Extreme High Vacuum: The Need, Production, and Measurement

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

What is XHV

- Extreme High Vacuum

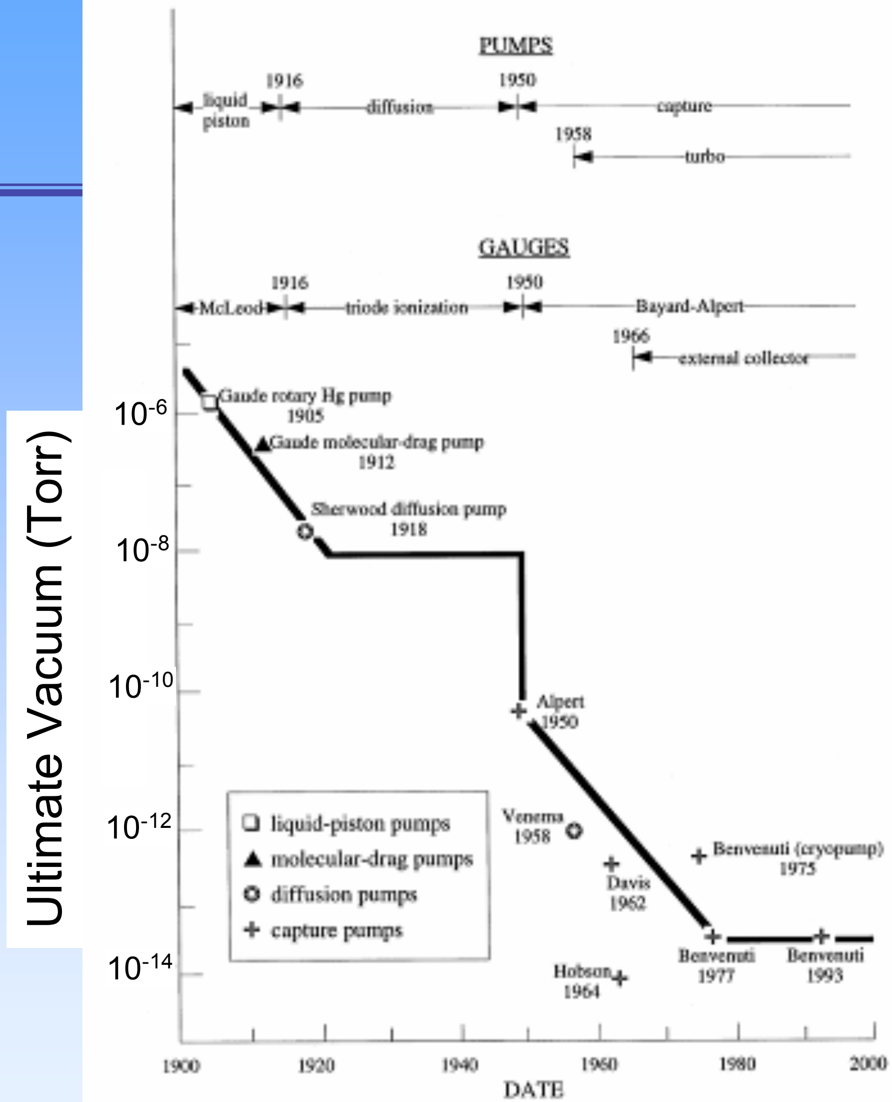
  $P < 1x10^{-10}$ Pa = $1x10^{-12}$ mbar = $7.5x10^{-13}$ Torr
- Baked, metal systems, low outgassing, coatings to reduce outgassing
- Combinations of pumping

  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 ~1×10^{-14} Torr for nearly 3 decades again

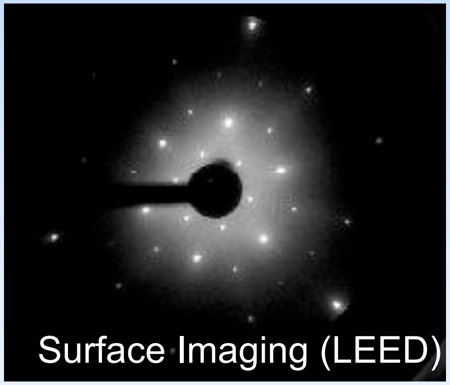
P.A. Redhead Vacuum 53 (1999) 137



Who needs XHV

- Storage Rings
  - CERN ISR:
    - beam lifetimes > 10 hours, pressure < 1×10^{-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

CERN aerial view



Surface Imaging (LEED)

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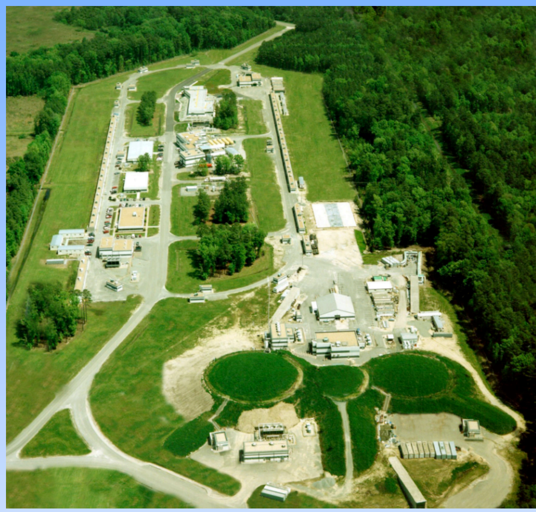
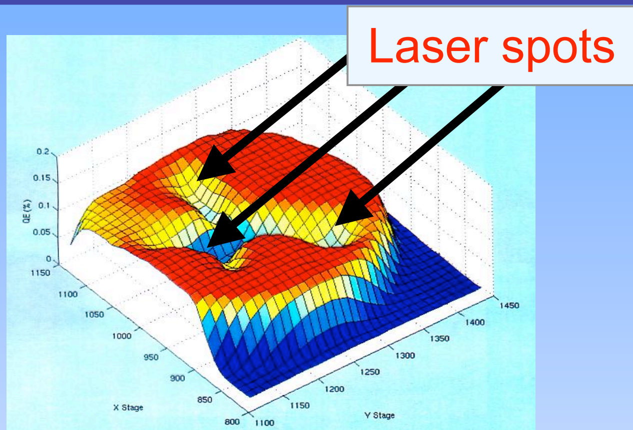
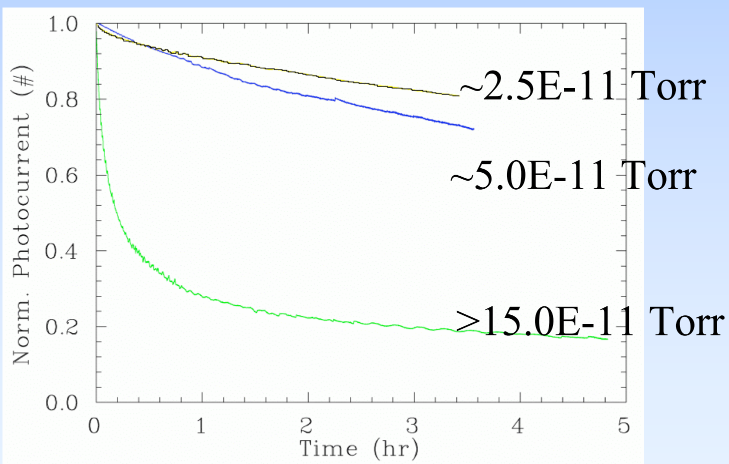


