LASER INTERFEROMETER GRAVITATIONAL WAVE OBSERVATORY
- LIGO -
CALIFORNIA INSTITUTE OF TECHNOLOGY
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LIGO Vacuum Compatibility, Cleaning Methods and Qualification Procedures

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This is an internal working note
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NOMENCLATURE AND ACRONYMS

ADP	Ammonium Di-hydrogen Phosphate [$(NH_4)H_2PO_4$]
AMU	Atomic Mass Unit
HC	Hydrocarbons
HF	Hydrofluoric acid
JPL	Jet Propulsion Laboratory
KDP	Potassium Di-hydrogen Phosphate [KH_2PO_4]
LIGO	Laser Interferometer Gravitational Wave Observatory
OFHC	Oxygen Free High-Conductivity Copper
NEO	Neodymium Iron Boron
PFA	Perfluoroalkoxy fluoropolymer (Du Pont)
PTFE	Polytetrafluorethylene (Du Pont)
PZT	Lead-Zircomate-Titanate
RGA	Residual Gas Analyzer
RTV	Room Temperature Vulconizing Silicone elastomer
TBD	To Be Determine
UHV	Ultra High Vacuum

1. PURPOSE

Outgassing and contamination potential must be considered in every aspect of interferometer construction, from design and choice of materials through preparation, bakeout, storage and installation procedures as well as during any subsequent handling or adjustment. To insure uniform application of the same criteria we are establishing the following Policy.

All items to be installed inside LIGO vacuum equipment or beam tubes are to conform to this policy for selection of components and exposed materials and for preparation, handling, testing and storage prior to assembly and during assembly.

2. VACUUM COMPATIBLE MATERIALS APPROVAL PROCESS

All materials/parts (commerical and custom designed) must go through the materials cleaning process as described. The temperature and duration of the bake are determine by LIGO, based sometimes on manufacturer's recommendations in section 4. The vacuum data of the tested materials/parts will be compared to the LIGO vacuum outgassing and contamination requirements before be put on the LIGO vacuum compatible materials approved list (E960050).

A component or subassembly is itself considered approved if all its exposed materials are approved and if its pre-installation treatment is consistent with the Procedures (Section 4) for those materials.

All blind holes and trapped volumes will be explicitly vented to avoid virtual leaks; provision for cleaning such volumes adequately (e.g., by solvent flushing) will also be considered in the design process.

A material is considered "exposed" unless it is encapsulated fully and hermetically within another material. All design using hermetic containment must be approved specifically by the Vacuum Standards Board.

Components composed of materials from a single class are to be prepared, handled and stored according to the corresponding procedure for that class (Section 5).

Irreducible subassemblies comprising more than one material class are to be prepared and handled according to the most stringent subset of procedures consistent with all materials involved.

If a function cannot be properly fulfilled, for reasons of cost, availability, or performance degradation, by approved components and materials, efforts should be undertaken to eliminate that function or minimize its incidence.

If vacuum and other requirements cannot be simultaneously met by application of approved or provisionally approved materials and components, Qualification and Screening tests (Section 6) are to be performed to establish provisional approval or rejection of candidate alternative materials, according to currently active criteria.

A qualification and screening tests report must be written for the candidate material/component after completion of tests. This report must include the amounts of materials, outgassing rates (approved or not), residual gas analyses, molecular species that is outgassed, amount of hydrocarbons outgassing, and surface contamination information if available. A material usage must be complied for every subassembly or component that is placed in the vacuum to be included in the report.

2.1 Commercially Produced Components

For commercially produced components with potentially many materials used in the construction, a detailed accounting of all of the materials and the amounts used must be submitted for review. It may be necessary for some components to obtain certifications (per article or serial number) for the materials employed in their manufacture, so that material substitutions by the manufacturer are visible to LIGO. The specific requirements/procedures to ensure that approved components do not have material substitutions by the manufacturers are ?

3. VACUUM STANDARDS BROAD

All materials/parts (commerical and custom designed) must undergo vacuum compatibility testing for outgasssing and contamination data collection. The outgassing data of the tested material/part are to be submitted to the Vacuum Standards Broad for review. The Vacuum Standards Broad will make the decision of approval or rejection of the tested material/part before to be included in the LIGO vacuum compatible materials approved list (E960050). The Vacuum Standards Broad members are selected by the Systems Engineering and the Detector System group management.

4. CLEANING AND PREPARATION OF MATERIALS PROCEDURES

A. Metals:

For all metals do the following:

- Machine all sides
- Ultrasonic clean in Alconox[1] for 10 minutes
- Rinse in distilled water
- Ultrasonic clean in ethanol[2] for 10 minutes.

