurately measure pressure under all circumstances.
- Processes, in particular coating or etching applications, affect the accuracy of the sensor in the long run by deposition of material or etching of sensor elements.
- Usually this causes sensor signal drift or in the worst case destroys the sensor.

# Goal

The goal of this investigation is

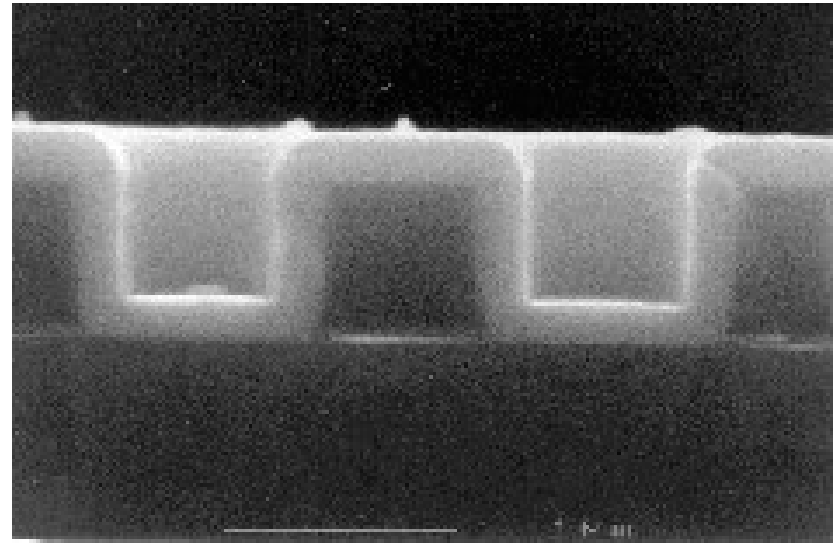
- to find methods to protect our gauges from process contamination in order
- to keep accuracy and
- to increase their life time on a process tool.

# TiN Uses in Semiconductor Industry

- Contact Liner - Ti / TiN

- Diffusion barrier material between substrate and metal interconnects
- Prevent the diffusion of metal ions into the semiconductor material
- Films in the order of few 100nm

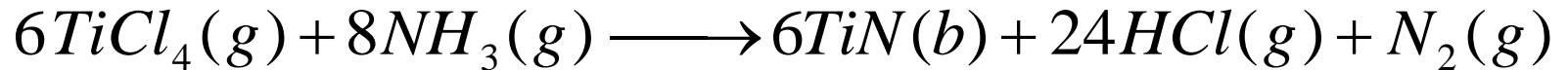
- Capacitor electrodes - TiN



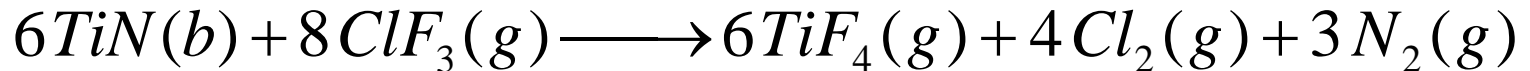


# TiN Process Basic Chemistry

Process:



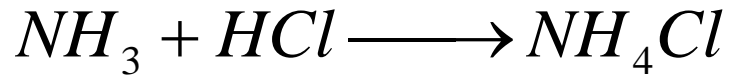
Dry clean: (ClF<sub>3</sub> + N<sub>2</sub> or Ar)



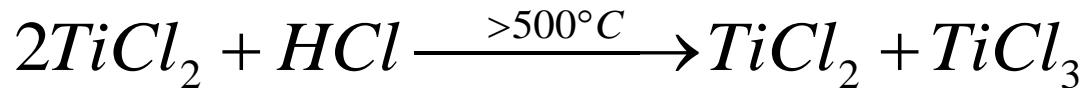
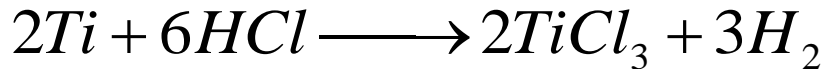
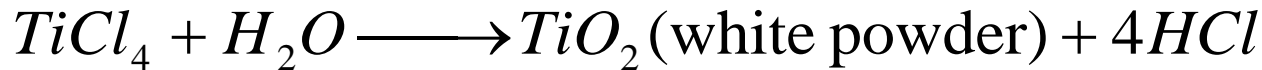
Wet clean:

*Isopropyl-Alcohol (C<sub>3</sub>H<sub>8</sub>O) + De-ionized water (H<sub>2</sub>O)*

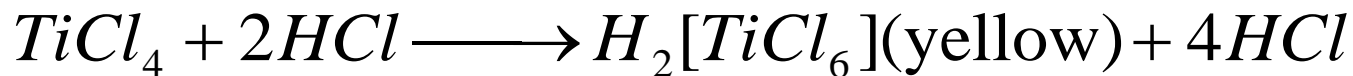
# By-product Reactions



= ammonium chloride  
is very reactive  
has a vapour pressure of 1Torr @160 C  
it sublimates and condensates at cold places



TiCl<sub>2</sub> = Very reductive  
May reduce water

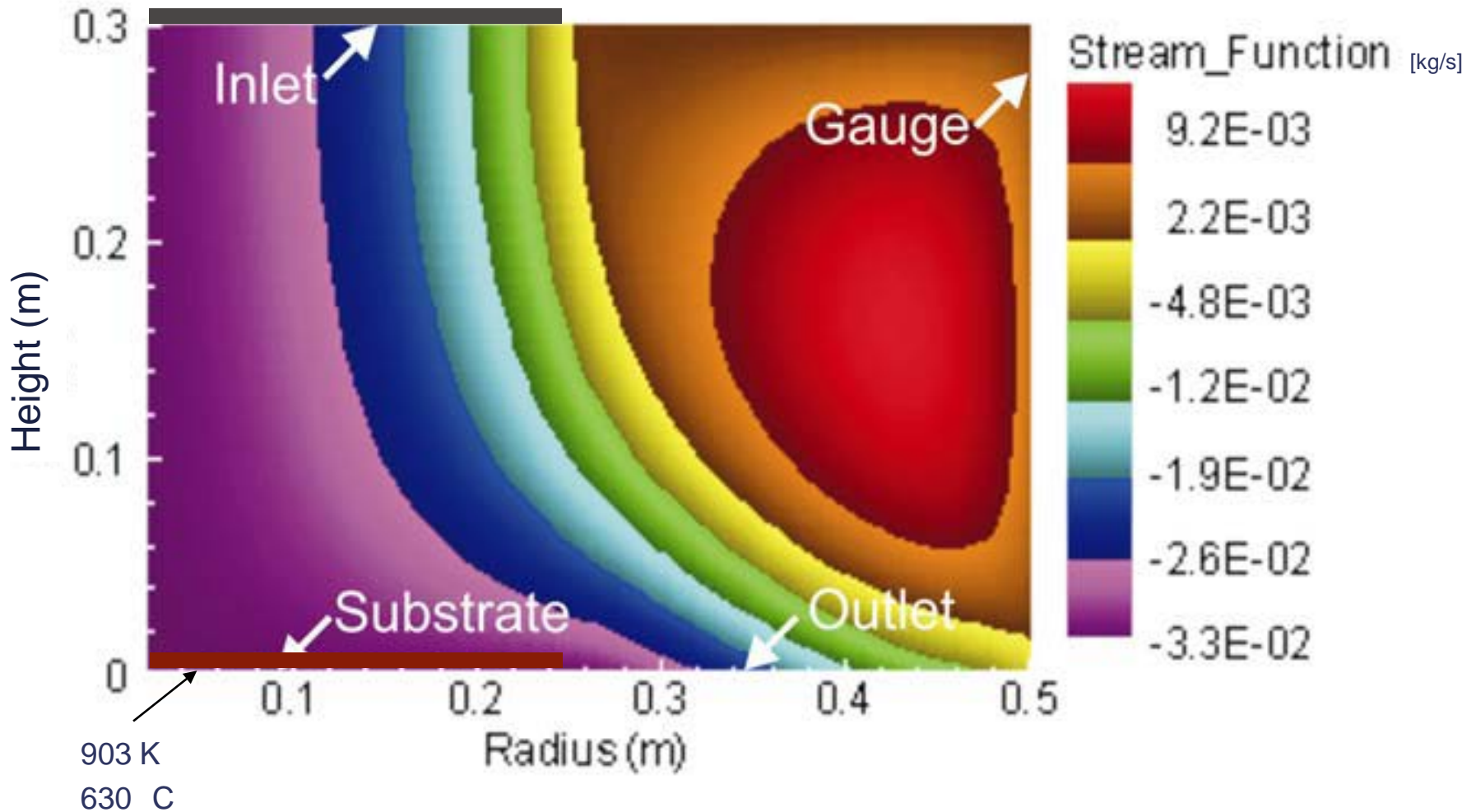


# Surface chemistry mechanism for TiN deposition

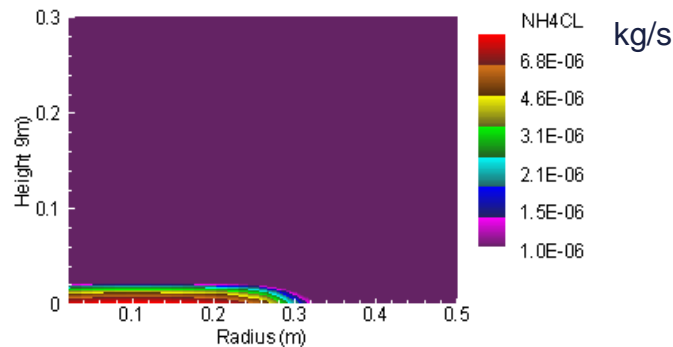
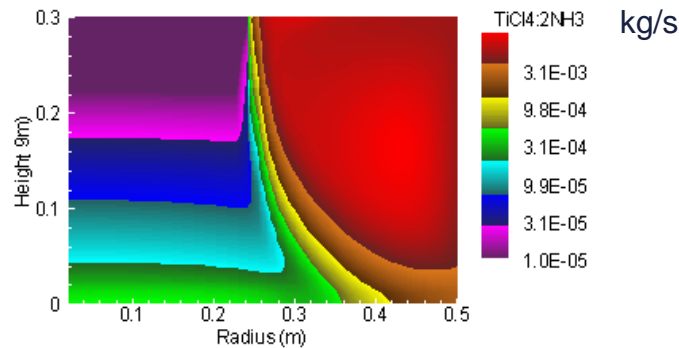
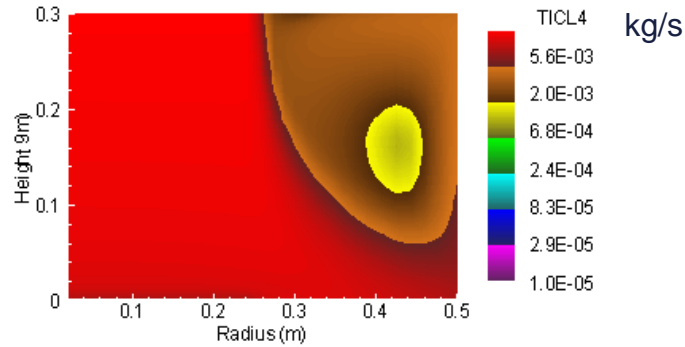
Gas:	TiCl <sub>4</sub> (g) NH <sub>3</sub> (g) HCl(g) N <sub>2</sub> (g)		
Surface:	TiCl <sub>3</sub> (s) TiCl <sub>2</sub> (s) TiCl(s) Ti(s) Ti*(s) NH <sub>2</sub> (s) NH(s) N(s) N*(s) N**(s)		
Bulk:	Ti(b) N(b)		
Titanium deposition:		A	E/R
1.	TiCl(g) + NH <sub>2</sub> (s) + Ti*(s) → TiCl <sub>3</sub> (s) + NH(s) + HCl(g) + Ti(b)	1.6 10 <sup>24</sup>	7500
2.	TiCl <sub>4</sub> (g) + NH(s) + Ti*(s) → TiCl <sub>3</sub> (s) + N(s) + HCl(g) + Ti(b)	1.6 10 <sup>24</sup>	7500
Nitrogen deposition:			
3.	TiCl <sub>3</sub> (s) + NH <sub>3</sub> (g) + N(s) → TiCl <sub>2</sub> (s) + NH <sub>2</sub> (s) + HCl(g) + N(b)	2.5 10 <sup>22</sup>	7000
4.	TiCl <sub>2</sub> (s) + NH <sub>3</sub> (g) + N(s) → TiCl(s) + NH <sub>2</sub> (s) + HCl(g) + N(b)	2.5 10 <sup>22</sup>	7500
5.	TiCl(s) + NH <sub>3</sub> (g) + N(s) → Ti(s) + NH <sub>2</sub> (s) + HCl(g) + N(b)	2.5 10 <sup>22</sup>	7500
Surface condensation:			
6.	TiCl <sub>3</sub> (s) + NH <sub>2</sub> (s) → TiCl <sub>2</sub> (s) + NH(s) + HCl(g)	4 10 <sup>13</sup>	7500
7.	TiCl <sub>3</sub> (s) + NH(s) → TiCl <sub>2</sub> (s) + N(s) + HCl(g)	4 10 <sup>13</sup>	7500
8.	TiCl <sub>2</sub> (s) + NH <sub>2</sub> (s) → TiCl(s) + NH(s) + HCl(g)	4 10 <sup>13</sup>	7500
9.	TiCl <sub>2</sub> (s) + NH(s) → TiCl(s) + N(s) + HCl(g)	4 10 <sup>13</sup>	7500
10.	TiCl(s) + NH <sub>2</sub> (s) → Ti(s) + NH(s) + HCl(g)	4 10 <sup>13</sup>	7500
11.	TiCl(s) + NH(s) → Ti(s) + N(s) + HCl(g)	4 10 <sup>13</sup>	7500
Bond breaking:			
12.	Ti(s) + N(s) → Ti*(s) + N*(s)	11 10 <sup>14</sup>	7500
13.	Ti(s)+N*(s) → Ti*(s) + N**(s)	11 10 <sup>14</sup>	7500
N <sub>2</sub> liberation:			
14.	2Ti(s) + 2N**(s)+2N(b) → 2Ti*(s) + N <sub>2</sub> (g) + 2N(s)	9.3 10 <sup>30</sup>	7500

M. D. Allendorf, A. Arsenlis, R. Bastasz, M. E. Colvin, G. Evans, G. Germann, C. L.. Janssen, R. S. Larson, C. F. Melius, T. H. Osterheld, D. A. Outka, M. L. Schulberg, P. Ho, and I. M. B. Nielsen, *Development of a process simulation capability for the formation of titanium nitride diffusion barriers*, Sandia National Laboratories, 1997, p. 40.

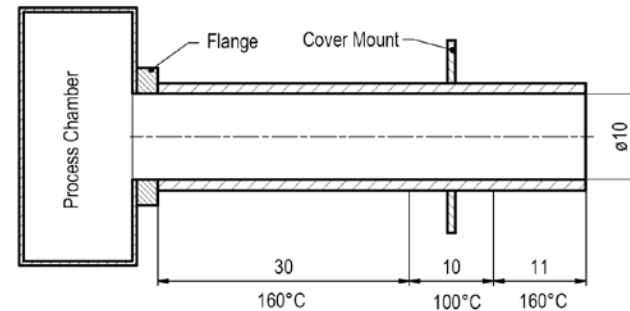
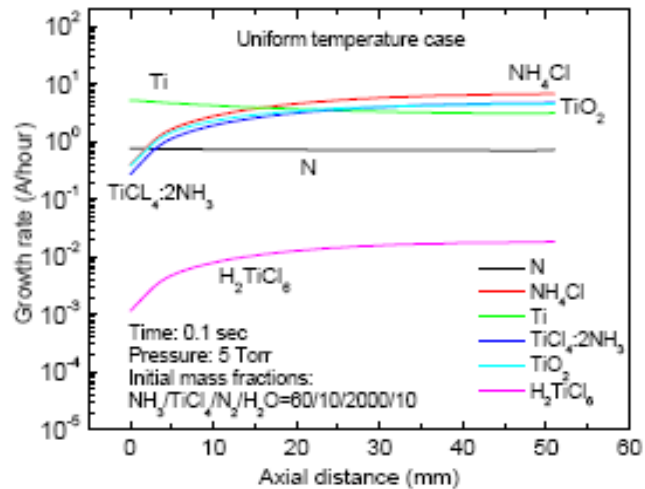
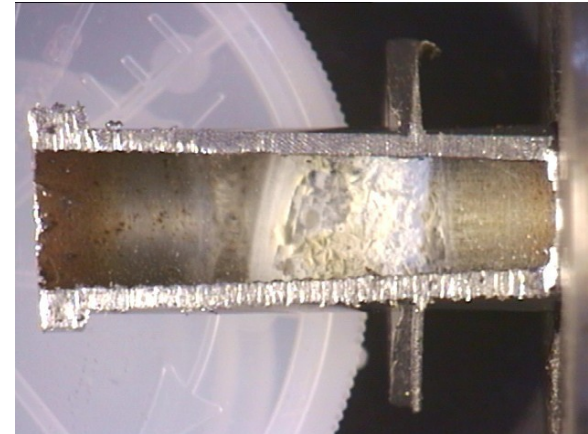
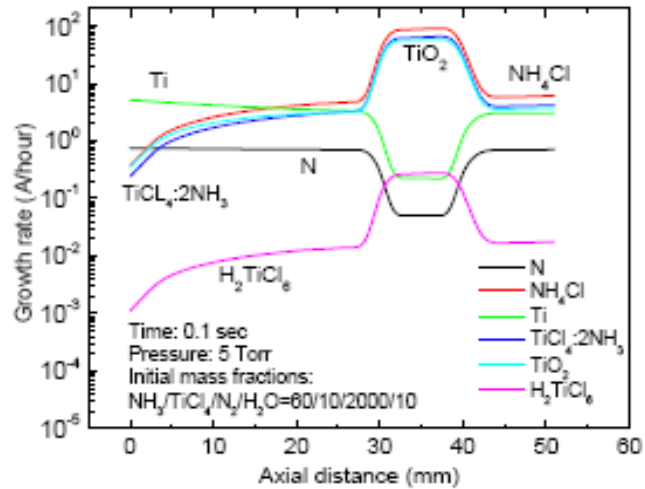
# Stream Function CVD Reactor



# Profiles of Mass Fractions



# Validation Results

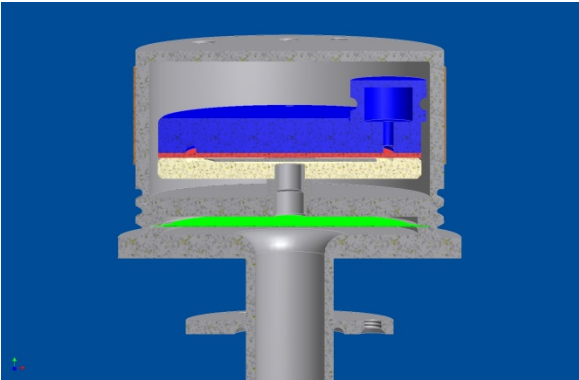




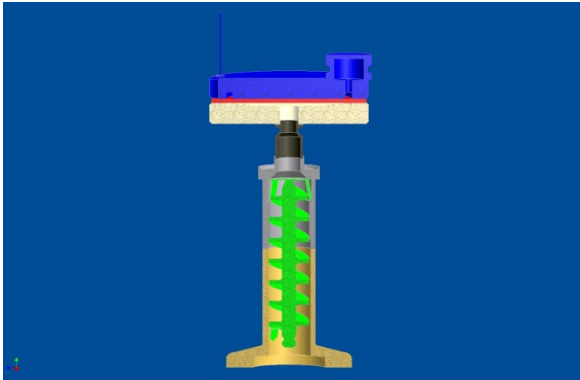
# Validation Results

- The surface and gas-phase mechanisms for the TiN chemical vapor deposition process were developed based on literature data.
- Simulations were performed for a typical geometry and operating conditions of a CVD reactor and also for the transport and chemistry inside a gauge.
- The performed simulations indicate that powder deposition rate could be similar or even exceed the TiN deposition rate inside the gauge.
- Cold spot of 60 °C in gauge tube causes deposition of by-products.
  - Can reproduce deposition profile
- TiN etching by  $\text{ClF}_3$  is not efficient since the products of etching quickly fill in the gauge and the etching stops. Cleaning is efficient only close to reactor wall.

