THE INFLUENCE OF ADSORPTION OF ALCOHOL AND WATER VAPORS ON CURRENTS OF A MIS STRUCTURE WITH AN INTERMEDIATE LAYER OF POROUS SILICON

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The influence of the adsorption of water and alcohol vapors on the current-voltage (I—V) characteristics of "metal–porous silicon-silicon" structures is analyzed theoretically and investigated experimentally. The analysis of the diffusion-drift equations allows us to derive expressions that show in what way the current depends on the variation of the dielectric constant and the built-in charge in porous silicon (PS). The highest current is reached for the 40% concentration of alcohol in water, which is explained by the competition between the injection of electrons into PS in the process of nondissociative adsorption of water molecules and the passivating properties of the adsorbed layer of alcohol molecules. We propose a model of recombination that explains the nonmonotonous character of the current variation in the process of adsorption of the mixture of water and alcohol.

1. Introduction

Recently, PS-based chemical sensors are widely investigated [1, 2]. They are characterized with a high sensitivity to low concentrations of adsorbed molecules due to, primarily, a large external area and specific properties of the PS surface [3]. The transduce parameters used in PS heterostructures are the intensity of photoluminescence, optical constants, and some electric parameters (current, capacity, etc.) [2—6]. The investigation of the influence of the water and alcohol adsorption on I—V characteristics of a MIS structure with an intermediate porous dielectric layer is interesting from the viewpoint of the production of sensors sensitive to moisture or other gases. There exist some papers devoted to the study of the I—V characteristics of PS-based structures and their dependence on the adsorption of methanol, acetone, hydrogen sulphide, and hydrogen [2]. These works indicate an important role of the morphology, surface treatment, porosity, PS thickness, and type of a sensitive structure in the achievement of the maximal sensitivity and selectivity of sensors. However, the mechanism of the influence of liquid vapors (for example, water or alcohol ones) on the properties of the structures as well as the kinetics of their variation in the process of adsorption requires a more detailed investigation.

In the present work, we report the results of studying the influence of the adsorption of water and alcohol vapors on the I—V characteristics of Al—PS—p-Si—Ni structures and the possibilities of producing moisture sensors and alcoholmeters on their basis.

2. Production of Structures and Experimental Technique

The PS films were formed by means of the electrochemical etching of a silicon plate (p-type conductivity, orientation (100), specific resistance of 4.5 $\Omega\times$cm). The etching of silicon plates was performed in the mode of constant current of 5 mA/cm^2 in the electrolyte HF(50%):C_2H_5OH:H_2O = 1:5:4. In order to form an ohmic contact, we coated the backside of the plates with Ni (0.4 μm) by means of the electron beam deposition at 150 °C. The thickness of the PS layer was equal to 0.15 or 0.45 μm. The thickness of the rectifying contact (Al) applied by means of thermal evaporation did not exceed 15 nm while the area of the contact approximated to 1 mm^2. The microstructure of the surface layer was investigated by means of the scanning electron microscope technique.

The influence of a gas medium on the samples was investigated in a chamber filled with water and ethyl alcohol vapors at a variable concentration of the latter. We measured the response of the structure on water and alcohol vapors at a frequency f of 9390 Hz which is optimal from the viewpoint of the signal/noise ratio.

3. Theory

3.1. The influence of the adsorption on the current flow in a metal—PS—Si structure

Let's analyze the influence of the adsorption on the dark I—V characteristic of metal—PS—Si structures using the expression for current obtained from the solution of the diffusion-drift equations in [7]:

$$j_p = \left[\left(p_0 e^{-\frac{e\varphi_\Sigma}{kT}} e^{-\frac{eV_d}{kT}}\right)\left(e^{\frac{eV}{kT}} - 1\right)\right]\times$$

$$\times\left[\frac{4}{ev_pP_p} + \frac{1}{e\mu_pE_l} + \left(\frac{4}{ev_p} - \frac{1}{e\mu_pE_l}\right)e^{-\frac{e\varphi_l - eV_l}{kT}}\right]^{-1}, \quad (1)$$

where j_p, μ_p, and p are the current density, mobility, and concentration of holes, respectively, E_l is the electric field strength, P_p denotes the transparency of the interface layer for the tunneling of holes, and v_p is the thermal rate. The total potential is redistributed between Si (V_L), PS (V_l), and the interface (V_d) : $V = V_d + V_l + V_L$, $e\varphi_\Sigma = e\varphi_0 + \Delta E_v + e\varphi_l$ (Fig. 1).

