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GAS PERMEABILITY OF NUCLEAR MEMBRANES AS A FUNCTION OF EFFECTIVE PORE DIAMETER

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We have examined experimentally and theoretically the transport of various gases in channels with a diameter comparable to that of the molecule dimensions.

Interest in the processes of gas transport in membranes with ultrasmall pores exhibiting diameters of $D < 10$ nm is associated with the possibility of achieving an optimum combination of their permeability with high selective properties. Such membranes may result, for example, as an intermediate product in the production of quartz glass [1]. However, the indeterminacy of the geometric structure of porous glasses limits their selectivity and hinders interpretation of experiments [1] into the gas permeability at the microscopic level of the description.

In recent times so-called nuclear membranes (filters) have gained ever-increasing acceptance, and these filters are produced through the irradiation of polymer films with fission fragments in nuclear reactions [2] or by heavy ions in charged-particle accelerators [2-4], and the subsequent chemical etching of the tracks. Pores most nearly cylindrical in shape are obtained here with limited dispersion by size as a consequence of the above-described method by irradiation of films made of polyethylene terephthalate (PETP) [2]. The regular pore geometry of such a nuclear filter, given their ultrasmall dimensions, comparable to the characteristic dimensions of the potentials of intermolecular interactions, offers a unique possibility of establishing the relationship between the parameters of gas permeability through the filter and the microscopic characteristics of the gas and of the channel surfaces.

Isothermal ($T \approx 300$ K) measurements of the permeability of He, Xe, Ar, and N_2 have been conducted in the present study on two specimens of nuclear PETP membranes whose pore diameters ranged in the interval 1.5-10 nm; these measurements have been carried out on these membranes in a series of "load—unload" cycles [5]. The experimental relationship of the relative permeability and the pore dimension is interpreted on the basis of a theoretical approach such as that covered in [6-8], within the scope of which we derive a solution to the system of interconnected kinetic equations for the functions of the distribution of free and adsorbed gas molecules within the channel.

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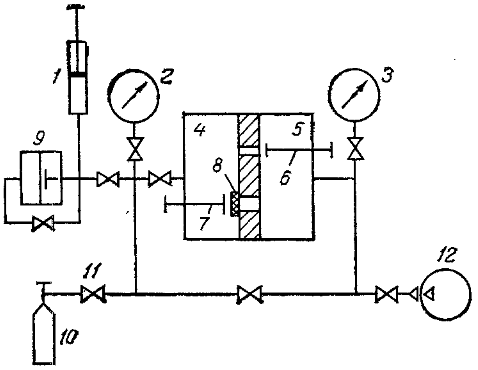

Fig. 1. Diagram of the experimental installation: 1) flow meter; 2, 3) reference manometers (vacuum gauges); 4, 5) working chamber cavity; 6, 7) valves; 8) nuclear filter; 9) capacitance micromanometer sensor; 10) gas tank; 11) overflow valve; 12) adsorption pump.

The basic diagram of the experimental installation can be found in Fig. 1. The gas, under a constant pressure $P_0 = 0.0133$ MPa, recorded by means of reference vacuum gauge 2, flows through the nuclear filter 8 into a vacuum. The volumetric flow of gas in the range $(10^{-5}\text{-}2)\cdot 10^{-6}$ m^3/sec was measured by means of piston flowmeter 1. Constancy of the gas pressure in front of the filter was monitored with a capacitance differential micromanometer 9 [9]. To prevent contamination of the pore surfaces by the various oil-pair combinations, adsorption pump 12 was used. The characteristics of the nuclear filters are as follows: area $S = (0.95 \pm 0.03)\cdot 10^{-4}$ m^2; film thickness $L = 10 \pm 0.5$ μm; pore density $n_1 = (4.8 \pm 0.6)\cdot 10^{13}$ m^{-2} (specimen 1) and $n_2 = (4.2 \pm 0.5)\cdot 10^{13}$ m^{-2} (specimen 2). The change in the effective pore diameters of the specimens was determined from the change in the flow of helium in the free-molecular regime through utilization of the Knudsen formula [10]:

$$Q = \frac{2\pi}{3}\frac{D_0^3}{L}\frac{v_t}{8}N\frac{\Delta P}{P_0}\frac{2-\varepsilon}{\varepsilon}. \tag{1}$$

