Measurement of outgassing rate for GTAW welded SS304 materials

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Abstract. Outgassing plays an important role to achieve Ultra High Vacuum (UHV) and to maintaining the required vacuum level of the vessel. For a large size machines like Tokamak, accelerators, space simulation chambers, outgassing from the structural materials and their welding sections need to be checked during the design. Hence studies were carried out for the measurement of outgassing rate for the Gas Tungsten Arc Welding (GTAW) welded SS304 materials at OutGassing Measurement System (OGMS) at IPR Extension Lab. The system consists of two chambers, pumping chamber and sample chamber, made up of 304 grades Stainless Steel pre-air baked at 400 ^{o}C. The pumping chamber connected to a turbo molecular pump having pumping speed 240 l/s, backed by a rotary vacuum pump having pumping speed 5m^3/hour. Pumping chamber and sample chamber connected through a 100 CF flange having a circular aperture of 5.2 mm diameter. The conductance of aperture is 2.47 l/s. Bare SS304 sample & GTAW welded SS304 samples are prepared with 100mmX50mmX5mm and 95mmX55mmX5mm dimensions respectively. The base outgassing rate of the blank system is 2.34x10^{-11} mbar l/s-cm^2. The calculated outgassing rate is 3.66x10^{-10} & 4.37x10^{-10} mbar l/s-cm^2 for bare sample & welded sample. From the partial pressure analysis it has been found that hydrogen and nitrogen are in the partial level of 3.35 x10^{-9} & 2.36 x10^{-9} for bare sample and 3.14 x10^{-9} mbar & 2.33 x10^{-9} mbar for welded sample. It has been observed that, the GTAW doesn't have major effect on outgassing rate and one can use welded joint for designing a large vessel welding sections.

1. Introduction

While designing an Ultra-High Vacuum (UHV) system, designer must select materials which are having low outgassing property even if they are welded. However, many factors have to be consider to select proper vacuum materials for vacuum systems i.e. thermal conductivity, surface roughness, mechanical strength like stability & hardness, vacuum performance, easy to fabricate and joining [1]. Outgassing phenomenon can be described in terms of the rate of desorption of gas from vacuum surface. In general Outgassing is defined as the evaluation of gas molecules from the surfaces exposed to vacuum medium. Due to Outgassing phenomenon, it is hard to achieve better ultimate pressure of the vacuum system. In large vacuum machines like Tokamak which is plasma confinement experimental device [2], LIGO- Laser Interferometer Gravitational-Wave Observatory [3], Space simulation chambers, outgassing from the welded materials plays an important role for the system performance and the cost.

Outgassing occurs mainly due to some physical processes like desorption, vaporisation, diffusion and permeation. Relatively, outgassing is more important in Ultra high vacuum than the rough vacuum region. Generally, vacuum materials like stainless steel (SS304) are used in the construction of high vacuum systems as it holds good outgassing properties with higher strength.

2. System description

Outgassing measurement system consists of two chambers i.e. pumping chamber and sample chamber. In construction of OGMS, four number of 4-way crosses of Stainless steel having grade 304L are used as vacuum material which were air baked at 400 ^{o}C before assembly. Following figure 1 shows established OGMS at IPR, Extension Lab. Main components of OGMS systems are sputter ion pump; turbo molecular pump with backup of rotary pump as well as RGA is used for quantitative partial pressure estimates for different species.

Figure 1. Established OGMS at IPR, Extension Lab

There are two TMPs are used having pumping speed 240 l/s and 70 l/s. Customize 100 CF flange is connected between pumping chamber and sample chamber having 2.47 l/s orifice conductance. Exposed sample chamber surface area of the OGM system is 3021.5 cm^2 and total volume of system is 15.6 litters. Schematic diagram of OGMS system with required dimensions are show in the figure 2.