Subsequent to the above steps bake the metal as follows:

Stainless Steel

- Bake in vacuum at 200 C° for 24 hours.

Aluminum

- Bake in vacuum at 120 C° for 24 hours.

NOTES: - In the case of gross contaminants, the above may be proceeded by an acid bath

1. Standard Alconox solution is 1 tablespoon in 1 gallon of water.
2. Methanol may be substituted for ethanol if availability is restricted or required volume is excessive.

(i.e., 20% Protex solution (diluted with distilled water) for aluminum or 2% Oakite solution for stainless steel), or an appropriate degreasing agent such as trichloroethane or acetone.
- Solvents must be 100% reagent grade.

B. Ceramics and Glasses:
- Clean off contaminants with soap and water or tricholorethane, be sure to rinse thoroughly
- Ultrasonic clean in ethanol for 10 minutes
- Soak in isopropyl alcohol for 10 minutes, agitating regularly
- Bake in a vacuum at 120 C° for 48 hours.

C. Composite Assemblies

1. Commercial Mechanical Assemblies:
   - Disassemble completely
   - Replace all non-approved materials by approved materials
   - Clean parts in ultrasonic cleaner with Alconox for 10 minutes
   - Rinse in clean water
   - Clean in ultrasonic cleaner with ethanol for 10 minutes
   - Use Krytox grease sparingly on surfaces that absolutely require lubrication
   - Reassemble stages
   - Bake in vacuum at 120 C° for 24 hours.

2. Electronic Components:
   - Clean with flux remover until no stains are visible
   - Clean with detergent and rinse
   - Bake in vacuum at highest temperature compatible with manufacturer's maximum storage rating.

3. Sensor/Actuator Head assemblies (with LED/PD assembly in place):
   - Ultrasonic clean in ethanol for 10 minutes
   - Soak in isopropyl alcohol for 10 minutes agitating regularly
   - Bake in vacuum at 120 C° for 48 hours.

5. HANDLING AND STORAGE PROCEDURES

Policy for Handling and Storage of Cleaned Parts and Assemblies:

- Nitrilite[1] gloves are to be worn for handling, assembly and installation of cleaned or partially cleaned parts.
- Gloves are to be changed when proceeding to handle components at different stages of processing.
- Tools and fixtures which may contact cleaned parts in assembly or transport are to be

1. Nitrilite 100% nitrile gloves from Ansell Edmont Industrial.

cleaned ultrasonically in Alconox solution and/or methanol (consistent with their construction) and air-dried.

- Processed parts awaiting installation or further assembly will be stored in bags or wrapping material made of contamination-free aluminum foil, or Ameristat 1.5^{TM} plastic film.
- Small parts may also be stored in stainless steel or glass containers, cleaned and prepared in the same way as vacuum equipment.
- Tables and work areas for cleaning, packing/unpacking, assembly, alignment and testing of cleaned parts are to be lined or covered with fresh contamination-free foil or film immediately before starting work. (Plastic film should not be used if an incompatible solvent is involved in the process). ANY STIPULATION ON ENVIRONMENT? CLEAN ROOM BENCHES NEEDED FOR FINAL STAGES OF CLEANING?

6. QUALIFICATION AND SCREENING TESTS FOR CANDIDATE MATERIALS, COMPONENTS AND PROCEDURES

All candidate materials must satisfy the criterion of screening and qualification tests before being consider for addition to the vacuum compatible “approved” or “provisionally approved” list. The screening test and the high power exposure (qualification) test of cavity mirrors are described in detail in the following paragraphs.

6.1 Screening Test

There are two steps of the screening test, 1) the vacuum bake for outgassing data collection and 2) the residual gas analysis.

6.1.1 Vacuum Bake

Vacuum baking of the candidate component/material is performed to obtain the hydrocarbon and other outgassing data information. The typical vacuum bake test setup is shown in Figure 1. Typical testing procedures are as follow:

1) Obtain sample of candidate component/material to be tested
2) Obtain a “Parts Cleaning Request” (see Appendix Form A1) form from Detector Systems for pre-bake cleaning. Follows the cleaning methods and handling procedures in Section 4 and 5 above according to the type of material, and indicate the procedures on the form.
3) Obtain a “LIGO vacuum bake oven procedure and check list” (see Appendix Form A2) from Detector Systems. Provide the component/material baking time and temperature and any requirements of temperature ramptime or soaktime. Baking temperature should follow written procedures (Section 4 and 5).
4) System calibration:
   a. Clean selected oven and preparation for calibration
   b. Calibrate the Residual Gas Analyzer (RGA) for the X & Y (pressure) axis
   c. Introduce a known test gas (e.g., AMU=28) into the test oven with the valve open and take pressure reading, than close the valve to have the oven pump down (25 liter/sec pump speed) and take another pressure reading after 10 minutes.

