Formation of self-assembled quantum dots during GaSbP deposition on AlP surface

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Formation process of the self-assembled GaSbP quantum dots (QDs) on the AlP(100) surface from flows of Sb_4, P_2 and Ga atoms was investigated by reflection high-energy electron diffraction technique. The dependences of the QDs nucleation critical thickness (D_{eff}), the elastic deformations in QDs and the composition of the GaSbP alloy of QDs, on the substrate temperature (T_S) and the pressure ratio in the flows of Sb_4 and P_2 molecules ($P(Sb_4):P(P_2)$) was studied. It was found that in wide ranges of T_S and $P(Sb_4):P(P_2)$ values (380−460°C and 0.07−27 accordingly), unstrained GaSbP/AlP quantum dots are formed.

Keywords: quantum dots, molecular beam epitaxy, solid alloy, plastic strain relaxation.

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1. Introduction

The energy spectrum of charge carriers in low-dimensional semiconductor heterostructures significantly differs from the spectrum of charge carriers in bulk materials because of the effects of quantum confinement [1]. The complete three-dimensional localization of charge carriers in semiconductor self-assembled quantum dots (QDs) allows for a prolonged storage of a charge in them [2,3]. The storage duration is determined by the energy of localization of the charge carrier (E_{loc}) and by the capture cross section in QD [4,5]. This QD property opens up the prospect of using QDs arrays as a floating gate in flash memory elements. The creation of a prototype of a memory element based on InAs/AlGaAs-heterostructures with QDs is discussed in Ref. [6]. The possibility of organizing direct capture of charge carriers in QD when recording a charge on a floating gate is an advantage of using III−V materials for creation of flash memory cells compared to traditional Si/SiO_x structures. This results in the fact that the information recording time is limited only by the time of relaxation of the energy of charge carriers in case of capture in QD amounting to ~ 1 ps [7], which is several orders of magnitude faster than in traditional Si/SiO_x flash cells. Moreover, the absence of the need to create hot charge carriers in case of recording a charge in III−V QD significantly increases the usage life of flash memory cells based on such heterostructures. Therefore, the use of III−V heterostructures with QD opens a prospect of creation of a universal memory combining high performance and the possibility of long-term storage of information.

The first prototypes discussed in Ref. [6] were characterized by a very short charge storage time in QDs, ~ 10 ms. This is caused by the insufficient E_{loc} amounting to ~ 0.8 eV. Therefore, an urgent task was to obtain and study new III−V heterostructures with QD characterized by a larger E_{loc}. Many heterosystems have been studied along this path, such as GaSb/GaAs [8–10], GaSb/AlAs [11–13], InSb/AlAs [14–16], GaAs/GaP [17,18] InAs/GaP [19–21], GaSb/GaP [22–24], InGaSb/GaP [25,26] and GaSb/AlP [27,28]. GaSb/AlP is the most promising heterosystem. The first experimental results of [28] concerning the obtaining of such QD showed that when GaSb is deposited on the surface of AlP, GaSb/AlP QD are formed with completely relaxed mechanical stresses due to the introduction of dislocations. These QD are characterized by E_{loc} of holes at the level of 1.65−1.70 eV, which allows expecting the charge storage time of $\gg 100$ years in QD. However, the question remains open of whether dislocations in QD have a negative effect on the charge storage time. At the same time, our theoretical calculations [27] show that E_{loc} can reach even higher values, up to 2.04 eV in pseudomorphically stressed QD. Additionally, the absence of dislocations in crystallically perfect QD will eliminate the factor of the impact of dislocations on the charge storage time in QD. Therefore, the priority task is the formation of heterostructures with pseudomorphically stressed QD.

