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 vessels or 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 and baked in vacuum with temperature recommended by the manufacturer's as 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-E).

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.

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, its outgassing rates (approved or not) and residual gas analyses. 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 get certifications (per article or serial number) of 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 STANDARD BROAD

All materials/parts (commerical and custom designed) must go through the vacuum compatibility testing for outgasssing and contamination data collection. The outgassing data of the tested material/part are submitted to the Vacuum Standard Broad for review. The Vacuum Standard Broad will make the decision of approval or rejection of the tested material/part before be put on the LIGO vacuum compatible materials approved list (E960050-A-E). The Vacuum Standard Broad members are selected by the Systems Engineering and the Detector System group. The current broad members are shown on the signatures approval block in page two of this document.

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 (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

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.

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 Stages:
- Disassemble and clean parts in ultrasonic cleaner with Alconox for 10 minutes
- Rinse in clean water
- Clean in ultrasonic cleaner with ethanol for 10 minutes
- Replace all plastic parts with appropriate metal or Teflon replacement part (Teflon PFA 440 HP pieces)
- Remove Teflon parts and clean thoroughly
- 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 detergent and rinse
- Bake in vacuum at highest temperature compatible with manufacturer's max rating.

3. Sensor/Actuator Head assemblies:
- 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

Handling and Storage of Cleaned Parts and Assemblies policy:

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 cleaned ultrasonically in Alconox solution and/or methanol (consistent with their

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

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 (available at JPL stores).

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).

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

All candidate materials must be satisfied with the criterion of screening and qualification tests before consider to be added 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

The screening test is the preliminary test to evaluate the candidate material for outgassing and contamination potentials. There are two steps of the screening test, 1) the vacuum bake for outgassing and contamination data collection and 2) the residual gas analysis.

6.1.1 Vacuum Bake

Vacuum baking of the candidate component/material to obtain the hydrocarbons and 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 the testing laboratory for pre-bake cleaning. Fillout the form by follows the cleaning methods and handling procedures in Section 4 and 5 above according to the type of material.
3) Obtain a “LIGO vacuum bake oven procedure and check list” (see Appendix Form A2) from the testing laboratory. Provide the component/material baking time and temperature and any requirements of temperature ramptime or soaktime. Baking temperature should follow manufacture recommendations.
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 ~6-10 minutes.

1. Calibration:

   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 pressure reading with valve open and subtract the pressure reading with the valve closed.

   Calibration pressure constant = $1.5\times10^{-8}/25$ torr

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

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

d. Obtain a background scan of $\sum(41, 43, 53, 55, 57) \leq 1.0\times10^{-11}$

5) 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:

P (pressure) = $\sum$mass / RGA gain

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

For LIGO pressure requirement

P (pressure) = (Outgassing rate) / LIGO vacuum pump speed (3000 liter/sec)

6.2 High Power Exposure Tests Of Cavity Mirrors

There are two cavities tests:

A. Control test
- One empty, very clean vacuum tank, quartz spacer
- mirrors very similar to LIGO mirrors T=10ppm, zero field on surface, S+A ~ 10ppm

B. Test
- Second, same cavity, very clean vacuum tank, contains material to be tested.
- Light storage time ~ 80 usec
- if storage time decreases (i.e., losses went up), e.g., t decrease by 2 usec - L increased by ~ 1 ppm
- if Control cavity test shows no sign of decreased light storage time but the test cavity does, - material outgasses something bad.

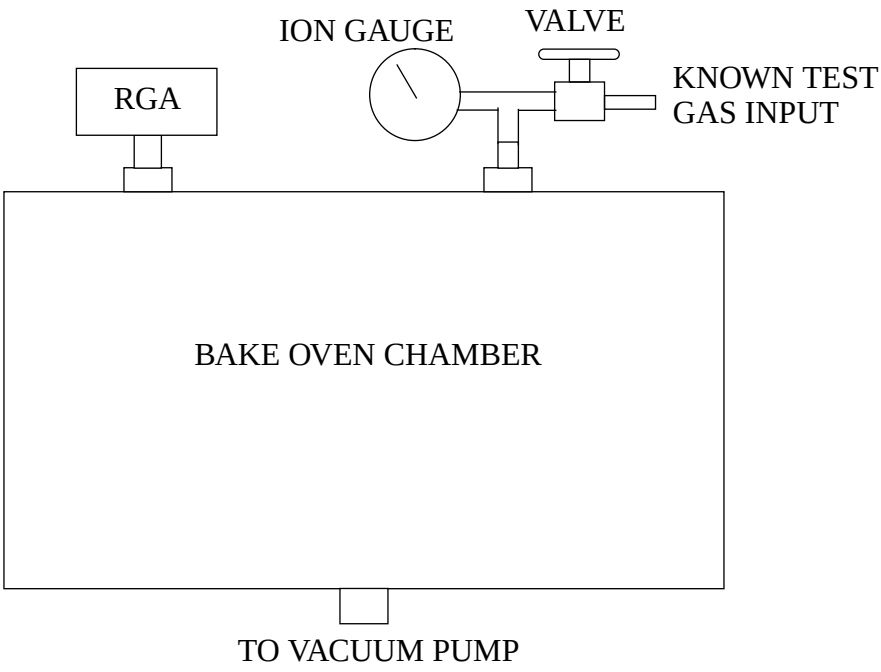

Figure 1. Typical Vacuum Bake Test Setup

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