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Researches of temperature dependencies in volt-ampere characteristics of CdHgTe photodiode grown on GaAs, Si, and CdZnTe substrates

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The temperature dependences of CdHgTe photodiode current-voltage characteristics have been investigated. The influence of the substrate for epitaxy on the charge transfer mechanisms in semiconductor structures has been identified and studied. It has been revealed that CdHgTe photodiodes on Si substrates have an increased generation-recombination current compared to photodiodes grown on CdZnTe and GaAs substrates.

Keywords: photodiode structure, CdHgTe, volt-ampere characteristic (VAX), dark current.

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1. Introduction

The fabrication of infrared focal plane arrays with high quantum efficiency makes it necessary for developers to design new architectures of photosensitive elements (PSEs) based on cadmium mercury telluride solid solutions [1–4].

Analysis of the current-voltage characteristics (I–V curves) is a powerful tool for understanding the dark current mechanisms, optimizing device designs, and predicting performance characteristics [5, 6]. An important scientific and technical task is to study of the temperature dependence of the I–V curves of CdHgTe photodiodes, since the operating cryogenic temperature determines the ratio of various charge carrier transport mechanisms and, ultimately, the limiting characteristics of the FPAs.

2. Description of the samples and the mathematical model

A study investigated three samples of CdHgTe photodiode structures with a composition of $x = 0.39$ with a size of the p – n junction area of $6 \times 6 \mu\text{m}$ and a pitch of $30 \mu\text{m}$.

The photodiode arrays were fabricated using CdHgTe heterostructures obtained by molecular beam epitaxy (MBE) and liquid phase epitaxy (LPE) on cadmium zinc telluride (CdZnTe), silicon (Si), and gallium arsenide (GaAs) substrates (Table 1).

Table 1

Samples under study

Sample number	Substrate	Epitaxy method
Sample 1	CdZnTe	LPE
Sample 2	Si	MBE
Sample 3	GaAs	MBE

The temperature dependences of the dark current-voltage characteristics of four test photodiodes from each sample were measured; the measurement results are presented below.

The experimental data were approximated by the photodiode current model:

$$I = I_{\text{off}} + I_{01} \left[\exp \left(\frac{V - (I - I_{\text{off}}) R_s}{V_T} \right) - 1 \right] + I_{02} \left[\exp \left(\frac{V - (I - I_{\text{off}}) R_s}{2V_T} \right) - 1 \right] + \frac{V - (I - I_{\text{off}}) R_s}{R_{sh}},$$

where R_s is the series resistance, R_{sh} is the shunt resistance, I_{off} is the current offset, I_{01} is the diffusion-drift dark saturation current in the reverse branch of the photodiode I–V curve, I_{02} is the generation-recombination dark saturation current in the reverse branch of the photodiode I–V curve, V_T is the thermal voltage across the p – n junction, $V_T = \frac{kT}{q}$.

Variable parameters of the model: I_{01} , I_{02} , R_s , R_{sh} , I_{off} .

This model enables the separation of the diffusion-drift and generation-recombination components of the dark current, isolating them from the contribution of the substrate series

resistance and the shunt leakage current. The absence of a tunneling current component in the model is due to its small contribution to the dark current of CdHgTe photodiodes with a composition of $x = 0.39$ in the studied voltage range from -400 mV to 200 mV [7].

3. Results

Processing of the experimental data according to the photodiode current model made it possible to obtain the temperature dependences of the following parameters: the diffusion-drift saturation current in the reverse branch of the photodiode I–V curve (Fig. 1), the generation-recombination saturation current (Fig. 2).

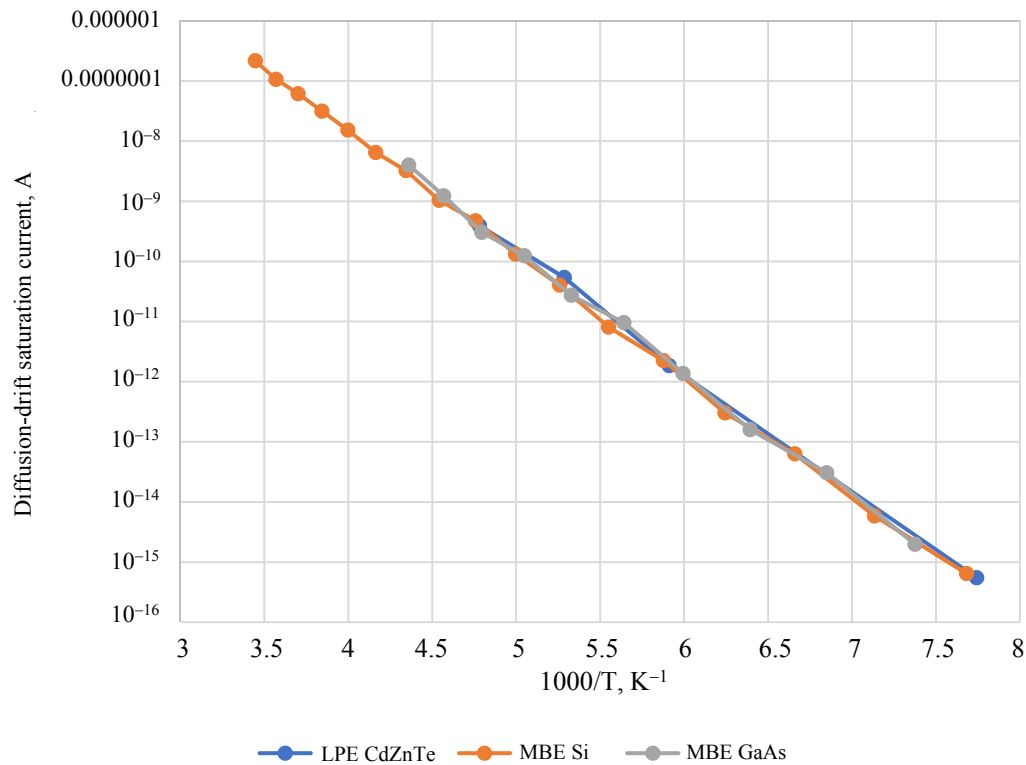


Fig. 1. Temperature dependence of the approximation parameter – the diffusion-drift dark saturation current in the reverse branch of the photodiode I–V curve (I_{01})

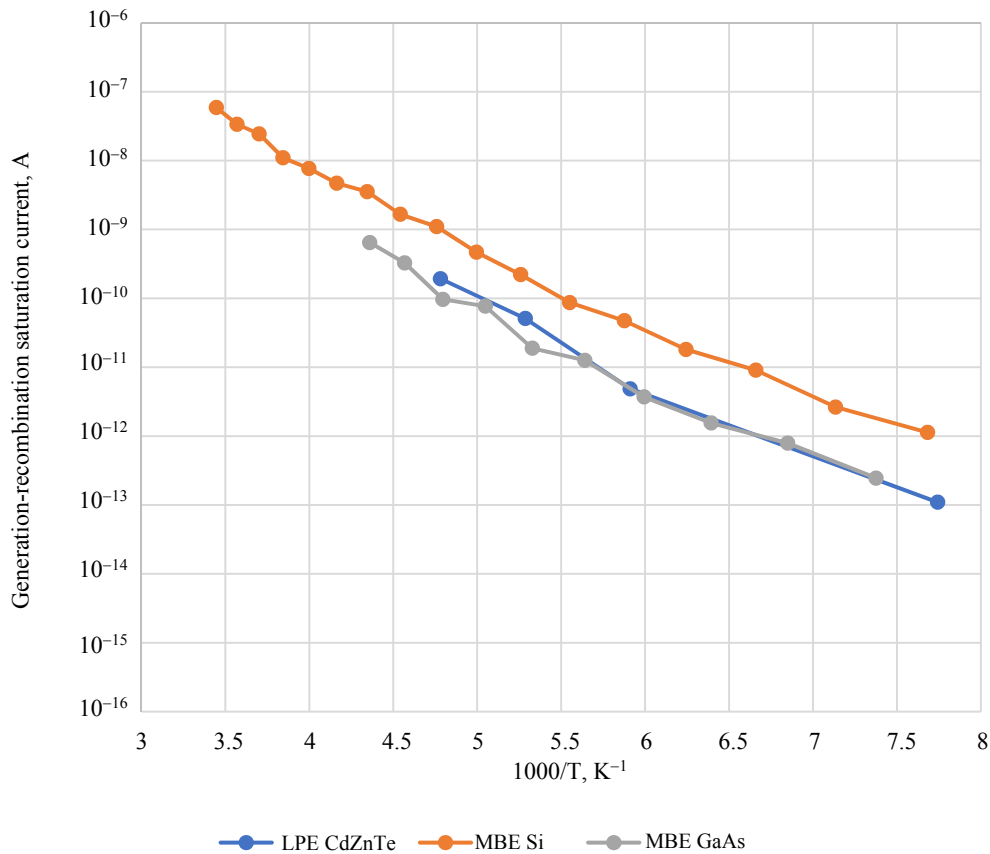


Fig. 2. Temperature dependence of the approximation parameter – the generation-recombination dark saturation current in the reverse branch of the photodiode I – V curve (I_{02})

According to the classical monograph by Sze [8], the following estimates of the temperature dependences of the dark current are valid:

$$I_{01} \sim q \sqrt{\frac{D_p}{\tau_p}} * \frac{n_i^2}{N_d} \sim T^{\frac{3+\gamma}{2}} \exp\left(-\frac{E_g}{kT}\right), \quad (1)$$

where D_p is the hole diffusion coefficient, τ_p is the hole lifetime, n_i is the intrinsic charge carrier concentration, N_d is the donor concentration in the n -region, E_g is the band gap, γ is a constant determined by the temperature dependence of

$$\frac{D_p}{\tau_p} \sim T^\gamma;$$

$$I_{02} \sim \frac{qn_i W}{\tau_e} \sim n_i \sim T^{\frac{3}{2}} \exp\left(-\frac{E_g}{2kT}\right), \quad (2)$$

where W is the width of the depletion layer, τ_e is the effective charge carrier lifetime.

As shown in Fig. 1, the diffusion-drift component of the dark current exhibits the same exponential temperature dependence for all three samples. Such a result expectedly follows from Equation (1), since the exponential term exerts a dominant influence compared to the power-law part, and the bandgap of the samples is the same.

The generation-recombination component of the dark current also exhibits a near-exponential temperature dependence for all samples; however, for sample 2 the parameter I_{02} is an order of magnitude larger than for the other samples.

According to the Shockley–Read–Hall (SRH) theory, the increased generation-recombination current in photodiodes on the silicon substrate may be due to a larger density of deep SRH centers in the middle of the bandgap, resulting from the lower structural perfection of the CdHgTe heterostructures grown on a silicon. . The large lattice mismatch and the difference in the space groups of the substrate and CdHgTe

epilayer induce a higher threading dislocation density, with dislocations serving as SRH recombination centers. The characteristic parameters of the crystal lattices of the materials used in the study are presented in Table 2.

The total dark saturation current in the reverse branch of the I–V curve, excluding the contribution of the series and shunt resistances, is calculated as the sum of the above current components

$$I_0 = I_{01} + I_{02}.$$

When considering the dependence of I_0 on temperature (Fig. 3), it can be noted that for the operating temperature $T = 170$ K (5.88 1/mK) the dark current of sample 2 (CdHgTe heterostructure on Si) is an order of magnitude greater than the dark current of samples 1 (CdHgTe heterostructure on CdZnTe) and 3 (CdHgTe heterostructure on GaAs). To achieve the same dark current level as that of photodiodes on GaAs and CdZnTe substrates, the photodiode on a silicon substrate must be cooled by 23 K lower compared to samples of photodiodes 1 and 3.

Table 2

Parameters of the materials used in the study

	Lattice constant	Lattice type	Space group	Dislocation density in CdHgTe
Si	5.431	diamond-like cubic	Fd-3 m	10^6 – 10^7
GaAs	5.653	zinc blende	F-43 m	10^5 – 10^6
CdZnTe	6.46–6.48	zinc blende	F-43 m	10^4 – 10^5
CdHgTe	6.461–6.482	zinc blende	F-43 m	—

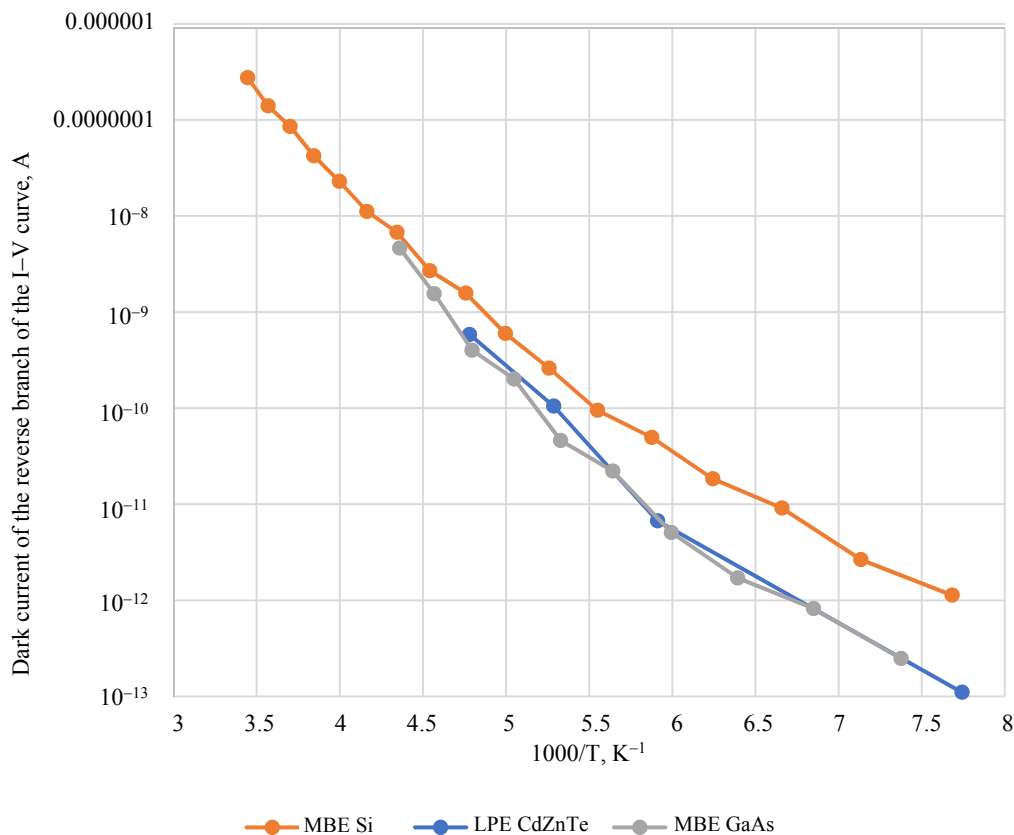


Fig. 3. Dependence of the total dark current I_0 on temperature for photodiode structures fabricated in CdHgTe heterostructures grown on Si, CdZnTe, and GaAs substrates

4. Conclusions

In the temperature range from 130 K to 290 K, the generation-recombination saturation current and the dark current of CdHgTe photodiodes on a Si substrate are an order of magnitude higher compared to those on GaAs and CdZnTe substrates. Thus, to achieve the same dark current value under the operating reverse bias voltage, sample 2 must be cooled by 23 K lower than the other samples.

