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Theoretical Consideration for the Outgassing Characteristics of an Unbaked Vacuum System

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Abstract

Recently the outgassing rate of an unbaked vacuum chamber has been measured with a variable orifice pumping by the author, and it has been shown that the outgassing rate q obeys the power law of pumping time t as $q = q_o(t_1/t)^x$, $0.8 < x < 1.1$ and the initial outgassing rate q_o is dependent on the pumping speed S of orifice as $q_o \propto S^m$, $0 < m < 1$. This paper describes the theoretical verification for the outgassing characteristics in the light of adsorption - desorption of molecules on the wall surface and the pump-down of gas phase molecules out of the chamber.

Key words: outgassing rate, gas desorption model, sticking probability, Freundlich isotherm, Temkin isotherm, extended Temkin isotherm.

1. Introduction.

Recently the outgssing rate of an unbaked vacuum chamber has been measured with a variable pumping orifice by the author and the coworkers[1,2,3,4]. According to thier reports, three results on the outgassing characteristics are shown as follows; (1) time dependence: the outgassing rate q (Torr$*l$/s$*$cm^2) obeys power law with respect to time

$$q(t) = q_1(t_1/t)^x \quad , < x > \sim 0.959 \qquad (1)$$

where q_1 is the initial outgassing rate at time t_1, x is a constant of $0.8 < x < 1.1$. (2) pumping speed dependence: the outgassing rate q at the fixed time t obeys power law with respect to pumping parameter

$$q = C(S/A)^m \qquad (2)$$

where C is a constant, S/A is the pumping parameter which is defined as the ratio of the pumping speed S(l/s) of the orifice to the surface area A(cm^2) of the chamber, m is a constant of $0 < m < 1$. (3) transition of m: the transition of the exponent m occurs depending on the region of pumping parameter and the constant C changes accordingly. These are arranged as for the upper and the lower region of the critical pumping parameter $(S/A)_t$

$$C_1, m_1 \text{ for } (S/A) > (S/A)_t, \qquad (3a)$$

and

$$C_2, m_2 \text{ for } (S/A) < (S/A)_t \qquad (3b)$$

where $(S/A)_t = 1.12 \times 10^{-3}$($l$/s$*$cm^2) , and the magnitude of m are $m_2 < m_1$ and $m_2/m_1 \sim 1/2$.

In this paper we attempt to verify theoretically these results in the light of adsorption - desorption of molecules on the wall surface and the evacuation of gas phase molecules out of the chamber.

2. Equation of outgassing rate.

Here we consider the outgassing rate during pumping-down of a vacuum chamber. In the usual theorey of desorption-adsorption the surface covarage θ, which is defined as the fraction of occupied site on the surface, is used. Then the outgassing rate is specified by the changing rate of covarage with respect to time $-d\theta/dt$ and is expressed as the difference of the desorption rate θ/τ of molecules from the surface and the adsorption rate $Nv/(4\sigma_m)\{s_o(1-\theta)\}$

of gas phase molecules on the surface:

$$-d\theta/dt = \theta/\tau - Nv/(4\sigma_m)\{s_o(1-\theta)\}\ , \quad (4a)$$

$$\tau = \tau_0 \exp(E/RT)\ , \quad (4b)$$

where τ is the mean residence time, τ_0 is the nominal period of vibration of an adsorbed molecule, E is the activation energy of desorption, Nv/4 is the arrival rate of gas molecules on the surface per unit area per unit time, N is the number density of gas phase molecules, v is the avarage velocity of a gas molecule, σ_m is the number of molecules per unit area in monolayer covarage and s_o is the sticking probability on empty site. For reversible adsorption the surface covarage may be expressed as a function of pressure by a suitable adsorption isotherm which may be represented in general as

$$\theta = f(p) = \sigma/\sigma_m, \quad (5)$$

where σ is the number of molecules per unit area.

