Research Article

Investigation of the Crystallographic Perfection and Photoluminescence Spectrum of the Epitaxial Films of \((\text{Si}_2)_{1-x}(\text{GaP})_x\) \((0 \leq x \leq 1)\) Solid Solution, Grown on Si and GaP Substrates with the Crystallographic Orientation \((111)\)

A. S. Saidov, D. V. Saparov, Sh. N. Usmonov, A. Kutlimratov, J. M. Abdiev, M. Kalanov, A. Sh. Razzakov, and A. M. Akhmedov

1Physical-Technical Institute, Scientific Association "Physics-Sun", Uzbek Academy of Sciences, Chingiz Aytmatov Street 2B, Tashkent 100084, Uzbekistan
2Chirchik State Pedagogical Institute, Chirchik 111700, Uzbekistan
3Institute of Nuclear Physics, Uzbek Academy of Sciences, Tashkent, Uzbekistan
4Urgench State University, 14, Kh. Alimjan Str, Urgench 220100, Uzbekistan
5Tashkent Institute of Irrigation and Agriculture Mechanization Engineers, Kory Niyoziy Str. 39, Tashkent 100000, Uzbekistan

Correspondence should be addressed to D. V. Saparov; dada@uzsci.net

Received 7 June 2021; Revised 13 September 2021; Accepted 17 September 2021; Published 18 October 2021

Academic Editor: Sefer Bora Lisesivdin

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Epitaxial layers of the solid solution of molecular substitution \((\text{Si}_2)_{1-x}(\text{GaP})_x\) \((0 \leq x \leq 1)\) on Si \((111)\) and GaP \((111)\) substrates are grown by liquid-phase epitaxy from an Sn solution-melt. Such graded-gap solid solutions allow the integration of well-established silicon technology with the advantages of III-V semiconductor compounds. The structural features, the distribution of the atoms of the components over the thickness of the epitaxial layer, the photoluminescence spectrum of the \((\text{Si}_2)_{1-x}(\text{GaP})_x\) \((0 \leq x \leq 1)\) solid solution, and the electroluminescence of the structure n-GaP-n+-(\text{Si}_2)x(\text{GaP})_{1-x}\) \((0 \leq x \leq 0.01)\) have been investigated. It is shown that the layers of the solid solution have a perfect single-crystal structure with the crystallographic orientation \((111)\), with the size of subcrystallites \(\sim 39 \pm 1\) nm. The epitaxial layer \((\text{Si}_2)_{1-x}(\text{GaP})_x\) \((0 \leq x \leq 1)\) is a graded-gap layer with a smoothly and monotonically varying composition from silicon to 100% GaP. The energy levels of atoms of Si\(_2\) molecules which are located 1.47 eV below the bottom of the conduction band of gallium phosphide are revealed. Red emission of n-GaP-n+-(\text{Si}_2)x(\text{GaP})_{1-x}\) \((0 \leq x \leq 0.01)\) structure which is caused by electron transitions with participation of energy levels of Si\(_2\) atoms is detected.

1. Introduction

In recent years, “silicon photonics” has very developed to combine the advantages of optical data processing with Si microelectronic technology. This merger is becoming increasingly important for future high-speed technologies [1]. Due to the indirect energy band of Si, the main focus is on the monolithic growth of direct-band III-V binary compounds on an Si substrate [2]. On the other hand, such technology is also necessary to avoid expensive III-V substrates with a small area, while allowing the use of new technologies on Si, such as integrated photonics [3, 4] and highly efficient and inexpensive multijunction solar cells [5, 6]. However, high-quality growth of III-V compounds on Si is difficult due to the mismatch in the lattice constant, thermal expansion coefficient, and polarity. A promising material from the point of view of integrating Si technology with III-V compounds is GaP, which is practically matched with Si in terms of the lattice parameter (lattice mismatch 0.37% at 300 K). However, growing GaP on Si is not an easy task. Incompatibility of intrinsic material properties such as lattice mismatch, interfacial heterovalence (polar/nonpolar),
thermal expansion mismatch, and interface chemistry creates electrically active crystal defects, including antiphase domains (APDs), stacking faults (SF), and microtwins (MT), as well as dislocations [7, 8]. Recent work aimed at careful control and design of Si substrate preparation and GaP nucleation has shown that APD, SF, and MT defects can be suppressed [9]. Most studies of the growth of GaP on Si have been focused on thin pseudomorphic GaP layers with an emphasis on reducing the growth of polar defects [9–14]. Consideration of the mechanics of the epitaxial film and the properties of heterovalent interfaces in the design of the epitaxial structure was used to create high-quality heteroepitaxial GaP/Si layers capable of supporting further epitaxy of III-V compounds for devices, in particular photovoltaic devices [15, 16]. In the above studies, GaP-on-Si epitaxial layers were grown by metal organic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE). However, despite the outlined successes in the integration of III-V compounds with Si technology and the emergence of instrumental applications, heteroepitaxy of III-V semiconductors on Si still remains a difficult problem [17].

