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Study of structural and electrical properties of the reconstructed Si(111) surface after lithium adsorption

© D.A. Tsukanov,[1,2] M.V. Ryzhkova[1]

[1]Institute of Automation and Control Processes, Far Eastern Branch of Russian Academy of Sciences,
690041 Vladivostok, Russia
[2]Far Eastern Federal University, Vladivostok, Russia,
690090 Vladivostok, Russia
e-mail: tsukanov@iacp.dvo.ru

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The results of a study of the crystal structure and surface conductivity of a Si(111) silicon substrate with a reconstructed surface after deposition of submonolayer doses of lithium are presented. We used the method of low energy electron diffraction to study changes in the structure of the crystal lattice of the surface, as well as the four-point probe method for measuring the conductivity of substrates under *in situ* conditions. As the initial surfaces, we used the Si(111)7 × 7 reconstruction of an atomically clean silicon substrate, and the reconstructions obtained by adsorption of 1 ML of gold, lead, and bismuth atoms: Si(111)$\beta-\sqrt{3}\times\sqrt{3}$−Au, Si(111)1 × 1−Pb and Si(111)$\beta-\sqrt{3}\times\sqrt{3}$−Bi, respectively.

Keywords: silicon surface, adsorption, surface reconstruction, surface conductivity, low-energy electron diffraction.

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Introduction

Recently, there has been an increased interest in the study of the structural and electrical properties of lithium films, as well as its silicon alloys, due to their potential use as elements for lithium-ion batteries [1]. In principle, studies of films of alkali metals, including lithium, on the silicon surface have been conducted for a long time [2–5]. It is already known that on the atomically clean surface of Si(111) silicon, the deposition of submonolayer coatings of lithium or other alkali metals leads to the formation of ordered reconstructions: 3 × 1, 6 × 1, 1 × 1, etc. [5]. However, recently a number of phenomena have been discovered that allow us to pay attention to the use of alkali metals as a material for adsorption on the surface with atomic reconstructions. One of the interesting features of the interaction of alkali metals with the reconstructed surface Si(111)$\sqrt{3}\times\sqrt{3}$−Au is the formation of a homogeneous surface and the appearance of metallic surface states in the semiconductor bandgap [6]. On the other hand, it was found that the interaction of sodium with the surface of atomically clean silicon Si(111)7 × 7 leads to the disappearance of metallic states and a shift in the band bending by 60 meV above the Fermi level [7].

In this paper, the task is to study the effect of adsorption of lithium atoms on the structure and properties, in particular, electrical conductivity, of silicon samples with a reconstructed surface. Si(111)$\beta\sqrt{3}\times\sqrt{3}$−Au, Si(111)$\beta\sqrt{3}\times\sqrt{3}$−Bi and Si(111)1 × 1−Pb systems were selected as the reconstruction for which lithium deposition was carried out. For example, in the latter, Si(111)1 × 1−Pb, lead atoms form an ordered superlattice on a single-crystal surface, there is no diffusion of lead atoms into the substrate volume and there is no chemical reaction with silicon atoms (there is no silicide formation) [8]. In principle, the submonolayer Pb/Si(111) system can be considered as a model system of a non-reactive interface. In addition, recently this system has been of interest from the point of view of the formation of two-dimensional lead-based alloys with elements such as gold [9], thallium [10], bismuth [11], etc. It has been shown that these systems demonstrate a whole set of promising properties, such as spin splitting, superconductivity, and so on. Surface Si(111)$\beta\sqrt{3}\times\sqrt{3}$−Au contains partially filled metallic surface states, but at the same time it has a band gap of 50 meV, and there is also a strong band bending in the area of space charge layer near the surface [12]. Thus, this phase represents a surface with a carrier-enriched subsurface layer with weakly filled metallic surface states. Moreover, this surface is characterized by the presence of a large number of defects in the form of domain walls caused by the tension of the upper atomic layer, which can be removed by additional adsorption of a small number of atoms, for example, indium, thallium, as well as alkali metals, sodium and caesium, resulting in a defect-free homogeneous surface of type Si(111)$h-\sqrt{3}\times\sqrt{3}$−(Au,A), where adsorbed atoms In, Tl, Na, Cs [6] can be used as component A. Surface reconstruction of Si(111)$\beta\sqrt{3}\times\sqrt{3}$−Bi contains 1 monolayer (ML) of bismuth atoms, but unlike the Si(111) 1 × 1−Pb phase has a semiconductor character of the band structure, and for this phase, the effect of spin splitting of the Rashba type [13] was previously detected. However, for the practical use of spin transport for this system, it is necessary to increase its electrical conductivity by adding charge carriers to unfilled spin-split states, which was previously demonstrated when sodium was deposited on this surface [14,15].