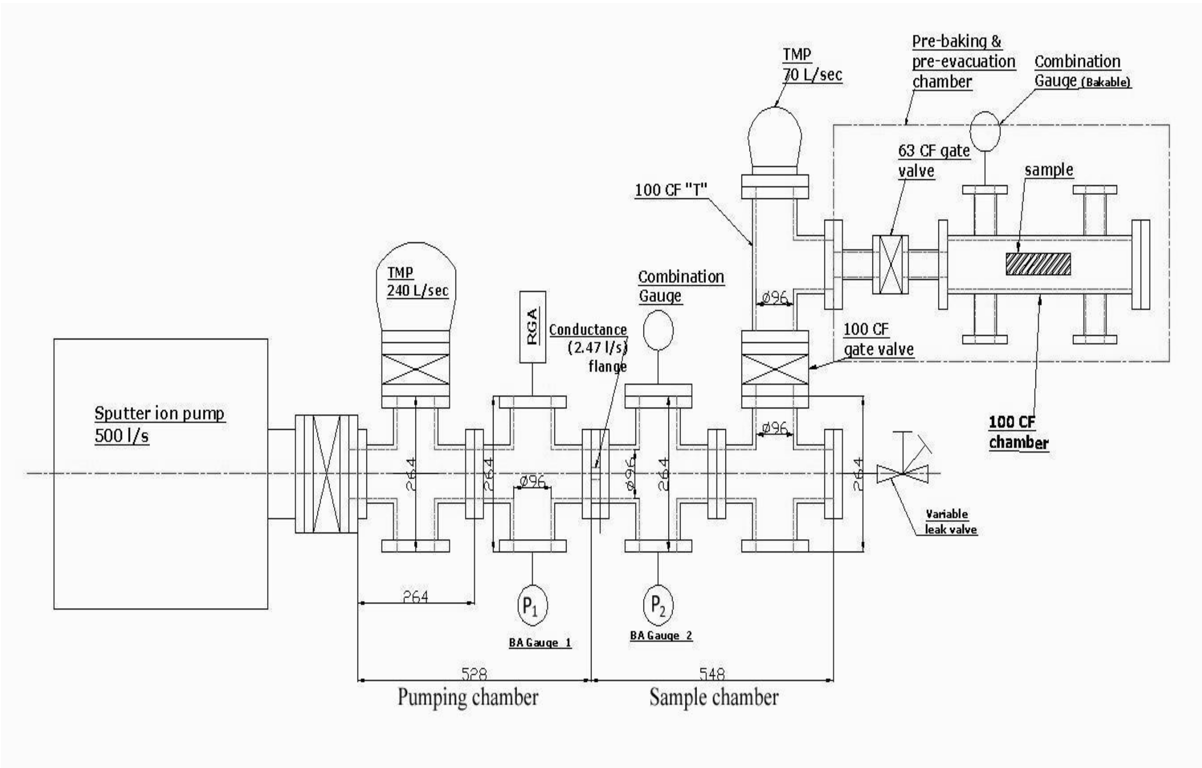

Figure 2. Schematic diagram of Outgassing Measurement system

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3. Experimental details

3.1. Sample Preparation

Two types of samples were prepared to measure outgassing rate.

i. Bare sample
ii. Welded sample

3.1.1. Bare sample preparation.

Bare sample was prepared from Stainless steel 304 material having dimension 100mmX50mmX5mm in rectangular geometry. For surface finish buffing process has done followed by hand polishing on emery paper D150. Surface roughness of sample was 0.267µm measured by surface roughness measuring tester SJ-210. For cleaning purpose, sample was cleaned by petroleum ether. After all these processes on sample, it was stored in ambient environment at room temperature and humidity was 40%. Welded sample preparation is shown below.

3.1.2. Welded sample preparation

Mostly for welding of vacuum materials Gas tungsten arc welding (GTAW), Plasma arc welding (PAW) and Gas metal arc welding (GMAW) are preferable. To insure good quality, free from weld defects, Vacuum facing parts will be welded by GTAW or PAW and other than vacuum facing part will be welded by GMAW. Good quality weld with less distortion and Arc stability is main requirement in the welding. So that in GTAW controlled pulsed DC with Direct current electrode positive, constant current source is used.

Hydrogen gas will never use as shielding gas in welding because it limits the ultimate pressure in UHV. So for GTAW, 100% Argon or Argon+Helium is most preferable for shielding gas. Here each welded sample with dimensions 95mmX55mmX5mm was fabricated from SS304 by using GTAW (Gas tungsten arc welding). Surface roughness of welded samples were 0.221 µm measured by surface roughness measuring tester SJ-210. After completion of GTAW on SS304 sample, grinding as well as buffing process were done for its surface finish as its roughness also causes outgassing measurement error. Following figures 3 & 4 shows the welded sample after grinding and its cross section with dimensions.

