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Identification of fast hydrogen permeability parameters of gas separation membranes

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The problem of parametric identification of some nonlinear models of fast hydrogen permeability of alloys for membrane technologies for the release of high-purity hydrogen (on the example of alloy B1) is solved. Not only diffusion in the bulk of the material is taken into account, but also physical and chemical processes on the surface: adsorption, desorption and rapid dissolution. The mathematical software and the results of numerical simulation for a three-stage breakthrough experiment with vacuum pumping at the membrane outlet from the structural material under study are presented. The influence of the accumulation of atomic hydrogen on the surface has been studied.

Keywords: hydrogen permeability, membrane technology of gas separation, parametric model identification.

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Introduction

Studies of the interaction of hydrogen isotopes with various structural materials have different nature [1–5]: protection against hydrogen corrosion, transportation of hydrocarbon raw materials, rocket production, hydrogen energetics, prospects of thermonuclear fusion.

In experimental practice, the methods of breakthrough are mainly used (with or without vacuum evacuation from the output side of the membrane from the material under study) and thermal desorption spectrometry (TDS). General analysis and practical recommendations are contained in reviews [6,7]. Kissinger's method is described in detail. The corresponding models operate with the concentration of absorbed hydrogen averaged over the sample volume (see, in particular, [8]). Note the review of models in the paper [9] and the analysis of the limits of the Kissinger formula applicability in comparison with the distributed model with reversible capture of the diffusant by traps (the McNabb−Foster model) [20]. The effect of trapping in various kinds of traps (heterogeneity of the material structure) is the subject of the paper [11]. The papers [12–14] present studies in the field of hydrogen materials science for thermonuclear power engineering. The general distributed model of dehydrogenation is presented in [15], applied problems and corresponding models are presented in [16–18]. Models taking into account various stages of hydrogen transfer and numerical methods for solving boundary value problems are described in [19–21].

In gas separation units, for example, for the separation of extra-pure hydrogen the rather thin membranes with a high throughput capacity are used. The materials are searched with the required strength characteristics and high capacity. According to preliminary experimental data or theoretical considerations it is advisable to conduct a series of computational experiments with various models to evaluate the values of parameters and the influence of various limiting factors (including those under extreme operating conditions of the material) in order to narrow the search range and identify the most effective modes, saving the cost of expensive experiments.

The present paper is a continuation [21], which describes in detail the experimental unit in IMET Ural Branch of the Russian Academy of Sciences, on the basis of which these studies were carried out. In this paper we developed an algorithm for estimating the parameters of the models under consideration without the stage of TDS degassing and breakthrough without pumping out at the outlet, which greatly simplifies the experiment. Let's briefly describe it. The membrane of the material under study is a partition of the vacuum chamber and is preliminarily degassed, the sample temperature is sufficiently high and constant. The outlet side is evacuated and the outlet hydrogen flow is measured using a mass spectrometer. A constant pressure of molecular hydrogen is step-wise created from the inlet side. After a certain time the output flow reaches a stationary value. After that, the inlet pressure level increases by two times. The experiment is carried out with at least three temperatures to estimate the parameters of the Arrhenius temperature dependences of the coefficients. With the temperature increasing it is not necessary to degas the sample again, i. e., the experiment can be carried out cyclically without depressurization of the diffusion cell and the membrane degassing. The chamber depressurization for repeated experiments can lead to a change in the surface structure of the material (strictly speaking, different materials). The cyclic option seems to be more correct and requires only a slight modification of the software. The uniqueness of the problem solution of estimating the hydrogen permeability parameters is of fundamental importance, since the experimental results on extra thin membranes are extrapolated to the protective structures of reactors, including the ITER project. Traps (capturing material heterogeneities) certainly exist. But the determining influence of capturing in the volume of the essentially applied problem must be substantiated. In the problem of gas separation for thin and rapidly permeable membranes the volume traps are neglected due to the relative small volume and the high degree of the material homogeneity.