Based on the measured values of Q_i for the volumetric flows of He, Xe, Ar, and N_2 we found the reduced flowrates ω_{ij}, independent of the gas molecule mass:

$$\omega_{ij} = \frac{Q_i}{Q_j}\sqrt{\frac{m_i}{m_j}}. \tag{2}$$

Figure 2 shows the experimental functions $\omega_{ij}(D_0)$ for three pairs of gases Xe–He (1, 2), Ar–He (3, 4), and N_2–He (5) [11]. Various symbols are used to identify the experimental points obtained on specimen 1 (1, 3, 5) and on specimen 2 (2, 4). We see that as the gases flow through pores with diameters of $D_0 > 6$ nm, ω_{ij} tends to a constant value on the order of 0.92, as is to be expected in accordance with formula (1) when $\varepsilon_{He} \approx 0.96$ and $\varepsilon \approx 1$ for the remaining gases. With a gradual reduction in the channel diameters we observe an increase in the reduced rate of flow for the Xe–He pairs and a reduction in that rate of flow for the Ar–He and N_2–He pairs. Such a behavior of the functions $\omega_{ij}(D_0)$ cannot be described within the scope of the existing model of "discontinuous" diffusion over the surface [1-3] and ap-

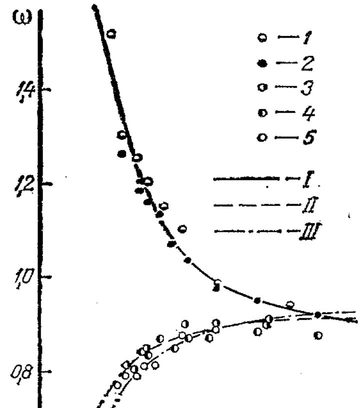

Fig. 2. Reduced flow rate as a function of the gasdynamic channel diameter. Experiment: 1, 2) Xe–He; 3, 4) Ar–He; 5) N_2–He. Theory: I) Xe–He; II) Ar–He; III) N_2–He; D_0, nm.

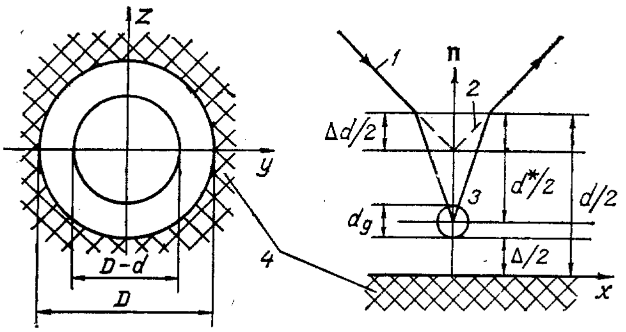

Fig. 3. Geometric parameters of gas flow in a cylindrical channel of ultrasmall diameter: 1) real three-molecule trajectory; 2) effective trajectory; 3) gas molecule; 4) PETP.

parently associated not only with the increasing contribution of the flow of adsorbed gas particles to the general flow through the filter, but also with the influence exerted by the static potential fields within the filter channels on the transport processes.

Let us examine in greater detail the processes of gas transport through a solitary cylindrical filter channel. As was demonstrated in [6, 8], the flows of free I_f and adsorbed I_a gas molecules through some arbitrary cross section of the channel can be represented in the form:

$$I_f = -\varkappa_{ff}\frac{1}{n_f}\frac{\partial n_f}{\partial x} - \varkappa_{fa}\frac{1}{n_a}\frac{\partial n_a}{\partial x},$$
$$I_a = -\varkappa_{af}\frac{1}{n_f}\frac{\partial n_f}{\partial x} - \varkappa_{aa}\frac{1}{n_a}\frac{\partial n_a}{\partial x}. \qquad (3)$$

Here and below it is assumed that the flow I_a is formed by nonlocalized adsorbed gas molecules, with no collisions occurring between the particles of phases f and a. The intersecting kinetic coefficients $\varkappa_{fa}=\varkappa_{af}$ describe the isothermal entrainment [8] and are governed by the fact that in molecular adsorption and desorption some transfer of mean tangential momentum takes place between phases f and a.

The localized molecules do not participate directly in the transport process, and their influence on the transport makes itself evident when the surface is filled to a considerable degree with heavy gases at relatively low temperatures, when the mobile molecules are scattered at the strongly bound localized molecules rather than at the components of the filter material.