The GaSb/AlP heterosystem is characterized by a high value of the lattice parameter mismatch of about 11.7% [29]. As we have suggested, the reduction of the mismatch between the lattice parameters of the deposited material and the AlP matrix can prevent plastic relaxation of deformations and the introduction of dislocations. The magnitude of the mismatch between the lattice parameters of the QD material and the AlP matrix can be reduced by forming QD from a solid GaSbP solution. This is supported by the experimental results obtained for the GaSb/GaP heterosystem, which is related to the studied GaSb/AlP

heterosystem. It is possible to form pseudomorphically stressed GaSbP/GaP QD characterized by high crystalline perfection as shown in [23].

The addition of P atoms to the composition of GaSb/AlP QD inevitably results in a decrease of E_{loc}. However, calculations of the energy spectrum of pseudomorphically stressed GaSbP/AlP QD [27] showed that the value of E_{loc} remains at the level of 1.5 eV when QD is formed from $GaSb_{0.65}P_{0.35}$, which is still sufficient to ensure a prolonged storage of the charge in QD at room temperature for more than 10 years [30]. At the same time, the mismatch of the parameters of lattice $GaSb_{0.65}P_{0.35}$ and AlP is already $\sim 6.5\%$, which is comparable to the mismatch value for the well-studied InAs/GaAs heterosystem, for which the possibility of forming pseudomorphically stressed QD is shown [31]. Therefore, there is some margin for diluting the QD material with P atoms without losing the practical significance of QD in terms of application for creation of non-volatile memory elements.

GaSbP/AlP QD can be formed both in case of the deposition of Ga atoms and Sb_4 molecules due to lateral and/or bulk diffusion of P atoms in QD, and in case of the deposition of Ga atoms and Sb_4 and P_2 molecules due to the incorporation of precipitated phosphorus into the QD composition. The effect of substrate temperature (T_S) and the pressure ratio in the molecular streams Sb_4 and P_2 ($P(Sb_4) : P(P_2)$) on the structural properties of the resulting QD have been studied for this reason. The structures with QD were grown by the method of molecular beam epitaxy (MBE). The basic information about the processes occurring on the AlP surface was obtained *in situ* by the reflection high-energy electron diffraction (RHEED) method. Unexpected results were obtained showing that unstressed QD of GaSbP/AlP are formed regardless of T_S and $P(Sb_4) : P(P_2)$, which vary widely.

2. Experiment

2.1. Growth of heterostructures

Heterostructures with GaSbP/AlP QD were grown using the MBE method on artificial GaP/GaAs substrates of orientation (100). The GaAs (100) substrates used for this purpose are characterized by a deviation from the plane of (100) $\leq 0.5^\circ$. The density of threading dislocations in the near-surface layers of the GaP/GaAs artificial substrate was $\sim 10^8\,\text{cm}^{-2}$ [32]. The details of the procedure for growing III–V (GaP, GaAs and GaSb) layers on inconsistent substrates by the MBE method are described in Ref. [32–36].

All heterostructures were grown using the MBE „Shtat“ facility in the ISP SB RAS. The facility is provided with crucible sources of flows of Al, Ga atoms and Sb_4 molecules with dressing valves, as well as two-zone valve sources of P_2 molecules. T_S was monitored according to the readings of the control thermocouple of the substrate heater, which was calibrated using the RHEED method for the transition temperatures of surface superstructures on GaP (100) in the absence of a flow of P_2 molecules. The pressure in the flows of P_2, Sb_4 molecules, as well as Al and Ga atoms was determined by the values of the ion current of an ionization gauge transducer introduced during measurements into direct flows on the substrate position. The ratio of $P(Sb_4) : P(P_2)$ was reduced during QDs formation by increasing the flow of P_2 molecules with a constant value of the flow of Sb_4 molecules. At the same time, the ratio of $P(Sb_4) : P(Ga)$ corresponded to the stoichiometric GaSb growth conditions for the studied range of substrate temperature values. The ratio of $P(Sb_4) : P(P_2)$ was reduced below 0.3 by reducing the flow of Sb_4 molecules upon reaching the limit values of the flow of P_2 molecules.

