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Outgassing rate of different materials and its measurement methods

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Abstract—The Outgassing due to the vacuum chamber wall materials determine the ultimate pressure to be achieved in a vacuum system in the absence of leaks, internal cracks and permeation through chamber wall. Outgassing rate is more important at lower pressure, i.e. the evolution of molecules from the surfaces exposed to the vacuum. Outgassing occurs mainly due to desorption and diffusion from the internal surface of the chamber and from the bulk material respectively. Hence selection of right candidate material with a known outgassing rate is required for the vacuum system designing. This paper reports outgassing properties of different structural materials and how outgassing rate is changing in different experimental conditions. In ultra high vacuum systems, it is required that they are constructed of materials which are having the best outgassing properties. Since such systems are normally made up with prebaked the materials, and hence have to withstand high temperatures. Hence the selection of the structural materials has a significant effect on the vacuum system's performance. In view of the above outgassing rates for various structural materials are presented in a tabular form.

Index Terms—Outgassing rate, High vacuum, Measurements, Structural materials

1 INTRODUCTION

The commercially available vacuum pumps and vacuum gauges do not cover complete range of vacuum. To cover complete vacuum range, a combination of vacuum pumps and gauges are required. It is very difficult to use only one pump or gauge and costly to achieve lower pressure. Also outgassing is very important in the lower pressure. So relatively outgassing is less important in the rough vacuum region and more important in the ultrahigh vacuum region.

The designer must select structural material with low outgassing properties during constructing a vacuum vessel. However, other properties like strength, easy fabrication, thermal conductivity, elasticity, optical properties, resistance to high temperature, and cost are important according to the particular application. Structural materials which are commonly used in vacuum system include stainless steel, aluminium, copper, titanium mild steel, glass etc.

Generally high vacuum systems are constructed of steel or aluminium because of their good outgassing properties and strength. Since ultra high vacuum systems are usually made up with prebaked the materials, hence need to withstand high temperature. So stainless steel is widely used for the vacuum vessel, connecting tubes and valve bodies.

In any vacuum system that has reached equilibrium and in which leaks have been eliminated the pressure depends on the total outgassing of the system and pumping speed of pumps.

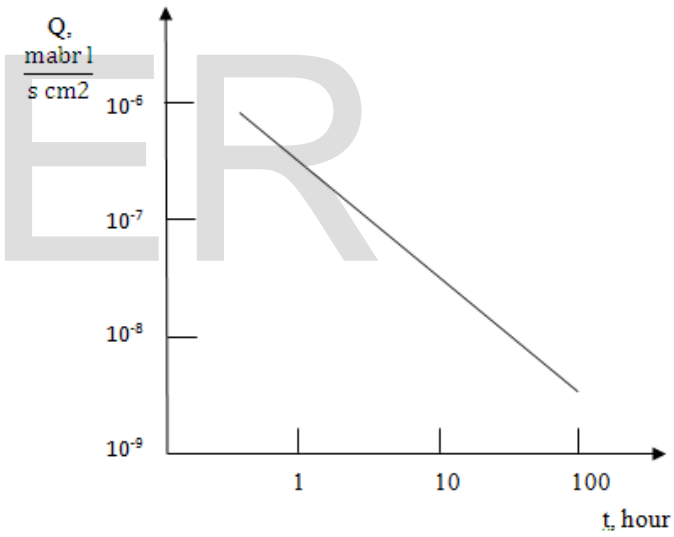

Fig. 1. Typical outgassing rate plot

$$p = \frac{Q}{S} mbar \quad (1)$$

Where Q= Outgassing rate (mbar l s-1)
S= pumping speed (l s-1)

Above formula is true only for the discrete part of the system where molecular flow prevails and where one can consider a volume into which gas is evolved from surfaces, and out of which gas flows due to pumping. But this formula is not applicable where large temperature differences are exist, e.g. near cryo surfaces of a cryopump.

In general the pressure in a pumped vacuum system will decrease with time due to the outgassing rate of the materials, reducing as gas is removed. This is because the rate depends on the surface coverage or on the concentration of gas dis-

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solved in the material. Figure 1 shows a typical plot of outgassing rate against time.