Under the conditions of fast hydrogen permeability and sufficiently high temperatures (hundreds of degrees Celsius), the following model of boundary conditions is accepted as the base:

$$dq_0/dt = 2\mu s p_0 - bq_0^2 + D\partial_x c|_{x=0}, \quad q_0(0) = 0, \tag{1}$$

$$dq_\ell/dt = 2\mu s p_\ell - bq_\ell^2 - D\partial_x c|_{x=\ell}, \quad q_\ell(0) = 0. \tag{2}$$

Let us refine the notations in (1), (2): $q_0(t)$ — concentration of atoms (after dissociative chemisorption) on the input surface $\big([q] = 1_H/\mathrm{cm}^2\big)$, q_ℓ — on output surface; $\mu = (2\pi mkT)^{-1/2} \approx 2.474 \cdot 10^{22}$ $\big([\mu] = 1_{H_2}/\mathrm{Torr} \cdot \mathrm{cm}^2 \cdot \mathrm{s}\big)$ — gas kinetic constant; not all hydrogen molecules dissociate into atoms, which reflects the sticking coefficient $s(T)$; $p_{0\ell}$ — pressures of molecular hydrogen in the inlet and outlet chambers of the diffusion cell; $b(T)$ — desorption coefficient; $D(T)$ — diffusion coefficient. We calculate concentrations and flows in atoms 1_H, the temperature dependences of the coefficients are assumed to be Arrhenius (but each experiment of a triple pressure jump at the inlet is carried out at a fixed temperature), $[T] = K$.

As coupling conditions on the surface and in the near-surface volume at sufficiently high temperatures, we take $c_{0,\ell} = g(T)q_{0,\ell}$ $(x = 0,\ x = \ell)$, where $g(T)$ is fast dissolution coefficient.

A more complex model: the diffusion equation (including with reversible capture by various kinds of defects z, including possible inclusions of hydride phases) [19]

$$\partial_t c = D\partial_x^2 c - \sum_{\nu=1}^{m}\Big[a_\nu^-\big[1 - Z_\nu\big]c(t,x) - a_\nu^+ z_\nu(t,x)\Big],$$

$$\partial_t z_\nu = a_\nu^-(T)\big[1 - Z_\nu\big]c(t,x) - a_\nu^+(T) z_\nu(t,x),$$

will not be used, since in a practically negligible small volume of homogeneous fast-permeable alloy the number of defects is relatively insignificant. Here $z_\nu(t,x)$ — concentrations of hydrogen atoms captured by defects of various types; $a_\nu^\mp$ — coefficients of absorption and release of H traps; $Z_\nu \equiv z_\nu(t,x)/z_{\max}^\nu$ — degree of saturation $\big(z_{\max}^\nu = \max z_\nu\big)$. For practical purposes, the capture is taken into account in the simplest „integral“ form; refinement of the geometry of defects and their distribution would significantly complicate the model. If the defect, for example, is not a microcavity, but an inclusion of a hydride phase, then at the degassing stage the corresponding coefficient $a_j^-(T)$ is identically equal to zero, and the value $a_j^+(T)$ is positive only after reaching the critical temperature: $T(t) \geq T_{\mathrm{crit}}$. Due to different bond energies (coefficients E_a), a given number of peaks can be achieved.

Specify the required numerical data. Membrane material — alloy V1 (in wt%): Pd–15Ag–25Au–0.7Pt–0.7Ru–0.2Al. Membrane thickness — $\ell = 0.01$ cm; surface area (each of two) — $S = 0.4\,\mathrm{cm}^2$; the input and output volumes are $V_1 = V_{\mathrm{out}} = 2840\,\mathrm{cm}^3$, $V_2 = V_{\mathrm{in}} = 1680\,\mathrm{cm}^3$ (notation $V_{1,2}$ correspond to Fig. 1 [21]). Fixed membrane temperature is $T_1 = 400°$C. The sample was preliminarily degassed. Three constant inlet pressures: 21.2, 50.3, 82.1 Torr. Repeated cycles were carried out at the following temperatures and pressures: 450°C (21.8, 39.9, 61.1 Torr), 500°C (14.9, 31.2, 51.5 Torr). There is no need to re-degas the sample: we change the temperature of the diffusion cell and again increase stepwise the pressure at the inlet, keeping them constant until reaching the next stationary state of the outlet flow.