As one can see from (1), the voltage dependence of the current for majority charge carriers corresponds to the classic exponential law $j_p = j_s \exp(eV/kT{-}1)$. However, the expression describing the saturation current j_s is more complicated and involves the transparency of the intermediate layer P_p, the drift velocity of holes in PS μ_pE_l, the distribution of the voltage drop between Si, PS, and the interface, as well the corresponding potential barriers. In the denominator of (1), we can separate the first term responsible for the tunneling through the interface and the second and third terms that describe the diffusion and drift through PS.

Taking into account that the hole drift velocity in PS is much lower than the thermal one $\mu_pE_l \ll v_p(v_p \sim 10^7$ cm/s, $\mu_p \sim 10^{-4}$ cm^2/(V·s) [8], $E_l \sim 10^5$ V/cm at $l = 100$ nm), the following inequality is valid:

$$4/evP_p \ll (1/e\mu_pE_l)[1 - \exp(-(e\varphi_l - eV_l)/kT)]. \quad (2)$$

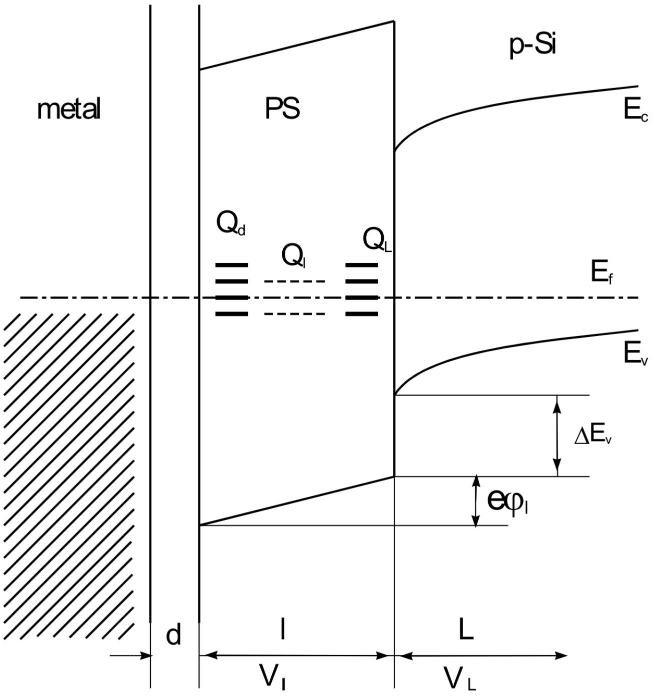


Fig. 1. Energy diagram of the " metal—tunnel-thin oxide—thin PS—p-Si" contact

In this case, the I—V characteristic can be described as

$$j = j_s[\exp(eV_L/kT) - \exp(-e(V_d + V_l)/kT)], \quad (3)$$

where the saturation current depends on the drift velocity μ_pE_l in PS:

$$j_s = ep_0\mu_pE_l \exp(-e\varphi_\Sigma/kT)\times$$

$$\times[\exp(-eV_l/kT) - \exp(-e\varphi_l/kT)]^{-1}.$$

The first and second terms in expression (3) describe the direct and inverse currents, respectively.

