Design and Performance of Differential Pumping System of Coating Unit

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Abstract. A box type coating unit has been developed in view of dual purpose of optical and reactive coating. The system is divided in two parts namely, substrate chamber (800mm × 800 mm × 100 mm) and gun chamber (800mm × 800 mm × 100 mm). Coating material is evaporated in the substrate chamber by traverse (270°) electron beams. Reactive gas is injected in the substrate chamber by up-stream pressure controller to reach set pressures in the range of 1×10^{-3} mbar to 1×10^{-4} mbar for gas flow rate in the range of 0-30 sccm. Traverse EB guns (10 kV, 15 kW, 2 No) are mounted inside gun chamber. The gun chamber vacuum should be better than 1×10^{-5} mbar for the operation of EB guns. Both these chambers are connected by the apertures provided on the intermediate bifurcation plate for the passage of electron beams. Through the apertures the reactive gas leaks from the substrate chamber to the gun chamber due to differential pressure. The differential pumping system consists of individual pumping modules for the substrate chamber and the gun chamber. The paper focuses upon the design of differential pumping system in view of determination of steady state differential pressures for different flow rates of reactive gas. It has been noticed that on introduction of reactive gas in the substrate chamber, the pressures in the substrate chamber and the gun chamber oscillates before converging to steady state values. Theoretically calculated values have been compared with the experimental values as design validation.

1. Introduction
Differential pumping is carried out to achieve steady state pressure differential among inter-connected volumes and also to control the pressure differential as per the process requirement. Reactive coating is one of such application where the substrate chamber pressure needs to be controlled at different set values as per the coating requirement while the gun chamber pressure is to be maintained at pressure lower than 1×10^{-5} mbar for discharge free operation of the EB gun. These chambers are connected through 15 mm × 18 mm × 2 no. apertures provided for the passage of EB. Upstream pressure controller has been connected to the substrate chamber to control the reactive gas flow rate in the substrate chamber to reach the set pressure. Individual pumping modules have been designed for the substrate chamber and the gun chamber. The pumping modules have been designed in view of achieving substrate chamber vacuum in the range of 1×10^{-3} mbar to 1×10^{-4} mbar for gas flow rate settable in the range of 0-30 sccm, keeping the gun chamber vacuum better than 1×10^{-5} mbar.

2. System Description
The coating unit consists of a box type vacuum enclosure, which is divided into two parts namely substrate chamber (800mm × 800 mm × 900 mm) and gun chamber (800mm × 800 mm × 100 mm). The chambers are isolated by a bifurcation plate. The substrate chamber mounts the substrate holder, ion cleaning module, quartz crystal thickness monitor, crucible shutter and different accessories. The
gun chamber accommodates the traverse EB guns (10 kV, 15 kW, 2 No) and water-cooled crucibles (2 No) for evaporation of coating material. Individual pumping modules are connected to the substrate chamber and the gun chamber. Vacuum system for the gun chamber consists of 12,000 lps DP backed by 175 m³/hr rotary pump. Vacuum system for the substrate chamber consists of 550 lps TMP backed by 292 m³/hr rotary pump.

Traverse electron beam (270°) passes through the rectangular aperture (15 mm×18 mm×2 No) provided on the bifurcation plate and falls on the crucible pocket. Coating material is evaporated in the crucible pocket and a conical vapor dome is generated. The substrate is exposed to this vapor dome by the substrate holder, which can impart rotation and up-down motion to the substrate. The crucible consists of multiple pockets (4 No.) for evaporation of different materials for multi layer coating. The crucible indexing mechanism can rotate the crucible and bring particular pocket in the evaporation zone. Figure 1 shows the schematic view of the system, whereas figure 2 shows the system erected and commissioned at BARC.
3. Nomenclature