Comparing this findings with the lattice parameters of the studied materials, it can be assumed that the discrepancies originate from the varying dislocation densities in the MCT material grown on a CdZnTe substrate matched in crystal structure and on alternative Si and GaAs substrates.

This conclusion is further validated by a qualitative analysis of the shunt resistance, which reveals higher leakage currents for the CdHgTe photodiodes on Si substrates compared to photodiodes on CdZnTe and GaAs substrates.

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The influence of growth modes on the characteristics of germanium-silicon microresonators on a thick silicon oxide layer

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In this study dependences of the structural parameters of germanium microresonators formed on an oxidized silicon surface (average island size, island size distribution, island height-to-diameter ratio, and island surface density) on post-growth annealing conditions were determined. Optical characteristics of the resulting heterostructures with microresonators were investigated. Their potential for use as absorbing structures as well as antireflection coatings for certain wavelengths was demonstrated. Methods for controlling the geometric and optical characteristics of microresonator ensembles by varying the parameters of their epitaxial synthesis were proposed.

Keywords: microresonators, silicon, germanium, molecular beam epitaxy, atomic force microscopy, transmission spectra, reflectance spectra.

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Introduction

Microresonators (Mie resonators) are capable of localizing and enhancing the electromagnetic field at certain resonant wavelengths. When integrated with quantum dots, such structures provide a substantial enhancement of the optical response, which increases the efficiency of detecting weak signals in photosensitive devices [1, 2].

An important factor determining the functional capabilities of microresonators is their antireflective properties, which make it possible to increase the efficiency and sensitivity of optoelectronic devices when integrated into active structures. The placement of microresonators on a surface leads to a significant reduction in the reflection coefficient in certain spectral ranges, which are set by their geometric parameters. This effect is due to the occurrence of Mie scattering

during the interaction of electromagnetic radiation with surface inhomogeneities whose characteristic dimensions are comparable to the wavelength of the incident radiation [3].

As a result of Mie scattering, the character of radiation reflection from the surface changes: instead of predominantly specular reflection, diffuse scattering arises, in which part of the incident radiation is redistributed in various directions. At the same time, a significant fraction of the energy propagates along the surface of the structure, promoting more efficient input of radiation into the material. Such a mechanism leads to an increase in transmission and a reduction in reflection losses over a broad spectral range [1].

Molecular beam epitaxy (MBE) makes it possible to obtain pure nanostructures with a minimal number of defects [4]. Reflection high-energy electron diffraction (RHEED) is used to

analyze the surface morphology of the samples [5]. RHEED makes it possible to monitor the surface morphology during growth with an accuracy of high precision (less than one monolayer (ML)) [6]. Control of epitaxial processes by changing the temperature and deposition rate and by using special substrates makes it possible to create self-organized structures with the required parameters and desired properties [7]. Recently, particular attention has been paid to the development and epitaxial creation of photosensitive elements [8, 9] based on germanium-silicon microresonators, including the creation of antireflective coatings on their basis [10, 11].

Methods

In the present work, germanium microresonators were created on an oxidized silicon surface by molecular beam epitaxy followed by annealing under ultrahigh vacuum conditions. Si(001) wafers were used as substrates for germanium deposition. The experimental studies were carried out on a Katun-100 MBE setup. After the pre-epitaxial preparation of the Si(001) wafer, a thick oxide layer of SiO₂ (about 100 nm) was deposited on it by the magnetron method. Then, a GeSi solid solution of various thicknesses with a Ge percentage content of 30% was deposited on the oxidized substrate. The deposition rate was determined by the method of reflection high-energy electron diffraction from the intensity oscillations during the deposition of materials onto a Si(001) substrate without an oxide layer. Deposition occurred at a temperature of 400 °C to exclude Ge segregation and create a uniform layer. After the deposition of the germanium and silicon layers, the obtained structures were annealed at a temperature of 800 °C with various annealing durations.

The state of the surface during growth was monitored using the method of reflection high-energy electron diffraction. After synthesis, the surface morphology was studied by atomic force microscopy. The optical characteristics of the obtained structures were studied by the method of optical spectrometry for diffuse reflection using a Shimadzu UV-3600 Plus spectrophotometer. The measured spectra were compared with the

calculated ones obtained in the Lumerical environment by the finite-difference time-domain method.

Results

Judging by the data of the RHEED studies carried out during the entire experiment, the GeSi film before annealing had an amorphous structure, as evidenced by the presence of two broad blurred diffuse halos. During annealing, the structure became polycrystalline: Debye–Scherrer rings were formed on the RHEED patterns. Upon completion of annealing, reflections additionally appeared on the patterns, corresponding to transmission diffraction from the periodic structure.

As a result of the annealing of structures with a deposited layer of silicon-germanium solid solution on an oxidized silicon surface, an ensemble of nanoislands was formed with characteristic sizes of 150–250 nm and a height of 100–250 nm. Figure 1 shows an SEM image of a sample with a deposited solid solution layer (effective thickness 33 nm) that was annealed at a temperature of 800 °C for 1 hour. Analysis of the image makes it possible to estimate the surface density ($\sim 1 \times 10^9 \text{ cm}^{-2}$) and the size distribution of the islands. The average size of the islands under these annealing conditions was 200 nm.

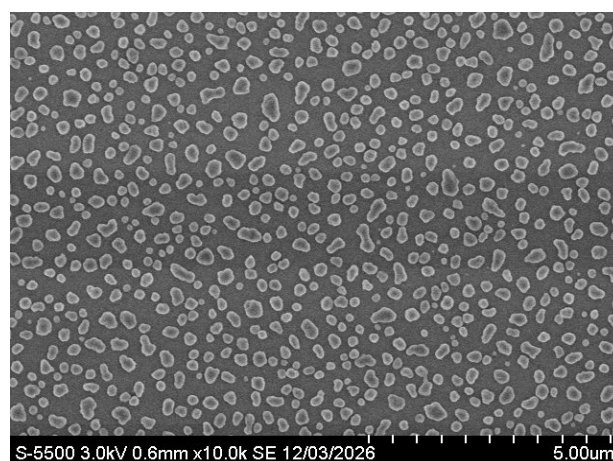


Fig. 1. SEM image of a sample with germanium-silicon microresonators (effective thickness 33 nm) on magnetron silicon oxide

For a more detailed study of the geometric characteristics of the obtained nanoislands

(including their height), the samples were examined by atomic force microscopy in the semi-contact mode using a cantilever with a tip radius of 2 nm. The study of the morphological characteristics of the formed microresonators by atomic force microscopy made it possible to determine the distributions of height and base diameter, as well as to estimate their surface density. Figure 2 shows the dependences of the average base diameter (d) and the height-to-diameter ratio (h/d) on the thickness of the deposited silicon-germanium layer for samples annealed at 800 °C for 1 hour.

In accordance with Figure 2, with an increasing effective thickness of the deposited solid solution, the island size increases. The island diameter d increases almost linearly, while

the height-to-diameter ratio, h/d , grows nonlinearly, gradually approaching unity.

Experiments on varying the parameters of post-growth annealing showed that an increase in the annealing temperature or duration leads to an increase in the diameter and a decrease in the parameter h/d .

To study the optical response of the structures, transmission and diffuse reflection spectra were recorded over a broad spectral range. As an illustration, Figure 3 presents the diffuse reflection spectra of two samples with average island sizes of 150 and 200 nm. A clear resonant character in the reflection spectra of such nanoislands is observed, as well as a substantial dependence of the spectral profile on the sizes of the microresonators.

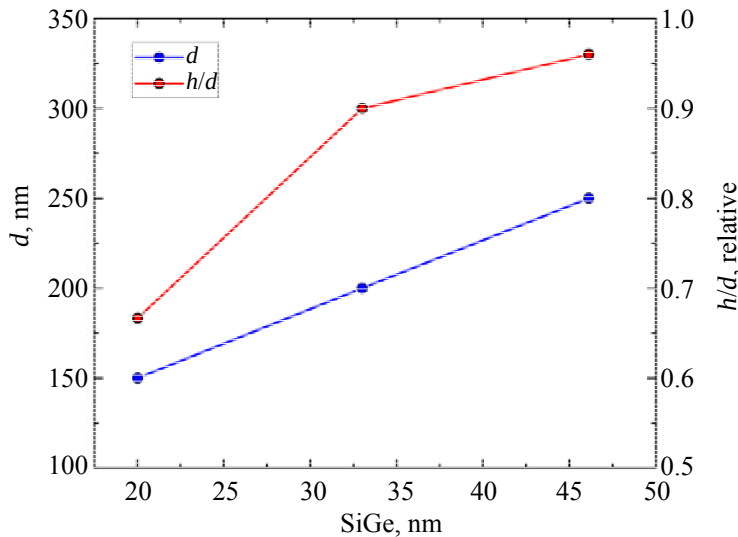


Fig. 2. Dependence of the base diameter d (blue curve) and the height-to-base-diameter ratio h/d (red curve) on the thickness of the deposited silicon-germanium solid solution

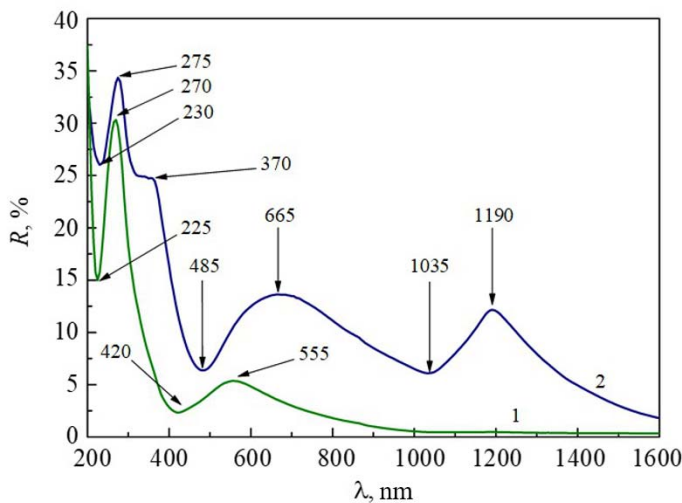


Fig. 3. Experimental diffuse reflection spectra of structures with silicon-germanium microresonators on a thick silicon oxide layer with an average size of 150 nm (curve 1) and 200 nm (curve 2)

In the reflection spectra, alternating maxima and minima are observed, caused by the

excitation of Mie resonances in the nanoislands. At the same time, for the initial silicon surface,

the contribution of diffuse reflection is negligible, since the reflection is predominantly specular in nature. Outside the resonance regions, the transmission spectra of the structures with microresonators practically coincide with the transmission of the initial silicon substrate.

The results of numerical modeling of the optical characteristics of layers with microresonators having diameters of 150 and 200 nm and a height-to-diameter ratio $h/d = 1$ are

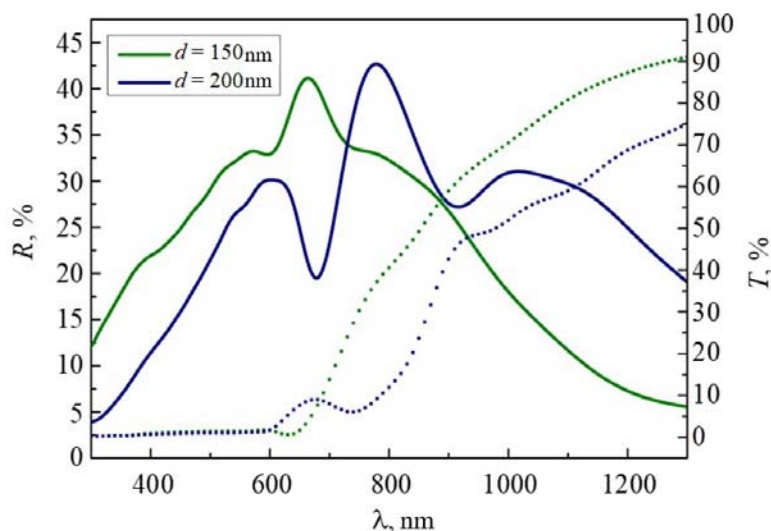


Fig. 4. Calculated reflection spectra (solid lines) and transmission spectra (dashed lines) of a layer with microresonators having diameters of 150 and 200 nm

Comparison of the experimental and calculated data (Figures 3 and 4) demonstrates qualitative agreement in the presence of intensity extrema only in a narrow wavelength range of 550–650 nm. The observed differences in the absolute values of the reflection and transmission coefficients, as well as in the number and width of the resonance peaks, are due to the inevitable spread in the sizes of the formed islands, which leads to smoothing of the spectral features.

