Extreme High Vacuum: The Need, Production, and Measurement

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Thomas Jefferson National Accelerator Facility (Jefferson Lab)

Polarized Electron Gun Group
Newport News, Virginia
Run by JSA for the US DOE
What is XHV

- Extreme High Vacuum
  \[ P < 1 \times 10^{-10} \text{ Pa} = 1 \times 10^{-12} \text{ mbar} = 7.5 \times 10^{-13} \text{ Torr} \]
- Baked, metal systems, low outgassing, coatings to reduce outgassing
- Combinations of pumping
  - Ion, Getter, Cryo, Titanium Sublimation, Turbo
- Measurement: Ionization gauges
Ultimate Vacuum

- Steady decrease interrupted by gauge limitations 1920-1950
- Bayard-Alpert gauge introduced in 1950
- Plateau $\sim 1 \times 10^{-14}$ Torr for nearly 3 decades again

![Graph showing the history of ultimate vacuum with key points and dates](image-url)
Who needs XHV

- **Storage Rings**
  - CERN ISR:
    - beam lifetimes > 10 hours, pressure < $1 \times 10^{-12}$ Torr
    - Vacuum in interaction region in the $10^{-14}$ Torr range
- **Large Detector Systems**
  - KATRIN (later in this session)
- **Surface Science applications**
  - Alkali metals on surfaces
    - surface contaminates within ~1 hour
  - Surface X-ray diffraction at synchrotrons, He scattering
    - low signal, long collection times
  - Dynamical surface analysis
- **High current polarized photo-electron guns**
Jefferson Lab

- CEBAF: Nuclear physics electron accelerator laboratory and Free Electron Laser (FEL)
- User community of 2000+ physicists
- GaAs Photoelectron gun (100 kV, 200 µA, 85% polarization) delivers beam simultaneously to three experimental halls
- Nuclear physics gun on up to 310 days/year, 24 hours/day
- CEBAF pressures ~1.2x10^{-11} Torr
- Guns pumped with combination of NEG and ion pumps
- FEL gun operates 350 kV, 9 mA unpolarized electron gun
Photocathode Lifetime

- Quantum Efficiency (yield) of GaAs photocathode decays
- Lifetime of inversely proportional to vacuum conditions
  - Residual gas ionized
  - Ion backbombardment damages
    - Crystal structure
    - Surface chemistry
- Lifetime very good: ~200 Coulombs, 85% polarization
- Future applications: higher currents
  - Electron/ion colliders: >1 mA polarized
  - Novel light sources: 100 mA unpolarized
  - Electron cooling applications: 1 A+, unpolarized
  - RF photoguns – GaAs photocathodes

Laser spots

~2.5E-11 Torr
~5.0E-11 Torr
>15.0E-11 Torr
Materials and Preparation

• Low outgassing
  – Stainless Steel
  – Titanium alloys
  – Aluminum
  – OFHC Copper, Cu/Be alloys
• 300 series austenitic steels (304L, 316L, 316LN)
  – low carbon, 316 series adds Mo for strength
• Coatings to reduce outgassing
• Coatings to add pumping
Hydrogen reduction through heating

Calder & Lewin 1967 calculate time and temperature to reduce stainless steel outgassing

Fick’s law governs diffusion of hydrogen from bulk metal

- Initial concentration
- Time
- Temperature
- Wall thickness
- Surface recombination

<table>
<thead>
<tr>
<th>$t$ (sec)</th>
<th>$D$ ($\text{cm}^2 \text{ sec}^{-1}$)</th>
<th>$T$ ($^\circ\text{C}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$1 \cdot 0 \times 10^6$ (11 days)</td>
<td>$3 \cdot 5 \times 10^{-6}$</td>
<td>300</td>
</tr>
<tr>
<td>$8 \cdot 6 \times 10^4$ (24 hours)</td>
<td>$3 \cdot 8 \times 10^{-7}$</td>
<td>420</td>
</tr>
<tr>
<td>$1 \cdot 1 \times 10^4$ (3 hours)</td>
<td>$3 \cdot 0 \times 10^{-6}$</td>
<td>570</td>
</tr>
<tr>
<td>$3 \cdot 6 \times 10^3$ (1 hour)</td>
<td>$9 \cdot 0 \times 10^{-6}$</td>
<td>635</td>
</tr>
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</table>
## Outgassing Rates for SS

### Table 1
Some published data of outgassing rates $q_{out}$ of stainless steel chamber walls after different pre- and in situ treatments.