The change of current in the process of adsorption has a form

$$\Delta j_s = j_{s\mathrm{a}}/j_s = (E_{l\mathrm{a}}/E_l)[\exp(-e\delta\varphi_\Sigma/kT)]\times$$

$$\times[\exp(-e\delta V_{dl}/kT)][\exp(e\delta V_l/kT], \quad (4)$$

where $V_{dl} = V_d + V_l$, $\delta V_{dl} = V_{dl\mathrm{a}} - V_{dl}$, $\delta\varphi_\Sigma = \varphi_{\Sigma\mathrm{a}} - \varphi_\Sigma$, (here and afterwards, the index "a" corresponds to the case of adsorption). The applied voltage is considered to be low, $e\varphi_l > eV_l$, or

$$\Delta j_s = (E_{l\mathrm{a}}/E_l)[\exp(-e\delta\varphi_\Sigma/kT)]\times$$

$$\times[\exp(-e\delta V_{dl}/kT)][\exp(e\delta\varphi_l/kT] \quad (5)$$

(high applied voltages, $e\varphi_l < eV_l$). That is, in the process of adsorption, the saturation current can be changed due to the variation of: a) the electric field in PS, b) the potential barrier, or c) the voltage drop across the dielectric and PS.

In order to analyze the influence of the adsorption on the variation of the voltage redistribution between the oxide layer, PS, and the space charge region (SCR) of silicon, we can use the equations from [7]:

$$V_d = (2d\varphi_0\varepsilon_L/L\varepsilon_d)[1-\{(\varphi_0 - V_L)/\varphi_0\}^{1/2}] + V_d^i, \quad (6)$$

$$V_l = (2l\varphi_0\varepsilon_L/L\varepsilon_l)[1-\{(\varphi_0 - V_L)/\varphi_0\}^{1/2}] + V_L^i. \quad (7)$$

In this case,

$$V_{d\mathrm{a}} - V_d = (2d\varphi_{0\mathrm{a}}\varepsilon_L/L_\mathrm{a}\varepsilon_{d\mathrm{a}})[1-\{(\varphi_{0\mathrm{a}} - V_{L\mathrm{a}})/\varphi_{0\mathrm{a}}\}^{1/2}]-$$

$$-(2d\varphi_0\varepsilon_L/L\varepsilon_d)[1-\{(\varphi_0 - V_L)/\varphi_0\}^{1/2}] + V_{d\mathrm{a}}^i - V_d^i,$$

$$V_{l\mathrm{a}} - V_l = (2d\varphi_{0\mathrm{a}}\varepsilon_L/L_\mathrm{a}\varepsilon_{l\mathrm{a}})[1-\{(\varphi_{0\mathrm{a}} - V_{L\mathrm{a}})/\varphi_{0\mathrm{a}}\}^{1/2}]-$$

$$-(2d\varphi_0\varepsilon_L/L\varepsilon_l)[1-\{(\varphi_0 - V_L)/\varphi_0\}^{1/2}] + V_{L\mathrm{a}}^i - V_L^i, \quad (8)$$

where

$$V_{d\mathrm{a}}^i - V_d^i = (d/\varepsilon_0)[(\Delta Q_{d\mathrm{a}} + \Delta Q_{L\mathrm{a}})/\varepsilon_{d\mathrm{a}}-$$

$$-(\Delta Q_d + \Delta Q_L)/\varepsilon_d],$$

$$V_{L\mathrm{a}}^i - V_L^i = (l/\varepsilon_0)[\Delta Q_{L\mathrm{a}}/\varepsilon_{l\mathrm{a}} - \Delta Q_L/\varepsilon_l]. \quad (9)$$

For the "metal—semiconductor" contact, the height of the potential barrier in the silicon SCR can be presented as

$$e\varphi_0 = e^2L^2p_0/2\varepsilon_0\varepsilon_L = (Q_s^2 - Q_{s0}^2)/2\varepsilon_0\varepsilon_L p_0 =$$

$$= \delta Q_s^2/2\varepsilon_0\varepsilon_L p_0, \quad (10)$$

where $Q_{s0} = Q_s$ at $V = 0$ is the charge in the semiconductor SCR, and $Q_s = ep_0L(V)$. For PS, we can write in a similar way

$$e\varphi_l = e^2l^2p_l/2\varepsilon_0\varepsilon_l = (Q_l^2 - Q_{l0}^2)/2\varepsilon_0\varepsilon_l p_l =$$