2 Specific Outgassing Rate

Outgassing is described in terms of the rate of desorption of gas from a vacuum surface. The measured (or net) outgassing rate is the difference between the intrinsic outgassing rate (of the component) and the rate of re-adsorption on the surfaces of the test chamber [1]. The specific outgassing rate defined as the total gas load generated per unit time due to gas desorbing from a vacuum surface due to the temperature of the surface per unit area of desorbing surface. It is represented here by q_{th}.

$$Q_{th} = q_{th} \bullet A \quad (2)$$

Where,

Q^{th} = Total outgassing rate (mbar l s^{-1})

A =Area of the desorbing surface (m^2)

3 Mechanism of Outgassing

It has been recognised for many years that outgassing process is controlling the lowest achievable pressure and gas composition in vacuum chamber at high and ultra high vacuum systems [2, 3]. The mechanisms to encourage outgassing are multiple.

I. Desorption: Desorption is simply the release of gas from surface. The atoms/molecules that later make up the gas can reach the surface by a variety of routes. Desorption is also the final stage of the diffusion and permeation processes.

II. Vaporisation: Vaporisation is the phase transition of material the vapour-pressure of a solid material, which increases with temperature, is the partial pressure of the vapour in thermodynamic equilibrium with its solid phase. Materials which have a high vapour pressure at the operating temperature of the chamber should not be used inside a vacuum chamber - these include: alloys containing zinc, lead, cadmium, selenium, sulphur and phosphorus.

III. Gas diffusion: Gas molecules are dissolved into the bulk of a material during its production and while it is exposed to air. If a surface is exposed to a lower pressure (e.g. it is inside a vacuum chamber), the dissolved gases will diffuse to this surface, where they can be desorbed.

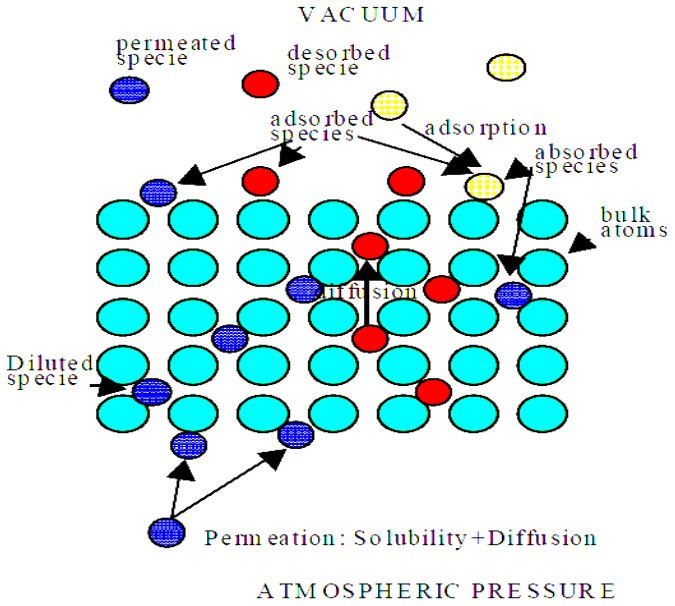

Fig. 2. Mechanisms of outgassing

IV. Gas permeation: Permeation is the mechanism by which gas passes through a solid wall from atmosphere. It is a three-step process consisting of: adsorption onto the outer wall; diffusion through the bulk material; desorption in vacuum from the inner wall.

4 Methods to Measure Outgassing Rate

Following methods are used for measuring outgassing rate. An experimentalist must choose the most suitable method for particular application and obtaining all outgassing data.

4.1 Throughput method

In this method vacuum chamber is connected to a pumping system through an orifice with conductance C. The pressure (p) in the chamber will be large relative to ultimate pressure of the system [4]. For a given gas, net pumping speed will be independent of pressure and speed of pumps (in molecular flow region). Also it will be different for the various gas components in a gas mixture under molecular flow condition. The net pumping speed can be computed from conductance formula, calculated by Monte Carlo methods or view factor analysis or measured.

