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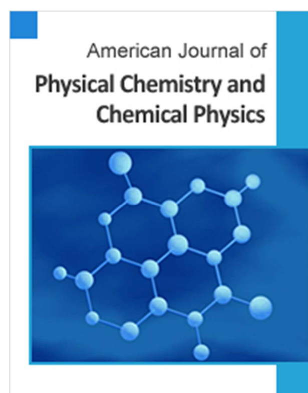

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Investigation of Influence of Cleanliness of the Surface Substrate on Formation and Transformation of Silicon-Germanium Nanoclusters

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Abstract
Established that the degree surface perfection to be seen as an essential part of the general task of preparing a clean surface before the process of formation of SiGe nanoclusters and the suppression of their transformation from nanoscale to microsizes. A small displacement of the surface atoms of complex structures on a clean surface with the formation of bonds of Ge -Ge or Si- Si is beneficial for self-organization of nanoclusters SiGe.

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1. Introduction

Study of the surface phenomena plays an important role in investigation of processes of nucleation, growth, and self-organization of nanoclusters [1–25]. The significance of the processes occurring on the blank substrate surface increases with decreasing their sizes. The specific properties of the near-surface area can even determine the characteristics of the nanocluster-based products. The difference in properties of atoms on the surface and in bulk exists in single crystals. The inner part of the single crystal can be described as a regular repetition of a unit cell, which typically contains a small number of atoms. The number of the nearest neighbors for the atom on the surface is less than the coordination number as a result of the sharp break of the crystal lattice. Surface atoms have unsaturated bonds and can therefore have a very high chemical activity. As a result, the crystal surface is usually coated with one or more layers of the compounds formed by the reaction between the surface atoms and the environment. It is often assumed that the position of atoms on the surface is different from their positions in normal crystal lattice of the given crystal. Thus, the properties of the solid body area immediately adjacent to the surface may differ from the properties of the inner part of the sample. In fact, the surface atoms of the single crystal are located absolutely in other surroundings than the atoms in bulk and thus have different activity and occupy different positions. The surface structure can also change as a result of chemisorption of foreign atoms. Research of clean surfaces is the best way to get information about the fundamental properties of the surface [9, 26–30]. In this case it is important not only to prepare a clean surface but also to keep it in this state in the process of self-assembly of nanoclusters. In particular, silicon-germanium (SiGe) nanoclusters.

In practice one can approximate to a perfectly clean surface only in one extent or another. A surface of the material, which chemical composition is uniform with the second inner layer of atoms and which is covered with not more than one hundredth of a monolayer of foreign atoms is considered clean surface. That is why the investigation of the effect of the surface treatment of blank substrates on the process of formation and transformation of SiGe nanoclusters is so important.

The purpose of this work – investigation of the influence of purity of surface of substrates on process of formation and transformation of the silicon-germanium nanoclusters.

2. The Methodology and the Experimental Part

The size of nanoclusters was determined by beveled samples and chips using scanning electron microscope S-4800 (Hitachi, Japan). The size of nanoparticles (nanoclusters) was also determined by using scanning probe microscope "Ntegra Prima" in atomic force microscopy mode.

The analysis of silicon-germanium nanoclusters was performed on the surface of plates at an angle of 30° without decoration and metal deposition.

The surface structure change of the blank substrates, Ge, and SiGe nanoclusters was analyzed on the basis of Raman spectroscopy (RS). Raman spectra by optical phonons were obtained by irradiating the structures with Ar-laser with a wavelength λ = 514.5 nm, and were recorded on DFS-52 spectrometer. The spectra were recorded at room temperature. Effect of the type of surface treatment of the blank substrates on the surface density and sizes of SiGe nanoclusters was investigated at a temperature of 540–560°C, which surface was cleaned: clean silicon substrates in hydrogen chloride environment (1a) followed by annealing in argon (b), and also in hydrogen environment (c) followed by annealing in nitrogen and hydrogen environment followed by annealing (d). Annealing was carried out at a temperature of 900–1000°C, substrates with ultrathin (4 nm) layer of silicon dioxide - with annealing in hydrogen environment (e), with ultrathin (4 nm) layer of silicon nitride layer with annealing in hydrogen environment (f) and substrates with ultrathin (4 nm) layers of dysprosium oxide (g) and yttrium (k).

