Water Vapor Outgassing and Readsorption Test Experiment

Overview

The LIGO beamtube modules were originally vacuum baked at 160 C for 1 month to reduce water vapor outgassing from approximately J(H2O) ~ 1×10^{-11} Torr-liter/s/cm^2 as fabricated, to J(H2O) < $1e^{-17}$ Torr-liter/s/cm^2 in operation. This final outgassing requirement was overestimated and is at least an order of magnitude better than required, even for Advanced LIGO. However, gate valve and tube wall failures in the interim will require limited reexposure to atmospheric water vapor, compromising this performance. To be economically feasible, recovery from such interventions may demand a less aggressive and less time-consuming bakeout protocol.

For the original bakeout, water vapor coverage and desorption were successfully modeled using the semi-empirical formalism of Dubinin-Radushkevich (DR) isotherms [LIGO T890001 (Weiss 1996), etc.]. The first goal of this experiment is to test this formalism, and if necessary develop extensions or corrections, in untested intermediate regimes of partial vapor reexposure. Repair scenarios may invoke such limited repopulation, for example by use of short-duration backfills with artificially dry gas. Our second goal for this experiment is to develop and test recovery protocols (e.g., bakeout or other degassing methods) that economically restore the resulting compromised surfaces to an acceptable outgassing rate.

It is expected that these experiments will inform a subsequent scale test, using a sample section of beamtube currently stored at LLO. This will in turn enable repair and/or replacement of leaking LIGO gate valves without excessive expenditures or loss of observing time.

DR Isotherm Experiment

Objective

This experiment is performed in the molecular flow regime.

The goal is to experimentally determine if the steel used in the construction of LIGO follows the surface coverage equation predicted by the Dubinin-Radushkevich (DR) equation

$$\sigma = \sigma_m \, e^{-\left(\frac{T}{T_0}\right)^2 ln^2\left(\frac{P}{P_0}\right)}$$

where

σ = surface coverage (molecules/area)

σ_m = surface coverage (molecules/area) at the saturation vapor pressure P_0 at temperature T

P is the pressure

P_0 is the saturation vapor pressure at T

T is the temperature

T_0 is the adsorption energy (temperature). T_0 is also a Gaussian distribution function of adsorption site energies, with a tail skewed towards the high energy part of the distribution.

We will determine the energy distribution function

$$\theta(T_{act}) = (2\frac{T_{act}}{T_0})^2 e^{-\left(\frac{T_{act}}{T_0}\right)^2}$$

by monitoring the equilibrium pressure versus temperature (T_{act}) behavior for steel samples degassed in a sealed vacuum chamber to which a known volume of water has been reintroduced.

In a (sealed) system the total number of molecules between the adsorption sites and the gas volume is conserved. With uniform energy distribution and surface sites depleted by thermal bakeout, the adsorption site residence time can be simply related to the equilibrium pressure (density). To keep the experiment time reasonable (residence time short) we want to perform the experiment in a regime where b<<1, with b defined as follows

$$b = \frac{\rho \, f_0 \, V_{thermal} \, \tau(T_{act})}{4 \, n \, \sigma_0}$$

 where

ρ = gas density (molecules/volume)

f_0= the sticking probability, $0 < f_0 < 1$

$\tau(T_{act})$ = the residence time at energy T_{act}

$V_{thermal}$ = the thermal speed $\sqrt{\frac{8kT}{\pi \, m}}$

n is the number of monolayers

σ_0 is the surface coverage (molecules/area) at the saturation vapor pressure P_0 at temperature T, ~ 3 x 10^{15} molecules/cm^2 per monolayer.

Procedure

Sample preparation

The samples shall be made of 1 inch wide 304L stainless steel strip and will receive the following processing.