The considerable length L of the channel in the experiment ($L/D_0 > 10^3$) allows us to assume that in any element of the channel lengths sufficiently removed from the ends there exist local equilibrium between the gas and adsorbed phases. In this case

$$\frac{1}{n_f}\frac{\partial n_f}{\partial x} = \frac{1}{n_a}\frac{\partial n_a}{\partial x} \approx \frac{\Delta P}{LP_0},$$

while the total flow I of particles through the cross section of the channel is equal to

$$I = I_f + I_a = (\varkappa_{ff} + 2\varkappa_{fa} + \varkappa_{aa})\frac{\Delta P}{LP_0}. \qquad (4)$$

In the absence of potential fields within the channel, the kinetic coefficient $\varkappa_{ff}$ is equal to

$$\varkappa_{ff} = \frac{1}{4} n_f v_t S_f l_f.$$

If we take into consideration [6] that the total energy of the gas particles changes significantly only as a result of their ineleastic collisions with the atoms of the polymer, the acceleration of the molecules in the potential hole of the surface leads, on the one hand, to a distortion of ℓ_f and, on the other hand, to a reduction in the effective area S_f of the transverse cross section. Indeed, the number of free particles incident at a velocity close to v per unit time per unit area of channel surface is equal to $v_n f(v) d^3v$. As a result of the acceleration of the free particles in the potential hole with the characteristic depth U their normal velocity increases: $v_n' = \sqrt{v_n^2 + 2U/m}$, but the flow of particles does not change until collision with the surface, so that

$$v_n' f(\mathbf{v}') d^3 v' = v_n f(\mathbf{v}) d^3 v.$$

Since the law of the conservation of energy is satisfied in this process, we have $v_n' d^3v' = v_n d^3v$, from which it follows that

$$f(\mathbf{v}') = f(\mathbf{v}).$$

In a state close to the equilibrium this equality indicates that the numerical density n_f of the gas at some distance from the surface and its density n_f^* in the region of effective static attraction potential of the quantity U are associated by the relationship

$$n_f^* = n_f \tau, \tag{5}$$

where $\tau = \exp(U/kT)\,\mathrm{erfc}\,(U/kT)^{1/2}$.

Let the surface potential have the shape of a rectangular potential hole with width d/2, and let the gas molecules represent solid spheres with diametr d_g. It is not difficult to see that the characteristic mean free path of the molecules in the potential surface field with $d^*/2$ is smaller than d/2 by the sum of the molecular radii $d_g/2$ and the characteristic thickness $\Delta/2$ of the layer of surface localized molecules (see Fig. 3), so that

$$d^* = d - (d_g + \Delta).$$

Then, with consideration of (5) we can write

$$\varkappa_{ff} = \frac{1}{4}(n_f S_f + n_f^* S_f^*) l_f' v_f = \frac{1}{4} n_f v_t S_f' l_f'. \tag{6}$$

Here the effective cross-sectional area for the free molecules is equal to

$$S_f' = \frac{\pi}{4}(D-d)^2\left(1 + \frac{2d^*(D-d) + d^{*2}}{(D-d)^2}\tau\right), \tag{7}$$

while the characteristic relaxation lengths of the free molecules, with consideration of the wall potential, can be written in the form

$$l_f' = \frac{4}{3}\frac{2-\varepsilon}{\varepsilon}(D - d + \Delta d), \tag{8}$$

where $\Delta d \approx d^*\tau$.

Having substituted (7) and (8) into (6), we finally obtain

$$\varkappa_{ff} = c_{ff}(D-d)^2(D - d + \Delta d) n_f v_t, \tag{9}$$

where

$$c_{ff} = \frac{\pi}{12}\frac{2-\varepsilon}{\varepsilon}\left(1 + \frac{2d^*(D-d) + d^{*2}}{(D-d)^2}\tau\right).$$

In analogy with (6) the surface flow of nonlocalized adsorbed molecules is determined by the following coefficient [8]:

$$\varkappa_{aa} = \frac{1}{4} n_a v_t S_a l_a. \tag{10}$$

If E is the characteristic magnitude of the potential barriers hindering the displacement of the adsorbed gas molecules along the surface, the density n_a under the condition E < U can be estimated to be, as in [12],

$$n_a = n_f\left(\exp\frac{U-E}{kT} - 1\right). \tag{11}$$

TABLE 1. Fundamental Parameters of Gas Permeability for the Investigated PETP in Nuclear Filters with Ultrasmall Pore Diameters

Parameters	Ar	N_2	Xe
d, nm	0,97	1,35	1,26
d^*, nm	0,69	1,03	0,68
βc_a	1,23	1,39	1,59
U, kcal/mole	2,4	2,8	8,0
$U-E$, kcal/mole	0,12	0,15	1,13

Assuming $\ell_a \approx c_a d^*$, we obtain

$$\varkappa_{aa} = c_{aa} d^{*2} (2(D-d) + d^*) \left(\exp \frac{U-E}{kT} - 1 \right) n_f v_t, \tag{12}$$

where $c_{aa} = c_a \pi/16$. Let us take note of the fact that unlike the model of the "discontinuous" diffusion containing the characteristic discontinuity length δ [2, 3], that is frequently employed to describe surface diffusion, in the model under consideration this role is played by d^*.

