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Ge/Si(001) heteroepitaxial layers doped in the HW CVD process by impurity evaporation from a sublimating Ge source

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Ge/Si(001) heteroepitaxial layers were grown by HW CVD and in situ doped with Ga or Sb using a separate resistively heated Ge source containing one of these impurities. Sublimation of the germanium source gave a concentration of $\sim 1 \cdot 10^{19}\,\mathrm{cm}^{-3}$ gallium atoms in the layers. The mode of introduction of this impurity into the epitaxial layers was investigated as a function of hot filament (Ta) temperature and growth temperature using *C-V* and Hall effect. To increase the maximum concentration of charge carriers in the Ge/Si(001) layers, a melt zone was formed on the Ge source during the growth of the layers, which made it possible to increase the concentration of impurities in the Ge layer by almost an order of magnitude.

Keywords: heteroepitaxial layers, evaporation by sublimation, HW CVD, Si, Ge, *n*- and *p*-type alloying.

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1. Introduction

Recently, germanium(Ge) has found new applications as a key material for the integration of micro- and optoelectronic devices on the silicon (Si) platform [1]. Solid source molecular beam epitaxy (SS MBE) [2,3] and chemical vapor deposition (CVD) are the most significant deposition methods for the Ge-on-Si system [4]. A two-temperature regime ($\sim 350/600^{\circ}\mathrm{C}$) is used for the growth of epitaxial layers (EL) Ge-on-Si in both of these methods [5]. However, the high temperatures of this second growth stage are undesirable for CMOS integration. This prompts an intensive search for alternative low-temperature methods for groing Ge-on-Si. In addition, low temperatures are desirable to improve the introduction of dopants and reduce their diffusion.

Previously, [6] we grew pure (undoped) epitaxial layers of Ge-on-Si on a substrate at a constant low temperature ($\sim 350^{\circ}\mathrm{C}$) using the CVD process with monogermane decomposition (GeH_4) on the „hot wire“ (Hot Wire CVD). In addition, we successfully *in situ* doped such layers with phosphorus atoms to the maximum electron concentration $\sim 1.3 \cdot 10^{20}\,\mathrm{cm}^{-3}$ [7].

At the same time, Ge/Si(001) EL are required for a number of applications both moderately doped ($\sim 1 \cdot 10^{17}\,\mathrm{cm}^{-3}$) and with a high concentration of electrically active impurities ($> 1 \cdot 10^{19}\,\mathrm{cm}^{-3}$). They are required for a number of applications: from nanoscale transistors with high mobility [8] to light-emitting devices based on Ge [9,10].

Currently, *in situ* doping of Ge/Si(001) EL with donor impurities such as phosphorus [7] and antimony [11] has been broadly studied. At the same time, despite the widespread use of the required Ge *p*-type of conductivity layers, their production is not well studied.

So, in the GS MBE method, *in situ* doping of the *p*-type using sources B_2H_6 and Ge_2H_6 was used for the growth of the Ge/Si(001) EL. The layers were grown at a temperature of $T_s = 300{-}400^{\circ}\mathrm{C}$ with a boron concentration between $3 \cdot 10^{16}$ and $4 \cdot 10^{19}\,\mathrm{cm}^{-3}$ [12]. Layers with a higher concentration of boron were grown in another paper of these authors ($8 \cdot 10^{21}\,\mathrm{cm}^{-3}$), but growth was carried out at a higher temperature of $T_s = 600^{\circ}\mathrm{C}$ [13].

In our opinion, it is advisable to use gallium (Ga), which has a greater solubility in the Ge matrix than boron and aluminum, as a dopant for growing Ge EL of *p*-type of conductivity using the HW CVD method [14].

It is necessary to select the appropriate source type to obtain a clean impurity stream for successful *in situ* doping of Ge layers. The evaporation of an impurity from an elementary state used in the MBE method is associated with a number of technical and fundamental challenges. The evaporated particles mainly consist of polyatomic molecules, the embedding of which into the Ge lattice is difficult and leads to the generation of defects [15].