300 nm thick GaP buffer layers were grown at $T_S = 600^\circ$C. Then T_S was reduced to 450°C and 50 nm thick AlP layers were grown in accordance with the results of studies of the epitaxial growth of AlP layers [37,38]. The rate of deposition of Ga and Al atoms during the growth of the GaP and AlP bulk layers corresponded to the growth rate of 1 monolayer per second (ML/s). The T_S corresponding to the selected conditions of QDs nucleation was set after growing of AlP. The substrate with the AlP layer was cooled in a flow of P_2 molecules.A solid solution of GaSbP was deposited for the formation of QD at various values of $P(Sb_4) : P(P_2)$ and T_S. The rate of Ga deposition during QD formation did not vary and corresponded to the rate of GaP(100) homoepitaxial growth of 0.23 monolayer per second (ML/s). First, the flow of Sb_4 molecules was switched on, and then, the flow of Ga atoms was switched on after 3 s. The RHEED pattern was recorded during the deposition of GaSbP, and the time of transition from a two-dimensional growth regime to a three-dimensional regime was recorded. The time was counted from the moment the flow of Ga atoms was switched on. The flows of Ga atoms and Sb_4 molecules were switched off after the formation of QDs. T_S was increased to 650°C for removal of the formed QDs. Next, 5 nm GaP was deposited, T_S was reduced to 450°C and 5 nm AlP was deposited, after which the QD formation was carried out under other conditions.

2.2. Analysis of RHEED images

In situ data on the time of transition of the GaSbP layer growth regime from two-dimensional to three-dimensional were obtained using the RHEED method. This allowed estimating the amount of substance required to initiate the formation of QDs. Subsequent analysis of the RHEED patterns provided information about the degree of relaxation of QDs and the lattice constant of their material.

A typical RHEED image obtained in azimuth [011] after QD formation is shown in Figure 1. The wide white arrow in Figure 1 indicates the position of the reflex (00) of the RHEED pattern. Short gray arrows indicate the positions of linear reflexes of two-dimensional diffraction from the face (100) of a crystal with a lattice constant

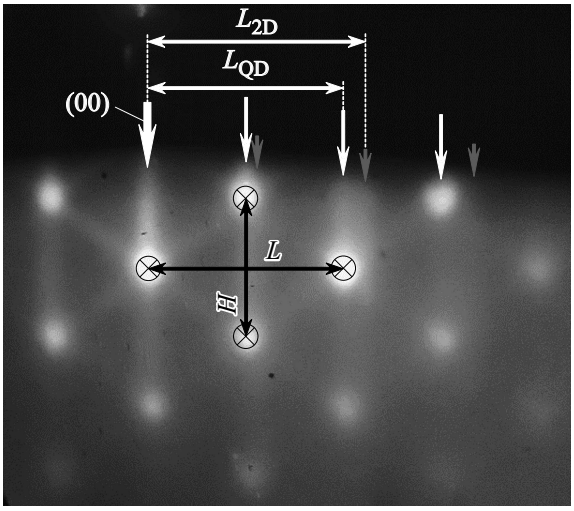

Figure 1. A typical RHEED pattern obtained during the formation of GaSbP/AlP QD (see explanations in text).

corresponding to AlP. Long white arrows indicate the positions of linear and associated point diffraction reflexes corresponding to a material with a lattice constant significantly greater than that of AlP. Point reflexes are the result of electron transmission diffraction on three-dimensional objects. It should be noted that the lateral surfaces of three-dimensional objects along the direction [011] are covered with facets {111}, as evidenced by inclined linear reflexes.

