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NONCONTACT LASER CONTROL OF ELECTRIC-PHYSICAL PARAMETERS OF SEMICONDUCTOR LAYERS

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Non-contact non-destructive laser-interferometric methods for measuring several electrophysical parameters of semiconductor and dielectric layers are proposed. They are the lifetime of charge carriers for electrons and holes separately; parameters of recombination centers, namely their concentration and capture cross-sections; bulk volume lifetime and rate of surface recombination, as well as the diffusion length of charge carriers. The methods are based on the interference-absorption interaction in a semiconductor of two laser radiations with different wavelengths. Short-wave injection radiation generates additional charge carriers in the material, which leads to a change in its optical constants at the wavelength of the other – long-wavelength probing laser radiation – and to modulation of this radiation as it passes through the sample of the studied material. The means for implementing the proposed methods and methods for processing the modulation signal for determining the parameters of the investigated samples are developed.

The methods have been successfully tested on samples of such materials as germanium, silicon, indium antimonide and cadmium-mercury-tellurium alloy. It is shown that the methods can be used both in scientific research and electronic industry.

Keywords: laser interferometry, noncontact control, electrophysical parameters

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Introduction. Metallurgy of semiconductors is a special sector of metallurgy [12]. The reason for this is that the use of semiconductors in the electronics industry requires high purity materials. It must comply with the rule of «nine nines»: the main substance should be at least 0.999999999 of the whole mass. Otherwise, the atoms of the impurities produced levels in the forbidden band of a semiconductor and unacceptably change its electrophysical properties. In produced addition, most semiconductors are produced by metallurgical plants in the form of single crystals with a given crystallographic line. After manufacturing, crystals that have very large dimensions are divided into 0.5 mm thick plates. Usually, these plates are ground and polished and already in this form are delivered from metallurgical plants to the electronic industry, where they go through several technological operations on plates, and electronic devices for various purposes are produced. The high sensitivity of semiconductors to almost all impacts requires monitoring of their electrical properties at all stages of production in both the metallurgical and electronic industries [1, 11]. And it is desirable that this control should be non-destructive, noncontact and local.

Most of existing methods of measuring the parameters of semiconductor materials are contact, i.e. require the creation of an electrical contact with the studied semiconductor [2, 5, 13, 31]. It is always either inconvenient or impossible. If many devices are placed on a single semiconductor wafer (for example, using planar technology), part of the plate must be designed for the manufacture of specially developed test structures. The technology of these structures should be compatible with the technology of production of basic devices and should not change the parameters of the semiconductor during production process.

The creation of test structures leads to unproductive consumption of the working area of the semiconductor plate and, therefore, to a decrease in the number of working units on one plate and to an increase of their cost. In addition, when using test structures, measurements are taken not in the working area of the unit but near, where the parameters of the semiconductor may differ. The very process of measuring test structures by establishing an electrical contact with them mechanically can lead to failure of the main units due to damage and contamination of the semiconductor plate.

That is why the efforts of developers are aimed at creating methods for measuring the parameters of semiconductor materials that do not require mechanical contact. For example, there have been developed methods using an electron beam to probe the structure of the measuring unit [21, 31]. Raster scanning methods of electron-beam sounding make it possible to obtain an exceptionally

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high resolution along the area of the sample, and to study its surface in detail. However, the use of the electron beam requires the vacuum treatment evacuation of the sample during the measuring process. This greatly increases the measurement time and cost. Because of this, electron-beam methods find application primarily in the research and development work in the field of semiconductor instrument engineering.

Recently there have been developed methods where electromagnetic radiation of ultrahigh frequencies is used to probe the electrophysical parameters of semiconductors [8, 15, 17, 24]. Unfortunately, all works in which electromagnetic radiation of ultrahigh frequencies are applied have a significant drawback – a low resolution in terms of area. As it is known, the resolving power is determined by the wavelength of the applied radiation, which in this case exceeds 1 mm. The resolving power of ultrahigh-frequency methods is within millimeters range, which in many cases is not enough, especially in the production of micro-sized instruments.

Optical methods of control. The use of optical (infrared and visible) radiation for probing semiconductors theoretically allows to increase the resolving power of non-contact methods of radiation probing tenfold. The radiation of modern lasers can be focused into a spot several microns in size. The question is how to use laser radiation to study the electrical properties of semiconductors.