At the same time, there is a known method of doping of Si EL, in which a dopant is evaporated from a solid state by sublimation [15–18]. Mainly single atoms are deposited on the substrate in case of sublimation. For example, mass spectrometric studies of the germanium molecular beam [19] show that it consists of 80% of Ge single atoms. Apparently, the dopant used for doping the germanium single crystal will sublimate in the form of single atoms. Since only single atoms of dopant elements can occupy

electrically active states in the lattice of the germanium layer, it is obvious that the doping efficiency will be determined precisely by the proportion of monatomic particles in the dopant flux.

Another important advantage of this doping method is the stationary composition of the molecular stream after a certain sublimation time.

Using a similar variant with a sublimation Si source, for growing high-quality layers *p*-Ge/Si(001) using the HW CVD method, we proposed using evaporation of gallium atoms (Ga) from a sublimation source cut from a monocrystalline germanium ingot, which is doped with this dopant [20]. When the source is heated to a temperature close to the melting point of Ge, mainly Ga atoms evaporate from it, since the pressure of saturated vapors Ga is almost 4 orders of magnitude higher than the vapor pressure Ge [21].

At the same time, for controlled *in situ* doping of Ge EL, it is necessary to study in more detail the patterns of transfer of Ga atoms and other dopants (for example, Sb) from a sublimating Ge source to a growing layer in the HW CVD process. Since the vapor pressure of Sb atoms, as well as the vapor pressure of gallium atoms, is also higher than the vapor pressure of Ge at a temperature close to its melting point [21], it can be expected that their evaporation from the Ge source will also be quite intense, which will allow *in situ* doping Ge/Si(001) layers with them.

It should be also noted that in case of doping of Ge/Si(001) EL from a sublimating source, the upper limit of the concentration of a dopant in them is limited by its concentration in a single crystal of Ge produced by industry ($1 \cdot 10^{19}\,\mathrm{cm}^{-3}$). However, Ge/Si(001) layers with a higher dopant concentration are sometimes needed. Therefore, we also applied in this study the method of evaporation of a dopant from a melt formed on a Ge source. The high density of the dopant flux from the melt in the germanium source should increase its concentration in Ge EL.

The purpose of this work is to study the process of doping by transferring a dopant (Ga and Sb) from a sublimating Ge source to a Ge/Si(001) EL in case of growth by the HW CVD method, as well as by evaporation of a dopant from a melt formed on a Ge source to increase the dopant flux density and an increase in the dopant concentration in the EL.

2. Experiment procedure

The doped Ge EL were grown using HW CVD method in an ultrahigh vacuum unit with a base pressure of $\sim 5 \cdot 10^{-9}$ Torr using a procedure similar to that described in [20]. Ge sources were cut out in the form of blocks with a size of $50 \times 5 \times 1\,\mathrm{mm}^3$ from ingots of monocrystalline Ge doped with Ga or Sb atoms. Such a source was placed on current leads cooled by running water and heated by passing current to a temperature of $\sim 900^\circ$C, which was controlled using an optical pyrometer.

In parallel to the Ge source, a Ta strip with a size of $70 \times 5 \times 0.1\,\mathrm{mm}^3$ was secured on another pair of current leads, which performed the function of a „hot wire“ for the growth of Ge EL layers by the HW CVD method. The third pair of current leads was used to attach a Si-sublimating source to it, from which a Si buffer layer was grown on a Si(001) substrate. All three sources were secured on one flange of the Du-160 growth chamber. A heater and a substrate holder with Si(001) plate were mounted on the same flange on the opposite side.

