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06

Effect of dislocations on heat transfer in germanium

© O.V. Malyshkina,[1] I.A. Kaplunov,[1] P.V. Mamaev,[1] V.E. Rogalin,[2] G.I. Kropotov[3]

[1] Tver State University,
170002 Tver, Russia
[2] Institute for Electrophysics and Electric Power, Russian Academy of Sciences,
191186 St. Petersburg, Russia
[3] TYDEX, LLC,
194292 St. Petersburg, Russia
e-mail: Olga.Malyshkina@mail.ru

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Comparative studies of the thermal characteristics of the germanium single crystals with dislocations and dislocation-free germanium in the dynamic mode of passage of a temperature wave through a sample were carried out. It is shown that samples of dislocation-free germanium have a higher thermal conductivity coefficient (by 18 % in the crystallographic direction $\langle 100\rangle$ and by 9 % in the direction $\langle 111\rangle$) compared to samples of germanium with dislocations. At the same time, the difference in the values of the thermal diffusivity coefficients of germanium samples with dislocations and dislocation-free is insignificant.

Keywords: crystalline germanium, dislocation-free single-crystal germanium, thermal conductivity, thermal diffusivity, TSW method for studying thermal characteristics.

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Introduction

Germanium (Ge) is the first and one of the purest, most sought-after and thoroughly studied classical semiconductor materials [1]. Currently, optics and electronics are the main applications of crystal germanium [1,2]. In the atmospheric transparency window $8.0-14\,\mu$m, germanium, due to its best physico-chemical properties, is the most sought-after optical and effective acousto-optic material [2,3]. In recent years, interest in Germany has intensified due to the expansion of the use of the material for the manufacture of photovoltaic converter substrates [2,4]. The use of germanium as high-energy radiation detectors is relevant [5,6].

For most applications of crystalline germanium, requirements are put forward not only for high purity or a certain level of alloying, but also for a minimum concentration of defects in the crystal lattice of the material. The main types of defects that have the greatest impact on the optical and electrophysical characteristics of germanium single crystals include, first of all, dislocations and their derivatives, small-angle boundaries and slip lines.

Dislocations mainly affect the mechanical properties of solids (elasticity, plasticity, strength, internal friction), for which their presence is often crucial. They change the optical properties of crystals, which is the basis for the method of observing isolated dislocations in transparent materials. Dislocations disrupt the optical uniformity of crystals, which leads to significant scattering of IR radiation and electric charge carriers [7–9]. The presence of dislocations in low-dislocation crystals used in photovoltaics leads to a mismatch between the parameters of Ge crystal lattices and compounds $A^{III}B^{V}$, preventing the growth of high-quality photosensitive epitaxial layers on a germanium substrate [10–12].

Dislocations indirectly affect the properties of crystals, depending on the nature of the distribution and movement of point defects (impurities, vacancies, color centers, etc.) in them [13].

It follows from the above that it is important to understand the thermophysical properties of germanium. At the same time, until recently, it was believed that thermal conductivity is a constant of a material that depends on environmental parameters, primarily on temperature, but not on the concentration of structural defects in the material. Engineering calculations usually used the reference value obtained during the initial study of the material, which in some cases did not correspond to reality. The development of power electronics and optics has revealed the need to obtain materials with high thermal conductivity, and this requires a more thorough study of this parameter in order to identify the possibilities of increasing it.

Germanium single crystals are actively used in semiconductor electronics and in infrared optics, which leads to a fairly good understanding of the properties of the material. In addition, Ge, due to its relatively simple, well-studied crystal lattice and high chemical and structural perfection, is often used to study insufficiently studied processes in solids.

The main contribution to Ge thermal conductivity is made by the phonon component. The phonon spectrum noticeably depends on various structural defects, although this is most often manifested at low temperatures [14]. From general considerations, it is clear that dislocations, being a noticeable defect in the crystal lattice, should affect phonon processes, which include thermal conductivity.