- \( A = \) Total cross sectional area of apertures (cm\(^2\))
- \( A' = \) Effective outgassing surface area of substrate chamber (cm\(^2\))
- \( A'' = \) Effective outgassing surface area of gun chamber (cm\(^2\))
- \( C = \) Conductance of the aperture (lps)
- \( L_i = \) Leak rate from the substrate chamber to the gun chamber through the apertures at \( i^{th}\) time step (mbar.lps)
- \( M = \) Molecular weight of gas
- \( P^* = \) Steady state pressure in the substrate chamber (mbar)
- \( P'_i = \) Steady state pressure of substrate chamber at \( i^{th}\) time step (mbar)
- \( P'' = \) Steady state pressure in the gun chamber (mbar)
- \( P'_g = \) Steady state pressure of gun chamber at \( i^{th}\) time step (mbar)
- \( Q = \) Gas injection in the substrate chamber (mbar.lps)
- \( Q'_i = \) Net gas throughput in the substrate chamber at \( i^{th}\) time step (mbar.lps)
- \( Q'_d = \) Outgassing throughput in the substrate chamber (mbar.lps)
- \( Q'_L = \) Gas throughput in the substrate chamber due to leak and permeation of gas (mbar.lps)
- \( Q'_g = \) Net gas throughput in the gun chamber at \( i^{th}\) time step (mbar.lps)
4. Design Calculation

Initially both the substrate chamber and the gun chamber are evacuated without any gas injection to reach respective steady state ultimate pressures. Introduction of gas inside the substrate chamber results in oscillation of pressures in the substrate chamber and the gun chamber before converging to individual steady state values. The gas leak though the aperture from the substrate chamber to the gun chamber is directly proportional to the differential pressure between them. Considering a particular gas load in the substrate chamber, as the gas leaks from the substrate chamber to the gun chamber, the differential pressure between them falls immediately resulting in reduced gas throughput in the next time step. This results in higher differential pressure and consequent higher gas throughput in the next time step. This fluctuation in the gas throughput in successive time steps results in pressure oscillation in the gun chamber and the substrate chamber.

The design calculation has been carried out to find steady state pressures in both the chambers for a particular gas flow rate and time step. Based on the steady state pressures leak through the aperture is corrected in the next time step calculation to find out the steady state pressures. The calculation is continued until the steady state pressures values do not change in successive time steps. Different steady state pressure values can be obtained for different gas flow rate into the substrate chamber. Air has been considered as the flow medium.

4.1 Governing Equations:

Total gas throughput in the system is the resultant of outgassing load, leak & permeation load, and leak through the aperture. Hence governing equation for the calculation of system throughput substrate chamber and the gun chamber can be expressed as follows:

\[ Q_i^s = Q + Q_i^{sl} + Q_i^L - L_i \]  
\[ Q_i^g = Q_i^{sg} + Q_i^L + L_i \]  

Where,

\[ L_i = C(P_{i-1}^{s} - P_{i}^{g}) \]  

Pressures in the respective chambers can be calculated as follows:

\[ P_i^{s} = \max \left[ \frac{Q_i^s}{S_{eff}^{s}}, 1 \times 10^{-6} \right] \], Considering the pump can reach maximum vacuum of \(1 \times 10^{-6}\) mbar

\[ P_i^{g} = \max \left[ \frac{Q_i^g}{S_{eff}^{g}}, 1 \times 10^{-6} \right] \] Considering the pump can reach maximum vacuum of \(1 \times 10^{-6}\) mbar
Values of different variables used in the above equations have been given in table 1.

| Variable | Details | Value |
|----------|---------|-------|
| A        | 1.8 cm × 1.5cm × 2No. | 5.4 cm³ |
| A⁺       | 80,204 cm² |       |
| A⁻       | 14,252 cm² |       |
| C        | $C = 3.64 \left(\frac{T}{M}\right) \times A$ [²] considering molecular flow region | 63.58 lps |
| M        | air | 28.96 [³] |
| $Q_d$    | $Q_d = R \times A$ | 2.8×10⁻³ mbar.lps |
| $Q^g_d$  | $Q^g_d = R \times A^g$ | 5×10⁻⁴ mbar.lps |
| $LQ$     | 1×10⁻⁶ mbar.lps |       |
| $LQ^g$   | 5×10⁻⁷ mbar.lps |       |
| $R$      | 3.5×10⁻⁸ mbar.lps/cm² [¹] |       |
| $S_{eff}$| 6000 lps |       |
| $S^g_{eff}$ | 450 lps |       |
| V⁺       | 590 liter |       |
| V⁻       | 65 liter |       |
| T        | 303 K |       |

### 4.1 Determination of flow regime:
Mean free path of air at ambient temperature $\lambda = 5\times10^{-3}/\text{Pcm}$, [²] where $P$ = pressure in torr. Considering the maximum value of $P = 1 \times 10^{-4}$ torr, $\lambda = 50 \text{ cm}$. Minimum dimension of aperture width $D = 1.5 \text{ cm}$. Knudsen number $\lambda/D = 33.33 > 1$. Hence the flow regime is molecular.