<table>
<thead>
<tr>
<th>Preprocessing</th>
<th>Processing in situ</th>
<th>$T$ (°C)</th>
<th>$t_0$ (h)</th>
<th>$F_0$</th>
<th>$T$ (°C)</th>
<th>$t_0$ (h)</th>
<th>$F_0$</th>
<th>$\Sigma F_0$</th>
<th>$q_{out}$ (mbar·L/s·cm$^2$)</th>
<th>Refs.</th>
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</thead>
<tbody>
<tr>
<td>950</td>
<td>2</td>
<td>39.6</td>
<td>150</td>
<td>168</td>
<td>0.12</td>
<td>39.7</td>
<td>2.5 x 10$^{-14}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>400 (air)</td>
<td>38</td>
<td>9</td>
<td>150</td>
<td>168</td>
<td>0.12</td>
<td>9.1</td>
<td>1.1 x 10$^{-14}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>390</td>
<td>100 (air)</td>
<td>3.3</td>
<td>150</td>
<td>24$^a$</td>
<td>0.03</td>
<td>0.03</td>
<td>3 x 10$^{-12}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>48</td>
<td>0.1</td>
<td>150</td>
<td>24</td>
<td>0.03</td>
<td>4.0</td>
<td>1.1 x 10$^{-14}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>950</td>
<td>2</td>
<td>43</td>
<td>200</td>
<td>48</td>
<td>0.1</td>
<td>0.1</td>
<td>4 x 10$^{-12}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>400 (air)</td>
<td>72</td>
<td>7.7</td>
<td>250</td>
<td>72</td>
<td>0.46</td>
<td>0.46</td>
<td>3.8 x 10$^{-12}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>550</td>
<td>72</td>
<td>46</td>
<td>250</td>
<td>72</td>
<td>0.46</td>
<td>46.5</td>
<td>4 x 10$^{-15}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>404</td>
<td>1.4</td>
<td>70</td>
<td>200</td>
<td>72</td>
<td>3</td>
<td>70</td>
<td>3 x 10$^{-16}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>72</td>
<td>3</td>
<td>200</td>
<td>72</td>
<td>3</td>
<td>70</td>
<td>1 x 10$^{-13}$</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$^a$ Estimated, since the exact in situ bake-out time was not specified.

Meaning of the columns: temperature ($T$) and duration ($t_0$) of pre- and in situ treatments and outgassing rate ($F_0$) of the wall material.

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**Other exceptional outgassing rates (in Torr·L/s·cm$^2$)**

- **BeCu alloy:** $4 \times 10^{-16}$  

- **Ti/steel alloy:** $7.5 \times 10^{-15}$  

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P. Marin Virgo, Vacuum 1998

M. Bernardini Virgo, JVSTA 1998

H. Hseuh Brookhaven JVSTA 1998

G. Messer, 1977

V. Nemanič thin walls JVSTA 1999

V. Nemanič JVSTA 2000
JLab Preparation

- 304 SS vacuum chambers
  - Untreated
  - Electropolished and vacuum fired 900°C 4 hours
- Baking
  - 30 hours, 250°C
  - Unfired chamber
    1x10⁻¹² Torr·L/s·cm²
    ~13 bakes
  - Vacuum fired chamber
    8.9x10⁻¹³ Torr·L/s·cm²
    3 bakes

Achieve modest outgassing rate for 304SS
Lower rates possible with better grade steel
Add heat treatment after final welding
XHV surface coatings

TiN, SiO₂, Chromium oxide
Diffusion barrier for hydrogen
Affect surface recombination
Can also reduce beam induced pressure rise in storage rings
See session VT-WeM

<table>
<thead>
<tr>
<th>Chamber #</th>
<th>Q (Torr·cm⁻³)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiN</td>
<td></td>
<td></td>
</tr>
<tr>
<td>#2A</td>
<td>2.5E-13</td>
<td>in-situ 250°C bake, without TiN coating</td>
</tr>
<tr>
<td></td>
<td>(120 hours, post-bake)</td>
<td></td>
</tr>
<tr>
<td>#3A</td>
<td>2.1E-13</td>
<td>in-situ 250°C bake, with high pressure TiN coating</td>
</tr>
<tr>
<td></td>
<td>(96 hours, post-bake)</td>
<td></td>
</tr>
<tr>
<td>#5B</td>
<td>1.9E-13</td>
<td>in-situ 250°C bake, with low pressure TiN coating</td>
</tr>
<tr>
<td></td>
<td>(72 hours, post-bake)</td>
<td></td>
</tr>
</tbody>
</table>

