

Optical, electrophysical and structural properties of polycrystalline germanium grown by horizontal directional crystallization method

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Studied are the conditions for the obtaining of polycrystalline germanium (poly-Ge) of optical quality by the method of horizontal directional crystallization. Peculiarities of the formation of the structural and electrophysical characteristics, as well as their influence on the optical properties of the polycrystals, are investigated. It is established that poly-Ge ingots have inhomogeneous structure and texturing with preferential growth of crystallites along the crystallographic direction [111]. The grain sizes vary within a wide range from ~ a millimeter to several centimeters along and across the ingot. It is shown that, likewise in the case of Ge single crystals, the electrophysical and optical properties of poly-Ge are essentially defined by the concentration of free charge carriers, the structural defects and the charge state of the donor activator. Annealing at $T = 850\text{--}900^\circ\text{C}$ raises the value of optical transmission of both pure and Sb-doped germanium not less than by 1–2 % within the whole of the transparency range. There is obtained poly-Ge:Sb with the coefficient of light attenuation $\alpha \sim 0.04 \text{ cm}^{-1}$ (for $10.6 \mu\text{m}$ wavelength) and high homogeneity of the distribution of the characteristics along the ingot.

Keywords: Stibium doped germanium, horizontal directional crystallization method, optical absorption, resistivity, carrier concentration.

Оптичні, електрофізичні та структурні властивості полікристалічного германію, вирощеного методом горизонтальної спрямованої кристалізації. *С.В.Ніжанковський, Л.О.Гринь, А.А.Козловський, О.О.Вовк*

Досліджено умови одержання методом горизонтальної спрямованої кристалізації полікристалічного германію (полі-Ge) оптичної якості, особливості формування структурних, електрофізичних характеристик і їх вплив на оптичні властивості полікристалів. Встановлено, що злитки полі-Ge мають неоднорідну структуру і текстуровання з переважним ростом кристалітів вздовж кристалографічного напрямку [111]. Розміри зерен варіюються у широкому діапазоні від ~ міліметра до декількох сантиметрів вздовж і за товщиною злитка. Показано, що подібно до монокристалів Ge, електрофізичні і оптичні властивості полі-Ge значною мірою визначаються концентрацією вільних носіїв заряду, структурних дефектів і зарядовим станом донорного активатора. Відпал при $T = 850\text{--}900^\circ\text{C}$ приводить до збільшення оптичного пропускання як легованого, так і нелегованого стибієм (Sb) германію не менше, ніж на 1–2 % у всьому спектральному діапазоні прозорості. Отримано полі-Ge:Sb з коефіцієнтом ослаблення світла $\alpha \sim 0,04 \text{ см}^{-1}$ (для довжини хвилі $10,6 \text{ мкм}$) і високою однорідністю розподілу характеристик вздовж злитка.

Исследованы условия получения методом горизонтальной направленной кристаллизации поликристаллического германия (поли-Ge) оптического качества, особенности формирования структурных и электрофизических характеристик, их влияние на оптические свойства поликристаллов. Установлено, что слитки поли-Ge имеют неоднородную структуру и текстурирование с преимущественным ростом кристаллитов вдоль кристаллографического направления [11]. Размеры зерен варьируются в широком диапазоне от ~ миллиметра до нескольких сантиметров вдоль и по толщине слитка. Показано, что подобно монокристаллам Ge, электрофизические и оптические свойства поли-Ge в значительной степени определяются концентрацией свободных носителей заряда, структурных дефектов и зарядовым состоянием донорного активатора. Отжиг при $T = 850\text{--}900^\circ\text{C}$ приводит к увеличению оптического пропускания как легированного, так и нелегированного стибием (Sb) германия не менее, чем на 1–2 % во всем спектральном диапазоне прозрачности. Получен поли-Ge:Sb с коэффициентом ослабления света $\alpha \sim 0,04\text{ см}^{-1}$ (для длины волны 10,6 мкм) и высокой однородностью распределения характеристик вдоль слитка.