This paper presents the results of a study of the reconstructed silicon surface after deposition of lithium atoms on it. A feature of the work is the use of the method of diffraction of low-energy electrons to observe the crystal structure of the surface, as well as to measure the surface conductivity of the obtained structures in ultrahigh vacuum immediately after the formation of ultrathin films without the removal of samples in air.

1. Experimental conditions

To conduct research in this study, the RIBER DEL-300 ultra-high vacuum chamber with a base pressure of $\sim 10^{-10}$ Torr was used. The unit is equipped with a low-energy electron diffractometer (LEED) from Omicron Nanotechnology and a four-probe head for electrical measurements. The head is placed on a retractable manipulator, tungsten probes are located at the corners of a square with an interprobe distance of 0.6 mm. The Keithley 6221 precision DC and AC power source with a low noise level and the Keithley 2182A nanovoltmeter were used as meters, which in the delta system mode provide reliable and reproducible characterization of the materials under study by separating current and voltage measurements. Measurements of the surface conductivity of the samples were carried out under stable conditions in ultrahigh vacuum and at room temperature. The value $\sigma = I/U$ [S] was used as the unit of measurement of surface conductivity, where I and U are the current values directly measured in the experiment (from 5 to $50\,\mu$A) and voltage, respectively, when passing direct current alternately through four pairs of probes with further averaging of measurement results.

Rectangular silicon plates of the size $15 \times 5 \times 0.45\,\text{mm}^3$, doped with phosphorus, with resistivity 300−1700 Ohm·cm were used as samples. Cleaning of the sample surface was carried out first by washing and cleaning in organic solvents, drying. Next, the samples were placed in an ultrahigh vacuum chamber, where they were first degassed at a temperature of up to 600°C for several hours by passing alternating current, and then a short-term heating (flash) was carried out to a temperature of 1250°C. The samples prepared in this way showed a clear 7×7 diffraction pattern (Fig. 1, *a*), characteristic of the atomically pure silicon surface.

In this paper, the following sources of adsorbed materials were used: lead and bismuth were sprayed from tantalum tubes, gold was evaporated from a tungsten basket, a getter dispenser from SAES Getters was used as a lithium source. The deposition rate of the adsorbed material was calibrated using LEED by diffraction patterns from the reconstructed surface with a known number of atoms (1 ML of the adsorbate coating corresponds to the concentration of atoms $7.8 \cdot 10^{14}\,\text{cm}^{-2}$ for the non-reconstructed surface Si(111)). The error in the coating of adsorbates calibrated in this way can be estimated as ± 0.05 ML. A Knudsen cell heated by direct current was used to evaporate fullerene. The deposition rate of fullerene was ~ 0.1 ML per minute and was determined by deposition of C_{60} on the surface of $\text{Si}(111){-}\alpha\sqrt{3} \times \sqrt{3}{-}\text{Au}$ with the formation of a fullerite-like lattice (1 ML corresponds to the concentration of fullerene molecules $1.15 \cdot 10^{14}\,\text{cm}^{-2}$ [16]).

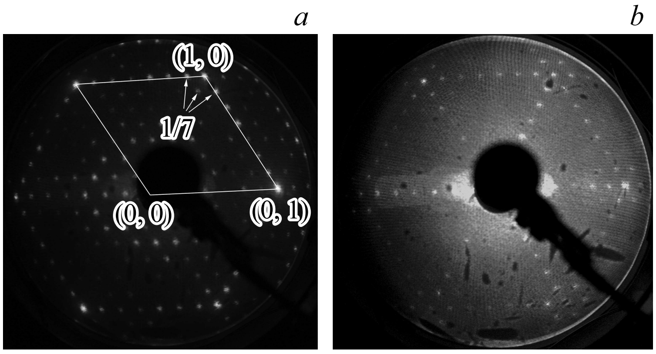

Figure 1. LEED patterns of atomically pure substrate surface $\text{Si}(111)7 \times 7$ (*a*) and surface $\text{Si}(111)\delta(7 \times 7)$ obtained after 1 ML of lithium deposition at room temperature (*b*). The energy of the primary beam is 40 eV.

