

Biosensor Properties of SOI Nanowire Transistors with a PEALD Al_2O_3 Dielectric Protective Layer

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Abstract—The properties of protective dielectric layers of aluminum oxide Al_2O_3 applied to prefabricated silicon-nanowire transistor biochips by the plasma enhanced atomic layer deposition (PEALD) method before being housed are studied depending on the deposition and annealing modes. Coating the natural silicon oxide with a nanometer Al_2O_3 layer insignificantly decreases the femtomole sensitivity of biosensors, but provides their stability in bioliquids. In deionized water, transistors with annealed aluminum oxide are closed due to the trapping of negative charges of $<(1-10) \times 10^{11} \text{ cm}^{-2}$ at surface states. The application of a positive potential to the substrate ($V_{\text{sub}} > 25 \text{ V}$) makes it possible to eliminate the negative charge and to perform multiple measurements in liquid at least for half a year.

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1. INTRODUCTION

Electronic biochemical sensors based on semiconductor transistors are attractive first and foremost because of the easy processing of data on the biochemical interaction, i.e., the reliably measured drain current which exponentially depends on the potential of a virtual gate consisting of a molecular “anchor” and an analyte bioparticle trapped by it on the surface of a semiconductor [1–3]. Unfortunately, the formation of a functional layer of anchors on the surface of a semiconductor, which is stable in air and in liquid to the adsorption of other atoms and molecules, is an extremely difficult problem due to the requirement to provide the minimum thickness of such a layer to retain sensor sensitivity [4]. The advantage of silicon-nanowire transistors is the formation of a native oxide film up to 2 nm thick on their surface in air, which provides femtomole sensitivity to adsorbed atoms and molecules [5, 6]. However, such a film is relatively loose and readily traps various atoms and molecules over a day in laboratory air, and is even dissolved in bioliquid [7]. The stability of the electrical characteristics of the sensor in liquid does not exceed a few hours, which leads to either sensor loss or the need for special chemical processing to regenerate its surface before measurements. At the same time, it is known [8] that dense insulator films, in particular, Al_2O_3 , grown by atomic layer deposition (ALD), have high barrier characteristics with respect to water-molecule diffusion. At the same time, our previous attempts to

deposit aluminum oxide on nanowire transistors from laser plasma by pulsed laser deposition (PLD) led to nanowire structure breakage on the silicon-on-insulator (SOI) surface of the sensors. The possible cause of breakage was elastic strains in the insulating PLD films, exceeding the critical values for separating the joined surfaces of the fabricated SOI structures [9, 10].

The objective of this work is to search for and study the properties of protective insulating coatings of the surface of silicon-nanowire transistors, providing sensitivity, long-term stability, and the simple regeneration of electronic biochemical sensors on their basis.

2. EXPERIMENTAL

In this study, an insulator consisting of sequentially deposited monomolecular layers of amorphous aluminum oxide $a\text{-Al}_2\text{O}_3$ was grown by plasma enhanced atomic layer deposition (PEALD) in which the first precursor sorbed on the surface of the nanowire structures was trimethylaluminum (TMA); oxygen plasma ions were the second precursor. A remote plasma source in the FlexAl reactor (Oxford Instruments, UK) provided an atomic oxygen O^* concentration of $10^{13}\text{--}10^{14} \text{ cm}^{-3}$ at a density of positively charged ions of $\sim 10^9 \text{ cm}^{-3}$ and a pressure of $p = 15 \text{ mTorr}$ in the working area. The sample stage was under zero potential, which, in combination with the above plasma parameters had a “soft” stimulating effect on the heterogeneous TMA oxidation reaction without damag-