1. Introduction

Now a days IR devices are being more and more widely applied in many fields of science, technology, medicine, military hardware. One of the materials most often used for the making of various elements of IR devices (lenses, input windows, objectives of thermal imaging systems, components of IR spectroscopy, etc.) is crystalline germanium [1]. The main requirements for the crystals used in IR optics include low optical absorption in the IR spectral region 3–12 μm and high optical homogeneity [2]. Absorption of germanium in the transmission region is defined by several processes, in particular, lattice absorption, as well as the one caused by free charge carriers and by structure defects and impurities [3, 4]. The main absorption mechanism in germanium is realized by free charge carriers, i.e. holes and electrons [4] which concentration is defined by the impurity atoms of acceptor and donor type. Thereat, cross-section of hole absorption exceeds the electron one more than by an order [5] that leads to an essential rise of light absorption. Therefore, to reduce the absorption caused by background impurities of acceptor type, germanium is doped with a donor impurity, most often with stibium (Sb). For the making of optical elements there are used stibium-doped germanium single crystals (Ge:Sb) which electrical resistivity ranges from 5 to 40 Ohm-cm, and optical transmission in the working wavelength is ~ 46 % for 1 cm thick samples [2, 4].

For IR optics there are predominantly used germanium single crystals [2]. The chief technique of the growth of Ge single crystals is the Czochralski method [2, 6–8]. There has been also reported the obtaining of Ge by the Bridgman [9] and Stepanov

[10] methods. However, the growth of large Ge single crystals for large-size ($\geq 200\text{ mm}$) windows is rather inefficient and accompanied with certain difficulties. Therefore, now researchers consider the possibility to use polycrystalline germanium (poly-Ge) and to find more effective methods of its obtaining [11]. The use of poly-Ge is limited by increasing optical loss caused by grain boundaries, clusters of inclusions and impurities around them, high concentration of dislocations and considerable residual stresses [12]. Another factor that influences the optical quality of poly-Ge and limits its application, in particular, for fabrication of lenses, is its optical inhomogeneity. The latter arises in the polycrystal due to the presence of grain boundaries which are drains for background impurities and electrically neutral stibium [13], as well as non-uniform distribution of residual stresses and dislocations.

Nevertheless, poly-Ge can be effectively used for the making of a variety of one-panel and plane-parallel elements, IR-shielding windows of thermal imaging and optoelectronic systems for which the said parameters are not so critical [14]. Therefore, development of effective methods and technologies for the obtaining of polycrystalline germanium is a topical task. One of most promising methods for the growth of poly-Ge meant for the making of flat-parallel windows is horizontal directional crystallization (HDC). The main advantages of this method include closeness of the geometric parameters of the ingot to the size and shape of the final product, as well as high effectiveness of the use of the expensive raw material. At present the said method is being updated for the obtaining of poly-Ge of optical quality, in particular, by doping with stibium and sodium [13, 15]. However, the available results on the regularities of

Table 1. Electrophysical characteristics of raw material for germanium

Raw material	Parameters		
	Specific electrical resistance ρ , Ohm-cm	Concentration of charge carriers N , cm ⁻³	Type of conductivity
Germanium purified by zone melting	52.2±2.6	5.8·10 ¹⁴	n

the formation of structural, optical and electrophysical characteristics of poly-Ge depending on the conditions of crystallization, annealing and other factors, are insufficient for development of an efficient technology for the obtaining of poly-Ge with required parameters.

The goal of the present work was to study the conditions for the obtaining of polycrystalline germanium of optical quality by the method of HDC, as well as to investigate peculiarities of the formation of structural and electrophysical characteristics and their influence on the optical properties of the said polycrystals.

2. Experimental

Pure and Sb-doped germanium polycrystals were grown in HDC setups with a thermal unit made from carbon graphite constructional materials and resistive tungsten heater. The crystallization process was realized in argon medium under a pressure of 0.1 MPa, in crucibles made from isostatically pressed graphite with a purity of 99.99 % and the dimensions 30 mm×30 mm×100 mm and 30 mm×30 mm×200 mm. The temperature gradient ($gradT$) at the crystallization front was ≈ 20 °C/cm. The crystallization rate (v) varied from 15 mm/h to 50 mm/h, the melt overheating temperature (ΔT_{melt}) ranged between 80°C and 120°C.

