Available online at www.sciencedirect.com

ScienceDirect

Procedia Materials Science 6 (2014) 272 – 277

Procedia Materials Science

www.elsevier.com/locate/procedia

3rd International Conference on Materials Processing and Characterisation (ICMPC 2014)

OGMS: A Facility to Measure Out-Gassing Rate of Materials

Ranjana Gangradey, Samiran Mukherjee, Paresh Panchal, Ravi Prakash N.

Institute for Plasma Research (IPR), Bhat, Gandhinagar-382428

Abstract

OGMS, the OutGassing Measurement System, is a facility to measure the outgassing rates of materials. The rate is highly important factor in vacuum science dealing with systems working at pressures below 10^{-6} mbar. In ultra- high vacuum range it plays role in system pressure at given pumping speed. For standard materials the data is available in literature but at times not under condition of specific application. Many application use very specific materials under vacuum conditions. Cryoadsorption Cryopump a project at Institute for plasma Research uses steel samples coated with activated carbon as sorbents. Various kinds of sorbents were used. Necessity to measure the outgassing rate of such unique materials established the OGMS. The OGMS facility has a known conductance of 2.46 l/s and base outgassing rate of ~ $3x10^{-12}$ mbar-ltr/s-cm^2. An ultimate vacuum of < 5 $x10^{-9}$ mbar was achieved in a sample chamber of volume ~7.5 liters. This paper describes OGMS, its calibration, experiments to find outgassing rate of steel samples and comparison with reported data to establish authenticity for new materials. It also reports results of outgassing rates of cryo-adhesives and activated charcoal coated steel samples.

Selection and peer review under responsibility of the Gokaraju Rangaraju Institute of Engineering and Technology (GRIET)

Keywords: vacuum, outgassing, sorbent, cryopump

Correspondence Email id: ranjana@ipr.res.in

1. Introduction

In Vacuum Science improper selection of material can test the nerves when it comes to operation in ultra high vacuum range. The relation Q (Gas Load) = S (Pumping Speed) x P (Pressure), shows that a lower Q will result in a lower P with any given S indicating the role of outgassing of material.

* Corresponding author.
E-mail address: ranjana@ipr.res.in

Selection and peer review under responsibility of the Gokaraju Rangaraju Institute of Engineering and Technology (GRIET)
doi:10.1016/j.mspro.2014.07.034

Outgassing is the evaluation of gas leaving per unit surface area per unit time at a specified temperature after the start of evacuation reference by Redhead P.A (2002). Outgassing gas load limits the lowest achievable pressure in a vacuum chamber and extends the time for high and ultrahigh vacuum to be reached. Criticality of the parameter is exhibited for materials that are used for example in Accelerators, Fusion reactor, Semiconductor industry etc. As already described, outgassing rate defines the pumping speed at ultimate pressure requirement of the system. For a given material it depends on surface treatments like chemical treatment, prebaking, electro polishing, grinding, horning etc. Although literature reports outgassing rates of some standard materials but at times it is not available for specific material and at specific temperature.

For the project of development of Cryopump which uses indigenously developed activated carbon sorbents, not much data is reported in literature about the outgassing rates especially for the steel substrates coated with various forms of activated carbon and glued with the help of adhesives compatible to work at cryogenic temperatures. The requirement for knowing the outgassing rate for such a compound unit is essential. It determines the ultimate pressure achieved for the Cryopump system which has base pressure below 10^{-6}mbar, a regime dominated by outgassing rate of the materials inside vacuum environment.

The outgassing measurement of samples was performed in OGMS established at Institute for Plasma Research. To establish the authenticity of results, outgassing measurement was carried out for various steel samples. The results were comparable to values reported in literature reference by E.D.Erikson et al. (1984), R J Elsey (1975). After carrying out the exercise, out gassing rate for activated carbon coated samples was carried out.

The structure of this paper describes the OGMS, its calibration with performance evaluation of the blank experimental system followed by results from cryo-adhesive and activated carbon on steel substrate.