Using Eq.(5) ,Eq.(4a) is expressed as

$$-d\sigma/dt = \sigma/\tau - (vN/4)\varepsilon\ , \quad (6a)$$

and

$$\varepsilon = s_o(1-\theta) = s_o(1-\sigma/\sigma_m)\ , \quad (6b)$$

where ε is an effective sticking probability. In the vacuum chamber of the volume V and the surface area A, the pumping rate of gas phase molecules out of the chamber is given by

$$V(dN/dt) = A(-d\sigma/dt) - avN/4, \quad (7a)$$

$$a = (4 \times 10^3 S)/v\ , \quad (7b)$$

where $a(cm^2)$ is the equivalent surface area to the pumping speed S(*l*/s) of a vacuum pump used in the pump-down of the chamber. Since we can put approximately that $dN/dt \sim 0$ in the quasi-steady state during pumping-down of the chamber, Eq.(7a) gives the relation of

$$Q = -d\sigma/dt = avN/(4A). \quad (8)$$

By combining Eqs.(6a) and (8), we obtain an equation of the outgassing rate during pumping-down of the chamber as

$$\sigma/\tau = \varepsilon Nv/4 + Q\ . \quad (9)$$

Q and σ/τ are called usually as the net outgassing rate and the true outgassing

rate,respectively. In addition, by neglecting the term of vN/4 from Eq.(9) using Eq.(8) we obtain the final form of Q as

$$\sigma/\tau = Q(\varepsilon A/a + 1). \qquad (10)$$

If we use the time variant expression for τ introduced by Moraw[5], since the activation energy of desorption E(t) is defined as

$$E(t) = E_c + \gamma RT^{*}\ln(t/\tau_c)\ ,\ 0 \leqq \gamma \leqq 1, \qquad (11a)$$

τ is expressed by substituting Eq.(11a) into Eq.(4b) as

$$\tau = \tau_c(t/\tau_c)^{\gamma}. \qquad (11b)$$

In order to obtain a time variant solution of Q, if we differentiate Eq.(10) with respect to time, since the left hand side is expressed with

$$d(\sigma/\tau)/dt = (1/\tau)d\sigma/dt + (-\gamma/t\tau)\sigma,$$

then using Eq.(11b) for τ and by eliminating σ from the calculation, we obtain a differential equation of

$$dQ/dt = -\ [\gamma/t + \{(A/a)/(\varepsilon A/a+1)\}(d\varepsilon/dt) + 1/\{\tau(\varepsilon A/a+1)\}]Q. \qquad (12a)$$

By replacing

$$(\varepsilon + a/A) = y, \qquad (12b)$$

Eq.(12a) is simplified further as

$$dQ/dt = -\ [\gamma/t + (1/y)(dy/dt) + (a/A)(1/y\tau)]Q\ . \qquad (12c)$$

This is a basic equation to describe Q as a function of time.

3. Time dependence of outgassing rate.

In order to solve Eq.(12c), we need to know a possible function for the parameter y. If we can put the third term $(a/A)(1/y\tau)$ of the right hand side of Eq.(12c) with β which is a constant independent of time as

$$(a/A)(1/y\tau) = \beta/t^{\lambda}\ ,\ 0 < \lambda \text{ and } 0 < \beta, \qquad (13)$$

then the possible function of y to satisfy the condition of $d\beta/dt = 0$ is given by

$$(1/y)(dy/dt) = (\lambda-\gamma)/t,\ \ 0 \leqq (\lambda-\gamma). \qquad (14)$$

The solution of this equation is expressed with an integration constant K as

$$y = \varepsilon + a/A = K{*}t^{\lambda-\gamma}. \qquad (15)$$

When the outgassing rate versus time characteristics in the pump-down of the

vacuum chamber is expressed with a power law of

$$Q = \text{const} * t^{-x}, \qquad (16)$$

according to the magnitude of the exponent x the adsorption isotherm which specified the desorption process is devided into following three classes; (I) Freundlich isotherm for $1 < x$, (II) Temkin isotherm for $x = 1$ and (III) extended Temkin isotherm for $x < 1$. Therefore, as long as the general solution of Eq.(12c) is expressed by Eq.(16), we may also devide the possible solution into three isotherms according to the valid ranges of parameter values for λ and γ.