In this work, we investigated the possibility of growing an epitaxial GaP layer on Si substrates through a buffer layer consisting of the substitutional solid solution (Si$_2$)$_{1-x}$(GaP)$_x$ (0 ≤ x ≤ 1).

To grow epitaxial GaP films on Si substrates, we chose the method of liquid-phase epitaxy from a limited volume of a solution-melt. The thermodynamic condition of the process is chosen so that when GaP and Si are dissolved in metallic solvents, the dissolved materials are in the form of molecules. This contributes to the formation of a solid solution (Si$_2$)$_{1-x}$(GaP)$_x$ of molecular substitution; that is, Si molecules are replaced by GaP molecules. This method makes it possible to grow layers of (Si$_2$)$_{1-x}$(GaP)$_x$ (0 ≤ x ≤ 1) solid solution with a smoothly and monotonically varying composition from Si to GaP. In addition, the process of solid solution crystallization occurs under conditions close to equilibrium. The cooling rate of the solution-melt is rather low. The time of migration of dissolved molecules of GaP and Si$_2$ along the crystallization front is much longer than the time of their transition to the crystalline state. Molecules migrating over the surface will crystallize exactly in those places where the energy of elastic distortion of the crystal lattice will be minimal. This facilitates the growth of an epitaxial film with a perfect crystal structure.

2. Methods and Materials

2.1. Growing Graded-Gap Layers of the Solid Solution (Si$_2$)$_{1-x}$(GaP)$_x$ (0 ≤ x ≤ 1) on Si Substrates from a Limited Volume of an Sn Solution-Melt. Growing epitaxial GaP directly on Si substrates does not allow obtaining high-quality layers of GaP, since Si and GaP coefficients of thermal expansion differ by more than 2.5 times. Therefore, it is advisable to grow epitaxial GaP layers on Si substrates through a buffer layer, which smooths out the mismatch between the thermal parameters of the substrate and the epitaxial film. In this work, a continuous solid solution (Si$_2$)$_{1-x}$(GaP)$_x$ (0 ≤ x ≤ 1) was used as a buffer layer, in which the composition changes in the direction of growth from Si to GaP (x varies from 0 to 1). As is known, when growing from a liquid phase, the components of an epitaxial film are preliminarily dissolved in a metal-solvent until saturation. After being brought into contact with the substrate, the saturated solution-melt is cooled at a certain rate, and the excess of the dissolved material begins to crystallize on the surface of the substrate in the form of an epitaxial film. The composition of the solution-melt consisting of Sn, Si, and GaP is determined from the phase diagram of Sn-Si and Sn-GaP and has the following ratio of components (in mass percent): Sn: 95.88%, Si: 1.73%, and GaP: 2.39%.

The possibility of the formation of a solid solution of molecular substitution between Si and GaP was studied. According to the criteria, for the formation of a substitutional solid solution, the following conditions must be met [18]:

(i) The sums of the valences of the atoms of the molecules of the substituting components of the solid solution must be the same

(ii) The sums of the covalent radii of the atoms of the molecules of the substituting components should not differ by more than 10%

According to the calculations, it can be said that Si and GaP can form a continuous substitutional solid solution with molecular substitution (Figure 1), but, with atomic substitution, they cannot.

