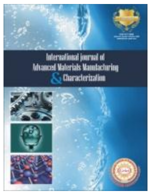

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Study the Effect of Heat Treatment on SS Material for LIGO-India UHV System

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ABSTRACT

LIGO-India under an international collaboration with LIGO-USA, has proposed for construction and operation of a large-scale international detector in India. The detector is 4 km arm-length Michelson Interferometer with Fabry-Perot enhancement arms, and aims to detect fractional changes in the arm-length smaller than 10^{-23}. Ultrahigh Vacuum (UHV) environment is required to detect such low fractional changes by this detector. The optics components, viz., mirrors, beams splitters and the interferometer will be installed in ultrahigh vacuum systems. The Ultrahigh Vacuum system consists of 8 kilometres of beam tubes of 1.2 meter diameter and large corner / end stations. The total volume of the vacuum system is about 10000 m^3, vacuum of ~ 10^{-7} Pascal. This UHV system will be the largest vacuum system in the southern hemisphere. To achieve UHV in such a large system, the selection of material and various surface treatments are very important. We have carried out the study of reduction of outgassing by applying various treatments to the selected material. An experimental system is installed in our lab for this purpose. A number of identical coupons are machined from ~ 3 mm thick SS 304L sheet. These coupons are treated in various heat treatment conditions. One sample with seam weld is also prepared. These coupons checked in a vacuum system for their outgassing rate. The surface of the coupons is analyzed by XRD and SEM. This paper presents the experimental set up and results of the test coupons.

Introduction

Impurities in UHV chambers of the Laser Interferometer Gravitational wave Observatory (LIGO) are main concern for detecting gravity wave [1]. Hydrogen, Oxygen and other hydrocarbon should be much less. It is highly prerequisite to have outgassing rates of SS material as low as 10^{-10}mbar l/s/cm^2 to achieve UHV. The partial pressure of all impurities should be less than 10^{-13}mbar. It is known that Stainless Steel 304L is the most suitable material for such type of construction of UHV chambers. Grade 304 is the most commonly used stainless steel. It is austenitic, corrosion resistant steel with excellent strength, toughness, fabrication characteristics and weldability. SS 304L is the low carbon version of Stainless steel. SS 304 L gives Hydrogenevolution when exposed to UHV. Suppression of the Hydrogen is necessary to achieve UHV using SS304L. Air baking of SS is known process to suppress Hydrogen. In this process the material is baked in presence of air at 450ºC up to 36 hours. We have carried out the process on SS304L test coupons in different environment like air, dry air, and oxygen to study the effect on Hydrogen out gassing.

It is well known from metallurgical science that hydrogen penetrates inside steel during the initial phases of production and fabrication, reaching weight concentrations of few ppm (parts per million)[2]. Even such a low hydrogen concentration is a fundamental problem in vacuum technology where the ultimate vacuum limit is determined by gases escaping from the exposed walls of the chamber.

The hydrogen outgassing can be reduced by reducing the hydrogen content in the bulk or by introducing a superficial barrier to hydrogen diffusion. The oxide layer normally present on stainless steel surface is one of the sources, as influencing hydrogen diffusion [3]. To increase layer formation of chromium oxide on the surface of SS 304, we considered various procedures for treating the test coupons, viz., 36 hours air baking, 36 hours dry air baking and 36 hours baking with oxygen gas concentration.

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There are two basic reasons to limit the outgassing rate of beam tube material of the LIGO vacuum system.

- To limit the phase noise associated with scattering from the residual gas species in the long Fabry-Perot arms.
- To limit mirror optical loss (scattering and absorption) due to condensed vacuum gas species.

The phase noise requirement must be satisfied by ensuring that the integrated outgassing from all of the LIGO in-vacuum components is within the pumping capacity of the system to keep the partial pressures of each gas species below requirements.

Optical loss due to adsorption/condensation of (high molecular weight) gas species and subsequent interaction with the incident laser light is a material compatibility issue more than an allowable outgassing rate issue.

The ultimate vacuum in the LIGO beam tubes is aimed the vacuum of ~ 1 ×10^{-9} mbar.

The outgassing rate measurement system

The Outgassing measurement system is designed to measure total gas load due to thermal out-gassing and composition of various gases evolving from the material test coupons exposed to different baking temperatures as a function of exposure time in vacuum.