Such optical characteristics of a semiconductor, as the absorption and refraction of optical radiation, in accordance with the electronic dispersion theory depend [10] on the concentration of charge carriers (electrons and holes). This dependence is stronger at large wavelengths, i.e. in the infrared optical range. In the same range, semiconductors usually have regions of transparency or weak absorption. Laser radiation with a wavelength corresponding to these transparency windows can act as probing.

An increase in the concentration of charge carriers, electrons, and holes in a semiconductor can be achieved by the generation of nonequilibrium electron-hole pairs. This can also be done with the help of optical radiations, but at other wavelengths corresponding to the internal photoelectric effect in the studied semiconductor.

In any way, in theory, the use of optical radiation makes it possible to build completely non-contact methods for studying the behavior of electric charges in semiconductor materials.

The appearance of additional electron-hole pairs in the semiconductor when exposed to radiation from the spectral region of the internal photoelectric effect of this semiconductor increases the absorption coefficient of a long-wave probing beam of infrared radiation. This leads to a change in the transmission of the probe beam by the sample. By the magnitude of the emerging modulation of the probing beam, one can judge the electrophysical properties of the semiconductor. Unfortunately, the method based on the measurement of the absorption modulation is not sensitive enough [27, 29, 30]. To use for studied samples, it is necessary to reach a concentration of additional charge carriers of more than 10^{16}-10^{17}cm^{-3}.

At the same time, in recent years, high-sensitivity non-contact laser methods for measuring the thickness of semiconductor and dielectric layers, based not only on absorption but also on the interference of probing optical radiation, have been intensively developed. The main purpose of these methods was to determine the geometric thickness of semiconductor and dielectric layers. At the same time, high sensitivity and speed were achieved [18, 26, 28]. In fact, these methods do not determine the geometrical thickness of the layer, but its optical thickness, which is the product of the geometrical thickness by the refractive index of the material. Usually, in optical studies of semiconductor materials, the interference of laser radiation in a semiconductor was either neglected or suppressed, only a small number of studies considered interference effects. As it turned out, the accounting and use of these subtle but reliably recorded effects allows us to create new research methods and obtain more detailed information on the recombination characteristics of semiconductors.

The physical principle of laser-interference identification of the electrical-physical parameters of semiconductors. The idea proposed by the authors, was that well developed for the

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study of geometrical parameters of the dielectric layers and semiconductor laser interferometric techniques can be modified so that they may be used to explore the kinetics of generation and recombination of charge carriers in semiconductors and identify electrophysical parameters of these processes. For this purpose, a new physical mechanism was proposed – the mechanism of interference-absorption interaction in a semiconductor of two laser radiations [7]. It consists in the following. One of the emissions has a photon energy greater than the width of the forbidden band of the semiconductor under investigation. The semiconductor absorbs it and generates electron-hole pairs, changing the concentrations of free charge carriers. The other radiation corresponds to the semiconductor transparency band. It is probing and has photon energy less than the width of the forbidden band of a semiconductor and, accordingly, is very weakly absorbed. The test sample is made in the form of a plane-parallel plate and is formed between two reflecting surfaces creating a Fabry-Perot interferometer. Such reflecting surfaces could be the faces of the sample itself (due to the refractive index discontinuity at the air-semiconductor boundary). The parameters of the probing radiation, both transmitted through the sample, and reflected, primarily its intensity, are determined by the result of interference. The generation of nonequilibrium carriers in the semiconductor when it is exposed to laser-injector radiation leads to a change in the refractive index and absorption coefficient at the wavelength of the probing beam. This, in turn, causes a change in the intensity of the probe beam passing through the sample (and reflected by it) because of the restructuring of the semiconductor interferometer wavelength and its increased absorption.

Mathematical patterns of laser-interference modulation. The calculations showed that the change in the power of the probing beam can exceed the value of the power of the injecting laser causing this effect. Thus, it is possible to implement optical amplification in a laser-semiconductor system [7]. Experiments on the detection and investigation of the interference modulation effect were performed on germanium [16]. During the experiments, the validity of the proposed model of the phenomenon was confirmed and the relationship between the value of interference modulation and the concentration of nonequilibrium current carriers was established. We also obtained formulas describing the relationship of magnitude of the modulation of the probing beam to both the concentration of nonequilibrium carriers and their lifetime, and to the interference characteristics of the samples, that are derivatives of its transmission along the phase angle of the probing radiation and the absorption in one pass [23]. The probing radiation modulation coefficient $M = \Delta I/I$ depends on the lifetimes of nonequilibrium charge carriers as follows:

$$M = \left[T_v'\left(\sigma_p \tau_p + \sigma_n \tau_n\right) + T_\delta' \frac{e^2 \lambda_z}{2\pi\varepsilon_0 n c^2}\left(\frac{\tau_p}{m_p^*} + \frac{\tau_n}{m_n^*}\right)\right]\beta L ,$$

where σ_n and σ_p – absorption cross section of probing radiation by electrons and holes; m_n^* and m_p^* – effective masses of electrons and holes; T_δ' and T_v' – the derivatives of the transmission of the sample along the phase angle $\delta = (4\pi n l \cdot \cos\varphi_2)/\lambda_z$ and parameter $v = \exp(-\alpha_z l)$; l – sample thickness; n – refractive index of the "unexposed" semiconductor; φ_2 – angle of refraction of probe radiation in a semiconductor; e – electron charge; ε_0 – electric constant; c – light speed in vacuum; β – quantum yield of internal photoelectric effect; L – the number of emission quanta of the injector absorbed in the sample. As you can see, this formula includes world constants and parameters of the semiconductor σ_n, σ_p, n, m_n^*, m_p^*.

The values T_δ' and T_v' can be obtained by differentiating the Airy function. In particular, for $v \approx 0$ (which is the case when λ_z is selected from the range of high transparency of the semiconductor) T_δ' and T_v' are defined by the expressions

$$T_v' = \frac{(1-R)^2(1-R^2)}{[(1-R)^2 + 4R\sin^2(\delta/2)]^2} ,$$

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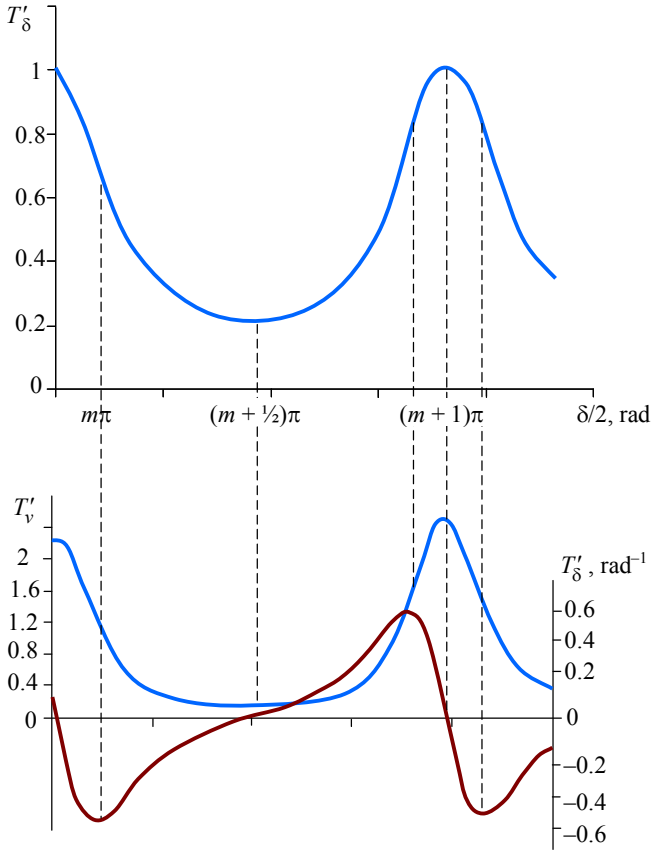


Fig.1. Dependencies of T_δ' and T_v' from phase angle δ for ideal Fabry-Perot interferometer

$$T'_\delta = \frac{-2(1-R)^2 R\sin\delta}{[(1-R)^2 + 4R\sin^2(\delta/2)]^2},$$

where R – reflection coefficient of the sample face

Graphically, these functions are shown in Fig.1.

Experimental schemes. Separate determination of the lifetimes of electrons and holes. An analysis of the established physical regularities of the interference modulation mechanism made it possible in one experiment to propose a method for separate determination of concentrations and lifetimes of nonequilibrium electrons and holes [14]. The main recombination parameter of a semiconductor is the lifetime of charge carriers: electrons and holes. The carrier lifetime, i.e. the time from generation (for example, from light) of an electron-hole pair to their mutual annihilation-recombination, it can be the same for both electrons and holes. The difference usually arises when recombination does not go directly, but through recombination centers, which are impurities and defects. The capture cross sections for such centers, especially charged ones, can differ greatly for electrons and holes. Therefore, in the beginning, the carrier of one sign is captured and loses its mobility, and after some time the same happens with another carrier. The concentrations of recombination centers and the capture cross sections of carriers are also important recombination parameters of the semiconductor.