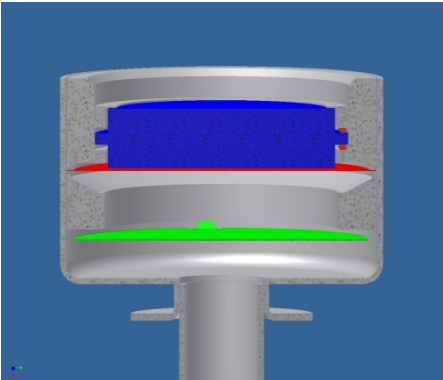
# Gauge Designs



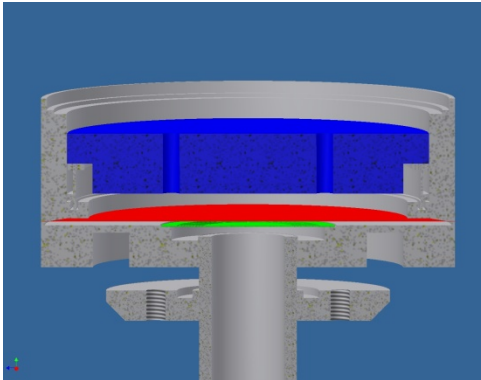
INFICON CDG160A  
Plasmashield  
Suprashield  
Crossshield



INFICON CDG160D  
Helix  
Crossshield



Competitor A

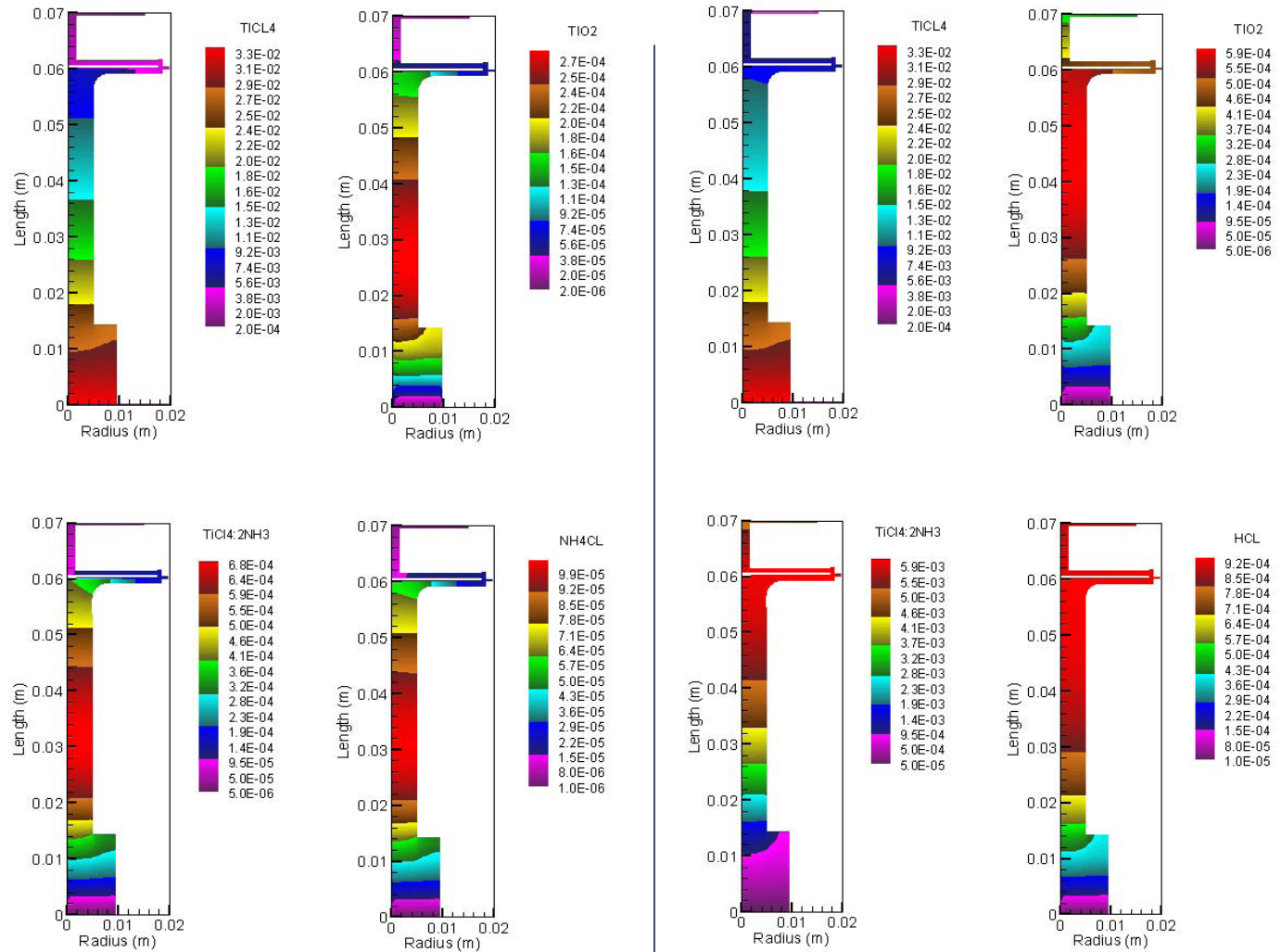


Competitor B

# TiCl<sub>4</sub> + NH<sub>3</sub> Process in a CDG 160A with Plasma Shield

## Spatial Mass Fraction Profiles

- Gas pressure 5 Torr
- Wall temperature 160 C
- Gauge filled with N<sub>2</sub> at start

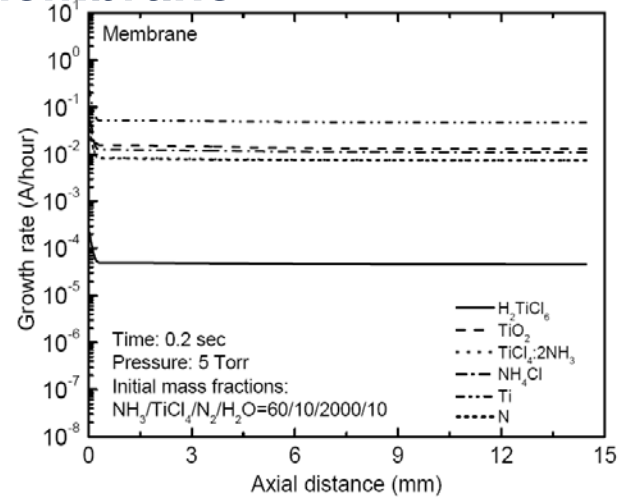
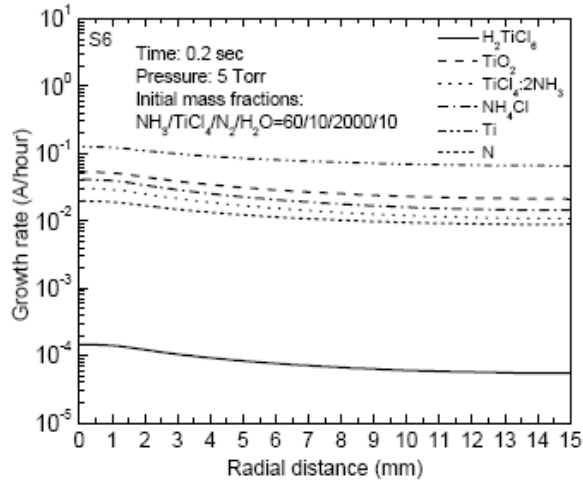


0.2 sec

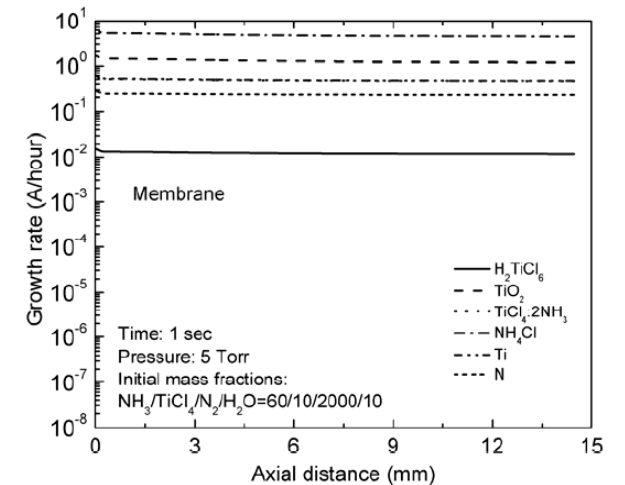
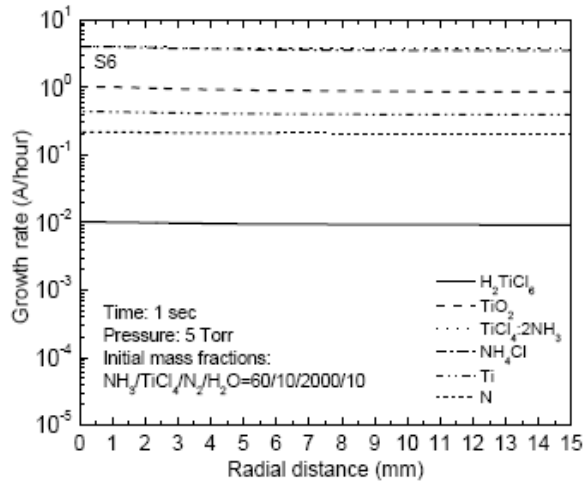
1 sec

# Comparison Deposition Profile along Membrane

0.2 s



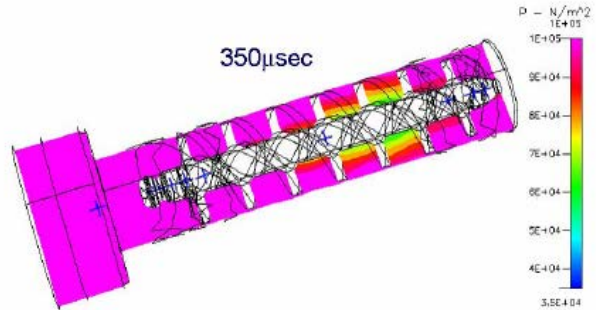
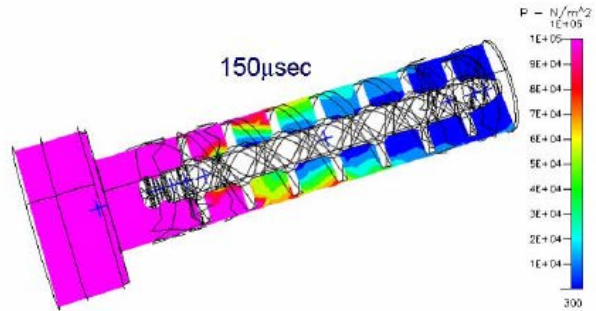
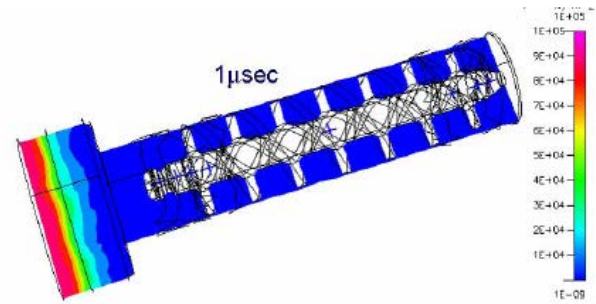
1 s



Plasmashield

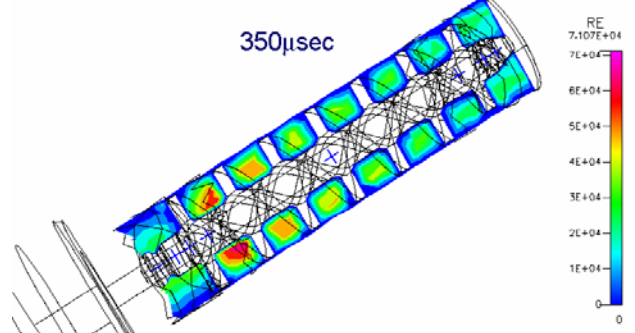
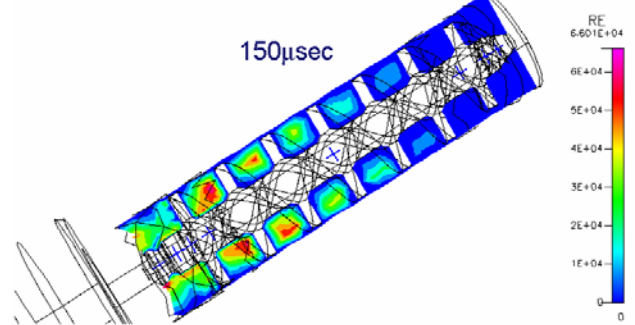
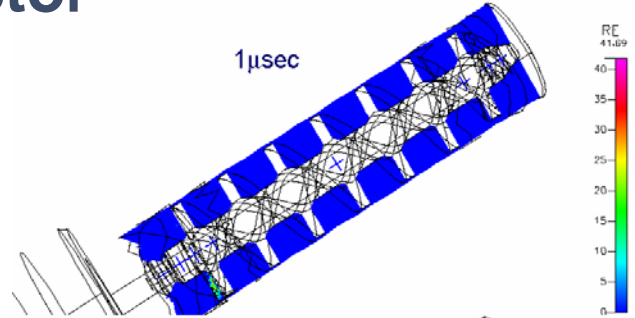
Helix

# 3D Pressure Profile During Pressure Change $10^{-6}$ mbar to 1000 mbar in the Reactor



Pressure

1 ms to reach equilibrium at end of helix

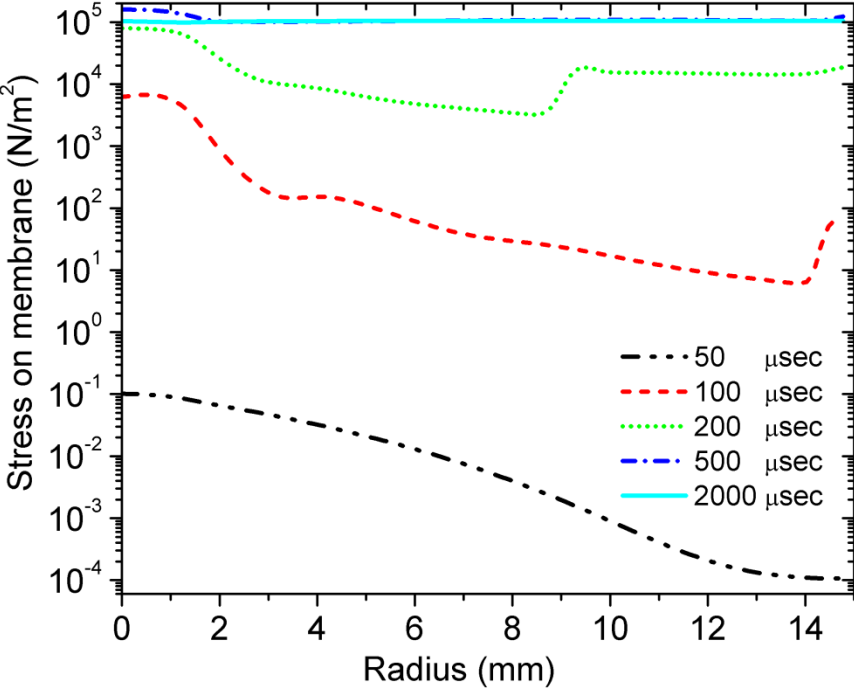


Re

Re < 2300 laminar  
 Re > 4000 turbulent

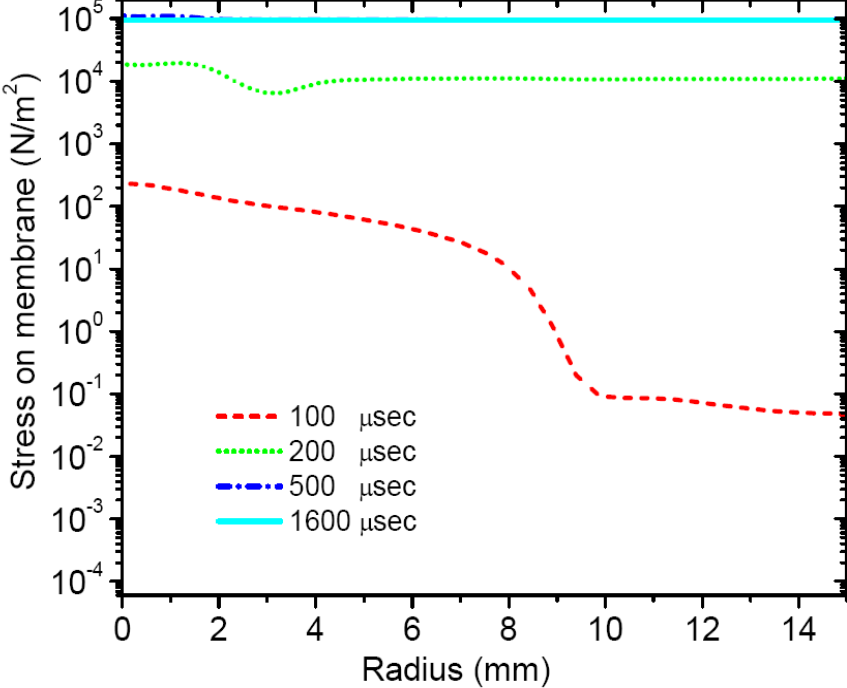
# Stress on Membrane as Function of Time