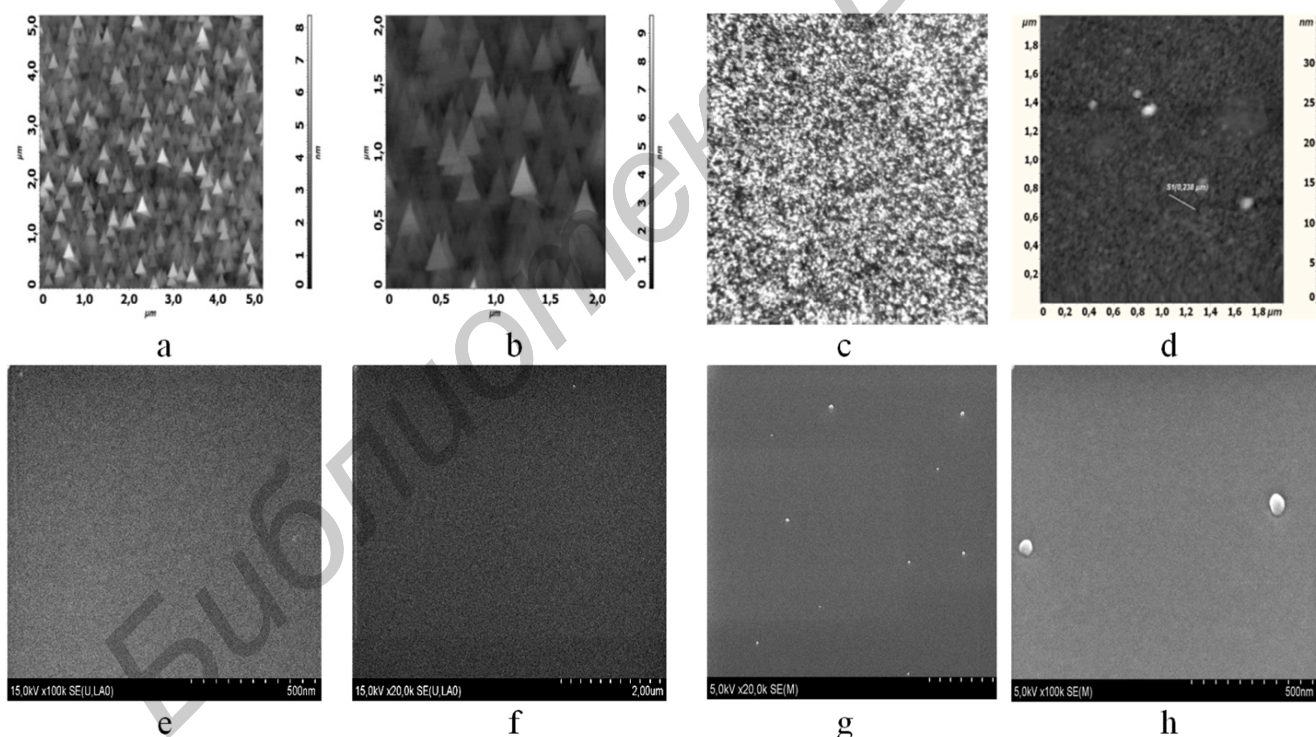

Figure 1. The topography of the surface substrates after various types of heat treatment: (a) – silicon substrates in hydrogen chloride environment, (b) – then further annealed in argon, (c) – in hydrogen environment, (d) – followed by annealing in nitrogen and hydrogen environment, (e) – substrates with ultrathin (4 nm) layer of silicon dioxide, (f) – with ultrathin (4 nm) layer of silicon nitride and (g), (h) substrates with ultrathin (4 nm) layers of dysprosium and yttrium oxide in hydrogen environment. Heat treatment was carried out at a temperature of 900–1000°C.

3. Results and Their Discussion

Surface topographies of the blank substrates, which were subjected to various types of processing, are shown in Figure 1. As can be seen from Figures 1–3, only processing of the blank substrates with superthin layers of silicon oxides and nitride in hydrogen environment allows obtaining practically clean surface without relief. The degree of increase of the surface cleanliness, which is observed in treatment of

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superthin (4 nm) coatings of silicon dioxide on the blank silicon substrate is, apparently, conditioned by reducing reactions between silicon dioxide and hydrogen. Removal of silicon oxide and opening of clean surface of the blank silicon substrate occurs as a result of such reactions. There is a sharp break of the crystal lattice on the clean surface of the silicon single crystal. As a consequence of this, the outer shell of the silicon (Si) surface atoms is not completely filled and, therefore, there are unsaturated bonds. It is assumed that the sequential adsorption of one or two monolayers of germanium leads to the formation of SiGe nanoclusters (Fig. 5–8). Thus, a rapid formation of the first SiGe monolayer, and then a slower formation of the second and third SiGe monolayers is observed in the beginning.

The amount of germanium atoms (monogermane) adsorbed on the surface can be represented as $N= a+b\ lgt$, where t- the time in minutes, a and b – parameters that depend on the temperature; a depends also on the pressure of monosilane (SiH_4) and monogermane (GeH_4), which are used in the formation of SiGe nanoclusters.

The value of parameters a and b is dependent on the cleanliness of the surface, level of doping and crystallographic orientation of the blank substrate, as well as on the type of superthin coating of the blank substrate on the planar side. When SiH_4 and GeH_4 are used as sources of germanium and silicon in the LPCVD process, after five-minute interaction with the blank substrate surface, on the average a=8,5x10^{14} atom/cm^2, b=1,2x10^{14} atom/cm^2. The extent of the surface coverage by SiGe nanoclusters is increased on the freshly prepared surface of the blank substrate in the row-clean substrate with treatment in hydrogen chloride environment and subsequent annealing in pure hydrogen (Fig. 2, 5, 6), the substrate with a superthin layer of dysprosium and yttrium oxides after heat treatment in hydrogen environment (Fig. 3, 7) and the substrate with a superthin layer of silicon oxide and nitride after heat treatment in hydrogen environment (Fig. 4, 8). Such an increase is connected with the high density of dangling bonds on the freshly prepared surface of the blank substrate. Increasing the amount of the connected germanium and silicon at self-assembly of SiGe nanoclusters is a consequence of this. In case of GeH_4 four bonds of Si-H are formed and four Si surface atoms are linked to one Ge atom. Four bonds of Ge-H are formed at SiH_4 molecule adsorption and four Ge surface atoms are linked to one Si atom. The nanoclusters of SiGe composition are formed as a result of such interaction.

Thus, the total number of bonds is the same for all gases and is simply equal to the number of the surface atoms of silicon (Si) at a monolayer occupation by monosilane (SiH_4) and monogermane (GeH_4) of the clean surface of the silicon substrate.