Si(001) substrate was annealed at a temperature of $\sim 1000^\circ$C for 10 minutes before the deposition of the layers for desorbing the oxide layer from its surface. Then the substrate temperature was reduced to $\sim 700^\circ$C and a silicon buffer layer with a thickness of $\sim 0.1\,\mu$m was grown from a sublimating Si source to heal substrate defects. After that, the substrate temperature was reduced to $\sim 350^\circ$C, the wire was heated to a temperature of $T_{\mathrm{Ta}} = 1300-1500^\circ$C, monogermane ($GeH_4$) was injected into the growth chamber to a pressure of $(4-9) \cdot 10^{-4}$ Torr, a sublimating Ge source was heated to a temperature of $\sim 900^\circ$C, a gate was opened separating the substrate from the sources, and the growth of the layer was carried out.

The substrate temperate ranged from 300 to $\sim 500^\circ$C to study the dependence of the dopant concentration in the Ge/Si(001) EL on the substrate temperature. The concentration of charge carriers in the Ge/Si(001) EL was measured by *C-V*-profilometry using LCR E7-12 meter at a frequency of 1 MHz. In some cases, the concentration of charge carriers and their mobility in doped Ge EL were determined using the Hall effect method. The structural perfection of Ge/Si(001) EL was studied by X-ray diffraction (XRD) using Bruker D8 DISCOVER diffractometer (Germany). The quantitative measure of the crystal structure of the Ge/Si(001) layers was the full width of half maximum (FWHM) of intensity of the X-ray peak from the Ge(004) layer. The morphology of the surface of the Ge/Si(001) EL was examined using atomic force microscopy (AFM).

3. Experimental results and discussion

3.1. Transfer of a dopant from a sublimating Ge source to the epitaxial layer

The following are the established quantitative patterns of transfer of dopant (Ga and Sb) from the Ge source to the Ge layers when they are grown using the HW CVD method on Si(001) substrates.

The EL doping efficiency is usually quantified using the coefficient of transfer of the dopant atoms:

$$k_n = \frac{N}{N_{so}}, \tag{1}$$

where N — dopant concentration in the EL, N_{so} — dopant concentration in the source.

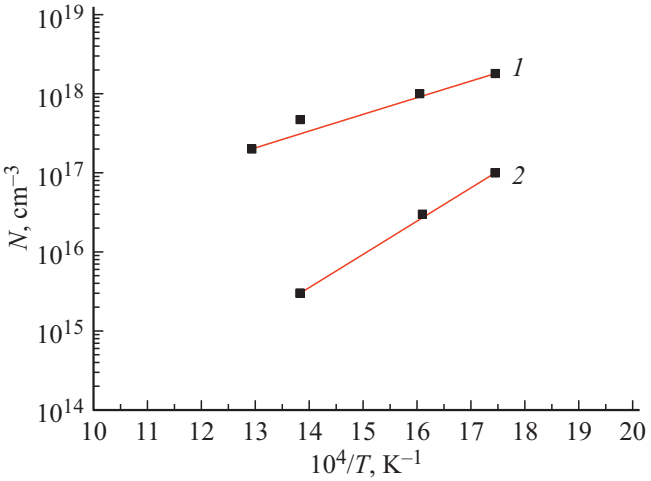

Figure 1. Dependence of the concentration of charge carriers in the Ge/Si(001) EL on the inverse temperature of the substrate. The Ge source is doped with: *1* —gallium, *2* — antimony.

We found that the coefficient of transfer of Ga or Sb atoms from Ge sources doped with these dopants to Ge EL strongly depends on the growth temperature and growth rate, as well as on the concentration of the dopant in the source.