## Plasmashield



2 ms to reach equilibrium

## Helix

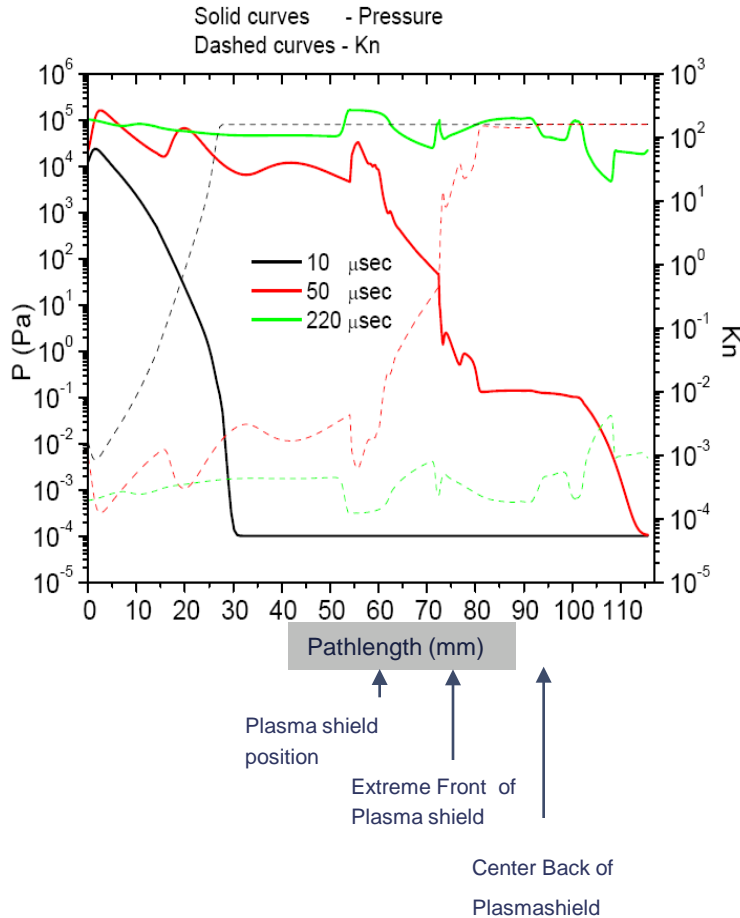


1.6 ms to reach equilibrium

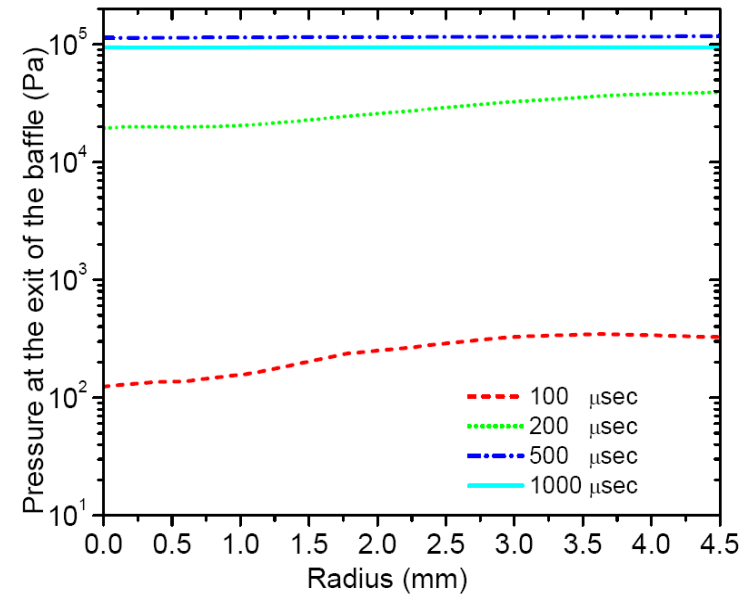


# Pressure/Knudsen Number (x, t)

## Plasmashield



## Helix



## At Exit of Helix

3D Sim

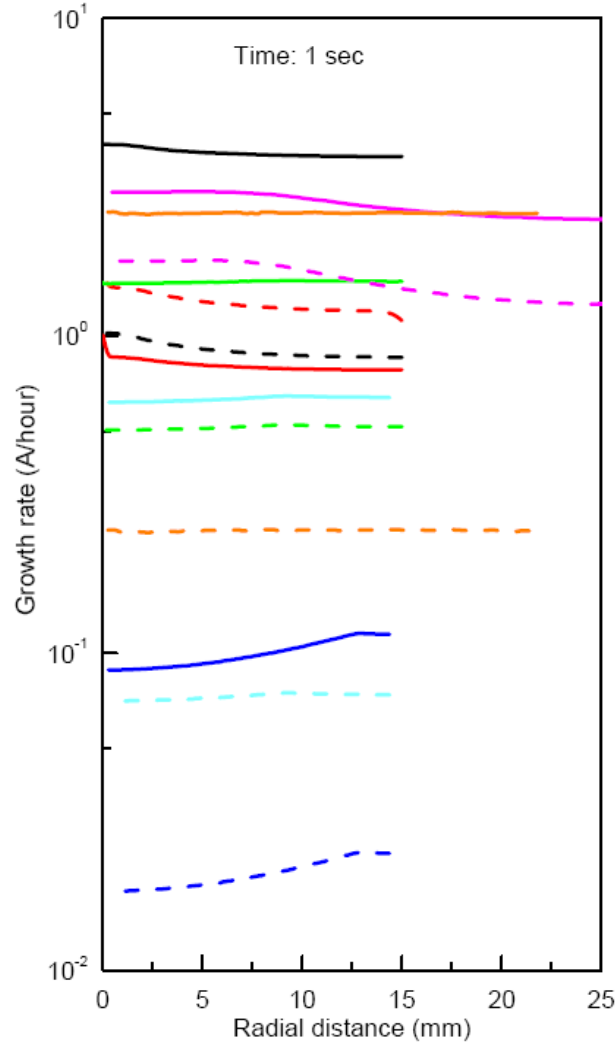
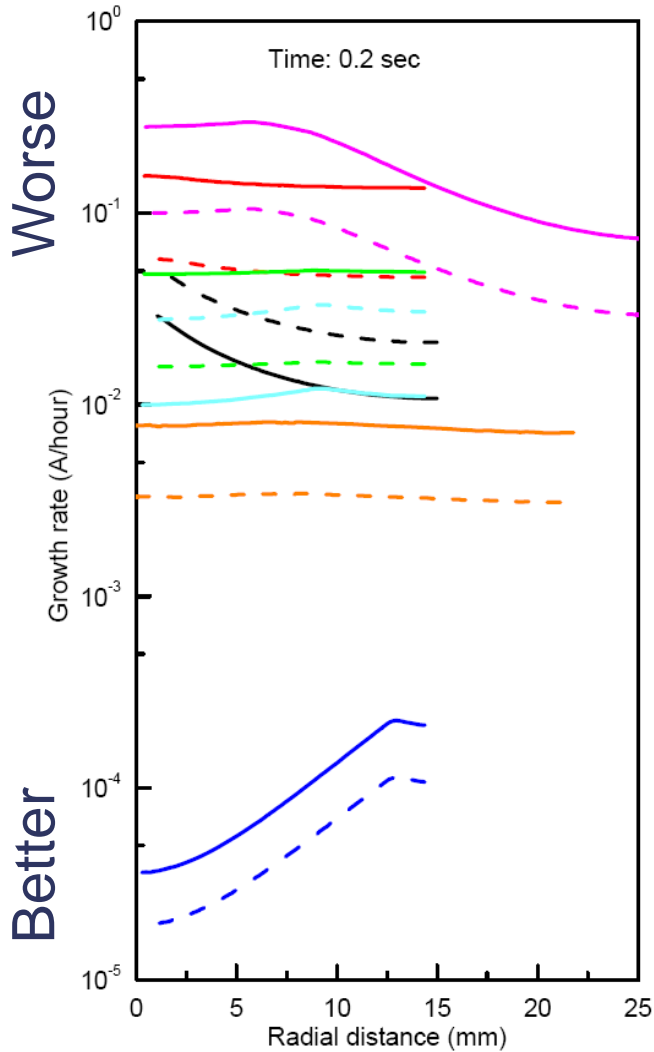
# Summary Dynamics

Response to step pressure change  $10^{-6}$  mbar to ATM

Ranking	Name of the gauge	Time ( $\mu$ s)
1	<b>CDG160D with spiral baffle system</b>	<b>1600 (CFD-ACE)</b>
2	<b>CDG160A gauge with plasma shield</b>	<b>2000 (CFD-ACE) 2000 (Fastran) 2000 (UFS)</b>
3	<b>CDG160D gauge with a spiral baffling system and cross shield</b>	<b>2200 (CFD-ACE)</b>
4	<b>Competitor B gauge</b>	<b>3000 (CFD-ACE)</b>
5	<b>CDG160A gauge with plasma and supra shields</b>	<b>6000 (CFD-ACE) 20000 (UFS)</b>
5	<b>Competitor A gauge</b>	<b>6000 (CFD-ACE)</b>

Significant difference between the results obtained with CFD-ACE and UFS for a CDG160A gauge with plasma and supra shields was attributed to different gas-surface models implemented in the codes.

# Membrane Contamination



- (G1) -  $\text{TiCl}_4 \cdot 2\text{NH}_3$  CDG160A+PS
- - (G1) -  $\text{TiO}_2$
- (G2) -  $\text{TiCl}_4 \cdot 2\text{NH}_3$  CDG160D + H
- - (G2) -  $\text{TiO}_2$
- (G3) -  $\text{TiCl}_4 \cdot 2\text{NH}_3$  CDG160D + H + CS
- - (G3) -  $\text{TiO}_2$
- (G4) -  $\text{TiCl}_4 \cdot 2\text{NH}_3$  CDG160A+PS+SS
- - (G4) -  $\text{TiO}_2$
- (G5) -  $\text{TiCl}_4 \cdot 2\text{NH}_3$  Competitor B
- - (G5) -  $\text{TiO}_2$
- (G6) -  $\text{TiCl}_4 \cdot 2\text{NH}_3$  Competitor A
- - (G6) -  $\text{TiO}_2$
- (G7) -  $\text{TiCl}_4 \cdot 2\text{NH}_3$  CDG160A+PS+CS
- - (G7) -  $\text{TiO}_2$

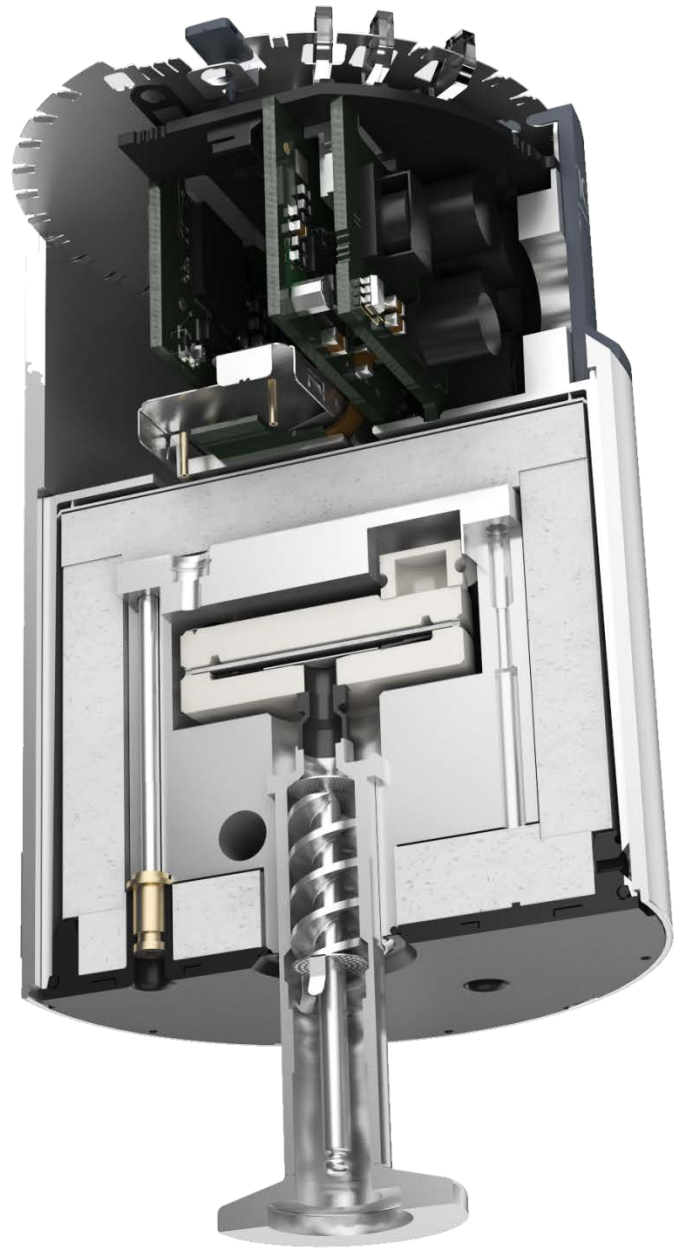
Pressure: 5 Torr

Initial gas:  $\text{N}_2$

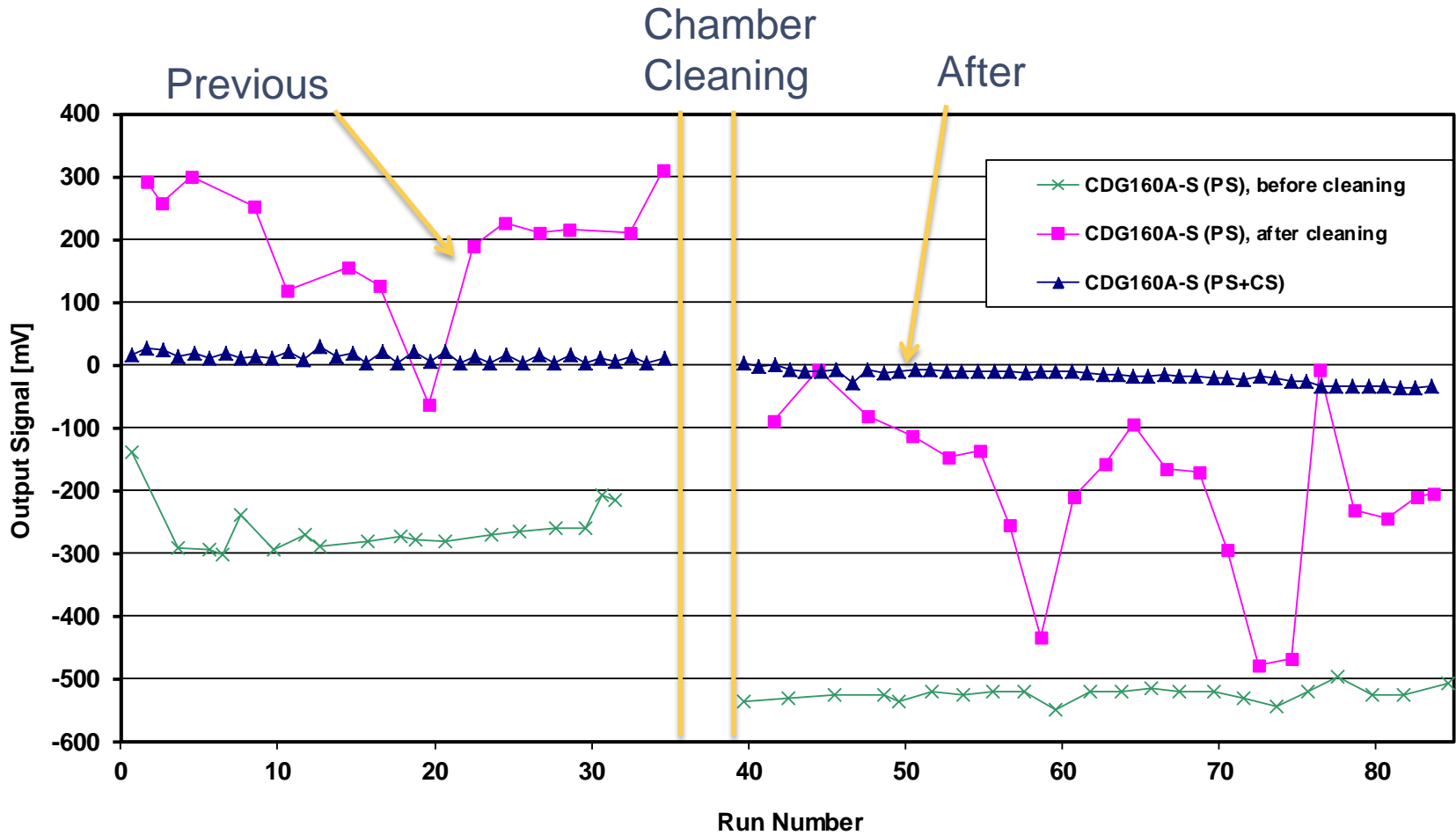
Mass fractions at reactor/gauge orifice:  
 $\text{NH}_3/\text{TiCl}_4/\text{N}_2/\text{H}_2\text{O} =$   
 60/10/2000/10