- CEBAF: Nuclear physics electron accelerator laboratory and Free Electron Laser (FEL)
- User community of 2000+ physicists
- GaAs Photoelectron gun (100 kV, 200 μA, 85% polarization) delivers beam simultaneously to three experimental halls
- Nuclear physics gun on up to 310 days/year, 24 hours/day
- CEBAF pressures ~1.2×10^{-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

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

Table 1. Bakeout times of 2 mm thick sheet at various temperatures for $\dot{Q}_r = 10^{-16}$ torr l. cm^{-2} sec^{-1}

t (sec)	D (cm^2 sec^{-1})	T (°c)
$1{\cdot}0 \times 10^6$ (11 days)	$3{\cdot}5 \times 10^{-8}$	300
$8{\cdot}6 \times 10^4$ (24 hours)	$3{\cdot}8 \times 10^{-7}$	420
$1{\cdot}1 \times 10^4$ (3 hours)	$3{\cdot}0 \times 10^{-6}$	570
$3{\cdot}6 \times 10^3$ (1 hour)	$9{\cdot}0 \times 10^{-6}$	635

Fick's law governs diffusion of hydrogen from bulk metal

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

Outgassing Rates for SS

Q = $10^{-12} – 10^{-16}$ mbar·L/s·cm^2

Table 1
Some published data of outgassing rates q_{out} of stainless steel chamber walls after differer

Y. Ishikawa, V. Nemanič Vacuum 69 (2003) 501

Preprocessing			Processing in situ					
T (°C)	t_0 (h)	F_0	T (°C)	t_0 (h)	F_0	ΣF_0	q_{out} (mbar l s^{-1}cm^{-2})	Refs.
950	2	39.6	150	168	0.12	39.7	2.5×10^{-14}	P. Marin Virgo, Vacuum1998
400	38 (air)	9	150	168	0.12	9.1	1.1×10^{-14}	
			150	24[a]	0.03	0.03	3×10^{-12}	M. Bernardini Virgo, JVSTA 1998
390	100 (air)	3.3	150	24[a]	0.03	3.33	5×10^{-15}	
			200	48	0.1	0.1	4×10^{-12}	H. Hseuh Brookhaven JVSTA 1998
950	2	43	200	48	0.1	43.1	4×10^{-13}	
			250	72	0.46	0.46	3.8×10^{-12}	G. Messer, 1977
400	72	7.7	250	72	0.46	8.16	4×10^{-15}	
550	72	46	250	72	0.46	46.5	1×10^{-15}	
			404	1.4	70	70	3×10^{-16}	V. Nemanič thin walls JVSTA 1999
			200	72	3	3	1×10^{-13}	V. Nemanič JVSTA 2000

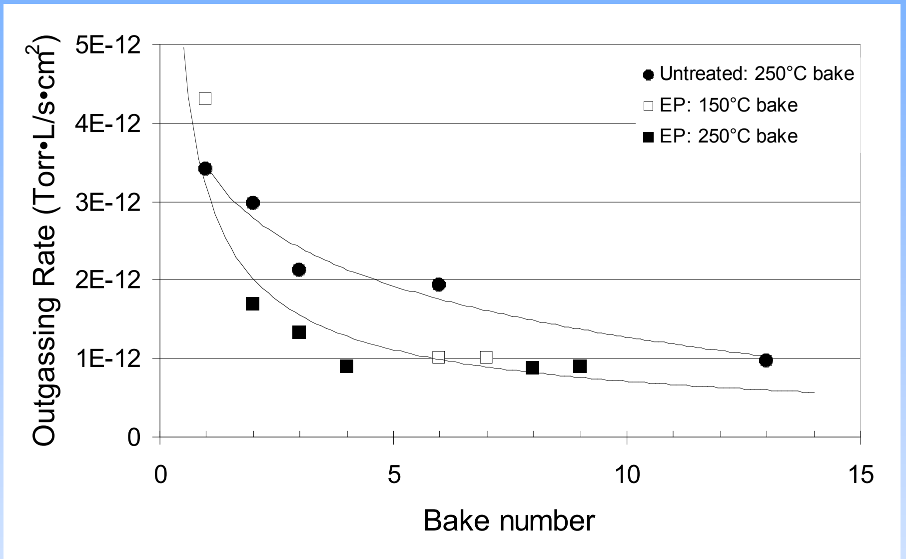
[a] Estimated, since the exact in situ bake-out time was not specified.
Meaning of the columns: temperature (T) a
pre-treatment, temperature (T) and durati
treatment.

Other exceptional outgassing rates (in Torr·L/s·cm^2)
BeCu alloy: 4×10^{-16} F. Watanabe JVSTA 22 (2004) 181, 22(2004) 739.
Ti/steel alloy: 7.5×10^{-15} H. Kurisu et al. JVSTA 21 (2003) L10.

JLab: 1×10^{-12} Torr·L/s·cm^2

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·cm^2 ~13 bakes
  - Vacuum fired chamber $8.9x10^{-13}$ Torr·L/s·cm^2 3 bakes



M.L. Stutzman *et al.* submitted to NIM 2006

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

XHV surface coatings

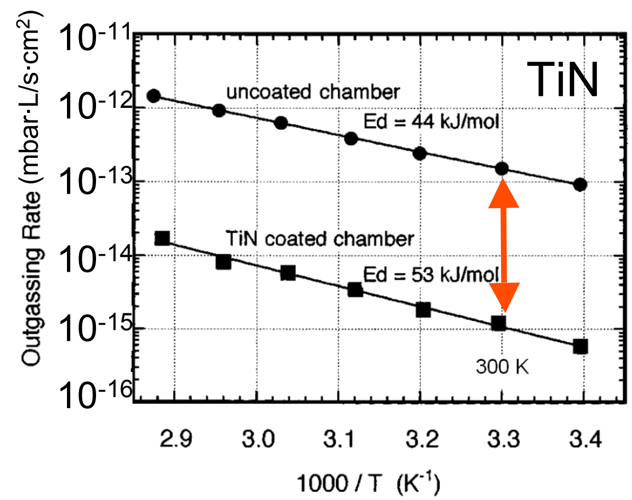
- TiN, SiO_2, Chromium oxide
- Diffusion barrier for hydrogen
- Affect surface recombination
- Can also reduce beam induced pressure rise in storage rings
- See session VT-WeM