For the formation of the solid solution between Si and GaP, it is necessary to create conditions for molecular substitution; that is, the GaP molecules must be replaced by Si molecules. These conditions were created on the basis of the model proposed in [19]. According to this model, semiconducting compounds III-V and II-VI and elementary semiconductors, such as Si and Ge, when dissolved in metallic solvents, at temperatures much lower than the melting point of these materials, are mainly in the form of molecules (Figure 2).

Epitaxial GaP layers on Si substrates, through buffer layers of the solid solution (Si$_2$)$_{1-x}$(GaP)$_x$ (0 ≤ x ≤ 1), were grown by liquid-phase epitaxy from a limited volume of an Sn solution-melt. The temperatures of the beginning and end of crystallization were −980°C and −830°C, respectively. The composition of the solution-melt, consisting of Sn, Si, and GaP, was determined from the phase diagram of the Sn-Si binary alloy, taking into account the solubility of GaP in Sn. The composition of the solution-melt is as follows: Sn: 100 gr, Si: 1.8 gr, and GaP: 2.5 gr.

For growth, we used Si substrates with the diameter of 20 mm, with the crystallographic orientation (111). Studies have shown that the highest-quality epitaxial films of continuous solid solution (Si$_2$)$_{1-x}$(GaP)$_x$ are obtained by cooling the solution-melt at the rate of 0.5–1.5 deg/min. The thickness of the solution-melt, that is, the width of the gap between two horizontally located substrates, d = 0.75 mm. The grown epitaxial films (Si$_2$)$_{1-x}$(GaP)$_x$ had n-type conductivity, and their thickness was 25 μm.
3. Results and Discussion

3.1. Structural Studies of the Solid Solution \((\text{Si}_2)_{1-x} \text{(GaP)}_x\) \((0 < x < 1)\). Structural studies of epitaxial films were fulfilled on the DRON-3M X-ray diffractometer (CuK\(_\alpha\) radiation, \(\lambda = 0.15418\) nm) according to the \(\theta - 2\theta\) scheme (reflection (111), FWHM = 6.2\(^\circ\)) in the step-by-step scanning mode.

In Figure 3, the X-ray diffraction pattern of the Si substrate is shown. The analysis showed that the substrate surface corresponds to the (111) crystallographic plane. The high intensity \((2 \times 10^5\) pulses/sec) and the selective nature of reflections of the (HHH) type indicate a sufficient perfection of the crystal lattice of the substrate (Figures 3, 4(a), and 4(b)). The experimental value of the lattice parameter of the substrate was \(a_{\text{Si}} = 5.4329\) Å, which is comparable with the tabulated value \(a_{\text{Si}} = 5.4307\) Å [20].

Good splitting of the (333) reflection, close to the calculated splitting with the characteristic ratio of intensities for \(\alpha_1\) and \(\alpha_2\) emissions [133 (\(\alpha_1\)) 2I333(\(\alpha_2\))] (Figure 4(b)) and the singlet shape of the (111) reflection (Figure 4(a)), indicates that the elastic stress is predominantly concentrated on the near-surface layer of the substrate and at the boundaries between the blocks [21].

In addition, the X-ray diffraction pattern at small scattering angles \(2\theta \approx 18.4^\circ\) exhibits a broad diffuse reflection caused by structural \(\text{SiO}_x\) fragments in the surface layers with unsaturated bonds.

In Figure 5, the diffraction pattern of the epitaxial film \((\text{Si}_2)_{1-x} \text{(GaP)}_x\) grown on an Si substrate is shown. It differs somewhat from the diffraction pattern of the substrate. In the X-ray diffraction pattern of the \((\text{Si}_2)_{1-x} \text{(GaP)}_x\) film, the intensity of the main (111) reflection increased by 12.5%
compared to the intensity of the same reflection from the Si substrate. The intensity of the second order (222) (forbidden reflection) increased by 25%, while the intensity of the third order (333) and the intensity of the (110) reflection from the SiO$_2$ impurity phase remained practically unchanged. The level of the inelastic background of the X-ray diffraction pattern of the film increased to average of 20% compared to the level of the inelastic background of the X-ray pattern of the Si substrate.

