

Features of Galvanomagnetic Effects in n -Ge Crystals

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It was shown that in weakly compensated oxygen-containing n -Ge crystals, the oxygen or oxygen-impurity complexes appearing during heat treatment play the role of statistically distributed inhomogeneities and cause an increase in the transverse magnetoresistance in the regions of classically strong and quantizing magnetic fields. It was revealed that during the annealing of n -Ge with oxygen impurity, the formation of oxygen (or oxygen-impurity) complexes is limited by diffusion of impurity atoms (primarily, oxygen atoms). It was established that the changes in the transverse magnetoresistance resulted from the thermal annealing of the n -Ge samples (with different oxygen concentrations in their volume) in the same time interval, and also from a gradual increase in the time interval in which the same sample is annealed.

topics: germanium, oxygen impurity, thermal annealing, magnetoresistive effect

1. Introduction

Despite the long history of research on germanium and its various structures, they remain relevant due to the unique properties of the material itself and the expansion of its applications [1, 2].

The high carrier mobility in germanium makes it a promising material for further development of CMOS technologies [3]. Heterocomposites based on germanium and Ge-Si solid solutions are widely used to manufacture sensors and microsystems [4]. High-speed heterobipolar transistors are based on strained Ge-Si heterostructures with a combined lattice [5]. Investigation of parameters and improvement of technologies, as well as the creation of highly doped and perfect germanium epitaxial layers, open new possibilities in the design of nanoelectronic and plasmonic devices with active elements in the terahertz (THz) spectral range [6].

High purity germanium is used for the production of ionizing radiation detectors, highly efficient and radiation-resistant photoelectric converters in satellite on-board power supply based on $A_{III}-B_V$ epitaxial compounds on Ge (GaInP/GaInAs/Ge) substrates [2, 7].

The two transparency windows of the atmosphere (3–5 μm and 8–14 μm) overlap well with the region of maximum Ge transmission. Moreover, Ge has a transparency window in the terahertz spectral range (~ 100 –300 μm). Such physicochemical properties make crystalline germanium one of the basic materials for the development of the infrared (IR) photonics [8].

It should be noted that even though the magnetoresistive effect has been studied in germanium for a long time [9, 10], the interest in this phenomenon has increased significantly due to the development of spin electronics, because changing the material's resistance by manipulating electron spins with external magnetic and electric fields is of decisive importance here [11–13].

In almost all applications, the influence of structural inhomogeneities and point defects in germanium-based materials is significant. For example, dislocation-free germanium substrates are required to create modern, efficient photovoltaic converters using GaInP/GaInAs epitaxial compounds [7], while space charge inhomogeneities in the contact regions of In/Ge/In heterostructures cause high magnetoresistance at room temperatures [14].

Studies of the optical characteristics of materials based on germanium for IR photonics indicate that an increase in the temperature stability of optical characteristics is observed after the heat treatment at temperatures 573–673 K. Such changes are explained by the transformation of defects associated with the presence of oxygen impurity in the source material during heat treatment [15].

Oxygen is one of germanium's main uncontrolled (weakly controlled) impurities. Passing from the atomically dispersed (electrically passive) state to the electrically active state, which is typical for thermal annealing of crystals, the oxygen impurity in Ge forms donor-type oxygen-containing complexes (thermal donors) with different depths

in the bandgap [16, 17]. The relevance of research on the manifestation of the effects associated with the presence of oxygen remains to this day due to the development of new technologies for obtaining materials [18, 19] and their subsequent heat treatment [7, 15].

The interrelationship of electrophysical and optical parameters of semiconductor materials allows, for example, to test materials for IR photonics and to optimize the methods for their production and processing using the results of measurements of electrical parameters [8].

Herring [20] theoretically dealt with the question of the effect of inhomogeneities statistically distributed over the volume of a crystal on the electrical properties of semiconductor materials. The so-called Herring's inhomogeneities are defects whose dimensions are much smaller than the geometric dimensions of the crystal but exceed various characteristic lengths (Debye length, diffusion bias length, etc.), which under certain conditions can cause noticeable fluctuations in macroscopic conductivity. Estimates, based on investigation for oxygen-enriched Ge in annealing at 573–723 K by infrared absorption spectroscopy, indicated that the thermal donor complex involves 16–19 oxygen atoms [21].