The analysis of L/H and L_{2D}/L_{QD} ratios (see Figure 1) allows estimating the degree of relaxation and the value of the lattice constant of epitaxial layers. In the considered geometry of obtaining the RHEED and observing its pattern, the distance is $L/2 \propto 2^{1/2}/a_{\parallel}$, and $H/2 \propto 1/a_{\perp}$, where $a_{\parallel}$ is the lattice constant of the epitaxial layer biaxially compressed in the directions [010] and [001], $a_{\perp}$ — its lattice constant in the direction [100]. For fully relaxed QD, when $a_{\parallel} = a_{\perp} = a$, the ratio $L/H = 2^{1/2}$, while for pseudomorphically stressed QD, the ratio L/H will be determined in accordance with the formulas of the model-solid theory [39]: $a_{\parallel} = a_{\mathrm{AlP}}$, $a_{\perp} = a_{\mathrm{GaSbP}}(1-2fC_{12}/C_{11})$, where $f = a_{\mathrm{AlP}}/a_{\mathrm{GaSbP}}-1$ the mismatch of the parameters of GaSbP (a_{GaSbP}) and AlP (a_{AlP}) lattice, C_{12} and C_{11} — the elastic constants of GaSbP.

L/H is close to $2^{1/2}$ in the case shown in Figure 1. This indicates that the crystal lattice of three-dimensional objects does not have significant biaxial deformations, i. e. deformations in QDs were almost completely relaxed. The ratio $L_{2D}/L_{QD} \propto a_{\parallel QD}/a_{\parallel AlP}$ and in the example under consideration is 1.12. This allows concluding that the QD lattice constant is close to the lattice constant of GaSb, i. e., the proportion of phosphorus in the epitaxial layer is small. Meanwhile, our data do not give us direct information about the incorporation of Al atoms into the QD composition, since the lattice constants of AlSb and GaSb, as well as AlP and GaP, are close [29]. However, it can be assumed based on the results of studies of AlP/GaP quantum wells, short-period superlattices and Bragg mirrors [38,40–42], that the diffusion of Al into the GaP layers is largely suppressed. Further, we will consider the studied QD under the assumption of suppression of incorporation of Al atoms into them.

3. Results

It should be noted that the critical quantity of substance required to start the process of QD formation (D_{eff}) is expressed in monolayers in this paper, and the rate of deposition of gallium atoms on the surface is represented by the rate of homoepitaxial growth of GaP(100) in ML/s. It should be borne in mind that 1 pseudomorphically stressed epitaxial layer (pseudomorphic monolayer — ML) of GaSb/GaP(100) is equivalent to 1.25 homoepitaxial layer of GaSb(100) in terms of the number of atoms per unit surface.

At the first stage, we performed the deposition of Ga atoms and Sb_4 molecules on AlP at various T_S (380−460°C). Figure 2 shows the results of the analysis of the RHEED patterns with a variation of T_S. As can be seen from Figure 2, *a* that shows the dependencies $D_{\mathrm{eff}}(T_S)$ and $f(T_S)$, it is enough to deposit 1.6−2.0 ML of GaSb for QDs formation regardless of T_S. The dependence of the ratio $L/H(T_S)$ is shown in Figure 2, *b*. It can be seen that regardless of T_S, this ratio is close to 1.414. This indicates that QDs are formed almost completely relaxed. In this case, the mismatch of the lattice parameters of the QD material and the matrix is 10−12% (Figure 2, *a*). This allows stating that QDs consists of GaSbP with a low content of P of $\leq 15\%$. It should be noted that in the case of the formation of pseudomorphically stressed QD, characterized by the observed mismatch of lattice parameters at the level of 10−12%, the ratio L/H would accept the value of ~ 1.77 (marked with a dotted line in the upper part of Figure 2, *b*), which is markedly different from what was observed in the experiment. This additionally indicates the reliability of determination of almost complete relaxation of deformations in QD, based on the analysis of RHEED patterns.

At the second stage of the work, the ratio of flows of Sb_4 and P_2 molecules was varied at a constant value of T_S. Figure 3 shows the dependencies of D_{eff}, f and L/H on $P(Sb_4) : P(P_2)$, measured at $T_S = 380°C$.