Thermal Out-gassing critically depends on,

- Real surface area and not only actual measurable physical area. So, for a given physical area Thermal Out-gassing depends on surface finish. For example, electro-polished surface has lesser real surface area than mechanically polished surface having same physical surface area.

- Absorption and adsorption of gases (particularly water vapour and hydrogen) in material when exposed to processed like manufacturing, heat treatment, cleaning etc.
- Duration and temperature of material exposed to vacuum.

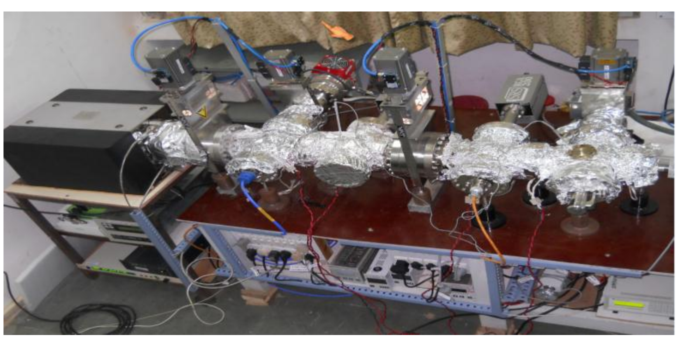

Figure 1. Outgassing rate measurement system

As per specifically indicated, outgassing measurements by dynamic conductance method is carried out with the vacuum system at room temperature (approx. 25^{o}C).

Test coupon preparation

We have prepared Test coupons from SS 304 sheet for studying the effect on outgassing rate after different procedures. All the test coupons has dimension of coupon is 200 mm × 50 mm × 3 mm. Aim of this testing is to investigate improvement of outgassing rate of SS 304 material due to formation of chromium oxide protecting layer after various heat treatments. According to available technical data, baking temperature for test coupon is considered as 450^{o}C at which surface of SS 304 can be oxidised and form chromium oxide layer. Heat treatments of test coupons are considered as follows.

(1) 450^{o} C Air baking for 12 hours
(2) 450^{o} C Air baking for 36 hours
(3) 450^{o} C Dry air baking 36 hours
(4) 450^{o} C baking with oxygen gas purging for 36 hours

Coupon baking has been carried out in industrial furnace at 450 ± 5^{o} C temperature. After the treatment, all the test coupons are protected to avoid any external contamination on the surface. Coupons are cleaned by Isopropyl Alcohol, before installing in outgassing measurement system.

Surface roughness analysis

As surface roughness of material plays very important role for outgassing rate of material and pump down time to achieve ultimate vacuum. The lesser surface roughness tends to low surfacecontamination and better cleanliness, hence weconsider minimum surface roughness ≈ 1 micron to minimise geographical surface area of test coupon. We measured surface roughness of various commercial available stainless steel samples to compare with test coupons.

The outgassing measurement system

Outgassing measurement system is designed and assembled to measure outgassing of test coupons. This system is shown in the figure 1. The schematic of the system is shown in the figure 2. The system comprises of two ultra-high vacuum chambers (Pumping chamber and Sample chamber) separated by gate valve. Material of construction of complete system is SS 304L. Pumping chamber is connected to Ion pump and Turbo molecular pump through gate valves. Turbo molecular pump is used to reduce gas load on Ion pump during initial pumping of the system from 1 × 10^{-3} mbar to 1 × 10^{-6} mbar and also during baking of the system. UHV gate valve between pumping chamber and sample chamber is modified with an aperture on its plate to act as calibrated conductance. Modified UHV Gate valve is kept open to effectively pump the sample chamber and closed to measure the gas flow through the calibrated conductance. Bayard-Alpert gauges and Quadrupole Mass Analyzer (QMA) type Residual Gas Analysers are used to measure total gaspressure and partial pressure of different gas species during the testing.

Two Bayard-Alpart gauges are mounted on two sides of the conductance to measure the differential pressure across the conductance

Measurement Procedure

Before starting of actual outgassing measurement, the system is pumped and baked to achieve vacuum in the ~1×10^{-9} mbar and measured gas load of the empty chamber.

The Out-gassing measurement is carried out as per the following steps.