Measurements of the reflection spectra performed at various angles of incidence and radiation detection did not reveal a significant angular dependence of the intensity. This indicates the predominantly diffuse character of reflection in structures with GeSi microresonators on an oxidized silicon surface. Analysis of samples with different values of h/d shows that a reduction in reflection and an increase in transmission are achieved with an increase in the ratio of the height of the microresonators to the diameter of their base. This is due to the improvement of the conditions for the simultaneous manifestation of electric and magnetic resonances in such structures [11].

presented in Figure 4. In the calculated spectra, the features associated with size resonances manifest themselves more clearly. It is shown that an increase in the island diameter leads to the appearance of additional resonant extrema in the reflection spectra. Nevertheless, the calculated data so far provide only preliminary information about the real spectra, since they do not take into account the large spread in islands size that inevitably exists in the grown samples.

Conclusion

As a result of the study, the dependences of the structural parameters of the formed germanium microresonators on a thick silicon oxide layer (average island size, island size distribution, height-to-island-diameter ratio, surface density of islands) on the post-growth annealing regimes were determined. The optical characteristics of the obtained heterostructures with microresonators were also determined. The prospects of their application not only as absorbing structures, but also as antireflective coatings for certain wavelengths are demonstrated. Ways to control the geometric and optical characteristics of the ensemble of microresonators by changing the parameters of their epitaxial synthesis are proposed.

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Influence of a strained InAlSb barrier on electron and hole transport in an InSb-based barrier-diode heterostructure

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This study presents a simulation of an InSb/InAlSb nBn heterostructure intended for use in mid-wave infrared photodetectors. The influence of the parameters of a strained InAlSb barrier layer on the band profile of the structure and on charge carrier transport is investigated. Particular attention is paid to achieving selective suppression of electron current while maintaining efficient hole transport, which is a key requirement for reducing dark current and increasing the operating temperature of photodetectors. The simulations were performed using the COMSOL Multiphysics software environment. Various barrier-layer designs were analyzed, and the configurations providing the most favorable balance of barrier properties for majority and minority charge carriers were identified. The obtained results can be used in the design of InSb photodetectors based on the nBn architecture for the mid-wave infrared range.

Keywords: IR photodetector, InSb, simulation, COMSOL Multiphysics, molecular beam epitaxy, barrier-diode heterostructures.

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Introduction

Infrared (IR) focal plane arrays are a basic element of modern thermal imaging and thermal radiation detection systems [1]. Increasing requirements for their sensitivity, speed, and energy efficiency make the development of new materials and device architectures for the mid-infrared range urgent.

One of the most common materials for creating mid-IR photodetectors is indium antimonide, a narrow-gap A_3B_5 semiconductor with a bandgap of about 0.23 eV at 80 K [2]. InSb remains one of the most in-demand materials for manufacturing high-speed photodetectors operating in the 3–5 μm range. On this basis, large-format photosensitive arrays have been created, and the development of epitaxial

technologies, in particular molecular beam epitaxy (MBE), has made it possible to improve the structural perfection of layers, reduce background impurity levels, and improve material uniformity [3].

However, the effective operation of InSb photodetectors, typically, requires cooling to cryogenic temperatures, since with increasing operating temperature the dark current sharply increases and the core device characteristics deteriorate [1, 2]. This leads to an increase in the mass, cost, and power consumption of the equipment. Therefore, a key direction in the development of InSb-based IR photodetectors is the creation of devices using barrier-diode heterostructures (nBn) [4]. A selective barrier that blocks the majority charge carriers (electrons) current makes it possible to reduce the dark

current without preventing the free movement of photogenerated holes in the valence band. Thus, an increase in the operating temperature is achieved without a significant deterioration in performance characteristics.

The most common material for the barrier layer of InSb-based structures is InAlSb [5]. This is primarily due to the successful combination of good lattice-parameter matching and a wide bandgap, which makes it possible to form a high potential barrier. This allows electrons to be effectively localized in InSb, reducing their leakage and suppressing parasitic currents, which is especially important for highly sensitive and high-speed devices. In addition, InAlSb provides high heterointerface quality and makes it possible to flexibly control the band structure by changing the layer composition. The study of its characteristics is fundamentally important, since barrier properties – such as the bandgap and band offset heights – largely determine the operating characteristics of the device. At the same time, strained InAlSb barrier layers, despite their practical importance for controlling the band profile and transport properties of the heterostructure, have so far been poorly studied. In this regard, it is of interest to analyze such strained barriers and assess their influence on electron and hole transport in nBn -type structures.

The present work is devoted to the modeling of an InSb/InAlSb heterostructure of the nBn type using the COMSOL Multiphysics software package. The work considers various designs of the InAlSb barrier layer and analyzes their influence on the transport of majority and minority charge carriers. Particular attention is paid to the choice of a barrier configuration that ensures effective suppression of electron transport while maintaining unimpeded hole transport. The obtained results make it possible to determine the optimal design of the barrier layer for application in mid-IR photodetector structures.

Band structure and strain-induced shifts of energy levels in InSb and InAlSb

The barrier-diode structures studied in the present work are traditionally grown by MBE. This is due to the need for precision control of the heteroepitaxial growth parameters that determine the band diagram and transport properties of the

structure. The MBE method provides high accuracy in specifying layer thicknesses, solid solution compositions, and doping profiles, as well as forming sharp and morphologically perfect heterointerfaces. This is especially important for narrow-gap InSb/InAlSb systems, in which even insignificant deviations in composition, barrier layer thickness, or interfacial blurring lead to a noticeable change in potential barrier heights, the position of band edges, and the carrier transport conditions. An additional advantage of MBE is the possibility of controlling the growth of strained and pseudomorphic layers with a reduced density of structural defects, which is fundamentally important for realizing the calculated energy profile of nBn structures and the reproducibility of their electrophysical and photoelectric characteristics.

At the same time, sufficiently thin InAlSb layers grown on InSb are formed in a pseudomorphic state, meaning they retain coherent conjugation with the substrate without the relaxation defect formation. This is due to the small lattice-parameter mismatch and is confirmed by structural data obtained both within the framework of this research direction by our scientific group in [6] for a 70-nm-thick $\text{In}_{0.8}\text{Al}_{0.2}\text{Sb}$ layer, and by independent teams in [5] for a 50-nm-thick graded $\text{In}_{1-x}\text{Al}_x\text{Sb}$ layer (*where* x gradually increased from 0.15 to 0.35). Establishing the pseudomorphic nature of growth is fundamentally important, since it is precisely in this case that the elastically strained state of the barrier layer is realized, which modifies the band structure and the energy profile of the heterosystem.

Further in this work, only fully pseudomorphic InAlSb barrier layers are considered, for which the coherent nature of growth is ensured either by reducing the aluminum content, and consequently, reducing the lattice-parameter mismatch with InSb – or by reducing the layer thickness. Such a limitation makes it possible to analyze heterostructures within the framework of the model of a fully elastically strained barrier model without considering relaxation and the its associated defects.

For a pseudomorphic InAlSb layer on InSb, the condition of equality of lattice parameters

($a_{\parallel} = a_{sub}$) is satisfied. Here $a_{\parallel}$ is the lattice parameter in the interface plane. Then the in-plane strain of the layer is determined by the expression

$$\varepsilon_{\parallel} = (a_{\parallel} - a_{layer}) / a_{layer},$$

where a_{layer} is the lattice parameter of the unstrained layer. The out-of-plane strain, within the framework of the linear theory of elasticity for cubic crystals [7], takes the form

$$\varepsilon_{\perp} = -2(C_{12} / C_{11}) \cdot \varepsilon_{\parallel},$$

where C_{11} and C_{12} are the elastic constants of the layer material. The values of the constants used in the present work, here and below, are taken from source [8]. To describe the elastic properties of the $\text{In}_{1-x}\text{Al}_x\text{Sb}$ solid solution, the elastic constants C_{11} and C_{12} can be estimated using Vegard's rule:

$$C_{11}(\text{In}_{1-x}\text{Al}_x\text{Sb}) = (1-x) \cdot C_{11}(\text{InSb}) + x \cdot C_{11}(\text{AlSb}),$$

$$C_{12}(\text{In}_{1-x}\text{Al}_x\text{Sb}) = (1-x) \cdot C_{12}(\text{InSb}) + x \cdot C_{12}(\text{AlSb}).$$

The presence of pseudomorphic deformation significantly affects the electronic structure of $\text{In}_{1-x}\text{Al}_x\text{Sb}$. In particular, strain leads to a change in the band gap, the splitting of heavy and light hole states, and shift in the effective masses of charge carriers and, accordingly, their mobilities.

The hydrostatic component of strain is determined as the trace of the strain tensor:

$$\varepsilon_h = \varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz} = 2\varepsilon_{\parallel} + \varepsilon_{\perp}.$$

In turn, the biaxial shear component of strain, responsible for lifting the degeneracy of the valence band, is given by the expression:

$$\varepsilon_b = \varepsilon_{\perp} - \varepsilon_{\parallel}.$$

To calculate the strain-induced shifts of the band edges, the deformation potentials a_c , a_v , and b are used:

$$\Delta E_c = a_c \cdot \varepsilon_h,$$

$$\Delta E_v(hh) = a_v \cdot \varepsilon_h - b \cdot \varepsilon_b,$$

$$\Delta E_v(lh) = a_v \cdot \varepsilon_h + b \cdot \varepsilon_b.$$

For the $\text{In}_{1-x}\text{Al}_x\text{Sb}$ solid solution, the values of the deformation potentials were determined in a linear approximation with respect to composition:

$$a_c(\text{In}_{1-x}\text{Al}_x\text{Sb}) = (1-x) \cdot a_c(\text{InSb}) + x \cdot a_c(\text{AlSb}),$$

$$a_v(\text{In}_{1-x}\text{Al}_x\text{Sb}) = (1-x) \cdot a_v(\text{InSb}) + x \cdot a_v(\text{AlSb}),$$

$$b(\text{In}_{1-x}\text{Al}_x\text{Sb}) = (1-x) \cdot b(\text{InSb}) + x \cdot b(\text{AlSb}).$$

The absolute positions of the conduction band and valence band edges of the materials relative to vacuum are determined by their χ electron affinity and the band gap E_g . At a temperature of 300 K, for the binary compound InSb the electron affinity is $\chi(\text{InSb}) = 4.59$ eV with a band gap $E_g = 0.17$ eV. For AlSb, the electron affinity is $\chi(\text{AlSb}) = 3.6$ eV with a fundamental band gap $E_g(X) = 1.62$ eV. However, for the $\text{In}_{1-x}\text{Al}_x\text{Sb}$ solid solution, it is necessary to take into account that with increasing Al content, approximately starting from $x \approx 0.45$, a transition from a direct-gap to an indirect-gap structure occurs. Therefore, when analyzing the band diagram, the dependences $E_g(\Gamma)$ and $E_g(X)$ should be considered separately: at small x the conduction band minimum is located at the Γ point, whereas at large x it is at the X point. Since the band gap of AlSb in the Γ valley is about 2.3 eV, to estimate the position of the conduction band level at the Γ point one can take $\chi \approx 2.92$ eV.

For the $\text{In}_{1-x}\text{Al}_x\text{Sb}$ solid solution, the χ dependence on composition in the first approximation can be described by Vegard's law:

$$\chi(\text{In}_{1-x}\text{Al}_x\text{Sb}) = (1-x) \cdot \chi(\text{InSb}) + x \cdot \chi(\text{AlSb}).$$

The influence of strain on electron affinity is due to the strain-induced shift of the conduction band. Under tensile strain, the bottom of the conduction band shifts downward in energy, which leads to an increase in electron affinity by the $\Delta E_c = a_c \cdot \varepsilon_h$ amount.

On the basis of the proposed model, energy diagrams of InSb/InAlSb *nBn* structures were constructed, consisting of an InSb absorbing layer 1 μm thick, two InSb contact layers 200 nm thick each, and an InAlSb barrier layer (see Fig. 1). The thickness of the barrier layer was determined depending on the Al content from the condition of preserving the pseudomorphic state.

The doping impurity concentration (*n*-type) was 10^{16} cm^{-3} in the sensitive layer, $2 \times 10^{16} \text{ cm}^{-3}$ in the upper contact layer, $4 \times 10^{16} \text{ cm}^{-3}$ in the lower contact layer, and 10^{14} cm^{-3} in the barrier layer. The temperature was set equal to 150 K.

Figure 1 shows that for all the considered structures the barrier manifests itself primarily as a jump in the conduction band. With an increase

in Al content from $x = 0.1$ to $x = 0.4$, the height of the electron barrier successively increases. In contrast to the conduction band, the valence band offset changes nonmonotonically and has a substantially smaller magnitude. For structures with $x = 0.1$ and $x = 0.2$, the formation of a potential well for holes is observed. At $x = 0.3$, the value of ΔE_v is close to zero, whereas at $x = 0.4$ a small barrier for holes arises. The nonmonotonic nature of the dependence of ΔE_v (in the inset to Fig. 1) is due to the competition of two factors: an increase in the valence band offset with growing Al content in the barrier layer and the contribution of the biaxial shear component of deformation, which leads to the lifting of the degeneracy of the valence band.