The grain and dislocation structures of the ingots were investigated by the method of etching (using H₂O₂:HF:H₂O solution in the proportion 1:1:3) by means of a microscope MIK-4.

Their crystallographic orientation was studied by the method of two-crystal X-ray diffractometry in CuK β radiation.

The optical transmission was measured within the range from 1.28 to 22 μ m by means of an IR Fourier spectrometer of Perkin Elmer type (the measurement accuracy was ± 0.5 cm⁻¹). The samples for the measurements were cut out both longwise and crosswise of the crystal. Finishing polish of the surface of the samples was realized using ACM 5/3 diamond powder. The thickness of

all the samples was 5 mm. The latter approximately corresponds to the standard thickness of optical windows of IR optical devices. The attenuation coefficient α was calculated taking into account the reflection coefficient according to the formula [16]:

$$\alpha = \frac{1}{h} \left[\ln \frac{(1-R)^2 + ((1-R)^4 + 4\tau^2 R^2)^{0.5}}{2\tau} \right], \quad (1)$$

where h is the sample thickness; R , the reflection coefficient; τ , the transmission coefficient of the sample.

The value of specific electrical resistance of the grown germanium crystals was measured by the two-contact method [17] on the samples shaped as parallelepipeds with the dimensions 5 mm×5 mm×25 mm. The error of determination of specific electrical resistance did not exceed 10 %. The type of charge carriers was established from the sign of the Seebeck coefficient using a thermoprobe [18]. The concentration of the main charge carriers was determined by calculations based on the data of the measurements of the Hall coefficient [19]. In accordance with the existing standards, the value of specific electrical resistance and of the Hall coefficient were measured at 23°C [20].

3. Results and discussion

Germanium ingots were obtained by the method of HDC from polycrystalline germanium preliminarily subjected to zone purification. The purity of the said starting material was $\geq 99,999$ %. The doping was realized using stibium of the type Cy0000 (99.999 %) introduced into the raw material in the form of highly concentrated ligature Ge:Sb.

The purity was controlled by measuring the electro-physical characteristics of the starting material (Table 1).

The value of specific electrical resistance (ρ) of the purified germanium is close to the corresponding value for pure germanium [21]. However, the concentration of free charge carriers is by an order of magnitude higher [22]. The latter may testify to the presence of excessive qualities of back-