1. Each coupon is prepared as such having the exposed surface area of ≈ 200 cm^2.

Table i. Surface roughness measurement of outgassing test coupons

Sample	Size in mm	Side	1	2	3	Average Ra µm
Long SS strip	180 × 24 × 2.5More	One surface	2.09	2.09	2.04	2.073
		Second surface	2.45	2.55	3.08	2.693
Short SS strip	60 × 38 × 2	One surface	0.27	0.20	0.23	0.233
		Second surface	0.40	0.32	0.29	0.336
Short SS strip	60 × 43 × 2	One surface	0.47	0.23	0.31	0.336
		Second surface	0.42	0.30	0.23	0.316
SS block	75 × 60 × 16	One surface	5.20	4.31	4.62	4.71
		Second surface	3.62	3.44	3.99	3.683
		Thickness side	8.4	--	--	8.4
Electro polished SS Stand	75 × 50 × 10	Random	0.18	1.31	0.97	0.82
		Thickness Side	0.11	0.068	--	0.089
63 CF I piece	--	Inside surface	0.43	0.52	0.60	0.51
Aditya Limiter Arc electropolished	--	Outer surface	0.23	0.12	0.31	0.22
35 CF Blank	--	Outside	0.80	0.70	0.79	0.76
		Inside	0.70	0.80	0.75	0.75
63 CF 'I' section with flange	--	Outside(Buffed surface)	0.15	0.12	--	0.13
		Inside(E.P.)	0.52	0.44	0.37	0.44
Air Baked SS Strip (450^{o}C)	200 × 50 × 3	A	0.25	0.24	0.18	0.22
		B	0.27	0.25	0.20	0.24
Unbaked	200 × 50 × 3	A	0.25	0.26	0.24	0.25
		B	0.27	0.27	0.28	0.27

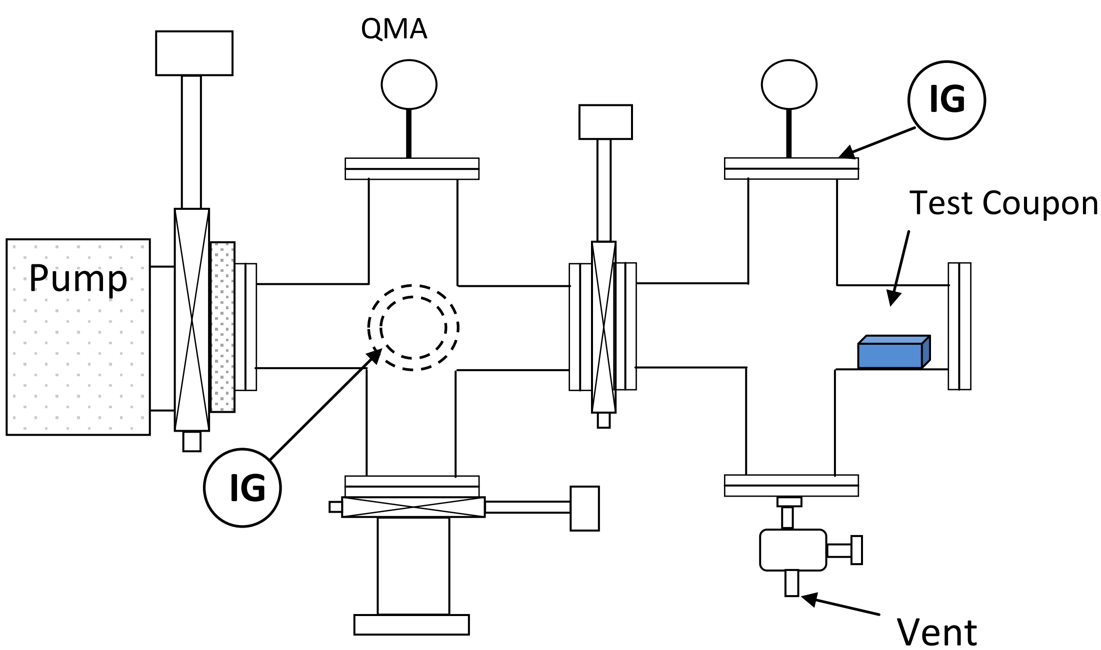

Figure2.Schematic of outgassing rate measurement system

2. The empty system is pumped as a “Blank Run” before actual experiment performed
3. The system is baked at 250° C for 50 hours.
4. The system is cooled down to room temperature.
5. The out-gassing rate of system is measured at room temperature.
6. The treated test coupon is installed in the sample chamber.
7. The system is pumped for 48 hours by Ion pump to get ultimate pressure at room temperature.
8. The out-gassing rate of system is measured at room temperature
9. The system is baked at 150° C for 48 hours.
10. The out-gassing rate of system is measured at room temperature.
11. Pressure and partial pressure in the system is recorded continues throughout the experiment.

