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Some problems of the vacuum technique development

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Abstract. In this work the problems of obtaining ultra-high vacuum without warming of the vacuum chamber are discussed. Numerical calculations have shown the possibility of obtaining within one hour the degree of coverage 10^{-2} in conventional vacuum chambers and $2 \cdot 10^{-3}$ in the chambers of special shape.

1. Introduction

The heyday of vacuum technique in the second half of the last century is over, but the possibilities of evolutionary development still exist. They are associated with the increasing use of vacuum technique in industry and scientific research. It is possible to identify ways of its further development on the basis of currently existing problems. The list of these problems in a large part is subjective and depends on the area of interest of the user. Let us consider some of them.

Vacuum physics offers a lot of methods of vacuum systems calculation, but it cannot provide the necessary for practice accuracy. This leads during the design of vacuum systems to do the calculations with a large margin, leading to a significant rise in the cost of the projects.

In the technique of producing vacuum pressure limit of scroll pumps is significantly worse than that of rotary vane oil pumps. Turbomolecular pumps typically can't have a direct exhaust into the atmosphere and have reserves to improve their pressure limit.

Combined vacuum converters have poor metrological characteristics and lower limit of measurement. Metrological basis for calibration of vacuum gauges and leak detectors needs significant modernization, and centralized verification and calibration of analyzers of partial pressure is absent.

Leak detectors have become so expensive devices that only large firms can afford them. The mass-spectrometer leak detectors have problems with the measurement of flows of more than 10^{-4} $m^3 \cdot Pa/s$. Use of partial pressures analyzers can be in addition addressed not only to measuring partial pressures, but also for the problem of leakage detection. Moreover, the cost of partial pressure analyzers is several times less than the cost of leak detectors. However, during the direct use of partial pressure analyzers as leak detectors, there are certain difficulties with the pumping of helium.

2. Obtaining ultra-high vacuum without warming of the vacuum chamber

Let us consider the problem of obtaining ultra-high vacuum without warming of the vacuum chamber. Requirement for the ultra-high vacuum is obtaining of clean surfaces free from adsorbed gases. In the middle of the last century it was believed that to obtain ultra-high vacuum the chamber should be warmed up to 450 °C. This value was associated with the softening temperature of the glass, which was necessarily present in the design of vacuum systems. Then, this number declined to 200 °C,

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providing desorption of the water vapor. Let us analyze is it possible at present to further reduce this temperature to room temperature, i.e. to exclude vacuum installation heating.

Let us define what minimum degree of coverage can be currently obtained without heating of the vacuum chamber for one hour of pumping. We choose water vapor as the main polluter of the surface. Let us conduct theoretical analysis, considering that pumping is carried out using an ideal pump, the pressure limit of which is equal to zero.

The amount of gas adsorbed on the internal surfaces of vacuum systems is determined by the isotherms of adsorption [1–3]. Frendlich equation [2] for isotherms of water vapor adsorption at room temperature on the surface of electropolished stainless steel can be written in the form:

$$\theta = dp^m / a_m, \quad (1)$$

where θ – degree of coverage; a_m = 0.116 $m^3 \cdot Pa/m^2$ – specific amount of adsorbed gas in a monolayer; d = 0.0176, m = 0.25 – constant coefficients [1]; p – pressure, Pa.

The degree of coverage strongly depends on temperature (figure 1), which makes heating of a vacuum unit an effective means of its reducing.

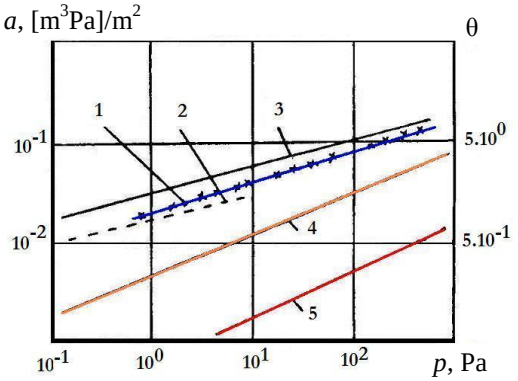

Figure 1. Dependence of the amount of the absorbed gas (a) and the degree of coverage (θ) of water vapors on stainless steel from pressure at different temperatures: 1 – 298 K [2]; 2 – 298 K [1]; 3 – 273 K; 4 – 350 K; 5 – 450 K.