When we pay attention to Eq.(14), from the relations of Eqs.(6b) and (8) we can show that for $\gamma < \lambda$,

$$d\varepsilon/dt = (s_o/\sigma_m)Q = K(\lambda - \gamma)t^{(\lambda - \gamma - 1)} . \qquad (17a)$$

If Eq.(17a) is acceptable for a solution of Eq.(12c), the exponent of x in Eq.(16) must be expressed as

$$x = (\gamma + 1) - \lambda . \qquad (17b)$$

Eq.(17a) shows that Q becomes 0 for $(\lambda - \gamma) = 0$. In addition Eq.(17b) shows that x becomes less than 1 (i.e., $x < 1$) for the condition of $(\lambda - \gamma) < 1$ and then the time dependence of Q corresponds to the extended Temkin isotherm. Now using Eq.(14) the general solution of Eq.(12c) is expressed as,

$$Q = \text{const} * F(t,\lambda) * t^{-\lambda}, \quad F(t,\lambda) = \exp\{-\int(\beta_\lambda / t^{\lambda})dt\} , \qquad (18a)$$

where

$$\beta_\lambda = a/(AK\tau_c^{1-\gamma}), \qquad (18b)$$

and for $\lambda = 1$,

$$F(t,1) = t^{-\beta_\lambda} . \qquad (18c)$$

Therefore, for the conditions of $\gamma < \lambda$ and $\lambda \neq 1$, the exponent x becomes

$$x = \lambda - \ln F(t,\lambda)/\ln(t), \qquad (18d)$$

and for the special condition of $\lambda = 1$,

$$x = 1 + \beta_\lambda . \qquad (18e)$$

Thus, if $\gamma < \lambda = 1$ and $\beta_\lambda = 0$, $x = 1$ is shown, i.e., the time dependence of Q is described with the Temkin isotherm. In addition if $\gamma < \lambda = 1$ and $0 < \beta_\lambda$, $1 < x$ is shown, i.e., the time dependence of Q is described with the Freundlich

isotherm. For the condition of $\lambda \neq 1$, we need the numerical analysis for Eq.(18d) to estimate the magnitude of x.

In some cases, it will be possible for the condition of $\gamma = \lambda$, i.e., dy/dt=0 to exist. Then the solution of Eq.(12c) is given by

$$Q = F(t,\gamma)*t^{-\gamma}, \text{ for } F(t,\gamma) = \text{const}*\exp\{-\int(\beta_\gamma/t^\gamma)dt\} , \quad (19a)$$

where $\beta_\gamma = \beta_\lambda$ for $\gamma \neq 1$. The exponent x becomes

$$x = \gamma - \ln F(t,\gamma)/\ln(t) \text{ for } \gamma \neq 1 , \quad (19b)$$

and

$$x = 1 + \beta_\gamma, \text{ for } \gamma = 1, \quad (19c)$$

where

$$F(t,1)= \text{const}*t^{-\beta_\gamma} \text{ and } \beta_\gamma = a/(AK). \quad (19d)$$

Thus if $\gamma = 1$ and $0 < \beta_\gamma$, then $1 < x$ holds, i.e., the Freundlich isotherm is shown. Also if $\gamma = 1$ and $\beta_\gamma = 0$, then $x = 1$ holds, i.e., the Temkin isotherm is shown. For $\gamma \neq 1$ we need the numerical analysis of the right hand side of Eq.(19b) to estimate the magnitude of x.

Our interest in this paper is to discuss the time dependence of outgassing rate when the sticking probability changes with time, because the Temkin isotherm and the extended Temkin isotherm must be directly derived from the condition of $dy/dt \neq 0$, as long as the condition of $\gamma < \lambda$ holds in Eq.(14). When we put $d\theta/dt = 0$ in Eq.(4a),the expression for θ becomes the Langmuir isotherm in which the heat of adsorption is constant i.e., τ is constant. The Temkin isotherm can be derived using the Langmuir isotherm with a heat of adsorption decreasing linearly with increasing covarage, i.e.,

$$\Delta E*\theta = E_m - E(t), \quad \text{for } \Delta E = E_m - E_c . \quad (20)$$

This relation shows that the heat of adsorption E(t) is the minimum E_c at $\theta = 1$ and is the maximum E_m at $\theta = 0$. We can express this equation using Eq.(11a) as

$$1 - \theta = \gamma(RT/\Delta E)*\ln(t/\tau_c), \quad (21)$$

where the relation $\gamma(RT/\Delta E)*\ln(t_m/\tau_c) = 1$ is used. Eq.(21) shows that $\theta = 1$ at t

$= \tau_c$ and $\theta = 0$ at $t = t_m$. Since Eq.(21) gives the surface covarage as a function of time, this is different from the usual expression of surface covarage as a function of pressure which has been derived by Kanazawa[6] or Readhead[7].