Figure 3. Welded sample after grinding process

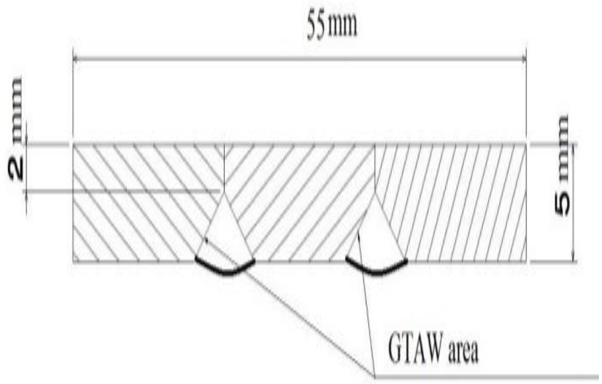

Figure 4. Cross-section of welded sample

In GTAW of SS304 samples, a tungsten electrode is used to heat the materials and argon gas to protect the weld from contaminants. SS304 material is cleaned by using a grinder and polished it. Details of welding procedure are given in table 1.

Table 1. GTAW procedure to weld SS304 samples

Sr. No.	Parameters	Measured Value
1	Characteristic of Current type	DCSP- Direct Current Straight Polarity
2	Torch nozzle material	Ceramic
3	Cup size	4 (11mm diameter)
4	Tungsten Electrode Diameter	2.4 mm
5	Filler Rod- SS 304L with	SS304L with1.6 mm diameter
6	Ampere range	50-80 A
7	Gas	Argon
8	Gas flow rate-	20 LPM

3.2. Blank run experiment of OGMS

By using throughput method measurement of blank system, , outgassing rate was carried out. In this procedure, whole system, without any sample, was pumped by using turbo molecular pump and rotary pump. The pressure P1 and P2 was measured at various intervals by B.A gauge 1 & 2 as shown in figure 2. Here outgassing rate is calculated by using throughput method as shown in equation 1.

According to throughput method, the outgassing rate is calculated by

$$Q = \frac{(P_2 - P_1)}{A} C \quad (\text{mbar l/s-cm}^2) \ldots\ldots\ldots\ldots\ldots (1)$$

Where, Q= Outgassing rate (mbar l/s-cm^2)
P_2= Sample chamber pressure (mbar)
P_1= Pumping chamber pressure (mbar)
C= Conductance of the orifice (l/s)
A= Exposed surface area of sample (cm^2)

Outgassing rate of blank system after 24 hours pumping at room temperature (RT) without baking was 2.55x10-10 mbar l/ s-cm^2. Then it was baked at 150 ^{0}C for 24 hours and then after 4 hours pumping, the outgassing rate obtained was 2.34x10-11 mbar l/ s-cm^2 which is of the same order as in literature for 304 references by E.D.Erikson (1984) [4].

Table 2. Calculated Outgassing rate of the blank system after regular interval and pumping condition

Experimental Condition	Cumulative pumping (hours)	Pumping chamber pressure (mbar)	Sample chamber pressure (mbar)	Calculated Outgassing rate (mbar l/ s-cm^2)
At RT w/o baking	01	3.48×10^{-7}	1.80×10^{-5}	1.44×10^{-8}
At RT w/o baking	05	4.5×10^{-8}	2.5×10^{-6}	2.00×10^{-9}
At RT w/o baking	24	1.12×10^{-8}	3.25×10^{-7}	2.55×10^{-10}
At 150 ^{0} C temp.	48	2.79×10^{-8}	4.28×10^{-7}	3.25×10^{-10}
At RT after baking	52	1.8×10^{-9}	3.05×10^{-8}	2.34×10^{-11}

Also partial pressure of present gases like N2, H2O, H2, Ar, O2 which were present in the system during blank run experiment measured by Residual Gas Analyzer. Table 3 shows partial pressure data of blank system at regular interval.

Table 3. Partial pressure of gases in blank system at regular interval

Experimental Condition	Cumulative pumping hours	Partial pressure (mbar)				
		N_2	H_2O	H_2	O_2	Ar
At RT w/o baking	01	1.62x10^{-8}	3.50x10^{-8}	4.56x10^{-8}	2.34x10^{-9}	3.03x10^{-9}
At RT w/o baking	05	4.30x10^{-9}	6.38x10^{-9}	8.62x10^{-9}	2.43x10^{-9}	2.44x10^{-9}
At RT w/o baking	24	3.01x10^{-9}	3.20x10^{-9}	4.15x10^{-9}	2.48x10^{-9}	2.38x10^{-9}
At 150 ^{0} C temp.	48	4.00x10^{-9}	3.43x10^{-9}	1.84x10^{-8}	2.33x10^{-9}	2.35x10^{-9}
At RT after baking	52	2.30x10^{-9}	2.12x10^{-9}	3.14x10^{-9}	2.06x10^{-9}	2.04x10^{-9}

There is a significant reduction of outgassing rate over pumping hours and it becomes almost steady after bake out. The main source of the outgassing is hydrogen after bake out, whereas water vapour is reduced after baking.