TABLE 2. Hydrogen Outgassing Rates for SNS Vacuum Chambers

TiN

Chamber #	Q (Torr-l/s cm^2)	Comments
#2A	2.5E-13 (120 hours, post-bake)	*in-situ* 250°C bake, without TiN coating
#3A	2.1E-13 (96 hours, post-bake)	*in-situ* 250°C bake, with high pressure TiN coating
#5B	1.9E-13 (72 hours, post-bake)	*in-situ* 250°C bake, with low pressure TiN coating

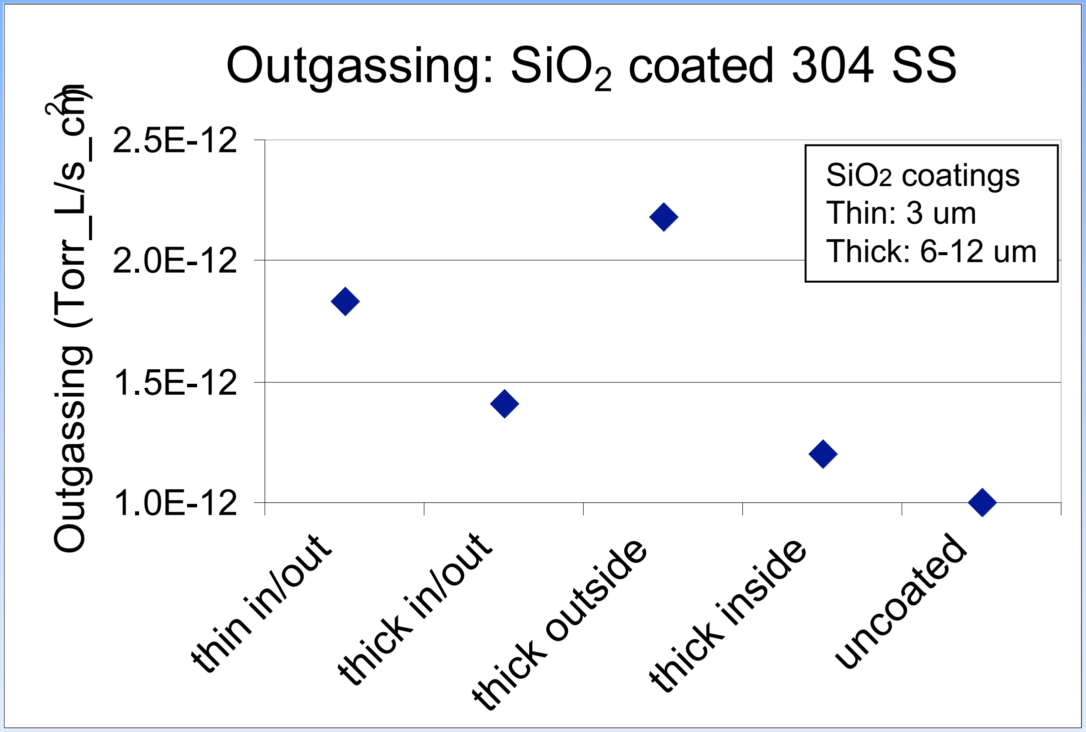
P.He, H.C.Hseuh, M.Mapes, R.Todd, N.Hilleret
Outgassing for SNS ring material with and without TiN coatings



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

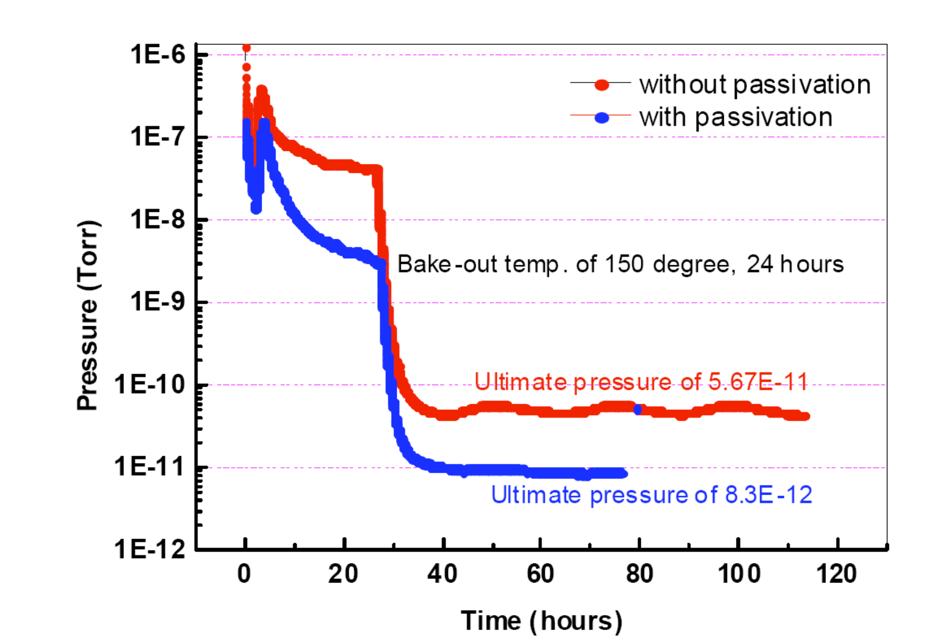


JLab: Y. Prilepskiy, G.R.Myneni, P.A. Adderley, M.L.Stutzman

Cr_2O_3 Surface passivation

304L Surface passivation
Vacuum fire 450°C, 24 hours
1×10^{-9} Torr O_2 partial pressure
5×10^{-7} Torr total pressure

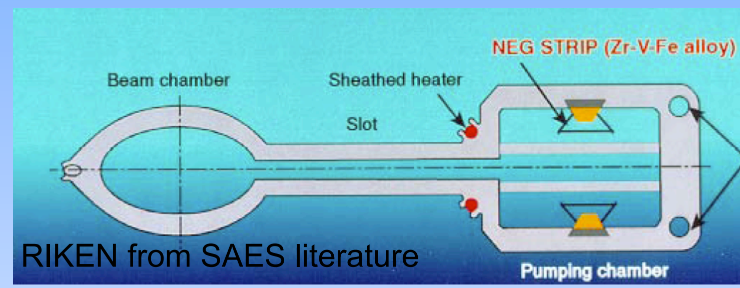
Cr_2O_3 is one component of air fired, low outgassing materials (VIRGO, LIGO)