# Best Membrane Protection

1. CDG160A with Plasmashield + Suprashield (G4 geometry)  
has the smallest powder deposition rates at both 0.2 s and 1 s.
2. CDG160A with Plasmashield + Cross shield (G7 geometry)
3. CDG025D + Helix (G2)
4. CDG025D + Helix + Cross shield (G3)
5. Competitor A (G6)
6. Competitor B (G5)  
largest at 0.2 s, but they are smaller than G1 at 1 s.
7. CDG160A + Plasmashield (G1)



# Process Drift Improvement

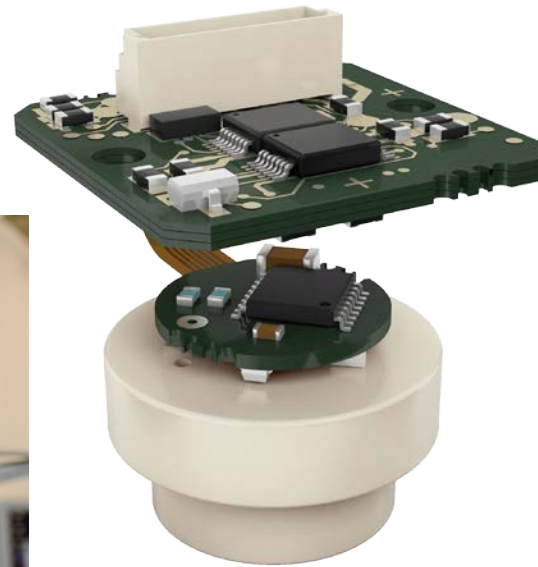


# Summary of Simulation

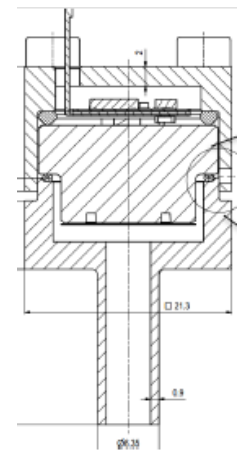
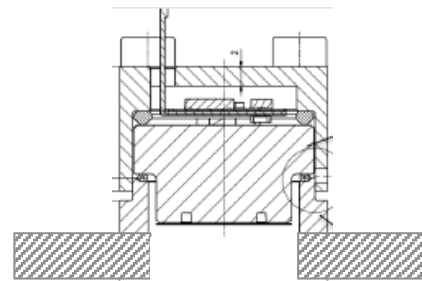
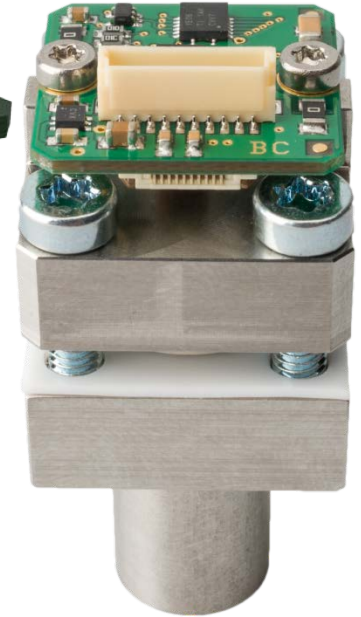
- Using a CFD computer code we have investigated membrane contamination and gas dynamical response time in a TiN process for 7 different geometries.
- Position of gauge in process has great influence on chemistry seen by gauge.
- Baffle geometry and gas dynamics in a gauge have a strong effect on the contamination of the membrane and the speed of response.
- INFICON has best in class gauge protection available.



# Small Scale CDGs

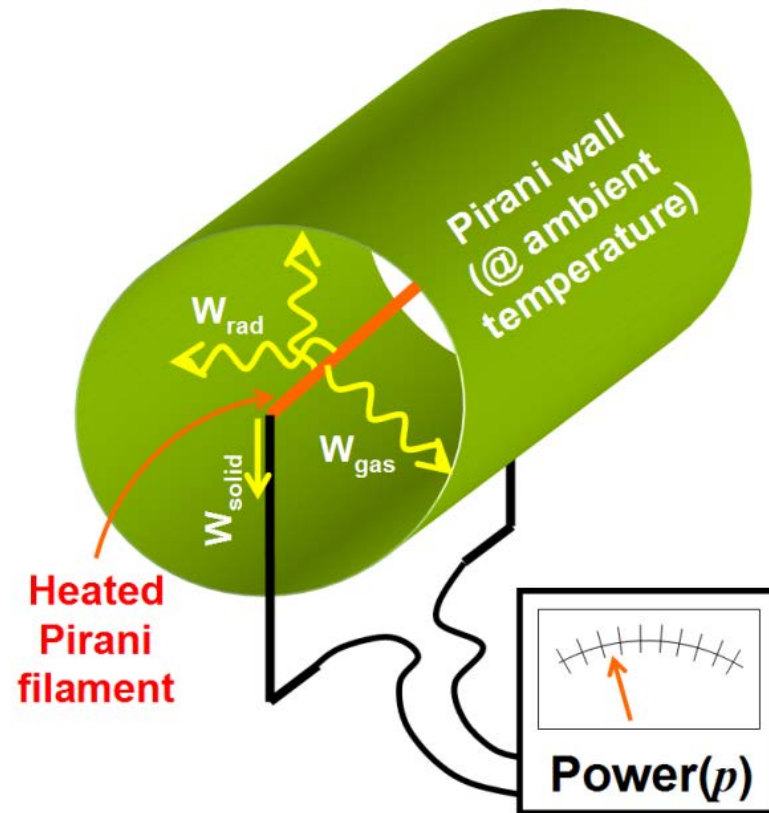


INFICON Spot CDS550D





# Pirani



# Thermal Conductivity Sensor

## a) Heat loss through radiation

$$W_{gas,v} \propto f(\epsilon_p, \epsilon_w) \times (T_p^4 - T_w^4)$$

## b) Heat conduction from filament to support

$$W_{solid} \propto \left. \frac{\partial T_p}{\partial x} \right|_{@ends}$$

## c) Heat conduction through gas

- Molecular flow ( $Kn > 0.5$ )

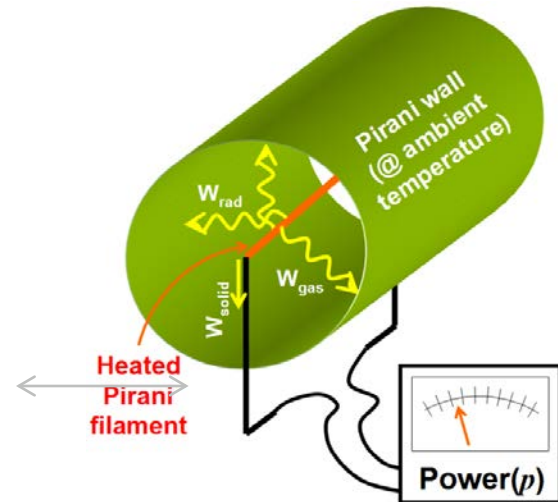
$$W_{gas,m} \propto \frac{a_E}{\sqrt{m}} \frac{(T_p - T_w)}{\sqrt{T_w}} \times p$$

- Viscous flow ( $Kn < 0.01$ )

$$W_{gas,v} \propto \frac{(T_p - T_w)}{\sqrt{m}} \times f(d)$$

- Transition flow ( $0.01 < Kn < 0.5$ )

## d) Heat transport through convection ( $Kn \ll 0.01$ )



# Pirani Sensor Elements



1970

TPR010

$10^{-3}$  to 100 mbar



1995

TTR211/216

$5 \cdot 10^{-4}$  mbar to ATM



1998

PSG400

$5 \cdot 10^{-4}$  mbar to ATM



2004

PSG500

$5 \cdot 10^{-5}$  mbar to ATM



2010

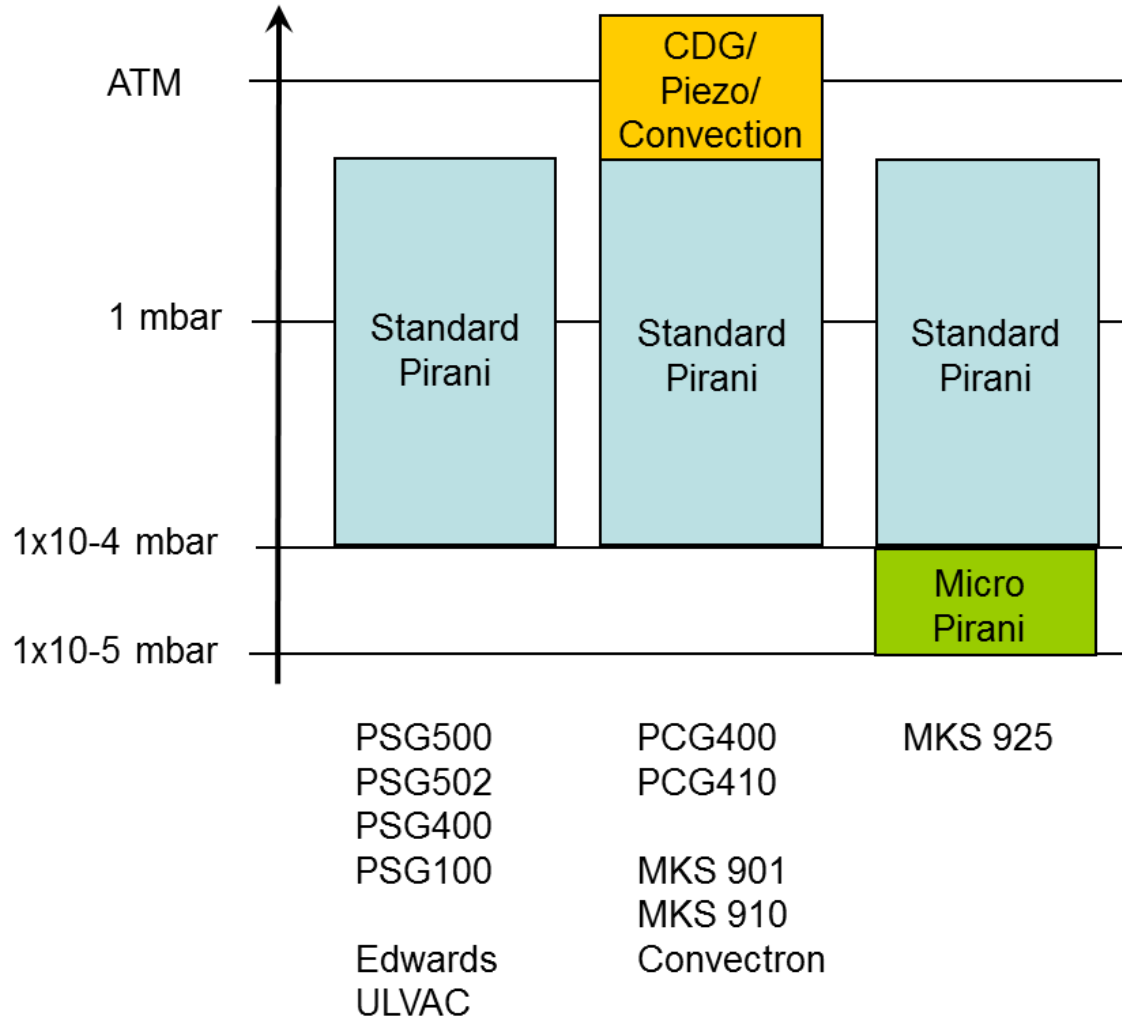
PSG550

$5 \cdot 10^{-5}$  mbar to ATM

# Issues

- Increase upper pressure limit
- Increase lower pressure limit
- Increase accuracy
- Increase process inertness
- Increase dynamics
- Decrease size

# Range Extensions





# Increasing the Upper Pressure Limit:

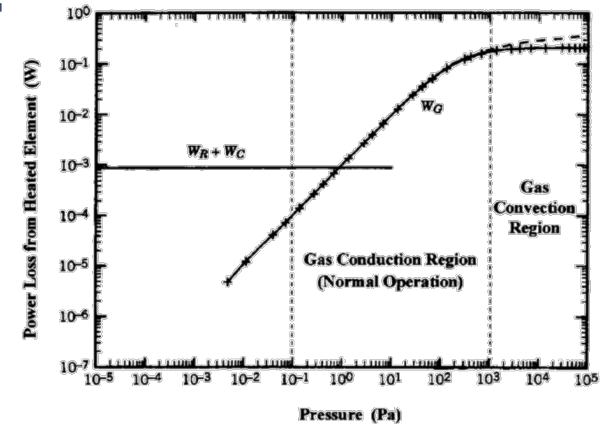
## 1. Use of convective transport

Convection enhanced gauge

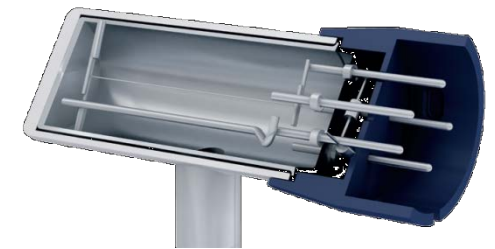
- ✓ Increased sensitivity at atmospheric pressure
- ✗ Orientation dependence
- ✗ Large volume needed
- ✗ Slow dynamics

Alternative designs, e.g. Edwards APGX

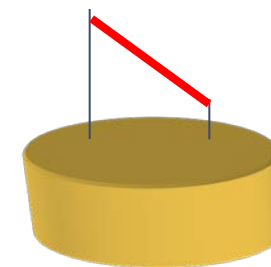
- ✓ Reduced orientation dependence
- Smaller but still substantial volume
- ✗ Slow dynamics



Ellefson and Miiller, JVSTA, 18, 2568-2577, 2000,



INFICON PGE050



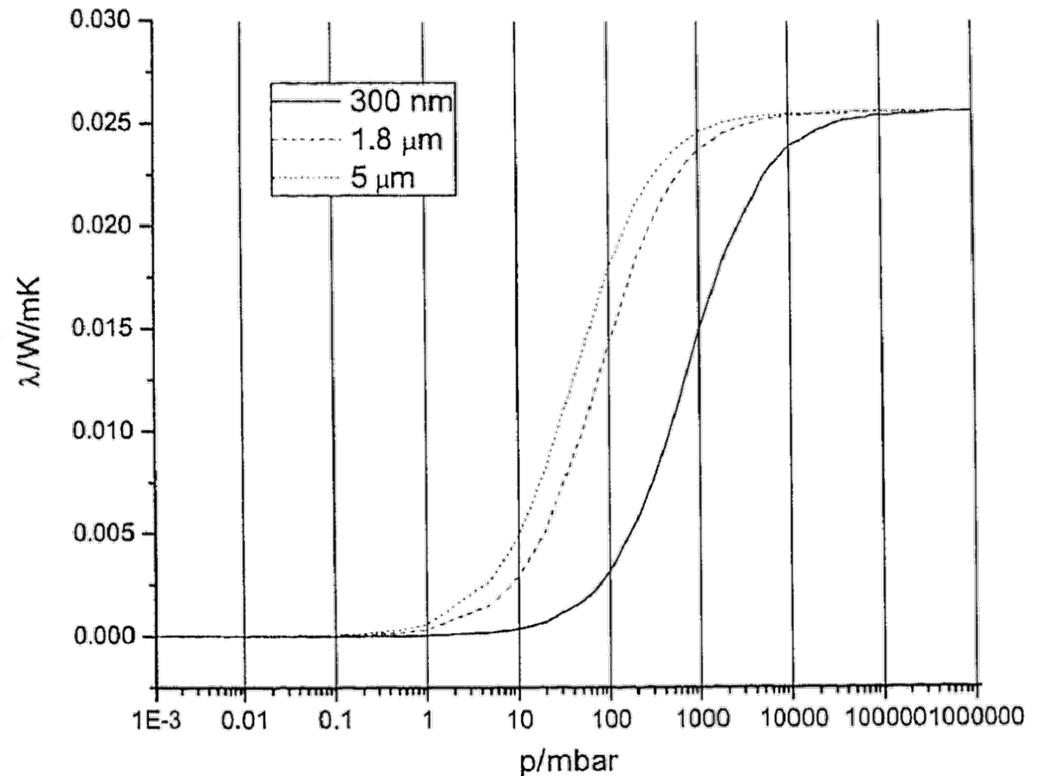
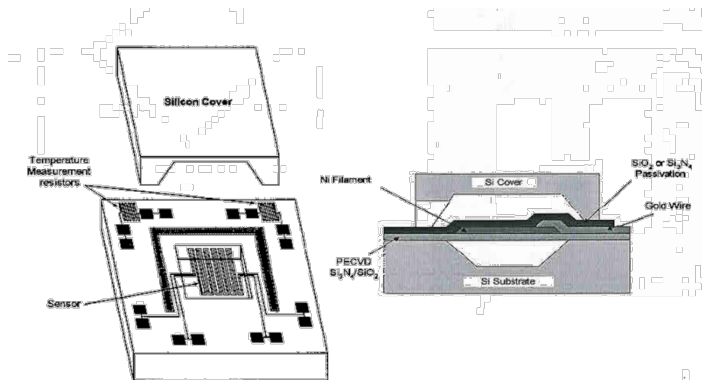
Pirani filament at tilted angle

# Increasing the Upper Pressure Limit:

## 2. Reduce mean free path

Reduce distance Pirani – Wall → MEMS

- ✓ Increased sensitivity at atmospheric pressure
- ✓ Good dynamics
- ✗ Sensitive to contamination



Doms et al., J. Micromech. Microeng., **15**, 1504-1510, 2005

# Increasing the Upper Pressure Limit:

## 3. Measurement of gas' heat capacity

Use time variable Pirani temperature  $T_p(t)$

### a) Pulsed heating mode

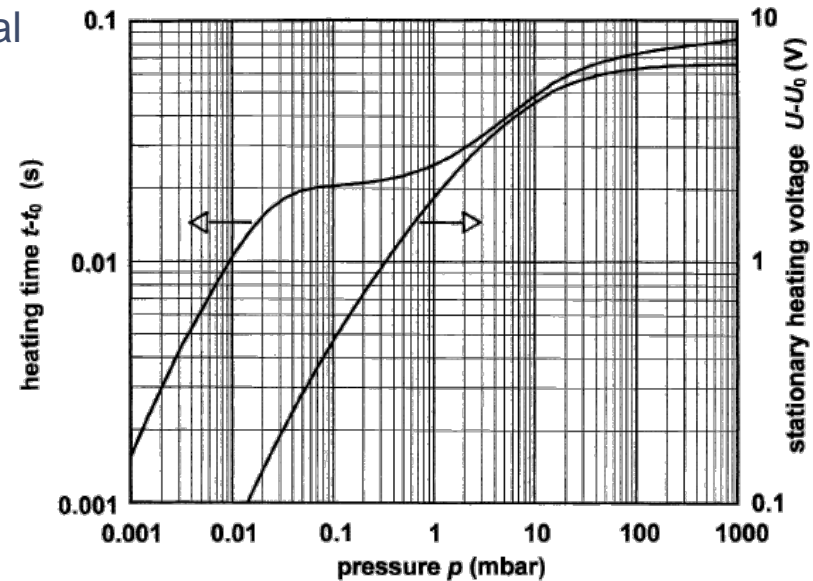
Jitschin and Ludwig, Vacuum 75, 169-176, 2004

### b) Superposition of AC temperature signal on stationary $T_p$

D.J. Seong et al., Key. Eng. Mat., **277-279**, 990-994, 2005.