If we differentiate Eq.(21), we have the expression of

$$-d\theta/dt = Q/\sigma_m = (\gamma RT/\Delta E)(1/t).$$

This is just the solution of Eq.(19a) when γ and β_γ are put to $\gamma = 1$ and $\beta_\gamma = 0$. From this result, we can say that the Temkin isotherm holds for the special condition of $\gamma = \lambda = 1$.

In addition, Eq.(21) can be put as

$$1 - \theta = \varepsilon/s_o = \gamma(RT/\Delta E)*\ln(t/\tau_c), \quad (22)$$

this relation shows that the stiking probability is a function of time. Therefore,we may consider that for the extended Temkin isotherm which holds under the condition of $\gamma < \lambda$, the coefficient γ must be transformed to a new coefficient γ^* as

$$\gamma^* = (\varepsilon/s_o)/\{(RT/\Delta E)*\ln(t/\tau_c)\} . \quad (23)$$

This coefficient is no longer constant with respect to time. Based on this new proposal, the mean residence time is also changed from the power function τ to an exponential function τ^* as

$$\tau^* = \tau_c \exp[\{\Delta E/(s_o RT)\}\varepsilon] . \quad (24)$$

If we write the equation of Q using τ^*, Eq.(12c) changes to the form of

$$dQ/dt = -[\{\Delta E/(s_o RT)\}(dy/dt) + (1/y)(dy/dt) + (1/y\tau^*)(a/A)]Q. \quad (25)$$

In order to solve this equation, if we put the third term of the right hand side as

$$(1/y\tau^*)(a/A) = \beta^*/t^{\Omega} , \ 0 < \beta^*, \quad (26a)$$

then for the condition that $d\beta^*/dt = 0$ must be satisfied, we obtain the relation of

$$\{\Delta E/(s_o RT)\}(dy/dt) + (1/y)(dy/dt) = \Omega/t . \quad (26b)$$

Then Eq.(25) is simplified as

$$dQ/dt = -[\Omega/t + \beta^*/t^{\Omega}]Q . \quad (27a)$$

The general solution of Eq.(27a) is given similarly to Eq.(18a) as

$$Q = \text{const}*t^{-\Omega}G(t,\Omega), \quad G(t,\Omega) = \exp\{-\int(\beta^*/t^{\Omega})dt\}. \quad (27b)$$

The solution of Eq.(26b) is given by

$$y = y_0 \exp(-B\varepsilon) * (t/\tau_c)^{\Omega}, \quad B = \Delta E/(s_o RT), \qquad (28a)$$

for the initial condition of $y = y_0 = a/A$ and $\varepsilon = 0$ at $t = \tau_c$, and the constant β^* is expressed as

$$\beta^* = \tau_c^{\Omega - 1}. \qquad (28b)$$

Since for the same initial condition, y of Eq.(15) can be expressed by

$$y = y_0 (t/\tau_c)^{\lambda - \gamma}, \qquad (29a)$$

by comparing exponents between Eqs.(28a) and (29a) we can show that

$$\Omega = (\lambda - \gamma)[\, 1 + (a/A)B]. \qquad (29b)$$

Therefore, if we calculate the outgassing rate Q using Eq.(26b) by putting

$$dy/dt = (s_o/\sigma_m)Q,$$

we have the expression for Q as

$$Q = (\sigma_m/s_o)\Omega(1/t)\{y/(1 + By)\}. \qquad (30)$$

If we can approximate that $By << 1$, the time dependence of Q for $\gamma < \lambda$ is expressed by $(1/t)^{1 - \Omega}$ and if $0 < (\lambda - \gamma) < 1$, $x = (1 - \Omega) < 1$ is shown, i.e., the outgassing rate Q is described with the extended Temkin isotherm. Recently Redhead [7] has discussed the pressure versus time characteristics during the pump-down of a vacuum system using the transformed function $\theta = f(p)$ from the variation of heat of adsorption with relative covarage, $E = f(\theta)$, in the Temkin isotherm. According to his conclusion the decrease of the exponent x results from the reduction of the variation of heat of adsorption ΔE. This suggestion is different from our discussion, because the exponent x in our model decreases with the increase of $(\lambda - \gamma)$.