2. Measurement technique

Outgassing rate as defined by American Vacuum is ‘the instantaneous net amount of gas leaves the material surface per unit of time’. ‘Amount of gases means effectively the ‘mass of gases. Outgassing rates of vacuum material are usually measured by the pressure rise method or the throughput method by Redhead P.A (2002). The throughput method is widely used, because the sample is maintained at a constant pressure to overcome the difficulties of re-adsorption of the pressure rise method. We used outgassing rate measurement by throughput method. Operating principal of this system is based on the technique to measure differential pressure across known conductance.

For the throughput method the outgassing rate per unit area (mbar l s-1 cm-2) is given by

$$Q = (P_2 - P_1) \cdot \frac{C}{A}$$

Where P_1 (in mbar) is the pressure in the test pumping chamber, P_2 (in mbar) the pressure at the sample chamber side of the known orifice, C (in l s-1) the conductance of the orifice; A (in cm2) the geometric surface area of the sample. A basic schematic of the OGMS system is shown in the fig.1

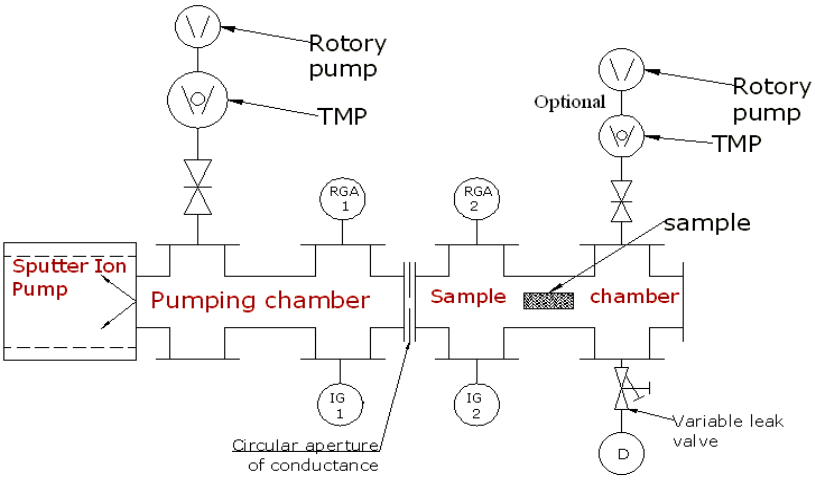

Fig. 1. Basic Schematic of OGMS

3. System Description

Two chambers (as shown in the schematic Fig.1) of OGMS are made of SS304L. Pumping system comprises turbomolecular pumping system operating in the range below 10^{-6} mbar. Ion gauges residual gas analyzers were used to measure total gas pressure and partial pressure of different gas species during testing. The orifice conductance is 2.47 lit/sec. Calibration of the conductance was performed by admitting the known gas through the leak valve into the sample vacuum chamber. The ΔP, change in pressure in time T, gave gas load (ΔP * V) / T. The gas load found by C x ΔP gives the measured gas load from the system. The percentage of error for experimental value & theoretical value of conductance is 5%. System used electro polished chamber made of SS304L having about 7.7 m^2 surface area with a volume of 15.6 liters, with aperture conductance of 2.46 liters/sec. Figure for the established system is shown below