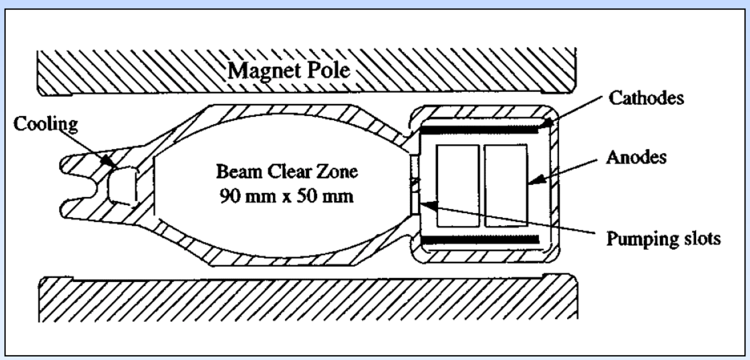
K.R. Kim et al
Proceedings of APAC
2004 Gyeongju, Korea

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

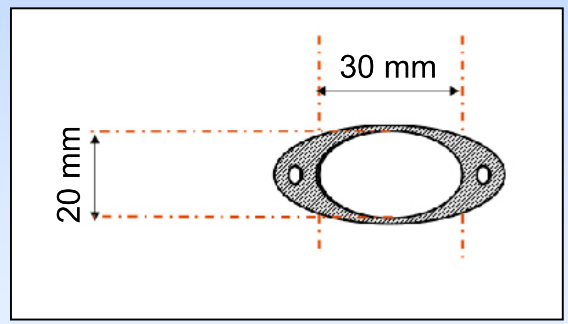


RIKEN from SAES literature



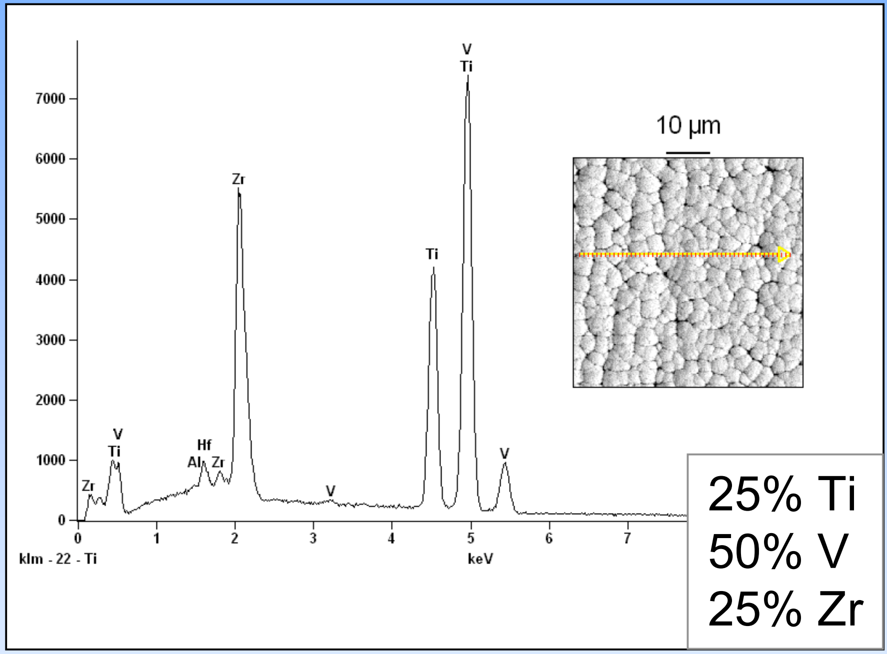
Distributed Ion pump: Y.Li et al., JVSTA 15 (1997) 2493.

