# X-RAY DIFFRACTION AND RAMAN SPECTROSCOPY ANALYSES OF GaSb-ENRICHED SI SURFACE FORMED BY APPLYING DIFFUSION DOPING TECHNIQUE<sup>†</sup>

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The paper studies the properties of surface and near-surface region of a single crystalline silicon sample doped with atoms of Ga ( $A^{III}$ ) and Sb ( $B^V$ ). n-type single-crystal Si wafers were chosen as substrates, and samples were size of  $8 \times 10 \times 0.5$  mm<sup>3</sup>. For diffusion into silicon, Ga and Sb impurities were used with a purity of 99.999 and 99.998, respectively. The authors propose that a new heterostructure might form in the near-surface region of silicon that could be engineered by applying a relatively cheap diffusion method. The experimental and analysis results show that the composition and absorption spectrum of silicon start manifest certain changes, and can be used in the future as a functional material for solar cells. The result showed that randomly located islands with an average diameter of 1–15 µm are formed on the substrate surface. X-ray diffraction analysis was carried out using a Rigaku diffractometer to study the crystallographic parameters of islands formed with the participation of Ga and Sb atoms on the silicon surface. The energy spectrum was studied on Nanofinder High End Raman spectrometer (LOTIS TII) in order to determine the presence of complexes of Ga and Sb atoms within islands formed as a result of diffusion. The optical emission spectra in the new structure were studied using a Lambda 950 spectrophotometer. The measurements were carried out at room temperature, i.e., at 300°K. Having studied the results of X-ray analysis, Raman spectroscopy, and optical spectroscopy, the authors have revealed that Ga and Sb atoms form new Si<sub>0.44</sub>(GaSb)<sub>0.56</sub> and Si<sub>0.75</sub>(GaSb)<sub>0.25</sub>-type binary compounds on Si surface.

Keywords: Silicon, Gallium; Antimony; Doping; Diffusion; Microsized islands PACS: 61.72.uf, 68.43.Jk

## **1. INTRODUCTION**

It is well known that silicon tends to be the main semiconductor material most commonly used in the field of electronics. Despite a number of advantages such as the abundance of silicon raw material on earth, the availability of a standard production technology, nevertheless, the main parameters of silicon, such as the band gap, charge carrier mobility, energy band structure, cannot meet the requirements of the current rapidly developing field of electronics [1-3]. Therefore, today it is of certain scientific and practical importance to study the effect of binary compounds on the crystal lattice, leading to a substantial alteration in the properties of silicon [4-5]. GaSb layers were obtained on various substrates by the authors [6-7] using the modern technique of molecular beam epitaxy, while their properties were studied by X-ray diffraction analysis [8], Raman spectroscopy [9], transmission electron microscopy (TEM) [10-11]. The possibilities of manufacturing high-precision electronic devices [12–14] and manufacturing infrared sensors [15–16] are shown. Previously unknown nanoscale effects have been discovered in GaSb-based structures [17–19], where layers are limited across one, two, or even three dimensions. However, the modern method of molecular beam epitaxy, which is currently used to obtain nanosized structures, requires the availability of expensive equipment and complex technological processes [20].

While growing thin layers, the quality of crystallization strongly depends on the crystallographic parameters of the base material, which means that the lattice constants of the substrate and growing crystals should be close to each other [21]. An analysis of the literature shows that, in most cases, the degree of interaction between the resulting GaSb layer and the substrate was not taken into account [22–24]. In this paper, we demonstrate that diffusion technology can be used to form thin layers of GaSb transferred onto a Si substrate, and the parameters of the obtained samples are given (Si and GaSb ~12% lattice mismatch).

### 2. MATERIALS AND METHODS

A Czochralski-grown n-type single-crystal Si wafer was chosen as the substrate, and several samples were made for further cutting using a Machine STX-402 (diamond wire cutting machine STX-402) brand. Samples were cut out with a size of  $8 \times 10 \times 0.5$  mm<sup>3</sup>. For diffusion into silicon, Ga and Sb impurities were used with a purity of 99.999 and 99.998, respectively.

Before diffusion, the substrate surface was subjected to standard cleaning and degreasing with HF-acid at 1-2 minutes intervals. The diffusion process was carried out inside AOT-GLS-1750X-type Vacuum Tube Furnace for

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Laboratory Material Burning. The diffusion process lasted for 5 hours and started from room temperature (30°C) up to 1200°C, then it was carried out in a gas-phase medium of impurity atoms at this temperature. The image of the silicon surface after diffusion was obtained using a DJ-SEM-type 150D-ST scanning electron microscope.