3.3. Outgassing measurement for the Stainless steel (bare) samples

After completion of blank run experiment of system, bare SS304 sample was tested in OGM System. Here during measurement of outgassing rate, test conditions were same as mentioned in above section. Calculated outgassing rate of SS304 was 4.30 x 10^{-8} mbar l/s-cm^2 after baking at 150 ^{o}C for 24 hour as shown in table 4. Moreover Partial pressure of hydrogen at room temperature after baking was 3.35 x10^{-9} mbar as shown in table 5.

Table 4. Calculated Outgassing rate of bare SS304 sample after regular interval and pumping condition

Sample Details	Experimental Condition	Cumulative pumping (hours)	Pumping chamber pressure (mbar)	Sample chamber pressure (mbar)	Outgassing rate (mbar l/s-cm^2)
Sample No./IPR/PCAC/OGMS/ SS304_Bare sample Size: 100mmX50mmX5mm Exposed Surface Area: 115 cm^2 Weight: 223.23 gm	At RT w/o baking	01	3.16x10^{-7}	1.85x10^{-5}	3.89x10^{-7}
	At RT w/o baking	05	4.32x10^{-8}	2.1x10^{-6}	4.40x10^{-8}
	At RT w/o baking	24	6x10^{-9}	3.05x10^{-7}	6.40x10^{-9}
	At 150 ^{o}C temperature	48	7.47x10^{-8}	2.4x10^{-6}	4.30x10^{-8}
	At RT after baking	52	2.52x10^{-9}	4.9x10^{-8}	3.66x10-^{10}

Table 5. Partial pressure of gases in bare SS304 sample at regular interval

Experimental Condition	Cumulative pumping hours	Partial pressure (mbar)				
		N_2	H_2O	H_2	O_2	Ar
At RT w/o baking	01	1.48x10^{-8}	3.4110^{-8}	4.21x10^{-8}	2.27x10^{-9}	2.79x10^{-9}
At RT w/o baking	05	4.13x10^{-9}	6.45x10^{-9}	9.36x10^{-9}	2.17x10^{-9}	2.13x10^{-9}

At RT w/o baking	24	$2.48x10^{-9}$	$3.03x10^{-9}$	3.0210^{-9}	$2.08x10^{-9}$	$1.9x10^{-9}$
At 150 °C temp.	48	$6.46x10^{-9}$	$4.89x10^{-9}$	$4.81x10^{-8}$	$2.19x10^{-9}$	$2.21x10^{-9}$
At RT after baking	52	$2.36x10^{-9}$	$2.25x10^{-9}$	$3.35x10^{-9}$	$2.14x10^{-9}$	$1.97x10^{-9}$

3.4. Measurement of Outgassing rate for SS304 welded sample

After the Outgassing rate measurement of bare SS304 sample and welded samples are tested in the same procedure as mentioned for the blank system & bare SS304 measurement. Cumulative pumping hour and measurement at baking temperature is also presented. There is cumulative reduction of outgassing rate over pumping. Measurement is also done at the bake out temperature 150 ^{0}C where hydrogen increases the total outgassing. Significant reduction of water vapour is observed after the bake out at room temperature measurement. Details of the calculated outgassing rates of welded sample are shown in the table 6 and the monitored partial pressure is shown in table 7.