1. Model refinement under experimental conditions

1.1. Model taking into account H accumulation on the output surface

In a short time, a practically linear distribution of dissolved atomic hydrogen will be established in a thin membrane with high hydrogen permeability (relatively slowly moving quasistationary state). The initial distortion is not critical, since integral relations will be used for $q_{0,\ell}$. The initial stage with a low output flow will not introduce significant distortion. Besides, the diffusion equation complicates the difference scheme, since the boundary conditions are not standard (I–II–III kind), and methods for integrating ordinary differential equations are enough in any mathematical package (the authors used the freely distributed Scilab 6, since the programming language is consistent with Matlab). Neglecting resorption at the output of the powerful vacuum system, we obtain a system of ordinary differential equations (ODE):

$$dq_0/dt = 2\mu s p - bq_0^2 - Dg\ell^{-1}[q_0 - q_\ell], \ \ p = p_0 = \mathrm{const}, \tag{3}$$

$$dq_\ell/dt = -bq_\ell^2 + Dg\ell^{-1}[q_0 - q_\ell], \ \ q_0(0) = 0, \ \ q_\ell(0) = 0. \tag{4}$$

Here we assume that atomic hydrogen $(\dot q_\ell(t) > 0,\ t > 0)$ can accumulate on the output surface. The concentration gradients quickly reach quasistationary state, which allows to approach them by a difference ratio.

1.2. Model not considering accumulation at the output

If H accumulation on the output surface is insignificant, then we arrive to the model

$$dq_0/dt = 2\mu s p - bq_0^2 - Dg\ell^{-1}[q_0 - q_\ell], \quad q_0(0) = 0, \tag{5}$$

$$bq_\ell^2 = Dg\ell^{-1}[q_0 - q_\ell], \quad q_\ell(0) = 0, \tag{6}$$

i. e. all diffusively penetrating hydrogen is immediately „carried away" by desorption.

In the presented models by measurements (using a mass spectrometer) we calculate the desorption flow density

$$J(t) = b(T)q_\ell(t), \quad t \in [0, t_*],$$

where t_* — the time when flow $J(t)$ reaches the stationary value $J(t) = \bar{J} = \mathrm{const}$. By shifting the origin of the time reference, we describe all three successive transient processes of reaching the stationary value.

The problem is to restore the parameter values from the experimental data for, in particular, determination how significant H accumulation on the output surface is. Estimation of D, g, b, s then allows one to experiment numerically without additional costs (including under extreme operating conditions of the material).

2. Stationary modes analysis

Let us equal the derivatives $\dot{q}_{0,\ell}$ in (3), (4) to zero and take into account the practically linear nature of the concentration distribution in the volume:

$$2\mu s p - b\bar{q}_0^2 = \bar{J} = -Dc_x|_0 \Rightarrow \bar{q}_0 = \sqrt{[2\mu s p - \bar{J}]b^{-1}},$$

$$b\bar{q}_\ell^2 = \bar{J} \Rightarrow \bar{q}_\ell = \sqrt{\bar{J}b^{-1}} \Rightarrow \bar{J} = Dg\ell^{-1}[\bar{q}_0 - \bar{q}_\ell]$$

$$\Rightarrow \bar{J} = X\left[\sqrt{2\mu s p - \bar{J}} - \sqrt{\bar{J}}\right], \quad X \equiv \frac{gD}{\ell\sqrt{b}}. \tag{7}$$

There are three such equations (7), corresponding to the pairs $(p, \bar{J})$ $(p \equiv \bar{p}_0)$, and at three different temperatures $T = T$.

Let's use, for example, the first two: $(p_1, \bar{J}_1)$, $(p_2, \bar{J}_2)$. We divide the corresponding equations (7) one by another $(\bar{J}_2 > \bar{J}_1)$, reducing the set of parameters X:

$$\frac{\bar{J}_1}{\bar{J}_2} - \frac{\sqrt{2\mu s p_1 - J_1} - \sqrt{\bar{J}_1}}{\sqrt{2\mu s p_2 - J_2} - \sqrt{\bar{J}_2}} = 0, \quad \mu s p_i > \bar{J}_i. \tag{8}$$

This equation is one-parameter and can be rewritten in a more convenient form

$$f(s) \equiv \Big[\bar{J}_1\Big\{\sqrt{2\mu s p_2 - \bar{J}_2} - \sqrt{\bar{J}_2}\Big\}$$

$$- \bar{J}_2\Big\{\sqrt{2\mu s p_1 - \bar{J}_1} - \sqrt{\bar{J}_1}\Big\}\Big] \cdot 10^{-24} = 0$$