We can also obtain the time dependence of Q for the Freundlich isotherm in the similar way using the covarage as a function of heat of adsorption but show the derivation in Appendix.

4. Pumping speed dependence of outgassing rate.

In the discussion for the time dependence of Q, we have set the initial outgassing rate constant. However, the measured outgassing rate depends on the pumping parameter from the beginning of pump-down with a variable

pumping orifice. Therefore, in order to explain the dependence of pumping speed on outgassing rate, we must describe theoretically the form of initial outgassing rate in the pump-down of the test chamber. In the experiment we first pumped the chamber with the rotary pump for the period from t = 0 up to t_0 = 0.16 h and once stopped the pump-down for a few minutes to switch the pumping line from the rotary pump to the turbomolecular pump, and then started the pump-down of the chamber with a variable pumping orifice. At the transient state in the switching of pumps the build-up of the outgassing rate from 0 to a steady value can be described from the conservation of mass as follows;

$$\text{at } t_0 = t,\ a = 0 \text{ and } Q = (\sigma/\tau)_0 - \varepsilon v N_0/4 = 0\ , \quad (31a)$$

$$\text{at } t_0 < t,\ a \neq 0 \text{ and } Q = (\sigma/\tau)_0 - \varepsilon v N/4 > 0\ , \quad (31b)$$

and time derivatives, dQ/dt and dN/dt are

$$dQ/dt = -dN/dt(\varepsilon v/4) \quad \text{and} \quad dN/dt = (A/V)Q - Nav/(4V)\ , \quad (31c)$$

where we assume for simplicity of discussion that ε and $(\sigma/\tau)_0$ are kept constant for the transient period and the change of pressure ($p = Nk_BT$) is given simply with

$$N(t) = N_0 \exp(-t/\tau_p). \quad (31d)$$

From Eq.(31c) the time dependent equation of Q is given by

$$dQ/dt + Q/\tau_W = \{(V/A)/(\tau_W \tau_p)\}N, \quad (32a)$$

where

$$\tau_W = 4V/(vA\varepsilon) \text{ and } \tau_p = 4V/(va). \quad (32b)$$

The solution of Eq.(32a) is

$$Q(t) = \{(V/A)/(\tau_p - \tau_W)\}N_0\{\exp(-t/\tau_p) - \exp(-t/\tau_W)\} \quad (32c)$$

for the condition of $\tau_W \ll \tau_p$.

By setting the initial outgassing rate as Q_1 at $t_1 = \tau_W$, we obtain

$$Q_1(\tau_W) = (N_0/\tau_p)(V/A) = \{a/(\varepsilon_1 A)\}(\sigma/\tau)_0\ . \quad (33)$$

Thus we can express the outgassing rate after the transient period using Eqs.(16) and (33) as

$$Q(t) = (a/\varepsilon_1 A)(\sigma/\tau)_0 (t_1/t)^X\ . \quad (34)$$

If we compare Eqs.(2) and (34) at a fixed pumping time, we have an important relation to explain the pumping speed dependence of Q as follows;

$$\varepsilon_1 = \varepsilon_0 (a/A)^{1-m} \quad (35a)$$

and

$$C^* = (C/k_BT)(v/4x10^3)^m = (1/\varepsilon_0)(\sigma/\tau)_0(t_1/t)^x \ . \quad (35b)$$

Therefore the final expression for the pumping speed dependence of Q becomes

$$Q(t) = C^*(a/A)^m \quad \text{for } t_1 \leq t \ , \quad (36)$$

using the constant C*. This is a scaling law which we propose here to express the dependence of outgassing rate on pumping speed in an unbaked vacuum system.