$$= \delta Q_l^2/2\varepsilon_0\varepsilon_l p_l, \quad (11)$$

where p_l is the concentration of holes in PS. The corresponding change in the height of the potential barrier can be expressed in the following way:

$$\delta e\varphi_0 = e\varphi_{0\mathrm{a}} - e\varphi_0 = (1/2\varepsilon_0\varepsilon_L p_0)(\delta Q_{s\mathrm{a}}^2 - \delta Q_s^2), \quad (12)$$

$$\delta e\varphi_l = e\varphi_{l\mathrm{a}} - e\varphi_l =$$

$$= (1/2\varepsilon_0)(\delta Q_{l\mathrm{a}}^2/\varepsilon_{l\mathrm{a}}p_{al} - \delta Q_l^2/\varepsilon_l p_l). \quad (13)$$

In addition, the ideality coefficient of a I—V characteristic can be also changed under the adsorption. Taking into account that the quantity n for a metal—PS contact is described by the equation

$$n = V/V_L = 1 + \varepsilon_L l/\varepsilon_l L + \varepsilon_L d/\varepsilon_d L + V_d^i/V_L + V_L^i/V_L, \quad (14)$$

the corresponding variation of this parameter in the process of adsorption can be presented as

$$\delta n = n_\mathrm{a} - n = l\varepsilon_L(1/\varepsilon_{l\mathrm{a}}L_\mathrm{a} - 1/\varepsilon_l L) +$$

$$+ d\varepsilon_L(1/\varepsilon_{d\mathrm{a}}L_\mathrm{a} - 1/\varepsilon_d L) +$$

$$+ (V_{d\mathrm{a}}^i/V_{L\mathrm{a}} - V_d^i/V_L) + (V_{L\mathrm{a}}^i/V_{L\mathrm{a}} - V_L^i/V_L), \quad (15)$$

where one should allow for the corresponding expressions (9) for the variation of the voltage drop on the surface electronic states.

Therefore, one can consider that the influence of the adsorption on the I—V characteristic of a metal—PS—silicon contact depends on the variation of the dielectric constant and the charges of the tunnel-transparent dielectric, PS, and the near-surface region of silicon. Moreover, if we compare this structure with an ordinary silicon MOS structure, it turns out that the additional sensitivity is reached exactly at the expense of the variation of δQ_l, δQ_L, δQ_d, and $\delta\varepsilon_l$.

3.2. Variation of the PS dielectric constant in the process of adsorption

Let's consider an additional effect of the dielectric constant variation that can be obtained at the expense of the use of PS. The averaged dielectric constant in PS ε can be presented in the form

$$\varepsilon = \Sigma U_i\varepsilon_i/\Sigma U_i, \quad (16)$$

where U_i stands for the volume of each heterogeneity, and ε_i is the dielectric constant in it. Using (16), we can estimate the change of ε for the PS region under the influence of the adsorption of gas molecules. Before the adsorption, the effective value of the parameter is equal to

$$Y_\mathrm{PS} = (U_sY_s + U_pY_p)/(U_s + U_p), \quad (17)$$

where in general case Y_PS denotes ε (or charge), and U_s and U_p are the corresponding volumes of Si and pores in PS. Taking into account the definition of porosity [1]

$$P = U_p/(U_s + U_p), \quad (18)$$

we obtain

$$Y_{\mathrm{PS}} = (1-P)Y_s + PY_p. \qquad (19)$$

After the adsorption of gas molecules, the effective value of the dielectric constant takes the form

$$Y_{\mathrm{PS}a} = (U_s Y_s + U_{\mathrm{a}} Y_{\mathrm{a}} + U_{p\mathrm{a}} Y_p)/(U_s + U_{\mathrm{a}} + U_{p\mathrm{a}}). \qquad (20)$$

Here, the index "a" signifies the parameters after the adsorption. We should also take into account that, after the adsorption of molecules, the volume of pores decreases: $U_{p\mathrm{a}} = U_p - U_{\mathrm{a}}$. Hence, the use of (20) together with (17) and (19) gives

$$Y_{\mathrm{PSa}} = (1-P)Y_s + PY_p + P(U_{\mathrm{a}}/U_p)(Y_{\mathrm{a}} - Y_p). \qquad (21)$$