2. Experimental results and their discussion

The lithium adsorption experiment was first conducted on an atomically clean silicon surface $\text{Si}(111)7 \times 7$ in order to clarify the effect of lithium atoms on the electrical conductivity of a silicon substrate. It is known that the adsorption of lithium at room temperature leads at the initial stage of deposition to the clustering of Li [17] atoms, which in the LEED picture manifests itself as a gradual attenuation of 7×7 superspots, and then when lithium coverage is about 1 ML, a LEED picture is formed, which is usually indicated as $\delta(7 \times 7)$ [18], where superspots of the order 1/7 remain only near the main spots 1×1, as well as on the line between these reflexes (Fig. 1, *b*). This behavior of the LEED patterns is explained in the study [19] by the fact that adsorbed lithium atoms gather in clusters inside cells with a structure of 7×7, displacing silicon adatoms, while the angular adatoms of these cells remain intact until the formation of clusters of the first three is completed, then six lithium atoms, and with a further increase in the concentration of lithium atoms, an unordered layer of lithium atoms is formed on the surface of the substrate. At the same time, there is a strong decrease in the work function on the surface, up to -1.74 eV for a surface with a diffraction pattern of $\delta(7 \times 7)$, when the lithium coverage is 1 ML [20]. The study of surface conductivity

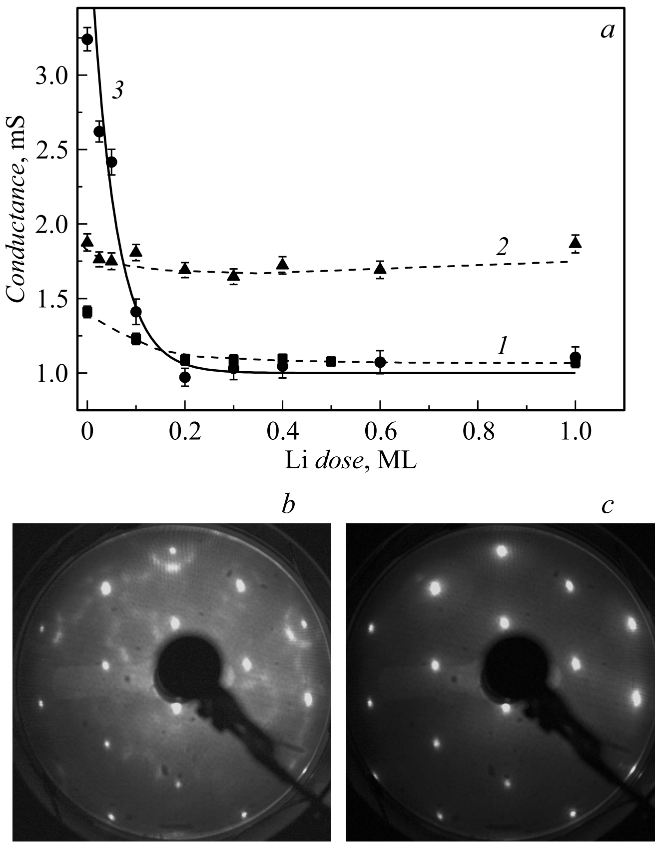

Figure 2. Surface conductivity of silicon samples after deposition of submonolayer lithium doses on the surface of Si(111)7×7 (graph *1*), Si(111)$\beta\sqrt{3}\times\sqrt{3}$–Au(graph *2*) and Si(111)$h\sqrt{3}\times\sqrt{3}$–(Au,Li) (graph *3*, approximation) at room temperature (*a*). LEED Patterns for the surface Si(111)$\beta\sqrt{3}\times\sqrt{3}$–Au (*b*) and Si(111)$h\sqrt{3}\times\sqrt{3}$–(Au,Li) (*c*). The energy of the primary beam is 40 eV.

showed (Fig. 2,*a*) that the electrical conductivity of the sample after lithium deposition changes slightly, there is only a slight decrease in electrical conductivity at the initial stage of sputtering. A similar decrease in conductivity was previously observed, for example, after sodium deposition on a Si(111)7×7 substrate in the study [7] for substrates as *n*-type ($1-10$ Ohm·cm), as well as *p*-type ($3900-6400$ Ohm·cm), and the changes in conductivity were explained as a result of the disappearance of metallic states on the surface due to the destruction of the ordered structure of the surface, as well as due to the band bending and the associated decrease in the work function, which is a characteristic phenomenon for alkali metals adsorbed on the surface as semiconductors, so are metal films [20–23]. In our case, a slight decrease in electrical conductivity is also observed approximately at the coverage of adsorbed lithium about $0.1-0.2$ ML, which can be interpreted as observation of the metal–insulator transition at room temperature [24].