Figure 3, *a* shows that the dependence $D_{\mathrm{eff}}\big(P(Sb_4) : P(P_2)\big)$ can be conditionally divided into 2 sections. It grows slightly from 1.7 to 2.25 ML with a decrease of $P(Sb_4) : P(P_2)$ from 27 to $0.6\,D_{\mathrm{eff}}$, while the critical thickness begins to grow sharply up to 27.5 ML with a decrease of $P(Sb_4) : P(P_2)$ below 0.6. In addition, it can be seen from Figure 3, *b* that regardless of $P(Sb_4) : P(P_2)$, the ratio L/H has a value close to 1.414, which indicates that QDs is formed largely relaxed. Figure 3, *a* also shows the experimental dependence $f\big(P(Sb_4) : P(P_2)\big)$. The figure also shows a smooth decrease of the value of f from 11−12 to 4−5% when $P(Sb_4) : P(P_2)$ decreases.

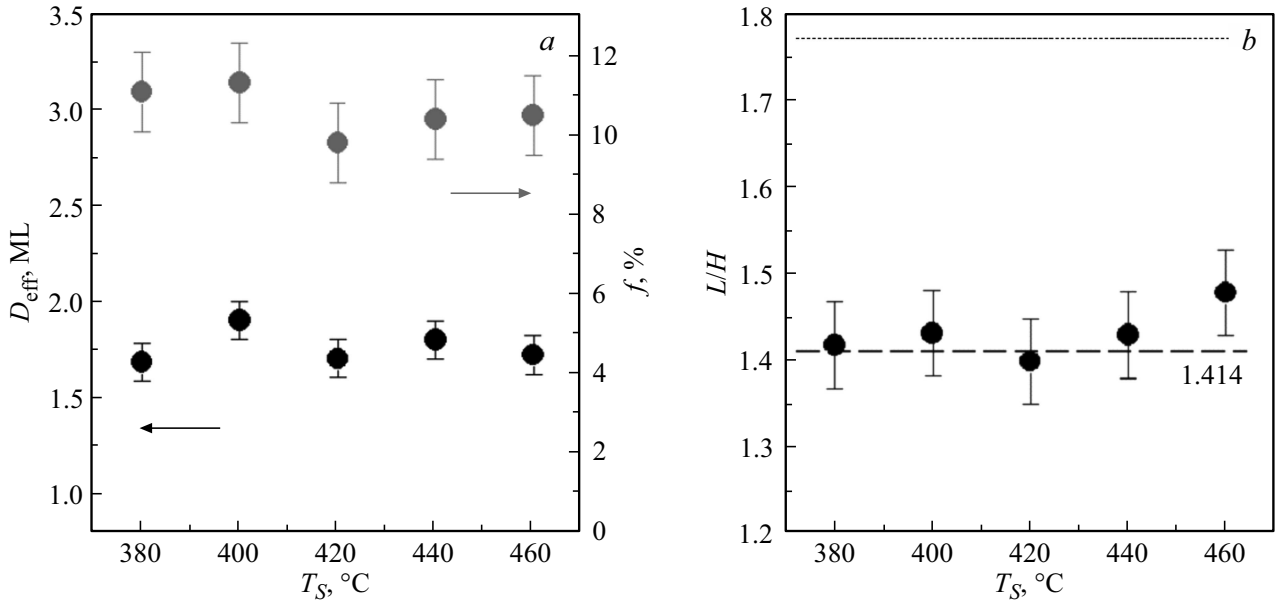

Figure 2. The obtained dependences $D_{\text{eff}}(T_S)$ and $f(T_S)$ (a) and $L/H(T_S)$ (b) in case of the deposition of Ga atoms and Sb_4 molecules at different T_S.

