

Mechanism of Formation of the Response of a Hydrogen Gas Sensor Based on a Silicon MOS Diode

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Abstract—Experimental data on the dependence of the flat-band voltage and relaxation time for the capacitance of the space-charge region in an MOS diode (Pd–SiO₂–*n*-Si) on the hydrogen concentration in a hydrogen/air gaseous mixture are discussed. It is assumed that variation in the flat-band voltage U_{fb} in an MOS structure with the thickness $d = 369$ nm subjected to a hydrogen/air gaseous mixture can be accounted for by the formation of dipoles in the Pd–SiO₂ gap due to polarization of hydrogen atoms (H_a). An analytical expression describing the dependence of variation in the flat-band voltage ΔU_{fb} on the hydrogen concentration n_{H_2} was derived. In MOS structures with $d \leq 4$ nm (or MOS diodes), the value of ΔU_{fb} is mainly controlled by passivation of the centers responsible for the presence of the surface acceptor-type centers at the SiO₂–*n*-Si interface by hydrogen atoms. Analytical expressions describing the dependences of ΔU_{fb} and the capacitance relaxation time in the space-charge region on n_{H_2} are derived. The values of the density of adsorption centers and the adsorption heat for hydrogen atoms at the Pd–SiO₂ and SiO₂–*n*-Si interfaces are found.

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1. INTRODUCTION

It is assumed in studies of the effect of molecular hydrogen on the capacitance–voltage (C – V) characteristics of metal–oxide–semiconductor (MOS) Pd–SiO₂–*n*-Si structures with a large thickness of the SiO₂ layer ($d \geq 100$ nm) that H₂ molecules dissociate to atoms (H_a) at the Pd electrode; it is also assumed that these atoms then diffuse and reach the Pd–SiO₂ interface [1]. Due to polarization of H_a atoms, a dipole layer is formed at this boundary; the electric field of this layer reduces the contact potential difference (U_c) between Pd and *n*-Si. As a result, the flat-band voltage (U_{fb}) is decreased and the C – V characteristic of the MOS structure shifts along the voltage axis. Eventually, a change in the capacitance of the MOS structure is observed at a fixed voltage.

In studies of MOS structures with a thin SiO₂ layer ($d \leq 4$ nm) [2, 3], it is assumed that the formation of a response to the effect of hydrogen is determined by electronic processes at the SiO₂–*n*-Si interface. It was shown in [3] that the relaxation time specifying the variation in the capacitance of a Pd–SiO₂–*n*-Si structure under the effect of hydrogen coincides with the relaxation time for diffusion hydrogen atoms from the Pd electrode to the SiO₂–*n*-Si interface. In this case, a decrease in the energy density of fast surface states is observed simultaneously.

It is stated on the basis of the above data that the defects responsible for the presence of fast surface states at the SiO₂–*n*-Si interface are passivated with hydrogen atoms. Possible mechanisms of passivation are discussed in detail, e.g., in [4].

It is also possible that H_a atoms are incorporated into the near-surface *n*-Si layer with subsequent passivation of the donor impurity [4, 5]. However, the passivating activity of hydrogen in *n*-Si is rather low at room temperature. A noticeable effect is observed at $T = 413$ K [6].

In this paper, we report the results of the studies whose aim was to estimate separately the contribution of electronic processes at the Pd–SiO₂ and SiO₂–*n*-Si interfaces to variation in U_{fb} under the effect of hydrogen on MOS structures with a tunneling-thin SiO₂ layer ($d \leq 4$ nm). In what follows, we refer to these structures as MOS diodes.

In order to solve the stated problem, we formed MOS structures with $d = 369$ nm and MOS diodes on the same *n*-Si substrate. The samples were fabricated using approximately the same technology as that described by Gaman et al. [7].