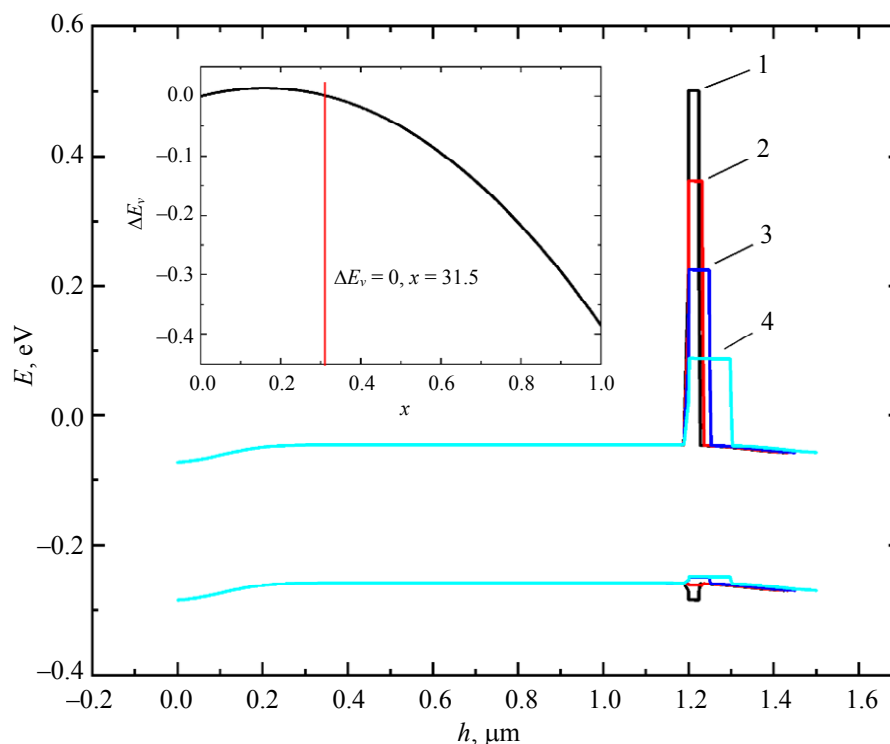


Fig. 1. Energy diagrams of InSb/In_{1-x}Al_xSb *nBn* structures with different barrier layers:

1 – $x = 0.4$, $h = 25 \text{ nm}$; 2 – $x = 0.3$, $h = 33 \text{ nm}$; 3 – $x = 0.2$, $h = 50 \text{ nm}$; 4 – $x = 0.1$, $h = 100 \text{ nm}$.

In the inset – analytically calculated dependence of the valence band offset of strained In_{1-x}Al_xSb on the Al content

For all the studied InSb/In_{1-x}Al_xSb *nBn* heterostructures, the temperature dependences of the electron and hole currents were calculated per pixel with a size of $15 \times 15 \mu\text{m}^2$. The modeling results are presented in Figure 2 for structures with different compositions and thicknesses of the barrier layer. Figure 2 shows that the electron current monotonically increases with temperature for all the considered structures, but its magnitude

depends substantially on the barrier layer parameters. With an increase in the Al content in the barrier, the electron current decreases by several orders of magnitude over the entire studied temperature range. This indicates enhanced barrier suppression of majority carrier transport with increasing barrier height due to the higher x content in the In_{1-x}Al_xSb layer.

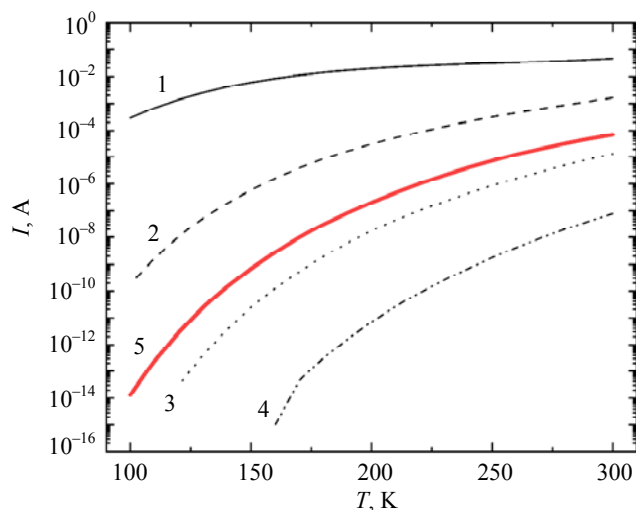


Fig. 2. Temperature dependences of the electron (black) current of InSb/In_{1-x}Al_xSb nBn structures with different barrier layers: 1 – $x = 0.1$, $h = 100$ nm; 2 – $x = 0.2$, $h = 50$ nm; 3 – $x = 0.3$, $h = 33$ nm; 4 – $x = 0.4$, $h = 25$ nm, as well as the common red curve 5 for the hole current of all structures

At the same time, the hole currents for all structures practically coincide, forming a single temperature dependence. Such a weak sensitivity of the hole current to changes in the barrier layer parameters indicates that the transport of minority carriers in the studied nBn structures remains practically unchanged when the composition and thickness of the barrier are varied.

At the same time, for structures with $x = 0.3$ and 0.4 , a transition to the regime $I_e < I_h$ is

observed, indicates the achievement of an effective electron-blocking configuration, making it the most favorable for the practical implementation of nBn devices.

Conclusions

This work investigated the influence of elastic deformation on the energy spectrum of InSb/In_{1-x}Al_xSb heterostructures, and established its role in the formation of band offsets at the heterointerface. It is shown that a change in the composition of the barrier layer leads to a substantial rearrangement of the band diagram, and the specific Al content at which the valence band offset ΔE_v disappears has been determined. The performed modeling of the current characteristics showed that the use of a pseudomorphic barrier layer with $x = 0.3$ – 0.4 makes it possible to effectively suppress the valence band offset, limit the transport of majority charge carriers, and achieve a regime in which the dark current is determined predominantly by the heavy-hole contribution. The obtained results confirm the promise of using pseudomorphic In_{1-x}Al_xSb barriers with increased Al content for optimizing InSb-based nBn structures and reducing the contribution of the electron component to the dark current.

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Electron beam generation efficiency in a high-voltage abnormal DC glow discharge in helium, neon, and argon

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The aim of this work was to carry out a comparative study of the current-voltage characteristics and the energy efficiency of electron beam generation in a high-voltage abnormal DC glow discharge in helium, neon, and argon at voltages above 1000 V. It is shown that, under ultraclean conditions and with thorough degassing of the cathode material, the current-voltage characteristics acquire a nonmonotonic form, while the total efficiency of electron beam generation reaches approximately 80 % in helium, approximately 90 % in neon, and 40 % in argon. The results of this work open up prospects for increasing the efficiency of electron beam generation in existing high-voltage discharge generators for applications in plasma technologies.

Keywords: gas discharge, helium, neon, argon, electron beam, efficiency.

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Introduction

Gas-discharge electron beam (EB) generators are of interest as compact sources of fast electron fluxes formed directly in a gas medium due to the acceleration of charged particles in strong electric fields [1–3]. The generation of EBs directly in a gas medium is a promising direction for the development of electron-beam and plasma technologies, since it makes it possible to dispense with the differential pumping systems and foil windows characteristic of vacuum accelerators. At the same time, considering both pulsed and stationary generators based on various types of gas discharges indicates the relevance of a comparative study of the mechanisms of beam formation, its energy efficiency, and the operating pressure ranges for discharges of different types. The simplest

representative of discharge types – the abnormal glow discharge (AGD) of direct current in inert gases – is characterized by a monotonically increasing current-voltage characteristic [4–6]. Works [7, 8] showed that, in high-voltage AGD in helium at pressures of 2–10 Torr, under conditions of minimized controlled and uncontrolled impurities in both the working medium and the cathode material, the current-voltage characteristics develop regions of negative differential resistance as the pressure increases. The electron-beam generation efficiency (EB) reaches about 80% [7].

Understanding the features and processes occurring in such fairly simple types of discharges is relevant for the development of plasma technologies. The current-voltage characteristics of the AGD depend to a large extent on the realized experimental conditions

[8]. Under uncontrolled conditions where the gas medium contains molecular components and the cathode surface is insufficiently cleaned, the current-voltage characteristics have a typical classical monotonically increasing form. Under clean conditions and with thorough degassing of the cathode material, the current-voltage characteristics acquire an *S*-shaped form in voltage U – current I coordinates. Contamination of the cathode surface and the presence of molecular components in the gas medium lead to a change in the role, composition, and energy of the particles bombarding the cold cathode. Under the condition that the emission properties of the cathode depend on the modification of the emitting layer due to the implantation of working particles into the surface, the functional parameters of the AGD (discharge voltage, current, the nature of its flow, and the efficiency of EB generation) will be determined by the purity of the working medium and the state of the emitting surface.

It is therefore of interest to investigate high-voltage AGD in other inert gases. The aim of the present work is a comparative study of the current-voltage characteristics and the energy efficiency of EB generation in an AGD in helium, neon, and argon in the direct current regime at elevated (more than 1000 V) voltages under conditions where controlled and uncontrolled impurities of the working medium and the cathode material are minimized.

Experimental Setup

The studies of the discharge were carried out in a discharge cell with flat polished electrodes: a cathode made of reaction-sintered silicon carbide and a titanium anode. The interelectrode distance was 3 cm. The electrodes were separated by a glass cylinder, as well as by a multilayer insulator made of Al_2O_3 ceramic plates placed near the cathode. The inner diameters of the holes in the plates were 1.6 and 2.3 cm, which limited the area of the active discharge zone to 2 cm^2 . The temperature of the cell was monitored during the experiment and maintained at a level of $T \leq 50^\circ\text{C}$ through the use of cooling elements – radiators and coolers.

The experiments were carried out in the gas-flow regime, which was supplied through a carbon

trap cooled with liquid nitrogen at a rate of $\sim 100\text{ ml/min}$. Inert gases were used with a volume fraction of at least: helium He – 99.9999%, neon Ne – 99.999%, argon Ar – 99.998%. The purity of the working gas was assessed by the ratio of the radiation intensities of the molecular bands of hydrogen, nitrogen, and oxygen and by mass-spectroscopic measurements using an SRS RGA 200 mass spectrometer. The gas pressures were measured with a Thyracont VCC200MA4 capacitive sensor with an accuracy of $\pm 0.25\%$. A direct current source with adjustable voltage $U = 0\text{--}4000\text{ V}$ and ballast resistance $R = 20\text{--}600\text{ k}\Omega$ was used.

Before each cycle of measurements of the discharge parameters, degassing of the cell was carried out. The procedure is considered in detail in [9]. Changes in the current-voltage characteristics during the degassing of the cell and conditioning of the cathode were characterized by a gradual decrease in the discharge current. The criterion for achieving the working regime was the constancy and stable reproducibility of the current-voltage characteristic parameters during all cycles and the measurement time. In the present work, after ~ 50 hours the current-voltage characteristics acquired a steady-state character.

The energy efficiency of electron beam generation, η , determined as a fraction of the discharge power transferred to the fast electron component, including the energy of electrons that reached the anode η_{col} and the energy lost by them in the gas during excitation, ionization, and elastic collisions η_{ds} . The measurement of η was carried out by the calorimetric method and was determined as the ratio of the power of the fast electron beam to the total power expended on maintaining the discharge: $\eta = \eta_{\text{ds}} + \eta_{\text{col}}$; $\eta_{\text{ds}} = P_{\text{ds}}/P$ и $\eta_{\text{col}} = P_{\text{col}}/P$, where P_{ds} is the power dissipated in the discharge gap, P_{col} is the power released at the anode, and $P = U \times I$ is the total discharge power, where U is the discharge voltage and I is the discharge current.

The methodology for determining η_{ds} and η_{col} was based on preliminary calibration [7, 10]. For this purpose, in the absence of a discharge, the dependences of the steady-state temperature increment ΔT on the outer wall of the cell and on the anode-collector on the power supplied to the built-in ohmic heaters were measured. During the experiment, the values of ΔT in the corresponding

zones were recorded, after which the values of P_{ds} and P_{col} were reconstructed from the calibration dependences. On the basis of the obtained values, the efficiencies η_{ds} , η_{col} , and the total efficiency of electron beam generation η were calculated.

Results and Their Discussion

In Fig. 1, as an example, a family of current-voltage characteristics of the AGD in the coordinates I – U in helium at pressure $p_{He} = 0.9$ and 10 Torr, neon $p_{Ne} = 0.5$ and 2 Torr, and argon $p_{Ar} = 0.5$ and 0.8 Torr, measured after performing the procedure of degassing the cell and training the cathode, is presented.

The experimental results demonstrated:

in helium, with increasing pressure, the dependences $I(U)$ change their character, and at $p_{He} \geq 4.3$ Torr they change character – they cease to be monotonically increasing. With increasing voltage, regions of decreasing current appear, and the magnitude of this drop increases with increasing gas pressure, reaching a maximum for the highest pressure. For example, for the current-voltage characteristic at $p_{He} = 10$ Torr, three regions can be traced:

- growth of I with increasing voltage up to $U \approx 400$ –500 V;
- decrease of I with increasing voltage up to $U \approx 1100$ –1400 V;
- repeated growth of I with increasing voltage.

In [11], it was experimentally shown that in a high-voltage discharge in helium, in which electron beams with $\sim$ keV energies are generated, the current-voltage characteristics have the power-law form $j \sim p^x U_c^y$ with exponents $x = 2$; $y = 3$; where j is the current density; U_c is the cathode potential drop, which under conditions of high values of U_c is practically equal to the voltage U applied to the discharge gap. The dynamics of change in the parameters x and y makes it possible to trace the transformation of the current-voltage characteristic when the discharge conditions change. In the present work, in a direct current discharge in helium, the exponent 'y' changes over the entire region of the current-voltage characteristic:

- for all pressures $y > 3$ at $U < 300$ V;
- at $U \approx 750$ –900 V it reaches its minimum with the value $y \approx 1$ at $p_{He} \approx 0.9$ Torr;
- becomes negative $y \approx -0.3$ at $p_{He} \approx 10$ Torr and increases, for example at $U \approx 2100$ V 'y' ≈ 1.8 and 1 at $p_{He} \approx 0.9$ and 10 Torr, respectively.