Since the characteristic energy of the nonlocalized absorbed states of the gas molecules exceeds the energy of the localized state by the quantity E, we might expect that the desorption and adsorption of the gas molecules is achieved primarily by the more probable transitions between the free and nonlocalized states. In this case, for the kinetic coefficients $\varkappa_{fa} = \varkappa_{af}$ we can use the estimate [8]:

$$\varkappa_{fa} = \varkappa_{af} = \frac{1}{4} S_f' l_a n_f v_t \beta. \tag{13}$$

Here β is the probability of gas adsorption at the given temperature of the system. Substituting (7) into (13), we obtain

$$\varkappa_{fa} = \varkappa_{af} = c_{af} (D-d)^2 d^* \beta n_f v_t, \tag{14}$$

where

$$c_{af} = \frac{3}{4} c_{ff} c_a.$$

The total flow of gas through the filter channel (4) assumes the form

$$I_g = -n_f v_t \frac{\Delta P}{LP_0} (a(D-d)^3 + 2b(D-d)^2 + c(D-d+d^*/2)), \tag{15}$$

where

$$a = c_{ff},\ b = c_{ff} \Delta d + \beta c_{af} d^*,\quad c = 2c_{aa} d^{*2} \left(\exp \frac{U-E}{kT} - 1 \right).$$

At the limit $U \ll kT$ (15) changes into the Knudsen formula (1)

$$I_0 = a_0 D_0^3 n_f v_t \frac{\Delta P}{LP_0}, \tag{16}$$

where $a_0 = \frac{\pi}{12} \frac{2-\varepsilon}{\varepsilon}$, $D_0 = D - d_g$ is the gasdynamic diameter of the channel. This condition, leading to formula (16) is valid for He, which remains virtually unadsorbed at the surface at room temperature [3].

The ratio of the flows of any gas and helium through the filter made up of a set of N identical channels of diameter D can be written in the form of the reduced flowrate (2)

$$\omega = A \left(1 - \frac{d'}{D_0} \right)^3 + \frac{B}{D_0} \left(1 - \frac{d'}{D_0} \right)^2 + \frac{C}{D_0^2} \left(1 - \frac{d' - d^*/2}{D_0} \right), \tag{17}$$

where $D_0 = D - d_{He}$ is the gasdynamic parameter with respect to He, $d' = d - d_{He}$, $\alpha = (2 - \varepsilon_g)\varepsilon_{He}/(2 - \varepsilon_{He})/\varepsilon_g$,

$$A = \frac{a}{a_0} = \alpha\left(1 + \frac{2d^*(D_0 - d') + d^{*2}}{(D_0 - d')^2}\tau\right), \tag{18}$$

$$B = \frac{2b}{a_0} = 2A(\Delta d + \beta c_a d^*), \tag{19}$$

$$C = \frac{c}{a_0} = \frac{3}{2}\frac{\varepsilon_{He}}{2 - \varepsilon_{He}} c_a d^{*2}\left(\exp\frac{U - E}{kT} - 1\right). \tag{20}$$

Formula (17) can be used to process the experimental data on the relative gas permeability through filters with ultrasmall pore dimensions $D_0 \geq d'$ for the case in which $U_{He} \ll kT < U$, $E < U$. In the general case formula (17) contains the unknown parameters d', d^*, α, β, c_a. However, the coefficient α can be easily found from the asymptote $\omega \approx \alpha$ for $D_0 \gg d'$. On the other hand, for gases with a low level of localized-state surface filling the characteristic thickness of the region of nonlocalized molecule flow is at its maximum and equal to $d^* = d - d_g = d' - (d_g - d_{He})$.