Assuming that oxygen-containing complexes (in the active electric state) will appear in the volume of thermally treated oxygen-containing *n*-Ge crystals, playing the role of Herring's statistically distributed inhomogeneities, the purpose of this work was to study the effect of heat treatment of *n*-Ge crystals with different oxygen content on the magnetoresistance phenomenon in the region of classically strong and quantizing magnetic fields.

2. Experimental details

A set of samples of *n*-Ge single crystals grown by the Czochralski method with different oxygen content, but approximately the same electrically active impurity concentration, was chosen for research. Overall, in terms of electrophysical characteristics,

the samples studied in this work were similar to the materials used in IR optics and studied in [8, 15]. All the *n*-Ge samples were doped with antimony (Sb), widely used as a well-controlled donor impurity in germanium [6, 8, 15]. For uncompensated samples, the concentration of the dopant (Sb) was assumed to be equal to the concentration of free carriers n_e (at 77 K) as determined by the standard Hall method. The set included uncompensated crystals (more precisely, initially weakly compensated, as their volume could contain an insignificant fraction of the acceptor-type background impurities) and compensated samples with different degrees of compensation.

Experimental determinations of the oxygen content in the atomically dispersed (electrically passive) state $[O_i]$ were carried out on an IFS-113v Fourier spectrometer using the conventional technique of Fourier transform IR spectrometry [15, 22].

The pulsed magnetic field was generated by discharging an 1800 μ F capacitor onto a beryllium bronze solenoid (with an inner diameter of 8 mm and a length of 40 mm) similar to those described in [23, 24]. For measurements at 77 K, the sample was mounted in a glass cryostat and placed inside the solenoid.

Measurements of the intensity of the pulsed magnetic field were carried out with the widely used technique [25] with the use of a measuring coil placed inside the solenoid. The measured signal values were calculated at the points of the magnetic field extremum ($dH/dt = 0$). The measurements were carried out for two (opposite) directions of the magnetic field and the sample current.

A digital current generator (Keithley-224) and multimeters (Keithley-2000) were used to measure n_e by Hall's method. The resistivity was determined from the value of the voltage between the ohmic contacts located along the sample, to the end contacts of which the stabilized direct current was applied. A digital storage oscilloscope was used to measure the amplitude and shape of the pulse signals. The digitized data was transferred to a computer for visualization and further processing.

TABLE I

Main parameters of the studied weakly compensated *n*-Ge samples with oxygen impurity. Note: the initial concentration of charge carriers $n_e \cong (2-5) \times 10^{14} \text{ cm}^{-3}$ corresponds to the electrically active doping impurity (Sb) concentration.

Sample	Stage	N_{O_i} ($\times 10^{-16}$) [cm^{-3}]	N_D ($\times 10^{-4}$) [cm^{-2}]	$T = 77 \text{ K}$					$T = 300 \text{ K}$		
				n_e ($\times 10^{-14}$) [cm^{-3}]	μ ($\times 10^{-4}$) [$\text{cm}^2/(\text{V s})$]	ρ [$\Omega \text{ cm}$]	K_{exper}	K_{theor}	n_e ($\times 10^{-14}$) [cm^{-3}]	μ ($\times 10^{-3}$) [$\text{cm}^2/(\text{V s})$]	ρ [$\Omega \text{ cm}$]
A.1	before	1	4	2.68	2.59	0.72	–	–	2.62	3.66	5.90
	after	–	–	2.66	2.54	0.73	15.9	14.1	2.61	3.67	5.95
A.2	before	9	1.5	3.68	2.54	0.55	–	–	3.55	3.72	4.25
	after	–	–	25.1	1.39	0.16	12.4	10.5	34.1	2.59	0.61
A.3	before	11	0.27	4.29	2.66	0.46	–	–	4.15	3.95	3.42
	after	–	–	35.4	1.39	0.11	10.8	9.8	50.7	3.15	0.33