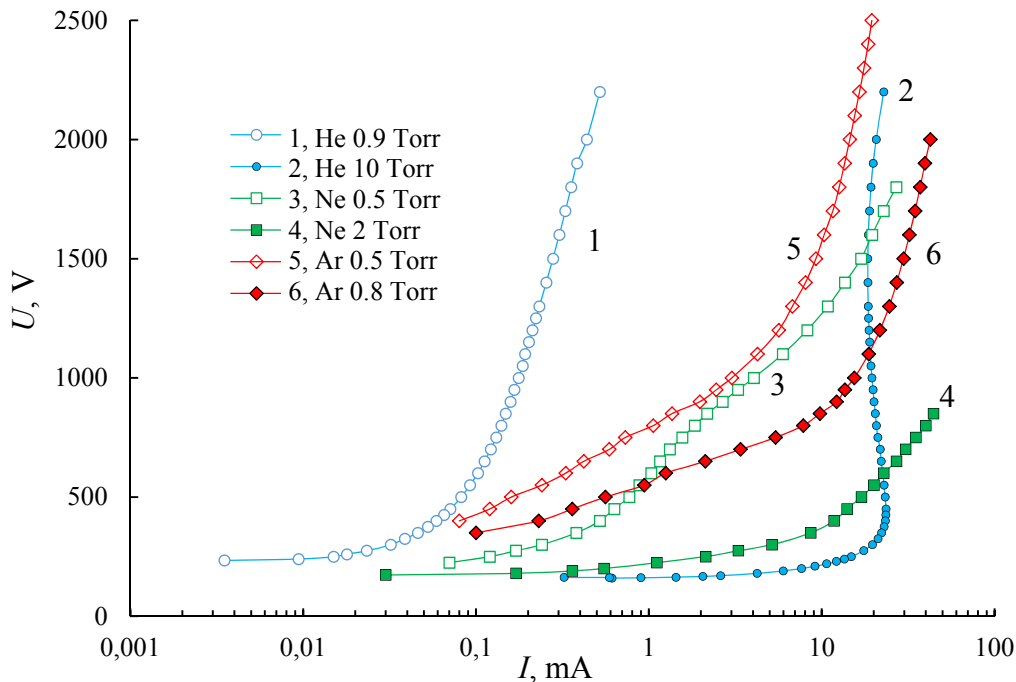


Fig. 1. Dependence of the discharge current I on voltage U at different pressures of the working gas p : He, $p_{He} = 0.9$ (1), 10 Torr (2); Ne, $p_{Ne} = 0.5$ (3), 2 Torr (4); Ar, $p_{Ar} = 0.5$ (5), 0.8 Torr (6)

In neon, the changes in the current-voltage characteristics during the operation of the discharge under clean conditions are less pronounced. With decreasing gas pressure, a transformation of the current-voltage characteristic is observed, but in the studied pressure range, regions with negative differential conductivity were not obtained. Up to voltages $U \approx 300$ V, the exponent $y > 3$; at $U \approx 500$ – 600 V the minimum value ' y ' ≈ 1.5 – 1.8 ; with a further increase in U , it increases. This, similarly to the case in helium, may indicate a change in the mechanism of electron emission [9]. At $p_{\text{Ne}} = 0.5$ Torr, the dependence $y(U)$ has a more complex form: at $U \approx 1000$ V the maximum value ' y ' ≈ 4 is reached, and with a further increase in U the parameter ' y ' decreases. Previously, such behavior of the dependence $y(U)$ was observed in an "open" discharge in helium [9].

In argon, no significant transformation of the current-voltage characteristic was observed, and the minimum value of ' y ' was not reached. At $U > 300$ V, the values of ' y ' are close for all the studied pressures and decrease from ' y ' ≈ 5 at $U \approx 300$ – 600 V to ' y ' ≈ 1.3 at $U \approx 1500$ – 2800 V.

In Fig. 2a–c, the measurement results the fractions of energy dissipated in the discharge gap η_{ds} , at the anode–collector η_{col} , and their total efficiency η in helium ($p_{\text{He}} = 4.7$ and 10.2 Torr), neon ($p_{\text{Ne}} = 0.5$ and 1.5 Torr), and argon ($p_{\text{Ar}} = 0.35$ and 1 Torr) are presented. The general tendency is an increase in the efficiency of fast electron generation with increasing U for all the studied gases and pressures. At the same time, the nature of the influence of pressure is different (Fig. 2c): in helium, with increasing p_{He} the efficiency grows as $\eta \sim p_{\text{He}}$, in neon this dependence is weakly pronounced, and in argon an inverse dependence is observed – $\eta \sim 1/p_{\text{Ar}}$. At $U \approx 3000$ V, the maximum efficiency is realized in neon $\eta \approx 90\%$, in helium it is $\eta \approx 80\%$, in argon, it does not exceed $\eta \approx 40\%$ at helium, neon, and argon pressures of 10.2 , 0.5 , and 0.35 Torr, respectively. The dependences of the fraction of beam energy released at the anode–collector $\eta_{col}(U)$ have a similar character. With increasing voltage, η_{col} increases monotonically. The maximum values are realized in the series $\text{Ar} \rightarrow \text{Ne} \rightarrow \text{He}$, while with increasing pressure for each of the gases η_{col} decreases. The

dependences of the fraction of energy released in the discharge gap $\eta_{ds}(U)$ have a more complex character. In the voltage range $U = 1000$ – 3500 V, for argon a monotonic weak growth is observed, for neon a sharp increase at $U = 1000$ – 1500 V with subsequent saturation at $U \geq 1500$ – 2000 V, and in helium this dependence has an extreme character with a maximum in the region $U \approx 1000$ – 1500 V.

It is clearly seen that the contributions of the quantities η_{ds} и η_{col} to the total efficiency of electron beam generation differ substantially depending on the kind of gas. For example, in helium at $p_{\text{He}} = 4.7$ Torr and $U = 2100$ V, the total generation efficiency $\eta \approx 66\%$ is formed as $\eta_{col} \approx 50\%$ and $\eta_{ds} \approx 16\%$. In turn, for neon at the same voltage $U = 2000$ V and $p_{\text{Ne}} = 1.5$ Torr, $\eta \approx 71\%$, $\eta_{ds} \approx 43\%$, and $\eta_{col} \approx 28\%$ are observed, and for argon at $p_{\text{Ar}} = 0.75$ Torr and the same voltage, $\eta \approx 31\%$, $\eta_{ds} \approx 23\%$, and $\eta_{col} \approx 8\%$.

The efficiency is related to the total emission coefficient $\langle \gamma \rangle$ as $\eta = \langle \gamma \rangle / (\langle \gamma \rangle + 1)$ [12]. At present, there are practically no reliable data on $\langle \gamma \rangle$ under clean conditions. For comparison, one can use the results of [13], in which the value $\langle \gamma \rangle \approx 0.98$ was obtained in helium at $U = 4000$ V, caused mainly by emission under the action of fast atoms. Under present investigation conditions, $\langle \gamma \rangle \approx 2$ at $U = 2000$ V, which indicates a significant contribution of photoemission to the electron current from the cathode. One can estimate the magnitude of the efficiency of electron beam generation proceeding from the fraction of energy losses in the discharge zone. In a high-voltage AGD, the main part of the applied voltage is concentrated in the cathode layer. In the quasi-neutral region beyond the cathode drop, the electric field is significantly smaller; therefore, the contribution of the low-energy electron component to the direct energy release is negligibly small. At a voltage at the cathode U , the maximum energy of runaway electrons can reach $E \approx eU$. The fraction of energy lost by such electrons in the region beyond the cathode sheath is $\sim (\eta_{ds} \cdot E)$. The fraction of energy η_{ex} of the electron beam expended on the excitation of resonant states of the working gas atom, without taking into account recombination population, has the values $\eta_{ex} \approx 0.3$ – 0.4 [14–16]. Given the geometrical

factor $R_s \approx 0.112$, characterizing the interception of radiation, the number of photons reaching the cathode is: $N_{ph} \approx eU \cdot \eta_{ex} \cdot \eta_{ds} \cdot R_s / h\nu$, where $h\nu = 19.8$; 16.7 and 11.6 eV is the energy of resonant photons in helium, neon, and argon, respectively.

Consequently, the number of generated photoelectrons, Ne, born thanks to radiation from the discharge gap:

$$N_e \approx eU \cdot \eta_{ex} \cdot \eta_{ds} \cdot \gamma_{ph} \cdot R_s / h\nu. \quad (1)$$

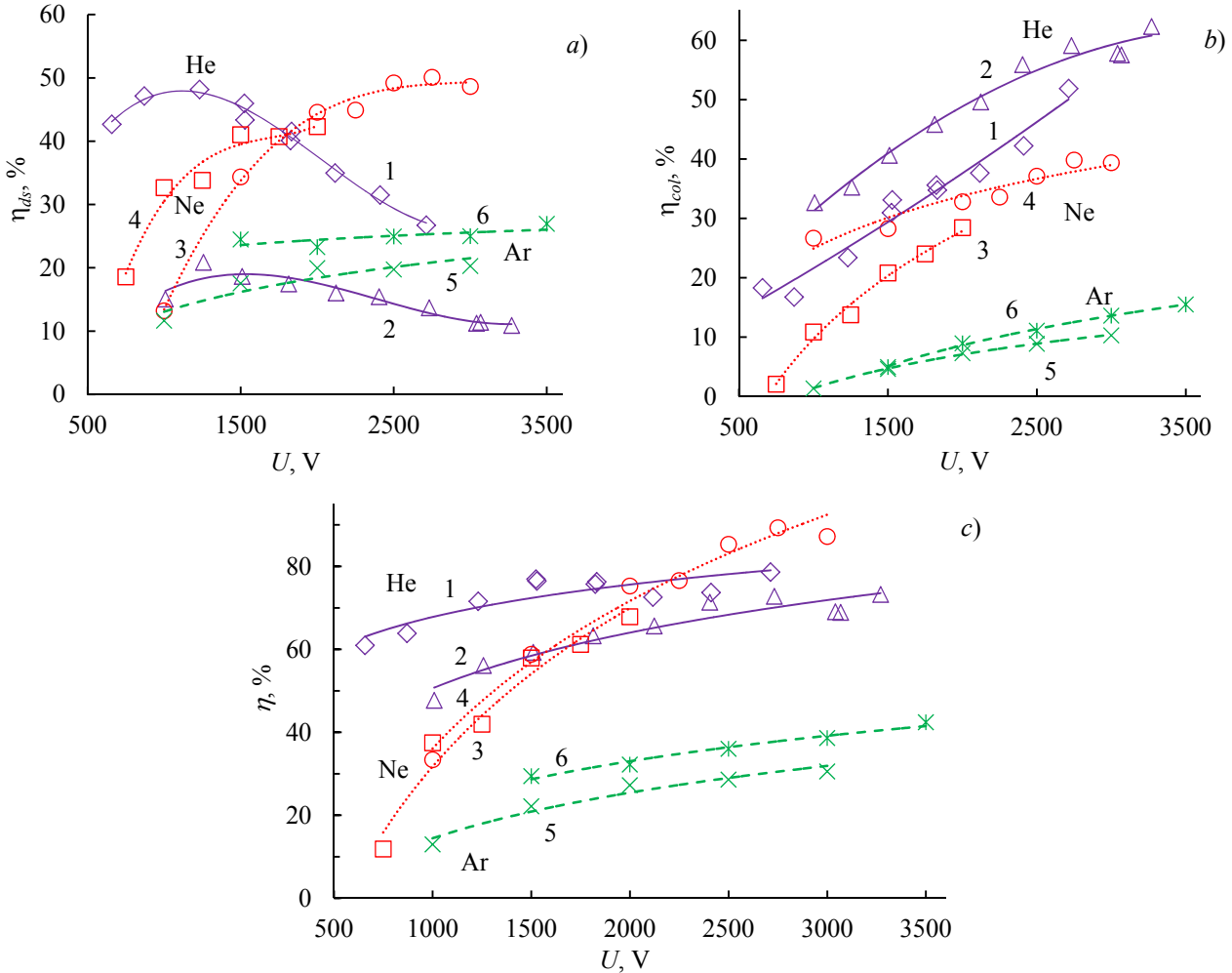


Fig. 2. Comparison of the dependences of the fractions of energy lost in the discharge gap $\eta_{ds}(U)$ – (a), at the collector $\eta_{col}(U)$ – (b), and their sum $\eta(U)$ – (c) on the voltage U at different pressures of the working gas p in He ($p_{He} = 10.2$ (1), 4.7 (2) Torr), Ne ($p_{Ne} = 1.5$ (3), 0.5 (4) Torr), and Ar ($p_{Ar} = 1$ (5), 0.35 (6) Torr)

Calculation by formula (1) shows that in He, Ne, Ar at $U = 2000$ V at $p_{He} = 10$ Torr, $p_{Ne} = 1$ Torr, and $p_{Ar} = 0.75$ Torr and values of the photoemission coefficient $\gamma_{ph} \approx 0.3$; 0.28, and 0.15 [17, 18], the number of photoelectrons born per one primary fast electron is $N_e \sim 0.505$; 0.50, and 0.196, respectively. Under the assumption that at $U \geq 2000$ V the beam is formed predominantly at the expense of photoemission, the calculated efficiency of electron beam

generation would be $\eta \approx 50.5$; 50, and 19.6%, respectively. From the obtained estimates it is evident that the calculated values are smaller than those obtained experimentally ($\eta \approx 72.6$; 75.5, and 30.6%). This indicates that under real conditions of the AGD, the contribution of photoemission is substantial, but not the only source of electron generation. An additional contribution may be made by other channels of secondary electron emission (primarily potential

emission under the action of ions and metastable atoms). Let us note that the geometrical factor R_s does not take into account the non-uniform excitation along the discharge gap, and η_{ex} in the proposed estimate does not depend on the electron energy and is taken as constant.

Conclusion

Thus, this work carried out a comparative study of the anomalous glow discharge in inert gases (He, Ne, Ar) at voltages above 1000 V under conditions of high purity of the gas medium and the cathode material. It is shown that the maximum efficiency of EB generation under the studied conditions is achieved in helium $\eta \approx 80\%$, neon $\eta \approx 90\%$, and argon $\eta \approx 40\%$ with a nonmonotonic form of the current-voltage

characteristic. Estimates of η under the assumption of operation under conditions of modification of the cathode by the working gas demonstrate that the main mechanism for sustaining the discharge is photoemission, and the discharge under such conditions is characterized by a high energy efficiency of the electron beam. Further development of this research direction presupposes the study of the AGD in the voltage range > 4 kV for obtaining beams with energies on the order of $\sim$ keV and a theoretical study of the mechanisms of electron emission.