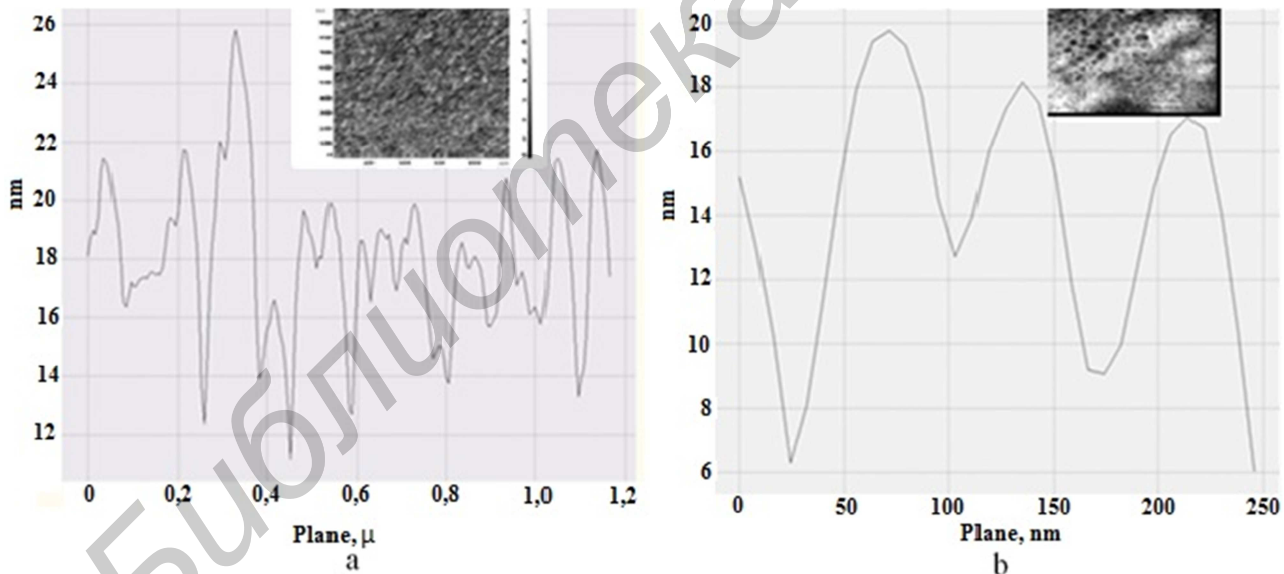

Figure 2. *The relief and profile of surface roughness of the blank substrates treated in hydrogen chloride (a) and hydrogen (b).*

Since the complete desorption of hydrogen occurs at the temperature close to the decomposition temperature of SiH_4, it is obvious that hydrogen is linked to the silicon surface in the same manner as in SiH_4 compound. Surface occupation can be calculated if one knows the number of silicon atoms per 1 cm^2. It is equal to 7,84x10^{14} for the plane (111), 9,58x10^{14} for the plane (110), 6,78x10^{14} for the plane (100). Therefore, to calculate the occupation the number of silicon atoms per 1 cm^2 was assumed to be 8x10^{14}. The divergence of values a and b for different methods of the surface treatment of the blank substrates can be connected with changing the number of crystals with the given surface orientation or the number of unsaturated bonds. In this case, coalescence does not limit the process rate, because both germanium and silicon are supplied from the gas phase. SiH_4 is decomposed into Si and H_2 at a temperature of 450°C, and GeH_4 into Ge and H_2 at 350°C. Hence, to form SiGe compound it is natural that the mixture of SiH_4–GeH_4 contacts the heated surface of the blank silicon substrate to the temperature of ≥ 450°C to form SiGe nanoclusters.

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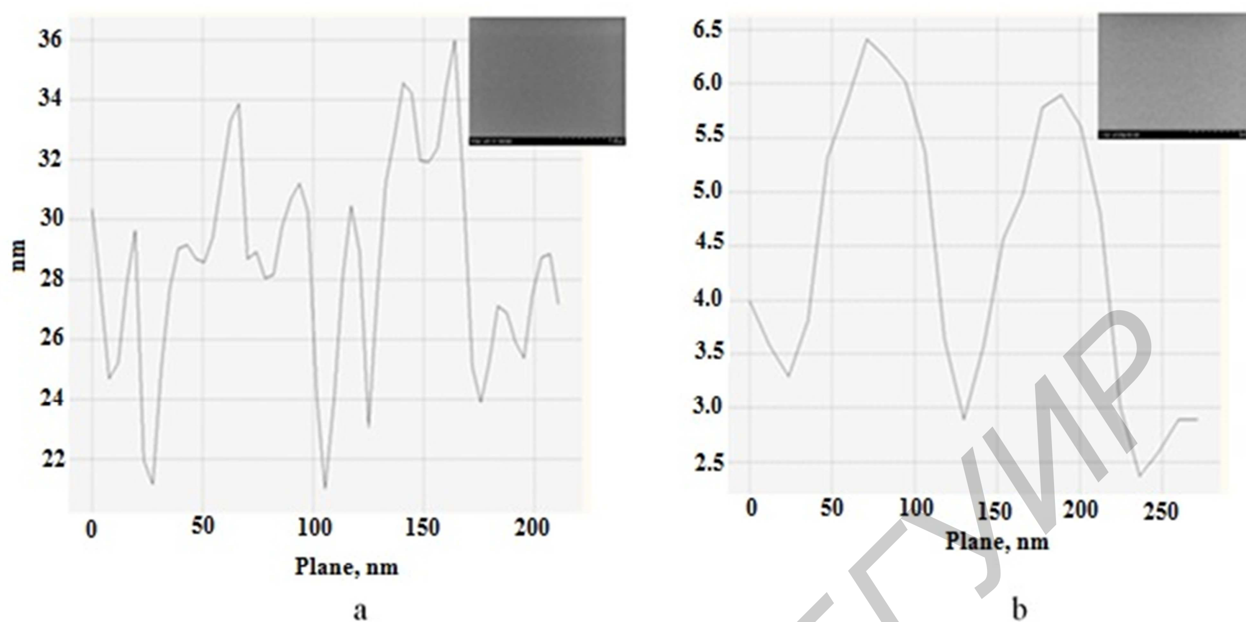

Figure 3. *The relief and profile of surface roughness of the blank substrates with dioxide dysprosium (a) and yttrium dioxide (b) treated in hydrogen.*