ESRF insertion device



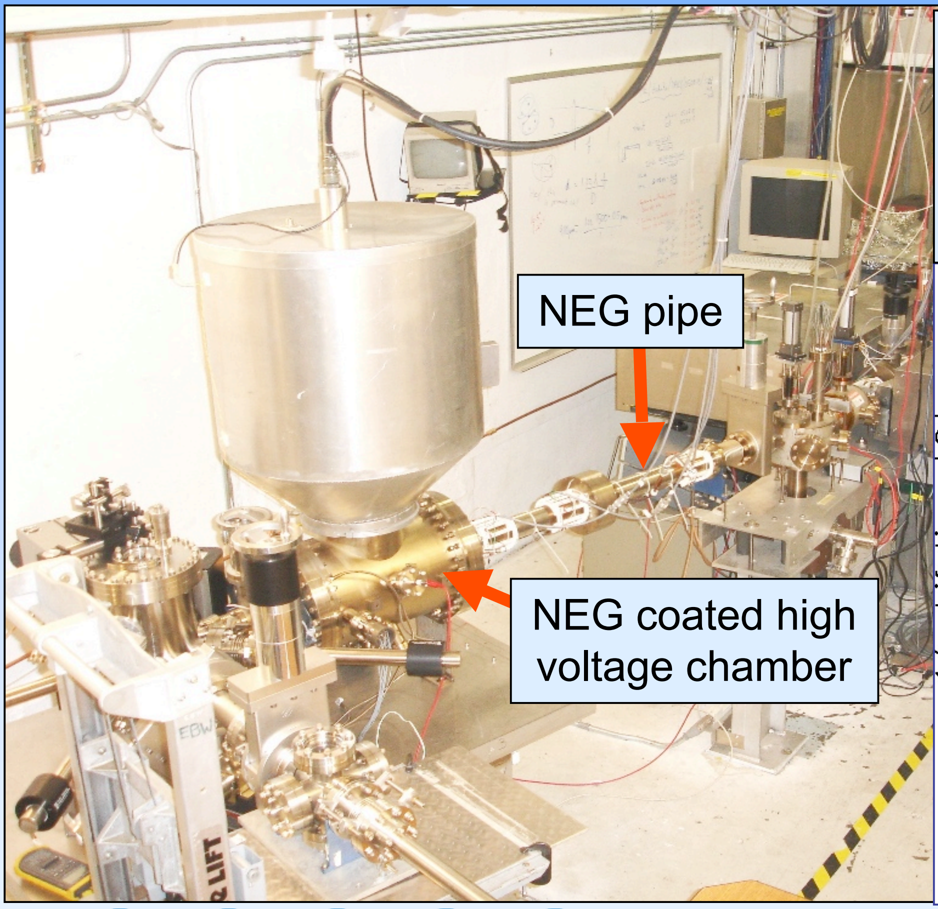
JLab's NEG coating

- 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

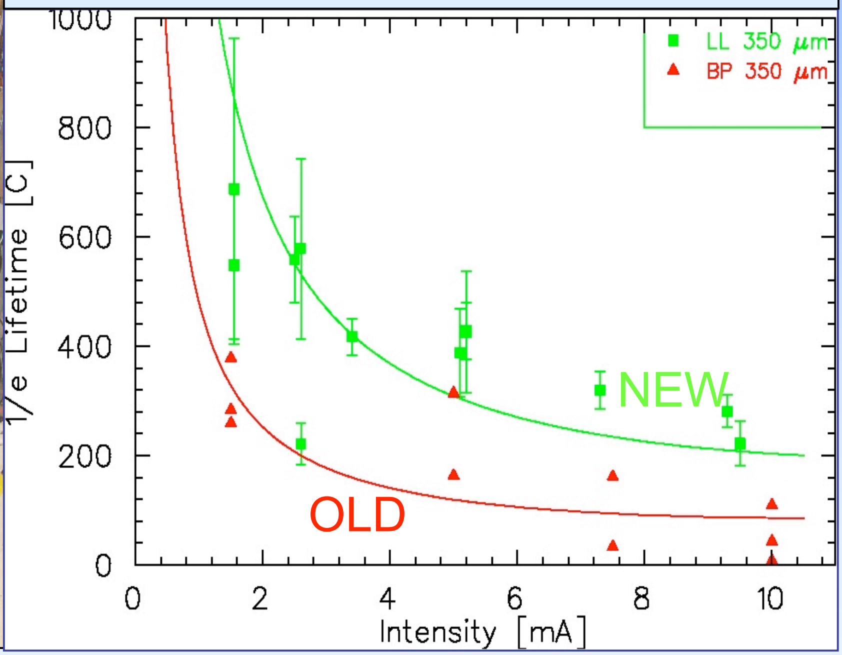


EDS analysis of getter coating composition

Load Locked Electron Gun



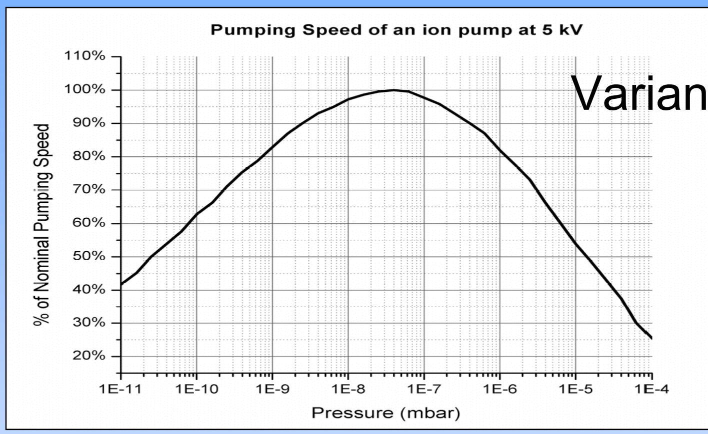
NEG coated HV chamber
Vacuum measured: ~1.2×10^{-11} Torr
Lifetime doubled
5-10 mA, 100 keV electron beam

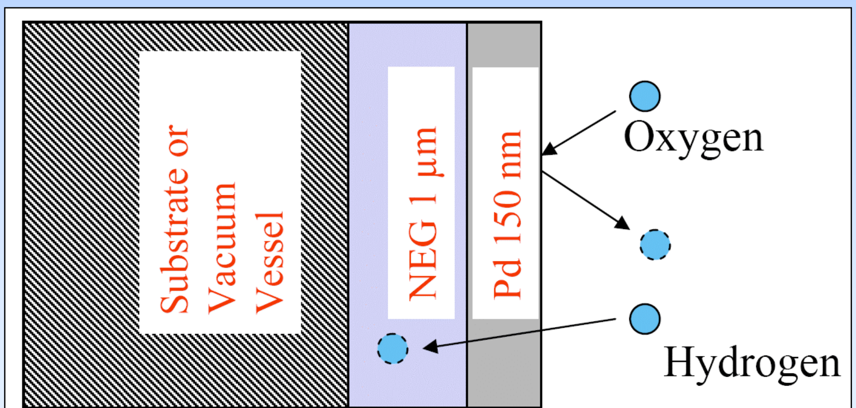


Pumps for XHV

- 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, N_2
  - Don't pump methane, noble gasses
  - Question about pump speed at base pressure
- Ti Sublimation
- Cryo pumps
- Turbo pumps – cascaded pumps

Ion pump limitations





Maruo Audi, 45th IUVSTA

- 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 2-6x10^{-11} mbar

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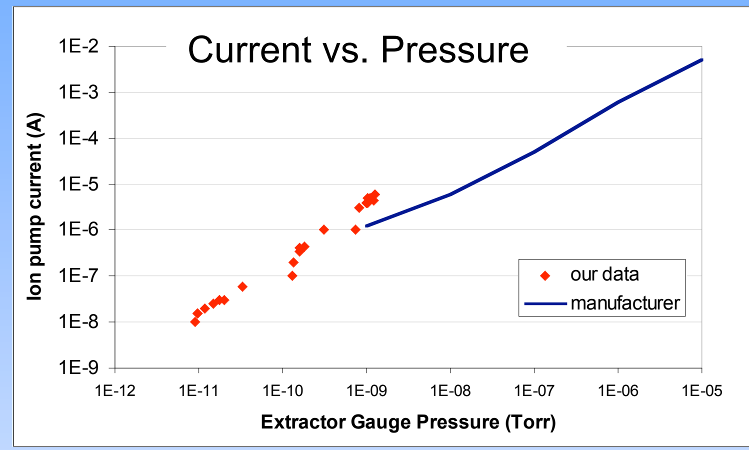
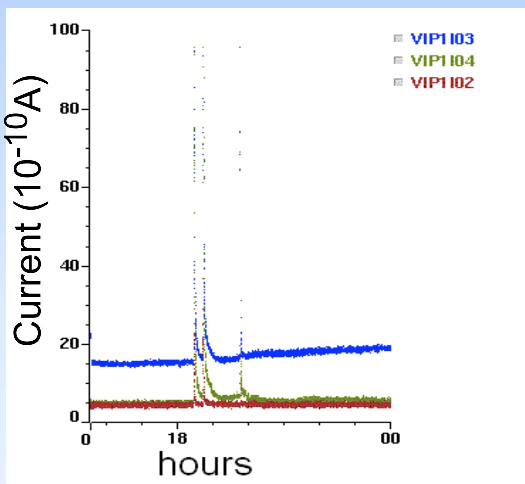
JLab UHV ion pump current monitoring