The proposed method is based on the different character of the two components of the relationship of transmission modulation of the probe beam by the sample from the increase in the carrier concentration. The component associated with absorption always increases with concentration growth. The component associated with the change in the refractive index, with carrier concentration growth, can both increase and decrease. This is a consequence of the transmission periodicity, depending on the optical thickness of the sample, and hence the refractive index. Thus, if we measure the modulation of the probing beam produced in the sample when it is illuminated with an optical injector, at two different optical thicknesses of the sample (Fig.2), we obtain a system of two linear equations for the nonequilibrium concentrations of electrons and holes, which is easily resolved.

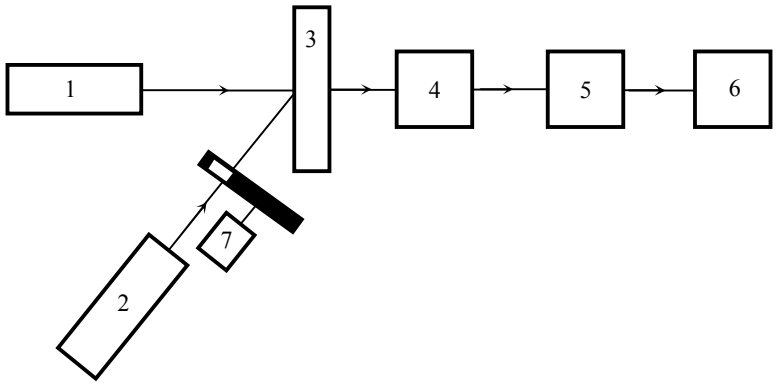


Fig.2. Scheme of experimental unit for separate identification of lifetime values of non-equilibrium electrons and holes

1 – source of probing radiation; 2 – source of injecting radiation; 3 – studied sample; 4 – photodetector; 5 – selective amplifier U2-8; 6 – synchronous detector V9-2; 7 – modulator of injecting radiation

Similarly, it is possible to obtain a system of two equations of the lifetime of electrons and holes, from which these two quantities are sepa-

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rately determined. The optical thickness of the sample can be changed in various ways: by turning, heating, and placing in an external interferometer. We used the rotation of the sample relative to the axis perpendicular to the probing beam.

We noted that laser interferometry makes it possible in one experiment not only to separately determine the nonequilibrium concentrations of electrons and holes and their lifetime, but also to make it non-contact and locally. For the first time the experimentally separate determination of the lifetimes of electrons and holes was carried out on germanium samples doped with antimony and compensated by an admixture of copper.

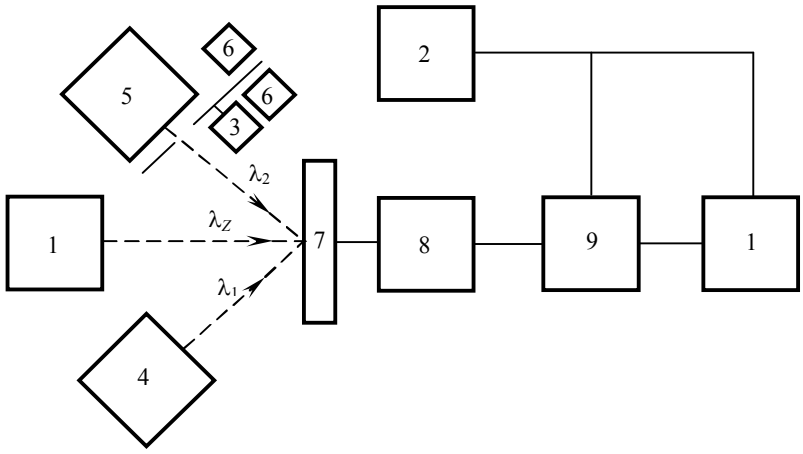


Fig.3. Scheme of experimental unit for measuring volume lifetime and velocity of surface recombination in silica samples

1 – laser LG-74; 2 – oscilloscope; 3 – obturator; 4 – optical injector based on light-emitting diodes AL-119A; 5 – optical laser-injector LG-126; 6 – optocoupler; 7 – studied sample; 8 – photoelectrical receiver; 9 – selective amplifier; 10 – synchronous detector

Determination of the volume lifetime and rate of surface recombination. Usually the sample surface is more defective than its volume. Therefore, the lifetime in a real sample depends on two quantities: the rate of recombination on the surface and the volume lifetime, which is an objective characteristic of the material. We proposed a method for noncontact laser-interferometric determination of the carrier lifetime, which is an objective characteristic of the material (in contrast to their lifetime in a given sample), and the determination of the rate of surface recombination of carriers [6, 19]. To carry out such investigations, it is necessary to have an optical injector with several wavelengths, at which the depths of the carrier generation regions are quite different.