✓ Conventional Pirani sensor may be used

✗ Medium to slow dynamics (1-10 Hz)



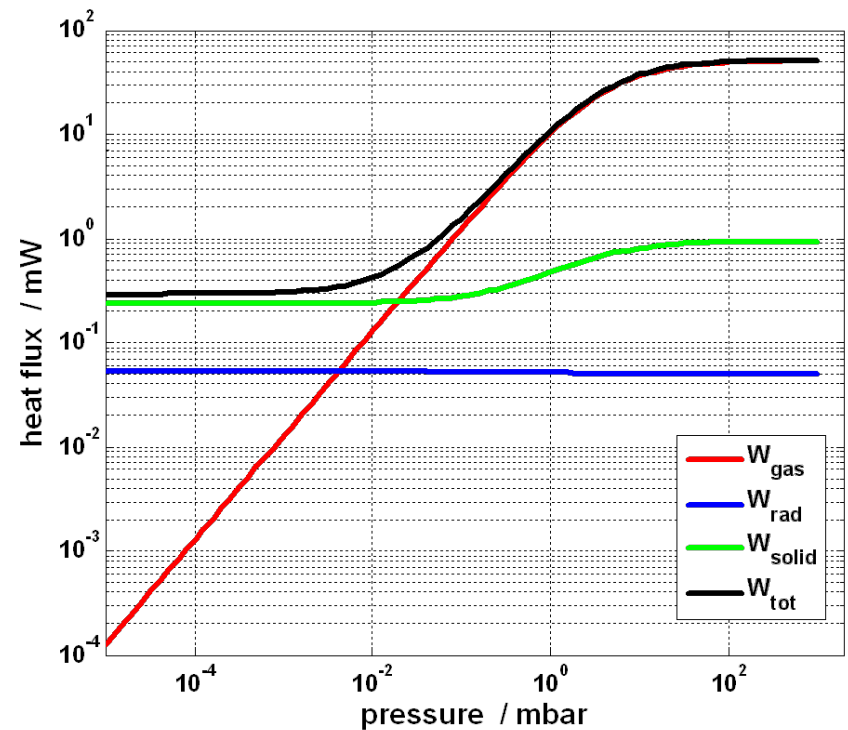
W. Jitschin et al., Vacuum, **75**, 169-176, 2004.

# Reducing the Lower Pressure Limit

$$W_{gas} \ll W_{rad}, W_{solid}$$

## 1) Radiation losses

- Reduce Pirani temperature
  - MEMS
  - use low emissivity materials



# Reducing the Lower Pressure Limit

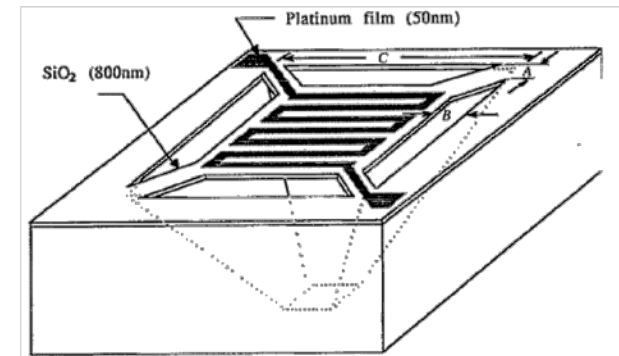
## 2. Heat losses through Pirani filament ends

- typically dominate over radiation losses

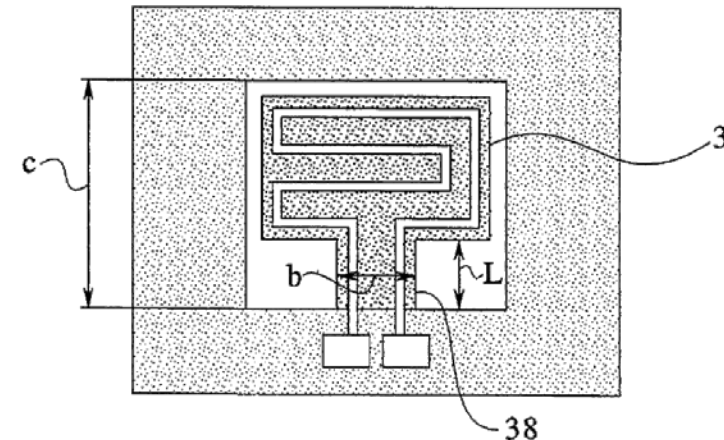
$$W_{solid} \gg W_{rad}$$

- increase filament length
  - helicoidal filament
- inhibit thermal flux from heated Pirani element to support through bridges
  - high thermal resistance bridges between support and Pirani-membrane in MEMS-Pirani
  - heated bridges

A. W. Van Herwaarden et al., *Sensors & Actuators*, **14**, 259-268, 1988.



P. K. Weng et al., *Rev. Sci. Instrum.*, **65**, 492-499, 1994.



J. Schieferdecker et al., *US005597957A*

# A Comparison

## Conventional Pirani with helicoidal filament

- ✓ Robust and reliable
- ✓ Good long term stability
- ✓ Good dynamics
- ✓ Low price
- ✗ Limited sensitivity @  $p < 10^{-4}$  mbar and  $p > 10^2$  mbar
- ✗ Gas type dependence

## Convection enhanced Pirani

- ✓ Increased sensitivity at  $p > 10^2$  mbar
- ✗ Large volume
- ✗ Slow dynamics
- ✗ Dependence on mounting orientation
- ✗ Gas type dependence

## MEMS-Pirani

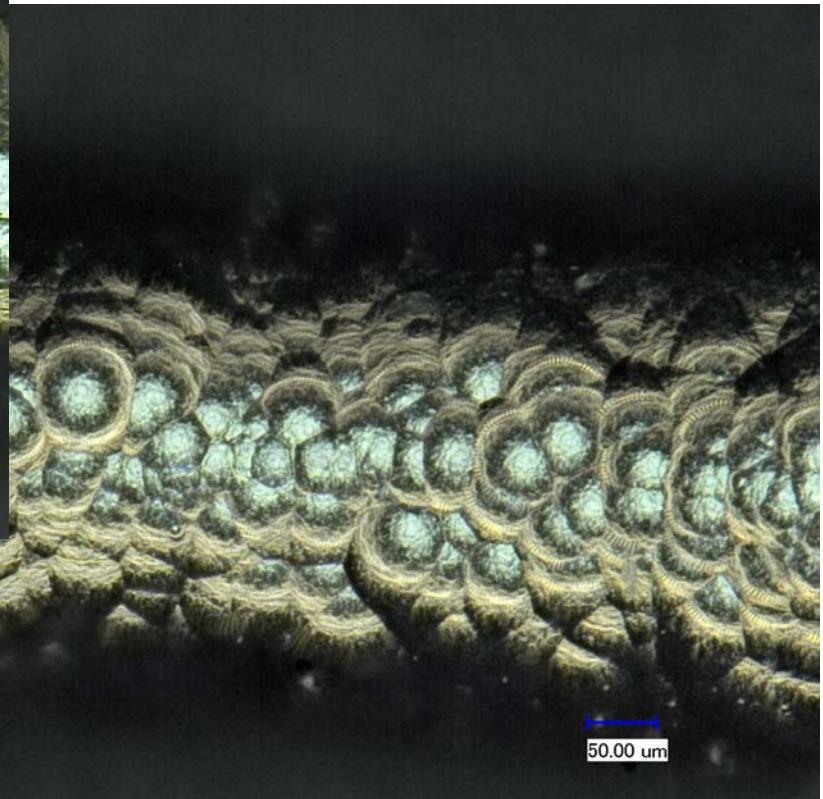
- ✓ Extended low pressure range when  $W_{\text{solid}}$  is reduced
- ✓ Better sensitivity at atmospheric pressure due to
- ✓ Small volume
- ✓ Good dynamics
- ✗ Price
- ✗ Contamination ➤ limited long term stability
- ✗ Gas type dependence

## Heat capacity measuring Pirani

- ✓ Increased sensitivity at  $p > 10^2$  mbar
- ✓ Low price as based on conventional Pirani
- ✗ Medium to slow dynamics
- ✗ Gas type dependence



# Carbonized filament





# Fluorine Etch of W Wire

Dry etch application:  $\text{CHF}_3$ ,  $\text{CF}_4$ ,  $\text{C}_4\text{F}_6$ ,  $\text{CH}_2\text{F}_2$ ,  $\text{C}_4\text{F}_8$

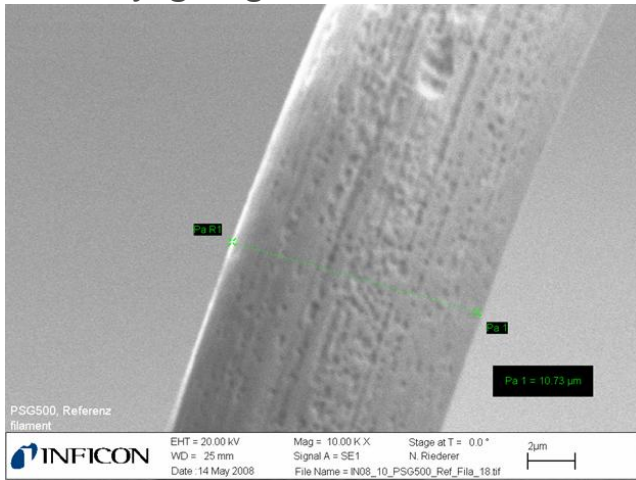
W is not resistant to those gases.

-> Wire gets thinner

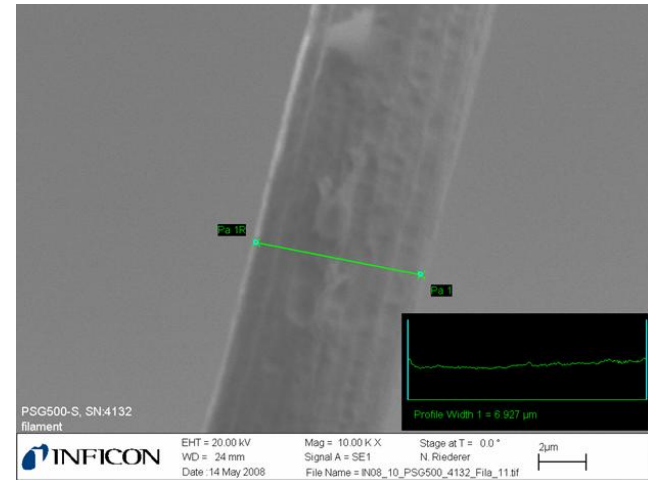
-> Resistance increases

Eventually gauge failure after 2 weeks

$$R = \rho \frac{l}{A} = \rho \frac{l}{\pi r^2}$$

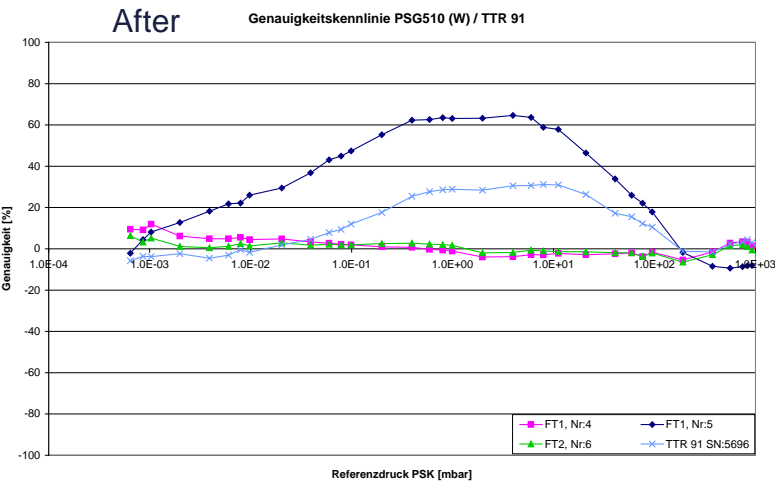
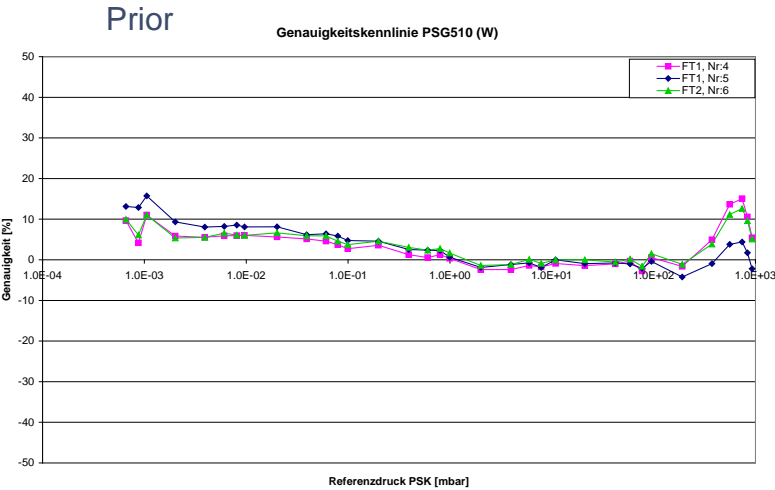


The measured diameter of a new W wire amounts to approx. 10.7 μm (with tolerances)



The measured diameter of the W wire of SN:4132 amounts to approx. 6.9 μm (with tolerances)

# W Filament 10 min in 40% HF Solution

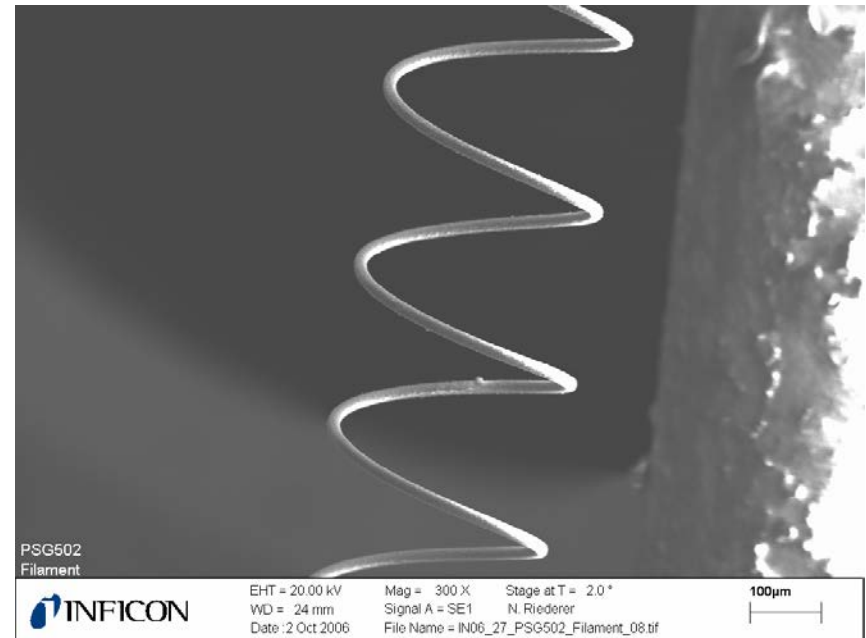
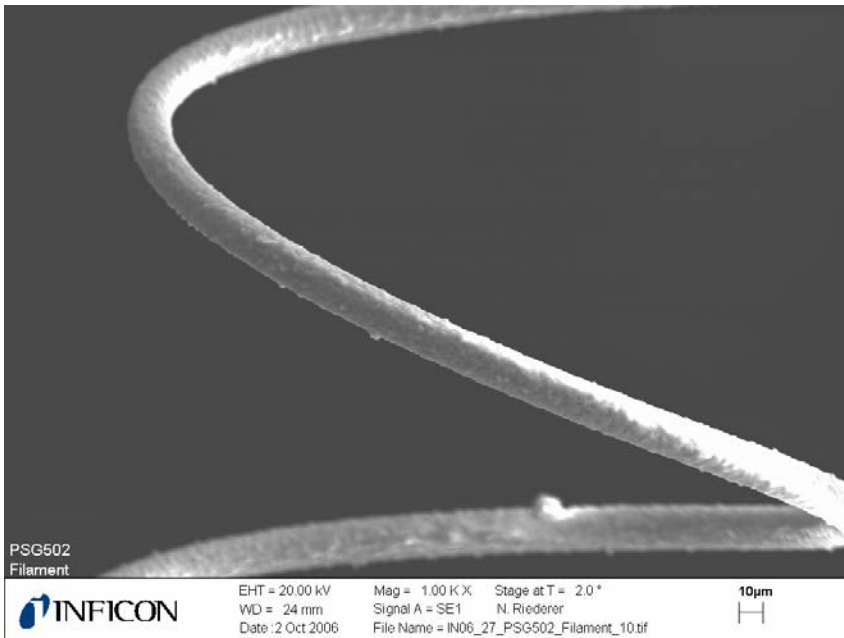