Table 6. Calculated Outgassing rate of the welded sample after regular interval and pumping condition

Sample Details	Experimental Condition	Cumulative pumping (hours)	Pumping chamber pressure (mbar)	Sample chamber pressure (mbar)	Outgassing rate (mbar l/s-cm^2)
Sample No./IPR/PCAC/OGMS/	At RT w/o baking	01	$4.62x10^{-7}$	$2.65x10^{-5}$	$5.36x10^{-7}$
SS304_1welded Sample Size: 95mmX55mmX5mm	At RT w/o baking	05	$8.34x10^{-8}$	$3.6x10^{-6}$	$7.24x10^{-8}$
Exposed Surface Area: 119.5 cm^2	At RT w/o baking	24	$1.76x10^{-8}$	$5.05x10^{-7}$	$1.00x10^{-8}$
Weight: 234.70 gm	At 150 ^{0}C temperature	48	$7.47x10^{-8}$	$1.91x10^{-6}$	$3.77x10^{-8}$
	At RT after baking	52	$1.78x10^{-9}$	$2.3x10^{-8}$	$4.37x10^{-10}$

Table 7. Partial pressure of Gases in Experiment on welded sample

Experimental Condition	Cumulative pumping (hours)	Partial pressure (mbar)				
		N_2	H_2O	H_2	O_2	Ar
At RT w/o baking	01	$2.32x10^{-8}$	$3.38x10^{-8}$	$6.01x10^{-8}$	$3.34x10^{-9}$	$3.46x10^{-9}$
At RT w/o baking	05	$7.44x10^{-9}$	$6.63x10^{-9}$	$1.40x10^{-8}$	$2.48x10^{-9}$	$1.77x10^{-9}$
At RT w/o baking	24	$3.59x10^{-9}$	$2.88x10^{-9}$	$6.97x10^{-9}$	$2.21x10^{-9}$	$2.23x10^{-9}$
At 150 ^{0}C temp.	48	$6.23x10^{-9}$	$5.39x10^{-9}$	$2.12x10^{-8}$	$3.25x10^{-9}$	$2.68x10^{-9}$
At RT after baking	52	$2.16x10^{-9}$	$2.03x10^{-9}$	$3.19x10^{-9}$	$2.04x10^{-9}$	$1.84x10^{-9}$

4. Results & Discussion

The calculated outgassing rate is 3.66×10^{-10} mbar l/s-cm^2 for bare sample of SS304 and 4.37×10^{-10} mbar l/s-cm^2 for welded sample. From the partial pressure analysis it has been found that hydrogen and nitrogen are in the partial pressure level of 3.14×10^{-9} mbar & 2.33×10^{-9} mbar for the welded sample. It is also noted that welding shows marginally low hydrogen partial pressure. This may be because of argon is used as inert gas during GTA Welding of SS304. So due to that shielding with argon, atmospheric gases will not be able to enter in the material and it protects from the contamination of material. Figure 5 shows the comparison of outgassing rate of blank run system, bare sample and welded sample in different conditions and pumping hours.

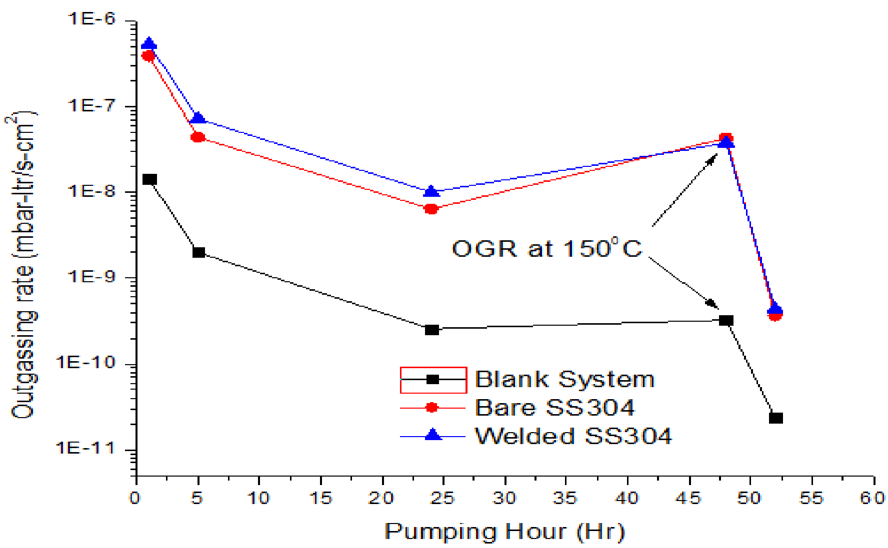

Figure 5. Comparison of outgassing rate in different conditions and pumping hours

Therefore it is concluded that, the welded sample prepared by GTAW does not have much effect on outgassing rate because it is almost equal to bare sample, so one can consider this for designing a large vessel welding sections. Of course, the result may differ depending on job preparation and workmanship of the welder.

5. Acknowledgement

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