The C – V characteristics and time dependences of the capacitance $C(t)$ for MOS diodes and MOS structures in the H₂/air gas mixture were measured using an automated setup at a frequency of 1 MHz. The depen-

dence of ΔU_{fb} on the hydrogen concentration n_{H_2} was studied in the range of concentrations from 450 to 12×10^3 ppm, and the time dependence of the capacitance of an MOS diode was studied within the concentration range from 450 to 22500 ppm.

2. DEPENDENCE OF THE FLAT-BAND VOLTAGE FOR AN MOS STRUCTURE ON THE HYDROGEN CONCENTRATION

The flat-band voltage in an MOS structure is described by the expression [3]

$$U_{fb} = -[Q_t(\varphi_s = 0) + Q_d]/C_d - U_c, \quad (1)$$

where $Q_t(\varphi_s = 0)$ is the surface density of the charge of electrons captured by acceptor surface states in the situation of flat bands, in which case the surface potential at the SiO_2 - n -Si interface $\varphi_s = 0$; Q_d is the surface density of charge in the insulator; $C_d = \varepsilon_d \varepsilon_0 / d$ is the specific capacitance of the SiO_2 layer; $\varepsilon_d = 3.9$ is the relative permittivity of SiO_2 ; $\varepsilon_0 = 8.85 \times 10^{-12}$ F/m is the permittivity of free space; $U_c = -(\chi_{\text{Pb}} - \chi_{\text{Si}})/e$; χ_{Pb} , and χ_{Si} are the thermodynamic work functions for Pd and Si, respectively; and e is the elementary charge. In the H_2 /air gaseous mixture, the flat-band voltage decreases and can be written as

$$U_{fbg} = -[Q_t(\varphi_s = 0) + Q_d + \Delta Q]/C_d - U_{cg}, \quad (2)$$

where $\Delta Q > 0$ is the variation in the charge density at the SiO_2 - n -Si interface due to neutralization of negatively charged acceptor surface states with hydrogen atoms and U_{cg} is the contact potential difference in the gaseous mixture. Subtracting expression (2) from expression (1), we obtain

$$\Delta U_{fb} = \Delta Q/C_d + \Delta|U_c|, \quad (3)$$

where $\Delta U_{fb} = U_{fb} - U_{fbg}$ and $\Delta|U_c| = |U_c| - |U_{cg}|$.

In accordance with published data, the diffusion coefficient D_H of hydrogen atoms in palladium is 10^{-7} cm²/s [8] and in SiO_2 is 1.9×10^{-15} cm²/s [2] at $T = 297$ K. At the thickness of the palladium layer $d_{\text{Pd}} = 300$ nm, the time of the H_a diffusion is approximately equal to 10^{-2} s. The time of diffusion through the SiO_2 layer in the MOS structure is 7×10^5 s. Experiments have shown that $n_{H_2} = 10^3$ – 10^4 ppm the steady-state value of the capacitance of an MOS structure is established in 30–40 s. The above data indicate that the SiO_2 - n -Si interface does not play a significant role in the formation in the response of the MOS structure, while the diffusion of H_a atoms through the Pd layer is not the process that limits the variation in U_{fb} . Then, the variation in U_{fb} can be completely caused by adsorption and polarization of H_a atoms at the first Pd– SiO_2 interface [1], so that $\Delta U_{fb} = \Delta|U_c|$.

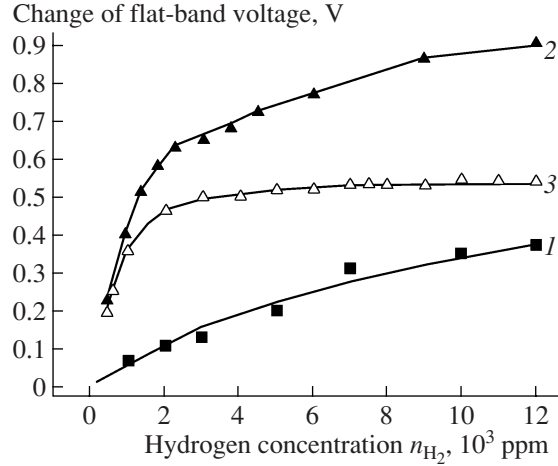


Fig. 1. Dependence of variation in the flat-band voltage on the hydrogen concentration: (1) $\Delta U_{fb} = \Delta|U_c|$ for an MOS structure (squares correspond to experiment, the solid line represents the results of calculation); (2) ΔU_{fb} for an MOS diode (experiment); and (3) $\Delta U_{fb}^* = \Delta U_{fb} - \Delta|U_c|$ (triangles represent the result of subtraction of curve 1 from curve 2, the solid line is the result of calculation).