We showed in Ref. [15–17] the dependence of the coefficients of thermal conductivity and thermal diffusion of Ge on the concentration of the alloying impurity and on the crystallographic direction in which the temperature wave passes through the sample. Thus, an increase in the concentration of the alloying impurity leads to a decrease in the values of the Ge thermal conductivity and thermal diffusion coefficients. This effect is more pronounced in Ge crystals of *n*-type. The maximum values of the coefficients of thermal conductivity and thermal diffusion of single-crystal Ge of *n*-type are observed in the crystallographic direction $\langle 111\rangle$, the minimum values correspond to polycrystalline germanium.

In addition, it is known that natural germanium contains 5 isotopes in different percentages. Such an isotopic disorder introduces distortions into the crystal lattice, which affects the thermal conductivity of Ge. The authors of Ref. [18] have shown that isotopically pure Ge at low temperatures has a thermal conductivity of 8.5 times higher than Ge of natural composition. Isotopically pure germanium single crystals differ from natural crystals in such characteristics as the phase transition temperature, unit cell parameters, absorption and reflection coefficients of radiation; there is also a significant difference in the propagation of sound waves [19,20].

Thus, at the moment it is relevant to conduct comparative studies of the passage of temperature waves (i.e., the values of the coefficients of thermal conductivity and thermal diffusivity) through single crystals with a perfect crystal structure — dislocation-free single crystals and through single crystals with dislocations, which usually occur when germanium single crystals are grown from a melt, unless special technological measures are used to reduce the number of dislocations.

1. Experimental methodology

Single-crystal and polycrystalline germanium samples of the electronic type of conductivity (antimony is impurity) were studied in this paper, which differed in the magnitude of the electrical resistivity (concentration of the alloying impurity), in the crystallographic direction, and in the presence and absence of dislocations in single crystals. The crystals were grown from the melt by the Czochralski method at Tver State University (polycrystals and dislocated germanium) and at the „Germaniy“ JSC (Krasnoyarsk). The electrical resistivity was determined by the four-probe method. The dislocation density of the studied samples was measured by counting the pits of chemical etching according to GOST 16153-80. The characteristics of the samples are given in sec. 2.

The method of determining the coefficient of thermal diffusion (thermal conductivity), which is based on periodic heating of one surface of a sample, was first proposed by A.J. Angstrom in 1863, as described in Ref. [21]. S. Lang [22] modified the method by suggesting that the upper surface of the material under study, located on a pyroelectric detector, be illuminated with a sinusoidally modulated heat flux. The temperature wave, passing through the sample under study, propagates in the detector. A change in temperature in the pyroelectric crystal of the detector causes a change in polarization, which leads to the flow of pyroelectric current in the external circuit. Using a synchronous amplifier (Lock-In amplifier), the amplitude of the pyroelectric current coming from the detector and the phase difference between the heat flow incident on the sample under study and the pyroelectric current are recorded. Using mathematical modeling methods, the frequency dependence of the pyroelectric current and the phase difference between the sinusoidally modulated heat wave incident on the sample and the pyroelectric response are calculated. The thermal conductivity coefficient of the test sample is selected in such a way that the calculated curve of the frequency dependence of the phase difference coincides with the experimental one [22]. The authors of Ref. [23,24] proposed to use the Thermal Square Wave Method at Single Frequency (TSW) to determine the thermal conductivity coefficient, when the sample surface is heated by a rectangular modulated heat flow. The use of square wave heat flow modulation, as shown in detail in Ref. [24], significantly simplifies the mathematical apparatus. The TSW method makes it possible to evaluate the coefficients of thermal conductivity and thermal conductivity of non-ferroelectric materials located on a ferroelectric crystal, regardless of the ratio: thickness of non-ferroelectric material–thickness of ferroelectric [15,23].

The TSW method is based on measuring the pyroelectric current induced in a ferroelectric crystal on which a non-ferroelectric material (in our case, a Ge sample) is placed using a rectangularly modulated heat flux (i.e., with periodic changes in the sample temperature) (Fig. 1). A lithium tantalate (TL) crystal was used as a ferroelectric crystal in this experiment. This choice is due to the fact that TL has a stable, homogeneous spontaneous polarization, which is practically impossible to change by exposure to an external field or temperature gradient.