The result showed that randomly located islands with an average diameter of  $1-15 \,\mu\text{m}$  are formed on the substrate surface. X-ray diffraction analysis was carried out using a Rigaku diffractometer to study the crystallographic parameters of islands formed with the participation of Ga and Sb atoms on the silicon surface. The energy spectrum was studied on Nanofinder High End Raman spectrometer (LOTIS TII) in order to determine the presence of complexes of Ga and Sb atoms within islands formed as a result of diffusion. The optical emission spectra in the new structure were studied using a Lambda 950 spectrophotometer. The measurements were carried out at room temperature, i.e., at  $300^{\circ}\text{K}$ .



Figure 1. SEM image of the surface of Si doped with Ga and Sb

## 3. RESULTS AND DISCUSSION 3.1. XRD analysis

The Figure 2 illustrates the comparative analysis of X-ray diffraction data of Si sample in which Ga and Sb atoms were diffusely embedded and the values extracted from COD crystallographic open database. The spectrogram of X-rays reflected from the surface of a silicon sample after diffusion shows 17 main peaks (Fig. 2-*a*). To identify the resulting peaks, the lines were superimposed corresponding to 2 theta angles (Fig. 2-*b*, *c*) from the International Open Crystallographic Database for GaSb and Si materials.



Figure 2. X-ray pattern: a – Si sample doped with Ga and Sb atoms; b and c – GaSb and Si semiconductor crystal databases from the COD (Crystallography Open Database) No: 96-900-8848 and No: 96-901-1999 respectively

One can judge that GaSb form on Si surface that is reflected by revealing peaks 25.25, 29.35, 41.89, 49.58, 60.78, 66.05, 67.5, 76.55, 82.25, 91.32, 96.82, 98.71, corresponding to crystallographic directions (111), (020), (202), (311), (040), (313), (402), (242), (511), (404), (513), (060). In the general spectrum in Figure 2-a, of course, there are peaks 56.23, 69.41, 76.55, related to Si, corresponding to crystallographic planes (131), (040), (313). Peak analysis showed that there were peaks No. 2, No. 9, which do not belong to either GaSb or Si. It's known that lattice constants of GaSb and Si are a=6,09 Å, a=5,34 Å respectively [25-26].

The peaks of GaSb (242) in Figure 2-*b* and Si (313) in Figure 2-*c* overlap. The authors of [27] indicate that peak No.4 belongs to Si (020). But since the No.2 and No.9 peaks are located between the GaSb 2 theta=25.25 (111) and Si 2 theta=28.45 (111) and GaSb 2 theta = 60.51 (040) and Si 2 theta = 69.25 (040) peaks, respectively, we can assume that that the detected peaks No.2 (111) and peak No.9 (040) probably belong to the new Si<sub>1-x</sub>GaSb<sub>x</sub> structure. Using these results, the lattice constant (*a*) and diffraction lattice constant (d<sub>hkl</sub>) of the GaSb binary compound formed on Si and its surface were calculated using the following expressions 1 and 2, respectively (Table 1)

$$a = d_{hkl} \cdot \sqrt{h^2 + k^2 + l^2} \,. \tag{1}$$

$$d_{hkl} = \frac{\lambda}{2 \cdot \sin \theta},\tag{2}$$

where  $\theta$  is Bragg's angle and  $\lambda$ =0.15402 nm (wavelength of Cu - K $\alpha$ ).

Table 1. Values of the lattice constant and diffraction lattice constant of new crystals formed in the sample, calculated based on the experimental results.

Crystal	Peak No.	2 theta	h	k	1	$d_{_{hkl}}, {\mathop{ m A}\limits^{ m o}}$	$a_{hkl}, \overset{\mathrm{o}}{\mathrm{A}}$
GaSb	1	25.25	1	1	1	3.52	6.10
	3	29.35	0	2	0	3.04	6.08
	5	41.89	2	0	2	2.15	6.09
	6	49.58	3	1	1	1.84	6.09
	8	60.78	0	4	0	1.52	6.09
	10	66.05	3	1	3	1.41	6.16
	11	67.5	4	0	2	1.39	6.19
	13	76.55	2	4	2	1.24	6.09
	14	82.25	5	1	1	1.17	6.08
	15	91.32	4	0	4	1.08	6.09
	16	96.82	5	1	3	1.03	6.09
	17	98.71	0	6	0	1.01	6.09
Si	4	33.21	0	2	0	2.69	5.39
	7	56.23	1	3	1	1.63	5.42
	12	69.41	0	4	0	1.35	5.41
	13	76.55	3	1	3	1.24	5.42
$\operatorname{Si}_{1-x}(\operatorname{GaSb})_x$	2	27.19	1	1	1	3.28	5.67
	9	61.55	0	4	0	1.50	6.02

### 3.2. Analysis of Raman spectroscopy

Raman spectrometry is the most useful method available for studying lattice vibrations and their interactions with other excitations. Changes after implantation of type III-V semiconductors with ions were considered based on the study of Raman spectra [28-30]. When Raman spectra are obtained at room temperature while samples were exposed to Ar laser with a wavelength of 5145 Å, the light penetration depth happened to be 1000 Å [28-30]. Therefore, it is possible to estimate the properties of the near-surface structure using the Raman spectrometry analysis [28-30]. Given that the Ga and Sb atoms in silicon are located at the sites of the crystal lattice [28-30], we can expect changes in lattice vibrations. Detecting such changes by studying the Raman spectra is of both scientific and practical importance. Therefore, during the study of Si samples doped with Ga and Sb atoms, the Raman spectrum was obtained at 3 different points depending on the areas on the image of the sample surface (Fig. 3).