Fairly tight binding between an electron and nucleus exists in a hydrogen atom. Therefore, we may assume that, as a result of polarization, the orbit of the electron shifts from the equilibrium position by the distance Δr , which is much smaller than the radius of the orbit. In this case, a dipole is formed with the moment $\mu_d = e\Delta r$. Assuming that the layer of dipoles is located in the air gap between Pd and SiO_2 and using the Ostrogradsky–Gauss theorem, we obtain

$$\Delta|U_c| = \mu_d N_{a1} / \varepsilon_0, \quad (4)$$

where N_{a1} is the surface concentration of H_a atoms adsorbed at the first interface. According to the solution of a kinetic equation for activated adsorption in the steady-state case, we have [9]

$$N_{a1} = N_{01} \eta_1 n_{H_2} / (1 + \eta_1 n_{H_2}), \quad (5)$$

where N_{01} is the surface density of the centers of adsorption of H_a atoms at the first interface, $\eta_1 = \eta_{01} \exp(-\Delta E_1/kT)$, η_{01} is a constant, $\Delta E_1 = E_{a1} - E_{d1}$ is the activation energy for the processes of adsorption and desorption of atoms (the parameter E_{d1} is also referred to as the differential adsorption heat, k is the Boltzmann constant, and T is the absolute temperature. When deriving expression (5), it was taken into account that the concentration of hydrogen atoms $n_H \approx n_{H_2}$. Using expressions (4) and (5), we obtain

$$\Delta|U_c| = \mu_d N_{01} \eta_1 n_{H_2} / [\varepsilon_0 (1 + \eta_1 n_{H_2})]. \quad (6)$$

The dependence of $\Delta|U_c|$ on n_{H_2} obtained experimentally is adequately described by expression (6) at $\mu_d N_{01} / \varepsilon_0 = 0.686$ V and $\eta_1 = 9.56 \times 10^{-5}$ ppm⁻¹ (Fig. 1,

curve 1). Using the value $N_{01} = 6 \times 10^{13} \text{ cm}^{-2}$ (see [1]), we obtain $\mu_d = 10^{-27} \text{ C cm}$. This value of μ_d corresponds to $\Delta r = 6.25 \times 10^{-9} \text{ cm}$, which is comparable to the radius of the electron orbit in a hydrogen atom ($r_0 = 5.29 \times 10^{-9} \text{ cm}$). This result makes it possible to assume that N_{01} exceeds the above value by approximately an order of magnitude. At $N_{01} = 6 \times 10^{14} \text{ cm}^{-2}$, we have $\mu_d = 10^{-28} \text{ C cm}$ and $\Delta r = 6.25 \times 10^{-10} \text{ cm} \ll r_0$. This value of μ_0 corresponds approximately to the estimate of this quantity reported in [1].

3. DEPENDENCE OF THE FLAT-BAND VOLTAGE IN AN MOS DIODE ON THE HYDROGEN CONCENTRATION

The time of diffusion of hydrogen atoms to the second interface at $d = 4 \text{ nm}$ is equal to 84 s. The time of establishment of steady-state values of capacitance and ΔU_{fb} for an MOS diode in relation to n_{H_2} is equal to $\sim(120\text{--}300) \text{ s}$. It then follows that the response to the effect of hydrogen in an MOS diode is formed due to adsorption of H_a atoms at both interfaces (Pd-SiO₂ and SiO₂-*n*-Si). Consequently, both terms on the right-hand side of expression (3) can play an important role in controlling the concentration dependence of ΔU_{fb} .