a b

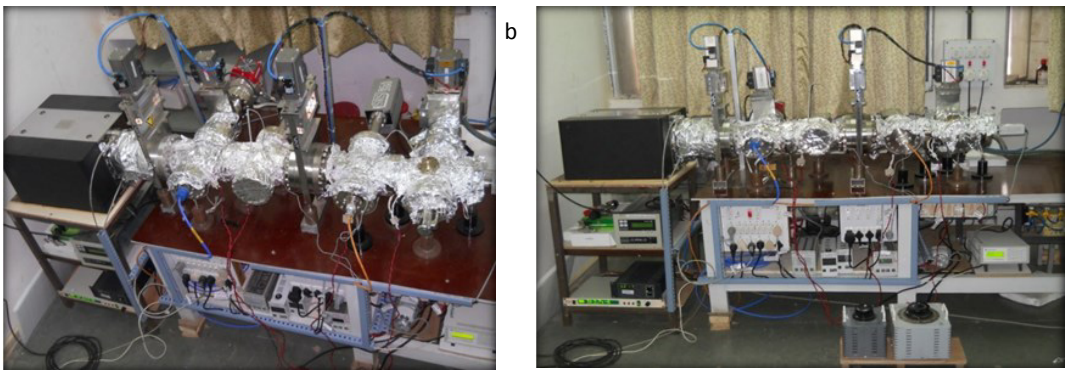

Fig. 2. Assembled OGMS facility a) “bird’s-eye” view b) side view

4. Performance enhancement of Blank system of SS304

Using the orifice method to determine the outgassing rate, the system was pumped, blank system outgassing measurement was done. After 50 hours of baking the system at 250^{0}C the system outgassing rate was 3.2x10^{-12} mbar lit/sec-cm^2 which is of the same order as quoted in literature for 304 references by E.D.Erikson (1984). Table.1 given below describes the outgassing rate of the system for different bake out and pumping conditions.

Table 1. Experimental results for the measurement of outgassing rate for the blank system for different condition

Experimental Condition for Outgasisng measurement	Outgassing rate (mbar l / sec-cm^2)
At 25 ^{0}C and pumping of 150 hrs	2.87x10^{-10}
At 25 ^{0}C after 50 hrs of Baking at 120 ^{0}C	5.24x10^{-11}
At 25 ^{0}C after 50 hrs of Baking at 160 ^{0}C	1.47x10^{-11}
At 25 ^{0}C after 50 hrs of Baking at 200 ^{0}C	9.64x10^{-12}
At 25 ^{0}C after 50 hrs of Baking at 250 ^{0}C	3.25x10^{-12}

On completing the exercise of a study on system performance with respect to blank measurements, studies were carried out for various steel samples and the outgassing rate measure in the order of 10^{-12} mbar lit/sec-cm^2, which is well acceptable for the working range.

5. Measurement of outgassing rates for various test samples

5.1. Measurement of outgassing rate for steel substrate

Before going for the measurement of outgassing rate for steel samples coated with adhesives and charcoal the outgassing rate measurement of bare steel sample was carried out. The measured outgassing rate is 2.86x10^{-11} mbar lit/sec and the details are given in table 2.

Table 2. Details of experimental results for the SS304L steel substrate

Sample details	Measurement condition	Cumulative pumping hrs	Outgassing rate (mbar l/s-cm^2)
Reference: IPR/PCAC/OGMS/S-001	At RT* w/o baking	01	2.77x10^{-08}
Type: AISI 304L plate	At RT w/o baking	05	1.67x10^{-08}
Size:100mmx 50mm x 1.5mm thickness	At RT w/o baking	24	9.84x10^{-10}
Sample Area:104.5 cm^2	At 150° C temp.	48	3.09x10^{-9}
Weight: 59.525 gm	At RT after baking	53	2.86x10^{-11}
Cleaning: with Petro. Spirit			

*RT: Room temperature

5.2. Measurement of outgassing rate for steel substrate with cryo-adhesive

Outgassing rate measurement was then carried out for steel sample coated with cryo-adhesive. Cryogenic adhesives are indigenously developed; therefore no literature database is available. To get the outgassing performance of the adhesive it is coated on a bare substrate of steel and the same studied for the measurement of outgassing rates.

Table 3. Details of experimental results for the steel substrate with cryo-adhesive

Sample details	Measurement condition	Cumulative pumping hrs	Outgassing rate (mbar l/s-cm^2)
Reference: IPR/PCAC/OGMS/ SA-001 Type: SS304L plate + Cryo-Adhesive Size: 100mm x 50mm x 1.5mm thick Sample Area:104.5 cm^2 SS sheet Weight:60.224 gm Adhesive weight:0.267 gm	At RT w/o baking	01	$3.56x10^{-6}$
	At RT w/o baking	05	$3.21x10^{-7}$
	At RT w/o baking	24	$3.69x10^{-8}$
	At 150° C temp.	48	$4.41x10^{-7}$
	At RT after baking	53	$7.17x10^{-10}$

5.3. Measurement of outgassing rate for steel substrate coated with activated carbon spheres glued with cryo-adhesive

Micro pores activated carbonaceous compounds are the sorbents suitable for adsorbing gases to cater the cryopumping. Coconut shell charcoal (of various granular size), activated carbon spheres (ACS), cloth (woven and nonwoven fabric) are some of sorbents serving the purpose. ACS offer very high surface area of the order of 1500 - 2000 square meters per gm for helium and hydrogen isotopes reference by H. Marsh (2006). ACS coated on steel substrate with cryoadhesives when cooled to the cryogenic temperatures offers very high sorption capacity which is mainly used for cryopumping. Therefore measurement of outgassing rate for such surfaces will give the proper estimation of gasload during various stages of operation and regeneration.