If we suppose that the adsorbed molecules and pores have a spherical cross section, then $U_p = \pi d^3 N/6$, where N is the number of pores, and d is a pore diameter; $U_{\mathrm{a}} = \pi d^2 d_{\mathrm{a}} N = 6U_p(d_{\mathrm{a}}/d)$, where d_a is the effective thickness of the adsorbed layer of molecules. Then we get

$$Y_{\mathrm{PSa}} = Y_{\mathrm{PS}} + 6P(d_{\mathrm{a}}/d)(Y_{\mathrm{a}} - Y_p). \qquad (22)$$

For example, in the case of the complete occupation of pores (the porosity $P = 0.6$, the diameter of pores $d = 5$ nm) with water molecules (the effective thickness $d_a = 1$ nm, and $\varepsilon_a = 80$), the effective dielectric constant ε_l of PS increases from 5.3 up to 62. That is, in comparison with the case of a solid film of semiconductor or insulator, the adsorption in PS also takes place in the volume, which allows one to obtain a much higher relative change of the dielectric constant $\Delta\varepsilon/\varepsilon$. Its variation $\Delta\varepsilon/\varepsilon$ can be determined from the increase of the capacity while measuring a capacity-voltage (C—V) characteristic in the mode of accumulation of a MIS structure during the adsorption of molecules, since $\Delta C/C = (\varepsilon_{l\mathrm{a}} - \varepsilon_l)/\varepsilon_l$ in this case.

3.3. Variation of the charge in PS in the process of adsorption

Considering the adsorption, we can allow for the variation of the charge density ($dQ = \rho dx$) with the help of the same equations (21) or (22) used when analyzing the variation of the dielectric constant in PS. For this purpose, we should denote the parameter $Y = \rho$, where

$$\rho = \Sigma U_i / \Sigma U. \qquad (23)$$

However, the change of the $C-V$ characteristic of MIS structures with PS will depend on the sign of the injected charge. For example, for a metal—PS—p-Si structure, the adsorption of water that takes place by the nondissociative mechanism: $\mathrm{H_2O}$ (gas) $\longleftrightarrow$ $\mathrm{H_2O^+}$(ad) + e$^-$ will result in an additional shift forward accumulation. In contrast, the dissociative adsorption accompanied with the injection of one electron: $\mathrm{H_2O}$ (gas)+ O_0^{2-}+V_0^- $\longleftrightarrow$ 2HO$^-$(ad)+e$^-$, (here V_0^- denotes an oxygen vacancy that can entrap one electron [9]) will shift the structure in the direction of depletion.

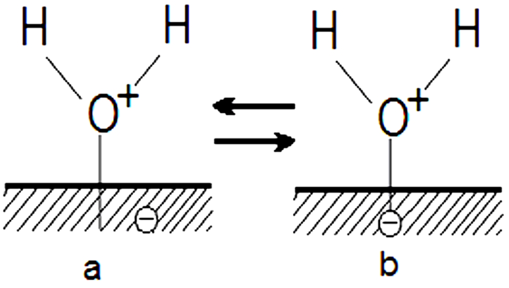


Fig. 2. Model of the adsorption of water molecules for weak (a) and strong (b) bonds

In the general case, a part of adsorbed molecules Q occupies the neutral state Q°, while another part does the positively or negatively charged ones, Q^+ and Q^-. As was shown in [8], for a free surface of a semiconductor, one can write the following relation between the concentrations:

$$Q^-/(Q+Q^-) = \left(1 + \exp\left(-E^-/KT\right)\right),$$

$$Q^+/(Q+Q^+) = \left(1 + \exp\left(-E^+/KT\right)\right). \qquad (24)$$

Here, E^- and E^+ stand for the energies of acceptors and donors with respect to the Fermi level.