One of the interesting features in the adsorption of alkali metals, as well as indium and thallium on reconstructed Si(111) surfaces is the ability to form a homogeneous surface from reconstructions that initially have a large number of domain walls, which include Si(111)$\alpha-\sqrt{3}\times\sqrt{3}$–Au, Si(111)$\beta-\sqrt{3}\times\sqrt{3}$–Au and Si(111)$6\times6$–Au [6,25,26], while filling of metal bands of surface states [27] is observed and, accordingly, an increase in surface conductivity [26,28]. It was found that lithium behaves similarly in these reconstructions. Thus, the results of the experiment show that the deposition of about 0.15 ML of lithium on the surface of Si(111)$\beta-\sqrt{3}\times\sqrt{3}$–Au at a temperature of 250°C also leads to the formation of a homogeneous surface, as can be seen from the LEED pictures in Fig. 2,*b* before lithium deposition and in Fig. 2,*c* after lithium deposition. Fig. 2,*b* shows the diffraction pattern of the initial surface Si(111)$\beta-\sqrt{3}\times\sqrt{3}$–Au, on which, in addition to reflexes of the order of $\sqrt{3}$, stripes are visible, the origin of which is caused by the presence of a large number of domain walls on the surface of [29]. After lithium deposition on the surface of Si(111)$\beta-\sqrt{3}\times\sqrt{3}$Au, heated to 250°C, the stripes on the diffraction pattern disappear, and only the clear spots of $\sqrt{3}\times\sqrt{3}$ remain (Fig. 2,*c*). For a given surface, denoted as Si(111)$h-\sqrt{3}\times\sqrt{3}$–(Au,Li) the value of the surface conductivity was measured, which was 3.24 ± 0.08 mS, which is significantly higher than for the initial surface with phase Si(111)$\beta-\sqrt{3}\times\sqrt{3}$–Au. However, lithium deposition at room temperature on the same surfaces only leads to a decrease in electrical conductivity. Fig. 2,*a* shows the changes in conductivity after lithium deposition on the surface of Si(111)$\beta-\sqrt{3}\times\sqrt{3}$–Au, and also to the surface Si(111)$h-\sqrt{3}\times\sqrt{3}$–(Au,Li), and the deposition in both cases was carried out only at room temperature. It can be seen that the conductivity decreases, the diffraction patterns at the same time demonstrate a gradual attenuation of reflexes. The decrease in conductivity can be described by the function

$$\sigma = \sigma_0 \exp(-D/D_0) + \sigma_S \text{ [30]},$$

where σ_0 — the initial value of the conductivity of the substrate Si(111)$h-\sqrt{3}\times\sqrt{3}$–(Au,Li), σ_S — conductivity of the pure substrate, D — dose of precipitated lithium, D_0 — saturating coating of adsorbed lithium atoms. Saturation in this case should be understood as the amount of adsorbed lithium required to completely remove the metallic surface states of the Si(111) $h-\sqrt{3}\times\sqrt{3}$–(Au,Li) surface phase, when the conductivity is carried out only through the volume of the substrate. Fig. 2,*a*, a solid line shows the approximation of the conductivity changes for the SSi(111)$h-\sqrt{3}\times\sqrt{3}$–(Au,Li), and the best value of the parameter D_0 is 0.25 ML.

It is well known that the interaction of alkali metal atoms is strongly manifested when they are adsorbed onto metal surfaces [31,32]. In the experiment, a common feature is clearly visible — the surface conductivity decreases with an increase in the dose of lithium atoms on the substrate. Thus, at the initial stage of adsorption, lithium atoms bind to the substrate, transfer a charge to it and become positively charged ions. In contrast to the semiconductor

