Challenges for Extreme High Vacuum (XHV)

H.F.Dylla
Thomas Jefferson National Accelerator Facility
(Jefferson Lab)
Newport News, VA USA 23606

CERN Accelerator School, Platja D'Aro,Spain
May 16-24, 2006

Challenges for XHV: Outline

- Definition
- Development History
- How does one obtain XHV?
  - materials
  - surface conditioning
  - pumping technology
  - leak detection
  - fabrication technology
- How does one quantify XHV?
  - Pressure and partial pressure analysis
  - understanding gas sources
- Who cares about XHV?
  - physics devices
  - materials processing?
  - electron tubes

XHV Definition

Definition:

UHV: 10^{-6} Pa (Europe) and 10^{-7} Pa (USA) -10^{-10} Pa

XHV: <1x10^{-10} Pa (or 10^{-12} mbar)

UHV:

- is routinely achieved worldwide with commercial equipment (largely with bakeable, stainless steel systems and “dry” pumps)
- Development largely driven by physics research and semiconductor manufacturing

XHV:

- Achieved in a only a few specialized laboratories (physics research) with difficulty, special equipment and procedures

Gas and Surface Parameters at XHV

Pressure (Pa)	Molecular Density (@298K)	Molecular Flux at 298K (molec.cm^{-2}s^{-1}	Molecular Mean Free Path λ* (N_2 at 295K)	Monolayer Time**
10^{-10}	2.5 x10^4 cm^{-3}	2.9x 10^8	3.4 x 10^4 km	44 days
10^{-13}	25 cm^{-3}	2.9 10^5	3.4 x 10^7 km	120 yrs
10^{-17} Interstellar space	0.25 m^{-3}	29	2.3 x 10^3 a.u.	1.2 x 10^6 yrs

*Mean free path for electrons, $\lambda_e = 4\sqrt{2}\lambda$ and for ions $\lambda_i = \sqrt{\lambda}$.

** Assuming 10^{15} sites per cm^2 and a sticking probability of 0.5

XHV Development History

1930: **P.A.Anderson** measuring the work function of barium in sealed glass tubes immersed in liquid H_2 no doubt achieved XHV (no measurement)

1950's **R.Gomer**'s work on field emission and field ion microscopes also achieved XHV in sealed glass systems

1958: **Hopson and Redhead** measured with an Inverted Magnetron Gauge

$< 10^{-10}$ Pa in a glass system with LHe cold finger

1962: **W.D Davis**, stainless steel system pumped with ion pumps (some LN_2 assist) measured 4×10^{-11} Pa with magnetic sector mass spectrometer

1964: **Hopson**, glass system (alumino-silcate) with Ti getter and inverted magnetron ion pump, measured ~1×10^{-12} Pa with LHe cold finger

1977: **Thompson and Hanrahan**: stainless steel UHV system immersed in LHe, measured ~10^{-12} Pa with an in-situ mass spectrometer

1989: **Ishamaru** with "Ex-Process" aluminum system measured 4×10^{-10} Pa

1977-93: **Benvenuti measured** $<5 \times 10^{-12}$ Pa with NEG pumping and extractor gauges