Outgassing rate of test coupon is calculated using the equation.

$$Q_t = Q_s / A$$

Q_s = Gas load from system
A = Area of test coupon

$$Q_s = C\,(P_2 - P_1)$$

C = conductance
P_2 = Pressure of pump chamber
P_1 = Pressure of test chamber

The measured partial pressure and Outgassing rate data of the test coupon are shown in table2.

TABLE 2 outgassing rate and partial pressure data of the test coupons

Test coupon Conditions	**Total Pumping duration (h)**	**Baking duration (h)**	**Outgassing rate of test coupon (mbar l/s cm²)**		**H_2 partial pressure (mbar)**	**H_2O partial pressure (mbar)**	**N_2 partial pressure (mbar)**	**H_2 outgassing rate(mbar l/s cm²)**
			Before baking	***After baking***				
Empty chamber	150	100 h at 250°C	4.71×10^{-12}	3.64×10^{-12}	2.77×10^{-09}	4.98×10^{-10}	9.94×10^{-10}	7.4×10^{-11}
With Unbaked coupon	96		7.53×10^{-10}	5.77×10^{-10}	3.31×10^{-09}	7.44×10^{-10}	1.11×10^{-09}	9.01×10^{-11}
With Unbaked coupon 12 H Air baked	96	48 h at 150°C	Not measured	1.62×10^{-10}	2.73×10^{-09}	2.75×10^{-10}	6.44×10^{-10}	7.34×10^{-11}
With Unbaked coupon 36 H Air Baked	96	48 h at 150°C	6.99×10^{-10}	1.25×10^{-10}	1.89×10^{-09}	2.60×10^{-10}	6.56×10^{-10}	5.08×10^{-11}

Partial Pressure Measurement

Partial pressure of various gas species has been continuously measured throughout the outgassing experiment. Fig. 3 shows partial pressure of Hydrogen, Nitrogen and Water in empty chamber which is the ultimate partial pressure achieved by baking of empty system at 250° C for 100 hours.

QMA scans of the chamber are shown in figures 4, 5 and 6. Figure 4 shows partial pressure ofHydrogen, Nitrogen and Water vapour inside the chamber with unbaked test coupon. Figure 5 shows partial pressure of Hydrogen, Nitrogen and Water vapour inside the chamber with 12 hours baked test coupon. Figure 6 shows partial pressure of Hydrogen, Nitrogen and Water vapour inside the chamber with 36 hours baked test coupon.

These QMA scans show improvement in partial pressures of Hydrogen, Nitrogen and Water vapour with baking.