Comparison of the developed theory with the experimental data on the permeability of the inert gases He, Xe, Ar, as well as of N_2 through the nuclear filter based on PETP is shown in Fig. 2. Processing of the data was accomplished by the method of least squares with three sweeping parameters, and for each gas, proceeding from an estimate of the level of surface filling, we initially chose the value of $d' - d^*$ and specified the quantity $\alpha = 0.92$. The molecular diameters of the investigated gases were taken from [13] and amounted to $d_{He} = 0.18$ nm, $d_{Ar} = 0.27$ nm, $d_{N_2} = 0.32$ nm, $d_{Xe} = 0.32$ nm.

At room temperature $T \sim 300$ K the degree of surface filling for Ar and N_2 is negligibly small. For these gases the physically noncontradictory values of the parameters d', βc_a, and C are obtained with a minimum magnitude of $d' - d^* = d_g - d_{He}$ ($\Delta = 0$). The degree of filling for the localized states under these same conditions for Xe, conversely, is close to unity. In addition to the estimates, this is also indicated by the fact that the values of d' are negative when $d' - d^* < 0.25$ nm, which is a result of the processing of the experimental data. The assumed value of $d' - d^* = 0.4$ nm ($\Delta = 0.257$ nm) has been obtained in the assumption that the centers of localized adsorption have been completely filled with Xe atoms, these centers forming a two-dimensional periodic lattice with an interval of $\delta \sim 1$ nm [3]. The calculation result can be found in Table 1.

The first term in (17) plays a decisive role when $D_0 \gg d'$, and the last term is significant when $D_0 \lesssim C^{1/2}$. The maximum contribution of the second term to ω pertains to that intermediate region of diameters and amounts to a quantity on the order of 0.65 when $D_0 \sim 1.6$ nm for Ar, 0.65 for $D_0 \sim 2.4$ nm in the case of N_2, and 0.3 for $D_0 \sim 2.9$ nm in the case of Xe. We should stress that in all of the cited experiments the required magnitude of the coefficient B cannot be achieved if for no other reason than because of the correction factor Δd in (7). A considerably greater contribution (on the order of 80%) to B is made by the isothermal effects of entrainment between the free and nonlocalized gas molecules (see Table 1). Thus, the results obtained can be regarded as a first experimental confirmation of the existence of the effects of entrainment in a heterogeneous gas system such as that examined in [6]. It follows from (19) that the entrainment effects are independent of the density of the nonlocalized gas molecules but are determined exclusively on the basis of their average macroscopic velocity along the surface (in terms of the Clausing factor $c_a d^*/L$) and the fraction β of the desorbed molecules in the surface-reflected flow of gas. Conversely, according to (20), the contribution of the surface flow to ω depends significantly on the density n_a and, in particular, is very sensitive to the height E of the potential barriers when compared to the depth U of the hole. The competition between these three contributions to the gas flow through the membrane leads to the appearance of local minima for Ar and N_2 (curves II and III) and a maximum for Xe (curve I) in the function $\omega(D_0)$ in the region $D_0 \sim 1.0$-1.5 nm (see Fig. 2). With a further reduction in the diameter we have a pronounced distortion of the potential field within the channels, and model (17) ceases to function. It is obvious that $\omega \to 0$ as $D_0 \to d_g \geq d_{He}$.

Unfortunately, the processing of the experimental data on the gas permeability of PETP-based nuclear filters [2, 3] which are available in the literature is made more difficult because of the small number (≤ 3) of experimental points in the region $D_0 < 5$ nm and the large relative error which may be as high as 30%. Nevertheless, calculation in accordance with formula (17) shows that with values for the parameters d, d*, and α shown in Table 1, the quantities U-E in the experiment [2] are larger approximately by a factor of 1.5-2 than those obtained here. It is not beyond the realm of possibility that this effect is associated with the spongelike structure of the filter channel walls obtained in [2, 3] by the method of lyophilic drying [14].

In conclusion, let us note that the relationship of the reduced flowrate $\omega(D_0)$ for the Ar—He and N_2—He pairs, such as observed here, leads to an increase in the ideal coefficient of separation in these systems with a reduction in D_0. For the Ar—He system the maximum value of the coefficient of separation is achieved in the case of $D_0 \sim 1$ nm, while for the N_2—He system it is achieved when $D_0 \sim 1.5$ nm.