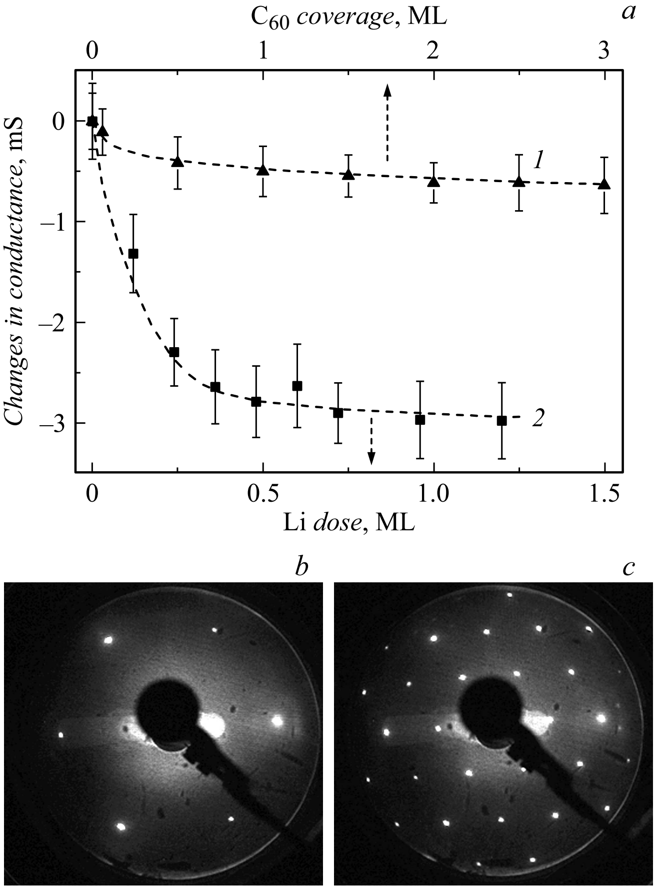

Figure 3. Changes in the surface conductivity of silicon samples after deposition of fullerene molecules C_{60} (graph *1*) and submonolayer lithium doses (graph *2*) on the surface of Si(111)$1\times1-$Pb (*a*). LEED Patterns for the surface Si(111)$1\times1-$Pb (*b*) and Si(111)$2\times2-$(Pb,Li) (*c*). The energy of the primary beam is 56 eV.

surface, the saturation coating estimate for alkali metals on the surface of metal films, for example, for caesium, ranges from 0.4 (in the case of caesium adsorption on the surface of Au(111) [33]) to 0.33 ML (in the similar case with adsorption caesium on Ag(111) [34]). This process is accompanied by a decrease in the electrical conductivity of the phase until saturation of the surface conductivity occurs after deposition of 0.2−0.3 ML of lithium.

Fig. 3, a shows the changes in conductivity after deposition on the reconstructed surface of Si(111)$1\times1-$Pb (the LEED picture is shown in Fig. 3, *b*) fullerene molecules C_{60} (graph *1*) and lithium atoms (graph *2*). This reconstruction is metallic, and the lead coating is 1 ML, when a reconstruction of 1×1 [8] is formed on the surface. This surface demonstrates a fairly high electrical conductivity through metallic surface states, i.e., in fact, the uppermost layer of [35] atoms participates in the conductivity. It is known that the adsorption of fullerene molecules onto this surface leads to the formation of a densely packed ordered layer of molecules C_{60} at room temperature [36]. Since the molecules C_{60} are electronegative and take on the electrons of the upper layer of the substrate [37], interacting with metallic surface states, they are in this sense an electron acceptor [38]. Such removal of electrons from the surface states also leads to a drop in the surface conductivity of the Si(111) $1\times1-$Pb reconstruction due to a decrease in the concentration of charge carriers in it. However, due to the fact that the concentration of fullerene molecules in 1 ML ($1.15\cdot10^{14}\,\mathrm{cm}^{-2}$) is significantly less than the atoms in the upper layer of the reconstructed surface ($7.8\cdot10^{14}\,\mathrm{cm}^{-2}$), a significant proportion of charge carriers remain in surface states and continue to participate in electrical conductivity.

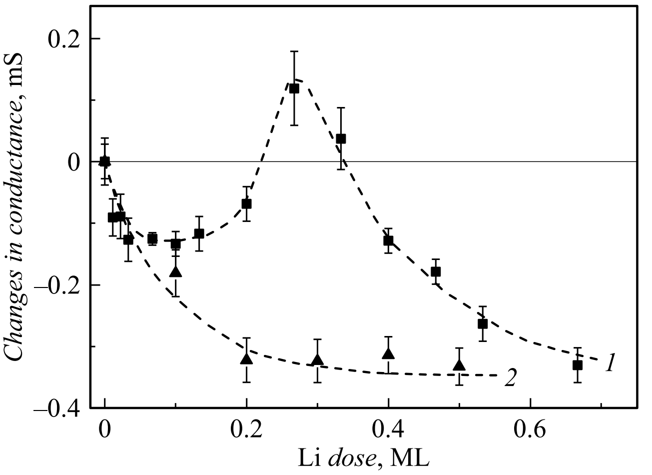

Figure 4. Changes in the surface conductivity of silicon samples after lithium deposition on the Si(111)$\beta-\sqrt{3}\times\sqrt{3}-$Bi surface (graph *1*). For comparison, a graph of changes in surface conductivity after lithium deposition on the surface of Si(111)7×7 is shown (graph *2*).