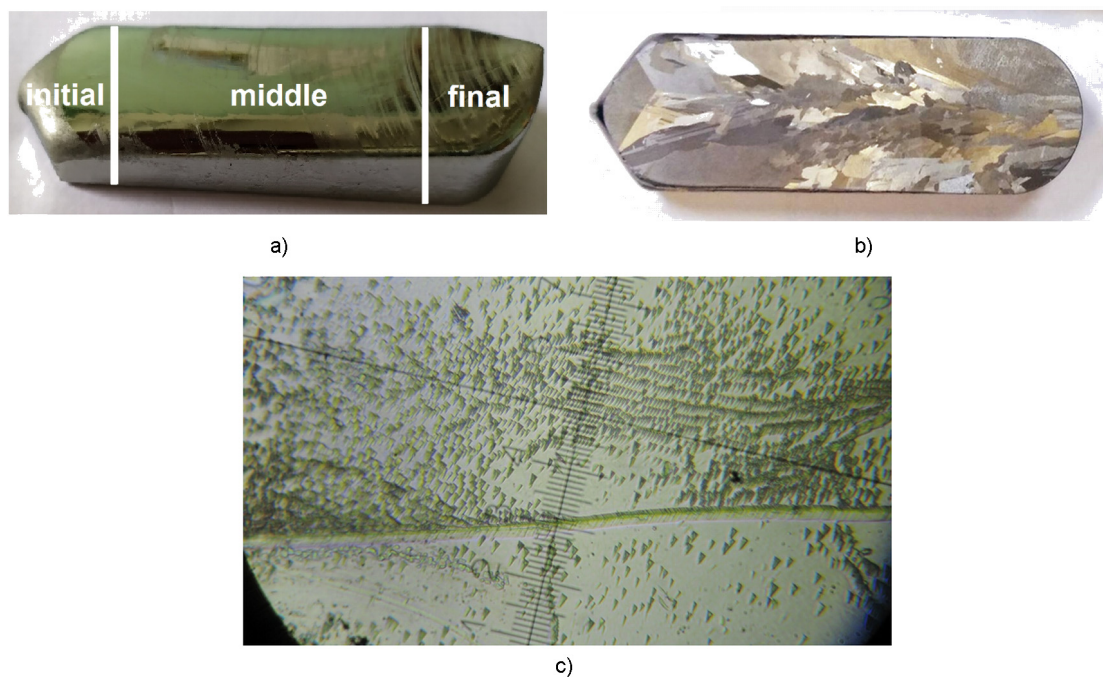


Fig. 1. Polycrystalline germanium grown by HDC method: a) external appearance; b) grain structure of poly-Ge; c) view of dislocation structure at the grain boundary.

ground impurities mainly of donor type (n type conductivity) in the starting material. Unlike germanium single crystals, the polycrystalline material does not show direct dependence of ρ on the concentration of free charge carriers [23]. This is due to the fact that in the polycrystals the value of ρ may be essentially influenced by the presence of intergranular boundaries, micro-pores, inclusions, aggregations of impurity atoms and inherent defects. Therefore, at a high charge carriers concentration which depends on the amount of impurities in germanium the value of specific resistance may increase (Table 1).

Fig. 1 presents the external appearance of the grown crystal with the dimensions 100 mm×30 mm×10–15 mm, as well as the grain and dislocation structures of the sample surface. Since the density of solid germanium is lower than the one of its melt, the thickness of initial region of the ingot is lesser than that of the final region (Fig.1a). As seen from the presence of growth steps on the ingot surface (Fig. 1a), the crystallization front is noticeably concave.

Examination of the etched surface of the ingots shows that they are polycrystals with clearly seen grain structure (Fig. 1b). The initial region of the ingots contains grains with a size up to 1 cm. In the process of crystal growth this size diminishes, the grains acquire elongated shape and are lo-

cated along the ingot axis. Only in the initial region of the ingot is it possible for grains to intergrow throughout its entire thickness. In other places, mainly grains with a transverse size up to half of the ingot thickness were observed.

The lower part of the ingot is characterized by a pronounced texturing along the growth direction with clear smooth boundaries. The upper part of the ingot does not contain grains with clear elongated shape. The size of the grains observed was much lesser. It seems most probable that the changes in the grain size and shape in the process of crystal growth is connected with violation of homogeneity of the temperature conditions at the crystallization front, as well as increase of the amount of background impurities rejected to the final region of the crystal ($K < 1$) and located mainly at the grain boundaries.

The performed investigations testify that with the rise of the crystallization rate and of the melt overheating temperature before the growth, the grain size in the ingot diminishes, and the amount of defects formed in the crystal increases. This reduces the value of optical transmission. As established experimentally, for the growth rate up to 30 mm/h and the melt overheating temperature up to 100°C, no essential worsening of the optical transmission of the examined samples was observed. Therefore,