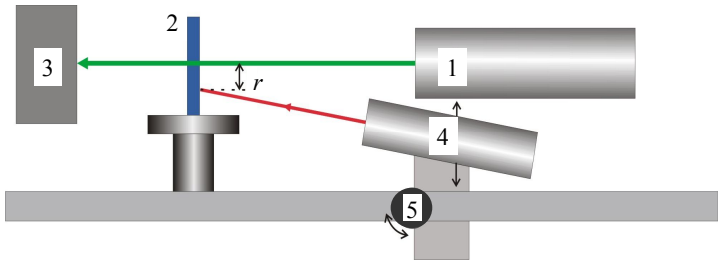


Fig.4. Scheme of experimental unit for controlling the diffusion length of charge carriers

1 – source of long-wave probing radiation; 2 – studied sample; 3 – photoelectrical receiver; 4 – source of short-wave injecting radiation; 5 – micrometric movement

The experiments (Fig.3) and calculations showed that measurements of the interference modulation of the probe beam, carried out at two different injector wavelengths (from which, for example, one corresponds to the surface one and the other, mainly to volume absorption), makes it possible to have two equations, their solutions define volume lifetime and rate of surface recombination.

Measurement of diffusion length of charge carriers. To measure such an important parameter of nonmetallic layers as the diffusion length of charge carriers, we had to complicate the scheme of the measuring device [9]. It was necessary to ensure that the beams of the injecting and probing lasers could be scattered along the surface of the sample at a controlled distance r (Fig.4). The proposed laser-interferometric method for measuring the diffusion length of charge carriers is a noncontact analog of the method of a traveling light probe with a point-sensing contact. Data on the diffusion length are obtained from the experimental dependence of the local concentration of excess charge carriers on the distance r between the point of its measurement and the carrier generation point by a short-wave laser 4. This distance is controlled by a micrometric movement 5. Injecting radiation from laser 4 generates additional charge carriers in sample 2 and leads to a change in the optical constants of the sample material and modulation of the longwave probing radiation transmit-

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ted through it, which is generated by the laser 1. This radiation is converted into an electrical signal by the photo-electric receiver 3. The detected signal decreases with increased distance *r* between the carrier generation points and the measurement of their concentration. The diffusion length is established by comparing the obtained experimental dependence with the analogous dependences calculated theoretically [30].

Determination of parameters of recombination centers. If one determines the lifetimes of both electrons and holes over a wide range of temperatures, in several cases it is possible to calculate such parameters of the recombination centers of electrons and holes as the cross sections for capture of electrons and holes at these centers, and the concentration of these centers. This type of research become more complicated due to the necessity of placing the sample in a cryostat cooled by liquid nitrogen with windows transparent in the middle infrared region (at a wavelength of 10.6 μm). Similar measurements were made on samples of cadmium-mercury-tellurium alloy, which is a material for creating infrared radiation receivers [19].

Methods of information signal processing. We proposed and patented various methods and optoelectronic schemes for processing the information signal for laser interferometry of semiconductors and gave their comparative analysis. In particular, when the lifetimes of electrons and holes are equal, the phase method is more favorable. The method is based on measuring the phase difference between the optical injector and the receiver of the probing radiation. This phase difference arises from the lag in the oscillations of the nonequilibrium concentration of carriers from the vibrations of the intensity of the radiation that generates them and carries information on the lifetime of the current carriers.

The amplitude method is based on measuring the amplitude of the variable component of the probing radiation transmitted through the sample (or reflected by it), which occurs when the sample is illuminated simultaneously with the light of an optical injector modulated at a fixed frequency. It is rational to use it when the lifetimes of electrons and holes are different, and when such parameters of the semiconductor, as its refractive index and the effective masses of electrons and holes, are known.

The frequency variant of laser interferometry is based on measuring the amplitude of the probing radiation after its interaction with the sample, depending on the frequency of the injecting radiation.