As shown by the authors of Ref. [24], when using a square-wave modulated heat flux in pyroelectric studies, the pyroelectric response of a uniformly polarized ferroactive material repeats its shape if the depth of penetration of the temperature wave into the sample (l) is less than one third of the sample thickness (h), otherwise the so-called „film“ response is observed. As an example of the latter, Fig. 2 (curve *1*) shows the pyroelectric response of a TL crystal with a thickness of 1 mm, observed when the heat flow is modulated with a frequency of 0.15 Hz.

When a non-ferroelectric material is placed on TL (in our studies, these are Ge samples), a „blockage“ appears at the beginning of the pyroelectric response pulse (Fig. 2, curves *2* and *3*), the value of which is determined by the thickness of the non-ferroelectric sample and the values of the thermal conductivity and thermal conductivity coefficients of the studied the material. As a result of calculating

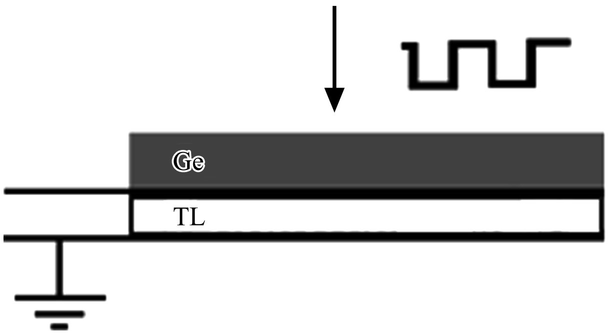

Figure 1. A scheme for registering a pyroelectric response when measuring the coefficients of thermal conductivity and thermal conductivity using the TSM method.

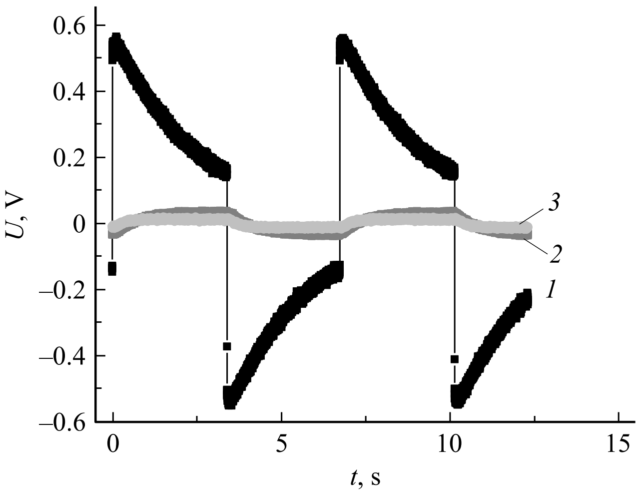

Figure 2. Pyroelectric response of a TL crystal observed in case of a direct exposure of a sample to a modulated heat wave (curve *1*) and when a temperature wave passes through a sample of a Ge crystal: with dislocations (curve *2*) and without dislocations (curve *3*) placed on TL crystal.

the temperature gradient over the thickness of the sample when the temperature changes in the dynamic mode (taking into account the parameters used in the experiment, the thickness of the Ge sample and the heat flux power), the gradient value of 0.001 K/mm was obtained. The gradient duration at the heat flux modulation frequency of 0.15 Hz does not exceed 3.5 s, and the experiments were carried out at room temperature ($\sim 25\,^\circ$C). Thus, the temperature gradient occurring in the experiments presented in the article cannot cause dislocation movement, since, according to Ref. [25], when holding a sample with dislocations under a temperature gradient of 0.08 K/mm (temperature ranged from 910 to 915 °C) for 50 h, the dislocation velocity was only $0.10{-}0.25\,\mu$m/s.