(Fig. 1) and is solved numerically, taking into account the inequalities indicated in (8). But s can also be found analytically from a somewhat cumbersome quadratic equation:

$$y = \sqrt{2\mu s p_1 - \bar{J}_1},$$

$$\sqrt{(y^2 - \bar{J}_1)p_2 p_1^{-1} - \bar{J}_2} = \sqrt{2\mu s p_2 - \bar{J}_2}$$

$$\Rightarrow F(y) \equiv a_2 y^2 + a_1 y + a_0 = 0,$$

$$y > \sqrt{\bar{J}_1}, \quad a_2 = p_2 p_1^{-1} - \bar{J}_2^2 \bar{J}_1^{-2},$$

$$a_1 = 2\bar{J}_2^{3/2}\bar{J}_1^{-1}\left[\sqrt{\bar{J}_2\bar{J}_1^{-1}} - 1\right],$$

$$a_0 = -\bar{J}_2\left[\sqrt{1 - \bar{J}_2\bar{J}_1^{-1}}\right]^2 - \bar{J}_2 + p_2 p_1^{-1}\bar{J}_1.$$

So, the parameter s is found, and other pairs $(p_i, \bar{J}_i)$ can be used, followed by averaging s values. Returning to equation (7), we define the complex X.

As a result, instead of (3), (4) we get the system

$$dq_0/dt = 2\mu s p - bq_0^2 - \sqrt{b}X[q_0 - q_\ell], \quad q_0(0) = 0, \tag{9}$$

$$dq_\ell/dt = -bq_\ell^2 - \sqrt{b}X[q_0 - q_\ell], \quad q_\ell(0) = 0. \tag{10}$$

Note that for already known s and X this is a system of two ODEs with one parameter b. Varying it we achieve a good approximation $bq_\ell^2 = J(t)$, where $J(t)$ — the corresponding experimental diagram. We emphasize that choosing b it is necessary to approximate three transient processes at once $0 \to \bar{J}_1 \to \bar{J}_2 \to \bar{J}_3$, $t \in [0, t_*]$, $t_* = t_{*3}$. The numerically one-dimensional problem of minimizing the root-mean-square residual error is relatively simple. Knowing s, b, it is impossible to determine g and D from the known $X = gD/[\ell\sqrt{b}]$. Therefore, it is necessary to look for additional equations for the parameters.

For the simplified model (5), (6) (without taking into account the atomic hydrogen accumulation on the output surface), the algorithm is simplified: we express $q_\ell(t)$ from the second equation and substitute it into the first one, reducing the numerical integration to one ODE.

3. Estimation of diffusion and dissolution parameters D, g

Let us use the fact that the stationary value $\bar{c}_0 = g\bar{q}_0$ is relatively quickly established at the inlet due to the high pressure. In this case, $\bar{q}_\ell \ll \bar{q}_0$. Usually the equilibrium value is used in accordance with the Sieverts law $\bar{c}_0 \propto \sqrt{p}$. But we will not use such an assumption, since for thin, fast-permeable membranes, the stationary value of the concentration at the input is much less than the equilibrium one.

The diffusion coefficient is estimated by the Daines–Barrer method. Asymptotically, the flow $J_1(t)$

reaches the stationary value $\bar{J}_1$. For certainty, we consider the initial stage, the initial pressure jump. Respectively

$$Q(t) \equiv \int_0^t J(\tau)\,d\tau \approx \bar{J}_1[t - \ell^2(6D)^{-1}] \quad \big(t \geq t_* \equiv t_{*1}\big).$$

The intersection of the asymptote with the t axis gives the so-called delay time $\tau_0 = \ell^2/(6D)$. Analytically

$$\tau_0 \approx t_* - \int_0^{t_*} J_1(\tau)\bar{J}_1^{-1} d\tau, \quad J_1 \approx \bar{J}_1,\ t \geq t_*.$$

Under the integral — a relative value that does not require absolute values of the penetrating flow. For uniformity, we count in atoms. The flow is calculated from the measured pressure in the output volume using a calibration multiplier, so that the ratio of the measured pressures can be taken under the integral. Moreover, the value τ_0 does not depend on the concentration level $\bar{c}_{01}$.

With a new time count ($t_* \to t_0 = 0$), integrating the expression $J_2(t)$, we obtain

$$\int_0^t [J_2(\tau) - \bar{J}_1]\,d\tau \approx [\bar{J}_2 - \bar{J}_1][t - \ell^2(6D)^{-1}],$$

where $t \geq t_* \equiv t_2^*$. Formally, changing not only the initial time, but also the flow reference level (exceeding the value $\bar{J}_1$), we obtain the same expressions for the delay time and estimate of D. It is expedient to average the values D over three transients.