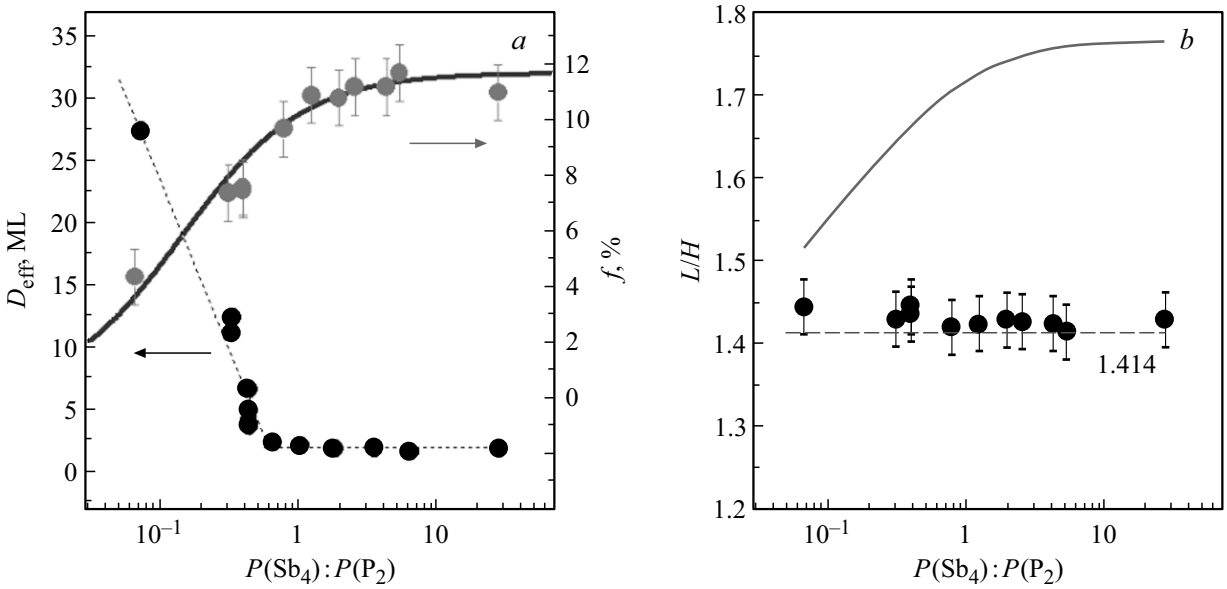

Figure 3. Obtained dependencies $D_{\text{eff}}\big(P(Sb_4):P(P_2)\big)$ and $f\big(P(Sb_4):P(P_2)\big)$ (a) and $L/H\big(P(Sb_4):P(P_2)\big)$ (b) in case of deposition of Ga atoms and Sb_4 and P_2 molecules at $T_S = 380^\circ$C. Solid lines show the theoretical dependencies (a) $f\big(P(Sb_4):P(P_2)\big)$, calculated using formula (3), and (b) $L/H\big(P(Sb_4):P(P_2)\big)$, calculated for pseudomorphically stressed QD according to the formula (4).

We calculated the composition of the growing layer of solid solution of $GaSb_xP_{1-x}$ to describe the obtained dependence:

$$x = \frac{1}{1 + \frac{2J_{P_2}S_{P_2}}{4J_{Sb_4}S_{Sb_4}}}, \qquad (1)$$

where J_i and S_i — the density of flow and the incorporation coefficient of Sb_4 and P_2 molecules, respectively. The pressure measured by an ionization vacuum meter in a molecular flow is directly proportional to its density. Therefore, taking into account the coefficients of sensitivity of the ionization vacuum meter to Sb_4 (η_{Sb_4}) and P_2 (η_{P_2}) molecules, it is possible to write:

$$x = \frac{1}{1 + \frac{2P_{P_2}\eta_{Sb_4}S_{P_2}}{4P_{Sb_4}\eta_{P_2}S_{Sb_4}}}. \qquad (2)$$

In our case, $\eta_{Sb_4}/\eta_{P_2} \approx 3$. Considering the direct linear relationship between the magnitude of the mismatch of the lattice parameters of the solid solution of $GaSb_xP_{1-x}$

and AlP and the composition of the solid solution x can be written as follows:

$$f = 11.7\% \times \frac{1}{1 + \frac{2P_{P_2}\eta_{Sb_4}S_{P_2}}{4P_{Sb_4}\eta_{P_2}S_{Sb_4}}}. \quad (3)$$

In accordance with the results of the work [43] devoted to studies of the formation of lattice-matched GaSbP/InP layers, the ratio of coefficients of incorporation of S_{P_2}/S_{Sb_4} is ~ 0.0125. In our case, the maximum matching of the calculated dependence obtained by the formula (3) with the experimental data of the RHEED was achieved at $S_{P_2}/S_{Sb_4} = 0.05$.