Since the materials of the field electrode and substrate in an MOS structure and an MOS diode are identical, the values of $\Delta|U_c|$ for these devices should be the same and feature similar dependence on n_{H_2} . Taking into account this circumstance, the problem amounts to an analysis of the concentration dependence of $\Delta U_{fb}^* = \Delta U_{fb} - \Delta|U_c| = \Delta Q/C_d$. In Fig. 1, we show experimental dependences of ΔU_{fb} (curve 2) and ΔU_{fb}^* (curve 3) on n_{H_2} . We assume that all hydrogen atoms adsorbed at the SiO₂-*n*-Si interface contribute to the variation in U_{fb} . Then,

$$\Delta U_{fb}^* = eN_{a2}/C_d, \quad (7)$$

where N_{a2} is the density of atoms adsorbed at the second boundary. In order to describe N_{a2} , we use expression (5), changing subscript 1 to subscript 2. After substitution of the expression for N_{a2} into (7), we obtain

$$\Delta U_{fb}^* = eN_{02}\eta_2 n_{\text{H}_2}/C_d(1 + \eta_2 n_{\text{H}_2}), \quad (8)$$

where N_{02} is the density of adsorption centers at the second boundary and η_2 and ΔE_2 have the same meaning as η_1 and ΔE_1 , but for the second boundary. It was established as a result of numerical simulation that, at $n_{\text{H}_2} \leq 4 \times 10^3 \text{ ppm}$, the dependence of ΔU_{fb}^* on n_{H_2} is described by formula (8) with $eN_{02}/C_d = 0.66 \text{ V}$ and $\eta_2 = 1.03 \times 10^{-3} \text{ ppm}^{-1}$. In the case of $n_{\text{H}_2} \geq 4 \times$

10^3 ppm , the quantity $\eta_2 n_{\text{H}_2}$ becomes constant: $\eta_2 n_{\text{H}_2} = 4$.

In Fig. 1, we show the data for the MOS diodes with $d \approx 3.7 \text{ nm}$ and $C_d = 9.3 \times 10^{-7} \text{ F/cm}^2$. Taking into account these data, it follows from the equality $eN_{02}/C_d = 0.66 \text{ V}$ that $N_{02} = 3.8 \times 10^{12} \text{ cm}^{-2}$. Thus, the density of adsorption centers at the SiO₂-*n*-Si interface is approximately two orders of magnitude lower than at the Pd-SiO₂ boundary.

The leveling-off of the dependence of ΔU_{fb}^* on n_{H_2} at $n_{\text{H}_2} \geq 4 \times 10^3 \text{ ppm}$ is caused by the fact that, in the range of hydrogen concentrations under consideration, a decrease in E_{d2} becomes noticeable as the occupancy of the adsorption centers $\theta = N_{a2}/N_{02}$ increases [1]. In this case, the value of E_{d2} decreases as θ is increased so that the product $[\exp(-\Delta E_2/kT)]n_{\text{H}_2}$ remains constant. It follows from obtained experimental data that we have $\theta = \eta_2 n_{\text{H}_2}/[1 + \eta_2 n_{\text{H}_2}] = 0.8$ at $n_{\text{H}_2} \geq 4 \times 10^3 \text{ ppm}$. The effect under consideration does not manifest itself at the Pd-SiO₂ interface. The dependence of $\Delta|U_c|$ on n_{H_2} does not level off until $n_{\text{H}_2} = 12 \times 10^3 \text{ ppm}$. This finding is accounted for by the fact that $N_{01} \gg N_{02}$ and $N_{a1}/N_{01} \approx 0.5$ at $n_{\text{H}_2} = 12 \times 10^3 \text{ ppm}$.