Figures 3-*b*, *d*, *f* represents an image of the surface of the sample, and also show the areas where laser light with a wavelength of 532 nm and a power of 20 MW is incident. As seen in the pictures, the main surface is gray with white islands and black closed-type curves. These three regions, which differ from each other, were chosen for exposer to laser beam. A graphical representation of each selected point of the Raman spectrum is shown in Figures 3-*a*, *c*, *e*, respectively. Analysis of the Raman spectrum obtained in the main gray area in Figure 3-*a* showed the presence of one peak at 519 cm<sup>-1</sup>, the intensity of which was very high (intensity = 15000 a.u.). The fact that this peak belongs to Si has been confirmed in literature [30]. However, figure 3-*c* shows the spectrum obtained at one of the points on the surface of the white island. As can be seen from the figure, one peak was found in the spectrum at 225 cm<sup>-1</sup> (intensity = 7000 a.u.), and it turned out that this peak belongs to GaSb (LO) crystal. Figure 3-*e* shows the spectrum obtained above the black line, in this case 2 completely different peaks were obtained. When comparing the peak values with the data given in the literature, it was found that the peak at 222.1 cm<sup>-1</sup> refers to GaSb, and the peak at 519 cm<sup>-1</sup> to Si. From the latest results obtained, we can assume that the region with a black closed curve is a crystal with the composition Si(GaSb).



e)

f) Figure 3. Raman spectrum and image of Si sample surface doped with Ga and Sb impurity atoms

## 3.3. Spectroscopic analysis

It is known that the band gap of GaSb binary compound and Si semiconductor material is Eg=0.726 eV, Eg=1.12 eV (at T=300 K), respectively. The band gap of semiconductors with complex compounds can theoretically be calculated using Vegard's law (expression 7)

$$E_{g,A_{(1-x)}B_x} = (1-x) \cdot E_{g,A} + x \cdot E_{g,B}$$
<sup>(7)</sup>

Where  $E_{g,A}$  is the band gap of material A,  $E_{g,B}$  is the band gap of material B,  $E_{g,A_{(1-x)}B_x}$  is the band gap of the compound material, x is the fraction of the material. Using expression 7, the band gap of the Si<sub>(1-x)</sub>(GaSb)<sub>x</sub> compound was calculated. The results of the study showed that the band gap of Si<sub>(1-x)</sub>(GaSb)<sub>x</sub> compound can be in the range of  $0.726 \div 1.12$  eV.

A Si sample doped with Ga and Sb atoms was characterized by light reflection using a Perkin-Elmer Lambda 950type UV-visible spectrophotometer, available at the laboratory of the Solar Energy Institute at EGE University (Fig. 5).



Figure 5. Reflective characteristic of a Si sample doped with Ga and Sb atoms

The Figure 5 shows the energies  $E_1=0.72 \text{ eV}$ ,  $E_2=0.896 \text{ eV}$  and  $E_3=1.02 \text{ eV}$ . The energy  $E_1$  is the band gap of the GaSb semiconductor material,  $E_2$  and  $E_3$  are the band gap values of the material with  $\text{Si}_{(l-x)}(\text{GaSb})_x$  compounds. Having determined the ratio of Si and GaSb using the inverse calculation of Vegard's law (formula 7) and taking into consideration that  $E_2=0.892 \text{ eV}$  is the band gap Eg of the material  $\text{Si}_{(l-x)}(\text{GaSb})_x$ , we calculate it in GFA as follows.

$$E_{g,Si_{(1-x)}(GaSb)_x} = (1-x) \cdot E_{g,Si} + x \cdot E_{g,GaSb}$$

if we substitute the energy values  $E_2$ ,  $E_{g,Si}$ ,  $E_{g,GaSb}$  into this equation, we get the following linear equation with one unknown

$$0.896 = (1 - x) \cdot 1.12 + x \cdot 0.726$$

This equation shows that x = 0.56. Thus, we can say that E<sub>2</sub> corresponds to energy of Si<sub>0.44</sub>(GaSb)<sub>0.56</sub> newly formed structure. Similarly, E<sub>3</sub> corresponds to the energy of Si<sub>0.75</sub>(GaSb)<sub>0.25</sub> structure.