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Gas bubbles chain in water and development of pulse electric discharge

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The development of an electric discharge in a liquid in the presence of a distributed gas phase can proceed differently than in a single-phase medium due to faster ionization processes in the gas. It was studied broadly in the numerical experiments by N. Yu. Babaeva and G. V. Naidis. They studied ionization within periodically distributed bubbles – two-dimensional arrays and one-dimensional chains. It was found the increase of electric field at the poles leads to increasing number of bubbles involved in the discharge development and it leads to expansion of the ionization region. However, numerical observation of a single discharge channel emerged from such ionized bubbles is currently impossible due to the multi-scale nature of the processes involved in the calculations. This work proposes a new method for generating ordered chains of gas bubbles in water. It is based on pulsed water electrolysis. Finally, effect of bubbles chain on the development of an electric discharge is examined.

Keywords: electrolysis, bubbles, chains, water, electric discharge.

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Introduction

The presence of a gas (vapor) phase in a liquid plays a major role in the initiation and development of an electric discharge in a liquid. Although direct ionization in the liquid state is theoretically possible at high field strengths and short rise times [1], the experimental realization of such conditions and the registration of ionization in the liquid state still remain a very difficult task [2]. A model concept of streamer propagation in liquid hydrocarbons was proposed in the works of G. V. Naidis [3, 4]. Much more often, the initiation of a discharge is preceded by the stage of formation in the liquid of a region of reduced density, in which ionization processes develop. Depending on the conditions, the formation of such a region is mainly caused by electrohydrodynamic flow [5–8], boiling [9], decompression (a sharp decrease in pressure) [10], or ultrasonic action [11].

The electrohydrodynamic flow arising in a liquid under the action of an electric field can lead to a disruption of continuity and the formation of a vapor-gas bubble in the region of strong field inhomogeneity [5, 6], which is realized, for example, near the tip of a high-voltage electrode [7]. Such a process occurs on nanosecond time scales.

The formation of a vapor-gas region through boiling is characteristic of lower voltages and long fronts of its rise [9]. The region of reduced density is formed where the greatest current flows, after which ionization occurs and a plasma channel appears, which grows into the liquid through evaporation and ionization.

Quite often, small and large bubbles may already be present in the liquid before the voltage is applied. This class of problems – with pre-existing bubbles – has received separate development. The most significant contribution to computational and theoretical studies was made

in the works of N. Yu. Babaeva and G. V. Naidis et al. [12–15], in which cases of both single bubbles and cases of their ordered one-dimensional chains and two-dimensional grids were studied. The development of a streamer inside a bubble is shown, as well as its influence on the initiation of discharges in neighboring bubbles (a kind of "chain reaction" effect). Experimental verification of the results obtained for ordered arrays of bubbles is currently complicated by the absence of a method for producing them. Breakdown of single or paired bubbles is being actively studied [16–19], however, the formulation differs somewhat from the computational one – as a rule, the bubbles are in contact with the electrode and with each other in order to fix them in space. Separately, it is worth mentioning an experiment in which a single bubble was held motionless in the bulk of a liquid using focused ultrasonic radiation [20].

In the present work, a method for producing chains of bubbles using pulsed electrolysis is proposed and studied. By varying the parameters of the electrolysis process, a high stability of the bubble diameter and the spatial repetition period can be achieved. For the most successful regime, shadow kinograms of the development of the electric breakdown of the gap in water with distributed vapor-gas bubbles were recorded.

Experimental Setup

To study the electric breakdown in water in the presence of gas bubbles, an experimental setup was used, schematically shown in Figure 1. The setup consists of a system for creating bubbles in the region between the electrodes, a system for supplying high voltage between the electrodes, a system for optical diagnostics, and measurement of the electrical parameters of the process.

The experiments were carried out in a dielectric container with a volume of 0.2 l, filled with water, with transparent walls (material – plexiglass). To ensure the electric breakdown of water in the region between the electrodes, a high-voltage electrode *E1* of spherical shape (diameter 3 mm) and a grounded electrode *E2* in the form of a disk with a diameter of 20 mm and a height of 5 mm are used. The grounded

electrode has a central through hole with a diameter of 2 mm, intended for the passage of bubbles into the region of electric breakdown. High positive voltage (+*HV*) from a capacitor bank *C* with a total capacitance of 1 μF, charged through a ballast resistor *R_b*, was supplied to electrode *E1* using a semiconductor switch *S* of the "half-bridge" type.

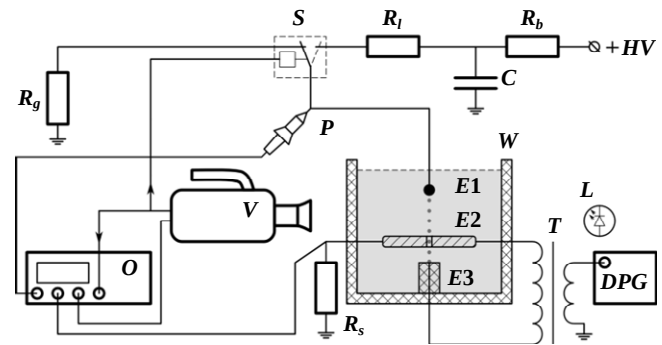


Fig. 1. Experimental setup diagram

To create bubbles in the region between electrodes *E1* and *E2*, an additional (low-voltage) electrode *E3* is used, located under the grounded electrode *E2*. When voltage pulses were applied between the low-voltage electrode *E2* and the grounded electrode *E2*, electrolysis of water occurred with the formation of gas bubbles. The diameter of electrode *E3* is 0.15 mm, the electrode itself is sealed in a dielectric sheath, which ensures an extremely low value of the contact area of the electrode with the liquid and a high local current density at this location. Ultimately, this leads to the localized appearance of gas bubbles near the cut of electrode *E3*. After the bubble detaches from the surface due to the action of buoyancy forces, it moves vertically upward through the hole in the grounded electrode. The opening of switch *S* was carried out by a signal from video camera *V*, used for visualizing the breakdown process. The discharge current was limited using load resistor *R_l*. Resistor *R_g* was used to limit the current through the switch in the "to ground" switching mode.

The mutual arrangement of the electrodes ensures the absence of electric breakdown between electrodes *E1* and *E3*; however, when a high potential is applied to electrode *E1*, the voltage at *E3* can still increase to a value of several hundred volts, which can damage the low-voltage part of the setup intended for electrolysis.

In order to avoid this, this part of the circuit is galvanically isolated from the volume of liquid using a pulse transformer T . The primary winding of transformer T is excited using a programmable digital generator (DPG), which has the capability of regulating the amplitude of the generated pulses. The generator operated in the mode of generating high-frequency pulses with a duty cycle close to 0.5, with a number of n pulses and with gaps of m pulses. Before applying high voltage, the numbers n , m , and the pulse amplitude were experimentally selected in the working container in order to achieve the required frequency of bubble creation and their size. The polarity of electrode $E3$ was positive.

To visualize the process and to determine the path of the electric arc, the shadow method was used, implemented using LED L , high-speed video camera V with a microscope as the objective, providing a spatial resolution of $10\text{ }\mu\text{m}/\text{pixel}$. The light emitted by the LED was collimated by a converging lens into the region between electrodes $E1$ and $E2$, then entered the microscope of the video camera. Registration of the shadow image of the process began after pressing the button of the video camera, and its recording rate was 140.000 fps, with an exposure of $0.3\text{ }\mu\text{s}$. The moment of pressing the button was determined by the operator when in the region between electrodes $E1$ and $E2$ there was a sufficient number of bubbles of suitable size.

To register the electrical parameters of the process, a four-channel digital storage oscilloscope O (2 GHz, 10 GSamples/s), a high-voltage probe P connected to the high-voltage electrode P , and a resistive shunt R_s with a resistance of $25\text{ }\Omega$ were used. The trigger signal for video recording of the video camera was simultaneously fed to the oscilloscope and to switch S , which ensured the start of their operation. To another channel of the oscilloscope, signals of the beginning of the video camera exposure were additionally fed in order to correlate the visible picture of the discharge with the electrical parameters of the circuit at that time. To the remaining two channels of the oscilloscope, signals from probe P and the voltage across shunt R_s were fed to measure the voltage at the high-voltage electrode $E1$ and the current in the circuit, respectively.

Findings and Discussion

At the first stage of the study, a search for an electrolysis regime that would ensure stable formation and detachment of bubbles was made. Figure 2 presents a photograph of a chain of bubbles with a diameter of about $50\text{ }\mu\text{m}$; the number of bubbles in the visible region is 11. The spatial period of succession is about $300\text{ }\mu\text{m}$.

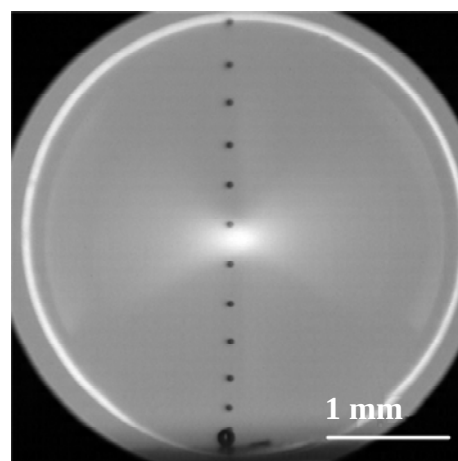


Fig. 2. An example of a chain of bubbles generated by means of electrolysis in a pulse-periodic regime

This regime can be considered the best since it combines a relatively large diameter and the absence of additional parasitic centers of bubble formation. The main difficulty in achieving the working regime consists in satisfying two oppositely directed requirements: on the one hand, to generate bubbles with a size of fractions of a millimeter and their chains, an increase in current is necessary; on the other hand, an increase in current leads to the spontaneous appearance of additional centers of bubble generation on the surface of electrode $E3$, which give rise to additional chains of bubbles of smaller diameter (about $10\text{ }\mu\text{m}$). At the same time, the continuous flow of current, even of a small amplitude, leads to chaotic generation and detachment of bubbles. In order to overcome the described difficulties, a pulse-periodic regime of voltage at electrode $E3$ is introduced in the work. The proposed voltage waveform – n pulses with a duration of about 500 ns and a period of about $1\text{ }\mu\text{s}$, followed by m skip pulses (voltage equal to zero) – solves the posed problem. The high frequency (about 1 MHz) together with a duty cycle of about 50% apparently ensure a pulsed

regime of gas formation in which each new portion of it manages to merge with the growing bubble. An increase in the duty cycle reduces the intensity of gas formation, which leads to longer periods of bubble detachment and larger distances between neighboring bubbles in the forming chain. A decrease in the duty cycle, on the contrary, leads to the appearance of additional centers of gas formation and the formation of undesirable side chains. In the proposed method, the detachment of bubbles after their growth is determined by the presence of m skip pulses in the voltage signal. The key here, apparently, is the influence of flows arising under the action of the electric field near the surface of electrode E_3 . This flow is clearly visible to the eye when observing small bubbles remaining near the electrode after a completed preceding experiment. The application of a new series of pulses leads to the formation of a bubble on the surface of the electrode, while the liquid around it rushes toward the electrode, thereby washing the bubble off the surface and ensuring its detachment. This circumstance is positive and important, since in the absence of a driving force, the detachment of a bubble from the surface and overcoming the forces of surface tension at the expense of buoyancy forces occurs at significantly larger diameters (on the order of 1 mm). The total time of the pulses ($n + m$) coincides in order of magnitude with the period of bubble detachment from the surface, which allows us to assume the main role of the mechanism described above in the detachment of bubbles from the electrode.

It should be noted that, despite the discovered parameters for controlling the process of bubble formation, it has so far not been possible to achieve chains of bubbles with a diameter on the order of 1 mm. The rapid growth of large bubbles with increasing voltage is accompanied by the appearance of secondary centers of gas formation and side chains of small bubbles. Slow growth of large bubbles is possible, however, the long growth time leads to very large distances between bubbles, at which they no longer constitute a chain and cannot serve the purposes of studying their mutual influence in the development of electric breakdown.

Despite the fairly high stability of the generation of the chain of bubbles, the parallel growth of one larger bubble is often observed (see Fig. 2, on the surface of the electrode), which grows over the course of 5–10 seconds and then detaches. No methods of combating this effect have yet been found, and this limits from above the distance at which the high-voltage electrode can be placed, since after detachment the large bubble floats up at a higher velocity and disrupts the structure of the chain obtained before its detachment. In the current implementation, this distance turned out to be less than the height of placement of the high-voltage electrode, chosen from considerations of the safety of the low-voltage (electrolysis) part of the electrical circuit. For this reason, the chain of bubbles along which the electric discharge develops (Fig. 3) turns out to be deformed.