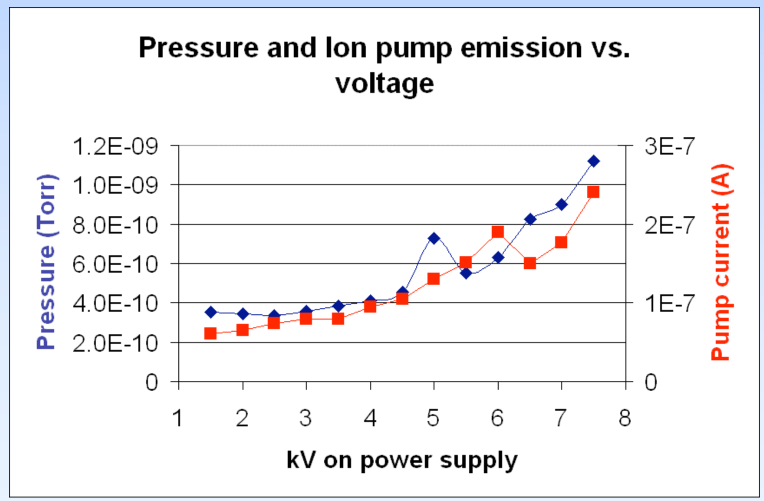
- Ion pumps current varies linearly with pressure as low as $1x10^{-11}$ Torr
- Real time monitoring of UHV vacuum
- Studying optimal voltage for pumping at low pressures





~10^{-10} Torr Full Scale

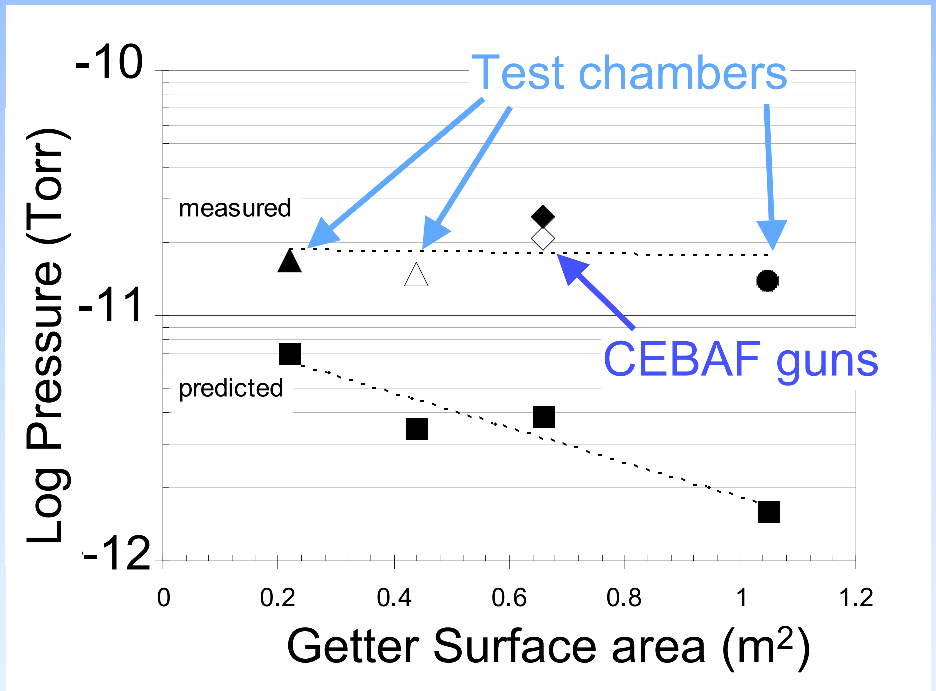
Discharge event in beamline



Base pressure in CEBAF guns

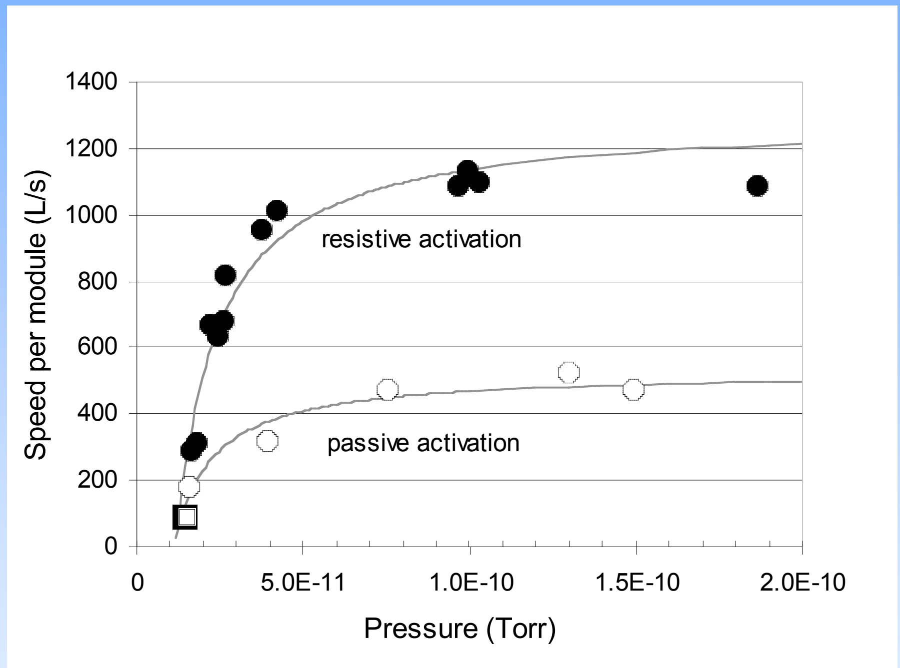
- Why isn't our chamber pressure as low as calculated?
  - Is outgassing much higher?
  - Is pump speed much lower?
  - Are we unable to measure lower pressures?
- First measured outgassing rate from chamber
  - 1×10^{-12} Torr·L/s·cm^2
  - Typical value for baked 304SS