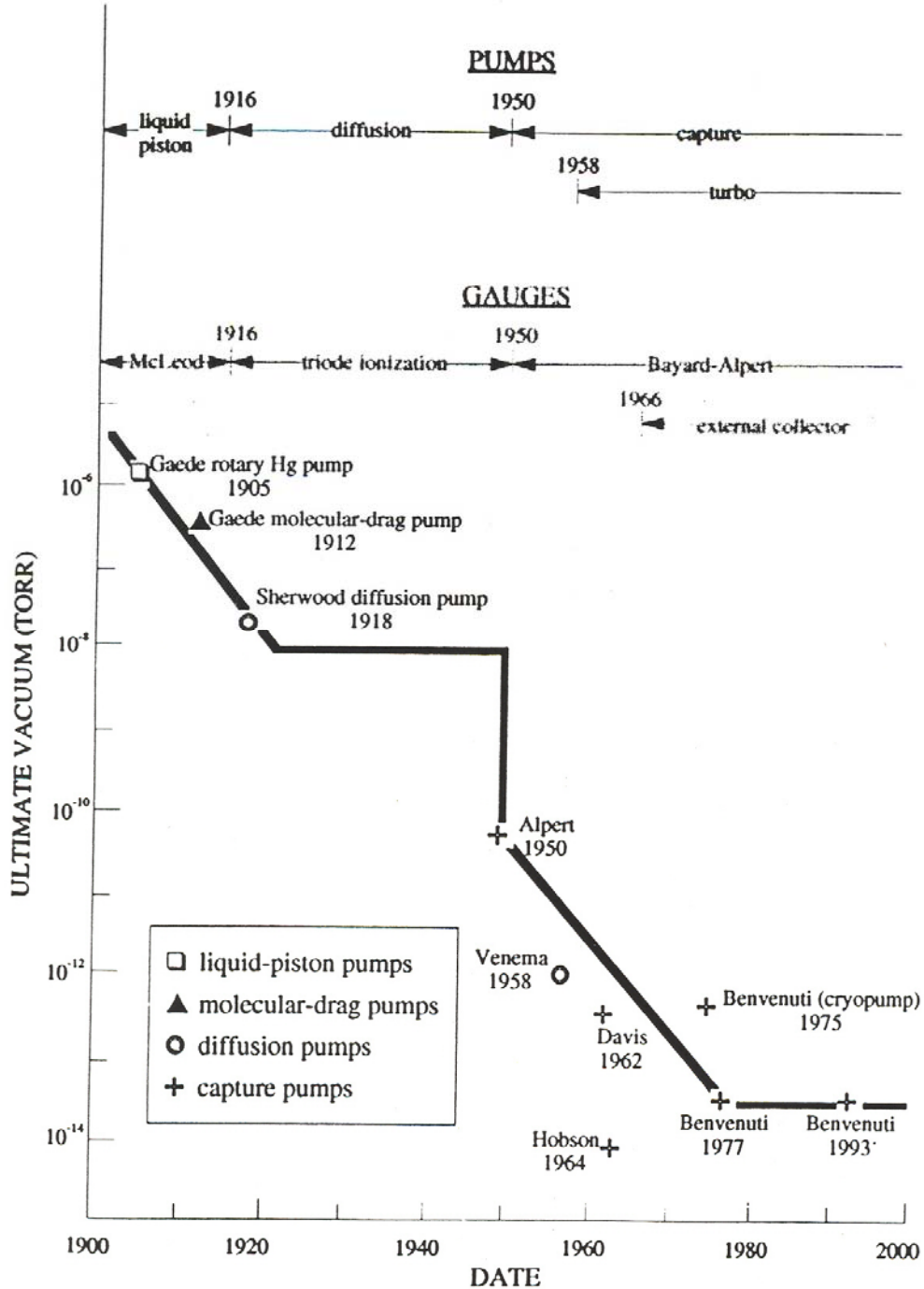
The Ultimate (Laboratory) Vacuum

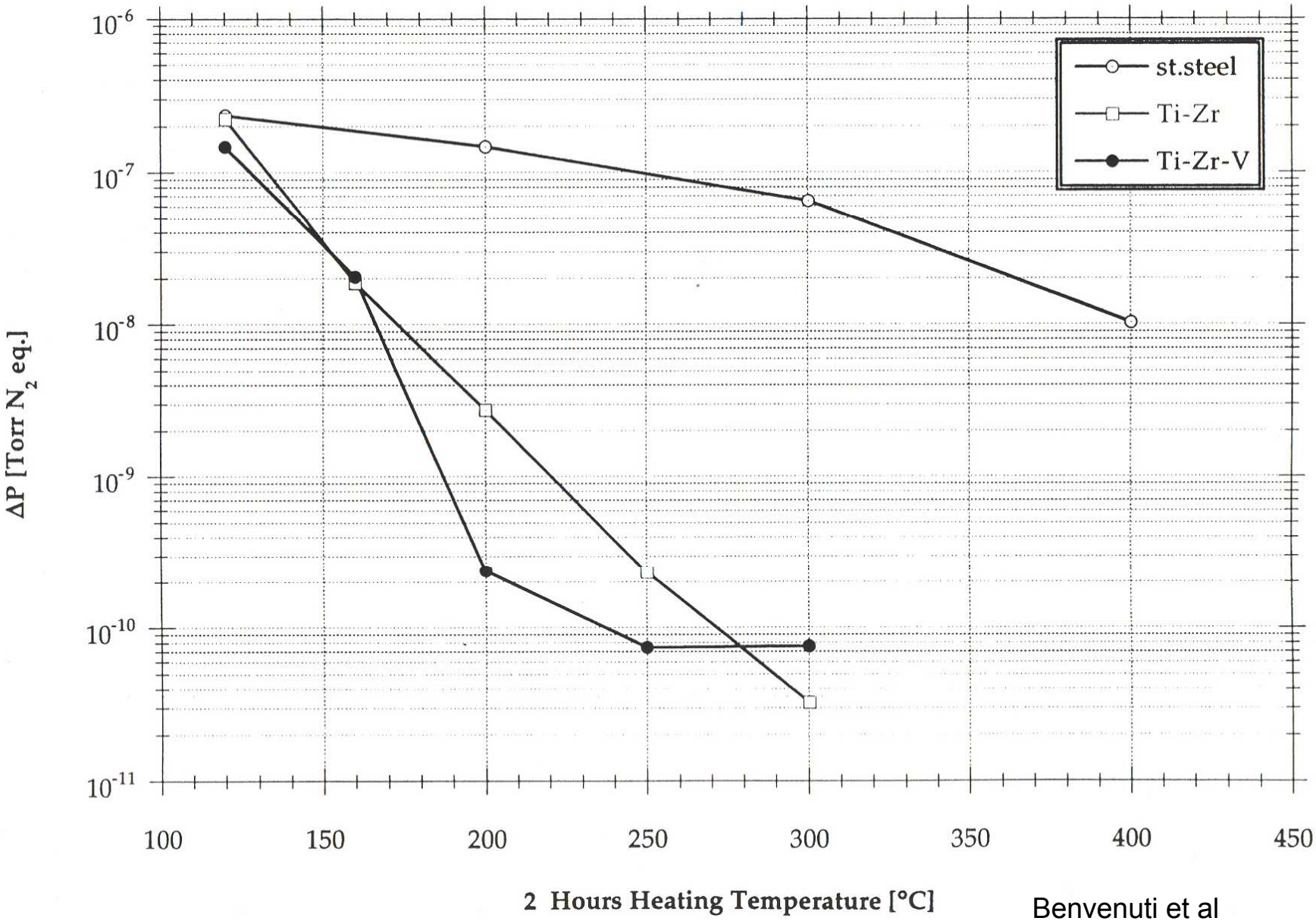
Hobson (1966): calculated the pressure in a small sealed vacuum system totally immersed in LHe (4.2K) should have a pressure of ~10^{-26} Pa based on the measured He isotherms

(assumes no in-leakage of He and no particle/radiation induced desorption of physisorbed He)

Weiss (2000): estimated that the residual vacuum level in a small cold (ie, LHe system) would have residual vacuum level of ~10^{-26} Pa due to background cosmic rays interacting with the vessel walls.

Note: there is no existing pressure (or density) measurement instrument for measurements in this range.


PUMPS
1916
1950
liquid piston
diffusion
capture
1958
turbo
GAUGES
1916
1950
McLeod
triode ionization
Bayard-Alpert
1966
external collector
Gaede rotary Hg pump
1905
Gaede molecular-drag pump
1912
Sherwood diffusion pump
1918
Alpert
1950
Venema
1958
Davis
1962
Benvenuti (cryopump)
1975
Benvenuti
1977
Benvenuti
1993
Hobson
1964
liquid-piston pumps
molecular-drag pumps
diffusion pumps
capture pumps
ULTIMATE VACUUM (TORR)
DATE
1900
1920
1940
1960
1980
2000



Benvenuti et al

Pumps for XHV

Kinetic Pumps (diffusion and turbomolecular)

* Provide continuous removal of gas from the system independent of integrated throughput and low pressure limit
- Capture pumps (ion, cryo and getter) don't remove gas from systems and have to be regenerated
- Kinetic pumps have potential back-streaming problems

Note: Some of the early XHV demonstrations were done with diffusion pumps but extreme care had to be taken with the intervening trap which prevents pump fluid back-streaming.