In this case, a formula is used to calculate the pyroelectric response of a ferroelectric, the conclusion of which is carried out provided that the depth of penetration of the temperature wave into the ferroelectric material is less than 1/3 of its thickness [24]:

$$I_{\mathrm{TL}} = \frac{S\beta_0 W_0 \gamma}{\rho c(d+h)} \operatorname{Re}\left\{\sum_{n=1}^{\infty}\left\{\frac{\sin(n\omega\tau/2)}{n\omega\tau/2}\exp(in\omega t) \times \frac{\mathrm{sh}[\varphi_2 h]}{\left[\mathrm{ch}(\varphi_1 d) + \frac{H}{\varphi_1}\mathrm{sh}(\varphi_1 d)\right] \times \mathrm{sh}(\varphi_2 h) + \left[\frac{k_1 H_1}{k_2\varphi_2}\mathrm{ch}(\varphi_1 d) + \frac{k_1\varphi_1}{k_2\varphi_2}\mathrm{sh}(\varphi_1 d)\right] \times \mathrm{ch}(\varphi_2 h)}\right\}\right\}. \qquad (1)$$

Here h is the thickness of ferroelectric, d is the thickness of non-ferroelectric material, I_{TL} is the pyroelectric current of ferroelectric, S is the area of illuminated surface, [m^2], W_0 is the heat flux power density, [W/m^2], γ is the pyroelectric coefficient of ferroelectric, [C/m^{2}K], ρ is its density, [kg/m^3], c is the specific heat, [J/(kg·K)], τ is the pulse light interval duration, [s], $\tau = T/2 = 1/(2f)$, f is the heat flux modulation frequency, k_1 and k_2 are thermal conductivity coefficients of a non-ferroelectric material and a ferroelectric substrate, respectively, [W/(m·K)], $H_1 = 4\beta_0\sigma T^3)0/k_1$ characterizes radiation losses, σ Stefan-Boltzmann constant $(5.6704 \cdot 10^{-8}\,\mathrm{W/(m^2K^4)})$, T_0 is the ambient temperature, [K], $\varphi_m = (1+i)\sqrt{n\omega/2\alpha_m}$, $\omega = 2\pi f$, $m = 1, 2$; α_1 and α_2 are thermal diffusivity coefficient of non-ferroelectric material and ferroelectric, respectively, [m^2/s].

We have previously shown in Ref. [15] that the use of the formula (1) allows varying two parameters in the calculation: the values of the coefficients of thermal conductivity and thermal diffusivity. This is due to the fact that the pyroelectric response value calculated using this formula (all other things being equal) increas, when the value of the thermal conductivity coefficient decreasing; and decreas, when the value of the thermal diffusivity coefficient decreasing. At the same time, if a change in the value of the thermal conductivity coefficient changes only the magnitude of the pyroelectric response, then a decrease in the thermal diffusivity coefficient also leads to a change in its shape— the tilting observed at the beginning of the response increases.

Thus, a comparison of the experimental forms of the pyroelectric response with those calculated using the formula (1) makes it possible to estimate the value of the coefficient of thermal diffusivity and thermal conductivity of the material through which the temperature wave passes.

A necessary condition of the experiment is that the area of a non-ferroelectric material should not exceed the area of a ferroelectric. In this study, we used TL and Ge crystals with an area of 10 × 10 mm. The thickness of the TL crystal was 1 mm, the thickness of Ge crystal was 5 mm.

2. Analysis and discussion of experimental results

Samples of Ge of n-type with a resistivity of $22 \pm 2\,\Omega\cdot$cm; crystallographic direction $\langle 100\rangle$ of single crystals with dislocations and dislocation-free single crystals; as well