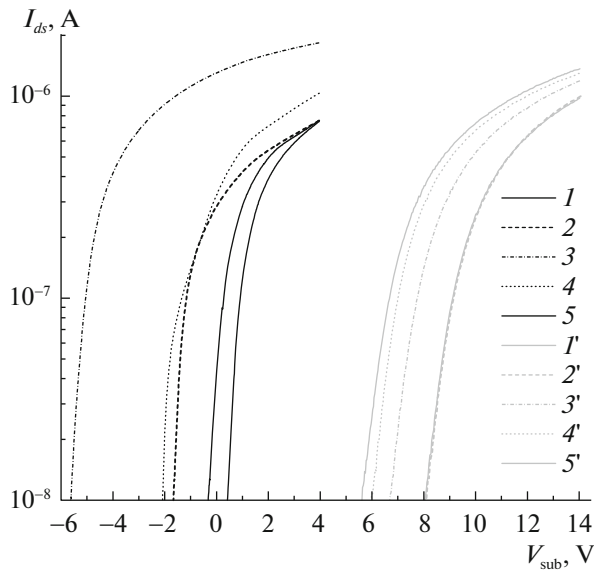


Fig. 1. Experimental gate–drain characteristics of n -channel transistors in the accumulation mode (I' – $5'$) before and (I – 5) after deposition of an Al_2O_3 layer 5.2 nm thick in the ALD process at 200°C and annealing in forming gas at 425°C . The curve numbers correspond to numbers 1–5 of transistors on the crystal.

ing the nanowire SOI sensors. The choice of this ALD version is also due to the possibility of performing the process at temperatures lower by 150 – 200°C than when using water vapor as the second precursor in the reaction with TMA at equal quality of the Al_2O_3 films.

The experiment was performed on two series of samples with different stage temperatures during Al_2O_3 deposition; the stage was in thermal contact with unhoused chips on the SOI wafer. At 100°C , 12 single layers (1.2 ± 0.1 nm) were deposited; and 200°C , 40 single layers (5.2 ± 0.1 nm) of α - Al_2O_3 were deposited onto the open surface of channels of the silicon nanowire transistors with SiO_2 native oxide, fabricated on SOI structures using the technology described in previous studies [5, 6].

The gate–drain characteristics of the transistors were measured in the experiment under different conditions on the nanowire surface. In this case, the substrate was used as the gate and potential sweep was applied to it. The transistor drain current was measured at a potential of $+0.1$ V at the drain and 0 V at the source. The wire surface state was controlled by the medium composition (air/water/bovine serum albumin solution) and the platinum-electrode potential (in the case of liquid on the wire surface). The platinum-electrode potential was varied from $+2$ V (when recording bovine serum albumin (BSA)) to -8 V (in the case of sample washing); however, it was zero in most measurements.

Measurements in water were performed in a “well”-type microfluidic cell immediately after pumping deionized freshly prepared water from a MilliQ system (18.2 M Ω cm, pH 7) with a rate of 2 – 8 $\mu\text{L/s}$ using a pump. The platinum contact was introduced into the cell itself and was in the immediate vicinity of the crystal surface. During measurements, water was not pumped (the pump was turned off). During recording of the BSA-molecule concentration, washing was not performed. Solutions with increasing BSA concentrations were sequentially pumped through the cell. Washing was performed after measurement with the highest concentration (10^{-8} mol/L or 0.66 $\mu\text{g/mL}$) of BSA molecules.

After depositing α - Al_2O_3 at a temperature of 100°C , all measured transistors were functional; however, the current–voltage gate–drain characteristics $I_{ds}(V_{\text{sub}})$ exhibited a shift of the threshold gate voltage from the substrate side to $-(200$ – $180)$ V and a decrease in the slope of the subthreshold characteristics, which indicates the presence of a positive charge to 3×10^{12} cm^{-2} in the aluminum oxide, and the formation of states with densities of up to 1×10^{12} cm^{-2} eV^{-1} in the intermediate layer at the $\text{Al}_2\text{O}_3/\text{Si}$ heterointerface, which is probably due to a low deposition temperature (Fig. 1).