The selective nature and high intensity ($2 \times 10^5$ pulses/sec) of the main reflection (111) indicate a sufficient degree of perfection of the crystal lattice of the (Si$_2$)$_{1-x}$(GaP)$_x$ film; that is, the grown film is single-crystal with the (111) orientation. The sizes of the subcrystallites (blocks) of the film, estimated from the width of this reflection by the Selyakov-Scherrer method [22], were about $\sim 39 \pm 1$nm. However, the intensity of the main reflection (111) of the film is 12.5% higher than the intensity of the same line of the substrate. This indicates the partial replacement of some paired Si ions by molecules of Ga and P ions in the Si lattice of the film. Since the intensity of dispersion of X-ray is proportional to the atomic number ($Z$) of the elements, such a replacement of ions should lead to an increase in the intensity of this reflection of the solid solution, because $Z_{Si} = 14$, $Z_{Ga} = 31$, and $Z_{P} = 15$. In this case, an unequal increase in the intensity of the main reflection (111) and its second (222) order shows an
inhomogeneous distribution of components with varying composition over the depth of the matrix lattice of the film. The absence of changes in the third-order intensity (333) indicates that GaP molecules replace paired Si₂ molecules in the film lattice only in the near-surface layer during epitaxy. The constancy of the intensity of the (110) structural reflection during epitaxy can be explained by the absence of replacement of the SiO molecule by the GaP molecule in the lattice of the impurity phase. These experimental facts allow us to conclude that, during epitaxy, GaP molecules replace Si₂ molecules only in the surface layer in the matrix lattice of the (Si₂)₁₋ₓ(GaP)ₓ solid solution.

In Figure 6, the shape of the diffraction reflection (111) of the epitaxial film is separately shown. It is seen that the examined structure line was formed because of the superposition of two similar reflections with each other. Analysis showed that this reflection is the result of the superposition of the separate diffraction reflection (111) from two sublattices of the film, that is, from the Si and GaP sublattices. Due to the closeness of the lattice parameters of Si (\(a_{Si} = 5.4307 \text{ Å}\)) and GaP (\(a_{GaP} = 5.4451 \text{ Å}\)), the angular distance between the (111) peaks from different sublattices (\(\Delta\theta_{GaP-Si} = 3.1'\)) of the film is apparently comparable to the angular distance between the (111) peaks from \(a_{\alpha}\) and \(a_{\alpha_2}\) copper radiation (\(\Delta\theta_{a_{2}-a_{1}} = 2.2'\)) in the given scattering angle. Therefore, the shape of the (111) reflection of the solid solution has a triplet form. Almost equal intensities of diffraction reflections (111) from different Si and GaP sublattices make it possible to determine the composition of the solid solution in the near-surface layer of the film as stoichiometric Si₂-GaP.

The overestimated value of the inelastic background level of the film in the entire angular range is also a result of the substitution of paired silicon ions by GaP molecules. Large ionic radii of Ga and P favor the replacement of paired Si atoms by molecules of the same GaP elements in the boundary regions between the film blocks, that is, in the defective regions of the film. Since in these regions the chemical bonds between the matrix ions are not saturated and the sites are somewhat displaced from their ideal positions, all these factors cause relaxation of the elastic energy of the distorted film lattice, which results in the formation of structural fragments of GaP in the subsurface layer and an increased level of inelastic background.

3.2. X-Ray Spectral Microprobe Analysis of the Chemical Composition of the Solid Solution \((Si_2)_{1-x}(GaP)_x (0 \leq x \leq 1)\). The distribution of the components across the thickness of the epitaxial layer of solid solution \((Si_2)_{1-x}(GaP)_x (0 \leq x \leq 1)\), grown on a Si substrate, has been determined by X-ray microprobe microanalyzer “Comeca.” Studies have shown that the Ga content in the near-surface region of the epitaxial film, the P content is 2 atomic percent less than the Ga content. This is due to the fact that, at the growing temperature of 950°C, the vapor pressure of P is much higher than the vapor pressure of Ga, and the P atoms partially evaporate.