TMP's with special modifications provide a more reliable option:

- Magnetic suspension removes oil/grease lubricated bearings
- Backing with (dry) molecular drag pumps eliminates all oil in such systems
- High emissivity (SiOx) coatings on the blades minimizes eddy current heating
- Tandem TMP's increases the compression ratio for H_2

  -Cho et al (1995) achieved pressures in the low 10^{-10} Pa range with a magnetic TMP backed by a molecular drag pump

Pumps for XHV

Capture Pumps (ion, cryo and getter)

Ion Pumps

- Useful in combination with getter pumps for pumping inert (Ar,He) and chemically inactive gases that are present in UHV systems

- Problems:

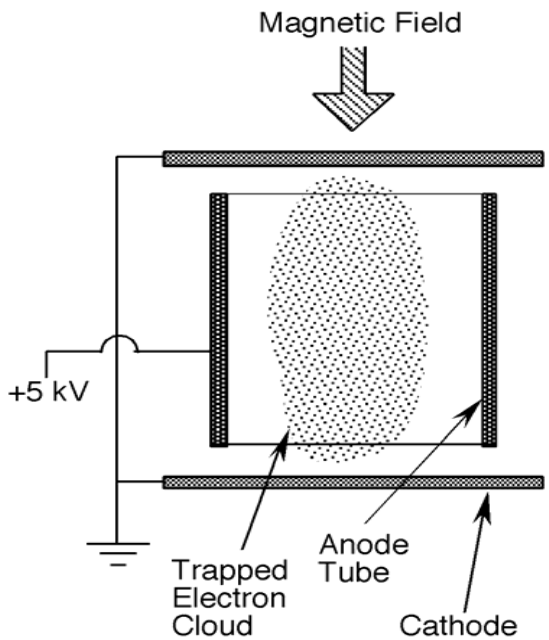
  (1) decreasing pumping speed (S) with decreasing pressures (p);

  $S \sim i_+/p$, a quantity proportional to the Penning discharge intensity

  (2) re-emission of gases implanted in the cathodes (esp., Ar)

  (3) generation of gases by ion bombardment of the cathodes

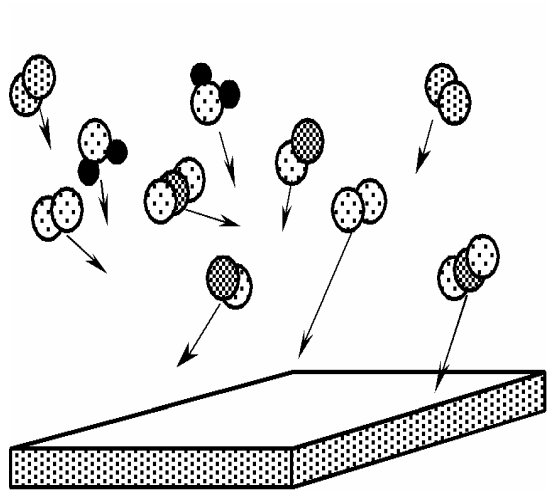
  (ex.: CH_4 and CO generation by H^+ bombardment



Pumps for XHV

Getter Pumps

- Widely used for XHV and for UHV-XHV accelerator applications
- Simplicity and ease of application to both large area and complicated geometries are virtues
- Need to be supplemented (by ion or TMP pumps) to pump chemically inert gases
- Sintered NEG geometries can increase both the effective pumping speed and capacity
- Evaporable getters allow conformation to the entire vacuum vessel
  (Benvenuti; “make the vessel a pump”)
- Benvenuti’s pioneering work in this area has achieved XHV both with discrete NEG strips and the conformal NEG coatings



Pumps for XHV

Cryopumps

Cry-condensation: pumping of a small number of physisorbed layers (2-3) on a cold surface
-low pressure limited by the adsorbent vapor pressure at the pump's temperature

Cryo-sorption: pumping on cold (large surface area) absorbent surface

-effective vapor pressure of the adsorbent given by the adsorption isotherm
(DR isotherm fits most residual gases)
-pumping capacity/area can be made large with suitable absorbents
(charcoal, anodized Al)

Problem:

Effectively shielding cold surface from radiation sources which will desorb gas and add to cryo-heat loads

Innovative solutions:

- the beam sheilds designed for the LHC

Minimizing the Gas Sources

XHV systems demand the choice of only a few UHV/XHV qualified materials:

- Stainless steels
- Aluminum
- Copper
- (Glass in the early demos)

and qualified conditioning techniques for these materials:

- Highly purified raw material
- Pre- and in-situ bakes
- Optional permeation or pumping barriers on the vacuum surface

Some measured outgassing rates at room temperature suitable for XHV

Material	Surface treatment	Outgassing rate (Torr l s^{-1} cm^{-2})	Reference
Stainless steel	V.F. 550°C/72 h B. 250°C/24 h	$1x10^{-16}[H_2]$	Grosse & Messer 1980
Stainless steel (304L)	A.F. 400°C/38 h B. 150°C/7 d	$8x10^{-16}[H_2]$	Marin 1998
	A.F. 390°C/100 h B. 150°C	$4x10^{-15}[H_2]$	Bernadini 1998
Stainless steel	V.F. 960°C/25 h B. 180°C/6 h	$8x10^{-16}[H_2]$	Fremery 1999
Aluminum	B. 525°	$8x10^{-16}[N_2]$	Ishikawa 1997
Copper (OFHC)	V.F. 550°C/72 h B. 250°C/24 h	$5x10^{-17}[H_2]$	Grosse & Messer 1980
Copper (OFHC)	B. 525°C	$1.5x10^{-16}[N_2]$	Ishikawa 1997
TiN on Stainless steel.	V.F. 430°C/100 h + 500°C/100 h	$8x10^{-17}[H_2]$	Ichimura 1999
Aluminosilicate glass	B. 500°C/18 h, 600°C/2h, 700°C/2h, 500°C/10 h	$2x10^{-16}[N_2]$	Hobson 1964

A.F. = Air fired
B = Bake
V.F. = Vacuum fired

1Torr l s^{-1} cm^{-2} = $1.33x10^{3}$ Pa m s^{-1}

References for Outgassing Rates

Bernadini M. et al., J.Vac.Sci.Technol.A, **16** ,188 (1998).

Fremery J.K., Vacuum, **53**, 197 (1999).

Grosse G and Messer G., Proc. 8th International Vacuum Congress, p. 399 (Cannes, 1980).

Hobson J.P., J.Vac.Sci.Technol., **1**, 1 (1964).

Ichimura.S., et al., Vacuum, **53**, 291 (1999).

Ishikawa Y., Paper at IUVSTA Workshop on Outgassing Properties of Materials, Gräftåvallen, Sweden 1997.

Marin P. et al., Vacuum, **49**, 309 (1998).

Nemanič V. and Šetina J., J.Vac.Sci.Technol.A, in press.

How to Measure XHV?