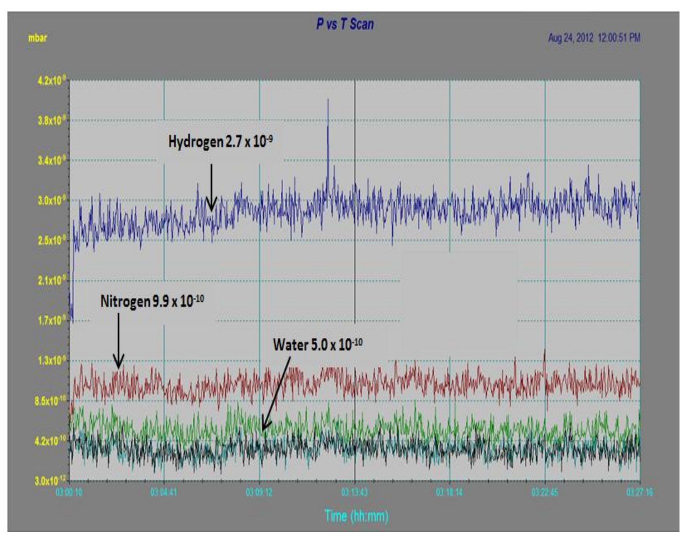

Figure3.Partial pressures scan for ≈ 15 min of empty chamber

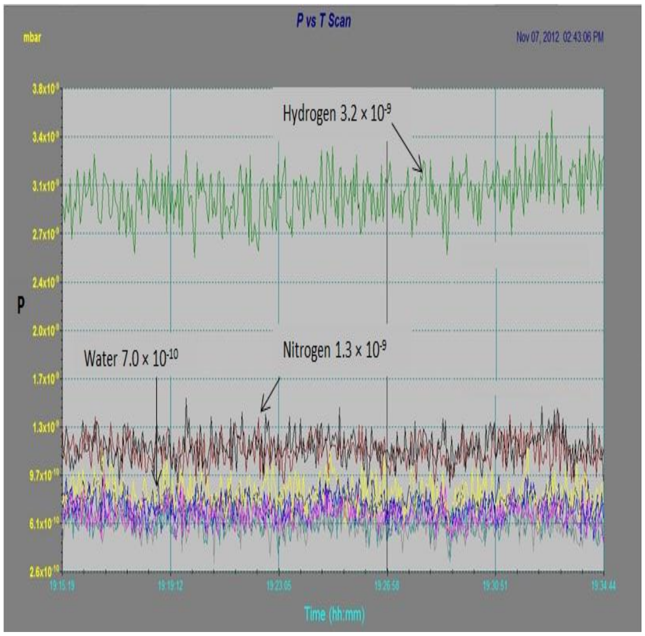

Figure4.Partial pressure of system with unbaked test coupons

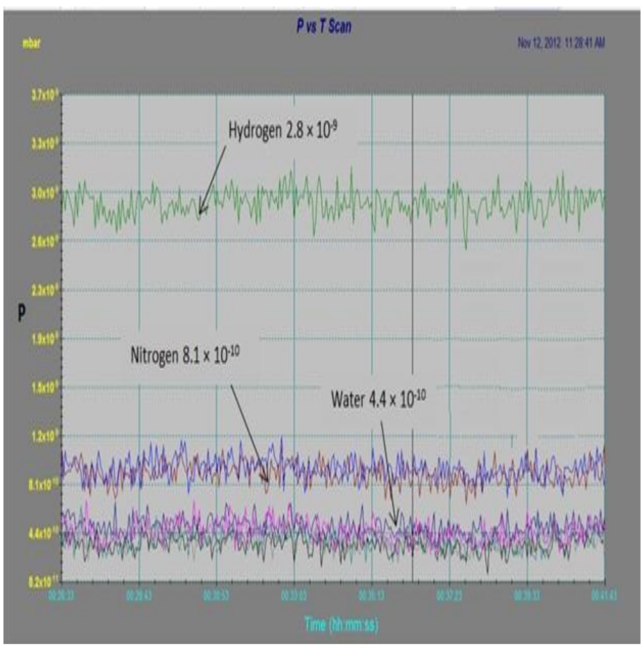

Figure 5.Partial pressure of system with 12 h air baked test coupon

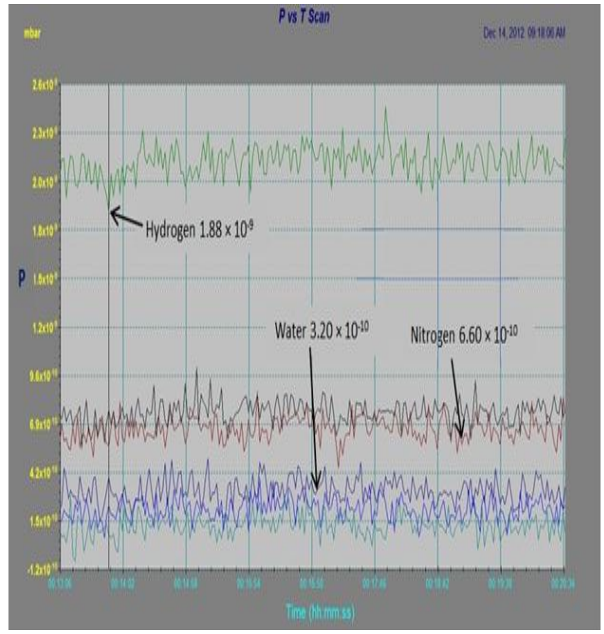

Figure6.Partial pressure of system with 36 h air baked test coupon

Conclusion

We carried out experiment to achieve acceptable out gassing rate after the treatment. Trend of the results shows that total outgassing rate and of test coupons reduces after air-baking for 36 hours. Measurement of improvement in outgassing rate is restricted due to large surface area of the measurement system compared to test coupons which is not treated.

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