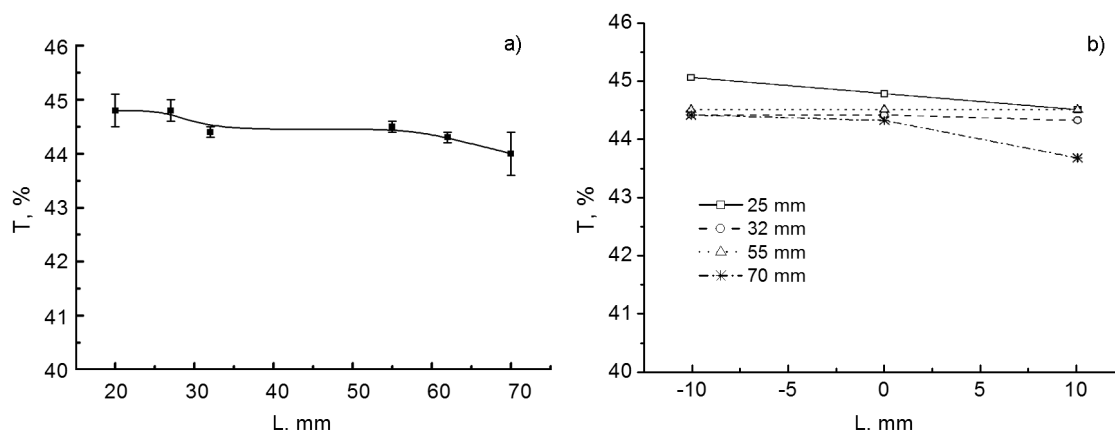


Fig. 2. Distribution of optical transmission ($\lambda = 10.6 \mu\text{m}$) along the length (a) and the width (b) of Ge ingot. (Shown in Fig. 2b is the distance from the ingot start where the distribution of optical transmission along the ingot width was determined).

for further experiments there were chosen the growth parameters: $\text{grad}T = 20^\circ\text{C}/\text{cm}$, $v = 30 \text{ mm/h}$, $\Delta T_{\text{melt}} = 100^\circ\text{C}$.

Both the pure samples and the ones doped with Sb cut out from the crystal perpendicularly to the growth direction had the following crystallographic orientations of the planes: (111), (311), (511). The predominant growth direction for the crystallites was [111]. In the crystallites the deviation of the axis [111] from the crystal growth direction did not exceed 4.7° at the initial region of the ingot and 8.25° up to final region, respectively.

Some grains (crystallites) in the grown crystals had a block structure with a disorientation between the blocks from $5'$ to 1.5° . Study of the dislocation distribution showed that at the final region of the ingot crystallites were strongly defective. The lowest dislocation density ($\sim 10^3 \text{ cm}^{-2}$) was observed in the middle region of the crystals (Fig. 1a), whereas at the final region of the crystal it was essentially higher ($5 \cdot 10^5 \text{ cm}^{-2}$). Even within one grain the dislocation distribution was nonuniform (Fig. 1c). The samples cut out from the final region of the ingots contained more dislocations located along the slip lines. This was probably due to non-stationarity of the growth-annealing conditions, and to the increased amount of the impurities rejected by the crystallization front and favoring the process of defect formation.

Shown in Fig. 2 is the distribution of the optical transmission longwise and crosswise the grown germanium crystal at $\lambda = 10.6 \mu\text{m}$.

The value of optical transmission of undoped poly-Ge crystal in $3\text{--}12 \mu\text{m}$ spectral region ranged between 43 % and 45 %. The

spread of the optical transmission values over the ingot did not exceed 5 %. The decrease of the transmission in final region of the crystal might be caused by the rise of the amount of the background impurities rejected by the crystallization front and due to other defects in the crystal bulk.

As known from [24], at the growth of optical germanium single crystals by the Czochralski method the concentration of Sb was $1 \cdot 10^{13} \text{ cm}^{-3}$ – $5 \cdot 10^{14} \text{ cm}^{-3}$. To establish the required concentration of the said impurity when growing such crystals by the method of HDC the concentration of Sb varied from $8 \cdot 10^{13} \text{ cm}^{-3}$ to $4 \cdot 10^{18} \text{ cm}^{-3}$. The dopant content was calculated taking into account the crystallization rate, the effective distribution coefficient and the coefficient of Sb diffusion in germanium according to the formula:

$$M_L = \frac{N_r \cdot M_{cr}}{N_L \cdot K}, \quad (2)$$

where M_L is the mass of the ligature, g; M_{cr} , the mass of the crystal, g; N_L , the concentration of charge carriers in the ligature; N_{cr} , the concentration of charge carriers in the crystal; K , the effective coefficient of Sb distribution in the crystal.