P. He, H.C. Hseuh, M. Mapes, R. Todd, N. Hilleret
Outgassing for SNS ring material with and without TiN coatings

K. Saito et al
JVSTA 13 (1995) 556
SiO$_2$ Coatings

- SiO$_2$ coated 304 SS (Restek prototype)
- SiO$_2$ coating applied to inside and outside, chemically stripped
- Accumulation method with spinning rotor gauge
- Outgassing no better with SiO$_2$ coating
  - Prototype coatings
  - Chemical stripping process
  - Increased surface roughness

Outgassing: SiO$_2$ coated 304 SS

<table>
<thead>
<tr>
<th></th>
<th>Thin</th>
<th>Thick</th>
</tr>
</thead>
<tbody>
<tr>
<td>in/out</td>
<td>1.0E-12</td>
<td>2.0E-12</td>
</tr>
<tr>
<td></td>
<td>1.5E-12</td>
<td>2.5E-12</td>
</tr>
</tbody>
</table>

JLab: Y. Prilepskiy, G.R. Myneni, P.A. Adderley, M.L. Stutzman
Cr$_2$O$_3$ Surface passivation

304L Surface passivation
Vacuum fire 450°C, 24 hours
1x10$^{-9}$ Torr O$_2$ partial pressure
5x10$^{-7}$ Torr total pressure

Cr$_2$O$_3$ is one component of air fired, low outgassing materials (VIRGO, LIGO)

K.R. Kim et al
Proceedings of APAC
2004 Gyeongju, Korea
Distributed beamline pumping

- Beamlines coated with getter material (Ti/Zr/V)
  - activated through bakeout temperature ~200°C
  - No conductance limitation
  - Reduces beam induced pressure rise

Distributed Ion pump: Y.Li et al., JVSTA 15 (1997) 2493.

ESRF insertion device

RIKEN from SAES literature
JLab’s NEG coating

- Ti/Zr/V NEG coating
- Sputtering without magnetron enhancement
- Beamline exiting CEBAF electron guns NEG coated since 1999
  - Enhanced photocathode lifetime: now achieving lifetime ~200 Coulombs
- High voltage chamber for new load locked gun coated

EDS analysis of getter coating composition

25% Ti
50% V
25% Zr
Load Locked Electron Gun

NEG coated HV chamber
Vacuum measured: $\sim 1.2 \times 10^{-11}$ Torr
Lifetime doubled
5-10 mA, 100 keV electron beam
Pumps for XHV

- **Ion pumps**
  - Ion pump performance vs. voltage
  - Ion pump current monitor at UHV pressures
  - Getter coating ion pumps

- **NEG**
  - Great pumping for hydrogen, also pumps CO, N₂
  - Don’t pump methane, noble gasses
  - Question about pump speed at base pressure

- **Ti Sublimation**

- **Cryo pumps**

- **Turbo pumps – cascaded pumps**
Ion pump limitations

Ion pump speed decreases at lower pressures
- Lower nA/Torr at lower pressures
- Re-emission of gasses
- Outgassing from pump body

Adding NEG pumping to ion pumping decreases hydrogen
Pd coated NEG films on inside of ion pumps reduced ultimate pressure to $2-6 \times 10^{-11}$ mbar
JLab UHV ion pump current monitoring

- Ion pumps current varies linearly with pressure as low as $1 \times 10^{-11}$ Torr
- Real time monitoring of UHV vacuum
- Studying optimal voltage for pumping at low pressures

~$10^{-10}$ Torr
Full Scale
Discharge event in beamline
Base pressure in CEBAF guns

• Why isn’t our chamber pressure as low as calculated?
  – Is outgassing much higher?
  – Is pump speed much lower?
  – Are we unable to measure lower pressures?
• First measured outgassing rate from chamber
  – $1 \times 10^{-12}$ Torr·L/s·cm$^2$
  – Typical value for baked 304SS

Measured and predicted pressure for 304 SS chambers and ST707 SAES getter modules

Log Pressure (Torr)

Getter Surface area (m$^2$)

Test chambers

CEBAF guns
Pump speed measurements

- Measured pump speed vs. pressure from base pressure of chamber to $2 \times 10^{-10}$ Torr
- Throughput method
  - conductance limiting orifice
  - RGAs to measure $H_2$ pressure
- Ultimate pressure method
  - Gas sources: outgassing from walls and gauge
  - Measure with extractor gauge
- Found very good pump speed at higher pressures
  - 500 L/s with bakeout
  - 1150 L/s activated (430 L/s quoted)
- Found drop in pump speed as function of pressure: WHY?

\[
S = \frac{C \left( P_{orf} - P_{orf}' \right) - \left( P_{main} - P_{main}' \right)}{P_{main}} + Q_{wall} + Q_{gauge}
\]
Alternate analysis of pump speed measurement

- $Q = S \times P$
- Plot $Q$ instead of $S$ vs. $P$
- Linear fit indicates constant pump speed throughout range
- Discrepancy:
  - Problem with throughput vs. pump speed at low pressures?
  - Problem with accurately measuring low pressures?