The solid curve in Figure 3, *b* shows the calculated dependence of the ratio $L/H\big(P(Sb_4) : P(P_2)\big)$ for the case of pseudomorphically stressed $GaSb_xP_{1-x}$/AlP QD. This dependence can be represented in the following form in accordance with the continuum theory formulas [39]

$$\frac{L}{H} = \sqrt{2}\,\frac{\left(1 - 2f\,\frac{C_{12}}{C_{11}}\right)}{1 + f}, \quad (4)$$

where f — the value of the mismatch of the lattice parameters of the solid solution and the matrix, calculated by the formula (3), C_{11} and C_{12} — elastic constants of $GaSb_xP_{1-x}$. The figure shows that this curve lies noticeably upper the experimentally observed points for all values of $P(Sb_4) : P(P_2)$. This additionally indicates the reliability of determination of almost complete relaxation of deformations in QD, based on the analysis of RHEED patterns.

4. Discussion

As our data show, QD are formed in case of deposition of the quantity of the substance that significantly exceeds 1 ML. In addition, D_{eff} increases with an increase of the proportion of P in the composition of QDs, which means a decrease of f. This suggests that QD are formed by the Stranski–Krastanov mechanism [44] and the relaxation of elastic deformations is the main driving force behind the formation of QDs. An unexpected result is that even a significant decrease of the proportion of Sb in the flow of deposited substances and, as a result, a decrease of the proportion of Sb in the composition of QD, as well as a wide variation of T_S, did not allow us obtaining pseudomorphically stressed QD. Even when QD is formed from a solid solution of GaSbP, the lattice parameter of which is 4% greater than AlP, plastic relaxation of deformations in QD occurs immediately after their formation.

Let's discuss the possible causes of the observed phenomena. First of all, we will pay attention to the results concerning the variation of T_S. We demonstrated the effect of change of the composition of QD depending on T_S for the GaSbP/GaP heterosystem, which is very close to the GaSbP/AlP heterosystem studied in this article [23]. The increase of T_S from 420 to 470°C results in an increase of the content of P in the composition of GaSbP/GaP QD from 60 to 80%. At the same time, we observe the formation of QD from GaSbP with a low content of P, $\leq 15\%$ in the studied GaSbP/AlP structures, when GaSb is deposited on AlP, which at the same time does not show a significant temperature dependence in a wide range of 380–460°C. This indicates that the final efficiency of incorporation of P atoms into a crystal from P_2 molecules in these conditions is significantly less than the final efficiency of incorporation of Sb atoms from Sb_4 molecules. The reason for this may be both the significantly more developed relief of the AlP surface compared to GaP [28], as well as differences of the superstructures and properties of the AlP(100) and GaP(100) surfaces. The initial superstructure can have a significant effect on the formation process, composition and conditions of plastic relaxation of the transition layer from phosphide to antimonide. Moreover, the transition layer is comparable to one monolayer in terms of the quantity of substance.

As our experimental results show, the mechanical stresses in the QDs completely relax regardless of the value of the lattice parameter of the solid solution of GaSbP QDs. This indicates that the magnitude of the mismatch between the lattice parameters of GaSbP and AlP does not play a decisive role in the transition of QD growth to the mode of plastic relaxation of deformations. We believe that the introduction of dislocations is affected not only by the factor of elastic energy stored in a stressed QD, but also by such factors as the energy of the QD/AlP hetero-interface and the free surface of the QD, which in turn depend on the chemical composition of the QD and the matrix, as well as on the microstructure of the hetero-interface (density of atomic steps and step breaks). In addition, the presence of threading dislocations in the AlP layers on which QD are formed may play a role. The density of such dislocations is $\sim 10^8\,\text{cm}^{-2}$, which can affect both the formation of QDs and the plastic relaxation of deformations in them.