The numerical value of K was calculated using the Burton-Prim-Slichter equation:

$$K = \frac{K_0}{K_0 + (1 + K_0) \exp\left[\frac{v \cdot \delta}{D_l}\right]}, \quad (3)$$

where v is the crystal growth rate, cm/sec; δ , the diffusive layer thickness, cm; D_l , the

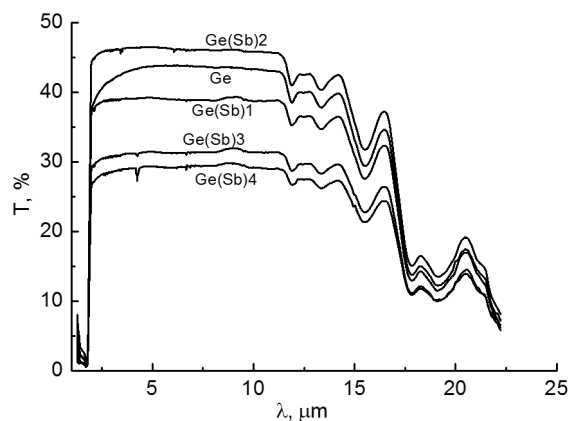


Fig. 3. Optical transmission of germanium doped with Sb of different concentrations (undoped — Ge; Ge(Sb)1 — $8 \cdot 10^{13} \text{ cm}^{-3}$; Ge(Sb)2 — $1.7 \cdot 10^{14} \text{ cm}^{-3}$; Ge(Sb)3 — $5 \cdot 10^{17} \text{ cm}^{-3}$; Ge(Sb)4 — $4 \cdot 10^{18} \text{ cm}^{-3}$).

diffusion coefficient of the impurity in the melt, cm^2/sec ; K_0 , the coefficient of equilibrium impurity distribution in Ge.

The transmission spectra in the IR-region for poly-Ge:Sb with different concentration of Sb are presented in Fig. 3. As is seen, the highest transmission value corresponds to the samples with Sb content equal to $1.7 \cdot 10^{14} \text{ cm}^{-3}$. Such samples were chosen for further investigations.

However, the doped poly-Ge:Sb ingots are characterized by a very high non-uniformity of the optical transmission distribution along the crystallization direction (Fig. 4).

The spread of the optical transmission values over the ingot rises up to 10 %. This may be caused by the formation of absorption centers connected with the dopant atoms present in the crystal in inactive state [13].

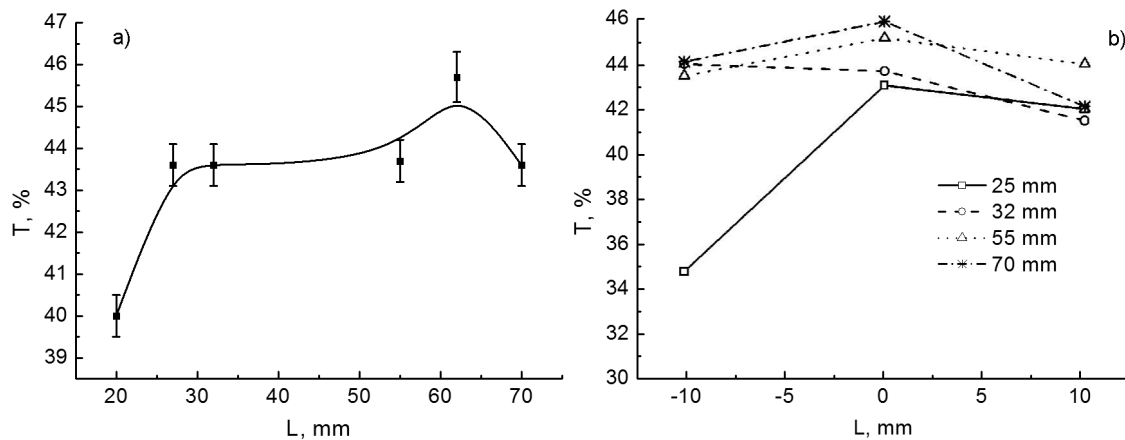


Fig. 4. Distribution of optical transmission ($\lambda = 10.6 \mu\text{m}$) longwise (a) and crosswise (b) poly-Ge:Sb with Sb concentration $1.7 \cdot 10^{14} \text{ cm}^{-3}$. (Shown in Fig. 4b is the distance from the ingot start).