- Recall from the historical overview that XHV was obtained in a number of labs long before these vacuum levels could be measured
- XHV gauging requires the same attention to proper choice of materials and materials conditioning as the XHV vessel

The gauges which have demonstrated sufficient sensitivity, and sufficiently small background effects to measure XHV conditions have been modified:

electron impact ionization gauges:

- Extractor
- Bent beam/Bessel Box
- Inverted Magnetrons

XHV Gauging: Room for Improvement

Novel solutions for XHV Gauging remain to be demonstrated:

- Laser ionization

  -resonant ring down spectroscopy as been applied to specific molecules (CO and H_2O) at the 10^{-7} Pa level (Looney)

  -non resonant LIF has been applied to 10^{-10} Pa level (Ichamura)

- Cross-beam ionizers

  -where the ionization and ion collection volumes are further removed from the XHV volume

- Surface flux monitors

  -early measures of XHV in the pre B-A gauge era

Characteristics of gauges suitable for XHV

Gauge	Electron Emission (A)	P_x** (Torr)	P_{min}*** (Torr)	Sensitivity (A $Torr^{-1}$)	References
Hot-cathode magnetron (*Lafferty gauge*)	$1x10^{-7}$ 10^{-9}	$8x10^{-16}$ $2x10^{-16}$	10^{-15} 10^{-15}	$5x10^{-2}$ [N_2] 0.5 [N_2]	Chen 1987 Gotoh 1995
Extractor (Leybold IE511)	$1.3x10^{-3}$	$1.5x10^{-12}$	$< 10^{-13}$	$7x10^{-3}$ [N_2]	Watanabe 1991
90° Bent-beam (*Helmer gauge*)	$3x10^{-3}$	$< 1.5x10^{-14}$	$8x10^{-15}$	0.13 [N_2]	Benvenuti 1980
180° Bent-beam* (*Ion spectroscopy gauge*)	$5x10^{-3}$	$< 1.5x10^{-14}$	$< 10^{-15}$	$6x10^{-2}$ [N_2]	Watanabe 1992
256.4° Bent-beam*	$1x10^{-4}$	$3x10^{-15}$	$3x10^{-15}$	$2.4x10^{-4}$ [H_2]	Oshima 1994
Bessel box* (*A-T gauge*)	$3.5x10^{-5}$	$6x10^{-13}$	$8x10^{-14}$ [H_2]	$1x10^{-4}$ [H_2]	Akimichi 1999

* These gauges have electron multipliers.

** Pressure at which the ion current equals the residual current (the X-ray limit).

*** Approximate lowest measurable pressure.

1 Torr = 133 Pa

References for XHV Gauges

Akimichi, H., Takeuchi, K., and Tuzi, Y., J.Vac.Sci.Technol.A, **17**, 753 (1999).

Benvenuti, C. and Hauer, M., Proc.8th International Vacuum Congress., vol 2, p.199 (Cannes, 1980).

Chen, J.Z., Suen, C.D., Kuo, Y.H., J.Vac.Sci.Technol.A, **5**, 2373 (1987).

Gotoh, N., Momose,T., Ishimaru,H., and Paitich, R., J.Vac.Sci.Technol.A, **13**, 2574 (1995).

Oshima, C. and Otuka, A., J.Vac.Sci.Technol.A, **12** , 3233 (1994).

Watanabe F., J.Vac.Sci.Technol. A, **9**, 2744 (1991).

Watanabe F., J.Vac.Sci.Technol. A, **10**, 3333 (1992).

Who Cares About XHV?

There is the “Reverse Everest Challenge”: How Low Can We Go?

There are applications that need XHV:

- Accelerators-you have heard about them at this Course

  -storage ring vacuum chambers to minimize beam-gas interactions and beam-vessel interactions

  -particle detectors- to minimize background

  -photoemission electron sources-to maximize lifetime
- Next Generation Gravity Wave Observatories

  -phase noise from residual hydrocarbons
- Electron Tubes, FE Displays, Materials Processing

  - probably no cost driver in these applications to go beyond UHV

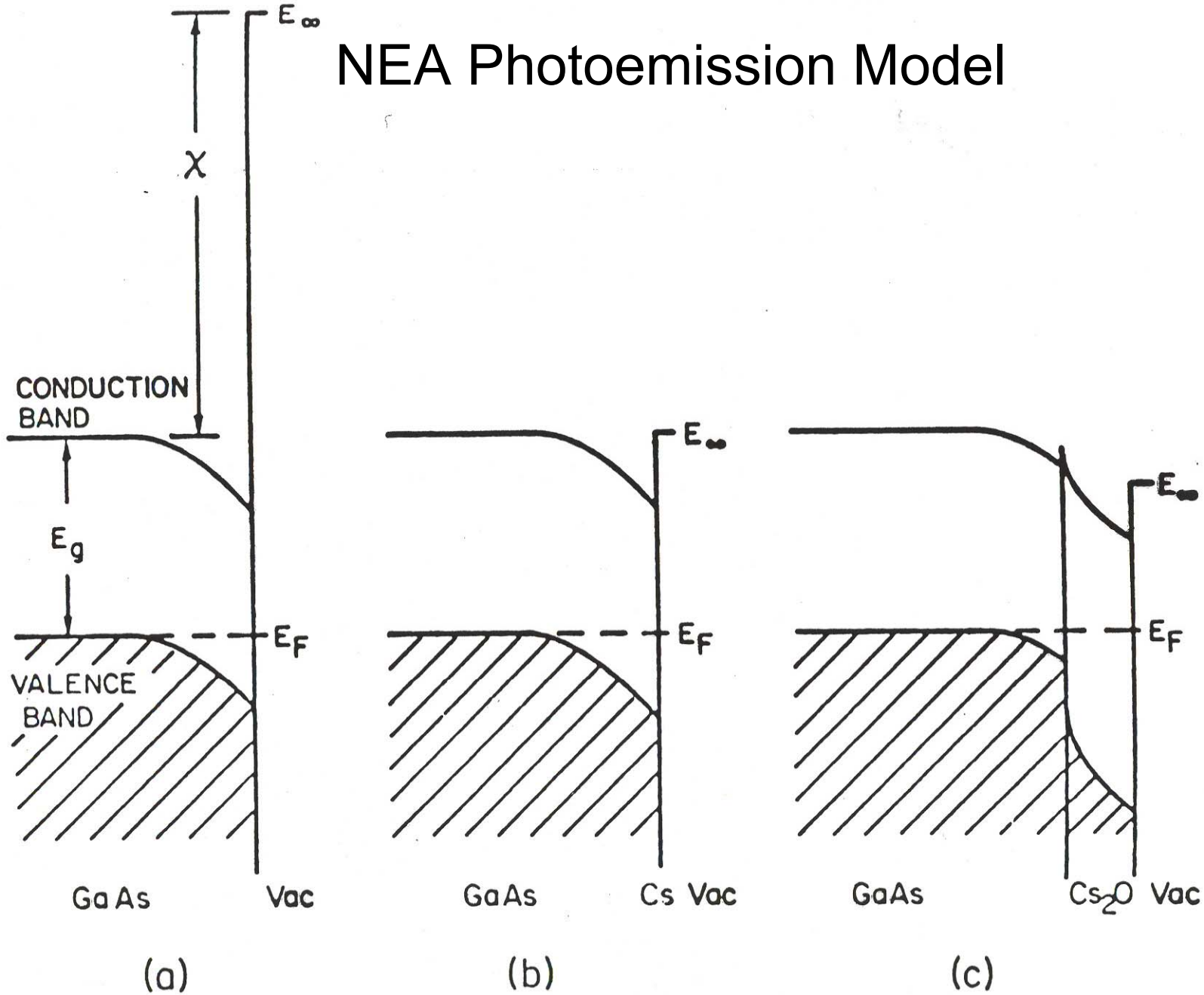
A Useful Application which Demands XHV: Photoemission Electron Guns