The dependence of the concentration of holes (Ga) in Ge EL on the growth temperature is shown in Figure 1 (curve *1*). The sources were cut from single crystals Ge with gallium concentration of $\sim 1 \cdot 10^{19}$ cm^{-3}. There is also a similar dependence of the concentration of charge carriers on the growth temperature in Ge EL doped with antimony (curve *2*). The initial concentration of Sb in the Ge source was also equal to $\sim 1 \cdot 10^{19}$ cm^{-3}. As expected, in both cases, the dependence of the concentration of charge carriers in EL on the inverse growth temperature is exponential. The values of the activation energy (E_a) of doping, calculated based on the slopes of the curves *1* and *2* in Figure 1, and the transfer coefficients of these dopants at $T_S = 350$°C are given in Table 1. Table 1 shows that the value of the activation energy of Sb is higher than the activation energy of Ga, and the transfer coefficient, on the contrary, is higher in case of doping Ge EL with gallium than in case of doping with antimony.

Our study showed that the Ge layers doped with gallium atoms were of the *p*-type of conductivity. At the same time, it turned out that the Ge layers grown using a source doped with Sb atoms (*n*-type) also turned out to be of *p*-type of conductivity. A number of researchers explain the presence of hole conductivity in deposited germanium layers by the impact of: 1) structural defects, 2) surface states, 3) dopants [22]. Moreover, the results of the study quite convincingly indicate in favor of the assumption of the predominant influence of the dopant on the properties of condensed germanium layers.

Table 1. Energy of activation of the EL doping process and the coefficients of transfer of Ga and Sb atoms from Ge sources to Ge/Si(001) EL

Element	E_a, kcal/mol	$K_{tr} = N_{la}/N_{so}$ at $T = 350$°C
Ga	16.6	0.18
Sb	35.6	0.09

In a number of cases, it is possible to compensate for the acceptor levels formed under normal conditions. Germanium layers were doped with dopants As, P and Sb in [23]. In the first two cases, it was possible to obtain layers of the *n*-type of conductivity, whereas the admixture of Sb did not provide such an effect. The authors explain this by the different solubility of these dopants in Ge: at the melting point, the solubility of phosphorus exceeded and solubility of arsenic was equal to 10^{19} cm^{-3}, whereas the solubility of Sb was only $5 \cdot 10^{17}$ cm^{-3}. Apparently, it is necessary to investigate in our future experiments the doping of Ge/Si(001) layers by sublimation of a Ge source with an dopant As.

At the same time, the unsuccessful use of a germanium source doped with antimony in our experiments could be explained by an insufficiently intense flow of this dopant from the source. Perhaps it was not sufficient to compensate for the *p*-type of conductivity, which is always observed in case of the growth of Ge/Si(001) heteroepitaxial layers.

We also studied the dependence of the concentration of holes in Ge EL doped with Ga atoms on the growth rate. For this study the growth was carried out at a constant temperature of Ge:Ga source of $T_{Ga} = 900$°C, and the temperature of the „hot wire“ was decreased from 1500 to 1400°C (to reduce the flow of Ga), while maintaining constant substrate temperature of $T_S = 350$°C. As a result, it was found that the concentration of holes in the GeGa/Si(001) EL increased from $6.5 \cdot 10^{16}$ to $1.5 \cdot 10^{17}$ cm^{-3}.

Figure 2 shows the distribution profile of the concentration of charge carriers (holes) over the depth of the epitaxial layer of Ge:Ga/Si(001). This layer with a thickness of $\sim 1\,\mu$m was grown at the temperature of the Si substrate of $T_S = 350$°C, temperature of „hot wire“ of $T_{Ta} = 1400$°C, pressure of GeH_4 $p = 6 \cdot 10^{-4}$ Torr and growth rate of $V_p = 0.6\,\mu$m/hour. The source of the Ga dopant was cut from a Ge ingot with a concentration of $1 \cdot 10^{19}$ cm^{-3}. The source was pre-annealed at $T = 850$°C for 30 min before the growth of the layers to stabilize the dopant flux. It can be seen from Figure 2 that the distribution of the concentration of charge carriers over the thickness of the layer is uniform. This was facilitated, apparently, on the one hand, by the presence of atomic hydrogen on the growth surface. Atomic hydrogen is released on the „hot wire“ in case of monogermane dissociation (GeH_4). It acts as a surfactant once on the surface of the growing layer and helps to suppress the surface segregation of the dopant [24]. On the other hand, the homogeneous distribution of charge