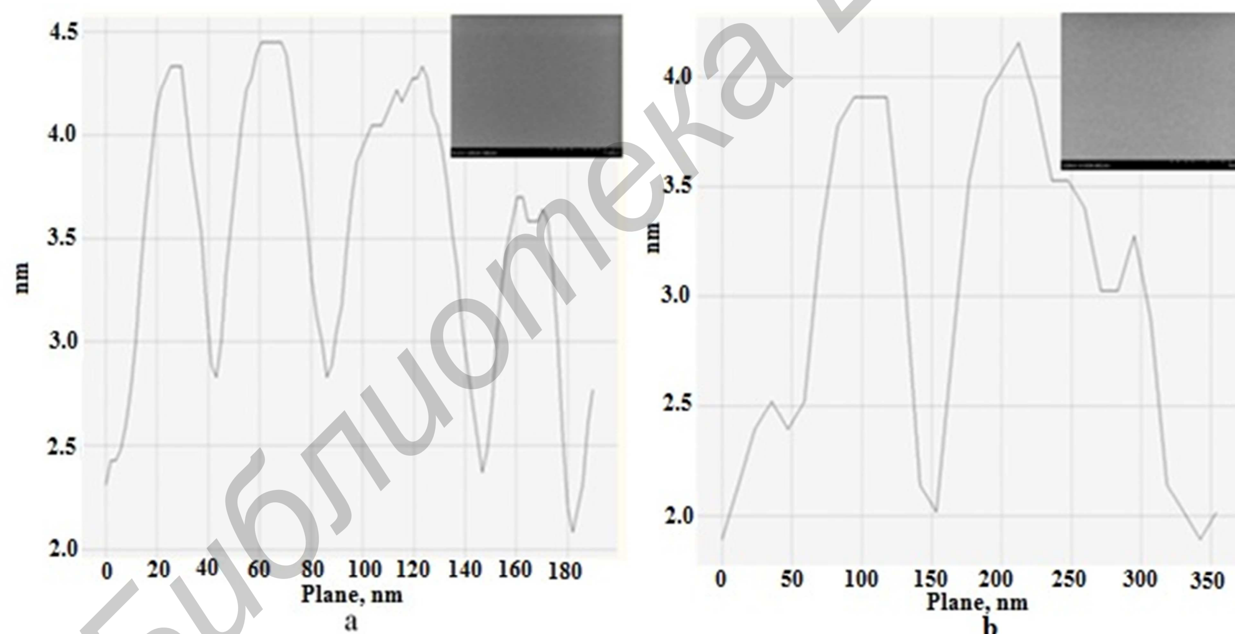

Figure 4. *The relief and profile of surface roughness of the blank substrates with dioxide silicon (a) and silicon nitride (b) treated in hydrogen.*

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The evolution of the surface relief changes after the corresponding treatment and nanoclusters on the surface of the blank substrates with changes of the surface treatment conditions of the blank substrates is shown in Fig. 1–8. It can be seen that the blank substrate surface is mostly covered with arrays of nanoscale, mainly conical formations (Fig. 6–8). It should be noted that the increase in temperature and time of heat influence on the system of freshly prepared nanoclusters leads to their joining in agglomerates which are larger both in height and in lateral dimensions - up to the microsize scale (Fig. 9).

Profiles of the surface roughness of the blank substrates treated in hydrogen chloride (a) and after that in hydrogen (H_2) (b) (Fig. 2), blank substrates with dysprosium dioxide (a) and yttrium dioxide (b) treated in (H_2) (b) (Fig. 3) and blank substrates with silicon dioxide (a) and silicon nitride (b) treated in (H_2) (Figure 4) are shown in Figures 2–4. The process of forming SiGe nanoclusters on the clean surfaces of silicon and germanium consists of the monolayer nucleation stage and the subsequent stage of slow

coalescence of the second layer. Speed of slow coalescence is proportional to the logarithm of time. These processes depend on the method of preparation of the clean surfaces of the blank substrates.

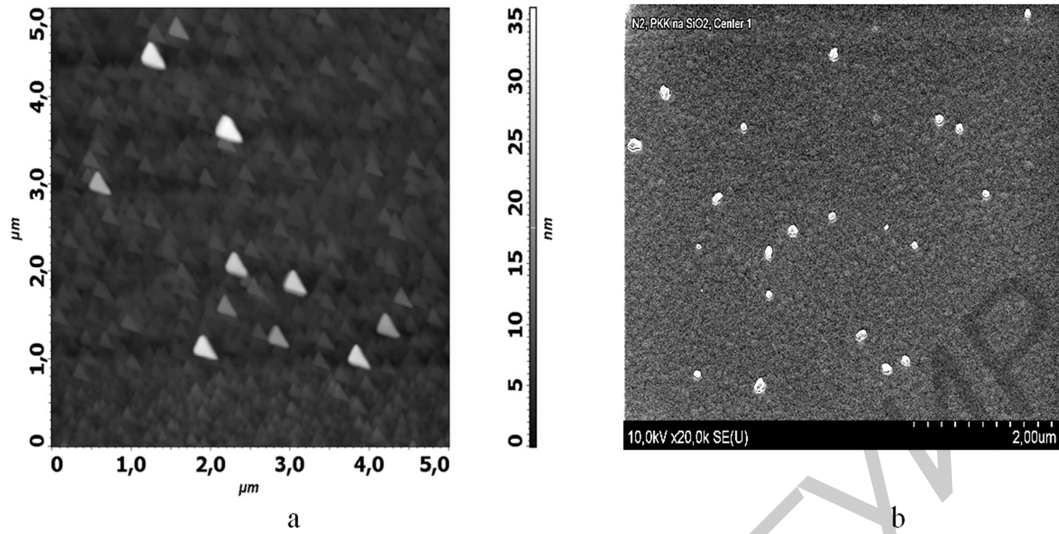


Figure 5. *Nucleation of SiGe nanoclusters on the clean silicon surface after treatment in hydrogen chloride (a) and on silicon dioxide (b) after heat treatment in hydrogen environment with nitrogen.*