- Photoemission electron guns offer unique characteristics:
- Ease of temporal and spatial modulation to very high frequencies (10's of GHz)
- Production of very high peak current and current density (10's to 100's of A/cm^2)
- Very high beam brightness, due to low cathode temperature, high current density, and lack of a grid
- Production of longitudinally polarized electron beams from particular GaAs cathodes (75-80% polarization)

Current and Future Applications

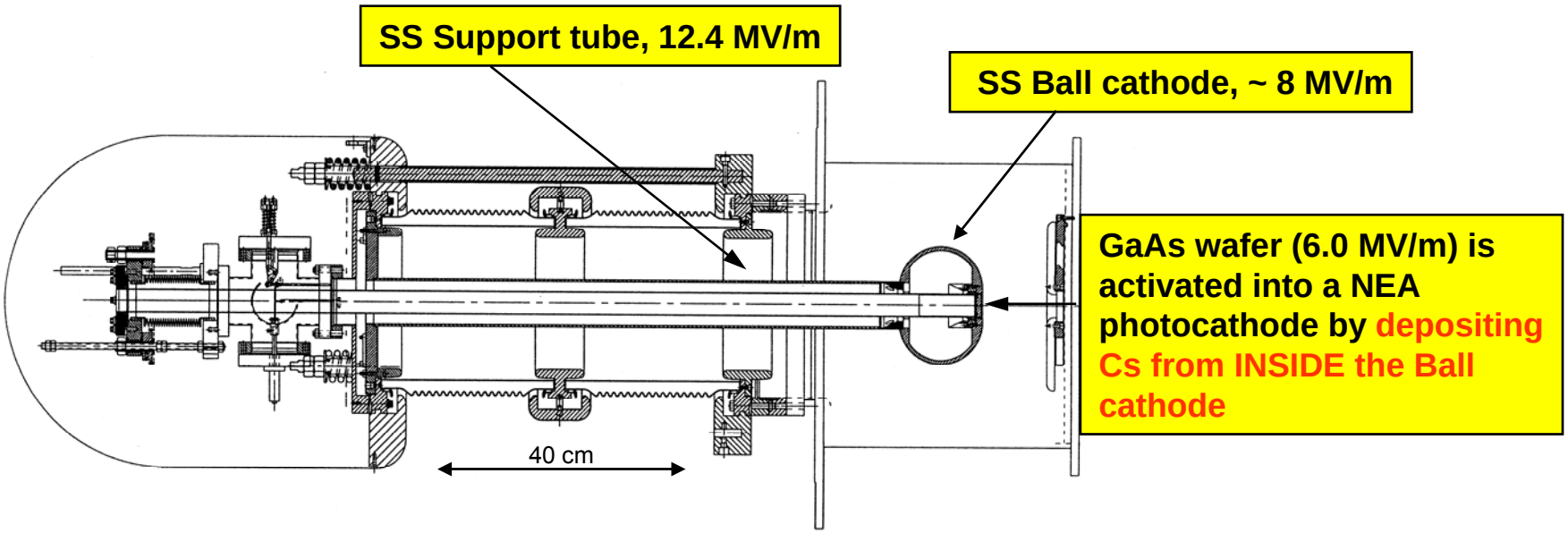
- CEBAF Accelerator – 200 μA average, 100 kV, polarized (1 mA in the future)
- JLab FEL – 9 mA average, 350 kV, unpolarized (10 mA at 500 kV in the future)
- Proposed for e-ion colliders – 170 mA, polarized
- Proposed for novel light sources – 100 mA, unpolarized
- Electron cooling applications – 1 A or more, unpolarized but very high brightness
- e- Beam Lithography – 100 nA, unpolarized, very high brightness and industrial reliability