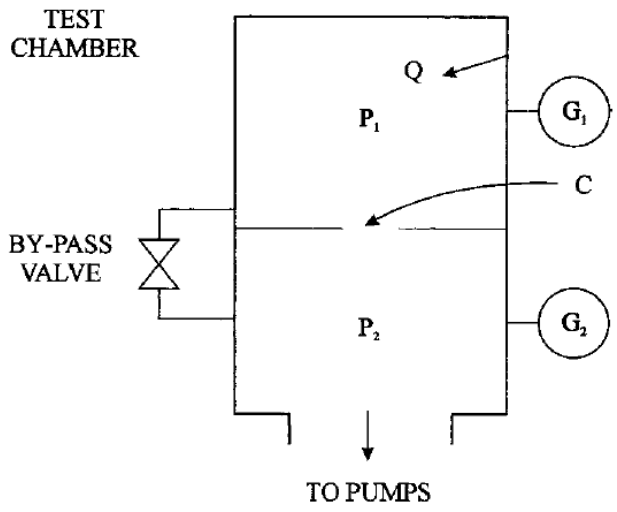

Fig. 3. Schematic diagram of the system used to measure outgassing rate by the throughput method.

In the above figure,
Q is the outgassing flux Q= q/A, where q= specific Outgassing rate A= geometric surface area of the desorbing surface. The two vacuum gauges G1 & G2 are used to measure the pressure drop (P1-P2) across the conductance C.
The orifice should be large enough i.e. ten times the pumping speed of the gauge if the latter has an appreciable pumping action for the gases liberated by the sample (e.g. an ionization gauge).
According to this method, Outgassing rate per unit area is given by

$$qA = (P1-P2)\frac{C}{A} \text{ (mbar l s-1 cm-2)} \quad (3)$$

The outgassing per unit mass is

$$qm = (P1-P2)\frac{C}{M} \text{ (mbar l s-1 gm-1)} \quad \ldots\ldots\ldots\ldots\ldots (4)$$

Where, P1= Test chamber pressure (mbar),
P2= Pressure at the pump side of orifice (mbar),
C= Conductance of the orifice (mbar l s-1)
A= Geometric surface area of sample (cm2)
M= mass of the sample (gm)
Now, if P2 << P1 then above Eqs. Become

$$qA = \frac{P1\,C}{A} \quad \ldots\ldots\ldots\ldots\ldots (3a)$$

&

$$qm = \frac{P1\,C}{M} \quad \ldots\ldots\ldots\ldots\ldots (4a)$$

4.2 Conductance modulation method

The conductance modulation method is a variant of the throughput method where the conductance of the pumping orifice is modulated [4, 5]. Here the known conductance of the orifice is modulated by the following ways,

(a) changing the opening of an iris diaphragm[4],
(b) exposing orifices of different size in a rotary disk, or
(c) by changing the separation of a circular plunger from a large circular opening in an annular disk

the latter method is outlined schematically in Fig. 4. The outgassing rate can be found from the test chamber by measuring the pressure P1 & P2 when the conductance is changed from C1 to C2 by moving the plunger from position 1 to 2. Outgassing rate of the sample is calculated by subtracting the blank system outgassing rate.

The outgassing rate per unit area is given by this method is,

$$qA = \frac{(P1-Pp)\,S1}{A} = \frac{(P2-Pp)\,S2}{A} \quad \ldots\ldots\ldots\ldots\ldots (5)$$

Where, P1 & P2= Pressure in the test chamber when plunger is in position 1 & 2 respectively,
S1 & S2= Effective pumping speed of the orifice when plunger is in position 1 & 2 respectively,
A= Surface area of the test chamber,
Pp= pressure in the pump chamber.
Now,

$$\frac{1}{S1} = \frac{1}{C1} + \frac{1}{Sp} \;\&\; \frac{1}{S2} = \frac{1}{C2} + \frac{1}{Sp} \quad \ldots\ldots\ldots\ldots\ldots (6)$$

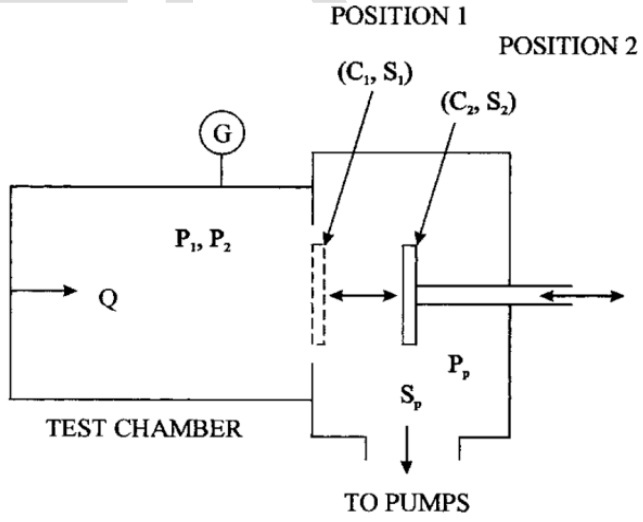

Fig.4 Schematic diagram of the system used to measure outgassing rate by Conductance modulation method.