NOTATION

D, diameter of membrane channels; Q, volumetric gas flow through the membrane; $v_t = (8kT/\pi m)^{1/2}$, average thermal velocity of the molecules; N, number of membrane channels; ΔP, pressure difference across the membrane; P_0, pressure at the filter inlet; ε, fraction of diffusion reflection at the wall; ω, reduced flowrate; m, gas molecule mass; I_f, flow of free molecules; I_a, flow of adsorbed molecules along the surface; n_f, n_a, numerical densities of the free and adsorbed molecules; κ_{ij}, kinetic coefficient; L, length of membrane channels; T, temperature; U, depth of potential surface hole; S_f, S_f^*, S_a, areas of transverse cross section of the regions of gas flow with densities n_f, n_f^*, and n_a, respectively; n_f^*, density of free molecules in the potential surface hole; S_f', effective area of lateral cross section of the channel for free gas molecules; v_n, normal component of molecule velocity; $f(v)$, function of gas distribution by velocities; $d^3v = dv_x dv_y dv_z$; x, coordinate along the channel; d/2, width of potential surface hole; d*/2, characteristic mean-free path of molecules in the surface potential field; $\Delta/2$, effective thickness of the layer of localized molecules; d_g, diameter of gas molecules; ℓ_f, ℓ_a, relaxation lengths of free and adsorbed molecules; ℓ_f', effective length of relaxation for the free molecules; Δd, correction factor for the relaxation lengths ℓ_f; c_{ff}, c_a, c_{af}, dimensionless coefficients; β, adsorption proability; D_0, gasdynamic diameter of the channel with respect to helium; c_α, proportionality factor; E, height of potential barriers blocking displacement of adsorbed molecules along the surface.

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CAPACITY AND ACOUSTIC-EMISSION METHODS OF STUDYING TURBULENT-CAVITATION FLOWS OF A FLUID IN POROUS PERMEABLE MATERIALS

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UDC 532.621

We present results from an investigation into the hydrodynamic flows of a fluid through porous materials; these investigations involve two developed methods and we have demonstrated their high sensitivity to the appearance of cavitation bubbles.

Porous permeable materials (PPM) are used extensively in liquid filtration and heat exchange in various branches of industry [1], and the forming flows [2] exert predominant influence on the efficiency of these processes. We presently have on hand a developed and tested range of various methods to study liquid flows in channels and these include mechanical [3], acoustic [4], electromagnetic (conductometric) [5], optical [6], and nuclear-physical [7]. However, the areas in which these methods can be employed to investigate the flows of a fluid in the microcapillaries of pores is extremely limited as a consequence of the specific nature of PPM structure. The goal of this study therefore involves the laying of a foundation for and the experimental verification of the capacity and acoustic-emission methods of examining turbulent-cavitation flows in PPM.

We know [8] that microcapillary channels in PPM are a form of a labyrinth with diversely distinct cross sections which are formed by the elements of the structure. The motion of a liquid about the curvilinear surface of the PPM channels is accompanied by deformation of the flow and the onset of turbulence [9], i.e., local fluctuations in velocity, pressure, and temperature; here we also find a reduction in the cavitational strength of the moving liquid [10]. When the local pressure is lower than the vapor saturation pressure, cavitation sets in [11], and this phenomenon is characterized by a pulsating vapor-gas phase which sends out acoustic waves into the surrounding medium. Consequently, having measured the cavitation index D, equal to the ratio of the component gas volumes ΔV_i of the phase in the liquid V_0 [12] and the amplitude-frequency characteristics (AFC) [2], we can qualitatively and quantitatively evaluate the turbulent-cavitation flows. On this basis, to achieve our stated goal, we have developed a PPM model to serve as a sensor, and this is a round disk of a sintered tantalum (niobium) powder. The structure of the formed materials pertains to the class of PPM and serves as an adequate analog of those materials used for filtration, heat exchange (Fig. 1). We know [1] that PPM exhibits large specific wetting areas, and the treatment of the tantalum (niobium) with H_3PO_4 acid forms thin current-insulating films on the material's surface. This makes it possible to come up with a high-capacity capacitor in which one of the plates is a PPM structure, while the current-conducting fluid (electrolyte) serves as the second. The capacitance here depends on the extent to which the PPM structure is completely filled, as well as on the uniformity of the liquid phase. Thus, we can make the assumption that with the onset of cavitation in the investigated PPM with a pore volume of V_0 the forming vapor-gas bubbles ΔV_i alter the initial capacitance C_0 of the capacitor by ΔC_i, and the quantities $V_0 \approx C_0$, $\Delta V_i \approx \Delta C_i$ will be equivalent. Connecting this device (Fig. 2)

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