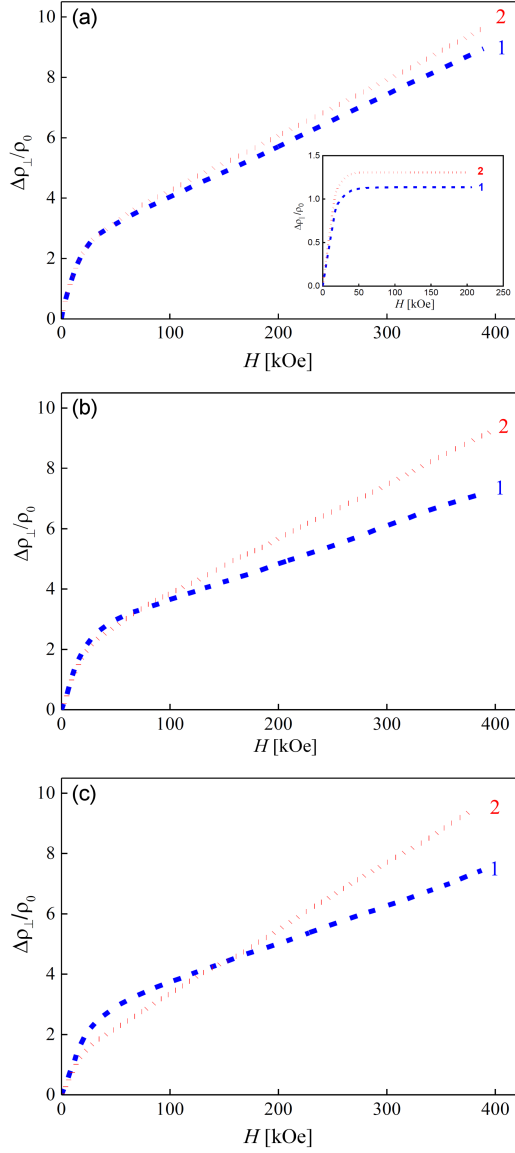


Fig. 1. Field dependences $\Delta\rho_H^\perp/\rho_0 \equiv \Delta\rho_\perp/\rho_0 = f(H)$ for the *n*-Ge samples (at $\mathbf{j} \parallel [111]$, $\mathbf{H} \parallel [110]$, $T = 77$ K) with different oxygen contents: curves 1 — initial samples; 2 — annealed at $T_{\text{ann}} = 623$ K during $t_{\text{ann}} = 60$ h. Sample numbers: (a) A.1, (b) A.2, (c) A.3 (see Table I). Insert shows field dependences $\Delta\rho_H^\parallel/\rho_0 = f(H)$ for the sample number A.1 at $\mathbf{j} \parallel \mathbf{H} \parallel [111]$, $T = 77$ K.

The field dependences of magnetoresistance $\Delta\rho/\rho_0$ were measured in a wide range of magnetic fields H (up to 400 kOe) at the sample temperatures $T = 77$ K before and after various thermal annealing in an argon atmosphere at a temperature $T_{\text{ann}} = 623$ K.

The magnetoresistance of a material characterizes the change in its resistance ρ when the magnetic field H is applied. The values of magnetoresistance $\Delta\rho/\rho_0$ is determined by the formula $\Delta\rho/\rho_0 = (\rho_H - \rho_0)/\rho_0$, where ρ_0 is the resistance of the sample in a magnetic field $H = 0$, ρ_H is the resistance

of the sample in a magnetic field $H \neq 0$. The field dependences of the longitudinal magnetoresistance $\Delta\rho_H^\parallel/\rho_0$ (at $\mathbf{j} \parallel \mathbf{H}$) and at the transverse magnetoresistance $\Delta\rho_H^\perp/\rho_0$ (at $\mathbf{j} \perp \mathbf{H}$) were studied.

3. Magnetoresistance of uncompensated (weakly compensated) *n*-Ge crystals before/after annealing at $T_{\text{ann}} = 623$ K

In the first series of experiments, samples with approximately the same donor concentration and different oxygen concentrations were annealed in an argon atmosphere at a temperature $T_{\text{ann}} = 623$ K for $t_{\text{ann}} = 60$ h. The parameters of the *n*-Ge samples are presented in Table I, where N_{O_i} is the concentration of atomic dispersed (electrically passive) oxygen $[\text{O}_i]$, N_D is the density of dislocations, n_e is the concentration of free charge carriers, μ is the mobility of carriers, ρ is the sample resistivity, K_{exper} and K_{theor} are, respectively, the experimental and theoretical values of the mobility anisotropy parameter $K = \mu_\perp/\mu_\parallel$. The values of K_{theor} can be obtained in the frame of the theory of anisotropy scattering [10]. The anisotropy parameter K_{exper} can be calculated using the saturation value $\Delta\rho_H^\parallel/\rho_0$ [10], for example $\frac{\Delta\rho_H^\parallel}{\rho_0} \Big|_{\text{sat}}^{[111]} = \frac{(2K+1)(K+8)}{3(7K+2)} - 1$ at $\mathbf{H} \parallel [111]$.