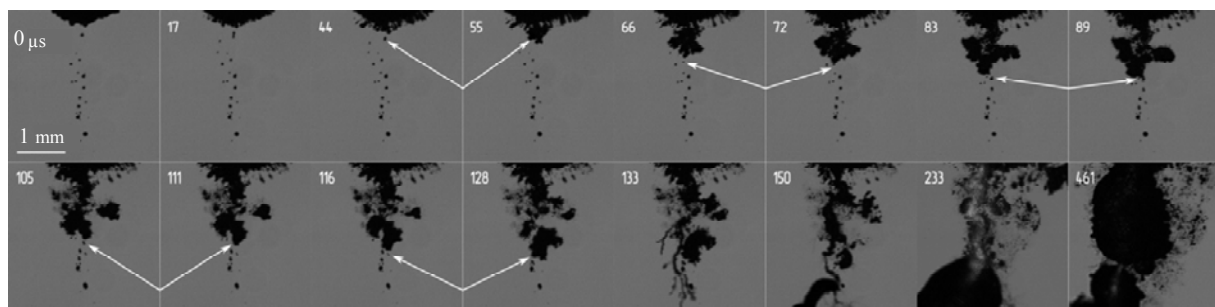


Fig. 3. Kinograms of the development of an electric discharge in water guided by a chain of bubbles

Figure 3 presents a kinogram of the development of a pulsed electric discharge in water with a conductivity of $45 \mu\text{S}/\text{cm}$ in the presence of a fragment of a chain of bubbles. The

magnitude of the applied voltage is 10 kV. At the upper boundary of the figure, a small part of the rounded anode is visible; the main part of the visible region is occupied by the chain of

bubbles; the cathode is outside the image, below the lower boundary. The corresponding oscillograms of voltage and current across the discharge gap are presented in Figure 4. Immediately after the application of voltage, the deformation of the electrolysis bubbles adhering to the surface of the high-voltage electrode *E1* during flotation begins. Their size along the field increases, while across the field it decreases. Then, inside one of them, the initiation of a discharge channel occurs ($44\text{ }\mu\text{s}$), which begins to grow downward in the direction of the grounded electrode *E2*. Comparing successive frames of the kinogram (44 and $55\text{ }\mu\text{s}$, 66 and $72\text{ }\mu\text{s}$, etc.), it is possible with a high degree of confidence to speak of the involvement of the bubbles in the chain (marked by arrows) in the process of advancement of the discharge channel. By the time $133\text{ }\mu\text{s}$, the channel turns out to be sufficiently close to the region of the inhomogeneous field associated with the presence of the edge of the central hole in electrode *E2*, and the relatively slow growth is replaced by a short-term and rapid elongation of the channel. On the oscillograms, this is accompanied by a short-term surge of current up to 0.5 A and a drop in voltage to 4 kV . The final closure of the gap occurs by $233\text{ }\mu\text{s}$, and the discharge transitions to the arc stage with a current of about 1.5 A , after which the high-voltage switch opens upon expiration of the pulse duration time of $500\text{ }\mu\text{s}$.

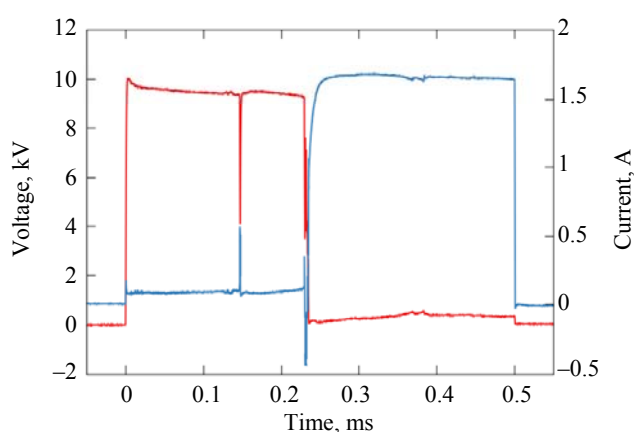


Fig. 4. Oscillograms of voltage and current across the discharge gap corresponding to the breakdown in Fig. 3

Conclusions

The proposed method for producing chains of single bubbles of submillimeter diameter by means of pulse-periodic electrolysis in water has shown its suitability and promise in studying the development of electric discharges in two-phase media. The advantages include the good reproducibility of the bubble diameter in the chain and the distance between neighboring bubbles, the possibility of influencing these quantities through the electrical parameters of the electrolysis process and establishing their required ratio.

The disadvantages include the complexity of tuning and the strong influence of the discharge on the electrolysis process at low water conductivity (about $5\text{ }\mu\text{S/cm}$) – after each application of high voltage, the electrolysis process stops for some time (minutes), which is explained by a deficit of ions after the flow of the discharge current. Increasing the diameter of the bubbles is difficult while preserving their repetition period in the chain, since the intensification of the gas formation process leads to the appearance of several growth centers. Each of them may begin to generate its own chain of small bubbles instead of generating one large bubble. A consequence of the instability of the process at a higher productivity is the periodic appearance and detachment of larger bubbles, whose motion disrupts the structure of the chain. For this reason, the initiation of the discharge process at large distances from the point of generation in a well-structured chain remains difficult.

In the most successful current formulation, the discharge was initiated along a chain of bubbles with a diameter of $50\text{--}100\text{ }\mu\text{m}$ and a repetition period of about $200\text{ }\mu\text{s}$. The propagation of the head part of the discharge channel along the distribution of bubbles in the interelectrode gap was recorded.

Further studies will be directed toward eliminating the above-listed disadvantages and visualizing the ionization processes inside the bubbles.

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Coating deposition processes in the gas and vacuum modes of a planar magnetron sputtering system

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The results of comparative studies of the deposition of coatings in the gas and vacuum modes of operation of a planar magnetron sputtering system are presented. Copper was used as the model material of the sputtered target cathode. It is shown that higher deposition rates of coatings are achieved in the vacuum mode, their adhesive properties are improved and their roughness is reduced.

Keywords: magnetron sputtering system, self-sputtering, plasma, vacuum (gassless) mode, magnetron discharge.

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Introduction

Since their appearance in the mid-1970s [1] planar magnetron sputtering systems (MSS) have been actively developing and improving. This development is driven by constantly growing requirements for coating quality and equipment productivity. Reducing the operating pressure of magnetron sputtering systems is one of the main directions of research, since it makes it possible to reduce the number of transport losses of sputtered atoms and thereby increase the energy efficiency of the deposition process, as well as to enhance the energy impact on the substrate during coating deposition. The limiting case of reducing the operating pressure is the vacuum or gasless mode of MSS, in which the magnetron discharge is maintained in the so-called self-sputtering mode [2]. In contrast to the known gasless MSS with a liquid-phase cathode [3, 4], in a vacuum magnetron the temperature of the sputtered target cathode is much lower than the threshold values of the evaporation or sublimation temperature of the cathode material [5]. From magnetrons with

thermal evaporation, the vacuum mode of MSS with a "cold" target features favorably by higher values of the directed energy of sputtered atoms (more than 10 eV versus 0.1 eV) [6], as well as a higher degree of ionization of the atoms of the sputtered target. At the same time, the coating deposition rate in the vacuum mode of MSS is comparable to the vacuum-arc method, but at the same time does not require filtration of the generated flux from the microdroplet fraction.

Our earlier studies [6] were focused predominantly on comparing the parameters of the magnetron discharge and discharge plasma in the gas and vacuum modes of operation. The aim of this work was to compare the deposition processes and a number of coating parameters in these modes at a fixed value of the discharge current.

Methodology and technique of the experiment

A magnetron with a direct water-cooled cathode-target [6] was mounted on a vacuum chamber 60 cm long and 40 cm in diameter

(Fig. 1). Copper was used as the model material of the magnetron cathode-target. The diameter of target 2 was 100 mm. Anode 3 of the magnetron was connected to the grounded walls of the vacuum chamber. The arched magnetic field of the magnetron was provided by a set of NdFeB magnets 1. The body of the magnetron and the vacuum chamber were made of nonmagnetic stainless steel. The vacuum chamber was pumped down to a residual pressure level of 2×10^{-5} Torr by a turbomolecular pump with a capacity of 500 l/s. A specialized power supply APEL-M-20DC-1000SS (Applied Electronics LLC) was used for the electrical supply of the magnetron discharge.

In the gas mode of MSS, argon was used as the working gas. The operating pressure was 3×10^{-3} Torr. To implement the vacuum mode of the magnetron, the initial initiation was carried out in the presence of the working gas at a pressure of 3×10^{-3} Torr, after which the magnetron discharge current was set at a level of 10 A and the admission of the working gas into the chamber was shut off by a mechanical valve installed directly on the vacuum chamber. In this case, the pressure in the vacuum chamber decreased to values close to the residual pressure (2×10^{-5} Torr).

In deposition mode No. 1, a movable holder with a ruler of samples 4 was used to measure the axial and radial distribution of the coating deposition rate, making it possible to change the distance to the target from 20 to 50 cm. Samples with a size of 3×3 cm² were placed at a radial distance R from the axis of the discharge system equal to from 0 to 20 cm with a step of 5 cm. Silicon and glass substrates were used as samples. The thickness of the glass samples was chosen to be at least 4 mm, which ensured sufficient strength of the substrate for measuring the adhesion of coatings by the method of pulling off a glued contact pad.

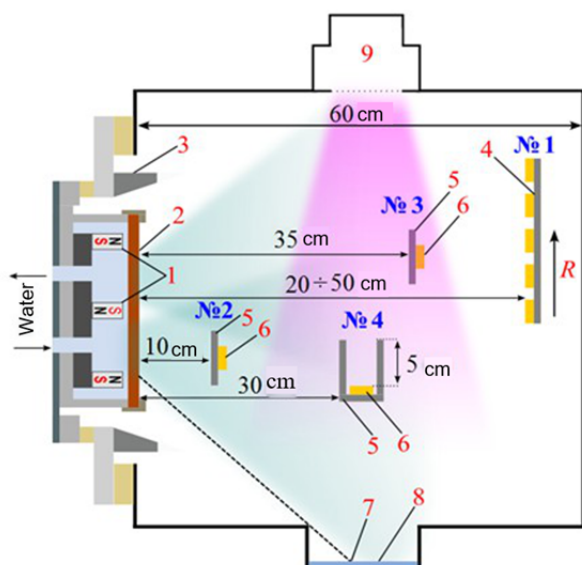
Before being placed in vacuum, the samples were cleaned with the use of an ultrasonic bath in alcohol and distilled water at a temperature of 60 °C for 60 min. Directly in the vacuum chamber, before the deposition process, the samples were subjected to additional ion cleaning

and surface activation using a plasma generator with electron injection [8]. The ion current density in the substrate region was $0.5 \div 1$ mA/cm² at a negative self-bias potential of the substrate of the order of 20–30 V. The duration of the ion treatment stage was 15 min. After that, the samples were covered with a shutter, the magnetron discharge was ignited, the deposition parameters were set, the shutter was opened, and the magnetron deposition process began. After the deposition process, the samples were held in vacuum for at least 60 min.

Experiments on deposition due to backscattering of sputtered atoms [9] were carried out in three modes. For deposition mode No. 2, a glass sample 6 with a size of 3×3 cm² was placed behind a screen 5 with a size of 5×5 cm² at a distance of 10 cm from the magnetron target. An similar experiment for mode No. 3 was carried out at a distance of 35 cm. In deposition mode No. 4, the sample was placed in a tube with an inner diameter of 7 cm at a depth of 5 cm from the end face, while the tube was rotated by 90 degrees relative to the magnetron axis.

The adhesion of coatings was measured by the method of pulling off along the normal to the sample surface on the so-called Sebastian machine, which is a second-class lever. The area of the contact pad was 0.2 cm². If the coating detachment did not occur over the entire surface of the contact pad, the surface area was recalculated to the actual one. The area of the glass samples made it possible to carry out multiple adhesion measurements on a single sample to collect statistics. The ultimate strength of the epoxy adhesive was 150 kg/cm².

An optical profilometer based on the Linnik interferometer (LOMO MII-4) was used to measure the coating deposition rate. A Solver P47 atomic force microscope (NT-MDT, Russia) using a CSG01 cantilever in contact scanning mode was used to study the surface morphology of the deposited coatings. Roughness was measured for three platform sizes: 1×1 μm², 5×5 μm², 15×15 μm². Subsequent image processing was carried out using the software packages Image Analysis 2.0 (NT-MDT) and Gwyddion.

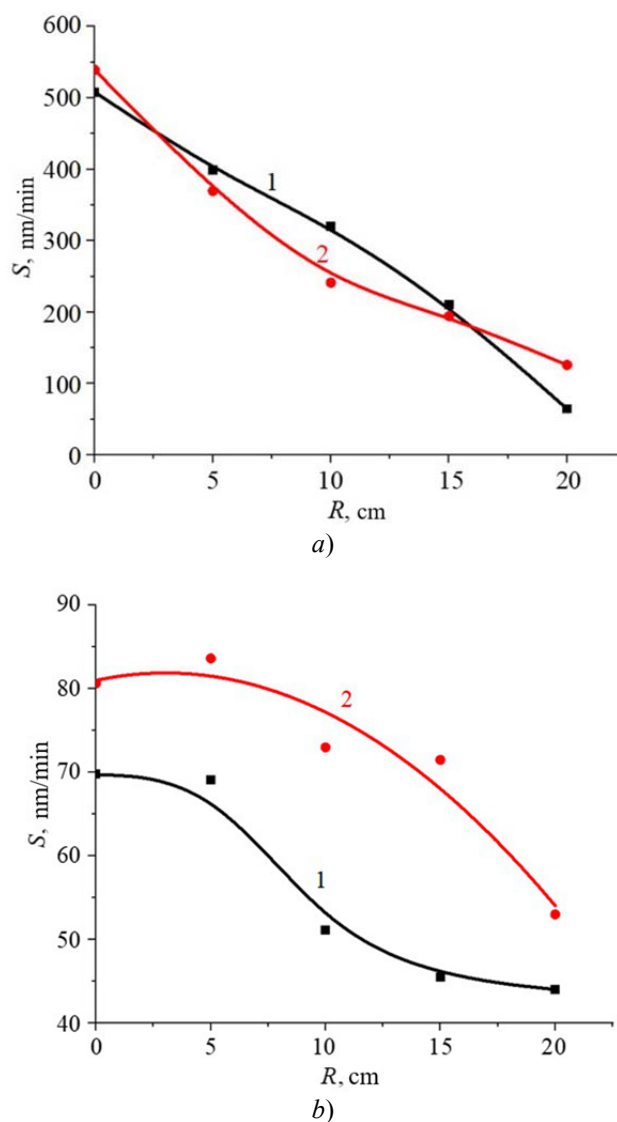


Results of experiments and discussion

Fig. 2 presents the radial distributions of the deposition rate for two values of the distance to the substrate. At distances of the order of two target diameters (20 cm), the deposition rate in the gas and vacuum deposition modes is comparable (Fig. 2a). The difference in the deposition rate in the vacuum and gas modes begins to manifest itself when distance increases. At a distance of 50 cm, the deposition rate in the vacuum mode exceeds the equal values in the gas deposition mode by a factor of 1.2–1.6 (Fig. 2b).