The electrophysical characteristics of the grown poly-Ge:Sb crystals are presented in Table 2. All the crystals have *n*-type conductivity that testifies to predominance of the donor impurities in them.

In all the studied samples the values of ρ at the final regions of the crystal are by 1.5–2 times lower than those in the middle region. In particular, in the process of crystal growth by directional crystallization the impurities with the distribution coefficient $K > 1$ (e.g. boron) will be located at the initial region of the crystal, whereas the ones with $K < 1$ (for Sb $K = 0.02\text{--}0.03$ [25]) will be contained in the final region. So, it may be assumed that the given change in the characteristics is caused just by the mentioned fact. However, such an assumption requires additional investigations.

The measured concentration of free charge carriers in the sample of undoped polycrystalline germanium ($1.5 \cdot 10^{14} \text{ cm}^{-3}$) is higher than the one in the standard single crystal [26]. At final region of the crystal the concentration of free charge carriers rises by more than two fold, that is probably bound up with rejection of the background impurities by the crystallization front.

In the doped germanium samples the concentration of free charge carriers is higher. However, if the influence of the background impurities is neglected, the said concentration is always lower than the calculated value. According to the data from [13], such a fact may be explained by incomplete transition of the introduced impurity atoms into active state. At the same time, proceeding from the proposed assumptions, the general dependence of the distribution of free charge carriers in the crystal remains valid.

Table 2. Electrophysical characteristics of grown germanium ingots

Sample	Part of crystal	Specific electrical resistance (ρ), Ohm-cm	Concentration of charge carriers (N), cm^{-3}	Type of conductivity
Ge	Initial region	25 ± 5.8	$1.5 \cdot 10^{14}$	n
	Middle region	45 ± 8.65	$1.5 \cdot 10^{14}$	
	Final region	35 ± 6.75	$3.5 \cdot 10^{14}$	
Ge(Sb)2	Initial region	22.2 ± 1.4	$2.7 \cdot 10^{14}$	n
	Middle region	42.8 ± 4.8	$2.7 \cdot 10^{14}$	
	Final region	–	$4.2 \cdot 10^{14}$	
Ge(Sb)3	Initial region	31.5 ± 3.25	–	n
	Middle region	62.4 ± 5.45	$3 \cdot 10^{14}$	
	Final region	44.2 ± 6.7	$5 \cdot 10^{14}$	
Ge(Sb)4	Initial region	5.25 ± 0.62	–	n
	Middle region	35.5 ± 7.0	$7 \cdot 10^{14}$	
	Final region	$18,44 \pm 3.63$	$2 \cdot 10^{14}$	

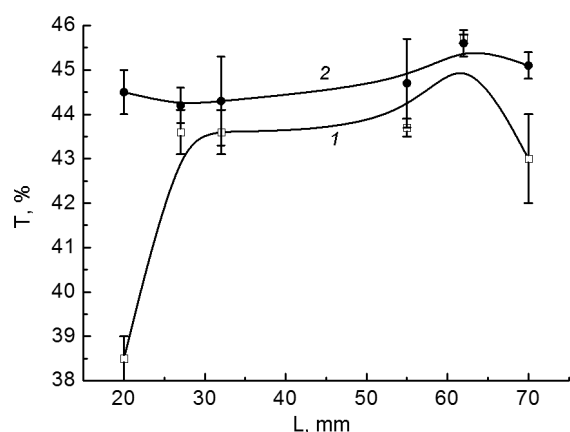


Fig. 5. Optical transmission ($\lambda = 10.6 \mu\text{m}$) along the length of poly-Ge:Sb ingot before (1) and after (2) annealing.