Nr. 0  
Filament Resistance after Test  
41.46  $\Omega$

Nr. 5  
40.39  $\Omega$

Nr. 6  
39.7  $\Omega$

# PSG502 Ni Filament with HF



# Pirani in liquid (!) HF

Nickel

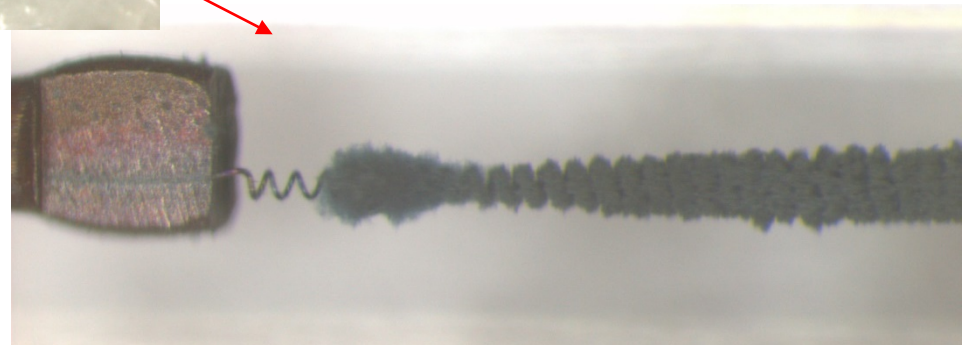
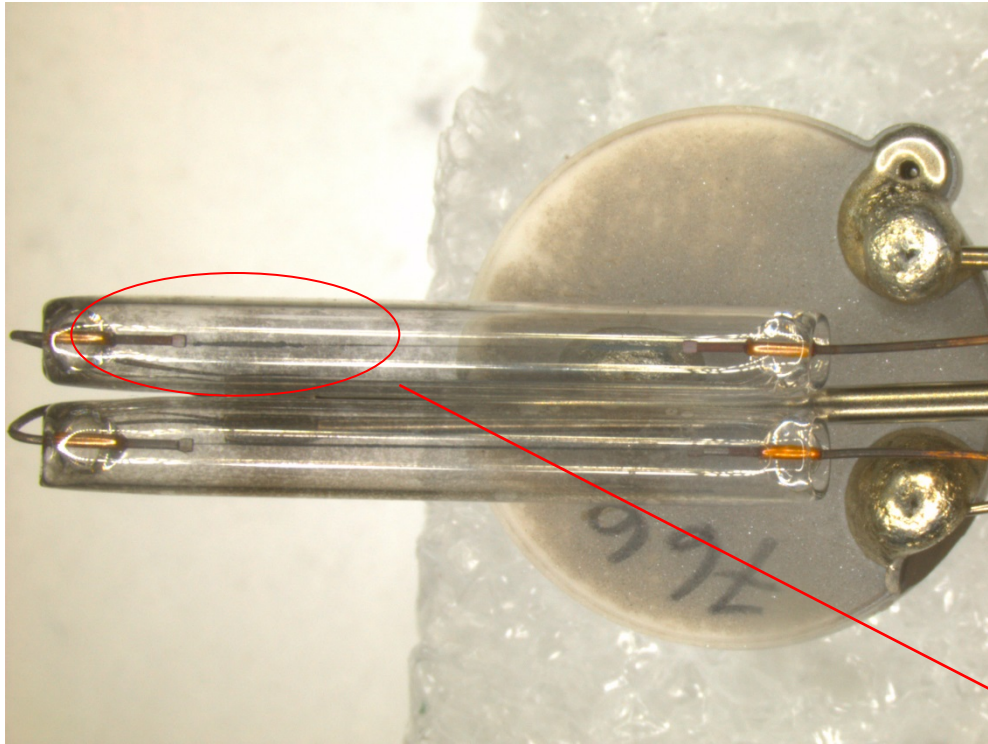


Tungsten



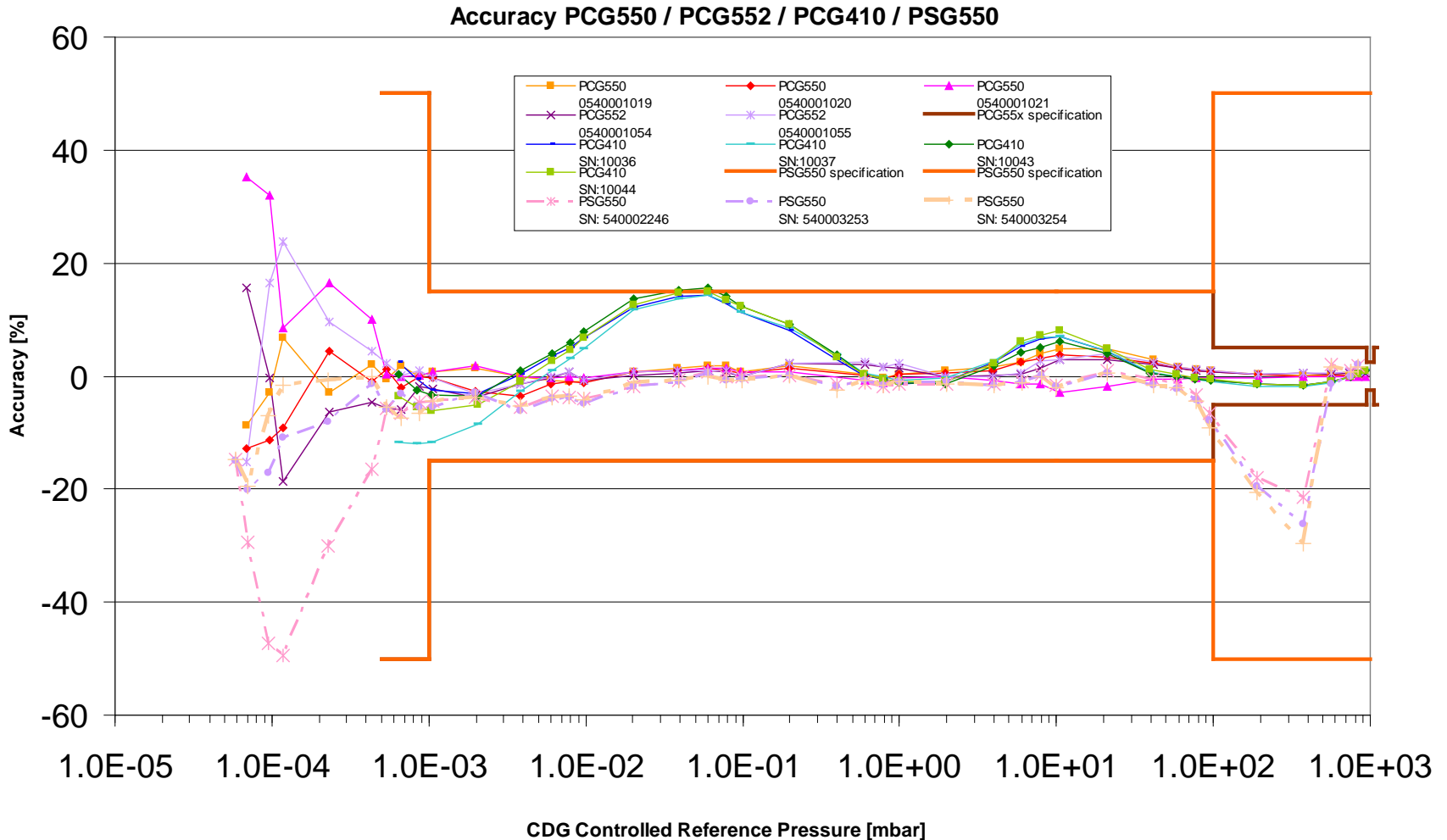
Experiment conditions: HF 40% 10 minutes

# Critical Materials: ZnO

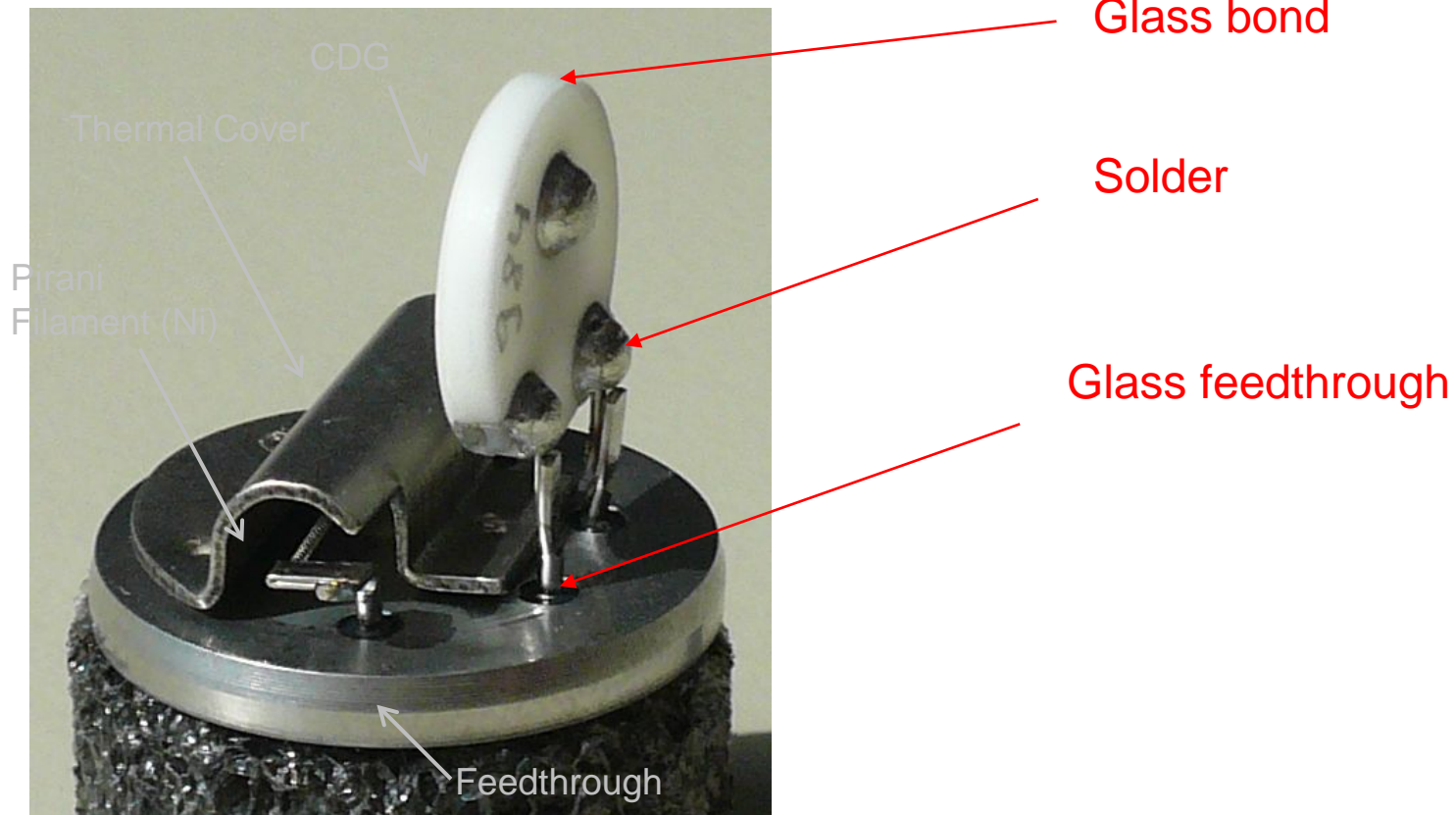




# Accuracy PSG vs. PCG



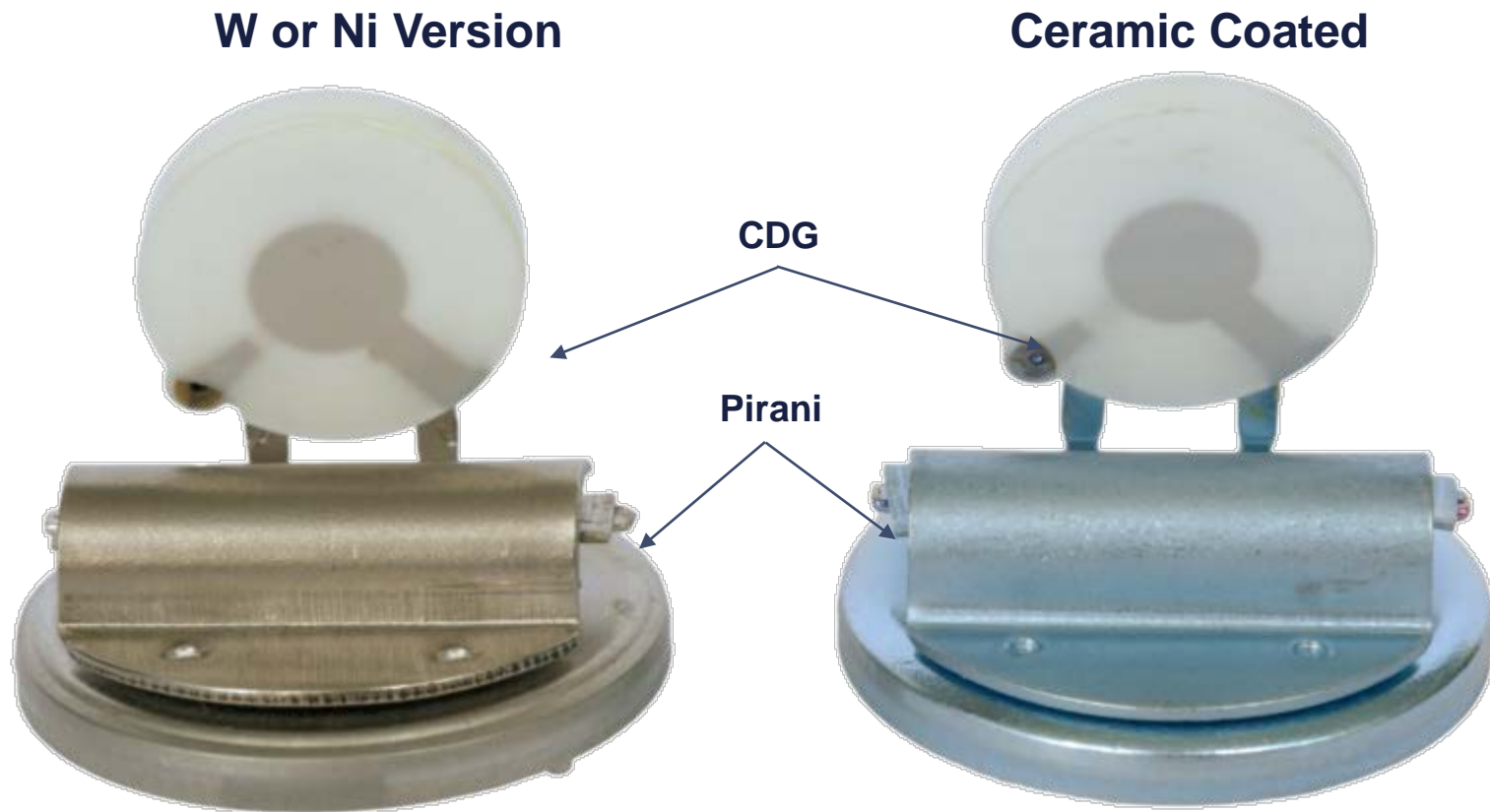
# Insufficient Corrosion Resistance





# What is the Ceramic Coated sensor?

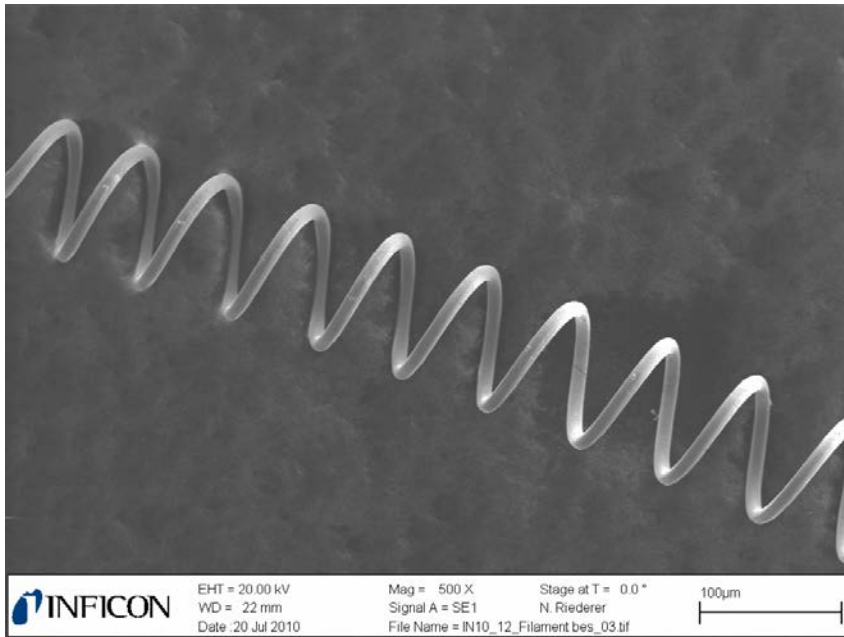
The Ceramic Coated Pirani is a fully  $\text{Al}_2\text{O}_3$  coated sensor. The ceramic coating prevents the sensor from corrosion in harsh environment.