A shift of the Fermi level in the direction of the conducting band results in the increase of Q^- and the decrease of Q^+. This implies that the number of molecules connected with the surface by acceptor bands rises while the number of those connected by donor bands falls. The quantity Q° has a maximum near the Fermi level. As a result, the adsorbability of the surface to a given kind of molecules depends on the position of the Fermi level that can be changed over the thickness of PS. This phenomenon can be used for the production of selective gas sensors based on PS.

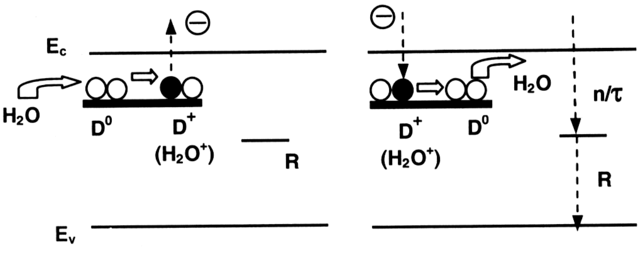


Fig. 3. Model of adsorption-recombination processes in PS

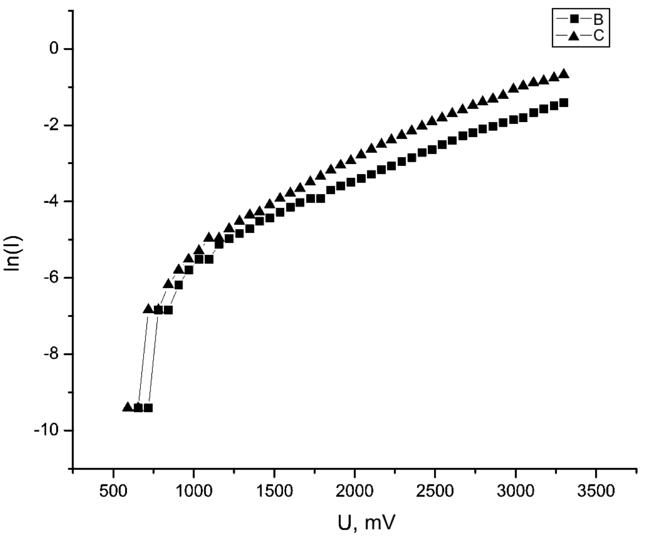


Fig. 4. Variation of the direct I—V characteristic structure under the adsorption in the presence of water vapors (▲) (■ — the data for the initial structure in dry air)

3.4. Kinetics of adsorption processes

In Fig. 2, we present a model of nondissociative adsorption of water molecules on the surface of a semiconductor. According to [10], the nondissociative adsorption of H_2O molecules can result in the injection of a free electron into the volume of a semiconductor accompanied by the formation of an H_2O^+ ion localized on the surface (Fig. 2,*a*) or the formation of an H_2O^+-electron dipole (Fig. 2,*b*).

Let's consider the kinetics of the current variation in a PS structure (formed on the basis of a p-type material) in the case of the injection of a free electron. Let the concentration of unoccupied donor states in PS, on which water molecules can be adsorbed, is m. Moreover, before the adsorption, let their concentration be equal to the total concentration of donors N_d. We suppose that, at the initial moment, the concentration of free carriers equals zero, $n = 0$. At the moment $t = 0$, water molecules begin to adsorb on the donor states in the form of H_2O^+ ions up to the concentration $N_d - m$. In the process of adsorption, there takes place the injection of free electrons into the conduction band, which results in an increase of the structure conductivity. A part of free electrons can recombine through recombination levels after a time interval τ or can be entrapped on the donor states occupied by H_2O^+ ions (the reaction of reduction of water molecules with their following desorption). Let's write the kinetic equations describing the variation of the concentrations of free carriers and unoccupied states:

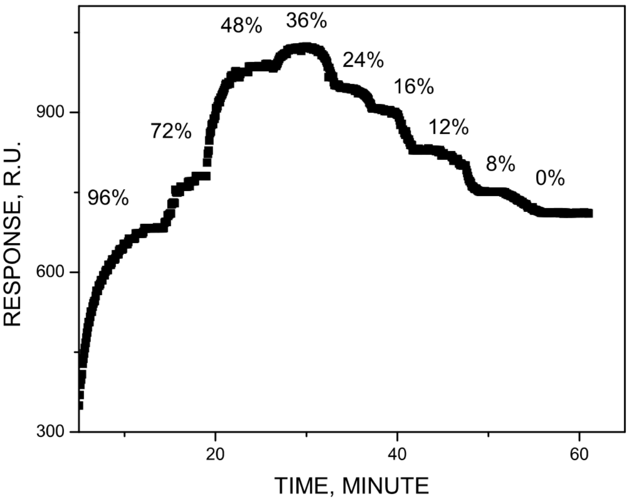
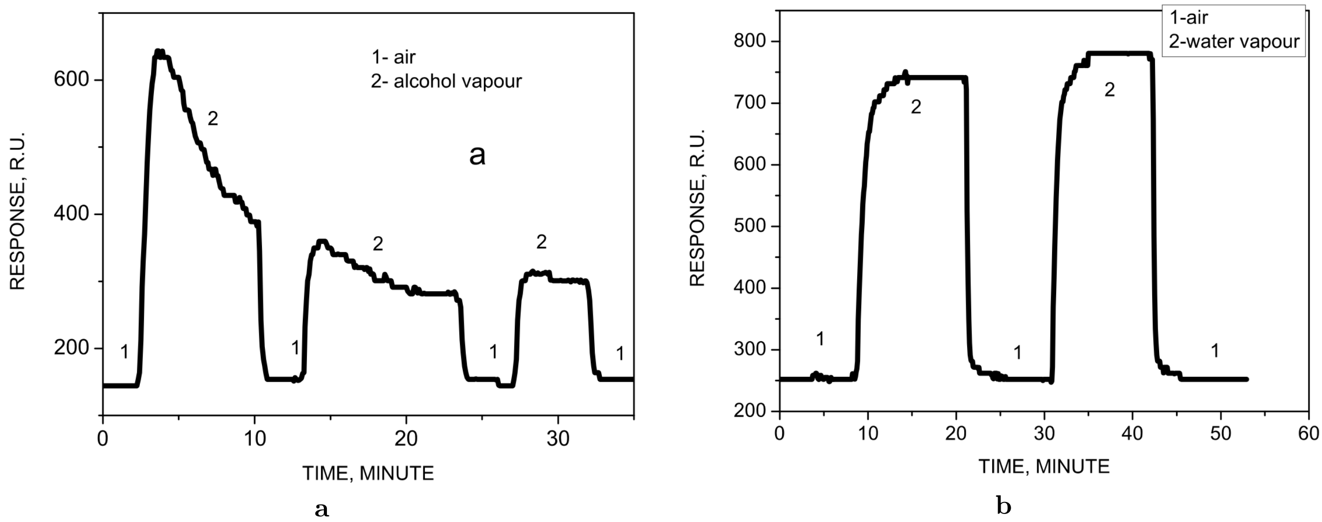


Fig. 5. Time dependence of the current through the MIS structure in the presence of a mixture of water and alcohol vapors of various concentrations over its surface

$$\frac{dn}{dt} = kNm - \frac{n}{\tau} - k_2 n(N_d - m), \qquad (25)$$

$$\frac{dm}{dt} = -kNm + k_2 n(N_d - m), \qquad (26)$$

where k, k_2 are the constants that characterize, respectively, the rate of adsorption of water molecules on the donor states accompanied with the formation of a free electron and the rate of entrapping of an electron by an H_2O^+ ion with the following desorption of the molecule, N is the concentration of water molecules, and τ is the lifetime of free electrons. In Eq. (25), the first term demonstrates that the rate of formation of free electrons is proportional to the product of the concentration of water molecules and that of unoccupied donor states. The second term describes a decrease of the concentration of nonequilibrium electrons due to the recombination through a recombination center. The third term shows that the rate of electron capture on a



a

b

Fig. 6. Influence of the adsorption of alcohol (a) and water (b) vapors on the conductivity of a MIS structure with an intermediate PS layer ($d = 150$ nm)

donor center is proportional to the product of the concentration of free carriers and the number of occupied donors. For the sake of simplicity, we consider that, at the beginning of the adsorption process, one can neglect inverse desorption processes. With regard for the initial conditions, the solution of the first equation gives $m = N_d \exp(-kNt)$, while the solution of the second one with regard for the initial conditions and the value of m allows one to obtain the variation of the concentration of free carriers.