3.3. Photoluminescence Spectrum of the Solid Solution \((Si_2)_{1-x}(GaP)_x\). Figure 8 shows a spectrum of the photoluminescence of the solid solution \((Si_2)_{1-x}(GaP)_x\) measured at 5 K. As can be seen from Figure 8, the photoluminescence spectrum has a wide band covering from 400 to 900 nm, with a maximum emission at \(\lambda_{max} = 525 \text{ nm}\), which corresponds to the photon energy of 2.28 eV. Photons with such energy are formed during radiative band-band recombination of electron-hole pairs located in the near-surface epitaxial layer of the solid solution. This maximum is due to the wide-gap component of the solid solution (GaP), which makes up the main part of the \((Si_2)_{1-x}(GaP)_x\) solid solution in the near-surface region of the epitaxial film, in which the energy gap (\(E_g\)) is \(E_g = 2.28 \text{ eV}\). In the long-wavelength region of the photoluminescence spectrum, one more peak is observed at 840 nm, which corresponds to the photon energy...
$E_{ph} = 1.47 \text{ eV}$. Apparently, this peak is responsible for the radiative transition of electrons from the conduction band to the impurity levels of atoms of Si$_2$ molecules located in the forbidden band of the solid solution. The Si content in the near-surface region of the film is less than 1 mol%, due to which the energy level at $E_{ph} = 1.47 \text{ eV}$ can be caused by radiative recombination of charge carriers with the participation of the Si–Si covalent bond, which is surrounded by the GaP-enriched sublayer of the tetrahedral lattice of the solid solution (Si$_2$)$_{1-x}$(GaP)$_x$ ($0 \leq x \leq 1$) (Figure 1). As is known, the breaking energy of the Si–Si covalent bond located in the tetrahedral lattice of ordinary silicon at 5 K is $E_{g, \text{Si}} = 1.2 \text{ eV}$ [23]. However, when Si$_2$ molecules are surrounded by relatively tightly bound atoms of GaP molecules, as a result of hybridization of the electron shells of atoms of Si$_2$ and GaP molecules, the energy of the Si–Si covalent bond increases to $E_i = 1.47 \text{ eV}$ at 5 K and gives rise to a diffuse band of the energy level of the atoms of the Si$_2$ molecules in the band gap of GaP. The Si–Si covalent bond in the crystal lattice of GaP (Figure 1) under the influence of the surrounding atoms of the base material is enhanced by $\Delta E = E_i - E_{g, \text{Si}} \approx (1.47 - 1.2) \text{ eV} = 0.27 \text{ eV}$ at the temperature of 5 K. Since the covalent bond of Si–Si atoms is saturated, that is, since the sums of the valences of the atoms of the Si$_2$ and GaP molecules are equal, the atoms of the Si$_2$ molecules in the tetrahedral lattice of GaP, apparently, have a donor character. Since the sums of the covalent radii of atoms of GaP molecules ($r_{Ga} + r_P = 2.36 \text{ Å}$) and Si$_2$ ($r_{Si} + r_{Si} = 2.34 \text{ Å}$) have similar values, consequently, the substitution of GaP molecules by Si$_2$ molecules does not lead to a strong deformation of the crystal lattice. The atoms of the Si$_2$ molecule located at the sites of the crystal lattice of GaP are not active centers of recombination.

These conclusions are based on the fact that we have previously observed the appearance of such peaks of atoms of Si$_2$ molecules in the photoluminescence spectrum of solid solutions of Si and binary compounds, such as (Si$_2$)$_{1-x}$(GaAs)$_x$ at 1.33 eV [24] and (Si$_2$)$_{1-x}$(ZnSe)$_x$ at 1.67 eV [25], as well as a similar luminescence peak of atoms of ZnSe molecules in the photoluminescence spectrum of (GaAs)$_{1-x}$(ZnSe)$_x$ solid solution [26] at 2.67 eV. A certain conformity to natural laws is observed here; the wider the band gap of the base material is, into which the Si$_2$ molecules are introduced as an impurity or form a solid solution with it, the higher the energies of Si atoms give a peak in the photoluminescence spectrum.