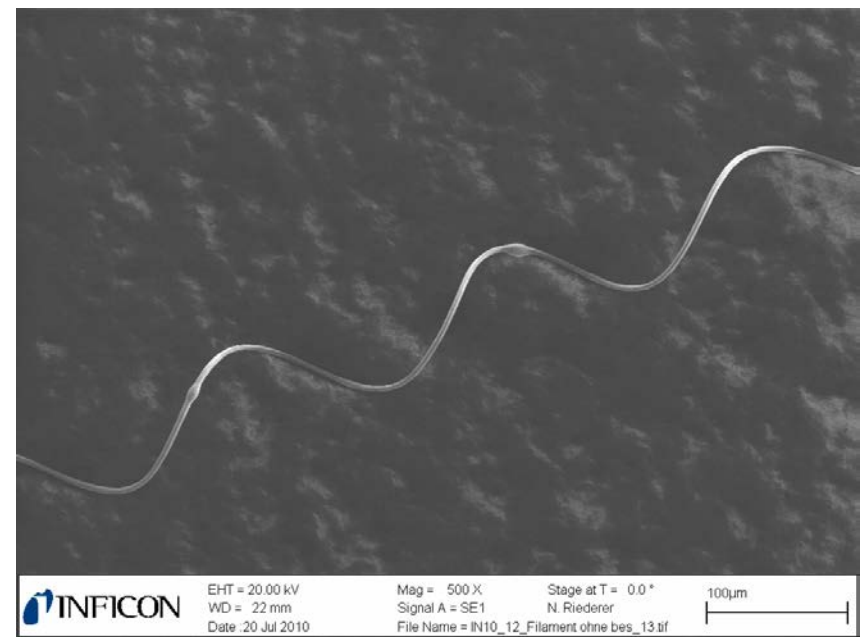
# Testing

To investigate that the coating is sealed, we run tests with  $H_2O_2$  and saw that the ceramic coated filament showed no dissolution or corrosion whatsoever.

Coated filament after 60 min in  
50 %  $H_2O_2$  at 25 °C



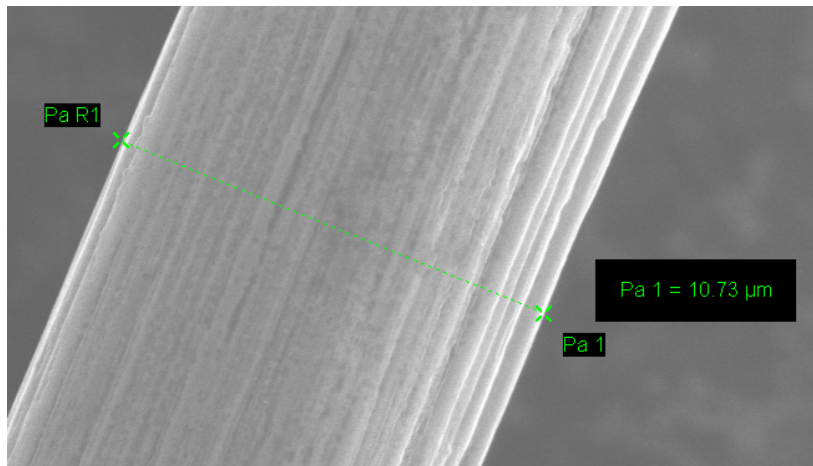
Uncoated tungsten filament after  
60 min in 50 %  $H_2O_2$  at 25 °C



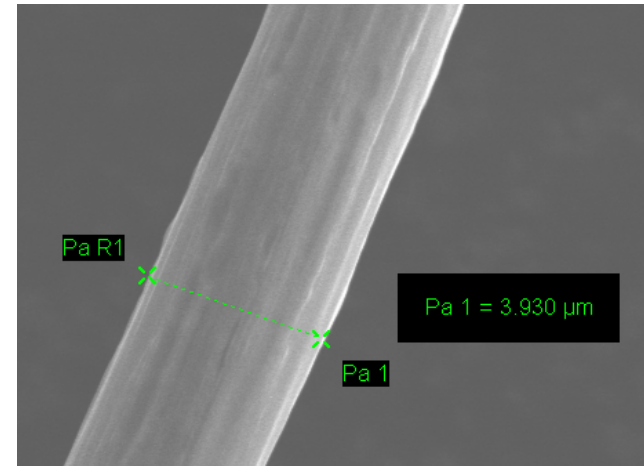
# Testing

When moving closer into the SEM picture we see that the coated filament still has the exact same diameter then before the test. The uncoated filament has been eaten down to more then the half of the original diameter.

coated filament after 60 min in  
50% H<sub>2</sub>O<sub>2</sub> at 25 °C



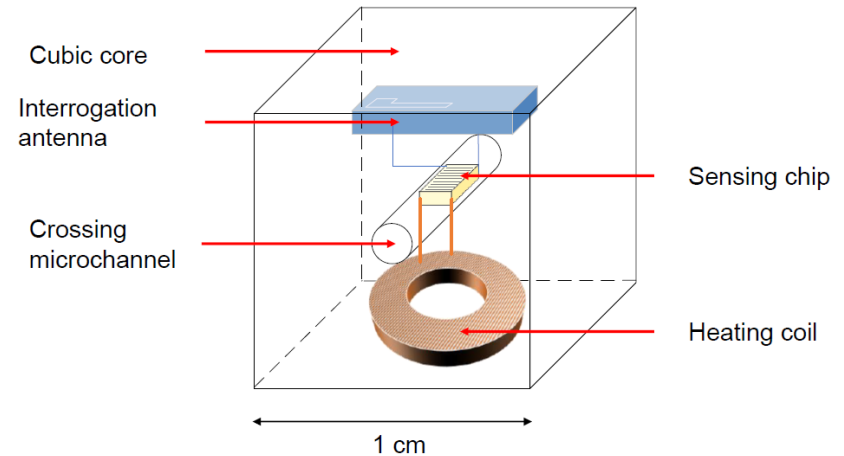
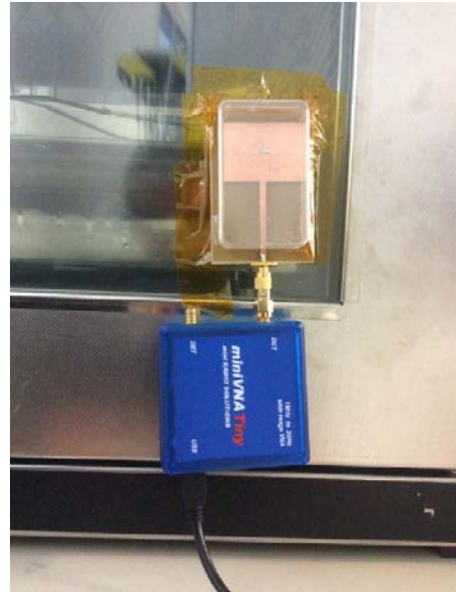
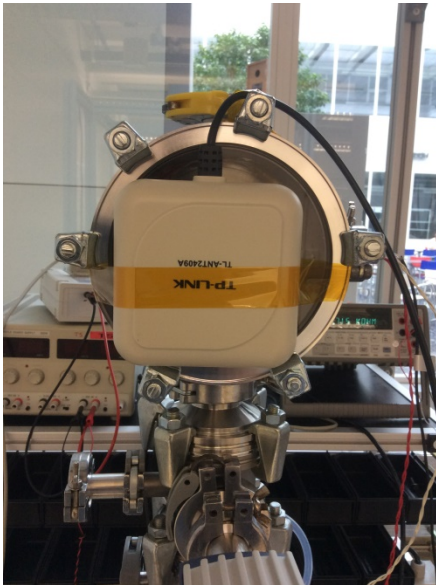
uncoated tungsten filament after  
60 min in 50% H<sub>2</sub>O<sub>2</sub> at 25 °C



Original wire diameter =  
Coating thickness =

10.6 μm  
0.08 μm

# Wireless Pressure Sensing



Sofia Toto, Ph.D. Thesis, Karlsruhe Institute of Technology, 2018



# Manufacturing

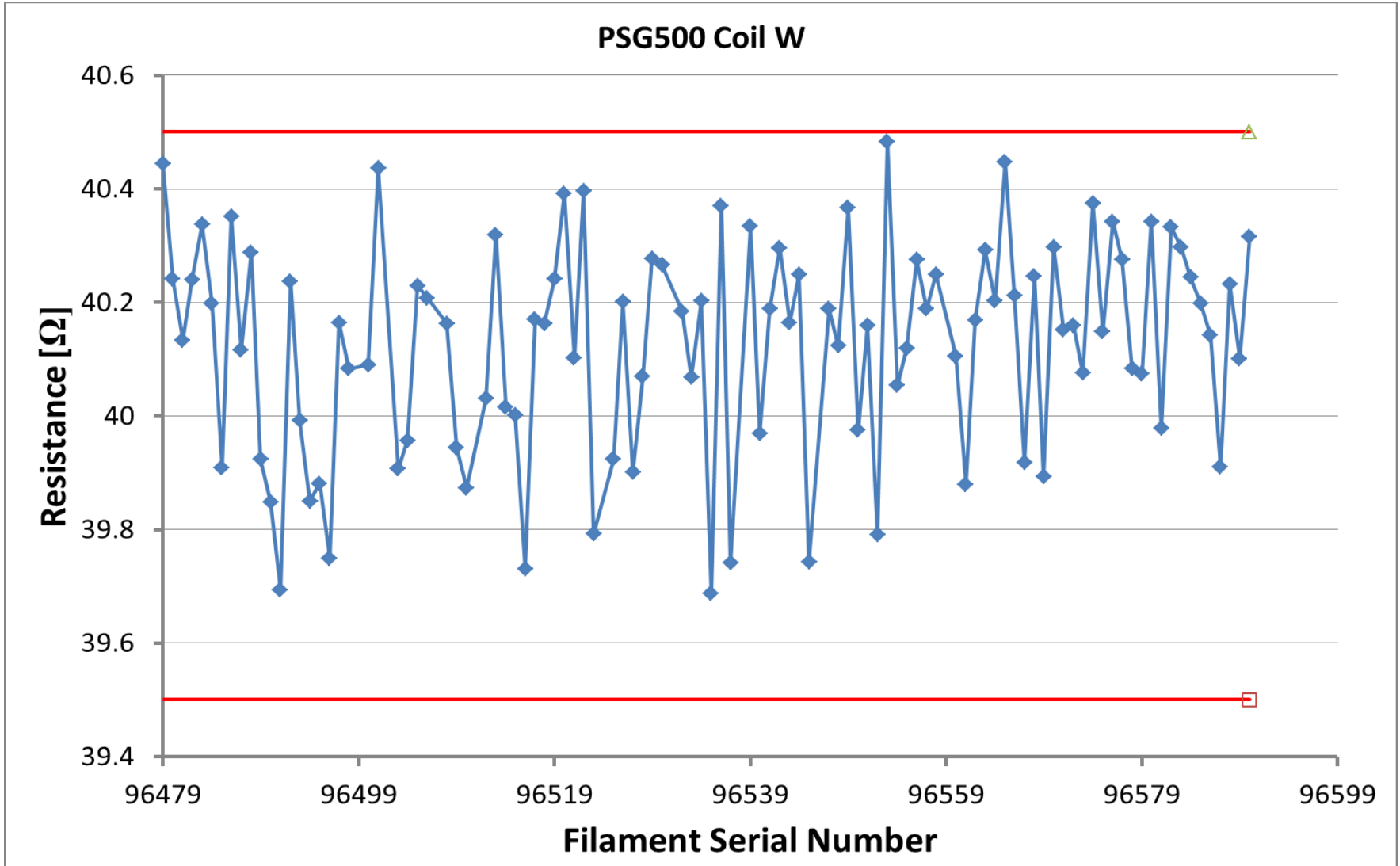




# Manufacturing: Automation



# Manufacturing: SPC Pirani





# Manufacturing: Optical Inspection Tool

Einrichtung | Produktion (Kontrolle) | Nachkontrolle | Benutzer Modus | Hilfe

Barcodenummer:   
Gefundene Einträge: A22J00066  
A22J00072  
Bsp. gut April 2011  
Bsp. schl April 2011  
Nov. 2010

Barcodenummer: Bsp. schl April 2011  
Seriennummer: 456  
Produktnummer: 3333  
Flanschtyp: testflansch  
Gehäusetyp: testgehaeuse

13.04.2011 09:31 Fehler -

Benutzername Flipper  
Matrixcode Fehler Details...  
Schweißnaht Fehler Details...  
Dichtungsfläche Fehler Details...  
Plasmablende Ok Details...

Bilder exportieren...

13.04.2011 09:26 Fehler +

Resultate Schweißnahtkontrolle-Test vom 13.04.2011 09:31  
Verarbeitungszeit 188 ms Information

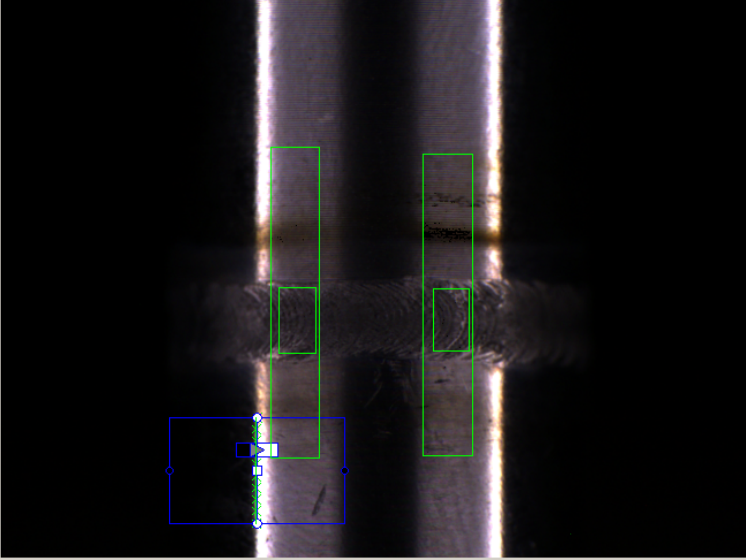
Für diesen Test wurden 20 Bilder auf dem Datenträger mit der ID 1 gespeichert. Momentan ist der Datenträger mit der ID 1 angeschlossen.

Aktuelle Konfiguration laden | Konfiguration beim Test laden | Test ausführen | Änderungen speichern