Calibration:
Using a calibrated leak, determine a correction factor for the vertical scale on the RGA to give an absolute calibration of RGA measurements. For example, given

- A calibrated leak *L*, of known AMU, typically $l = 1.5\times10^{-8}$ torr liter/sec at AMU 28
- A measurement of the apparent leak size, given by the difference of the peak size at AMU 28 with the leak valve open and the leak valve closed; call this $P_{\text{indicated}}$
- A known pumping speed for the system *S*, typically *S* = 25 liter/sec the correction factor will be given by

$$C = \frac{S}{L} P_{\text{indicated}}$$

IS THIS A CORRECT INTERPRETATION OF THE ORIGINAL TEXT?

Given a known leak rate of the valve = 1.5×10^{-8} torr liter/sec
Peak Mass = known test gas pressure reading from the RGA, obtained by the difference between the pressure reading with leak valve open and with the leak valve closed.

Calibration pressure constant = $1.5\times10^{\circ}$ / 25 torr

= 6×10^{-10} torr, with the Peak Mass, P(pump speed) = 25 liter/sec

through RGA gain = (Peak Mass) / 6×10^{-10}

d. Obtain a background scan of the sum of AMU 41, 43, 53, 55, 57; ensure that is less than $\sum(41, 43, 53, 55, 57) \le 1.0\times10^{-11}$

5) Perform the vacuum bake of candidate component/material
6) At the end of the vacuum bake period, obtain a graph print out of "Peak Mass" vs. "Mass Number" and a print out of pressure reading of the masses (41, 43, 53, 55, 57) from the RGA test instrument.

6.1.2 Residual Gas Analysis

Cleaning and baking of components/materials must be followed by a residual gas analysis. A set of outgassing calculation parameters need to be obtained for the residual gas analysis. The required calculation parameters are:

- The mass of AMU 41, 43, 53, 55 and 57
- $\sum$mass = $\sum(41, 43, 53, 55, 57)$
- LIGO vacuum pump speed = 3000 liter/sec.
- Test oven vacuum pump speed = 25 liter/sec.
- RGA gain (from paragraph 6.1.1 above calculation)

Measurement of outgassing rate from the RGA reading:
Obtain the $\sum$mass from the print out of pressure reading of the masses (41, 43, 53, 55, 57) from the RGA instrument.

Calculate:

Pressure (torr) = ∑mass / RGA gain

thru Outgassing rate = Pressure (torr) x [Test oven pump speed (25 liter/sec)]

For LIGO pressure requirement

Pressure (torr) = (Outgassing rate) / LIGO vacuum pump speed (3000 liter/sec)

6.2 High Power Exposure Tests of Cavity Mirrors

The exposure test is to evaluate the candidate material for optical contamination potentials. Outgassing can lead to contamination of the optics with the result of increased optical losses and ultimately failure due to heating. The amount of outgassing is less important than the molecular species that is outgassed. There are two test procedures in the exposure test, which are briefly described below; a complete procedure is yet to be developed.

A. Control cavity test

The control cavity test is setup to establish a known mirror cavity baseline for the material exposure portion of the exposure test.

1) Setup an empty, very clean vacuum tank, with a quartz spacer between two mirrors (see Figure 2). The mirrors are very similar to LIGO mirrors with T~10ppm, zero field on surface, and S (scattering) + A (adsorption) ~ 10ppm.
2) Measure the storage time T_E for the laser light travel between the two mirrors. Light storage time within T_E ~ 80 usec

B. Material sample cavity test

- Material sample is placed inside the same vacuum tank cavity (see Figure 2) and the same test procedures as the control cavity test described above are followed.
- Light storage time T_E should be ~ 80 usec
- If storage time decreases (i.e., light losses went up), e.g., T_E decrease by 2.5 usec - L (losses) increased by ~ 1 ppm
- If the Control cavity test shows no sign of decreased light storage time but the material cavity test does show a decreases, then the material sample outgassing affects the performance of the cavity mirrors.