NEA Photoemission Model



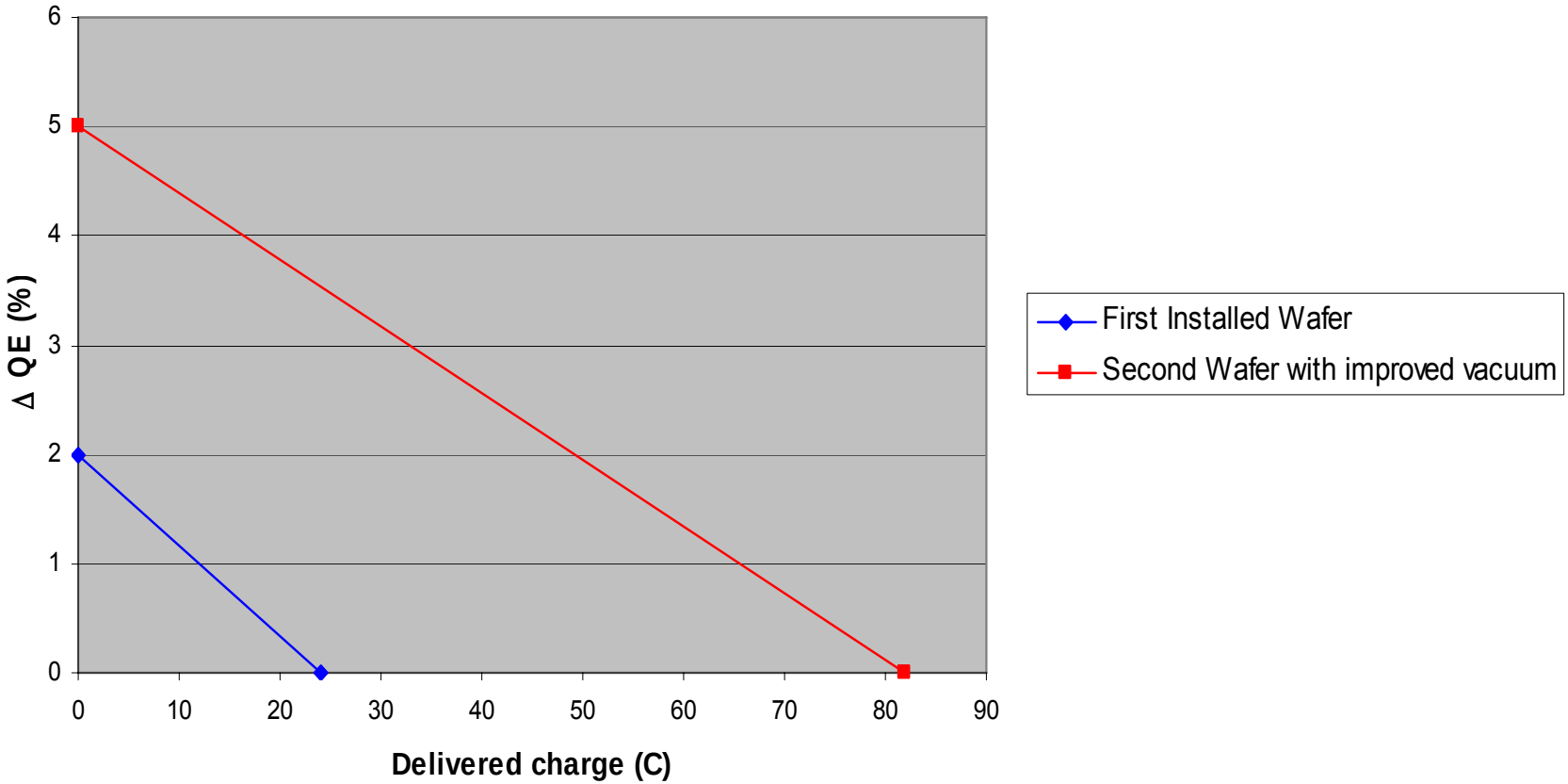
The 10 kW Upgrade IR FEL DC photocathode gun

Note: Field strengths calculated for 500 kV



- The photogun has been working very reliably since first operation on May 7, 2003
- Reproducible production of high QE (~5%) photocathodes with only in vacuo heat cleaning and re-cesiation
- Gun performance with Parmella modeling

FEL Photocathode performance

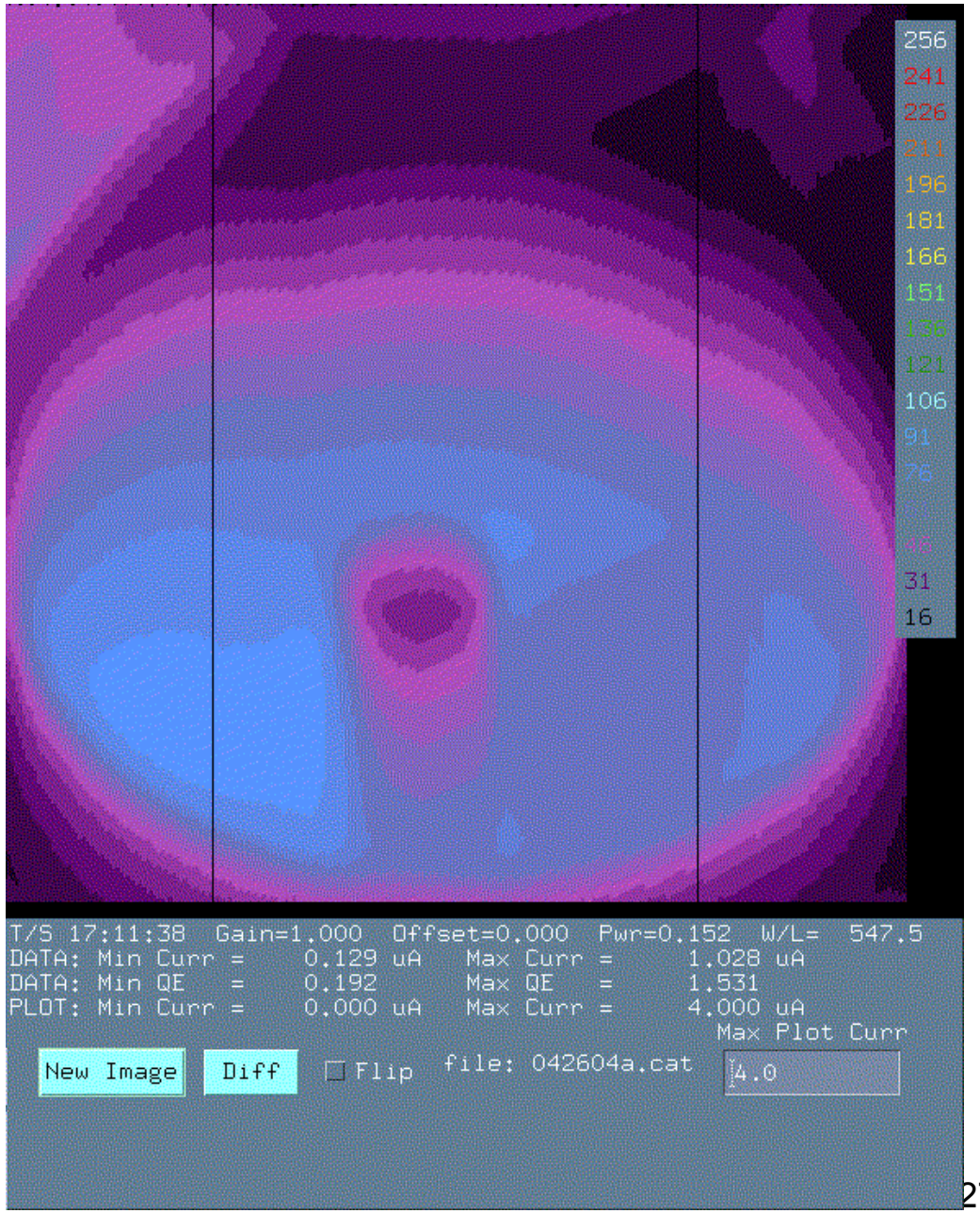


Photoemission Guns: Lifetime Limitations

- Emission lifetime (illumination and high voltage)
  - All electrons ionize residual gas molecules. The ions created in the cathode – anode gap are accelerated back to the cathode, where they cause QE degradation by a variety of mechanisms.
  - Some electrons may follow extreme trajectories and strike the system walls, desorbing gas. The desorbed gas may be a source of chemical poisoning or ion back bombardment damage to the cathode.