![Graph showing throughput vs. pressure with linear fit indicating pump speed]

Slope = Pump Speed = 1150 L/s

Throughput Ultimate

M. Stutzman et al. submitted to NIM 2006
XHV Pressure Measurement

- Ionization Gauges
  - Hot Cathode: Extractor, Improved Helmer, Axtran, Modulated BA, spectroscopy and bent beam gauges
  - Cold Cathode: Magnetron, inverted magnetron, double inverted magnetron
  - Laser ionization gauges
- X-ray limits
- Electron stimulated desorption limits
- Gauge outgassing
X-ray limit

Ionized gas molecules collected, proportional to gas pressure

Electrons strike grid, generate x-rays
X-rays striking collector photoemit

Collector current is sum of ionized gas and photoemitted electrons

Bayard-Alpert gauge 1950’s led to UHV measurements
• smaller collector
• modulation techniques
Extractor gauge geometry reduces measurement limits to ~XHV range
Improved Helmer gauge, Watanabe gauges optimize geometry
Extractor Gauge X-ray limits


<table>
<thead>
<tr>
<th>Gauge</th>
<th>X-ray Limit (Torr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Watanabe A</td>
<td>2.1 x 10^{-12}</td>
</tr>
<tr>
<td>Watanabe B</td>
<td>1.6 x 10^{-12}</td>
</tr>
<tr>
<td>Watanabe C</td>
<td>1.9 x 10^{-12}</td>
</tr>
<tr>
<td>JLab A</td>
<td>0.63 x 10^{-12}</td>
</tr>
<tr>
<td>JLab Gun 2</td>
<td>&gt;2 x 10^{-12}</td>
</tr>
<tr>
<td>JLab Gun 3</td>
<td>&gt;2 x 10^{-12}</td>
</tr>
</tbody>
</table>
Extractor gauge comparison

Three extractor gauges
Factor of 8 difference in readings
  • Identical ports
  • Symmetric positions
  • Multiple degas cycles

Divergence in pressure readings below $5 \times 10^{-11}$ Torr
Electron Stimulated Desorption

- ESD ions
  - Have energy higher than gas phase
  - Energy discrimination
- ESD neutrals
  - Same energy as gas phase
- Hotter grid: less adsorbed gas
  - Electron bombardment
    - More outgassing
  - Resistive heating
    - ESD and outgassing decoupled
- Watanabe heated grid gauges: total pressure and residual gas analyzer
  - BeCu walls
    - Low emissivity
    - High thermal conductivity
  - Cold cathode
    - Decouple grid temperature from filament

Ref: Fumio Watanabe
JVSTA 17 (1999) 3467,
Gauge solutions

• Extractor commercially available
  – X-ray limits can be in the $10^{-13}$ Torr range (barely XHV)
  – Reasonable residual current caused by ESD due to geometry
  – Work needed to ensure accuracy over time, between gauges

• Improved Helmer gauge used at CERN
  – Frequent pressure measurements in $10^{-14}$ Torr range quoted

• Watanabe proposes heated filament gauges
  – Separate ESD, outgassing problems

• Laser ionization gauge
  – Ionize gas with powerful laser, count ions: direct gauge of low pressures

Calibration techniques
Calibration Techniques

- Careful calibration needed for measurements below $5 \times 10^{-11}$ Torr
- Cross calibration with transfer standards
- Dynamic or static expansion methods
  - Relatively complex systems
  - Not common in gauge user laboratories
- Reported XHV pressure measurements should make note of the calibration method

C. Meinke and G. Reich JVST 6 (1967) 356.
Future work at JLab

- Get best available material
- Polish, vacuum fire after welding
- Optimize and calibrate extractor gauges, or
- Replace extractor gauges with better XHV gauge
- UHV ion pump supplies
  - Optimize voltage, geometry for pressure
  - Investigate NEG coatings in ion pumps
- Use cathode lifetime as a relative gauge
- Gauge exchange / cross calibration at different facilities
Future of XHV

- Gauging issues are coming along, but still an art, calibration critical
- Materials exist – many different “recipes” to get very good outgassing rates
- NEG, TiN coatings becoming widespread
- Pumping technologies
  - existing technologies can achieve XHV
  - room for improvement and study
- When XHV becomes routine, high current electron guns, surface science, accelerators, semiconductor industry, and others will benefit