### 3.4. Radiative Properties of the n-GaP–n$^+$-(Si$_2$)$_x$(GaP)$_{1-x}$ (0 \leq x \leq 0.01) Structures

To study the effect of Si atoms on the radiative properties of GaP, epitaxial layers of the (Si$_2$)$_x$(GaP)$_{1-x}$ (0 \leq x \leq 0.01) solid solution were grown by liquid-phase epitaxy from an Sn solution-melt in the temperature range of 800–750°C on n-GaP substrates with crystallographic orientation (111). In this growth mode, the molar concentration of Si in the (Si$_2$)$_x$(GaP)$_{1-x}$ solid solution is no more than 1 mol%. The grown layers had n-type conductivity. An isotropic structure n-GaP–n$^+$-(Si$_2$)$_x$(GaP)$_{1-x}$ (0 \leq x \leq 0.01) was fabricated on the basis of the (Si$_2$)$_x$(GaP)$_{1-x}$ solid solution. The area of the fabricated structure was $S = 4 \text{ mm}^2$. Ohmic contacts were made from an indium-gallium solution. When a reverse bias of 7 to 12 V was applied to the structure, a red light was observed (Figure 9). GaP has the band gap of 2.26 eV, and it corresponds to yellow light. The energy of the observed red light corresponds to $\sim 1.6 \text{ eV}$. The appearance of the red light, apparently, is associated with the band of energy levels of atoms of Si molecules, formed in the band gap of the solid solution (Si$_2$)$_x$(GaP)$_{1-x}$ (0 \leq x \leq 0.01), as indicated above: $E_i = 1.47 \text{ eV}$. Studies of the temperature dependence of the current-voltage characteristic of the p-Si–n-(Si$_2$)$_x$(GaP)$_{1-x}$ (0 \leq x \leq 1) heterostructure showed that the band width of the energy levels of Si atoms located in the band gap of the solid solution is $\Delta E = 0.14 \text{ eV}$. Consequently, the lower boundary of the band of energy levels of Si atoms will be located at $E_{Si} = E_i + \Delta E = 1.47 \text{ eV} + 0.14 \text{ eV} = 1.61 \text{ eV}$ below the bottom of the conduction band of the solid solution. With a sufficient level of injection of electrons from the n–n$^+$ junction to the base region of the n-GaP–n$^+$-(Si$_2$)$_x$(GaP)$_{1-x}$ (0 \leq x \leq 0.01) structure, apparently electrons will pass from the bottom of the conduction band to energy levels of atoms of Si molecules: $E_{Si} = 1.61 \text{ eV}$. Such electronic transitions emit red light (Figure 9).
4. Conclusion

The principal possibility of growing continuous graded-gap solid solutions $\text{(Si}_2\text{)}_{1-x}\text{(GaP)}_x$ ($0 \leq x \leq 1$) on Si and GaP substrates with crystallographic orientation (111) by liquid-phase epitaxy is shown for the first time. The grown films have a perfect single-crystal structure with the crystallographic orientation (111), with the size of subcrystallites $\sim 39 \pm 1$ nm. It is shown that in the epitaxial layer there is a smooth change in the composition of the solid solution from Si to GaP, with pure GaP on the film surface. A study of the photoluminescence spectrum at the temperature of 5 K revealed a band of energy levels of atoms of Si molecules located in the band gap of the solid solution $\text{(Si}_2\text{)}_{1-x}\text{(GaP)}_x$ ($0 \leq x \leq 1$). These energy levels are located 1.47 eV below the bottom of the conduction band of the solid solution. An isotype structure $n\text{-GaP}^-\text{-n}'\text{-GaP}^-$ $\text{(Si}_2\text{)}_x$ ($0 \leq x \leq 0.01$) was fabricated. When a reverse voltage is applied, red emission with the wavelength of 770 nm is observed; it occurs with the participation of the energy levels of atoms of Si molecules.

Data Availability

Anyone can use the scientific news, formulas, and graphs which obtained results in the research paper provided that they provide citation in the references of their work to our study.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

This work was performed on the basis of the fundamental research program of the Uzbekistan Academy of Sciences on the topic “Photovoltaic, thermal-voltaic, photo-thermal-voltaic and radiative effects in two and multicomponent semiconductor solid solutions with nanocrystals, obtained on silicon substrates from the liquid phase.” The work is dedicated to the 90th anniversary of the academician of the Academy of Sciences of the Republic of Uzbekistan, M. S. Saidov.

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