4. THE TIME DEPENDENCE OF THE CAPACITANCE OF AN MOS DIODE UNDER THE EFFECT OF HYDROGEN

In the case of fast inlet of hydrogen into the measurement chamber, we observe a transient process of establishment of steady-state values of $U_{fbg}(t)$ and the capacitance of the space-charge region (SCR) in an MOS diode $C_g(t)$. For an MOS diode in the mode of depletion, the dependence of the SCR capacitance on $U_{fbg}(t)$ is described by the expression reported in [3]. If the relatively slight dependence on the time t of the derivative $d(C_g^{-2})/dU$ is taken into account, the corresponding expression can be written as

$$C_g^{-2}(t) - C_{gst}^{-2} = 2[U_{fbg}(t) - U_{fbg}^0]/[K_0 S^2 \epsilon_r \epsilon_0 N_d b], \quad (9)$$

where $C_g(t)$ and C_{gst} are the capacitances of the SCR and gaseous mixtures at the time point t and at $t \rightarrow \infty$, U_{fbg}^0 is the steady-state value of U_{fbg} , K_0 is a constant, S is the area of the Pd electrode, ϵ_r is the relative permittivity of silicon, N_d is the donor-impurity concentration in Si, $b = 1 + e^2 N_t / C_d$, and N_t is the energy density of acceptor negatively charged surface states. Substituting the results of solution of the nonstationary kinetic equation for adsorption [9] into expressions (4) and (7)

(instead of N_{a1} and N_{a2}), we obtain the following time dependence of U_{fb} under the effect of a gaseous mixture:

$$\Delta U_{fb}(t) = U_{fb} - U_{fbg}(t) = \Delta|U_c| [1 - \exp(-t/\tau_{a1})] + \Delta U_{fb}^* [1 - \exp(-t/\tau_{a2})], \quad (10)$$

where

$$\tau_{a1} = \exp(E_{d1}/kT)/\nu(1 + \eta_1 n_{H_2}), \quad (11)$$

$$\tau_{a2} = \exp(E_{d2}/kT)/\nu(1 + \eta_2 n_{H_2}), \quad (12)$$

are the times of the adsorption relaxation of H_a atoms at the first and second interfaces and ν is the frequency of thermal vibrations of adsorbed atoms. It follows from expression (10) that

$$U_{fbg}(t) - U_{fbg}^0 = \Delta|U_c| \exp(-t/\tau_{a1}) + \Delta U_{fb}^* \exp(-t/\tau_{a2}). \quad (13)$$

After substituting expression (13) into (9), we obtain

$$C_g^{-2}(t) - C_{gst}^{-2} = M[\Delta|U_c| \exp(-t/\tau_{a1}) + \Delta U_{fb}^* \exp(-t/\tau_{a2})], \quad (14)$$

where

$$M = 2[K_0 S^2 \varepsilon_r \varepsilon_0 e N_{db}]^{-1}. \quad (15)$$

For an MOS structure, we have $\Delta U_{fb}^* = 0$ and

$$\ln[C_g^{-2}(t) - C_{gst}^{-2}] = \ln(M\Delta|U_c|) - t/\tau_{a1}. \quad (16)$$

Experimental data obtained at $n_{H_2} = 10^3$ ppm and $T = 297$ K confirm the linear dependence of $\ln[C_g^{-2}(t) - C_{gst}^{-2}]$ on t . In this case, $\tau_{a1} = 7.4$ s.

Setting $\nu = 10^{13} \text{ s}^{-1}$ in expression (11), we obtain $E_{d1} = 0.82$ eV at $T = 297$ K; this result is in good agreement with the heat of adsorption at $\theta \ll 1$ for the Pd-SiO₂ interface (see [1]).