To achieve a sensitivity to proteins on the order of 10^{-15} mol/L, the charge–state density of the top insulator and the protein-binding layer on the silicon surface should not exceed $\sim 5 \times 10^{11}$ cm^{-2} eV^{-1} , and leakage currents should be < 10 nA/ cm^2 [4]. To improve the electrical characteristics of the silicon and insulator heterojunction, to achieve the required sensitivity, selective trapping, and regeneration of the functional nanowire sensors based on SOI nanotransistors, a comparative study of the electrical and electrochemical properties of protective insulators based on aluminum oxide Al_2O_3 with increased permittivity was carried out at various deposition and subsequent annealing temperatures (Fig. 1). It was shown that as the ALD process temperature is increased to 200°C , the charge and density of surface states decrease by an order of magnitude to $\sim 2.0 \times 10^{11}$ cm^{-2} , according to the data of measurements of the current–voltage (I – V) characteristics of the transistors (Fig. 1).

Furthermore, as seen in Fig. 1, aluminum oxide annealed in the forming gas (4% H_2 and 96% Ar at 425°C , 15 min) provides the largest slope of the I – V characteristic, the least density of the built-in charge Q_{FB} and surface states N_{SS} ($< 5.0 \times 10^{11}$ cm^{-2} eV^{-1}) at the heterointerface even in comparison with the native silicon oxide.

One of the ways of further decreasing the charge and density of states in structures with metal dioxides on silicon, is the use of rapid pulsed heat treatments at temperatures of $\sim 900^\circ\text{C}$ [11].