Requirements for the test samples. Usually ideas about interference measurements are associated with high requirements for the optical quality of the samples. Real semiconductor samples are not an ideal Fabry-Perot interferometer because of the lack of parallelism of the faces (wedging of the sample) and their roughness. Special mechanical preparation of samples for research changes their properties, including electrophysical ones. We theoretically investigated and experimentally proved the possibility of studying such nonideal samples. The wavelength of the probing laser can and should be large enough. Usually this is 10.6 μm (a laser on carbon dioxide), or 3.39 μm (helium-neon laser). At such values of the wavelength and the parameters of the sample imperfections can be of comparable magnitude. It turned out that the criterion for the suitability of samples for interference measurements is their contrast, i.e. the ratio of the maximum and minimum values of the interference dependence of the transmission of the probing beam. Its value should be at least 1.2. This corresponds to a possible heterogeneity of the sample over a thickness of 1.3 μm on the width of the probing beam, which is not a strict requirement. It is almost always in agreement with semiconductor plates (even polished) used in the manufacture of micro- and nano-electronic devices, and they do not require special preparation for measurements. The contrast is determined quite simply by measuring the interference curve observed when the sample is rotated about an axis perpendicular to the probing beam.

Serial measurements can also be carried out on polished samples, and their interferential quality is even higher than polished ones, due to better flatness.

Results of experiments. For the first time, the proposed measurement principle was used to study germanium crystals doped with antimony and having a specific resistance of 0.16 Ωm. It was found out that the lifetimes of electrons and holes differed by a factor of four and amounted to 0.8 and 0.2 microseconds, respectively [14].

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Experiments to determine the volume lifetime as an objective characteristic of the material and the rate of surface recombination characterizing the processing of a specific sample were carried out jointly with the staff of the Physical-technical Institute, they were done on single crystals of silicon with a resistivity of 130 Ωm. All samples were from the same billet but had different surface treatments. It was found that with an error of about 5%, the volume lifetime in all samples is the same and is 340 μs. The rate of surface recombination was determined by the sample surface treatment and varied from 103 cm/s for polished samples and to 105 cm/s for ground samples [6].

Together with the staff of the State Institute of Rare-Metal Industry, we investigated the technological possibilities of increasing the lifetime of charge carriers in indium antimonide single crystals using proposed methods. It was found that when the crystals are treated with atomic hydrogen in the surface layer of the sample at a depth of 8-15 μm, the lifetime increases by a factor of 6-8 compared with the volume one, which does not change and is approximately 4 μs. It was also found that the growth of single crystals in a strong magnetic field (B = 0.15 T) increases the volume lifetime of charge carriers in indium antimonide by approximately a factor of two [22].

In the alloy cadmium-mercury-tellurium $Cd_{0,3}Hg_{0,7}Te$, it was possible to calculate the concentration in the investigated material of the recombination centers from the experimental temperature dependences of the lifetimes obtained by the proposed method for electrons and holes, which amounted to $1.8 \cdot 10^{14}$ cm^{-3} [20].

In silicon single crystals with a specific resistance of 160 Ohm·m we investigated the diffusion length of charge carriers with laser-interferometric method. It was found that the protection of the silicon surface by a dielectric layer increases this parameter by a factor of 1.5 [27]. We also studied the super-pure silicon [26].

In addition to scientific applications, the proposed methods have made it possible to create a semiautomatic laser tau meter for industrial use, which allows to sort the cassette with 25 standard silicon wafers by the life time of carriers in 10 minutes.

Conclusion. Thus, the theoretical calculations and experimental studies have shown that the proposed physical mechanism of the absorption-interference interaction of two laser radiations with different wavelengths is a good basis for creating methods and means for non-contact monitoring of the electrical parameters of semiconductors and dielectrics.

They are the lifetime of charge carriers (for electrons and holes separately); parameters of recombination centers, namely their concentration and capture cross-sections; volume lifetime and rate of surface recombination of charge carriers, as well as their diffusion length of charge carriers. The measurements are local and non-destructive. Locality is determined by the diameters of the used laser beams. It is also important that no special preparation of samples is required for the measurement, it is possible to use plates directly from the technological process. Preservation of the material parameters after the measurements makes it possible to carry out technological processes on the samples, in particular, the manufacture of semiconductor devices.

The method has been successfully tested on a series of samples of such materials as germanium, silicon, indium antimonide and cadmium-mercury-tellurium alloy with various surface conditions, including those covered with transparent protective layers. It can be used in scientific research in the metallurgical and electronics industries.

At the same time, many well-known and newly created semiconductor materials and structures require a wide variety of methods and means of measurement, so the process of searching for and developing such methods continues [30, 31].

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