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On the Outgassing Rate versus Time Characteristics in the Pump-down of an Unbaked Vacuum System

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Abstract

In order to derive theoretically the outgassing rate versus time characteristics(Q vs t) in the pump-down of an unbaked vacuum system, a thermal desorption model in the light of adsorption - desorption of gas molecules on surface has been proposed. In this model the outgassing rate is described as a function of time using the conservation of mass equation for gas molecules in the vacuum system. In order to solve this equation, it is essential to introduce an auxiliary equation which enables to determine the surface covarage as a function of the heat of adsorption. When this auxiliary function is expressed with the variation of heat of adsorption in the Temkin isotherm and the Freundlich isotherm, the solution for the outgassing rate gives a power law $Q = \text{const.}(1/t)^x$ where x is a constant as much as 1, which is usually familiar in the pump-down of a vacuum system.

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

1. Introduction

The outgassing characteristics of the unbaked vacuum system have been investigated experimentally [1] and theoretically using the thermal desorption model [2]. Then we have proposed a scaling law, which describes the outgassing rate Q (molecules/cm^2) as the product of a function f(a/A) of pumping parameter a/A and a function g(t) of time:

$$Q = f(a/A) * g(t) \qquad (1a)$$

$$f(a/A) = C(a/A)^m, \quad 0 < m \ , a = S/S^*, \quad (1b)$$

$$g(t) = (1/t)^x, \quad x \sim 1, \qquad (1c)$$

where a is the normalized area as the ratio of the pumping speed S to the ideal pumping speed S^* (= 11.7 $l/s \cdot cm^2$ for N_2 at 293 K), A is the surface area of the vacuum system, C , m and x are constants.

In the previous report [2], we have shown that the function g(t) can be derived by solving the conservation of mass equation for gas molecules in the vacuum system, when we use the time variant expression proposed by Moraw[3] to connect the mean residence time and the surface covarage with the heat of adsorption. However, the logical description to connect these parameters and the mathematic approach to solve the conservation of mass equation are not adquate. Therefore, the additional discussion on the theoretical derivation of time variant function g(t) is described in this paper.

2. Equation of outgassing rate

Here we consider the outgassing rate during pumping-down of a vacuum chamber. In the usual theory of desorption-adsorption the surface covarage θ, which is defined as the fraction of occupied site with adsorbed molecules on the surface, is used. Then the outgassing rate is described as the time derivative of surface covarage $-d\theta/dt$ and is expressed as the difference of the desorption rate θ/τ of molecules from the surface and the adsorption rate $N\upsilon/(4\sigma_m)\{s_o(1-\theta)\}$ of gas phase molecules on the surface:

$$-d\theta/dt = \theta/\tau - N\upsilon/(4\sigma_m)\{s_0(1-\theta)\}\ , \quad (2a)$$

$$\tau = \tau_0 \exp(E/RT)\ , \quad (2b)$$

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, $N\upsilon/4$ 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, υ is the avarage velocity of a gas molecule, σ_m is the number of molecules per unit area in monolayer covarage and s_0 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

$$\theta = f(p) = \sigma/\sigma_m, \quad (3)$$

where σ is the number of molecules per unit area.

Using Eq.(3) ,Eq.(2a) is expressed as

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

and

$$\varepsilon = s_0(1-\theta) = s_0(1-\sigma/\sigma_m)\ , \quad (4b)$$

where ε is an effective sticking probability. In the vacuum chamber of the volume $V(cm^3)$ and the surface area $A(cm^2)$, the pumping rate of gas phase molecules out of the chamber is given by

$$V(dN/dt) = A(-d\sigma/dt) - a\upsilon N/4, \quad (5)$$

where $a(cm^2)$ is the area of a pumping orifice which is defined as the ratio of the conductance $S(l/s)$ of orifice to the ideal pumping speed $S^* = 11.7(l/s\cdot cm^2)$ at 20 ℃ for nitrogen. Since we can put approximately that $dN/dt \sim 0$ in the quasi-steady state during pumping-down of the chamber, Eq.(5a) gives the relation of

$$Q = -d\sigma/dt = a\upsilon N/(4A). \quad (6)$$

By combining Eqs.(4a) and (6), we obtain an equation of the outgassing rate during pumping-down of the chamber as

$$\sigma/\tau = \varepsilon \upsilon N/4 + Q . \qquad (7)$$

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 $\upsilon N/4$ from Eq.(7) using Eq.(6) we obtain the final form of Q as

$$Q = \{(a/A)/(\varepsilon + a/A)\}(\sigma/\tau). \quad (8)$$

In oder to obtain a time variant solution of Q, if we differentiate Eq.(8) with respect to time, we obtain a basic differential equation of Q as

$$dQ/dt = - [(1/\tau)(d\tau/dt)+ (1/y)(dy/dt) + (a/A)/(y\tau)]Q, \quad (9a)$$

where

$$y = \varepsilon + a/A. \qquad (9b)$$

3. Time dependence of outgassing rate

3.1 Temkin and extended Temkin isotherms

In order to solve Eq.(9a), we need to know possible functions for parameters of y(t) and τ(t). We observe usually in the pump-down of a vacuum system that the outgassing rate versus time characteristics obeys a power law of