In the case where the entire photocathode is not uniformly illuminated, mapping the QE as a function of position can easily discriminate between ion back bombardment damage and chemical poisoning. (see next slide)

Cathode scan taken at the end of the usable QE (~1%).
The “hole” in the QE map is a result of back-ion bombardment.
(JLab FEL Data)



An Estimate – Hydrogen at 10^{-11} mbar

The ionization cross section for H_2 decreases approximately as $E^{-0.84}$ from ~ 100 eV to over 100 keV. If we assume that the electron gains energy linearly through a 5 cm cathode-anode gap, then the integral is easy, and gives about 3.7×10^{-12} ions/e^-, or 2.3×10^7 ions per coulomb.

In our present guns at JLab, which we believe to be at about 10^{-11} mbar of hydrogen, our 50,000 C/cm^2 gives a 1/e dose of about 1.2×10^{12} ions/cm^2.

Challenges to XHV

- It should be clear: to push the XHV frontier we have to be concerned with:
  - -XHV materials and the source of thermal and particle induced outgassing from these materials
  - -effective net pumping of these gases
  - -high sensitivity, non-contaminating gauging

- In dealing with all three of these development items understanding and controlling the source of the predominant residual gases

  are the critical problems:

  (H,CH_x and CO)
- The source for H_2 is obvious (major solute in all metals)
- The source of residual C,O is less obvious

  - ”accessible” impurities in near surface layers?

    C,CH,CH_x

    O,OH,MO_x

Challenges to XHV

Some thoughts on the source of residual gases in UHV/XHV systems:

Is the out-diffusion of hydrogen from the bulk a significant source term for the CH_4 and CO seen in XHV systems at 10^{-2} of the H_2 pressure?

Is H a carrier or recombinant in the passivation oxide layer to form or release these more complex species?

$H \rightarrow CH \rightarrow CH_4$

$H \rightarrow OH \rightarrow H_2O$

Challenges to XHV

Two intriguing observations:

1.) Residual surface concentrations of C and O remain quasi-static on stainless steel surfaces (~10 at.%) after high fluence H^+ bombardment

[eg, K.G.Tschersich and J.von Seggern,Proc.5th Intern. Symp. Plasma Chemistry, Ed. B.Waldie,Edinburgh (1988)]

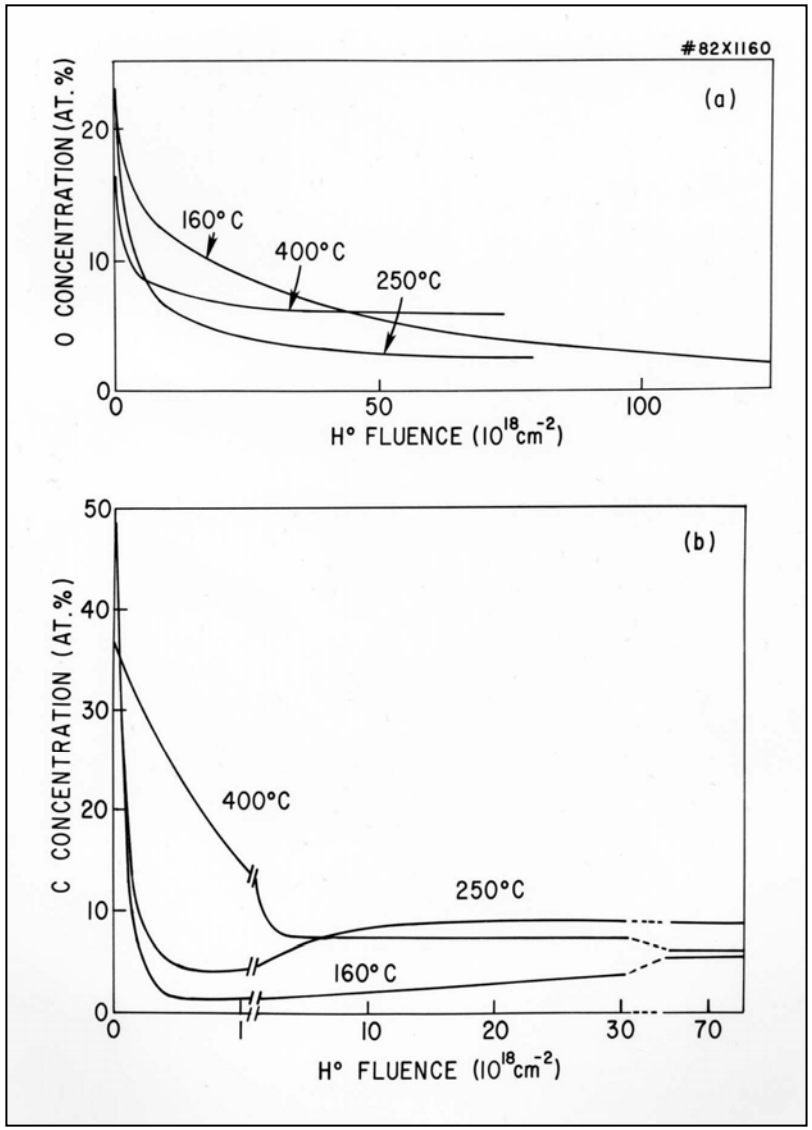
2.) H^+ (H^o) bombardment of "clean" metal (SS) surfaces yields CH_4 and CO desorption products (

[eg, H.F.Dylla, J.Vac.Sci.Technol.A6,1266(1988)]

Reaction mechanisms are complicated:

$$H\,(MO) \rightarrow CH \rightarrow CH_2 \rightarrow CH_3 \rightarrow CH_4$$

$$H\,(MO) \rightarrow OH \rightarrow H_2O$$


#82X1160
(a)
O CONCENTRATION (AT.%)
20
10
0
160° C
400° C
250°C
0
50
100
H° FLUENCE (10^{18}cm^{-2})
(b)
C CONCENTRATION (AT.%)
50
40
30
20
10
0
400°C
250°C
160° C
0
1
10
20
30
70
H° FLUENCE (10^{18}cm^{-2})