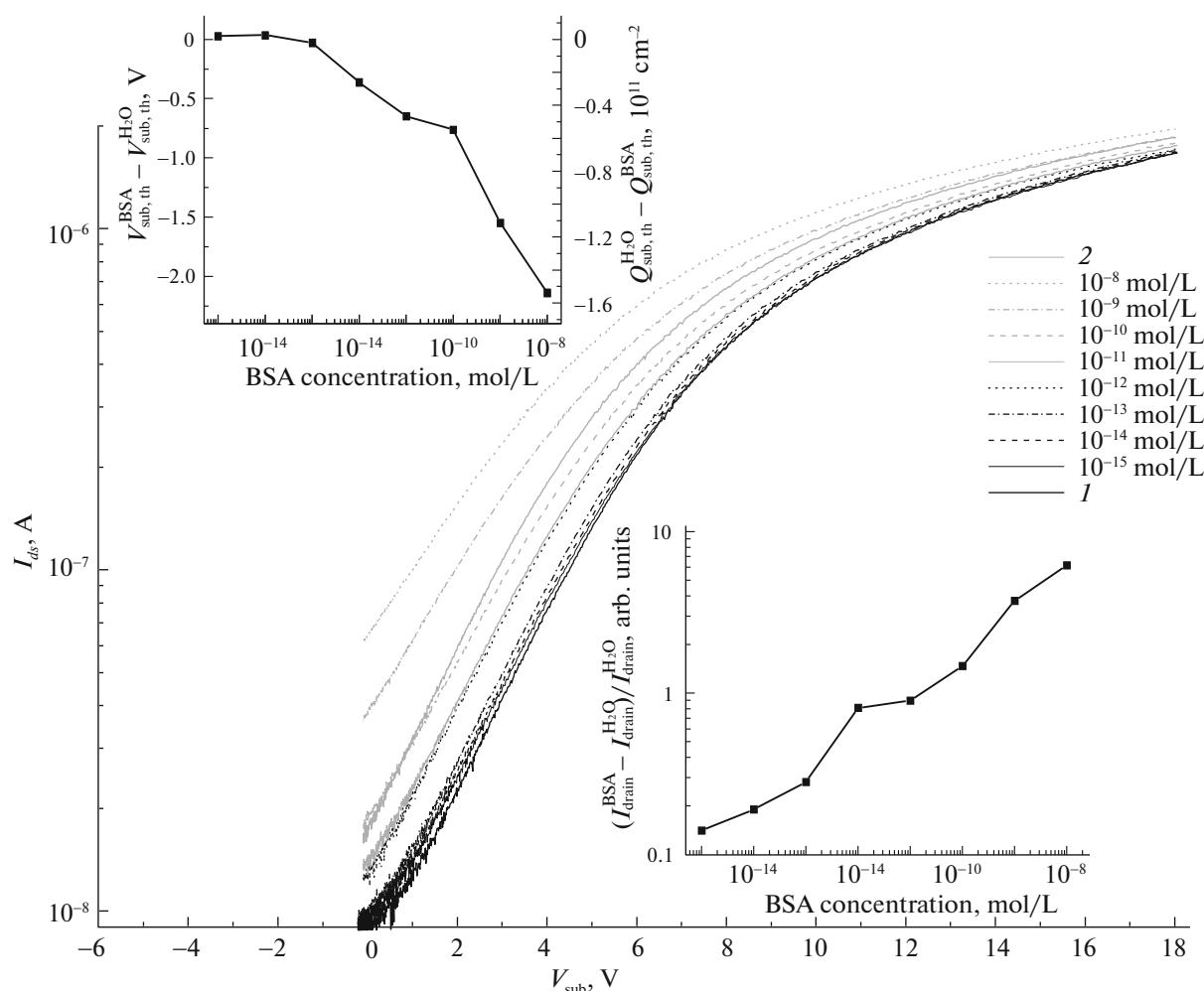


Fig. 2. Gate–drain characteristics of *n*-channel transistors with an ALD– Al_2O_3 layer 5 nm thick in the symmetric mode with a positive potential of +2 V in bioliquids with various BSA concentrations (indicated). Characteristics in water (1) before BSA recording and (2) after washing. The concentrations were measured sequentially beginning with low and ending with high ones. The insets show the BSA concentration dependences of the relative change in the drain current I_{drain} at +2 V applied to the substrate (bottom), the difference in the threshold voltages $V_{\text{sub,th}}$ with and without BSA molecules, and the corresponding difference in the charges $Q_{\text{sub,th}}$ with and without BSA molecules according to formula (1) (top); superscripts BSA and HgO indicate the presence or absence of BSA.

3. RESULTS AND DISCUSSION

Aluminum-oxide deposition significantly increases the slope of the gate characteristics and decreases the density of surface states and the negative charge trapped at the native-silicon-oxide surface (Fig. 1). However, an increase in the scatter of the threshold characteristics of the transistors, especially after annealing, points to the necessity of additional surface preparation of the nanowire structures with native oxide before depositing the Al_2O_3 insulator by the PEALD method. Figure 2 shows the results of determination of the sensitivity to BSA molecules for sensors with an additional insulating Al_2O_3 layer deposited by the ALD method, which makes it possible to

retain close-to-femtomole sensitivity to test proteins at a positive potential applied to the electrode in liquid.

Although it is known that the antisymmetric mode of two-gate transistor operation is more sensitive to surface states in comparison with the symmetric mode [12, 13], nevertheless, the best results in the case at hand were obtained at a positive potential applied to the substrate and in water. At a positive potential applied to the substrate and water, sensitivity according to a change in the threshold shift begins with 10^{-13} mol/L of BSA molecules (see the inset in Fig. 2); at the same time, sensitivity according to a change in the subthreshold current is observed for both 10^{-15} mol/L of BSA molecules and at other substrate potentials (see the inset in Fig. 2). Determination of the shift of the threshold