In the second series of experiments, the monocrystalline *n*-Ge samples with the carrier concentration $n_{e77\text{K}} = 5.2 \times 10^{14} \text{ cm}^{-3}$ and the initial oxygen concentration $N_{\text{O}_i} = 1.1 \times 10^{17} \text{ cm}^{-3}$ were annealed during different annealing intervals at a constant temperature $T_{\text{ann}} = 623$ K.

The field dependences of magnetoresistance at 77 K for the uncompensated (or weakly compensated) oxygen-containing *n*-Ge samples (before and after thermal annealing) are shown in Figs. 1 and 2.

As a result, several experimental facts were obtained indicating that the oxygen (or oxygen-impurity) complexes formed during thermal annealing of oxygen-containing *n*-Ge manifest themselves as Herring inhomogeneities. These facts include the following:

1. an intensification of changes $\Delta\rho_H^\perp/\rho_0$ in annealed crystals corresponds to a sequential increase in oxygen concentration in their volume (Fig. 1, $a \rightarrow b \rightarrow c$) or an increase of the annealing time;
2. the calculated experimental values of the mobility anisotropy parameter K_{exper} exceed the value K_{theor} in annealed crystals (see Table I);
3. there is a linearity of changes $\Delta\rho_H^\perp/\rho_0$ with H (which is superimposed on a similar dependence for the orientation $\mathbf{H} \parallel [110]$, caused by quantization effects at $H > 100$ kOe [9]).

Indeed, regardless of whether the oxygen or oxygen-impurity complexes are formed during low-temperature ($T_{\text{ann}} = 573\text{--}673$ K) annealing of *n*-Ge with an oxygen impurity, the process of their

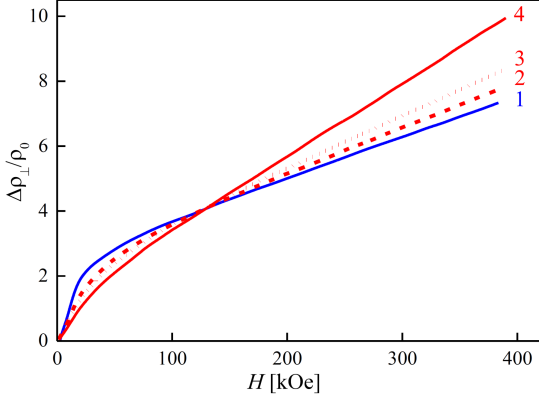


Fig. 2. Dependences $\Delta\rho_{\perp}^{\perp}/\rho_0 = f(H)$ for the n -Ge sample (at $\mathbf{j} \parallel [111]$, $\mathbf{H} \parallel [110]$, $T = 77$ K) with the initial oxygen concentration $N_{O_i} = 1.1 \times 10^{17} \text{ cm}^{-3}$: 1 — initial sample, $n_{e 77\text{K}} = 5.2 \times 10^{14} \text{ cm}^{-3}$, and for different annealing time intervals at $T_{\text{ann}} = 623$ K (t_{ann} [h], $n_{e 77\text{K}}$ [cm^{-3}]): 2 — 20, 1.63×10^{15} ; 3 — 40, 2.7×10^{15} ; 4 — 60, 4.08×10^{15} .

formation will be limited by the diffusion of impurity atoms (primarily, of oxygen atoms). However, under such conditions, the nature of changes in $\Delta\rho_{\perp}^{\perp}/\rho_0$ that arise as a result of thermal annealing of n -Ge (with different oxygen concentration in its volume) for the same time (Fig. 1), as well as a result of a gradual increase in the time interval in which the same sample is annealed (Fig. 2), are qualitatively different. Comparing the data shown in Fig. 1 and Fig. 2, it can be stated that the revealed shift in the position of the intersection of the curves 1 and 2 along the H axis with a change in the oxygen concentration in the crystals (see Fig. 1) in the second case (see Fig. 2) changes to a “fan” of curves intersecting practically in one point. As is known, the diffusion process at a given thermal annealing temperature depends differently on the thermal annealing time and the interatomic distances, which in turn are determined by the concentrations of the impurity atoms. This circumstance manifests itself in the considered experiments.