Challenges to XHV

Model* for Quasi-Static Residual Surface Concentrations (n_s) of C,O

What values of diffusion constants for C (O) are necessary to account for observed steady-state surface concentrations if the ion (or thermal atom) induced desorption rates are balanced by near-surface diffusion?

$$\frac{dn_s}{dt} = F_g + F_d + F_n$$

where F_g = adsorption flux

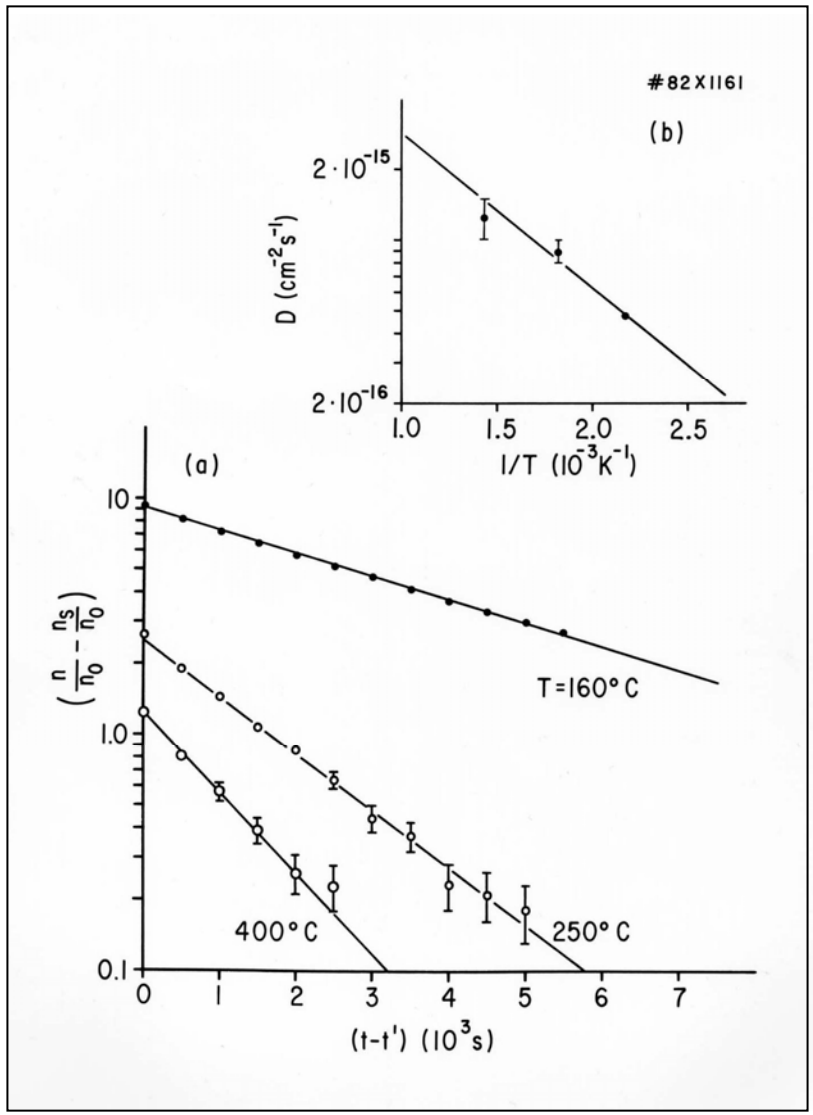
F_d = diffusive flux

F_n = desorption flux

For oxygen D~ 0.4-1.2 x 10^{-15} $cm^{-2}s^{-1}$

- consistent with literature values for pure transition metals
- grain boundary or pore diffusion would have larger D-values

*Ref: Dylla and Blanchard, J.Vac.Sc.Technol. A1,1297 (1983)


#82X1161
(b)
$2 \cdot 10^{-15}$
$2 \cdot 10^{-16}$
D ($cm^{-2}s^{-1}$)
1.0
1.5
2.0
2.5
1/T ($10^{-3}K^{-1}$)
(a)
10
1.0
0.1
$\left(\frac{n}{n_0} - \frac{n_s}{n_0}\right)$
T=160° C
400° C
250° C
0
1
2
3
4
5
6
7
(t-t') (10^3s)

Challenges to XHV

What would be the “ideal” conditioned surface for XHV:

If the passivation oxide layer were completely removed,

OR

If the surface had a perfect gas barrier,

OR

If the surface inhibited H (and H-C) recombination,

Would the resulting surface be ideally conditioned? ie, minimal gas release under thermal excursions or particle bombardment?

Challenges to XHV

The other source of residual C and O in XHV systems:

- ion pumps are known generators of CH_4 and CO
  (ion and e-impact surface reactions and Penning discharge reactions)
- so are all e-impact ion sources in ion gauges and RGA's
  (ion and e-impact surface reactions, esp. on hot surfaces)

- if small conversion rates exist for H to heavier gases in the passivation oxide layers of SS,CU and Al, then the process is no doubt at work at some level with the active metal interfaces on getter surfaces

Challenges to XHV

The physics behind the existing XHV demonstrations:

- chamber at LHe or LH_2 temperature
  - perfect pump in limited circumstances (no high gas loading and no radiation/particle bombardment)
  - surface diffusion and surface chemistry is frozen

- chamber coated with an active metal
  - perfect pump for active gases (within limits)
  - low initial C,O concentrations in near-surface region and relatively high surface recombination rates

Challenges to XHV: the Future

Further development of pumped surfaces:

LHe and active metal

Development of non-interacting XHV gauges:

- laser based?
- surface monitors?

References

C.Benvenuti, “Production and Measurement of Extreme High Vacua”
Proc.7th Int.Vac.Congr.p.1,Vienna(1977)

J.P.Hopson,”Methods of Producing Ultralow Vacuums and Measuring Ultralow Pressures,Surf. and Coll.Sci.,11,187(1979)

C.Benvenuti, “Extreme Vacua: Achievements and Expectations”
Physica Scripta, T22,48 (1988)

P.A. Redhead, “Ultrahigh and Extreme High Vacuum” in “Foundations of Vacuum Science and Technology”,p.625,Ed. J.M.Lafferty, Wiley,NY (1998)