The analysis of experimental data for the MOS diode is more complicated. Diffusion of hydrogen atoms through the SiO₂ layer was not taken into account when deriving expression (14). The concentration of hydrogen atoms at the SiO₂- n -Si interface grows at $t \geq 10$ s after the onset of the effect of gaseous mixture in accordance with the expression [3]

$$n_H(d, t) \approx n_H(0)[1 - \exp(-t/\tau_d)], \quad (17)$$

where $n_H(0)$ is the concentration hydrogen atoms at the Pd-SiO₂ interface and

$$\tau_d = 4d^2/\pi^2 D_H. \quad (18)$$

For an MOS diode with the dependence of ΔU_{fb} on n_{H_2} shown in Fig. 1, the calculated value $\tau_d \approx 33$ s.

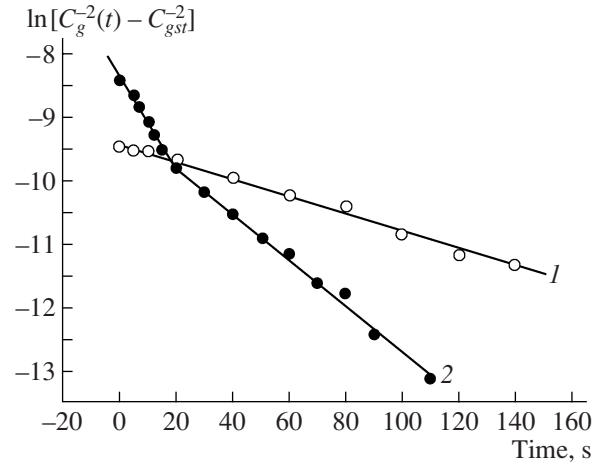


Fig. 2. Dependences of $\ln[C_g^{-2}(t) - C_{gst}^{-2}]$ on time at the hydrogen concentrations $n_{H_2} = (1) 900$ and $(2) 9 \times 10^3$ ppm. Circles correspond to experimental data and straight lines represent the results of calculation.

It follows from experimental data for an MOS diode that, in the range of n_{H_2} from 450 to 3×10^3 ppm, the difference $C_g^{-2}(t) - C_{gst}^{-2}$ depends exponentially on time t (Fig. 2, curve 1). However, at $n_{H_2} = 450$ ppm, the relaxation time of $C_g(t)$ exceeds τ_d by a factor of 4. This fact indicates that the time dependence of C_g in the region of small values of n_{H_2} is governed by adsorption (capture) of hydrogen atoms at the SiO₂- n -Si interface rather than by diffusion of these atoms through the SiO₂ layer; the relaxation time is equal to τ_{a2} . Dependence of τ_{a2} on the hydrogen concentration at $n_{H_2} < 10^3$ ppm can be described by expression (14) if we take into account that

$$E_{d2}(\theta) = E_{d2}(0) - \kappa\theta, \quad (19)$$

where $E_{d2}(0)$ is the activation energy at $\theta \ll 1$ and $\kappa = dE_{d2}/d\theta$. Experimental data for τ_{a2} at 450 and 900 ppm correspond to the following values of the parameters in expression (19): $E_{d2}(0) = 0.91$ eV and $\kappa = 0.0368$ eV.

At $n_{H_2} > 3 \times 10^3$ ppm, two linear portions are observed in the dependence of $\ln[C_g^{-2}(t) - C_{gst}^{-2}]$ on t (see Fig. 2, curve 2). The relaxation time τ_1 corresponding to the first portion of the curve decreases to approximately 10–11 s at $n_{H_2} = 7500$ ppm and remains constant until 22500 ppm (Fig. 3, curve 1). The relaxation time τ_2 corresponding to the second portion of the curve is independent of n_{H_2} and, on average, equals 33 s (see Fig. 3, curve 2), which coincides with τ_d . We would have assumed that $\tau_1 = \tau_{a2}$. However, it is at small values of t that adsorption of hydrogen atoms at the