Status ●  
Verarbeitungszeit: --

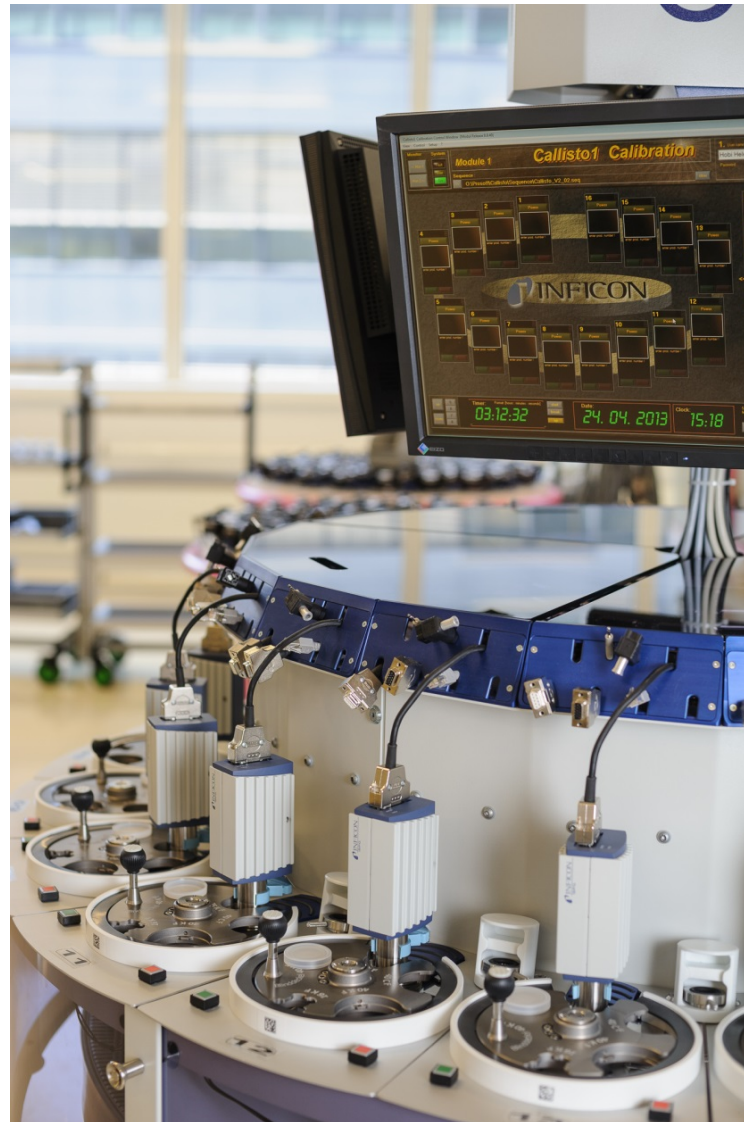
< 7 > / 20

Position Testregionen  
Schwelle 10  
Zentrum (438, 810)  
Winkel -89.6 °



Motor ● SPS ● Inficon PC ● Kamera ● Externe - HD ● Not Aus ● Automatik Betrieb ●

# Factory Calibration



# Optical Methods



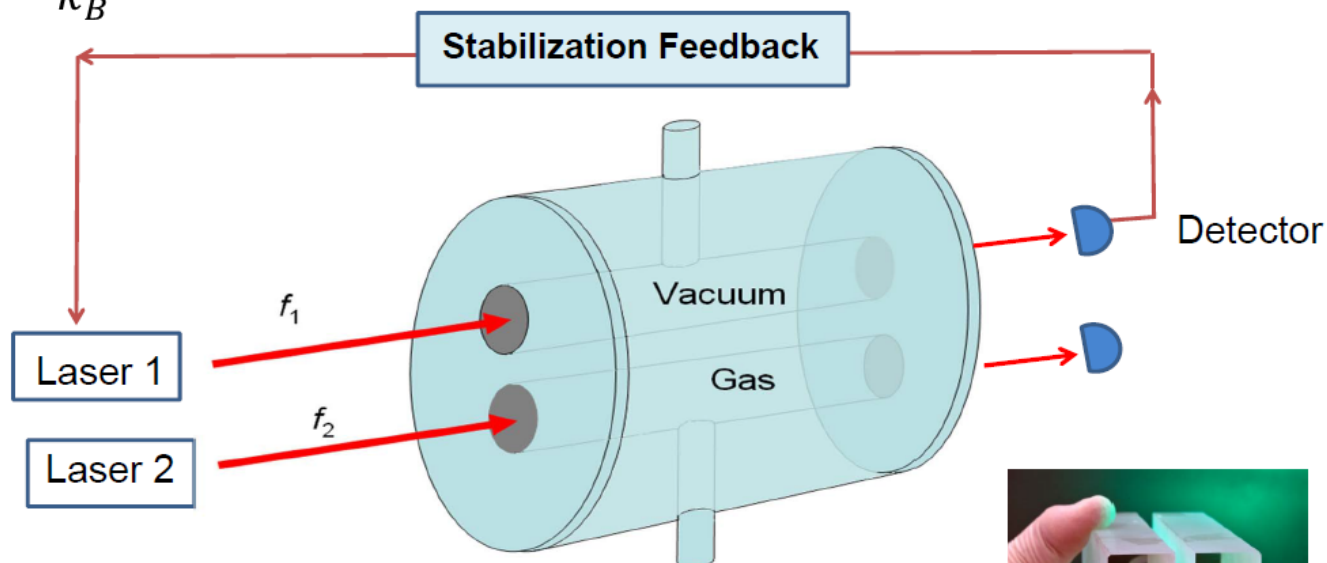
INFICON Optical Diaphragm Gauge

# NIST FLOC

$$\lambda_{gas} = \frac{c}{nf}$$

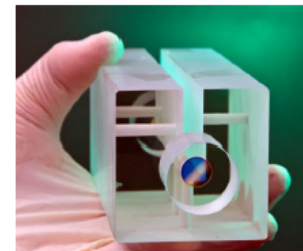
$$n - 1 \propto \frac{p}{k_B T}$$

## The Fixed Length Optical Cavity FLOC

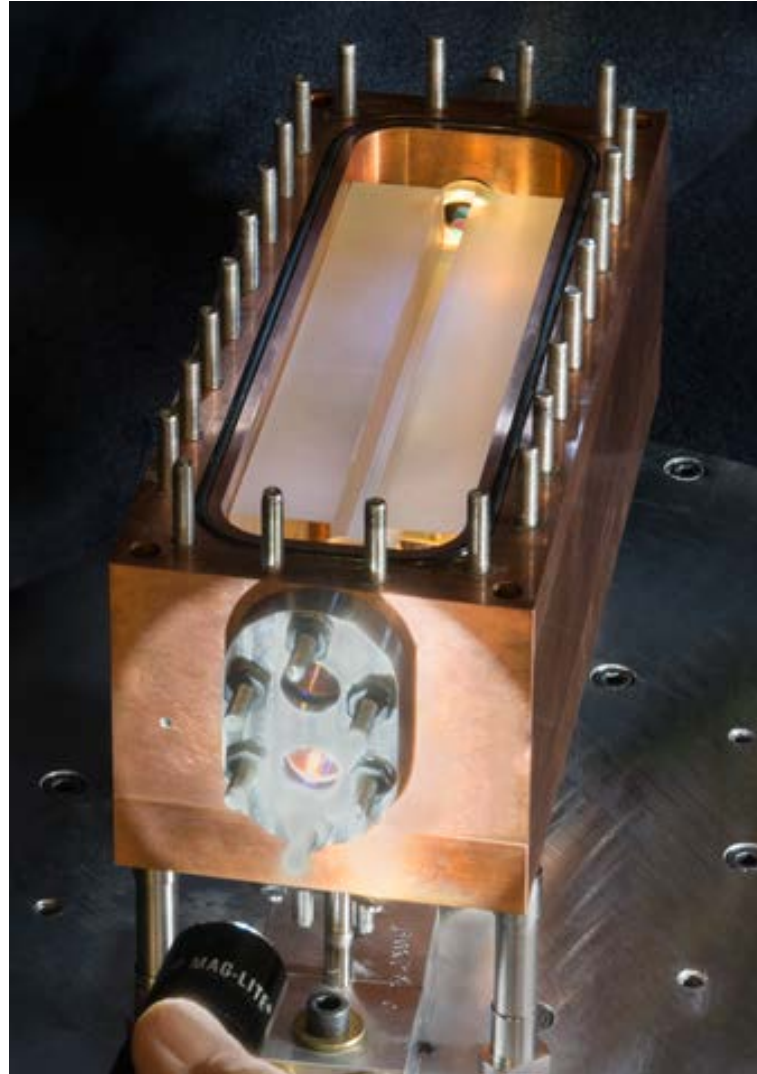


Pressure determined from  $f_1 - f_2$

Uncertainty goal ( $k=1$ ): 2.1 ppm at 1 atm

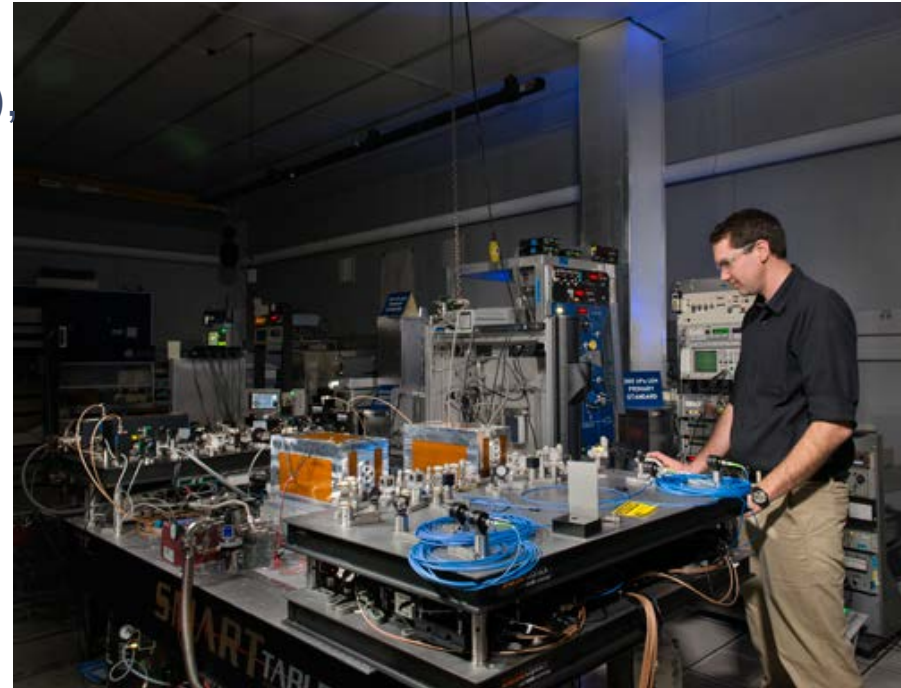


# NIST FLOC



# NIST FLOC

- Compared to NIST Hg Standard
  - 35x better resolution 0.1 mPa ( $10^{-6}$  mbar)  
Cube: 0.95 ppm F.S. =  $9.6 \times 10^{-4}$  mbar @ Atm)
  - 100 x faster
  - Covers 8 decades of pressure in one instrument
  - Negligible hysteresis 8  $\mu$ Pa/hr
- Customer calibrations will be done now on new system

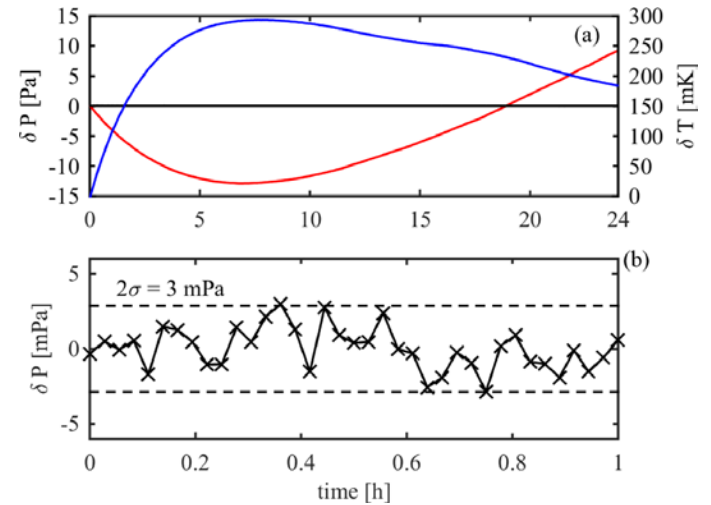
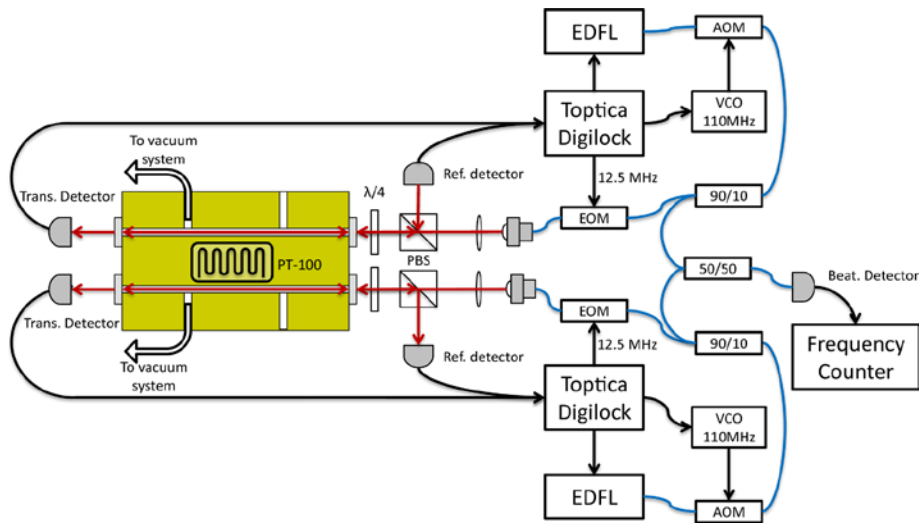


FLOC: 15.5 x 5 x 5 cm<sup>3</sup> + laser optics

Cube: 19.45 x 11.0 x 10.3 cm<sup>3</sup> incl. all electronics



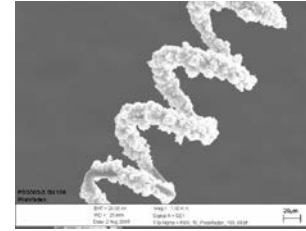
# University of Umea: GAMOR



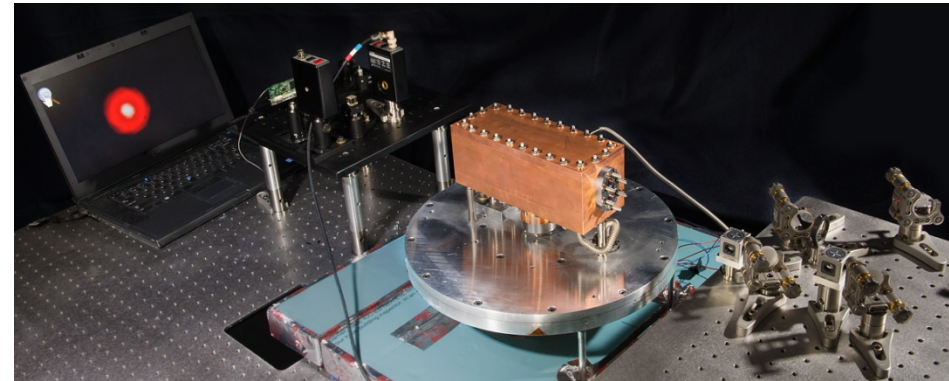
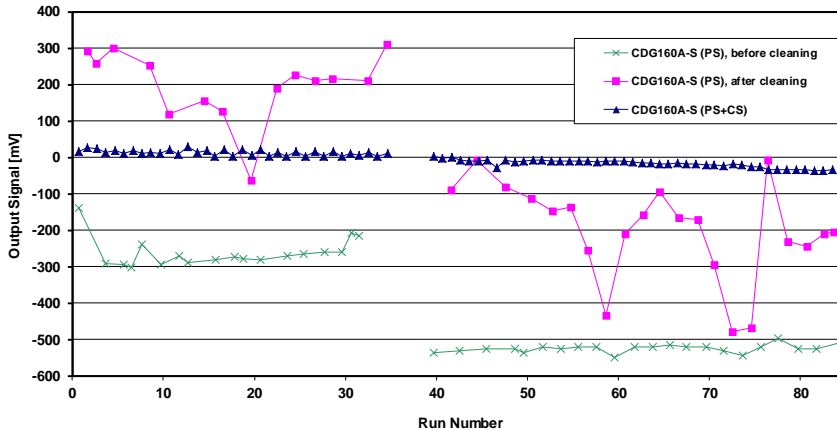
Silander et al., Gas modulation refractometry for high-precision assessment of pressure under non-temperature-stabilized conditions, JVST A36, 03E105 (2018)



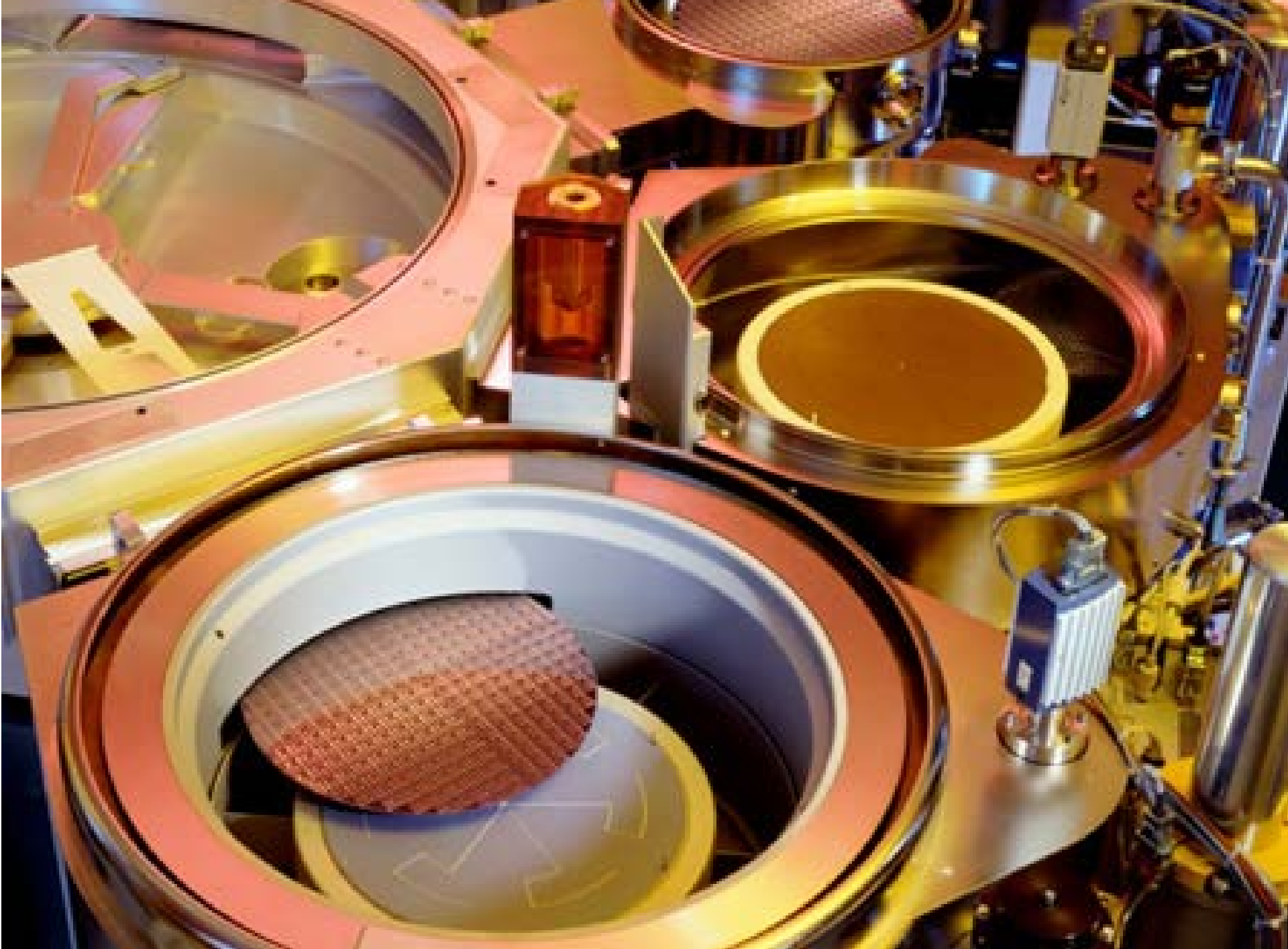
# Summary



- Gauges are becoming more robust for industrial processes
- To achieve this requires a deep understanding of many different fields of science
- Optical methods are being established for standards laboratories



[www.nist.gov/https://www.nist.gov/pml/nist-chip-photonic-sensors-pressure-and-light](http://www.nist.gov/https://www.nist.gov/pml/nist-chip-photonic-sensors-pressure-and-light)



# Vacuum Control in Liechtenstein