Table 2. Technological and structural parameters of Ge and Ge : Ga layers grown on Si(001)

№	Type of structure	T_S, °C	d, μm	XRD, FWHM, deg
1	Ge/Si(001)	300	2.7	0.13
2	Ge/Si(001)	300	2.0	0.14
3	Ge/Si(001)	300	1.9	0.13
4	Ge : Ga/Si(001)	300	1.5	0.11
5	Ge : Ga/Si(001)	400	1.6	0.064
6	Ge : Ga/Si(001)	450	1.9	0.063
7	Ge : Ga/Si(001)	500	1.4	0.048

Note. T_S — substrate temperature during layer growth, °C; d — EL thickness, μm; FWHM, deg — rocking curve full width at half maximum of Ge(004).

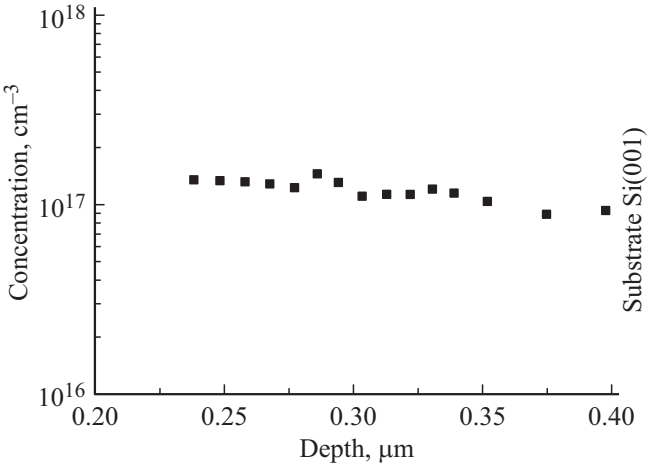

Figure 2. Distribution of the concentration of charge carriers over the thickness of the gallium-doped Ge layer (GDG-0.003).

carriers over the thickness of the layer was also facilitated by the fact that the dopant flux from the Ge source became stationary after its pre-annealing.

3.2. Structural perfection of Ge/Si(001) doped by dopant transfer

Table 2 shows the parameters of the structural perfection of Ge : Ga layers grown on a Si(001) substrate measured using XRD method. For comparison, FWHM parameter data for undoped Ge/Si(001) layers are additionally provided there. As can be seen from Table 2, the Ge/Si(001) layers doped with gallium atoms are more structurally perfect than the undoped ones: the value of the FWHM parameter is lower than that of the undoped Ge/Si(001) layers. Better structural perfection can explained taking into account that the Ga atoms in case of heteroepitaxial growth act as a surfactants [25] and facilitate the layered growth.

The morphology of the surface of the Ge : Ga layers is quite smooth: according to AFM data, their RMS value was < 1 nm on the scan area of $10 \times 10\,\mu m^2$. A slight increase of roughness was observed only in the layer grown at elevated temperature (500°C).

3.3. Impurity transfer from a partially molten Ge source to Ge EL

We developed a method for forming an increased dopant flux density from a Ge source to grow thin ($\leq 0.5\,\mu$m) strongly doped germanium layers that can be used as contact areas in instrument structures. It is possible to increase the dopant flux density by increasing its diffusion coefficient in the Ge source. Thus, the diffusion coefficient of a number of volatile dopants (including Ga, Sb) in a semiconductor increases by several orders of magnitude in case of transition of Ge source from a solid state to a melt compared with the diffusion coefficient in solid state [26].

It is necessary to increase the amount of current passing through the Ge source until a section of melt appears on its surface to form a melt zone on a sublimating source. At the same time, germanium usually melted along the longitudinal centerline of the source from the attachment point on one current lead to the attachment point on the other. As a result, the melt zone was located in the central part of the Ge source, and the rest of it preserved a solid state. A dopant with an increased flux density evaporated from this melt zone.