On the other hand, the adsorption of lithium onto this surface should have the opposite effect: the adsorbed lithium, being electropositive, gives electrons to the metal, where they are in close proximity to the surface and shield the adsorbed ions. It turns out that as a result of such interaction, the metallic surface states are destroyed, and a localized charge is formed on the surface. Usually, this process is visible on angular-resolution photoemission spectra as a blurring of the zone of surface states up to their complete disappearance, in addition, a change in the position of the bulk states is observed due to a decrease in the work function [26]. Thus, for this phase, as for the previous case with the surface Si(111)$h-\sqrt{3}\times\sqrt{3}-$(Au,Li), the electrical conductivity decreases to values close to the value of the electrical conductivity of the pure substrate due to the disappearance of the conduction channel through the metal layer. The difference is that when lithium is deposited on the surface of Si(111)$1\times1-$Pb a change in the concentration of adsorbed lithium atoms is accompanied by a change in the crystal structure of the surface. At a lithium dose corresponding to the coating of the adsorbed atoms 0.25 ML, a LEED pattern of 4×1 is observed, and then with an increase in the lithium dose above 0.3 ML, a surface structure of 2×2 is formed (Fig. 3, c).

Consider another surface reconstruction, Si(111)$\beta-\sqrt{3} \times \sqrt{3}-$Bi, which, unlike the previous two-dimensional systems, is semiconductor. This reconstruction contains 1 ML of bismuth atoms forming trimmers at positions T_1 on a volume-like silicon lattice [39]. The electronic structure of this surface has hole surface states below the Fermi level, as well as free surface states above the Fermi level, and none of them crosses the Fermi level [13]. The surface conductivity for this substrate, as our measurements show, almost does not differ from the conductivity of the original pure surface Si(111)7×7. Fig. 4 shows the changes in conductivity after lithium deposition on the Si(111) $\beta-\sqrt{3} \times \sqrt{3}-$Bi surface at room temperature, and for comparison, conductivity measurements after lithium deposition on a clean surface of the Si(111)7×7 substrate are presented. It can be seen that at the initial stage of dosing the surface with lithium (up to 0.2–0.3 ML), the surface conductivity decreases, however, when lithium dose is 0.3 ML on the surface of Si(111)$\beta-\sqrt{3} \times \sqrt{3}-$Bi there is a small peak of conductivity. In the study [40], using calculations from first principles, it was shown that the adsorption of 1/3 ML of alkali metals on the surface of Si(111)$\beta-\sqrt{3} \times \sqrt{3}-$Bi leads to the filling of the previously free surface states, as a result, this zone with an increase in the alkali metal coating falls down and crosses the Fermi level. The surface conductivity in this case increases due to an increase in the number of carriers, which we observe on the graph.

Conclusion

As a result of experiments conducted to study the effect of lithium adsorption on the crystal structure and electrical conductivity of Si(111) silicon with reconstructions previously formed on its surface, the possibility of controlled changes in the surface conductivity of substrates depending on the dose of deposited lithium, as well as the structural and electronic properties of the initial surface is shown. In this paper, new surface reconstructions were observed in the (Pb,Li)/Si(111) system: 4×1 and 2×2. It has been found that in the general case, lithium deposition on surface reconstructions with filled surface states having a metallic character, such as Si(111)$h-\sqrt{3} \times \sqrt{3}-$(Au,Li) or Si(111)$1 \times 1-$Pb, leads to a decrease in surface conductivity. If there are unfilled surface states on the surface, then under certain conditions, for example, lithium deposition on the Si(111)$\beta-\sqrt{3} \times \sqrt{3}-$Au surface reconstruction at a temperature of 250°C, or adsorption of 1/3 ML of lithium on the surface of Si(111)$\beta-\sqrt{3} \times \sqrt{3}-$Bi at room temperature, the electrical conductivity of the substrate increases. In general, these results demonstrate that at the initial stages of adsorption (at the concentration of adsorbed lithium corresponding to the coating of the material up to 1 ML), under certain conditions, lithium causes both structural changes on the surface and affects the electrophysical properties of the substrate surface, in particular, its surface conductivity.

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Conflict of interest

The authors declare that they have no conflict of interest.

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