1. Cut the samples into 20" (inch) lengths. 282 samples minimum. This gives 11,300 inch^2 of sample surface area or approximately 10x the chamber surface area of 1100 inch^2.
2. Check that deionized (DI) water resistivity is a minimum of 2 MΩ-cm. Handle samples with nitrile or latex gloves.
3. Degrease and the samples using Mirachem 500 undiluted and at room temperature. Residence time is 30 minutes. Rinse with DI water and air dry. See C-CMBS1, "Coil Material Bake Specification", LIGO-E950023-x0 modified by C961636, "CBI Request for Information (RFI) #21:

Coil Material Bake Temperature Range" to 444C ± 12C (approved by LIGO Lab in document C961612-x0) for additional information.

4. Stack the samples into bundles and tightly wire tie them. This step approximates coiled samples.
5. Air bake the samples at 444 C for 36 hours. Cool to room temperature (RT). Cut tie wires and destack the bundles.
6. Clean samples with Mirachem 500 and cold DI water 50% dilution. Rinse in cold DI water.
7. Rinse samples with 240 F (115 C) water/steam. An alternate process is to rinse in 65 C DI water (65 C was the final temperature of the LIGO beamtube during steam cleaning).
8. Air dry under a laminar flow bench.
9. Rinse several samples in reagent grade isopropanol, air dry.
10. Collect a FTIR sample using reagent grade isopropanol, a dilution of ~0.2 cc/cm^2 sample area (50 cc/sample) matches LIGO beamtube FTIR dilution.
11. Compare surface finish, oxide blushing, and cleanliness of the processed strips to legacy LIGO material.
12. Store clean samples in nylon bags.
13. Collect several 1-gram samples for H2 hot extraction measurement. See https://dcc.ligo.org/T880048 for historical data.

Test chamber preparation

The chamber shall be of all-metal construction. Prepare the chamber as follows.

1. Assemble components using UHV techniques; pre-clean flanges and chamber using acetone followed by an isopropanol rinse. Handle all components using gloves. Do not cross contaminate clean and dirty parts. Bottom all UHV (CFF Conflat) flanges until line to line contact is achieved.
2. Evacuate system and leak test using in-situ mass spectrometer. Vent system.
3. Clean, assemble (stack) and tie wire together approximately 20 to 50 strips as a thermal load and place in chamber. This stack will be used to measure the thermal time constant between the load and the external heaters, and to tune the PID loop.
4. Assemble heating mantle and tapes, heat system to 150 C and bake out. Target pressure < 5x10^{-9} Torr (Bayard-Alpert gauge). Evacuate and heat gas fill system simultaneously. Monitor all thermocouples continuously.
5. Record residual gas spectra at pre, hot and post bake. Water peak should be less than 1x10^{-9} Torr (system at room temp), peaks at 2 amu (H2) and 28 amu (CO) should dominate the spectrum. Air leaks, hydrocarbons, etc. should not be evident. Monitor pump down time and note valve/pump configuration. Compute H2 and CO outgassing rates using pump speed/conductance values.
6. Test programmed heating and cooling, tune PID loop if required. Profile should be as close to a linear ramp as possible (linear cooldown preferred). Temp range is 500K to RT. Perform post-bake helium leak test using QMS tuned to He peak.
7. Calibrate/crosscheck pumping speed measurement arm with calibrated leak, known conductance.
8. If available, test ultra-pure gas backfill (future experiment) system for water carryover by using QMS. Record dew point if available.

9. If doing water injection experiment: Set up the gas inlet system for water backfill. Evacuate any air in the headspace above the water column using a LN2 freeze-thaw (Schlenk-line) technique, minimum 3 cycles.
10. If doing water injection experiment: Perform water backfill. Monitor mass 18 partial pressure with QMS and calibrate. The backfill is done without the pumps vavled-in. Target pressure is 3×10^{-7} Torr partial pressure. Valve off QMS and hold with 8 l/s ion pump when completed.
11. Vent system using GN2 backfill.
12. Install previously prepared and cleaned samples in chamber. Place thermocouples at edge and center of steel strips.