In case of evaporation of gallium from a Ge source with a formed melt zone, it was possible to increase the concentration of this dopant in the Ge by almost an order of magnitude compared with the concentration in the layer obtained by evaporation in the sublimation mode. The maximum concentration of holes in Ge : Ga/Si(001) layers grown by evaporation from the melt was $\sim 8 \cdot 10^{19}$ cm^{-3}. This result is in quite consistent with the data of [27], that reported about successful doping of Ge EL with Ga atoms during evaporation from a germanium melt strongly doped with Ga atoms (Ge source GDG-0.0006).

Thus, growing of Ge : Ga/Si(001) epitaxial layers doped with gallium atoms were grown using HW CVD method *in situ* using evaporation of Ga from a germanium source by sublimation or evaporation from a melt allowed for achieving a hole concentration in the range between $2 \cdot 10^{17}$ and $8 \cdot 10^{19}$ cm^{-3} in them. This allows using these Ge/Si(001) EL data in the creation of a number of micro- and optoelectronics instruments.

4. Conclusion

The structural and electrical properties of Ge/Si(001) EL grown by low-temperature ($\sim 350^\circ$C) HW CVD method and doped with various dopants (Ga and Sb), which evaporated from a Ge sublimating source, were studied in this paper. The source was cut in the form of a rectangular plate from an ingot of monocrystalline germanium doped with a given dopant, and heated to a temperature close to the melting point Ge (sublimation mode) or above this temperature (evaporation from the melt).

Quantitative patterns of transfer of dopant (Ga and Sb) from a germanium sublimating source to the Ge/Si(001) layers were studied. It was found that the transfer coefficient of the dopant strongly depends on the temperature and growth rate, as well as on the concentration of the dopant in the source. The values of the dopant activation energy, which were 35.6 kcal/mol for Sb and 16.6 kcal/mol for Ga, were determined using the exponential dependence of the concentration of charge carriers in Ge EL on the inverse temperature of the substrate.

It was also found that the Ge EL grown using the HW CVD method using a source doped with a donor (Sb), contrary to expectations, had a *p*-type of conductivity, which is probably due to the insufficiently high flux density of this dopant from a sublimating Ge source.

Maintaining the steady-state flow of (Ga) from a sublimating Ge source by pre-annealing ensured a homogeneous dopant distribution over the layer thickness in the growing Ge/Si(001) EL, as shown by measurement of the concentration of charge carriers in the layer using *C-V*-profilometry.

The uniform distribution of the concentration of charge carriers over the EL thickness apparently was also facilitated by the presence of atomic hydrogen on the surface of the growth (a product of thermal decomposition of monogermane), which, as a surfactant, suppresses the surface segregation of the dopant.

XRD study of the structural perfection of the doped Ge : Ga/Si(001) EL showed that it is higher than the structural perfection of the undoped layers, which is probably explained by the action of gallium atoms as a surfactant.

It was also found that the evaporation of dopant atoms (Ga) from a Ge source with a formed melt zone on it allows for increasing the dopant concentration in Ge EL by almost an order of magnitude compared with their concentration in Ge EL doped *in situ* by evaporation of dopant (gallium) from a sublimation source. The maximum concentration of holes in such layers was $\sim 8 \cdot 10^{19}$ cm^{-3}.

In general, Ge layers of *p*-type with a concentration of charge carriers (holes) from $2 \cdot 10^{17}$ to $8 \cdot 10^{19}$cm^{-3} can be obtained by doping Ge EL in the HW CVD process by evaporation of a dopant from a sublimating source (from the solid phase) and by evaporation from the melt. This allows using such Ge : Ga/Si(001) EL for production of *p*-MOS transistors and *p-i-n* photodetectors.

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Conflict of interest

The authors declare that they have no conflict of interest.

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