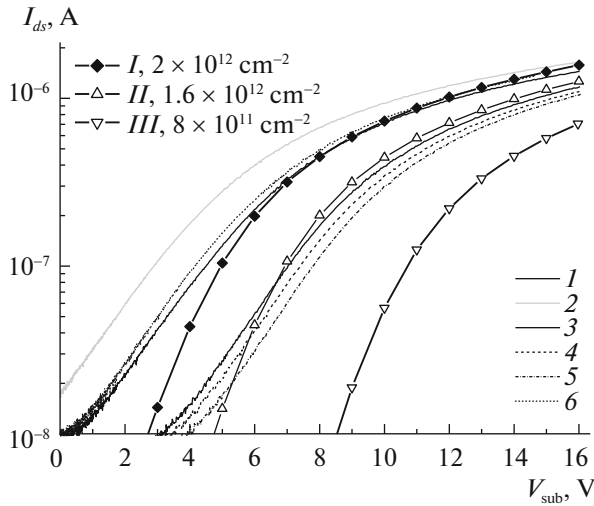


Fig. 3. Gate–drain characteristics of *n*-channel transistors with an ALD- Al_2O_3 layer 5.2 nm thick after measurements in bioliquid with BSA and cleaning in a deionized water flow at a potential of -8 V applied to the electrode in water: (1) measurement in water before BSA recording, (2) measurement immediately after recording BSA and cleaning in flowing water, (3–5) three sequential measurements after cleaning in flowing water at -8 V applied to the contact to the water, (6) measurement after 1000 min of storage in stagnant water. For comparison, calculated characteristics I–III are presented.

voltage $\Delta V_{\text{sub,th}}$ is useful to estimate the sensitivity of sensors operating in the key mode of the comparison of transistor currents with analyte (An) molecules on the surface and without them in a buffer solution (Buf)

as $\Delta V_{\text{sub,th}} = V_{\text{sub,th}}^{\text{An}} - V_{\text{sub,th}}^{\text{Buf}}$. We determined the change in the effective charge ΔQ_{eff} on the surface as

$$\Delta Q_{\text{eff}} = \frac{C_{\text{ox}}}{q} \Delta V_{\text{sub,th}}, \quad (1)$$

where q is the elementary charge, C_{ox} is the capacitance of the buried insulator of the SOI structures, $\Delta V_{\text{sub,th}}$ is the transistor threshold-voltage shift determined from the sensor's gate characteristics $I_{\text{ds}}(V_{\text{sub}})$ as $V_{\text{sub,th}} = \max[d^2 I_{\text{ds}}/dV_{\text{sub}}^2]$.

The cause of the detected dependence of the charge on the potential in liquid can be the structural features of the protective insulator Al_2O_3 . Aluminum oxide has more polar bonds than silicon dioxide, hence, a higher sensitivity to the charge of adsorbed particles. It is known that the mechanism of BSA-molecule adsorption on aluminum oxide depends also on its structural modification [14]. For example, the interaction with a BSA molecule occurs by the electrostatic mechanism for the high-temperature and most stable α - Al_2O_3 phase, whereas adsorption occurs by the ion–ion mechanism for the δ - Al_2O_3 phase, as well as for hydroapatite (HPA). Al_2O_3 films grown by ALD are

initially amorphous, but annealing at 425°C can cause γ - Al_2O_3 phase nucleation similar in properties to the δ - Al_2O_3 phase [15, 16]. In the experiments described, upon annealing of an amorphous aluminum oxide α - Al_2O_3 layer 5.2 nm thick, nanoscale metastable phase nuclei can also be formed; however, this problem calls for additional investigation.

According to the published data and performed capacitance–voltage measurements, the ALD deposition of high-*k*-insulators decreases the flat-band voltage in all cases, which indicates a decrease in the negative charge on the native-silicon-oxide SiO_2 surface after long-term storage. In this case, according to the data of the capacitance–voltage and spectroellipsometric measurements, additional 15-min annealing of ALD- Al_2O_3 at 425°C in the forming gas (Ar 96% and H_2 4%) barely changed the insulator properties, except for an additional decrease in the negative charge. For annealed ALD- Al_2O_3 , a significant decrease in the density of surface states, to $N_{\text{SS}} = (1\text{--}2) \times 10^{11} \text{ cm}^{-2} \text{ eV}^{-1}$, was achieved as well. The permittivities ϵ measured using the capacitance–voltage characteristics and spectral ellipsometry were less than the reference values in almost all films, which is probably caused by the decreased density ($\sim 90\text{--}95\%$) of the thin ALD films.