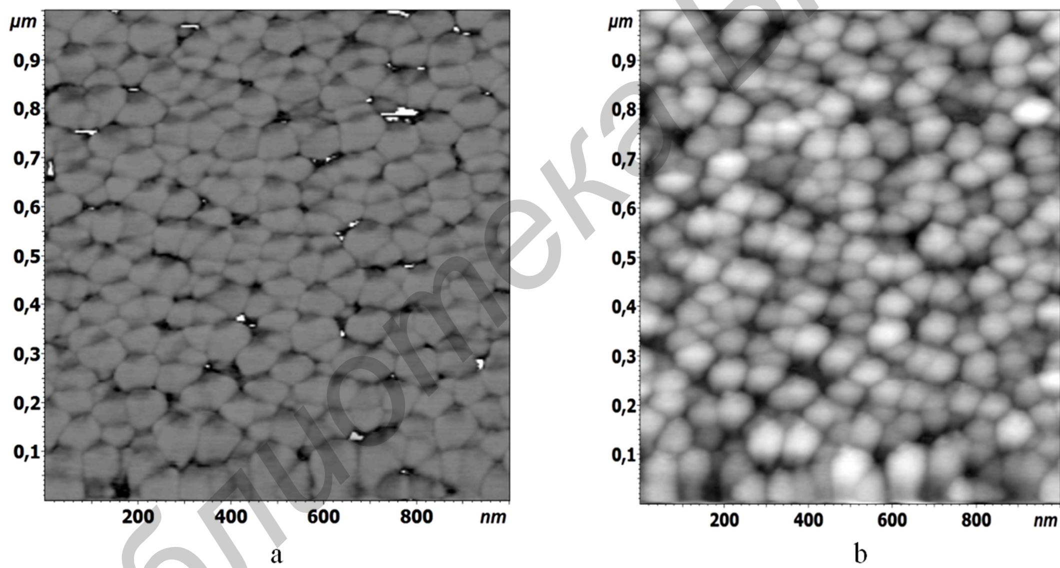
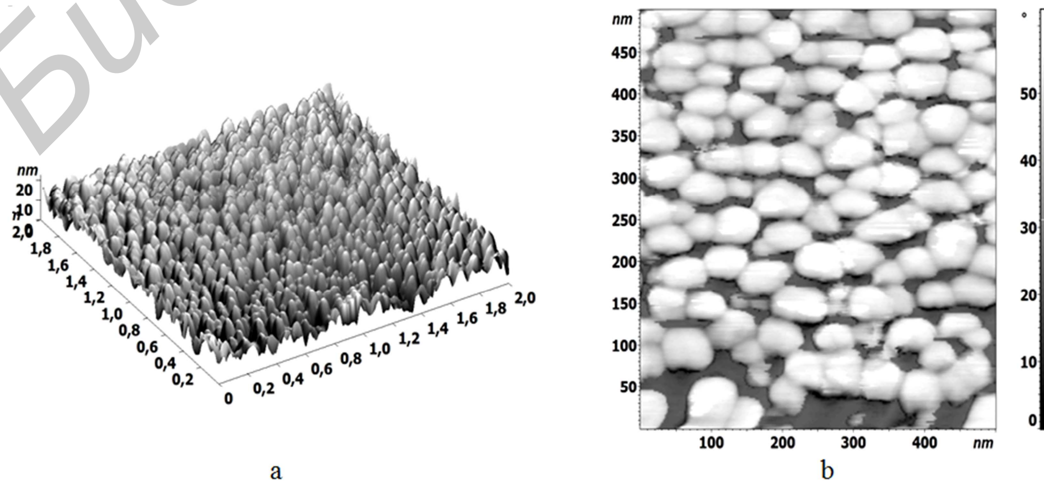


Figure 6. *Formation of SiGe nanoclusters on the blank silicon substrates annealed in argon (a) and hydrogen (b).*



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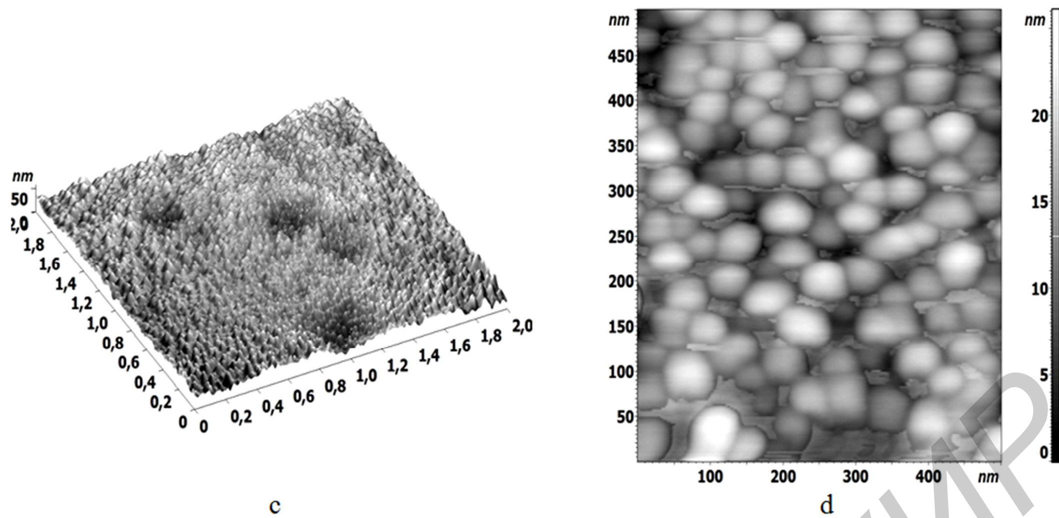


Figure 7. Topography of SiGe nanoclusters on dysprosium dioxide (a, b) and yttrium dioxide (c, d) without heat treatment of the blank substrate (a, c) and with heat treatment in hydrogen environment (b, d).