4. Magnetoresistance of highly compensated n -Ge crystals with an oxygen impurity in the passive and electrically active states

In n -type semiconductors, the compensation degree k is determined as $k = N_a/N_d$, where N_a and N_d are the corresponding concentrations of acceptors and donors. The compensation degree k can be evaluated using the experimental values of the Hall mobility μ_H and the Hall coefficient R_H and the theoretical dependence of the Hall mobility μ_H and the hall-factor $r = \mu_H/\mu$ on the concentration of ionized impurities $N_i = N_d + N_a$ [10, 26]. By experimentally determined μ_H and using the theoretically calculated dependence $\mu_H(N_i)$ we can evaluate the value of N_i , and after this by the

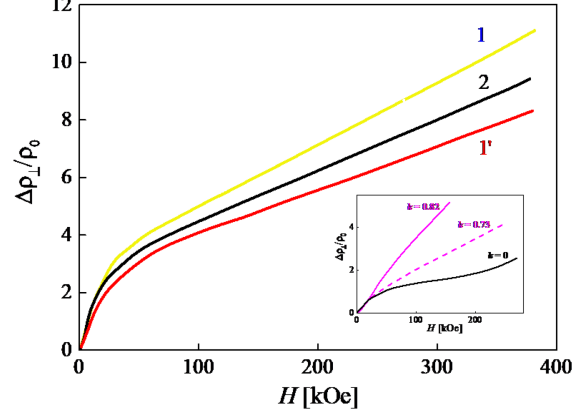


Fig. 3. Field dependences $\Delta\rho_{\perp}^{\perp}/\rho_0 = f(H)$ at $\mathbf{j} \parallel [111]$, $\mathbf{H} \parallel [110]$, $T = 77$ K for the n -Ge samples B.1 and B.2 (see Table II): 1 — initial sample B.1; 1' — annealed sample B.1; 2 — sample B.2 (uncompensated). Inset shows field dependences $\Delta\rho_{\perp}^{\perp}/\rho_0 = f(H)$ for the n -Ge crystals (at $\mathbf{j} \parallel [100]$, $\mathbf{H} \parallel [010]$, $T = 77$ K) with different compensation degree $k = N_a/N_d$.

theoretically calculated dependence $r(N_i)$ [10] we can obtain the corresponding values of the hall-factor $r(N_i)$ and the carrier concentration $n_e = r(N_i)/(ceR_H)$. Taking into account that $N_i = N_d + N_a$ and $n_e = N_d - N_a$, the value of the degree of compensation is obtained using

$$k = \frac{N_a}{N_d} = \frac{N_i - n_e}{N_i + n_e}. \quad (1)$$

At sufficiently high compensation levels of n -Ge crystals ($k = N_a/N_d$), the transverse magnetoresistance increases significantly and becomes a linear function of H (Fig. 3, inset) in a strong and quantizing magnetic field ($H > 100$ kOe, $T = 77$ K). The orientation selection $\mathbf{j} \parallel [100]$, $\mathbf{H} \parallel [010]$, in this case, made it possible to identify changes in the values of $\Delta\rho_{\perp}^{\perp}/\rho_0$ with an increase in the level of compensation, as well as the presence of the straightening effect of the dependence $\Delta\rho_{\perp}^{\perp}/\rho_0 = f(H)$. According to [9], this dependence for the chosen orientations \mathbf{j} and \mathbf{H} in quantizing magnetic fields cannot be linear when compensation is absent.