The effect of regeneration of the sensor-element surface with an unannealed amorphous insulator α - Al_2O_3 on its parameters, in particular, on the possibility of cleaning the sensor surface by changing the potential polarity in the bioliquid, was additionally studied. It was shown that the analyzed test BSA molecules (Fig. 3) and particle adsorbed after three-day storage in stagnant water (Fig. 4) are removed in a deionized water flow at a potential of -8 V applied to the electrode in liquid for 10 min, as suggested by the transistor-threshold return to the initial value of ~ 4 V for $< 10^3$ min, which then remains almost unchanged for 4×10^3 min (Figs. 3 and 4). The charge changes from 0.5×10^{12} to $2.0 \times 10^{12} \text{ cm}^{-2}$ according to the results of numerical calculations using the TCAD Synopsys package (Figs. 3 and 4) and in accordance with formula (1). The slope of the experimental curves is smaller due to the measuring resistor (Fig. 5). Furthermore, calculation by formula (2) also yields the water-trapped charge $\Delta Q = -1.5 \times 10^{11} \text{ cm}^{-2}$, which corresponds to the assumed deprotonation.

The experiments suggest that the threshold voltage is almost unchanged after the cleaning of BSA molecules for 4×10^3 min; then, after the repeated application of a negative potential to the electrode in water, it slowly returns to the initial value in the time $t > 10^2$ min (Fig. 4), and according to the data of Fig. 3, in $t < 10^3$ min. Immediately after immobilization of the test BSA molecules ($\sim 10^{-8} \text{ mol/L}$), the threshold voltage decreases by ~ 3 V (Fig. 2); however, then these molecules are removed by a deionized-water flow at a potential of -8 V applied to the electrode in liquid. For

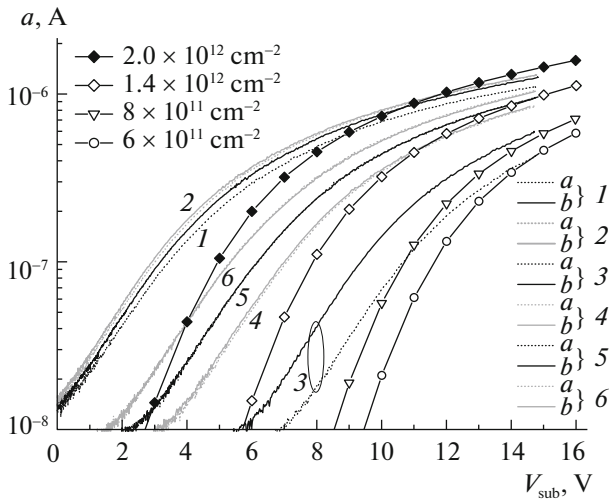


Fig. 4. Gate–drain characteristics of *n*-channel transistors with an ALD–Al₂O₃ layer 5.2 nm thick: (1) after 3-day storage in water, (2) after 10-min cleaning in a deionized water flow, (3) after additional 10-min cleaning in a water flow at a potential of –8 V applied to the electrode in water, (4) after additional 30-min storage in stagnant water, (5) after additional 30-min storage in stagnant water, (6) after additional 60-min storage in stagnant water. All experimental curves were measured two times successively: first, curve *a*, right after it, curve *b*, the time interval between measurements was several minutes. For comparison, the calculated characteristics are presented.