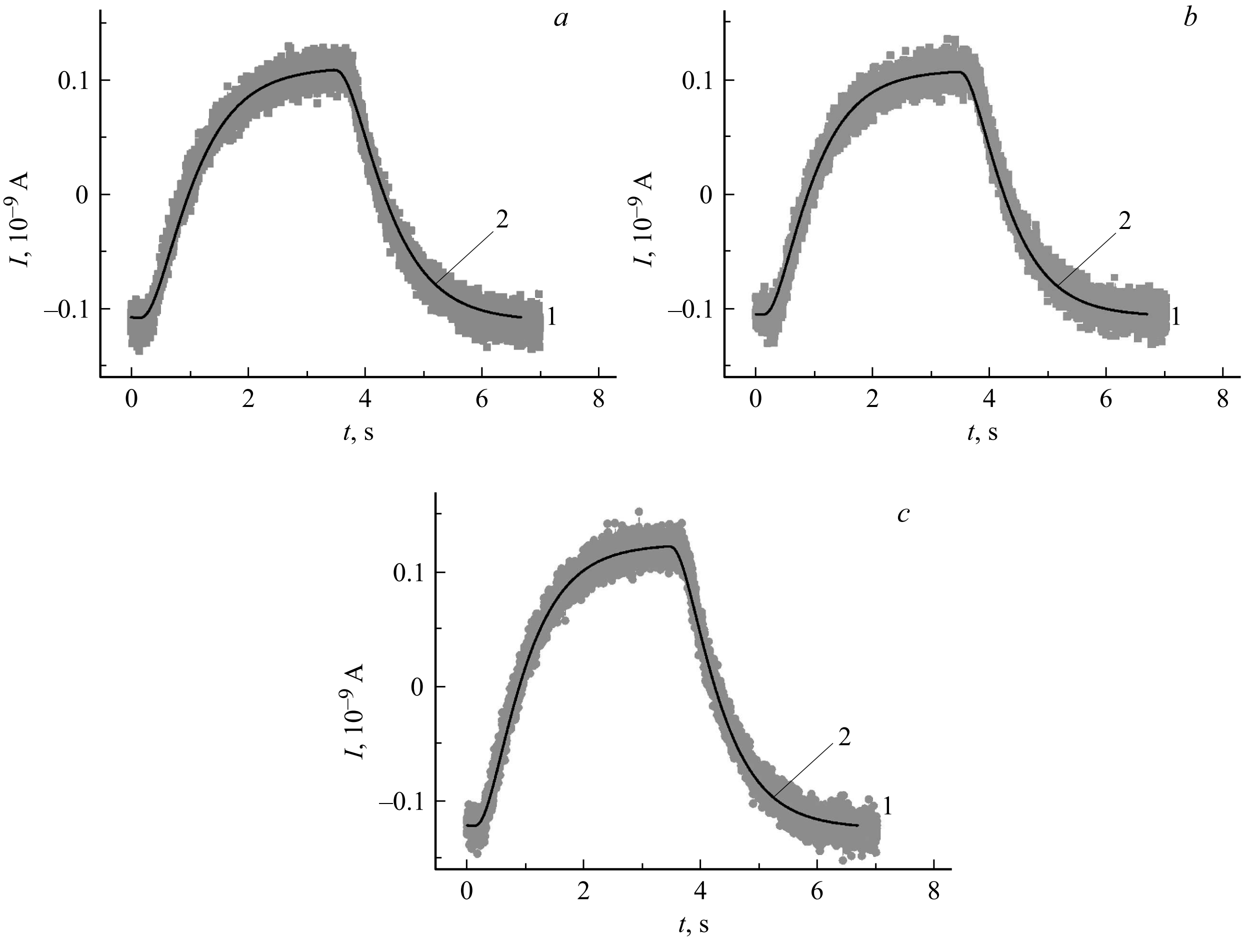

Figure 3. TL pyroelectric response, observed experimentally (curves *1*) and calculated (curves *2*) when a temperature wave passes through the samples of single crystals with dislocations (*a*) and dislocation-free single crystals (*b*) in the crystallographic direction $\langle 100\rangle$, and a sample of polycrystalline germanium (*c*).

as polycrystalline germanium were selected for comparative studies of thermal characteristics. The concentration of the n dopant (antimony) in these samples was $\sim 10^{14}\,\mathrm{cm}^{-3}$. Single crystals with dislocations and dislocation-free single crystals single crystals with a resistivity of $0.75\,\Omega\cdot\mathrm{cm}$ ($n \sim 2.5\cdot 10^{15}\,\mathrm{cm}^{-3}$) of the crystallographic direction $\langle 111\rangle$ were also studied. The dislocation density in all single crystals with dislocations was in the range of $\sim (0.5-1.0)\cdot 10^{4}\,\mathrm{cm}^{-2}$. The dislocation density in dislocation-free single crystals, according to GOST, is less than 10 cm^{-2}, it was less than $5\mathrm{cm}^{-2}$ in the studied samples.