Note. The asymptotic formula $\tau_0 = \ell^2/[6D]$, strictly speaking, is derived for the model $c_t = Dc_{xx}$, $c_0(t) = \hat{c} > 0$, $c_\ell(t) = 0$, $c(0, x) = 0$. The solution should be understood as generalized due to the mismatch between the initial and boundary conditions at $x = 0$. Here $\hat{c}$ — any positive constant. Formally, the input concentration instantly makes a jump at $t = 0$, and zero concentration is maintained at the output. We apply this asymptotics to a slightly different model: a stationary concentration (less than the equilibrium one) is very quickly established at the input, and only a relative zero $c_\ell(t) \ll c_0(t)$, $t > \varepsilon \ll 1$ is at the output. This introduces an additional error into the approximate formula for τ_0. At the same time, the problem is posed in a unique way to find approximations of the parameters D, g, b, s with subsequent local variation to minimize the root-mean-square residual error between the model and experimental graphs of the output desorption flow. The drive to „complete“ coincidence of the graphs is partially justified, since the accuracy of the experimental data is hardly less than 10%.

After evaluating D $\big([D] = \text{cm}^2/\text{s}\big)$ from $X = gD/[\ell\sqrt{b}]$ we find the value g $\big([g] = 1/\text{cm}\big)$.

4. Results of numerical modelling

Fig. 1 illustrates the uniqueness of the parameter s determination. We present the results mainly for the temperature $T = \bar{T} = 400^\circ$C. For 450 and 500°C (to determine that model coefficients are Arrhenius one), the results are similar.

Fig. 2 shows that it is necessary to take into account the hydrogen atoms accumulation on the output surface. It is noticeable that the first model has the best approximation capabilities. The parameters of the Arrhenius dependences are presented in Fig. 3.

Fig. 4 shows the dependence $\bar{J} = \bar{J}(\sqrt{p})$ $\big(\sqrt{p} = \sqrt{p}(\bar{J}),\ \mu s p > \bar{J}\big)$ in accordance with formula (7). It is noticeable that this dependence differs from $\bar{J} \propto \sqrt{p}$.

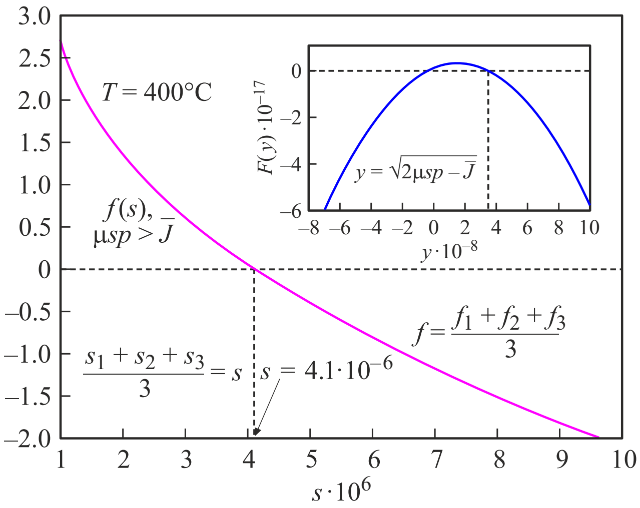

Figure 1. Parameter s determination.

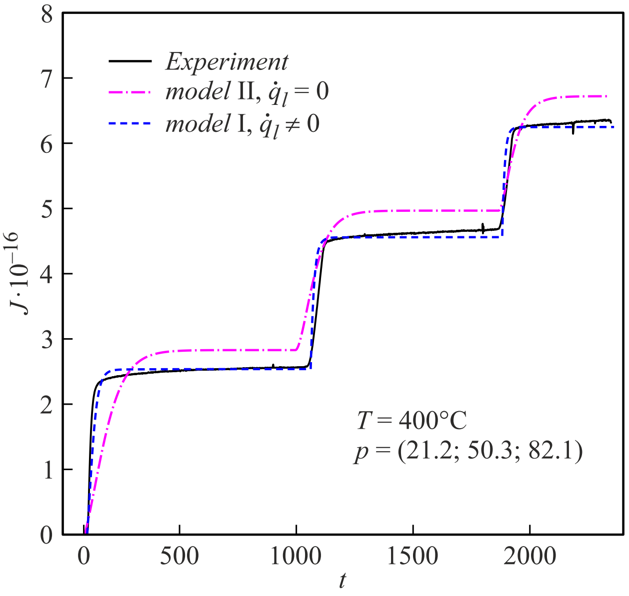

Figure 2. Approximation of experimental data.