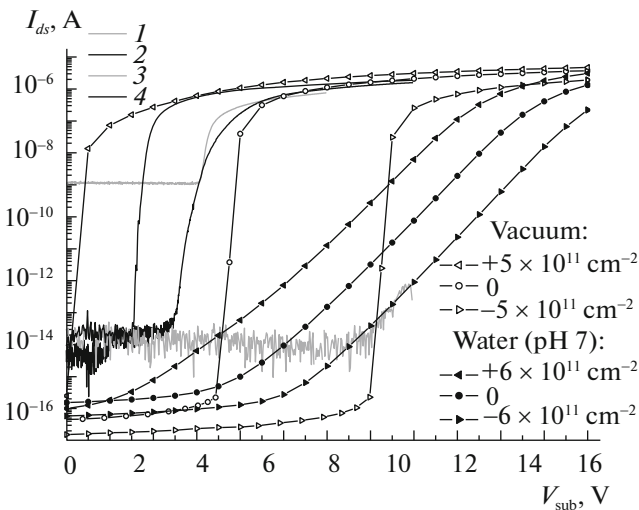


Fig. 5. *I*–*V* characteristics of the transistor no. 2 with an annealed ALD–Al₂O₃ layer 5.2 nm thick: (1, 2) the first measurement and measurement after 2-month storage in air, respectively, (3) the characteristic after half a year of experiments, measured in the range from +8 to –8 V, (4) measurement with a sweep from +8 to –8 V after several minutes of crystal storage at +80 V on the substrate. Measurements were performed in air. For comparison, the calculated characteristics for the wire surface contact with water and free space.

the transistor with *a*-Al₂O₃, it is possible to shift the characteristic to the right over 10 min by applying a negative voltage to the liquid, while BSA molecules shift it to the left due to effective positive charge. The results of a similar experiment with native silicon oxide showed that 30-min storage in air after special chemical cleaning of the sensor surface and washing in deionized water leads to an increase in the negative charge to (2–4) × 10¹² cm^{–2}, and then its slow relaxation over ~10⁴ min [14]. The native oxide is stable in air, although the density of surface states increases from 3 × 10¹² to 4 × 10¹³ cm^{–2} in ~10³ min in this case. However, it rapidly degrades in water or in bioliquid within a time of <10³ min [7]. The data we obtained count in favor of the predominantly electrostatic nature of the interaction of BSA molecules with unannealed insulator *a*-Al₂O₃ grown by PEALD, unlike the ion–ion interaction for the δ-Al₂O₃ phase [15].

The field and temporal drift of the electrical properties of transistors with Al₂O₃ insulator annealed in the forming gas was also studied. The hysteresis of the source–drain characteristics of such transistors in air did not exceed 1 V at sweeps from –8 to +8 V (Fig. 4). Figure 5 also shows the calculated and experimental *I*–*V* characteristic of transistor no. 2, measured with a 2-month interval for two limiting resistors with different limits of current measurements, 10^{–9} and 10^{–16} A. A comparison of the calculated and experimental *I*–*V* characteristics shows that the change in the threshold voltages is also no more than 0.4 V which corresponds to a change in the surface charge of <1 × 10¹¹ cm^{–2}.

Significant changes in the transistor characteristics were observed both during measurements in water with a bias *V*_{sub,max} increased to 24, 50, and 80 V and during subsequent long-term storage in air, which caused blocking to 70% of the transistors. Blocking of all transistors in water occurs due to a change in the surface potential defined by an expression related to a solution with pH_{PZC} at which charge is lacking on the insulator surface [16],

$$\psi_0 = -2.303 \left(\frac{RT}{\Delta z F} \right) (\text{pH} - \text{pH}_{\text{PZC}}), \quad (2)$$

where pH 7 and pH_{PZC} 8 for sapphire (*a*-Al₂O₃), Δ*z* is the change in the charge of surface groups (+1 or –1) during protonation/deprotonation, and *F* = *qN*_A is the Faraday number. Then the change in the potential is ψ₀ = ±59 mV which is identical to recent experimental data [17]. Since the transistor is blocked in the case at hand, the change was Δ*z* = –1. It was reversible, if the substrate potential was increased to *V*_{sub,max} > +24 V. The dependence *V*_{sub,th}(*V*_{sub,max}) in water was ambiguous; however, the characteristics are restored after pumping with water for ~10³ min, while an increase in the substrate potential to *V*_{sub,max} = 80 V reduces the recovery time to 10² min. However, at such a potential in water, half the transistors fail after 10⁵ min, and the

others that remain functional show a density of states higher by a factor of 3–4. After the next 10^5 min, $\sim 25\%$ of the transistors remain functional.