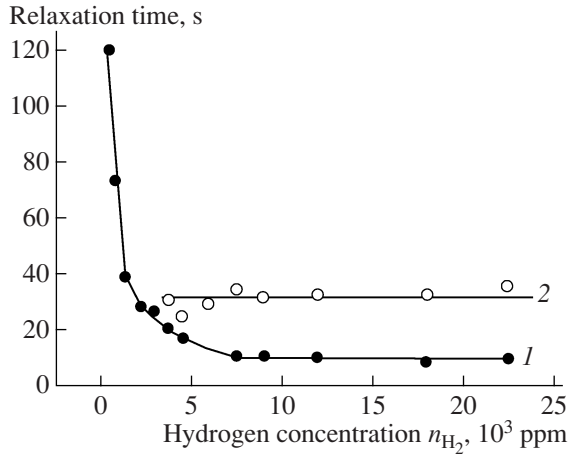


Fig. 3. Dependences of relaxation times for the SCR capacitance in an MOS diode (1) τ_1 and (2) τ_2 on the hydrogen concentration n_{H_2} .

SiO_2 - n -Si interface should be limited by diffusion through the SiO_2 layer.

We can adequately describe experimental curve 2 in Fig. 2 assuming that, at $n_{H_2} \geq 7.5 \times 10^3$ ppm, adsorption is completely controlled by diffusion of H_a atoms through the SiO_2 layer, since τ_{a1} and τ_{a2} are much smaller than τ_d . Taking into account this circumstance, we may assume that adsorption is of quasi-stationary character. At any point in time, the number of H_a atoms adsorbed and desorbed at the SiO_2 - n -Si interface are equal to each other. Writing this equality with expression (17) taken into account, we obtain

$$\Delta U_{fb}^*(t) = \frac{eN_{02}\eta_2 n_{H_2} [1 - \exp(-t/\tau_d)]}{C_d \{1 + \eta_2 n_{H_2} [1 - \exp(-t/\tau_d)]\}} \quad (20)$$

and

$$C_g^{-2}(t) - C_{gst}^{-2} = M \Delta U_{fb}^* \frac{\exp(-t/\tau_d)}{1 + \eta_2 n_{H_2} [1 - \exp(-t/\tau_d)]}, \quad (21)$$

where ΔU_{fb}^* is described by formula (8).

The results of calculation based on expression (21) at $n_{H_2} \geq 9 \times 10^3$ ppm adequately describe the experimental curve starting with $t \approx 5$ s (see Fig. 2, curve 2). In the range of the values of n_{H_2} from 10^3 to 7500 ppm, as the value of τ_{a2} decreases, we observe the transition from predominance of adsorption in the effect on the dependence $C_g(t)$ to the prevalence of the effect of diffusion of hydrogen atoms through the SiO_2 layer.

5. CONCLUSIONS

According to our studies, a decrease in the flat-band voltage U_{fb} as a result of the effect of hydrogen on MOS structures with a thick (369 nm) SiO_2 layer can be accounted for (in accordance with the hypothesis widely used in previous studies) by the formation of a dipole layer at the Pd- SiO_2 interface due to polarization of hydrogen atoms. In this case, the surface density of polarized H_a atoms should be $\sim 6 \times 10^{14}$ cm^{-2} , while the moment of the electric dipole is equal to 10^{-28} C cm. The rate of establishment of the steady-state value of capacitance of the MOS structure in the gaseous H_2 /air mixture features a relaxation time of adsorption of H_a atoms at the Pd- SiO_2 interface equal to 7 s.

In MOS structures with a thin SiO_2 layer ($d \leq 4$ nm) or in MOS diodes, a variation in U_{fb} is mainly caused by passivation of the centers responsible for the presence of fast acceptor surface states at the SiO_2 - n -Si interface. The surface density of these centers is 3.8×10^{12} cm^{-2} . The time dependence of the MOS-diode capacitance at the hydrogen concentration $n_{H_2} \geq 4 \times 10^3$ ppm is governed by diffusion of hydrogen atoms from the Pd electrode to the SiO_2 - n -Si interface. In the region of low hydrogen concentrations, establishment of steady-state values of the capacitance is delayed owing to large values of the relaxation time for adsorption (capture) of hydrogen atoms at the SiO_2 - n -Si interface.

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