$$Q = \text{const}*(1/t)^{x}. \qquad (10)$$

According to the magnitude of the exponent x, the adsorption isotherm which characterized the desorption process during pumping-down is devided into following three classes; (I) Freundlich isotherm for $1 < x$, (II) Temkin isotherm for $x = 1$ and (III) extended Temkin isotherm for $x < 1$. Redhead [4] has demonstrated recently that if the surface covarage θ in the Langmuir isotherm can be rewritten using the variation of heat of adsorption E in the Temkin isotherm , since θ can be expressed as a function $\theta = f(P)$ of pressure P in the vacuum chamber and as a result the outgassing term in the pump-down equation can be transformed to a function of pressure, the pressure versus time characteristics (i.e., P vs t curve) of $x \leqq 1$ can be explained. Then, he has classified the P vs t curve of $x < 1$ as the outgassing characteristics corresponding to the extended Temkin isotherm. In this study, in order to solve Eq.(9a), we

also need an auxiliary equation to describe the surface covarage θ for the variation of heat of adsorption in a suitable adsorption isotherm. We here also consider the Temkin isotherm. In the Temkin isotherm it may be considered that the heat of adsorption E(t) decreases monotonously with increasing covarage θ as

$$E(t) = E_m - \Delta E*\theta, \text{ for } \Delta E = E_m - E_c \quad (11)$$

where E_m and E_c are the maximum and the minimum of the heat of adsorption E(t) at the covarage of $\theta = 0$ and 1. Here we may consider the logarithmic function which has been proposed by Moraw[3] as a possible function of E(t);

$$E(t) = E_c + \Omega RT*\ln(t/\tau_c), \quad (12a)$$

where we assume that the valid range of coefficient Ω is limited by

$$0 \leqq \Omega \leqq 1, \quad (12b)$$

and from Eq.(2b) the characteristic times for $E(t) = E_c$ and E_m are given by

$$\tau_c = \tau_o \exp(E_c/RT) \text{ and } t_m = \tau_c \exp\{\Delta E/(\Omega RT)\}. \quad (12c)$$

Now, by substituting Eq.(12a) into Eq.(11), the surface covarage θ is expressed as

$$1 - \theta = (RT/\Delta E)*\ln(t/\tau_c)^{\Omega}. \quad (13)$$

Thus, using Eqs.(4b)and (13) the sticking probability ε is expressed as

$$B\varepsilon(t) = \ln(t/\tau_c)^{\Omega}, \quad (14a)$$

where

$$B = \{\Delta E/(s_o RT)\}. \quad (14b)$$

In addtion, using Eq.(12a) we can transform the mean residence time τ of Eq.(2b) as

$$\tau = \tau_{c*}\exp(B\varepsilon) = \tau_c(t/\tau_c)^{\Omega}. \quad (15)$$

The Eq.(15) shows that the mean residence time of an adsorbed molecule on the wall during pumping-down of a vacuum system is expressed with an increase function of sticking probability, when the

readsorption of molecules on the wall is not negligible. Then let us solve Eqs.(9a) using Eqs.(14a) and (15). Since the sum of first and second terms in the blacket of right hand side of Eq.(9a) can be replaced as

$$d(1/y\tau)/dt = -(1/y\tau)[(1/y)(dy/dt) + (1/\tau)(d\tau/dt)], \quad (16)$$

the integration is given by

$$\ln Q = \ln (1/y\tau) - (a/A)\int (1/y\tau)dt. \quad (17)$$

The second term of right hand side is also replaced as

$$- (k/\Omega)\lambda I[z,\Omega], \quad (18a)$$

where

$$k = (aB)/A\ ,\ \lambda = [\exp(k)]^{(\Omega-1)/\Omega} \text{ and } z = \{\exp(k)\}(t/\tau_c)^{\Omega}. \quad (18b)$$

$$I[z,\Omega] = \int z^{(1/\Omega-2)}[1/(\ln z)]dz\ . \quad (18c)$$

Since this integration $I[z,\Omega]$ is given by for the condition $\Omega \neq 1$

$$I[z,\Omega] = \ln\{\ln z\} + (1/\Omega - 1)\ln z + [(1/\Omega - 1)\ln z]^2/4 + \text{-----},$$

then if we take as dominant terms by the second term, the general solution is given as

$$Q = \text{const.}(t/\tau_c)^{-x} * (\varepsilon + a/A)^{-(1+\beta)}, \quad (19a)$$

where

$$x = \Omega + \beta(1 - \Omega) \text{ and } \beta = (k\lambda)/\Omega. \quad (19b)$$