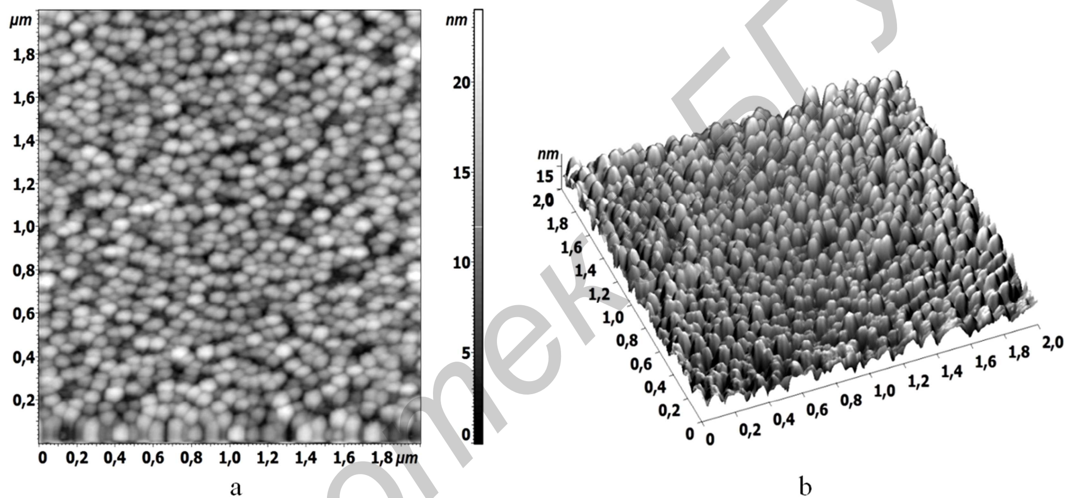
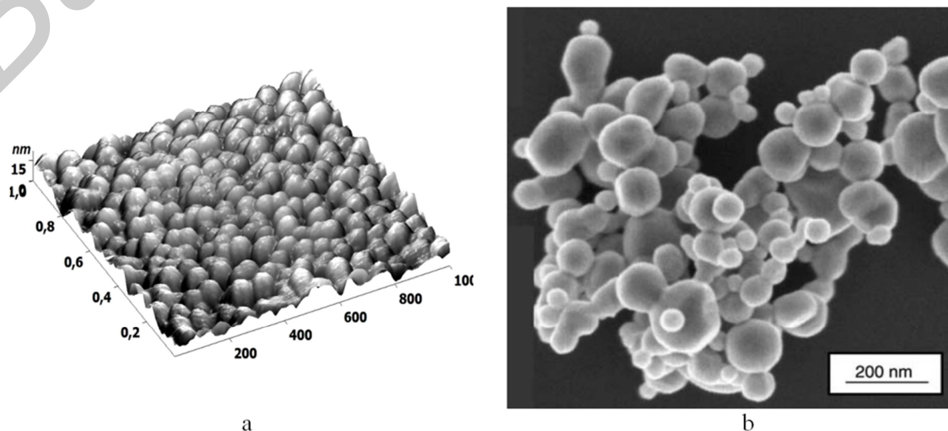


Figure 8. Formation of SiGe nanoclusters on silicon oxide and silicon nitride (b) surfaces annealed in hydrogen (a) on the blank silicon substrates.

Fillings achieved at the stage of nucleation of SiGe nanoclusters using Si and Ge SiH_4 and GeH_4 as sources on clean Si and Ge surfaces, which are characterized by the ratio of ½ SiH_4 and ½ GeH_4 per one surface atom of Si or Ge. It follows that on the clean surfaces of Si and Ge adsorption must occur with dissociation and formation of bonds Ge-H, Si-H and Si-Ge, respectively. Hydrogen is completely desorbed from the surface of Si and Ge at a temperature of 300°C which is substantially the same for all the adsorbed hydrides. This temperature for silicon is equal to 500°C. These temperatures are close to the decomposition temperatures of SiH_4 and GeH_4. There is a clear correlation between the partial pressure of SiH_4 and GeH_4 and the surface density of SiGe nanoclusters.



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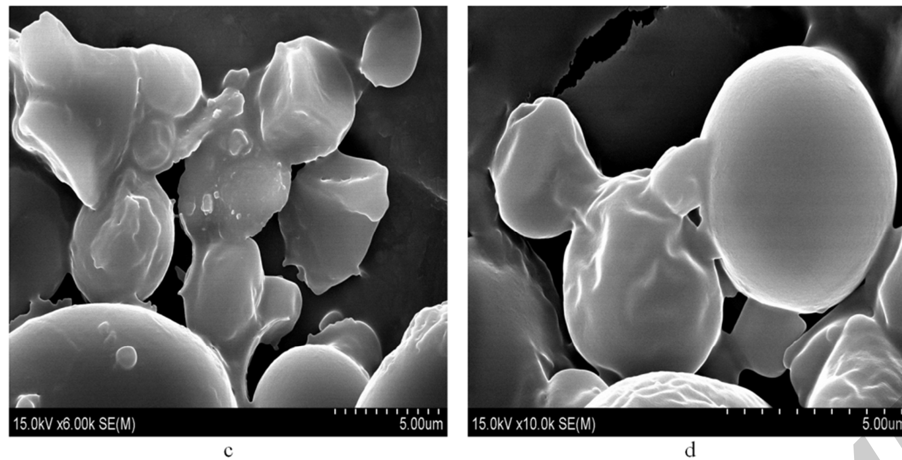


Figure 9. *Transformation of SiGe nanoclusters on the surface of the silicon dioxide film on the blank silicon substrate: a – initial clusters after self-assembly, b – after treatment in hydrogen-nitric mixture at a temperature of 560°C, c – at a temperature of 740°C and d – at a temperature of 900°C. Duration of the heat treatment process – 30 minutes.*

The gases, adsorbed on the clean surface of the blank substrate in large quantities, have also a profound effect on the surface density of the nanoclusters (Fig. 10).