Let us use a mathematical model of pumping of vacuum chambers with absorbent walls [4], which implies the equation for calculating the time of pumping of the water vapors:

$$t = 2.3v \lg \frac{p_i}{p_f} + \frac{dmf}{1-m}\left(p_f^{m-1} - p_i^{m-1}\right), \quad (2)$$

where t – pumping time, s; $v = V_c/S$, s; $f = F_c/S$, s/m; $S = S_m K_u(1 - p_m/p_f)$, m^3/s; V_c – volume of the vacuum chamber, m^3; F_c – area of the inner surface of the chamber, m^2; S – effective pumping speed, m^3/s; S_m – maximum pumping speed, m^3/s; K_u – coefficient of the pump usage; p_m – pump pressure limit, Pa; p_i – initial water vapors pressure, Pa; p_f – final pressure of the water vapors, Pa: d, m – coefficients from the Frendlich equation for adsorption isotherms. Pumping curves calculated using equations (1) and (2) for the pressure and the degree of coverage are shown in figure 2.

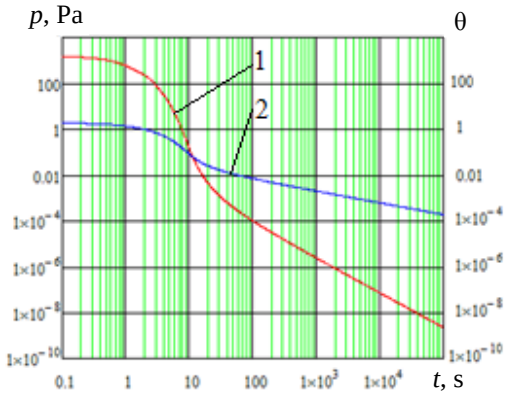

Figure 2. Curves of water pumping at 298 K for v = 1 s, f = 20 s/m: 1 – pressure; 2 – amount of absorbed gas.

Amount of absorbed gas during pumping decreases more slowly than pressure. To reduce the degree of coverage by 10 times it is necessary to reduce the pressure by 10^3 times (figure 2). The first term in the formula (2) takes into account the time of pumping from the volume of the chamber, and the second – time of pumping of the gases adsorbed on the surface. Effect of the chamber volume on the pumping curve is shown in figure 3.

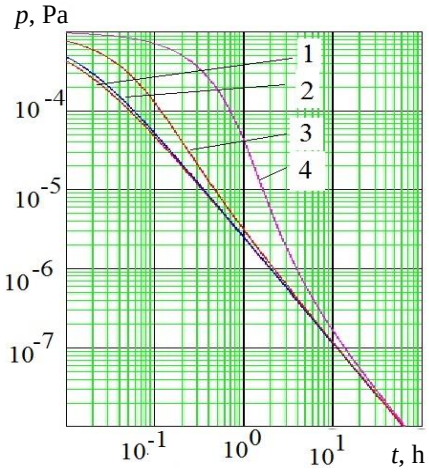

Figure 3. Curves of pumping of the vacuum chamber, made from electropolished stainless steel (d = 0.176; m = 0.25; f = 37.5 s/m; $p_i = 10^{-3}$ Pa), for water vapors at different values of the ratio of chamber volume to effective pumping speed of the pump $v = V/S$: 1 – 1 s, 2 – 10 s, 3 – 100 s, 4 – 1000 s.

If the following condition is met:

$$2.3v \lg \frac{p_i}{p_f} << \frac{dmf}{1-m}\left(p_f^{m-1} - p_i^{m-1}\right), \tag{3}$$

pumping curve does not depend on the volume of the chamber. In a typical vacuum system, the influence of the volume of the working chamber for pumping curve becomes insignificant after one hour after the start of the pumping process. Influence of the initial pressure on the pumping curve is shown in figure 4.

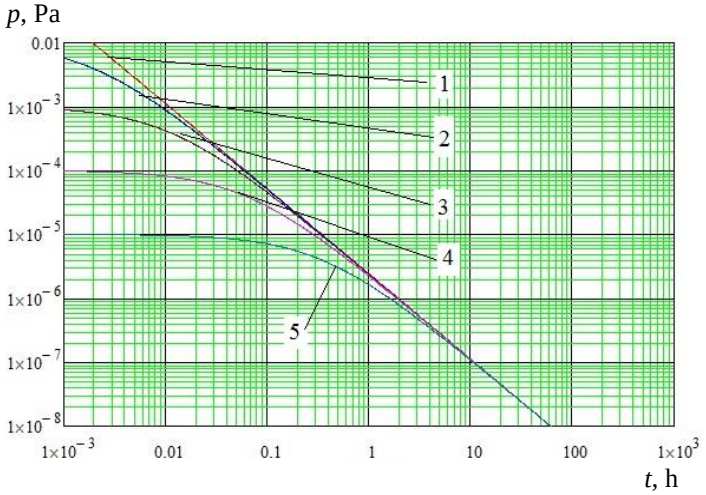

Figure 4. Curves of pumping of the chamber from electropolished stainless steel (d = 0.176; m = 0.25; f = 37.5 s/m; v = 1.25 s) for water vapors at different values of the initial pressure p_i: 1 – 10 Pa; 2 – 10^{-2} Pa; 3 – 10^{-3} Pa; 4 – 10^{-4} Pa; 5 – 10^{-5} Pa.