Where, Sp= Pumping speed of the pump,
C1 & C2= Conductance of the orifice when plunger is in position 1 & 2 respectively
Then, provided that pressure in the pump chamber, Pp re-

mains unchanged on moving the plunger, the outgassing rate per unit area is given by

$$qA = \frac{P1-P2}{A\left(\frac{1}{C1}-\frac{1}{C2}\right)} \text{ (mbar l s-1 cm-2) } \ldots\ldots\ldots\ldots\ldots (7)$$

Where, A= geometric surface area (cm2)

The outgassing rate per unit mass is given by,

$$qM = \frac{P1-P2}{M\left(\frac{1}{C1}-\frac{1}{C2}\right)} \text{ (mbar l s-1 g-1) } \ldots\ldots\ldots\ldots\ldots (8)$$

Where, M= Mass of the sample (kg)

4.3 Two-path method

Another variant of throughput method is the two-path method. This method is especially suitable for the very low outgassing rate measurement [6]. It also has the advantage that the x-ray limit and outgassing rate of the gauge are cancelled out by the difference method used. The outgassing rate of the test chamber is determined from the difference in outgassing rates, measured by the throughput method, when the gas flow is switched between two paths.
A schematic diagram of the measurement system for the two-path method is shown in Fig. 5. The test chamber is pumped through one of the two pumping paths by opening valve Va or Vb during the measurement.
Here the outgassing rate is measured by opening valve Va or Vb. Then the corresponding measured outgassing rates are qa & qb. And the outgassing rate of the test chamber is given by the difference of the two measured outgassing rate, i.e q= qa-qb.

During the measurement, P2 remains constant because P2 is determined by the total gas flow rate of the system (Q+Qa+Qb) & the pumping speed of the pumps, i.e P2a=P2b.

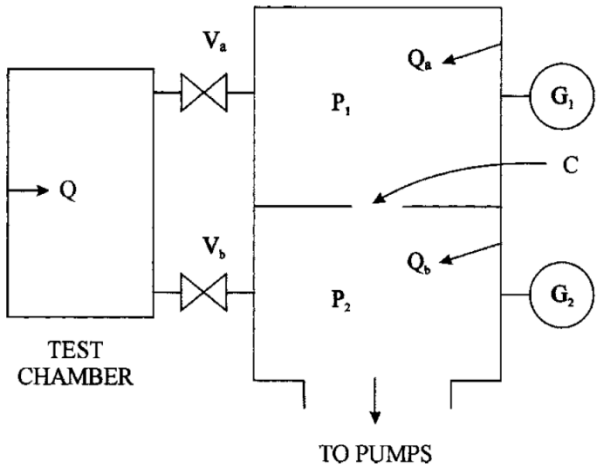

Fig. 5 Schematic diagram of the system used to measure outgassing rate by two path method.

Thus the outgassing rate per unit area of the test chamber is given by

$$qA = \frac{C}{A}\{(P1a-P2a)-(P1b-P2b)\} = \frac{C}{A}(P1a-P1b) \ldots\ldots\ldots\ldots\ldots (9)$$

$$qA = \frac{C}{A}(P1a-P1b) \text{ (mbar l s-1 cm-2) } \ldots\ldots\ldots\ldots\ldots (10)$$

And the outgassing rate per unit mass is,

$$QM = \frac{C}{M}(P1a-P1b) \text{ (mbar l s-1 g-1) } \ldots\ldots\ldots\ldots\ldots (11)$$

Where C =orifice conductance (mbar l s-1)
M is the mass of the sample (gm)
A is the geometric surface area (cm2)

The pressures P1a and P1b are measured by the same gauge G1 so that the residual current of the gauge (x-ray and electron stimulated desorption effects) is cancelled in the term (P1a-P1b). The pressure increase due to outgassing from the gauge is also cancelled.
The two-path method can be used to measure very low outgassing rates, for example, with an orifice conductance of 6x10-3 m3 s-1. The lowest outgassing rate measurable by the two-path method has been shown to be less than 10-11 Pa m s-1; this is a factor of about 100 less than that of the conventional throughput method [6].