The heat flux incident on the germanium sample was modulated by rectangular pulses with a frequency of $f = 0.15\,\mathrm{Hz}$. A continuous semiconductor IR laser emitting at a wavelength of 920 nm was used as a source of thermal radiation. The radiation was modulated by rectangular pulses using a power converter. The surface of the samples was blackened with fine graphite powder to ensure 100% heat absorption.

When a temperature wave passed through the samples of single crystals with dislocations and dislocation-free single crystals (Fig. 2, curves *2* and *3*), there was a difference in the shape of the TL pyroelectric response, which means a difference in the values of the thermal diffusivity and thermal conductivity of these samples (i.e., the influence of the material structure on their thermal characteristics).

The values of the thermal conductivity coefficients (k) and thermal diffusivity coefficients (α) of the studied Ge samples were determined by comparing the calculated formula (1) and the experimental pyroelectric response formula (Fig. 3, 4). During the calculation, the values of k and α varied as a parameter.

The values of the thermophysical characteristics of Ge, at which the calculated shapes of the pyroelectric response coincided with the experimentally observed ones, are given in the Table. 1 and 2 for Ge samples with a resistivity of $(22 \pm 2)\,\Omega\cdot\mathrm{cm}$ and $0.75\,\Omega\cdot\mathrm{cm}$, respectively. The error in calculating the thermal characteristics indicated in the tables was estimated using the methodology described in detail in Ref. [17].

As can be seen from the presented results (Table 1 and 2), if the values of the thermal diffusivity coefficient of the studied materials slightly differ, then the value of

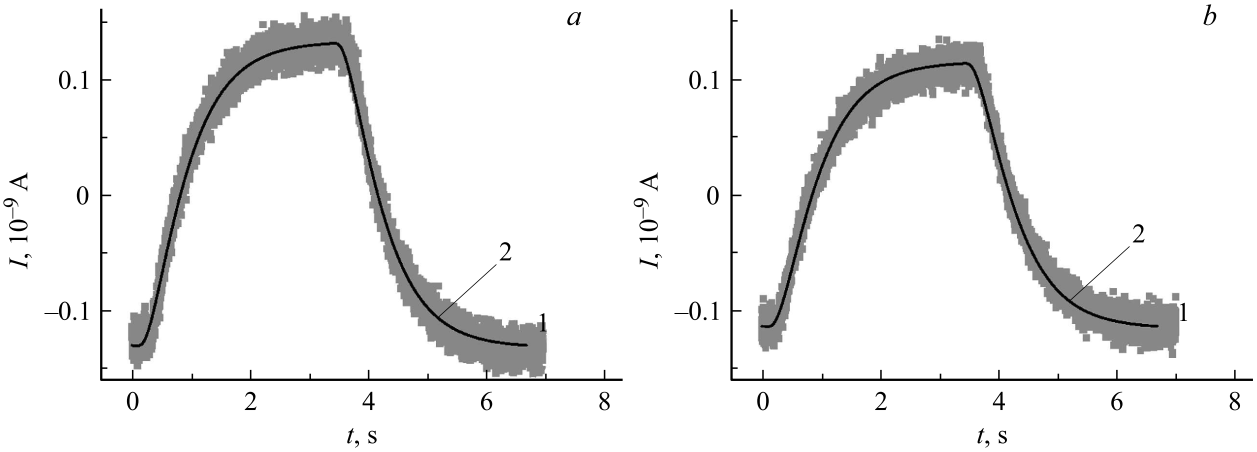

Figure 4. The experimentally observed (curves *1*) and calculated (curves *2*) TL pyroelectric response, when a temperature wave passes through the samples of single crystals with dislocations (*a*) and dislocation-free single crystals (*b*) in the crystallographic direction ⟨111⟩.