Experiment procedure

1. Set up the gas inlet setup for water backfill.
2. Evacuate vacuum chamber and cross over to the 300 liter/sec turbopump at P~100mTorr. Also evacuate the back to air system to the (closed) GN2 regulator valve. The 8 and the 60 liter/sec ion pumps are to be valved-off.
3. At P ~ 1×10^{-5} Torr open valve to QMS.
4. Monitor pressure reduction of mass 18, to convert to monolayers. Pump speed for water vapor is approximately 250 liter/second. Also monitor Bayard-Alpert gauge (BA gauge sensitivity should be set for N_2 ionization, and correct gauge constant).
5. Perform thermal bakeout to 200 C, (500K) continuously monitor mass 18. Want to achieve 0.01 < b < 0.1 for T ~ 1.5×10^4 K activation energy.
6. Stop pumping (valve off) the system when P < 3.8×10^{-7} Torr. Sample calculation (Table 1) is for b~.05 and T_{act}=1.5×10^4 K. Additional values can be calculated by using spreadsheet "gas law calculates b.xlss".

Table 1. Sample calculation for water vapor valve-off pressure versus activation energy. Accessible value (highlighted) has a residence time of 1 second, b<<1, T=500K. Pressure must be well below 3.8×10^{-7} Torr before pumping system is valved-off.

| Highest adsorption site activation energy	Lattice Constant	Sample Temperature	Boltzman factor residence time (sec)	Log (residence time)	b= 0.05 density	Pressure to stop pumping
T_{act} (K)	tau_0 (sec)	T_{sample} (K)	$tau_0 e^{Tact/T}$	$Log(tau_0 e^{Tact/T})$	molec/m^3	P_{stop} (Torr)
10	1.00E-13	500	1.02E-13	-12.99	7.67E+28	3.98E+06
100	1.00E-13	500	1.22E-13	-12.91	6.41E+28	3.32E+06
1000	1.00E-13	500	7.39E-13	-12.13	1.06E+28	5.50E+05
2000	1.00E-13	500	5.46E-12	-11.26	1.43E+27	7.44E+04
3000	1.00E-13	500	4.03E-11	-10.39	1.94E+26	1.01E+04
5000	1.00E-13	500	2.20E-09	-8.66	3.55E+24	1.84E+02
8000	1.00E-13	500	8.89E-07	-6.05	8.81E+21	4.57E-01
10000	1.00E-13	500	4.85E-05	-4.31	1.61E+20	8.37E-03
12000	1.00E-13	500	2.65E-03	-2.58	2.95E+18	1.53E-04
15000	1.00E-13	500	1.07E+00	0.03	7.32E+15	3.80E-07
18000	1.00E-13	500	4.31E+02	2.63	1.82E+13	9.42E-10
20000	1.00E-13	500	2.35E+04	4.37	3.33E+11	1.73E-11
25000	1.00E-13	500	5.18E+08	8.71	1.51E+07	7.83E-16

7. Introduce "n" moles of water by raising partial pressure (see tabulation from spreadsheet for partial pressure required) of mass 18 as indicated by mass spectrometer.

8. Start programmed temperature ramp to room temp. Continuously monitor equilibrium pressure of mass 18 vs temperature.

9. Calculate T_{act} from pressure vs temperature data. Assume constant energy with $T=T_m$. This procedure is outlined in LIGO MEMO 61558.TEX or LIGO T890001. Tabulate the initial pressure (density), and equilibrium pressure (density) after water injection and temperature ramp.

Equipment List: Consumables

1. Steel strip per UNS S3043 Finish 2D (anneal and acid pickle), 0.030 (22 gauge) thick x 1" width x 20" length, 350 pcs.
2. Isopropyl alcohol reagent grade (2-propanol) CAS 67-630 >99.5%.
3. Acetone (2-propanone) reagent grade CAS 67-64-1 >99.5%.
4. Wire UNS3043 Finish 2D or BA, 20 gauge.
5. Backfill gas: GN2 UHP 99.99 (backfill only).
6. Test Gas: GN2 Research Grade <20 PPB H20, Air Products.
7. Water, deionized, 2 MΩ-cm.
8. Mirachem 500, Mirachem Chemical, Phoenix AZ.
9. Thermocouple, Type K.
10. Air, compressed, desiccant dry and hydrocarbon-free for furnace atmosphere.
11. Argon calibrated leak, LACO Technologies, Salt Lake City, UT.