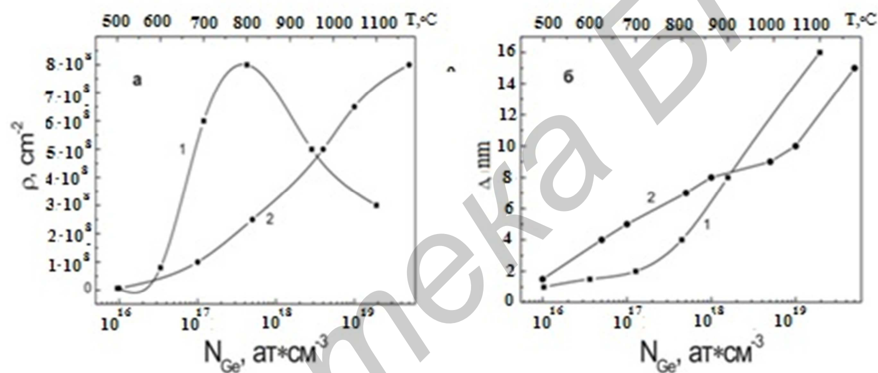
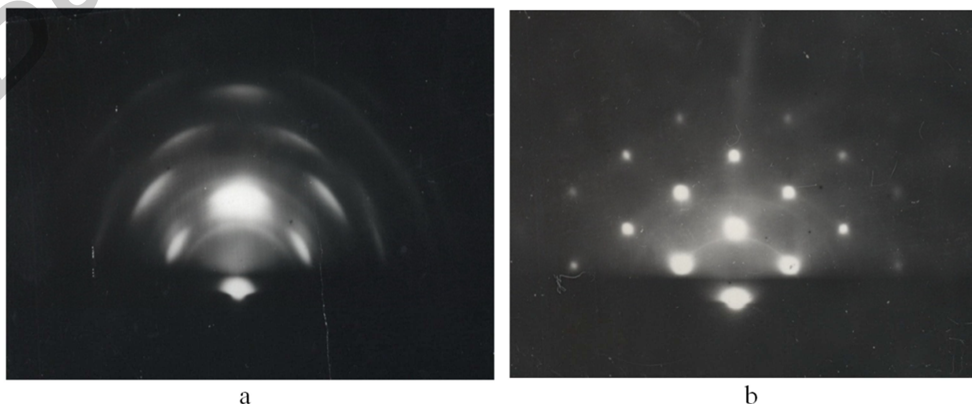


Figure 10. *Dependence of density (a) and height (b) of germanium nanoclusters under heat treatment in wet oxygen environment on temperature (1) and the initial content of germanium in SiGe (2).*

Chemisorption of SiH_4 GeH_4 initially leads to an increase of nanoclusters density and then to its decrease in all cases. Atoms with free bonds, which have acceptor properties, can be formed at first on the crystal surface in the preparation of clean surfaces of the blank substrates. This situation is very energetically unfavorable. Creation of complex structures on the clean surface with the formation of bonds of Ge–Ge or Si–Si type as a result of small displacement of surface atoms is more favorable. The experiments on diffraction of slow electrons really showed the existence of structure – 8 on the plane (111) of Ge (surface Ge (111) – 8x8) and structure – 7 on the plane (111) of clean Si surface (surface of Si (111) – 7x7), which is considered stable for silicon (Figure 11).



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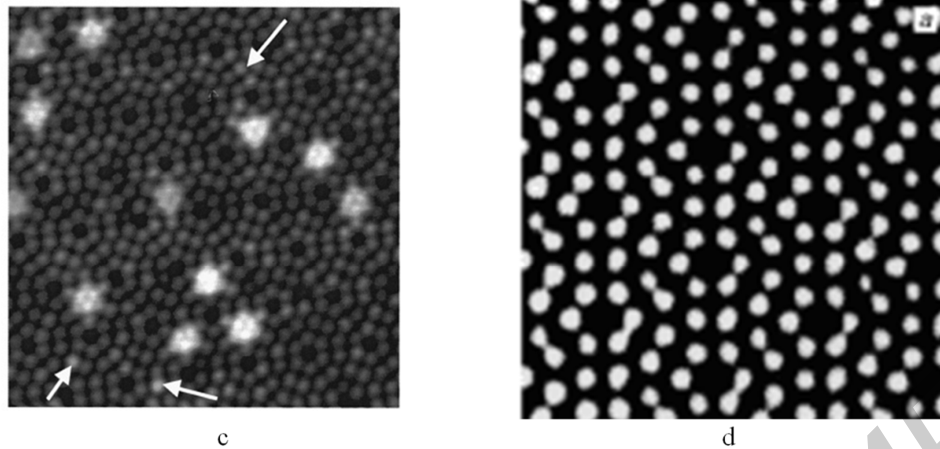

c d

Figure 11. *Diffractions of slow electrons of structure – 8 on the plane (111) of Ge (a, b) and structure –7 on the plane (111) of clean Si surface (c, d).*

Formation of these structures on the clean surfaces can weaken their acceptor properties. However, since the situation on the surface is energetically unfavorable, atoms on the clean surface have high activity.