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



Pump speed measurements

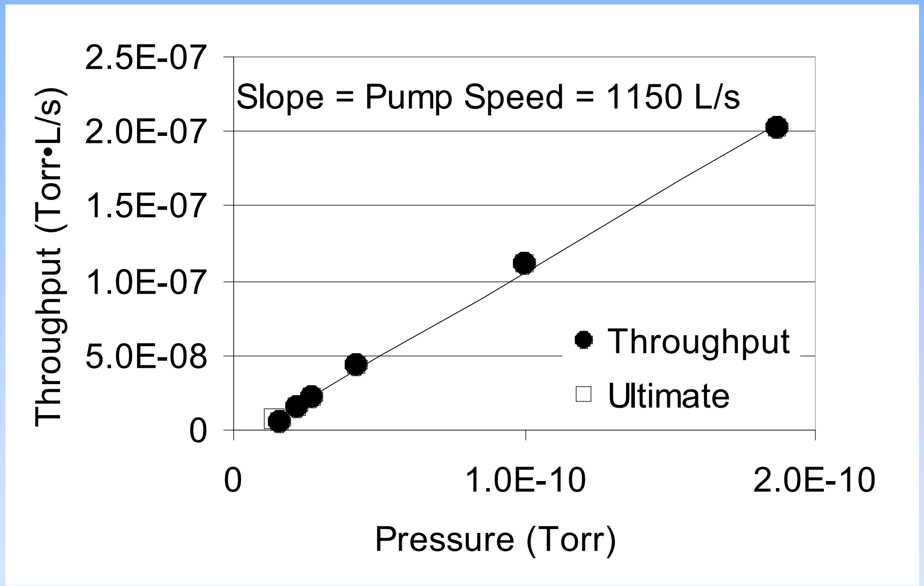
- Measured pump speed vs. pressure from base pressure of chamber to 2×10^{-10} Torr
- Throughput method
  - conductance limiting orifice
  - RGAs to measure H_2 pressure
- Ultimate pressure method
  - Gas sources: outgassing from walls and gauge
  - Measure with extractor gauge
- Found very good pump 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?



$$S = \frac{C * \left[\left(P_{orf} - P_{orf}' \right) - \left(P_{main} - P_{main}' \right) \right] + Q_{wall} + Q_{gauge}}{P_{main}}$$

Alternate analysis of pump speed measurement

- Q=S*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?

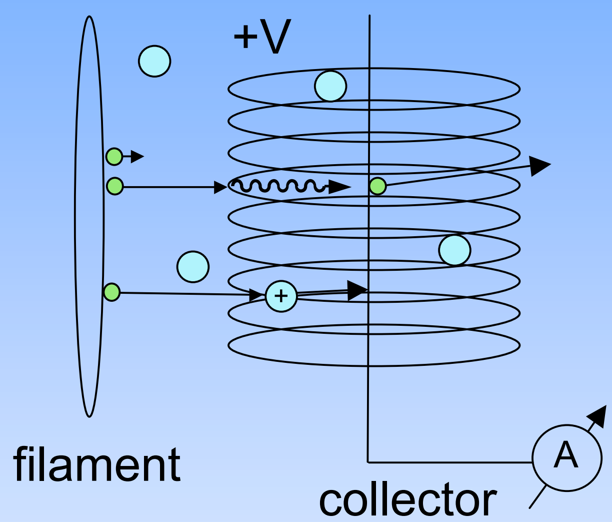


M. Stutzman *et al.* submitted to NIM 2006

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

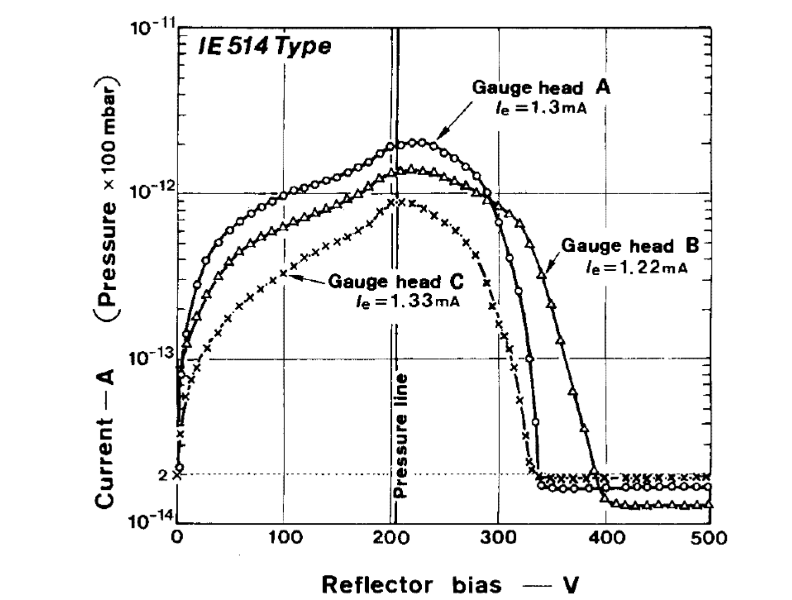
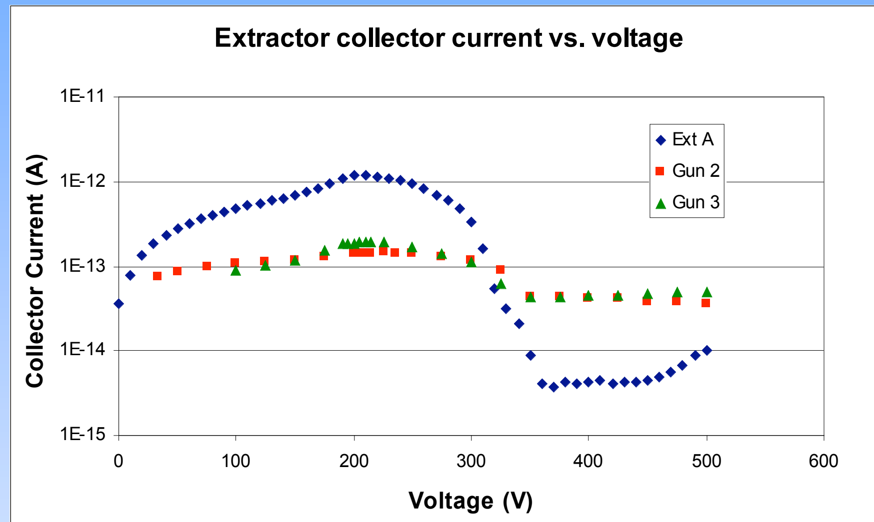


FIG. 3. Reflector bias effect for various IE514 gauge heads.

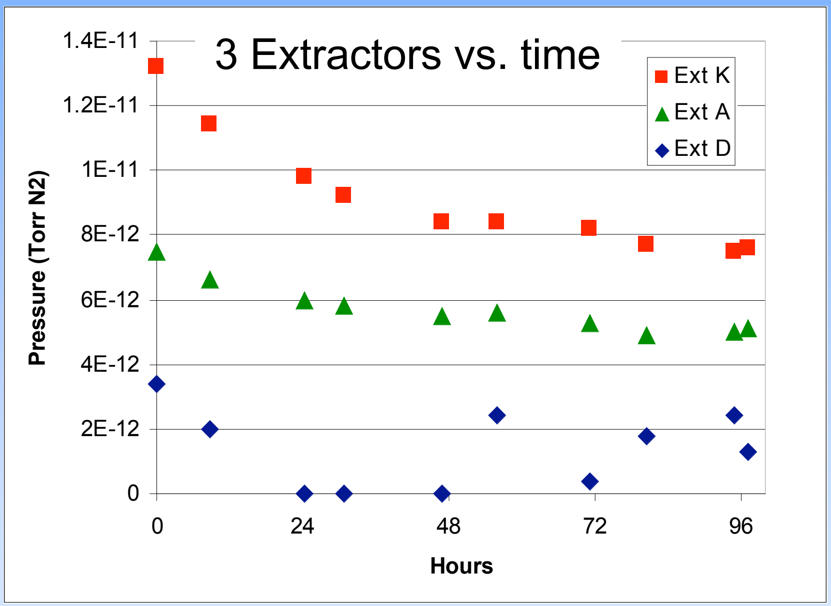
Fumio Watanabe JVSTA 9 (1991).