Now let's discuss the nature of the obtained dependencies $D_{\text{eff}}\big(P(Sb_4) : P(P_2)\big)$ and $f\big(P(Sb_4) : P(P_2)\big)$. The dependence of the composition of the QDs solid solution, and hence the magnitude of the mismatch of the QD/matrix lattice parameters on $P(Sb_4) : P(P_2)$, is well described by the formulas (2) and (3) as shown above. This dependence is smooth, without any special points at which the nature of dependence would change. At the same time, the growth of D_{eff} sharply accelerates with a decrease of $P(Sb_4) : P(P_2)$ below 0.6, which suggests a change of the mechanism of formation of QD. However, a mismatch of the lattice parameters of the QD material and the matrix is the main physical parameter affecting the formation of QD by the Stranski–Krastanov mechanism [44]. Therefore, we constructed the dependence $D_{\text{eff}}(f)$ using the experimental dependencies $D_{\text{eff}}\big(P(Sb_4) : P(P_2)\big)$ and $f\big(P(Sb_4) : P(P_2)\big)$, which is shown in Figure 4. As can be seen from the figure, the dependence $D_{\text{eff}}(f)$ does not have any highlighted points and is well described by a single function $D_{\text{eff}} \sim f^{-3}$.

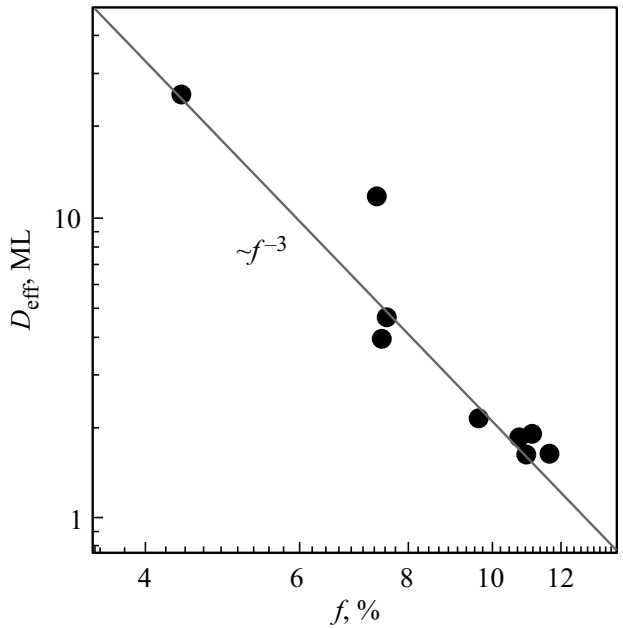

Figure 4. Dependence of the QDs formation critical thickness D_{eff} on mismatch of the lattice parameters of the solid solution of $GaSb_xP_{1-x}$ which compose the QD, and AlP f. The solid line corresponds to the dependency $D_{\text{eff}} \sim f^{-3}$.

Therefore, there is no reason to assume a change of the mechanism of formation of QD, based on our RHEED data.

5. Conclusion

In situ experiments were carried out to study the processes of formation of self-assembled QDs during deposition of Ga atoms and Sb_4 and P_2 molecules on the AlP surface by the MBE method. It was shown that QDs is formed from a solid solution of GaSbP. The data obtained suggest the formation of QD by the Stranski–Krastanov mechanism. The best match between the experimental and calculated dependences of the composition of the formed GaSbP/AlP QD on $P(Sb_4) : P(P_2)$ is achieved with the ratio of incorporation coefficients $S_{P_2}/S_{Sb_4} = 0.05$. It was found that, despite the incorporation of P atoms into the composition of QDs, which results in a decrease of the mismatch of the lattice parameters of GaSbP and AlP up to 4%, mechanical stresses in QDs almost completely relax.

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Conflict of interest

The authors declare that they have no conflict of interest.

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