### 3.4. Discussion

In times of diffusion of more than one type of impurity element atoms into silicon, under certain thermodynamic conditions, the formation of complex compounds can theoretically be predicted. In this work, to study diffusion into silicon of elements with similar diffusion parameters, solubility, and diffusion coefficient, such as Ga and Sb, were chosen as impurities. In addition, GaSb itself is a semiconductor material, the crystal parameters of which are fundamentally different from those of Si. Its cost is 1.5 times greater than the cost of Si. The formation of GaSb crystals on the silicon surface by diffusion of atoms of the elements Ga and Sb in Si without the use of a priori GaSb material was confirmed by X-ray diffraction analysis.

The results of the experiments revealed the formation of a new type of crystal, consisting not only of GaSb crystals, but also of components of Si and GaSb crystals. This is of great scientific and practical importance, since a new Si-based material can be obtained by simple diffusion process. It can be assumed that the properties of the material might reflect new previously unknown properties of Si and GaSb semiconductor materials. The graph of the comparative XRD and Raman spectra shows the formation of a new peak. Based on the calculation results, we assumed that the newly formed compound contains SiGaSb, and its lattice constant was equal to 5.67 Å and 6.02 Å. The properties of the new crystal further expand the scope of Si, differing from the fundamental parameters of Si. Based on the results obtained, it can be concluded that Si can also be widely used in optoelectronics.

### 4. CONCLUSION

The formation of a new  $Si_{0.44}(GaSb)_{0.56}$  and  $Si_{0.75}(GaSb)_{0.25}$ -type crystal structures on the surface of a Si sample doped by the diffusion method with impurity Ga and Sb atoms, was confirmed using three modern spectroscopic methods: X-ray phase analysis, Raman spectroscopy, and UV-VIS-NIR spectroscopy. The results of the experiments showed that a new crystal structure containing GaSb on the Si surface can be formed using an inexpensive and widely used diffusion doping technology. The crystallographic parameters of the new crystal were presented for the first time on the basis of experimental results.

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### АНАЛІЗ РЕНТГЕНОВСЬКОЇ ДИФРАКЦІЇ ТА РАМАНІВСЬКА СПЕКТРОСКОПІЯ ПОВЕРХНІ Si, ЗБАГАЧЕНОЇ GaSb, СФОРМОВАНОЇ ШЛЯХОМ ЗАСТОСУВАННЯ МЕТОДУ ДИФУЗІЙНОГО ЛЕГУВАННЯ Халмурат М. Ілієв<sup>а</sup>, Володимир Б. Оджаєв<sup>ь</sup>, Собір Б. Ісамов<sup>а</sup>, Бобір О. Ісаков<sup>а</sup>, Байрамбай К. Ісмайлов<sup>а</sup>, Кутуб С. Аюпов<sup>а</sup>, Шахзодбек І. Хамрокулов<sup>а</sup>, Сарвіноз О. Хасанбаева<sup>а</sup>

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Досліджено властивості поверхневої та приповерхневої області зразка монокристалічного кремнію, легованого атомами Ga (AIII) та Sb (BV). Як підкладки були обрані пластини монокристалічного кремнію п-типу, розмір зразків  $8 \times 10 \times 0,5$  мм<sup>3</sup>. Для дифузії в кремній використовували домішки Ga i Sb з чистотою 99,999 і 99,998 відповідно. Автори припускають, що нова гетероструктура може утворитися в приповерхневій області кремнію, яку можна створити шляхом застосування відносно дешевого методу дифузії. Результати експерименту та аналізу показують, що склад і спектр поглинання кремнію на початку зазнають певних змін і можуть використовуватися в майбутньому як функціональний матеріал для сонячних елементів. Результат показав, що на поверхні підкладки утворюються хаотично розташовані острівці із середнім діаметром 1-15 мкм. Рентгеноструктурний аналіз проводили на дифрактометрі Rigaku для дослідження кристалографічних параметрів острівців, утворених за участю атомів Ga та Sb на поверхні кремнію. Енергетичний спектр досліджували на спектрометрі Nanofinder High End Raman (LOTIS TII) з метою визначення наявності комплексів атомів Ga i Sb в острівцях, утворених в результаті дифузії. Спектри оптичної емісії в новій структурі досліджувалися на спектрофотометрі Lambda 950. Вимірювання проводили при кімнатній температурі, 300°К. Вивчивши результати рентгенівського аналізу, спектроскопії комбінаційного розсіювання та оптичної спектроскопії, виявилено, що атоми Ga та Sb утворюють нові бінарні сполуки типу Si<sub>0.44</sub>(GaSb)<sub>0.56</sub> та Si<sub>0.75</sub>(GaSb)<sub>0.25</sub> на поверхні Si.

Ключові слова: кремній, галій; сурма; легування; дифузія; мікроострови