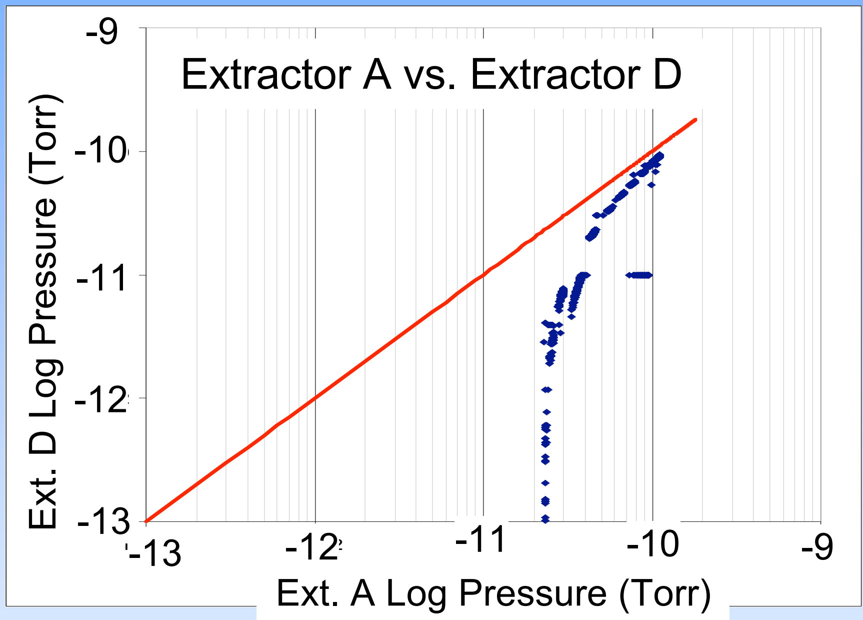


JLab 2006

Gauge	X-ray Limit (Torr)
Watanabe A	2.1×10^{-12}
Watanabe B	1.6×10^{-12}
Watanabe C	1.9×10^{-12}
JLab A	0.63×10^{-12}
JLab Gun 2	$>2 \times 10^{-12}$
JLab Gun 3	$>2 \times 10^{-12}$

Extractor gauge comparison





Three extractor gauges
Factor of 8 difference in readings

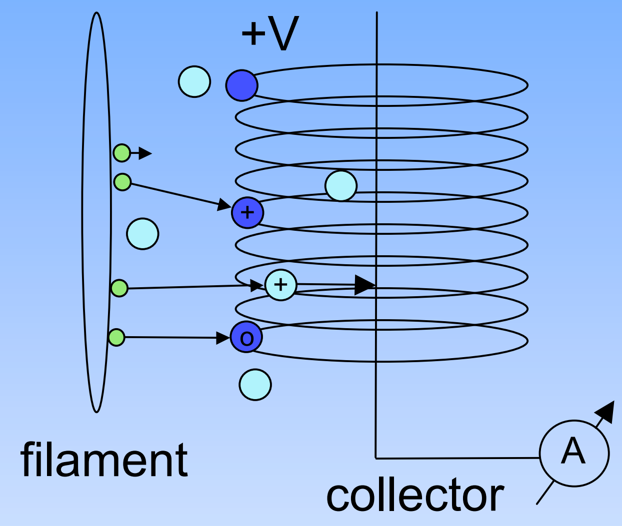
- Identical ports
- Symmetric positions
- Multiple degas cycles

Divergence in pressure readings below 5×10^{-11} Torr

Marcy Stutzman
AVS 53rd International Symposium
14 November 2006

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



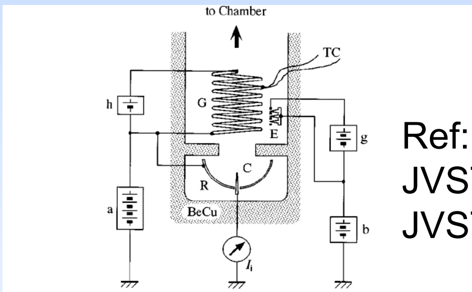


FIG. 1. Schematic diagram of the heated-grid gauge. G: Pt-clad Mo spiral grid, E: Spindt-type field emitter array, TC: R-type thermocouple, h: grid-heating power supply, a: grid-bias power supply, g: gate-bias power supply for emitter, b: emitter-bias power supply. All electrodes are immersed in a beryllium-copper-alloy envelope.

Ref: Fumio Watanabe JVSTA 17 (1999) 3467, JVSTA 20 (2002) 1222.

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

<u>Calibration techniques</u>

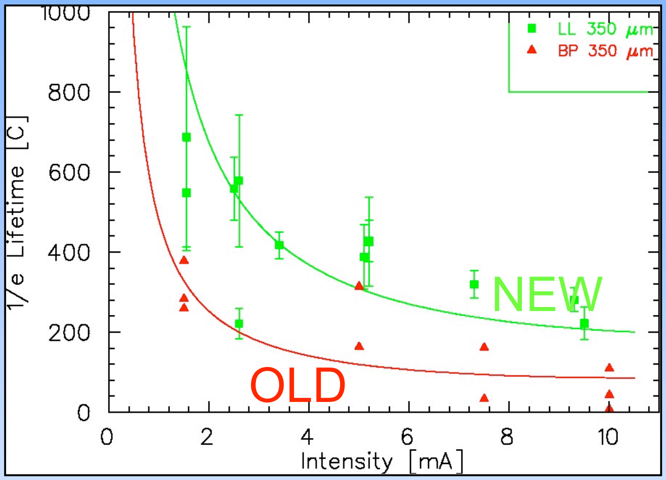
Calibration Techniques

- Careful calibration needed for measurements below 5×10^{-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.
A. Berman and J.K. Fremerey JVSTA **5** (1987) 2436.
W. Jitschin *et al.* JVSTA **10** (1992) 3344.
S. Ichimura *et al.* Vacuum **53** (1999) 291.
P. Szwemin *et al.* Vacuum **73** (2004) 249.

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