Equipment List: Vacuum System
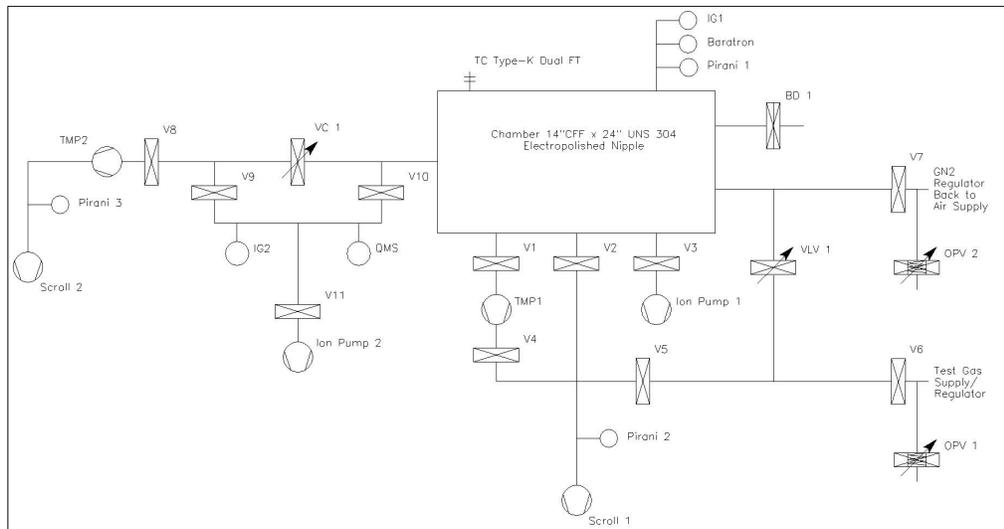
Part Identification

Valves

V1	6" CFF o-ring seal
V2	2.75 CFF all metal
V3	6" CFF o-ring seal
V4	KF25
V5	KF25
V6	VCR metal bonnet
V7	VCR metal bonnet
V8	4.5 CFF o-ring seal
V9	2.75 CFF all metal
V10	2.75 CFF all metal
V11	2.75 CFF all metal
VLV 1	Varian Leak Valve 2.75-1.33 CFF
VC 1	Conductance control (retractable disk with precision hole)
OPV 1	Gas supply overpressure 1 ATM
OPV 2	Gas supply overpressure 1 ATM
TV1	Turbo stack vent (not shown)
TV2	Turbo stack vent (not shown)

Pumps

TMP 1	300 l/s turbo with drag stage 6" CFF
TMP2	30 l/s mini- turbo 4.5 CFF
Ion 1	60 l/s diode or Star-cell 6 " CFF
Ion 2	8 l/s diode with low field shunt 2.75 CFF
Scroll 1	NW 25 Iwata or Tri-Scroll
Scroll 2	NW 25 small scroll Varian IDP2

Gauges

IG1	Nude MKS Stabil-ion 2.75 CFF
IG2	Nude MKS Stabil-ion 2.75 CFF
Pirani 1	MKS 275 Convectron 1.33 CFF
Pirani 2	MKS 275 Convectron KF25
Pirani 3	MKS 275 Convectron KF25
Baratron	1 K capacitance manometer 1.33 CFF
QMS	128 AMU Pfeiffer Mass spectrometer

Feedthru

TC1	2-channel Type-K 1.33 CFF
E1	Electrical (not shown) 2.75 CFF if internal heaters needed
MD 1	Microdot S-50 (not shown) for QCM

Misc

BD 1	Burst disk UHV 2.75 CFF