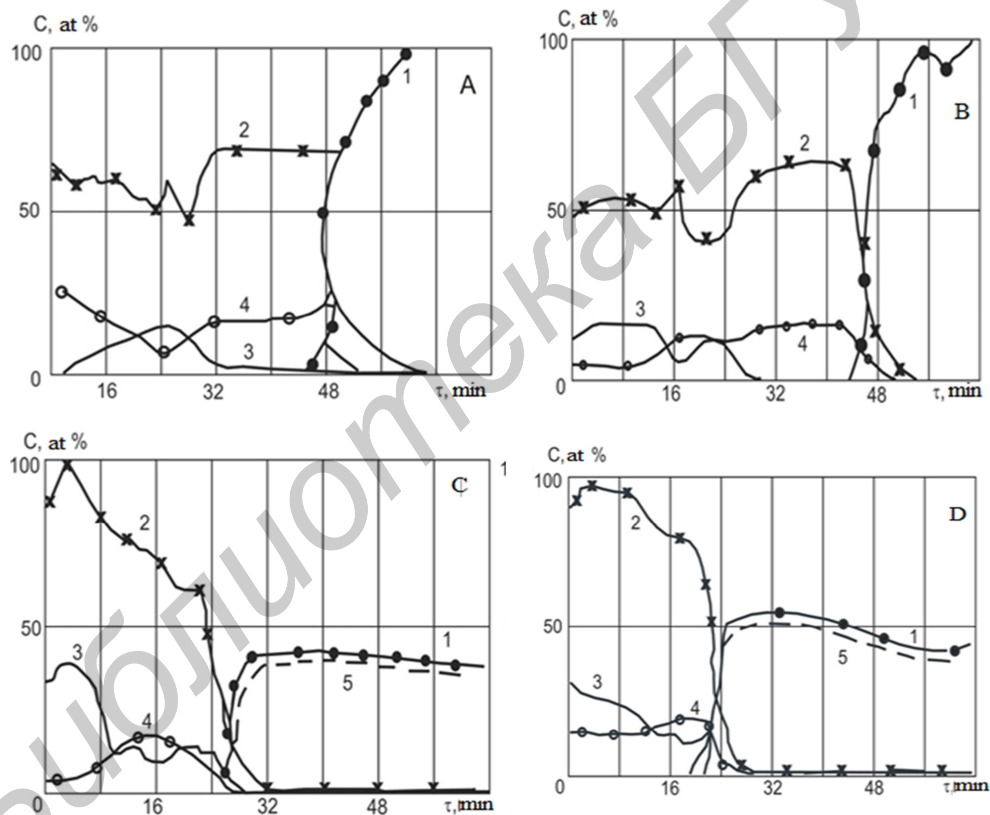

Figure 12. *Profiles of elements distribution in height of SiGe nanoclusters on the layer thickness 50 nm, deposited on silicon dioxide of thickness 4.2 nm (a, b) and silicon nitride of thickness 5.0 nm (c, d) and after its heat treatment at a temperature of 950^oC (b, d). 1 – Si (substrate) 2 – O, 3 – Ge, 4 – Si (oxide), 5 – N.*

Chemisorption of small amounts of SiH_4 and GeH_4 pulls out some atoms from the surface structure. These atoms are in energetically less favorable conditions, characterized by stronger adsorption properties. This leads to the capture of a larger number of Si and Ge atoms. The number of free surface atoms decreases in the adsorption of a large amount of SiH_4 and GeH_4, and the surface density of SiGe nanoclusters decreases with an increase in their size.

Thus, the degree of the surface perfection should be considered as an essential part of the general problem of preparing the clean surface before the process of formation of SiGe nanoclusters and suppression of their transformation from nanosizes to microsizes. It would be ideal to obtain an atomically smooth surface, parallel to a given system of crystallographic facets and not containing the lattice defects or contamination. In practice, it is essential to know the permissible deviations from the ideal case in this process and to determine the feasibility of the required conditions. To

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obtain the desired degree of the surface perfection in massive single-crystal substrates or films it is necessary to use special methods of surface treatment. Heat treatment and cooling rate of the substrates with SiGe nanoclusters can influence their structure, sizes and even composition. The annealed samples of nanoclusters undergo transformation as a result of which the transition from ~~of~~ nanoscale to microscale occurs (Fig. 9).

Root-mean-square value of the surface roughness (R_{rms}), which does not exceed 4 nm, was determined by AFM data. Increase of R_{rms} value from 0.5 to 4 nm with increasing temperature and time of thermal exposure (Fig. 1–4) is noted. Increasing the area of continuous surface sections and R_{rms} with increasing temperature and time of heat exposure in the LPCVD process is a characteristic of self-assembly of SiGe nanoclusters in the deposition of ultrathin films of polinanocryslalline silicon doped with germanium.

This is due to the fact that in the process of heat treatment the atoms, deviating from the ideal position in the crystal lattice, create additional force fields that leads to change in elastic properties of the whole nanocrystal. The nanocrystal surface itself is a defect. Relaxation of near-surface atoms changes stiffness of the crystallite in this area, therefore, the compressibility of nanocrystals depends on the condition of their surface and size of the whole nanocrystal.

The temperature range of heat treatment process above is limited by the process of transformation of nanoclusters into ultrathin films as a result of recrystallization, and below is limited by very low rate of their mobility, that ensures the nanosize scales of clusters.

The analysis of the Auger spectra (Fig. 12) of SiGe nanoclusters on the silicon substrate with SiO_2 and Si_3N_4 showed that a sharp transition between SiGe layers and the substrate is created during oxidation of nanoclusters, almost the same as that between SiO_2 and the silicon substrate. At least up to the temperature of 950°C visible interaction of germanium with other dielectric layers, such as SiO_2, Si_3N_4 and its diffusion is not detected (Fig. 12).

Fundamental research of growth rate of nanoclusters depending on the temperature and time of heat treatment has shown that the mobility of nanoclusters and their joining to agglomerates are multi-step, depending on heat treatment conditions.

The temperature ≥ 900°C should be considered the critical temperature of heat treatment of initial nanoclusters, because transformation of nanoclasters into micro-sized agglomerates begins at this temperature (Fig. 9).

This fact indicates increase in the contribution of coalescence of the clusters to their merging into larger formations.

The temperature of 540°C is the critical temperature of self-assembly of dense array of nanoclusters.

4. Conclusion

The purity of surface of the initial of the substrate is a fundamental factor in the self-organization of SiGe nanoclusters. Temperature rise and increase the duration of the heat treatment in a gaseous environment of starting surface of silicon substrate promotes the formation of SiGe nanoscale clusters and increasing their density. Small displacement of the surface atoms of complex structures on the clean surface with the formation of bonds of Ge–Ge or Si– Si type is favorable for self-assembly of SiGe nanoclusters.

Thus, the degree of the surface perfection should be regarded as an essential part of the general problem of preparing the clean surface before the process of formation of SiGe nanoclusters and the suppression of their transformation from nanosizes to microsizes.

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