It can be assumed that annealing of the compensated (in the initial state) n -Ge crystals with oxygen impurity will lead to the decrease in their compensation degree due to the generation of electrically active donor centers. Taking into account the data in Fig. 3 (inset), the decrease in the compensation degree will lead to a decline in the slope of the field dependence of the transverse magnetoresistance $\Delta\rho_{\perp}^{\perp}/\rho_0 = f(H)$ (and the absolute values of $\Delta\rho_{\perp}^{\perp}/\rho_0$). Such a decrease may be more effective than the increase in the slope of the dependence $\Delta\rho_{\perp}^{\perp}/\rho_0 = f(H)$, associated with the formation of complexes upon an annealing of oxygen-containing n -Ge in the absence of compensation.

TABLE II

 Initial parameters of *n*-Ge crystals ($T = 77$ K) and their changes after heat treatment ($T_{\text{ann}} = 623$ K, $t_{\text{ann}} = 60$ h).

Sample	Impurity [cm^{-3}]	Treatment	n_e ($\times 10^{-13}$) [cm^{-3}]	μ ($\times 10^{-3}$) [$\text{cm}^2/(\text{V s})$]	ρ [$\Omega \text{ cm}$]
B.1	$N_{\text{O}_i} = 8 \times 10^{16}$	initial (curve 1)	3.43	30.9	5.90 (compensated)
	$N_{\text{Sb}} = 3.43 \times 10^{13}$	after annealing (curve 1')	41.7	24.6	0.61 (compensated less)
B.2	$N_{\text{Sb}} = 2.14 \times 10^{13}$	initial (curve 2)	2.14	31.4	9.30 (uncompensated)

With the decisive role of the decrease in the compensation degree during annealing of the compensated (in the initial state) *n*-Ge crystals with oxygen impurity, the slope of the dependence $\Delta\rho_H^\perp/\rho_0 = f(H)$, and the value of $\Delta\rho_H^\perp/\rho_0$ (at a given value of H) should be lower than that of in the initial (unannealed) crystals, which is reflected in the experimental results (Fig. 3, curves 1 and 1') obtained for the region of strong magnetic fields.

Defining the compensation degree by the technique described above in the low-doped region is complicated. To additionally make sure that the crystal B.1 (Table II) is initially compensated, it is helpful to compare the results of the dependence measurements of $\Delta\rho_H^\perp/\rho_0 = f(H)$ on it (see Fig. 3, curve 1) with a similar dependence obtained on the guaranteed uncompensated crystal (Fig. 3, curve 2). Taking into account the data in Fig. 3 (insert), which are typical for compensated crystals, it can be argued that the relative position of the curves 1 and 2 in Fig. 3 is reasonably reliable evidence that the sample B.1 (with oxygen impurity) was indeed compensated in the initial state. Note that the compensation significantly influences the parameters of the IR optic [8], and such investigation can be helpful in detecting the compensation in the initial germanium material.

5. Conclusions

The effect of oxygen impurity in neutral and electrically active states on the transverse magnetoresistance of *n*-Ge with different compensation degrees was studied in classically strong and quantizing magnetic fields.

It was shown that heat treatment of weakly compensated *n*-Ge crystals with an oxygen impurity promotes the appearance of oxygen and oxygen-impurity complexes, manifested themselves as the Herring inhomogeneities, which increase the transverse magnetoresistance measured under the conditions $\mathbf{j} \parallel [111]$, $\mathbf{H} \parallel [110]$, $T = 77$ K in the region of classically strong ($\mu H/c \gg 1$) and quantizing ($H > 100$ kOe) magnetic fields.

It was revealed that in quantizing magnetic fields ($H > 100$ kOe, $T = 77$ K, $\mathbf{j} \parallel [100]$, $\mathbf{H} \parallel [010]$) the linear dependence of the transverse magnetoresistance on H is associated with the manifestation of the influence of the compensation of the *n*-Ge crystals.

It was found that the annealing ($T_{\text{ann}} = 623$ K) of weakly compensated *n*-Ge crystals (both with high and low oxygen content) leads to an increase in the slope of the field dependences of the transverse magnetoresistance in the region of classically strong and quantizing magnetic fields.

It has been shown that in compensated oxygen-containing crystals, after similar annealing, the increase in the slope of the field dependence of the transverse magnetoresistance can be replaced by a falling, as a result of a decrease in the compensation degree of the crystal due to the formation of donor-type oxygen complexes.

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