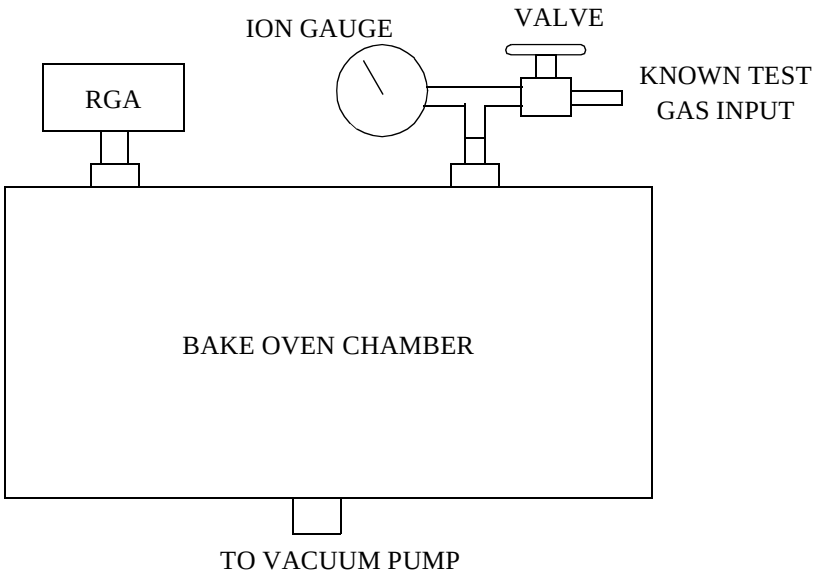

Figure 1: Typical Vacuum Bake Test Set

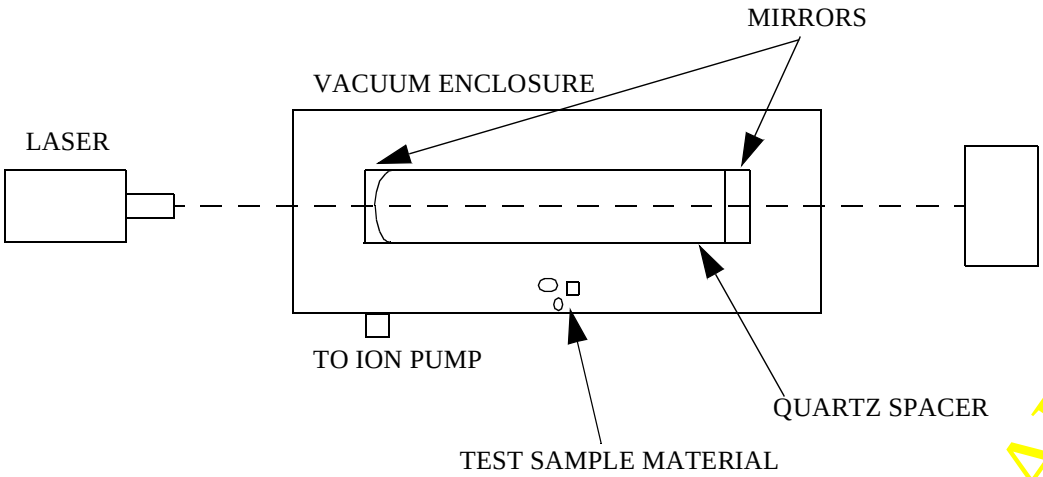

Figure 2: Exposure Test Setup

APPENDIX

Parts Cleaning Request

Name _____ Phone _____ Date _____

Parts Description, Dwg # _____

_____

Used In (next higher assembly) _____

Material: AL SST CST Bronze

Macor Teflon Viton Glass

Other: _____

Special Handling: _____

_____

Baked In Oven: _____ Load # _____ Temp.: _____ C°

Date In _____ Date out _____

Form A1. Parts Cleaning Request

LIGO Vacuum Bake Oven Procedure and Check List

Oven: _____ Load # _____ Date: _____

Load Contents: _____

Cap Torqued: _____ ft/lbs Leak Checked: Y N

All Metal Valve Open: Y N Vent Valve Closed: Y N TP ON _____:_____

Pressure: _____ Torr Date & Time: _____ _____:_____

Pressure: _____ Torr Date & Time: _____ _____:_____

Pressure: _____ Torr Date & Time: _____ _____:_____

NOTE: Do not turn heat on when pressure is above 5E-5 Torr.

AUTO/MANUAL:

RampTime - Oven: _____ Hrs., Pumpline: _____ Hrs.

SoakTime - Oven: _____ Hrs., Pumpline: _____ Hrs.

BAKE TEMPERATURE (C°):

Oven: _____ Pumpline: _____ TurboPump Heat On: Y N

TEMPERATURE (C°):

	P-Line	End	Body	Cap	Date & Time	Pressure (Torr)
1						
2						
3						
4						

TP Heat Off: Y N Temp Cont. sw Off: Y N Reset PROG OFF: Y N

DEGAS:

Fil On? Y N W/Dycor# _____ Date: _____ Time On: _____ Time Off: _____

SCAN:

Oven: ____:_____ Temp: _____ C° P-Line: ____:_____ Temp: _____ C°

All Metal Valve Closed: Y N N-Line Open: Y N Vent: Y N

Comments: _____

_____

_____

_____

Form A2. LIGO Vacuum Bake Oven Procedure and Check List