### 4.2 Estimation of real leak in the substrate chamber and the gun chamber ($Q_d$ and $Q^g_d$):
Holding time curve for the substrate chamber and the gun chamber has been shown in Figure 3 & 4.

![Substrate Chamber Pressure](image-url)
It can be noticed that the pressure increases at very fast rate in the high vacuum regime of $1 \times 10^{-6}$ mbar to $1 \times 10^{-3}$ mbar range, which is predominantly due the contribution of outgassing from internal surfaces. However the rate of pressure increment decreases with time as the outgassing throughput reduces with time and pressure. In the higher pressure region (0.01 mbar to 0.1 mbar) the outgassing throughput becomes negligible and the pressure increases mainly due to real leaks. The real leaks can be determined by calculating average pressure increment in the range of 0.01 mbar to 0.1 mbar (considering the last 11 data points) from the curves shown in Figure 3 and 4, as per the following formula,

$$Q^g_i = \frac{\partial P^g_i}{\partial t} \times V^g = \frac{0.052 - 0.022}{3000} \times 590 = 59 \times 10^{-4} \text{ mbar.lps}$$

$$Q^g_s = \frac{\partial P^g_s}{\partial t} \times V^g = \frac{0.05 - 0.02}{3000} \times 65 = 6.5 \times 10^{-4} \text{ mbar.lps}$$

### 4.2 Calculation Procedure:

1. Calculate $Q^g_0$ and $Q^g_0$ considering $L_0 = 0$ from equation (1) and (2)
2. Calculate $P^g_i$ and $P^g_s$ from equations (4) and (5)
3. Calculate $L_i = C(P^g_i - P^g_s)$
4. Calculate $Q^g_i$ and $Q^g_s$ from equation (1) and (2) considering $L_i$ from step 3
5. Repeat step (2) to (4) until $P^g_i = P^g_i$ and $P^g_s = P^g_s$

Typical design calculation for gas injection of 10 sccm in the substrate chamber is described below.

#### Step-1: Estimation of initial condition:
Both the chambers are considered isolated.

Hence, $L_i = L_0 = 0$.

$Q = 10 \text{ sccm} = 0.1874 \text{ mbar.lps}$

Putting the values in equation 1, 2, 4 & 5 following values can be obtained,

$Q^g_i = 0.1874 + 2.8 \times 10^{-3} + 59 \times 10^{-4} = 0.196 \text{ mbar.lps}$

$Q^g_s = 5 \times 10^{-4} + 6.5 \times 10^{-4} = 0.00115 \text{ mbar.lps}$

$P^g_i = 4.36 \times 10^{-4} \text{ mbar}$

$P^g_s = 1 \times 10^{-5} \text{ mbar}$
**Step-2: Iterative calculation:**
Apertures between the chambers are opened. Gas flows from the substrate chamber to the gun chamber due to differential pressure.

\[ L_0 = 63.58(4.36 \times 10^{-4} - 1 \times 10^{-4}) = 0.0276 \text{mbar.lps} \]

\[ Q^I_0 = 0.196 - 0.0276 = 0.168 \text{mbar.lps} \]

\[ Q^f_0 = 0.00115 + 0.0276 = 0.0288 \text{mbar.lps} \]

\[ P^I_0 = 3.74 \times 10^{-4} \text{mbar} \]

\[ P^f_0 = 4.8 \times 10^{-6} \text{mbar} \]

\[ L_1 = 63.58(3.74 \times 10^{-4} - 4.8 \times 10^{-4}) = 0.0235 \text{lps} \]

\[ Q^I_1 = 0.196 - 0.0235 = 0.173 \text{mbar.lps} \]

\[ Q^f_1 = 0.00115 + 0.0235 = 0.0246 \text{mbar.lps} \]

\[ P^I_1 = 3.84 \times 10^{-4} \text{mbar} \]

\[ P^f_1 = 4.11 \times 10^{-6} \text{mbar} \]