Table 4. Details of experimental results for the steel substrate coated with activated carbon spheres glued with cryo-adhesive

Sample details	Measurement condition	Cumulative pumping hrs	Outgassing rate (mbar l/s-cm^2)
Reference: IPR/PCAC/OGMS/SAACS-001 Type: SS304L plate+ Adhesive + ACS Size:100mmx 50mm x 1.5mm thickness Sample Area:104.5 cm^2 SS sheet Weight:58.717 gm Adhesive weight:0.510 gm SAACS-3 weight:1.284 gm	At RT w/o baking	01	$1.21x10^{-5}$
	At RT w/o baking	05	$3.26x10^{-6}$
	At RT w/o baking	24	$7.16x10^{-7}$
	At 150° C temp.	48	$2.30x10^{-6}$
	At RT after baking	53	$4.85x10^{-10}$

6. Comparison of results

As shown in the fig.3 a comparison of the measured outgassing rates are shown. It is very clear that there is

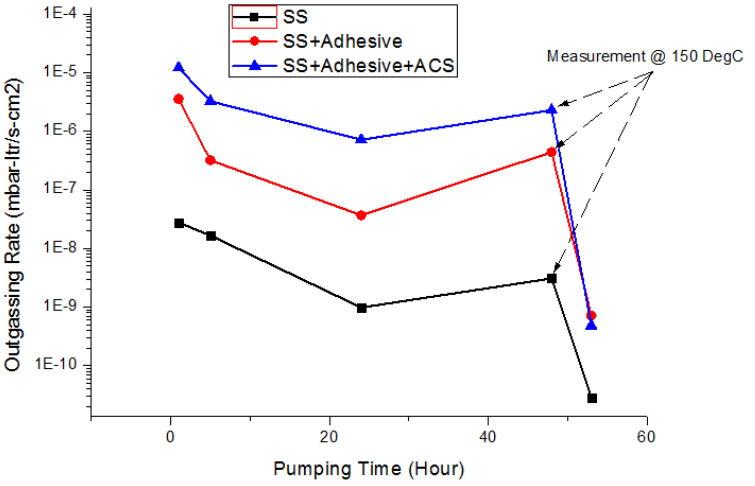

Fig. 3. Comparison of measured outgassing rates for the different samples

a subsequent rise of outgassing rates because of the adhesives and also for the ACS coating. Outgassing rates are measured at room temperatures for the initial 1, 5 and 24 hours of pumping therefore shows decrease in the outgassing rate. After that samples were heated for a period of 24hours upto 150Deg C, due to that degassing also takes place hence the measured outgassing rates shows an increased value. The measured value will help for the gas load estimation during vacuum system bake-out or cryopoump regeneration. Finally when the system cools down to the room temperatures outgassing rates are measured again at 53hours of cumulative pump down time.

Conclusion

The OGMS is producing quality results as it has been thoroughly checked for all kinds of calibrations. The system can now be used for measuring outgassing rates for any material qualified for vacuum applications. Future plan is to still improve its performance on targeting the micro level accuracies by air baking and increasing the duration of baking of samples. Result of outgassing rate of steel shows good agreement with the literatures. Studies also helped to know the outgassing rates of the materials to be used specifically for the Cryopump materials.

References

E.D.Erikson, T.G.Beat,D.D.Berger and B.A.Frazier, vacuum outgassing of various materials, J.Vac.Sci.Technol.A2.206(1984)
H. Marsh, and F. Rodríguez-Reinoso, Activated Carbon Elsevier pp1-12 (2006).
Redhead, P.A .Recommended practices for measuring and reporting outgassing data';J.Vac.Sc.Technol.A20,1667(2002)
R J Elsey. Outgassing of vacuum Materials-II, vacuum/Volume 25/Number8, 1975
P. A. Redhead, Extreme high vacuum, Proceedings of the CERN Acceleration School, Snekersten, Denmark, CERN report, edited