4.4 Gas accumulation method

The gas accumulation method is also known as the rate of rise method [7]. When a test chamber is isolated from the pumps during the evacuation process, the pressure within the system will begin to increase. The pressure will be minimum at instant of isolation and then it will gradually increases, till the outgassing rate equals to surface readsorption rate. Usually at

the time of isolation, the outgassing rate will be proportional to this initial instantaneous rate of pressure rise. Pumping down of the system may be continued by opening the valve to the pumps before the pressure has increased significantly. This cycle is repeated at suitable intervals for obtain sufficient data to plot outgassing rate as a function of pump down time.

To measured very low outgassing rate, the test chamber may be pumped to its ultimate pressure before the isolation valve is closed and the rate of pressure rise measured.

In the gas accumulation method, the outgassing rate is calculated from the following equations:

$$q_A = \left(\frac{dP}{dt}\right)_{t=0} \frac{V}{A} \text{ (mbar l s-1 cm-2)} \quad \ldots\ldots\ldots\ldots\ldots\ldots (12)$$

the outgassing rate per unit mass is

$$q_M = \left(\frac{dP}{dt}\right)_{t=0} \frac{V}{M} \text{ (mbar l s-1 g-1)} \quad \ldots\ldots\ldots\ldots\ldots\ldots (13)$$

Where, $\left(\frac{dP}{dt}\right)_{t=0}$ = the initial rate of press. Rise on isolation from the pumps (mbar s-1)
V= Volume of the test chamber (l)
A= geometric surface area of the sample (cm2)
M= mass of the sample (gm).

4.5 Mass- loss measurements

The obtaining of outgassing data by mass-loss measurements is often referred as weight-loss measurements [8]. This method involves the measuring of the loss in mass of a material over specific time interval and at the recommended temperature. Continuous recording is preferred as the results give instantaneous rate measurements. A schematic diagram of the measurement system for the mass loss method is shown in Fig. 6.

The mass loss measurements are made by using a continuous type of mass evolution of the sample while under continuous exposure to surface to vacuum. This is necessary because intermittent air weighing the exposed sample to possible moisture sorption and handling contamination. When vacuum microbalances are employed, care must be taken to evaluate thermal transpiration effects

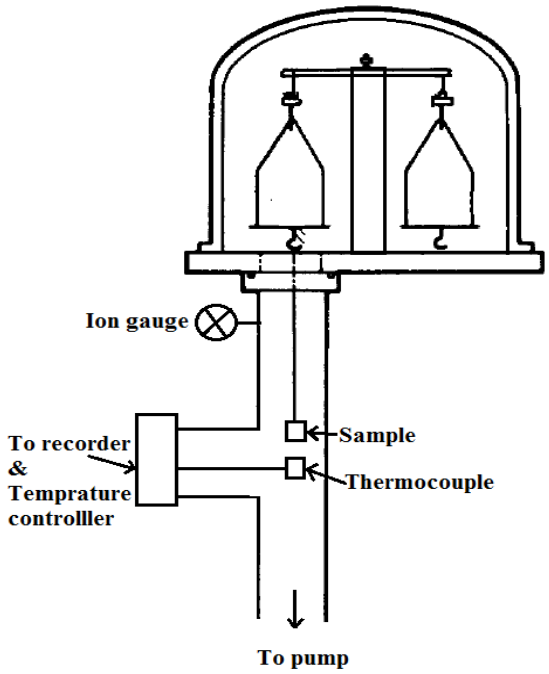

Fig. 6 Schematic diagram of the system used to measure outgassing rate by mass loss method

5 Conclusion

It should be noted that the outgassing of materials and its mechanism are not well understood and its complex.
It is observable from the results of outgassing tests published by different workers (see Table 1) that different outgassing rates have been obtained for structural materials evidently the same. Some of this variation can be attributed to differences in the methods measurement but here most of the variations are due to differences in sample conditions at the start of the experiments. The initial state of the samples depends on manufacturing process, pre-treatment and storage.

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