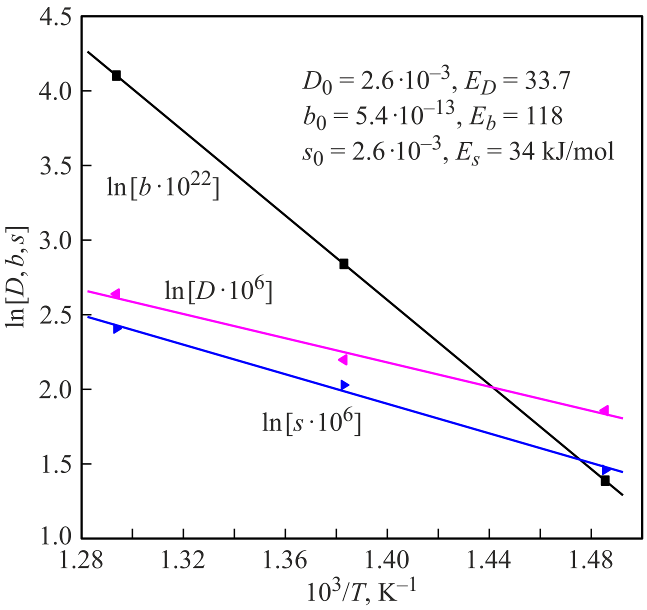

Figure 3. Arrhenius dependence of model coefficients.

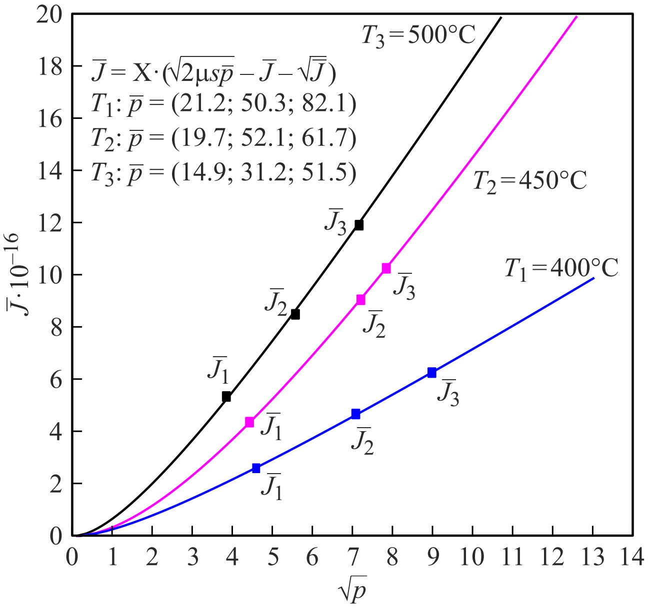

Figure 4. Dependence $\bar{J}(\sqrt{p})$ different from $\bar{J} \propto \sqrt{p}$.

Conclusion

The algorithm for estimating the parameters of the hydrogen permeability of gas separation membranes (on the example of alloy V1) based on the results of a breakthrough experiment with three pressure jumps of molecular hydrogen on the input side and evacuation at the output is presented. This then makes it possible to numerically simulate the permeability in a wide range of parameters for possible necessary adjustment of the experimental conditions, identification of limiting factors, and assessment of sensitivity to parameters variations (alloy composition). The algorithm guarantees the uniqueness of estimates, which is important when recalculating the results for other sample thicknesses. Only integral measurement processing operators are used, which predetermines a rather high noise immunity of the calculation results.

The Arrhenius dependence of the model coefficients on temperature and the difference between the dependence $\bar{J}(\sqrt{p})$ and $\bar{J} \propto \sqrt{p}$ are presented.

The expediency of taking into account the accumulation of dissolved atomic hydrogen on the output surface of the membrane, despite the evacuation of the outlet chamber of the diffusion cell, is shown.

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Conflict of interest

The authors declare that they have no conflict of interest.

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