Table 1. Values of thermal conductivity coefficients (k) and thermal diffusivity coefficients (α) of samples of dislocation-free single crystals, single crystals with dislocations and polycrystalline germanium with a resistivity of $(22 \pm 2)\,\Omega\cdot$cm ($n \sim 10^{14}\,\mathrm{cm}^{-3}$). Crystallographic direction ⟨100⟩

Sample	k, W/(m·K)	α, $10^{-6}\,\mathrm{m}^2/\mathrm{s}$
dislocation-free single crystal	87 ± 5	7 ± 0.5
single crystal with dislocations	72 ± 5	6 ± 0.5
polycrystalline germanium	75 ± 5	7 ± 0.5

Table 2. Values of thermal conductivity coefficients (k) and thermal diffusivity coefficients (α) of samples of dislocation-free single crystals and single crystals with dislocations with resistivity $0.75\,\Omega\cdot$cm ($n \sim 2.5 \cdot 10^{15}\,\mathrm{cm}^{-3}$). Crystallographic direction ⟨111⟩

Sample	k, W/(m·K)	α, $10^{-6}\,\mathrm{m}^2/\mathrm{s}$
dislocation- free single crystal	87 ± 5	7.5 ± 0.5
single crystal with dislocations	80 ± 5	8 ± 0.5

the thermal diffusivity coefficient of the dislocation-free single crystals sample in the crystallographic direction ⟨111⟩ exceeds the similar value for the sample of single crystals with dislocations by 9 %, and by 18 % in the direction ⟨100⟩.

The same values of the thermophysical characteristics of different dislocation-free single crystals samples attract attention. It should be borne in mind here that, firstly, the studied samples had different impurity concentrations, and, secondly, the thermal characteristics were studied in different crystallographic directions (⟨100⟩ and ⟨111⟩). Consequently, the absence of an increase in the value of k in the crystallographic direction ⟨111⟩ compared to the crystallographic direction ⟨100⟩ (which should have been observed, according to the result of our study in Ref. [15]) is „compensated“ by its a decrease due to the dependence of the values of the thermal conductivity and thermal diffusivity coefficients of the Ge of n-type on the concentration of the dopant [16].

It is interesting to note that Ge samples with a resistivity of $(22 \pm 2)\,\Omega\cdot$cm (Table 1), for which the thermophysical characteristics of the polycrystalline germanium were also measured, the values of the thermal diffusivity coefficient of the samples of dislocation-free single crystals and polycrystalline germanium coincide, whereas it is less for the sample of single crystals with dislocations.

Conclusion

A comparative analysis of the values of the coefficients of thermal coefficients and thermal diffusivity of crystalline germanium samples showed an increase in the coefficient of thermal conductivity in the dynamic mode of passage of a temperature wave through the dislocation-free single crystals sample compared with the sample of single crystals with dislocations. The higher value of k in dislocation-free single crystals is due to a more perfect crystal lattice. The increase in thermal conductivity occurs both in the crystallographic direction ⟨111⟩ and in the direction ⟨100⟩, but in the second case it is much greater.

The difference in the values of the thermal diffusivity coefficient is less significant. For samples with low resistivity ($0.75\,\Omega\cdot$cm), the measurements for which were carried out during the passage of a temperature wave in the crystallographic direction ⟨100⟩, they differ within the error limits.

For samples with high resistivity ($(22 \pm 2)\,\Omega\cdot$cm), when measured in the crystallographic direction ⟨111⟩, the difference in the values of the thermal diffusivity coefficient of samples of dislocation-free single crystals and single crystals with dislocations is 8 %. The coincidence of the values of the thermal diffusivity coefficient of samples of dislocation-free single crystals and polycrystalline germanium can probably be explained by the presence of micropores in dislocation-free germanium single crystals, which are assumed to occur due to clustering of vacancies during crystal growth [26].

Conflict of interest

The authors declare that they have no conflict of interest.

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