$$n = \frac{kNN_d}{1/\tau - kN}(e^{-kNt} - e^{-t/\tau}), \qquad (27)$$

From formula (27), one can see that, if the probability of the recombination is high, $1/\tau > kN$, then the conductivity of a PS sample rises sharply at the beginning of the process of adsorption of water molecules with the following gradual decrease.

4. Experiment

Figure 4 shows the $I-V$ characteristics for a MIS structure with an intermediate layer of porous silicon in air and under the action of water vapors. The direct and inverse currents increase in the presence of water vapors.

In Fig. 5, we present the dependence of the current (in relative units) during the pumping of the mixture of water vapors and alcohol of various concentrations. We can see that, with decrease in the alcohol concentration, the current first increases, reaches the maximum at a concentration of alcohol of about 40% and then falls. Such a form of the dependence can be explained by the fact that the adsorptions of alcohol and water vapors influence the PS conductivity in different ways.

While the adsorption of water molecules on the PS surface results, first of all, in an increase of the conductivity due to the injection of free electrons, the precipitation of alcohol molecules decreases the adsorption rate of water molecules and changes the near-surface recombination. (The effects of the influence of organic molecules on the recombination properties of PS were observed earlier in our investigations of photoluminescence and DLTS [11,12]). In this case, the rise in the current which accompanies the increase of the alcohol concentration in the mixture from 0 to 36% is explained by the dominant process of passivation of recombination centers. But, at high alcohol concentrations (higher than 48%), adsorbed alcohol molecules on the PS surface block the adsorption of water molecules, which results in a decrease in the number of injected electrons and, correspondingly, in the current.

At high alcohol concentrations, the inequality $1/\tau > kN$ is satisfied (due to a decrease in the number of adsorbed water molecules), and we can observe a peak of conductivity in the kinetics at the beginning of the pumping of the gas mixture. In contrast, in the case where only water molecules are adsorbed, the current increases up to saturation monotonously (Fig. 6).

5. Conclusions

The solution of the diffusion-drift equations allowed us to analyze theoretically the influence of the adsorption on the electric properties of metal—tunnel dielectric—PS—silicon structures. In the process of adsorption, the current depends on the variation of the dielectric constant and the charges of the tunnel-transparent dielectric, PS, and the near-surface region of silicon. The proposed model of the adsorption processes is confirmed experimentally for the adsorption of a water-alcohol solution. The dependence of a response of the metal—PS—silicon structure on the alcohol concentration has a maximum at a concentration of alcohol of about 40% in the water solution. At the beginning of the adsorption process, we observed the effect of a abrupt increase in the conductivity followed by its gradual decrease up to saturation. That's why the barrier structures can be used as effective alcoholmeters.

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ВПЛИВ АДСОРБЦІЇ ПАРІВ СПИРТУ ТА ВОДИ НА ВОЛЬТ-АМПЕРНІ ХАРАКТЕРИСТИКИ МДН-СТРУКТУРИ З ПРОМІЖНИМ ШАРОМ ПОРУВАТОГО КРЕМНІЮ

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Резюме

Теоретично проаналізовано та експериментально досліджено вплив адсорбції парів води та спирту на вольт-амперні характеристики структур метал — поруватий кремній—кремній. З аналізу дифузійно-дрейфових рівнянь отримано вирази, які показують, як величина струму залежить від зміни діелектричної сталої та вбудованого заряду в поруватому кремнії (ПК). Максимальний струм досягається для 40%-вої концентрації суміші спирту у воді, що пояснюється конкуренцією між інжекцією електронів у ПК при недисоціативній адсорбції молекул води та пасивуючими властивостями адсорбованого шару молекул спирту. Запропоновано модель рекомбінації, що пояснює немонотонний характер зміни струму в процесі адсорбції суміші води зі спиртом.