Provided that

$$p_i / p_f > 10^2, \tag{4}$$

pumping curve becomes independent of the initial pressure of pumping. Usually the condition (3) is fulfilled within one hour after the start of the pumping process. Thus, we can assume that after the time of pumping more than one hour occurs a regular mode, in which the pumping curve in a logarithmic scale asymptotically approaches a straight line, which is described by the following equation:

$$p(t) = \left[\frac{dmF}{3600S(1-m)t}\right]^{\frac{1}{1-m}} = A_1 t^{-B}, \quad (5)$$

where t – pumping time, h; $A_1 = \left[\frac{fdm}{3600(1-m)}\right]^{\frac{1}{1-m}}$, $B = \frac{1}{1-m}$.

Using the experimental coefficients A_1 and B (5), which are determined from the regular mode, the coefficients of the Frendlich adsorption isotherms (1) could be found:

$$d = 3600\frac{(1-m)A_1^{1-m}}{mf}, \quad m = \frac{B-1}{B}. \quad (6)$$

Degree of coverage from (1) and (5) can be calculated by the following equation:

$$\theta = \frac{d\left[dm\frac{f}{3600(1-m)}\right]^{\frac{m}{1-m}}}{a_m}. \quad (7)$$

Management of the degree of coverage, using the parameters a_m, m and d is difficult, because they are the properties of the material. The f parameter is a characteristic of the vacuum system and can be selected more freely. The dependence of the degree of coverage from the various parameters of equation (7) shows (figure 5) the possibility of obtaining degree of coverage in the range 10^{-2}…10^{-3}.

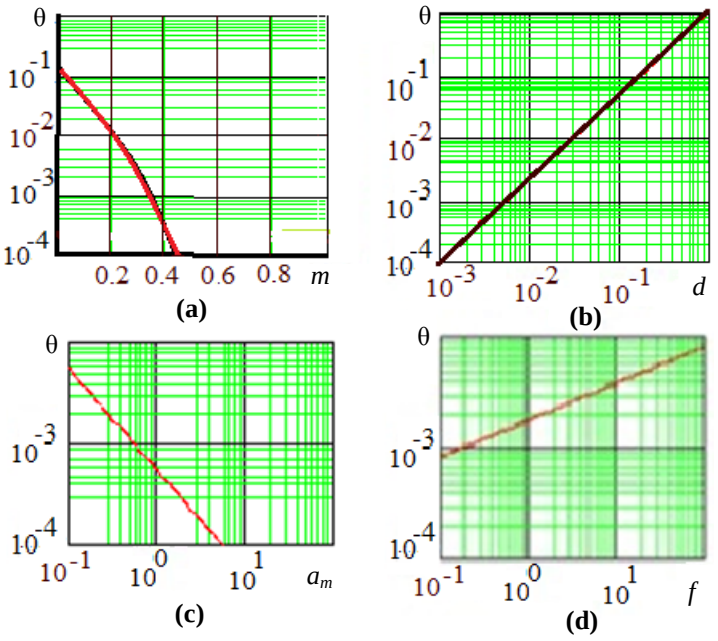

Figure 5. Dependences of the degree of coverage from: **(a)** – m at d = 0.0176, a_m = = 0.116 m^3·Pa/m^2, f = 20 s/m; **(b)** – d at m = 0.25, a_m = = 0.116 m^3·Pa/m^2, f = 20 s/m; **(c)** – a_m at m = 0.25, d = = 0.0176, f = 20 s/m; **(d)** – f at m = 0.25, a_m = 0.116 m^3·Pa/m^2, d = 0.0176.

Value of $\theta = 10^{-3}$ ($2\cdot10^{-9}$ Pa), if f = 0.2 s/m. For the vacuum chamber in a form of a cube (figure 6(a)), through one of the sides of which within one hour pumping by an ideal pump is conducted value f = 0.5, that from figure 2 corresponds to $\theta = 2\cdot10^{-3}$ (10^{-7} Pa). For the pump, closed with a plug (figure 6(b)) value f = 0.1, this corresponds to $\theta = 9\cdot10^{-4}$ ($5\cdot10^{-8}$ Pa).

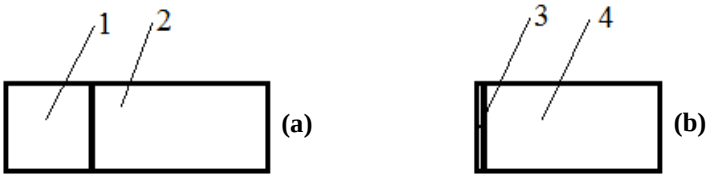

Figure 6. Vacuum chambers: 1 – cubic cell; 2 – pump; 3 – plug; 4 – pump.