As is known [27], annealing of crystals diminishes the value of residual stresses and the amount of other defects which have an effect on the value of optical transmission. We performed high-temperature (850–900°C) annealing of poly-Ge samples during 20 h. As a result, the optical transmission increased within the whole spectral range of transparency of germanium by 1–2 % and more along the full length of the crystal (Fig. 5, curve 2). Moreover, the uniformity of the optical transmission distribution along the crystal also improved. However, the required optical transmission level of was not achieved.

A low value of optical transmission within the whole of spectral transparency range for undoped germanium is mainly

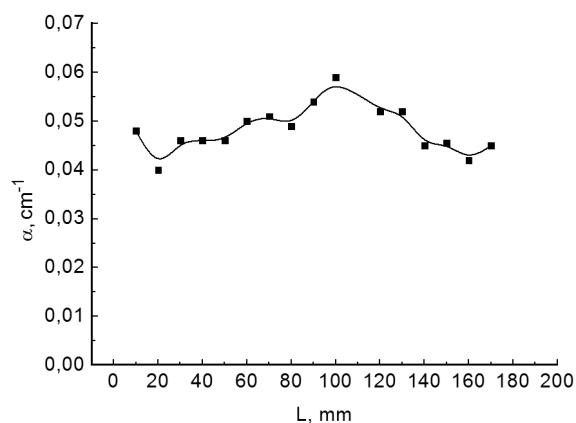


Fig. 6. Distribution of the attenuation coefficient ($\lambda = 10.6 \mu\text{m}$) along the length of poly-Ge:Sb grown with additional purification of the raw material at zone melting.

caused by a high concentration of free charge carriers. Therefore, it is necessary to reduce the amount of background impurities in the raw material. The method of HDC makes it possible to realize additional purification of the raw material before crystallization at zone melting (a narrow zone of the melt, a high temperature gradient).

For additional purification there was performed double re-crystallization of the raw material at zone melting. Afterwards the initial and final regions of the obtained ingot were removed. The middle region of the ingot was placed into a crucible to carry out the growth process with the earlier mentioned growth parameters. The optical transmission ($\lambda = 10.6 \mu\text{m}$) of the samples obtained after additional purification of the

raw material increased along the whole ingot length up to 45–46 %. The optical homogeneity improved, too.

During HDC additional purification of the raw material at zone melting and subsequent crystal growth can be realized in one process, without withdrawal of the crucible from the thermal zone. Besides time saving, such a technique reduces the possibility of contamination of the raw material during the operations bound up with reload of germanium after additional purification of the raw material. Fig. 6 shows the distribution of the attenuation coefficient along the axis of poly-Ge:Sb ingot grown after such a purification of the raw material.

As a result, there was obtained the value of attenuation coefficient close to 0.04 cm^{-1} , corresponding to the standard requirements to polycrystals for optical applications [13], as well as a high uniformity ($\Delta\alpha \sim 0.01 \text{ cm}^{-1}$) of its distribution along the ingot axis at a length of $\sim 200 \text{ mm}$. The value of specific electrical resistance and the concentration of free charge carriers within the limits of measurement error also remained practically unchanged along the whole of the ingot length.

4. Conclusions

As found in the present study, during the growth of polycrystalline Ge by the method of HDC the value and non-uniformity of the distribution of the attenuation coefficient in the polycrystal weakly depends on the grain size and other macro-defects. The main contribution to the optical absorption value belongs to free charge carriers which concentration depends on the doping addition present in active state, and on background impurities in Ge. The choice of the growth conditions and of the amount of the dopant, as well as thermal high-temperature annealing increase the value of optical transmission. There was obtained poly-Ge:Sb with the light attenuation coefficient $\alpha \sim 0.04 \text{ cm}^{-1}$ (for $\lambda = 10.6 \text{ }\mu\text{m}$) and a high uniformity of distribution of optical characteristics along the ingot axis.

The results obtained during the present study show that the use of the method of HDC is promising for the obtaining of germanium of high optical quality.

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