The current–voltage characteristic of transistor no. 2 after half a year of measurements in water and storage (2.6×10^5 min) in laboratory air is shown in Fig. 5 for comparison with the calculated characteristics in water and in air. Initially, the transistor is blocked almost in the entire measurement range from -8 to $+8$ V due to a negative charge of 6×10^{11} cm $^{-2}$ adsorbed on the insulator surface. After applying a high positive potential to the substrate ($+80$ V) in air, the negative charge in the insulator became positive to 8×10^{11} cm $^{-2}$ and then slowly (~ 30 min) restored to the initial value of $\sim 6 \times 10^{11}$ cm $^{-2}$. As follows from comparison of the experimental and calculated characteristics in free space, the subthreshold slope for experimental transistors with protective ALD- Al_2O_3 insulator is no less than that for the calculated transistors (Fig. 5).

After long-term storage in air, an adsorbed layer with negative charge on amphoteric surface states is formed on the surface of the transistor's sensitive area, which decreases after the application of a high positive bias to the substrate, $V_{\text{sub,max}} = 80$ V. The cause of negative-charge removal due to charge coupling in fully depleted two-gate transistors is the same as when applying a negative potential of -8 V to the electrode in water [12]. It is associated with water-molecule dissociation in an electric field in the water film existing on the transistor surface in air, followed by negative charge neutralization due to the formation of γ - and δ - Al_2O_3 phase inclusions in the insulator, which feature catalytic activity after annealing [15, 18, 19].

4. CONCLUSIONS

It was shown that the PEALD technology makes it possible to grow insulating aluminum oxide layers on already fabricated silicon-nanowire transistor structures before being housed. As the temperature of the PEALD process of Al_2O_3 -layer growth is increased to 200°C , the density of states at the heterointerface and the built-in charge decrease. In this case, the number of defect nanotransistors does not increase.

The native-silicon-oxide coating with a protective aluminum-oxide layer of nanometer thickness allows the formation of nanowire-transistor biosensors which are potentially more sensitive due to the subthreshold characteristic slope, than sensors with a protective layer of the same thickness, but made of silicon oxide and even sensors without a protective layer, but with a much higher density of states on the surface. Furthermore, the protective aluminum-oxide layer promises the higher stability of biosensors to bioliquids and more stable electrical characteristics. The native-silicon-oxide coating with aluminum oxide of nanometer thickness insignificantly lowers the femtomole sensi-

tivity of nanowire-transistor biosensors, but provides their stability in bioliquids. An additional aluminum-dioxide coating allows, at least for BSA-type proteins without their trapping at markers, sensor-element regeneration and washing by simple polarity reversal of the electrode in the bioliquid.

The additional annealing of PEALD- Al_2O_3 in the forming gas at 425°C provides the maximum slope and the least density of built-in charge Q_{FB} and surface states N_{SS} ($< 5.0 \times 10^{11}$ cm $^{-2}$ eV $^{-1}$) at the heterointerface with silicon even in comparison with transistors with only native silicon oxide, but increases the scatter of their threshold voltages.

In deionized water, transistors with annealed aluminum oxide are blocked due to trapping of the negative charge of $< (1-6) \times 10^{11}$ cm $^{-2}$ at surface states, which biases the threshold voltage $V_{\text{sub,th}} > +30$ V. A positive potential applied to the substrate ($V_{\text{sub}} > +24$ V) makes it possible to remove this charge and to perform multiple measurements in liquid at least for half a year.

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