Theoretical results (5) and (6) can be checked on the experimental setup (figure 7), including a vacuum chamber made of stainless steel 12KH18N10T with electropolished inner surface of volume V = 50 l and internal surface area F = 1.875 m^2; spiral Edwards XDS35i pump with a pumping speed of 10 l/s; turbomolecular pump Edwards nEXT 300 with pumping speed 300 l/s; quadrupole mass spectrometer EXtorr XT300M; vacuum valve Du-100 with metal seal.

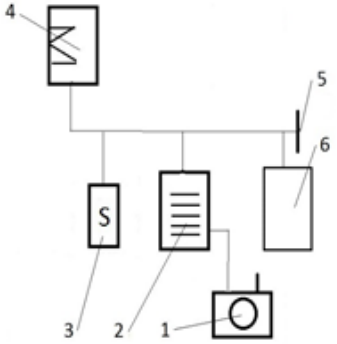

Figure 7. Experimental setup: 1 – spiral pump; 2 – turbomolecular pump; 3 – mass-spectrometer; 4 – cryosorbtion pump; 5 – entrance; 6 – inlet system.

The vacuum chamber was sealed using metal seals. Effective pumping speed was 1 l/s for spiral pump and 50 l/s for turbomolecular pump. Thus, for this installation v = 1 s and f = 37.5 s/m.

Mass spectrum of residual gases (figure 8) for the experimental setup (figure 7) consists of hydrogen, methane, water, nitrogen and carbon monoxide, oxygen, argon and carbon dioxide, but the main component, as expected for non-heated systems, is the water vapors, pressure of which at the end of pumping was $1.5 \cdot 10^{-6}$ Pa.

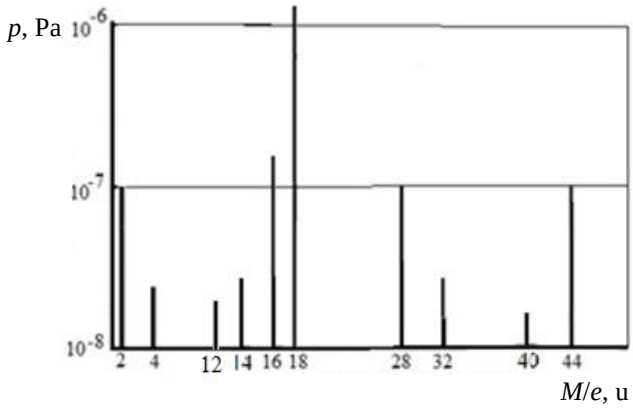

Figure 8. Mass spectrum of residual gases at the end of pumping process.

The results of calculations and experimental data (figure 9) correspond to each other, which confirm the possibility of using equations (5) and (7) to determine the extent of coverage of the internal surfaces of the vacuum chamber by water molecules.

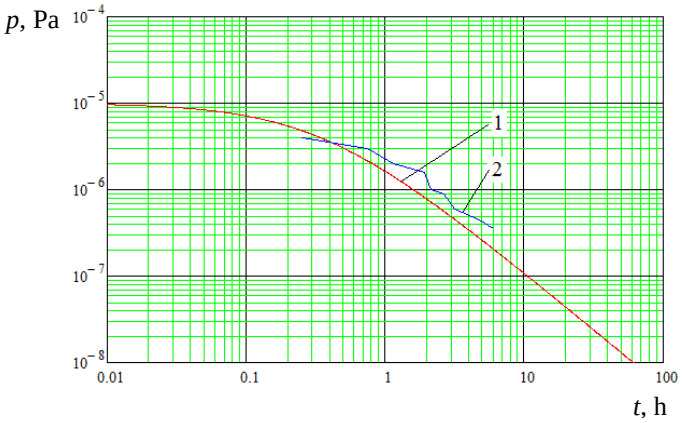

Figure 9. Pumping curve for the experimental setup at v = 1 s, f = 37.5 s/m: 1 – according to equation (5); 2 – experimental results.

After a brief purge of the atmosphere into the chamber and pumping by a spiral pump to the limit pressure for 850 s, the water vapor pressure was 10^{-5} Pa. After one hour of pumping with a turbo molecular pump was achieved a water pressure of $2 \cdot 10^{-6}$ Pa, which corresponds to the degree of coverage $\theta = 5.7 \cdot 10^{-3}$.

3. Conclusion

Obtaining of the ultra-high vacuum ($\theta = 10^{-2}$) without warming of the vacuum chamber from stainless steel and in the absence of the technological gas release is possible by means of modern vacuum pumps for a one hour high vacuum pumping. A further reduction of the degree of coverage ($\theta = 10^{-3}$) can be achieved by the reduction of f factor or increasing the duration of pumping.

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