5. Transition.

The remaining explanation is for the occurence of transition of the exponent m. When the parameter value a/A of the orifice is reduced to the one of the rotary pump, the pressure given by Eq.(31d) must be given by

$$N(t) = (4/v)\{a/(\varepsilon^2 A)\}(\sigma/\tau)_0 \exp(-t/\tau_p), \quad (37)$$

then we obtain the relation of $\varepsilon = \varepsilon_0(a/A)^{1-m/2}$ and as a result, $m_2 \sim (m_1)/2$ is shown.

6. Sammary and conclusion.

In order to explain the time dependence of outgassing rate, we have first derived the basic equation of the outgassing rate as a function of time in the light of adsorption - desorption of gas molecules on the surface and the evacuation of gas phase molecules out of the chamber. The important parameters in the equation are the mean residence time, the sticking probability and the pumping parameter. We can solve the equation by using the variation of heat of adsorption for the suface covarage which is considered in the Temkin isotherm. The author proposes to obtain the time variant solution of outgassing rate that the coefficient γ in the expression for the residence time τ proposed by Moraw must be replaced with the time variant sticking probability $\varepsilon(t)$. We can show that the outgassing rate versus time characteristics which have been observed in our experiment are able to be explained with the constant coefficient $\gamma = 1$ for the Temkin isotherm of $x = 1$ and with the time variant coefficient γ^* for the extended Temkin isotherm of x

< 1. The dependence of outgassing rate on pumping speed appears at the transient state when the pump-down of the chamber is switched from the rotary pump to the variable orifice pumping. Although the initial outgassing rate of the chamber builds up in the period from the transient state to the qasi-steady state, it must be determined corresponding to the change of the pumping speed. In this situation the initial out gassing rate is determined at the balance between the adsorption rate on the wall and the pumping rate by the pumping orifice. Therefore the sticking probability of the wall plays an important role for determination of the initial outgassing rate. It is made clear that the transition of the exponent m results similarly from the change of sticking probability.

Appendix

We can also verify the outgassing rate versus time characteristics corresponding to the Freundlich isotherm. Since we can write the heat of adsorption for the Freundlich isotherm as

$$E(t) = - E_m * \ln \theta , \qquad (a)$$

using Moraw's formula for E(t); $E(t) = E_c - (\gamma RT)\ln(t/\tau_c)$, the covarage θ is given by

$$\theta = \exp(-B_1) * (t/\tau_c)^{-\gamma B_2} , \qquad (b)$$

where

$$B_1 = E_c/E_m \quad \text{and} \quad B_2 = (RT/E_m). \qquad (c)$$

By differentiating Eq.(b), we obtain the expression for the outgassing rate Q as

$$- d\theta/dt = Q/\sigma_m = \exp(-B_1) * (\gamma B_2/\tau_c)(t/\tau_c)^{-(\gamma B_2 + 1)} . \qquad (d)$$

Now we return to the basic equation of Q of Eq.(10), if we set dy/dt = 0 (i.e., ε = const.), then Eq.(10) is given by

$$Q = \{a/(y_o A)\}(\sigma/\tau), \qquad (e)$$

where

$$y_o = \varepsilon_o + a/A \text{ and } \tau = \tau_c(t/\tau_c)^{\gamma} .$$

Then using Eqs. (5) and (b), we can rewrite Eq.(e) as

$$Q = \{a/(y_o A)\}(\sigma_m/\tau_c)\exp(-B_1) * (t/\tau_c)^{-(\gamma B_2 + \gamma)} . \qquad (f)$$

On setting the equivalence for the exponent of power function between Eqs.(d) and (f), we obtain the relations of $\gamma = 1$ and $B_2 = a/(y_0 A)$. Therefore, we can say that the magnitude of exponent x in the power function of Q is $x = 1 + B_2$ and $1 < x$. As a result the heat of adsorption and the constant sticking probability are given as

$$E_m = RT/(x-1) \qquad (g)$$

and

$$\varepsilon_0 = (a/A)[\{1/(x-1)\} - 1]. \quad (h)$$

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