This result shows that although the sticking probability increases logathmically with time and as long as the term $(\varepsilon + a/A)^{-(1+\beta)}$ changes at most within factor of 2 in the range of pumping time from t = 1 to 100 h , the time dependence of Q is determined mainly by the $(1/\tau)^{x/\Omega}$ law. Cosequently, we can expect that the time dependence of Q corresponds to the Temkin isotherm for the condition of x = 1, and corresponds to the extended Temkin isotherm for that of $x < 1$. The change of Ω depends on the variation of heat of adsorption. This point agrees with the result of numerical analysis made by Redhead[4] for explaining the extended temkin isotherm. He has suggested that in the Temkin isotherm when the minimum heat of adsorption E_c increases and the energy width ΔE becomes gradually small, the pressure versus time characteristics can be

explained by the extended Temkin isotherm. In addition if we consider the condition of dy/dt = 0 and y = (a/A) in Eq.(9a) , the integration I[z,Ω] becomes

$$I[z,\Omega] = \{1/(1 - \Omega)\}z^{(1 - \Omega)}, \qquad (20a)$$

then the solution is given by

$$Q = \text{const.}(\tau_c/t)^{\Omega}\exp[- \{1/(1 - \Omega)\}(t/\tau_c)^{(1 - \Omega)}]. \quad (20b)$$

This solution is the one derived by Moraw. If the exponential function decreases rapidly with time, Q does not obey a power law of time.

3.2 Freundlich isotherm

We can also show the outgassing rate versus time characteristics corresponding to the Freundlich isotherm.The heat of adsorption for the Freundlich isotherm is given by

$$E(t) = - E_m * \ln \theta . \qquad (21)$$

Then, using Eq.(12a) if we write E(t) as

$$E(t) = (\Omega RT) * \ln(t/\tau_0). \quad (22)$$

The surface coverage of Eq.(22) is expressed as

$$\theta = (t/\tau_c)^{-\Omega B^*} , \qquad (23a)$$

and the mean residence time is

$$\tau = \tau_0(t/\tau_0)^{\Omega} , \qquad (23b)$$

where

$$B^* = (RT)/E_m . \qquad (23c)$$

Using Eq.(23a),we can express the sticking probability as

$$\varepsilon = s_0(1 - \theta) = s_0\{1 - (t/\tau_0)^{-\Omega B^*}\} . \quad (24)$$

Since Eq.(9a) is expressed as

$$\ln Q = \ln [1/\{(\varepsilon + a/A)\tau)\}] - (a/A)\int dt/\{(\varepsilon + a/A)\tau)\} , \quad (25a)$$

by substituting Eqs.(23b) and (24) into the above equation, the integral of the right hand side becomes, by putting $z = (t/\tau_0)$,

$$- (a/A)\int dz/[z^{\Omega}\{s_0(1 - z^{-\Omega B}) + a/A\}] . \quad (25b)$$

By letting $Y = z^{\Omega B} - C$, $1/C = a/(As_0) + 1$, Eq.(25b) is transformed as

$$(a/As_0)\{1/(\Omega B)\}(1/C)\int \{dY/[Y *(Y + C)^{(\Omega - \Omega B)/\Omega B}]. \quad (25c)$$

If we can assume that $(\Omega - 1)/(B\Omega) = 1$ or $\Omega \sim (1 + B)$, (26a)

we can integrate Eq.(25c) and have the solution of Q as

$$Q = \text{const.}(\tau_0/t)^{\Omega}(1/y)^{1 + (a/As_0)(1/B\Omega)} \quad . (26b)$$

Thus if the term $(1/y)^{1 + (a/As_0)(1/B\Omega)}$ changes slowly with time, we can say that the outgassing rate obeys the power law to time as

$$Q \sim (1/t)^x, \quad x = \Omega = 1 + B. \quad (27)$$

Since Ω is a number slightly larger than 1, thus under the assumption of Eq.(26a) Freundlich isotherm predicts the characteristics of Eq.(27).

From the condition for the exponent x, the heat of adsorption is given by

$$E_m = (RT)/(x - 1). \quad (28)$$

4. Summary

Based on the thermal desorption model in which the readsorption of gas molecules from gas phase on surface is taken into account, we have derived a basic equation to describe the outgassing rate as a function of time. The main parameters used in this equation are the mean residence time τ, the sticking probability ε and the pumping parameter a/A. In order to solve analytically the outgassing equation, we need to know possible time variant functions for two parameters of τ and ε. Since these two parameter are usually expressed as a function of heat of adsorption in a suitable adsoption isotherm, we attempt to express the heat of adsorption as a function of time using the Moraw's formula. Then these parameters are successfuly expressed as time variant functions. For the analysis of the outgassing equation when the variation of heat of adsorption in the Temkin isotherm is taken into account, a typical solution shows that the outgassing rate obeys the power law to time $Q \sim (1/t)^x$ which is a familiar characteristics observed in the pump-down of an unbaked vacuum system. The time dependence of the outgassing rate is mainly determined by the inverse of the mean residence time $(1/\tau)$ but the contribution of the pumping action on that is

not negligible, and the change of the sticking probability on that is rather very weak. For the variation of heat of adsorption in the Freundloch isotherm, the solution shows also the time dpendence of $1 < x$ in the power law of the outgassing rate. Thus we can conclude that the gas desorption model presented in this study is useful to describe the outgassing rate versus time characteristics.

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