Similarly,

\[ L_2 = 0.0241, Q^I_2 = 0.173, Q^f_2 = 0.0253 \]

\[ P^I_2 = 3.82 \times 10^{-4} \text{mbar} \]

\[ P^f_2 = 4.21 \times 10^{-6} \text{mbar} \]

\[ L_3 = 0.024, Q^I_3 = 0.172, Q^f_3 = 0.0252 \]

\[ P^I_3 = 3.82 \times 10^{-4} \text{mbar} \]

\[ P^f_3 = 4.2 \times 10^{-6} \text{mbar} \]

\[ L_4 = 0.024, Q^I_4 = 0.172, Q^f_4 = 0.0252 \]

\[ P^I_4 = 3.82 \times 10^{-4} \text{mbar} \]

\[ P^f_4 = 4.2 \times 10^{-6} \text{mbar} \]

It can be seen that both the substrate chamber and gun chamber pressures oscillate before converging to steady state values of 3.82\times 10^{-4}\text{mbar} and 4.2\times 10^{-6}\text{mbar} respectively. Similar design calculations have been carried out for different gas flow rates in the range of 0-30 sccm. Steady state pressure values for different flow rates for the substrate chamber and the gun chamber have been shown in Figure 5 and Figure 6 respectively.

5. **Experimental evaluation of differential pumping:**
Steady state differential pressures in the substrate chamber and the gun chamber have been demonstrated for different gas flow rates. Upstream pressure controller has been used to control the mass flow rate of reaction gas to maintain the set value of vacuum in the substrate chamber. During the process, following steps are followed in cycle:

- A capacitance manometer senses pressure in the substrate chamber. MKS make baratron capacitance manometer (model: 627B) has been used.
- The pressure is compared to the desired set point pressure in the process controller. MKS make 4-channel gas flow and pressure controller (model: 647B) has been used.
- The pressure controller commands the mass flow controller valves to open or close, changing the mass flow rates of reaction gases to vary the chamber pressure and bringing it to the desired process pressure set point. MKS make MFC (model: 1179A) has been used.

Experiments have been carried out to achieve different set pressures in the range of 1\times 10^{-3}\text{mbar} to 1\times 10^{-6}\text{mbar} for gas flow in the range of 0-30 sccm. Variation of pressures in substrate chamber and gun chamber with respect to the gas flow rate has been shown and compared with the theoretically calculated values in Figure 5 and Figure 6 respectively.
6. Observations:

It is observed from the design calculation and the experimental results plotted in figures 5 and 6, that both the substrate chamber and the gun chamber pressures vary linearly with the gas flow rate. By linear curve fitting the relation can be derived as follows:

\[ P^s = a_1 + b_1 \times Q \]  
\[ P^g = a_2 + b_2 \times Q \]

where \( a_1, a_2, b_1 \) and \( b_2 \) are constants, which have been shown in table 2.

|               | \( a_1 \)   | \( b_1 \)   | \( a_2 \)   | \( b_2 \)   |
|---------------|-------------|-------------|-------------|-------------|
| Theoretical   | 1.72×10^{-5}| 3.65×10^{-5}| 3.65×10^{-5}| 3.83×10^{-7}|
| Experimental  | 5.6×10^{-6} | 3.87×10^{-5}| 4.51×10^{-6}| 3.825×10^{-7}|
It is seen from table 2 that slope of the pressure vs. gas flow rate curves \( (b_1 \text{ and } b_2) \) are almost equal for the theoretical and experimental cases. However there is finite difference between the curve intercepts \( (a_1 \text{ and } a_2) \) as measured theoretically and experimentally. This difference can be attributed to the degassing load and real leaks in the system which is varying from the values considered in the theoretical calculations. The gun chamber is connected with multiple feed throughs for the crucible indexing mechanism, cooling water lines and electrical connections to the EB gun, which contributes to the higher real leak. However the substrate chamber shows better vacuum than the theoretical values due to ion cleaning operation under rough vacuum (about \( 5 \times 10^{-2} \) mbar) and better degassing of the substrate and internals by the substrate heater under high vacuum.

The design calculation and experimental observations confirms that the gun chamber vacuum remains in the order of \( 1 \times 10^{-5} \) mbar or better for gas flow range of 0-30 sccm, which is satisfactory in view of operation of the EB gun.

7. Conclusion:
Differential pumping module for the application of reactive coating has been designed and developed. Design procedure to calculate the steady state pressures in the substrate chamber and the gun chamber has been established. The theoretically calculated values of the pressures varying with the gas flow rate have been compared with the experimentally measured values and found comparable. It is found that on introduction of reactive gas, both the substrate chamber and gun chamber pressures oscillates before reaching the steady state values. The steady state pressures vary linearly with the gas flow rate.

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