Simple evaluations show that the mean free path of a sputtered atom in the gas mode does not exceed 1 cm. Thus, even for deposition mode No. 2 (10 cm from the target), a sputtered metal atom can experience several collisions with working gas atoms with a change in the initial velocity vector. This conclusion is confirmed by the results of experiments on coating deposition on the back side of the sample. The small (< 100 nm) thickness of the deposited layer did not allow correct measurements using the MII-4 microinterferometer, but visually the dusting of the samples in the gas mode was many times

higher (Fig. 3) both in mode No. 2 and in mode No. 3. An equal effect was obtained for deposition mode No. 4 in the lateral direction at an angle of 90 degrees relative to the axis of the discharge system.



The ballistic deposition mode manifested itself in the vacuum mode of the magnetron as partial dusting of the viewing window with a characteristic sharp boundary 7 (see Fig. 1), determined by the protruding angle of the flange of the vacuum chamber in the path of propagation of the sputtered copper atoms. The part of the viewing window 8 located in the shadow region of the protruding angle of the vacuum chamber remained practically free from dusted even during

continuous operation of the magnetron for several hours. At the same time, in the first few minutes of magnetron operation, two characteristic boundaries of the deposition region were observed on the viewing window. Straight lines drawn from these coating boundaries through the protruding angle of the vacuum chamber pass through the far and near sections of the magnetron racetrack. The distance from the indicated sections of the racetrack to the boundary of the film on the surface of the viewing window was 50 cm. The formation of a sharp boundary 7 (Fig. 4b) of the deposited coating in the vacuum mode indicates the practically collisionless nature of the motion of sputtered atoms at the indicated distances. In the gas mode of the magnetron, the dusting of the

viewing window occurred uniformly over the entire surface, which can be explained by the scattering of the flux of sputtered atoms in collisions with working gas atoms (Fig. 4a).

The results of measuring the adhesion of coatings show that for all the studied values of the distance from the magnetron, the adhesion of coatings obtained in the vacuum mode is higher than for coatings obtained in the gas deposition mode (Table 1). With increasing distance from the magnetron target, the ratio of adhesion values in the vacuum and gas modes increases from 1.2 (at a distance of 20 cm) to 1.9 (at a distance of 50 cm). The decrease in the adhesion of coatings may be due to the loss of directed energy of sputtered atoms in the drift space in collisions with working gas atoms in the gas mode.

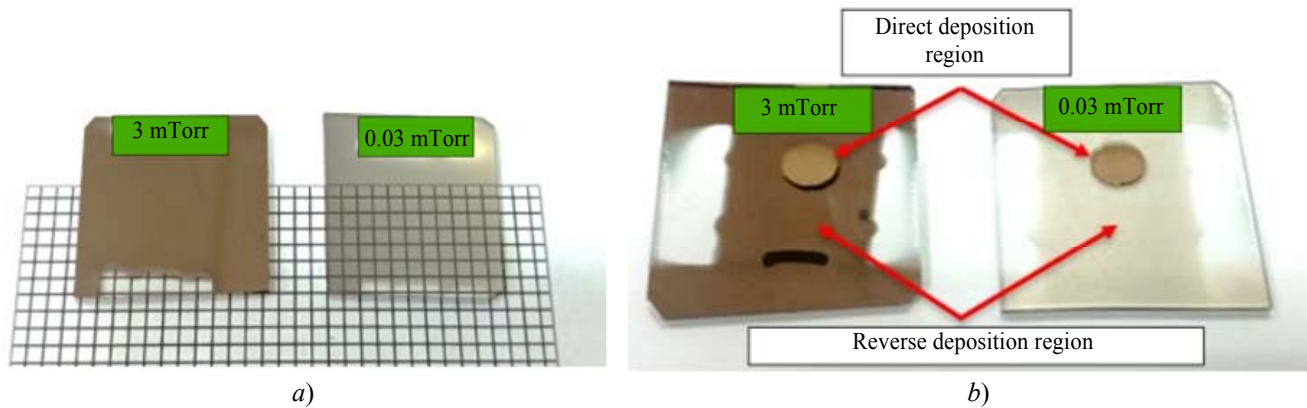


Fig. 3. Photographs of glass samples after deposition in the gas and vacuum modes of the magnetron:
a) $L = 10$ cm; b) $L = 35$ cm

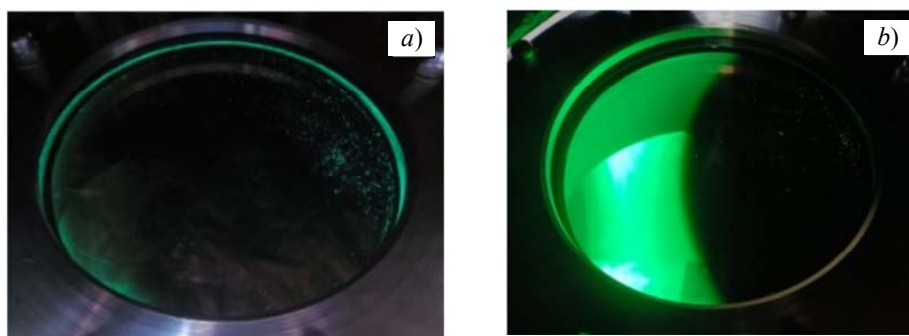


Fig. 4. Photographs of the deposited coatings on the viewing window after 30 min of magnetron discharge operation in the gas (a) and vacuum (b) modes. Discharge current 10 A

Table

Results of measurements of the adhesion of copper coatings for the gas and vacuum deposition modes depending on the distance to the magnetron

Distance from target to substrate	20 cm		30 cm		40 cm		50 cm	
	Gas mode	Vacuum mode	Gas mode	Vacuum mode	Gas mode	Vacuum mode	Gas mode	Vacuum mode
Coating adhesion, kg·f/cm ²	37	46	39	47	45	61	21	40

Coatings obtained in the vacuum mode are characterized by lower (on average by a factor of 1.5–2) values of the mean and root-mean-square coating roughness R_{mean} and R_{rms} , which amounted to 9 and 2.8 nm, respectively. This correlates with the results obtained earlier when reducing the operating pressure in the magnetron deposition process and may be due to the higher energy of the atoms arriving at the substrate and, as a consequence, greater mobility and denser packing of atoms [10].

Conclusion

The results of comparative studies of the coating deposition process in the gas and vacuum modes of operation of a planar magnetron sputtering system with copper as the model cathode material showed that the vacuum mode provides higher coating deposition rates, improves their adhesive properties, and reduces

surface roughness. The achieved parameters are due to the ballistic (collisionless) nature of the motion of the sputtered target atoms in the region from the magnetron cathode to the substrate being deposited. A consequence of this is weak effects of lateral and back scattering of sputtered atoms, which increases the efficiency of the coating deposition process in the vacuum mode of the magnetron using collimators. The obtained results unambiguously testify to the attractiveness of using the vacuum mode of MSS in the technology of creating metal coatings.

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Conflict of interests

The authors declare that they have no conflict of interests.

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The influence of conditions of formation of films of the W-C system during magnetron sputtering of tungsten with the introduction of volatile hydrocarbons into the plasma discharge region

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This paper presents experimental studies on the formation of a WC compound during magnetron sputtering of tungsten in a hydrocarbon environment on the surface of a glass substrate. It has been established that different magnetron operating modes affect the formation and surface structure of the growing film. In the range of voltages applied to the tungsten target, all hydrocarbons used in the experiment completely dissociate in the discharge plasma. Free carbon dissolves in the growing tungsten film, and at relatively low magnetron voltages, two chemical compounds are mainly formed in the W-C system: α -WC and W_2C , with WC predominating. As the magnetron voltage increases, the sputtering rate increases, and the proportion of W_2C increases. In this paper, changes in the surface microstructure of WC and W_2C films are assessed using atomic force microscopy (AFM). The potential of this method for producing compounds of refractory metals with carbon is demonstrated.

Keywords: magnetron sputtering, tungsten, graphite, surface modification, plasma-chemical synthesis.

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Introduction

Carbides of refractory metals have long found wide application in modern technology. One of the most important materials of this series is tungsten carbide, WC. At present, with the development of technologies, the field of application of tungsten carbide is constantly expanding. Modern achievements in materials science make it possible to modify the properties of WC and W_2C by adding, when necessary, various alloying additives to their composition. Such additives make it possible to obtain new properties of well-studied tungsten compounds – such as increased heat resistance or corrosion resistance. Such modifications find application in a wide variety of industries, including medical instruments, aviation and space technology.

Tungsten carbide in its pure form or in a mixture with W_2C and with additions of nickel or cobalt makes it possible to ensure maximum durability of surfaces coated with such materials. Along with this, tungsten carbide is actively used in electronics, where its conductive properties and heat resistance are employed. Such compounds are used in various electronic components as substrates for creating semiconductor devices. In cryogenic technology, tungsten and tungsten-carbon thermoelectric elements are used as sensitive elements for ultrasensitive thermometers.

Every year, research and development in this field become increasingly relevant. Various synthesis methods are used for depositing the above-listed coatings. Vacuum processes – such as a combination of pulsed laser ablation and

magnetron sputtering [1], a combination of reactive RF magnetron sputtering with ion implantation from a plasma source [2], sputtering using a magnetron arc source [3], or co-sputtering of WC and C targets in an argon (Ar) atmosphere [4]. Among these methods, of particular interest is the hybrid technology PECVD (plasma-enhanced chemical vapor deposition), PVD (physical vapor deposition), including simultaneous sputtering of a tungsten target in a reactive hydrocarbon atmosphere – such as C_2H_2 [3, 5], CH_4 [6], or C_6H_6 [7, 8]. Undoubtedly, of particular interest is the dissociation of organic molecules in plasma, leading to the incorporation of WC nanoparticles into a hydrogenated carbon matrix WC/C:H. These results, in combination with atomic force microscopy, make it possible to describe the surface of the deposited film as an aggregate of W_xC_{1-y} crystallites corresponding to an aggregate of small nanograins of W_2C and WC_{1-x} diluted in the hydrogenated carbon matrix. At the same time, the ratio between the hard crystallized WC phase and the softer hydrogenated carbon matrix decreases with increasing hydrocarbon flow. In the present work, the authors obtained tungsten carbide films by magnetron sputtering of a pure tungsten target in a medium of hydrocarbons and the working gas argon Ar at room temperature under various magnetron operating regimes. The authors applied similar methods for obtaining various compounds of the metal with carbon. In previous works [9–13], for example, composite targets of copper and solid graphite were used. However, the sputtering coefficients of copper (2.4 in argon) and graphite (0.12 in argon) at Ar^+ ion energies of 500 eV differ greatly, so it is very difficult to select a suitable regime for the magnetron operation. When sputtering a tungsten target, this difference is noticeably smaller (0.6 in argon), but the process of WC film growth is still poorly controllable. For this reason, a method was chosen of introducing a mixture of readily evaporating hydrocarbons into the region of the plasma discharge. In this region, as is known from the literature, plasma-chemical pyrolysis should occur, that is, the process of decomposition of organic compounds carried out in a low-temperature plasma with average molecular energies on the order of 0.1–50 eV [14–18]. The growth of WC films in our case proceeded at low substrate temperatures (~ 100 °C).

The mobility of the atoms of the deposited substance at such temperatures is low, and crystal growth is inhibited. The resulting fine-crystalline structure was observed by us using an atomic force microscope (AFM).

Experiment procedure

WC coatings were formed on glass substrates by means of reactive magnetron sputtering of a tungsten target in an Ar + C + H plasma. Nefras of grade BR-1 was used as the carbon source. Nefras is a mixture of paraffinic and naphthenic hydrocarbons.

In the experiments, a tungsten plate with a content of 99.93 wt. % of the main element, with a diameter of 70 mm and a thickness of 2 mm, was used as the target. A constant voltage from –600 V to –1400 V, depending on the experiment, was applied to the magnetron. In magnetron sputtering, argon was used as the plasma-forming gas. The experiments were carried out at a constant pressure of argon and the carbon-containing component in a ratio of 1:1. Each gas was supplied through a separate leak valve, after which the gases were mixed and entered the plasma zone in the form of a mixture. The pressure of the gas mixture in the vacuum chamber was maintained at a constant level and amounted to $\sim 10^{-3}$ Torr. Thus, the process of reactive deposition of atoms on the substrate surface was realized. As a result, in the medium of the reactive gas in the plasma, the required compound is formed on the substrate surface. The sputtering rate of the target depended on the voltage applied to it. In the experiment, at the first stage, at three different voltages (560 V, 900 V, 1400 V), a pure tungsten target was sputtered. Thus, at a voltage of 560–600 V, the deposition rate was ~ 1 nm/s, and at a voltage of 880–900 V – ~ 2.6 – 2.7 nm/s; at 1400 V, the deposition rate was 10–12 nm/s [19–21]. For the process of reactive deposition in a medium of argon and carbon at the same voltages, the deposition rates change. In the region of low voltages at the magnetron, the growth of the WC film occurs both on the substrate surface and on the surface of the tungsten target. Therefore, at low voltages the sputtering rates of the target are lower, since sputtering occurs from the already formed WC phase on the surface of the W target,

where the rates of formation of the WC phase and the rates of its sputtering compete [22–24]. Such a state is observed partially also for the average values of the applied voltage.

Only at a voltage of 1400 V is the growth rate of the WC film comparable to the growth rate of the W film. The structural and electrophysical parameters of films deposited under conventional deposition regimes are, in the general case, analogous to the structures of bulk samples. A characteristic feature of WC films is the presence of a minimum of electrical resistivity

in films of stoichiometric composition. It corresponds, for tungsten, to $0.56 \mu\Omega\cdot\text{cm}$ and, for tungsten carbide, to $0.19 \mu\Omega\cdot\text{cm}$, under the condition of good pumping of the vacuum chamber to residual pressure values of $\sim 5 \times 10^{-6}$ Torr, and further reduction of the oxidizing components of the residual atmosphere – by degassing. The surface morphology of the films was studied by the method of atomic force microscopy. Figure 1 presents AFM images of the surface of a tungsten film at different deposition rates.

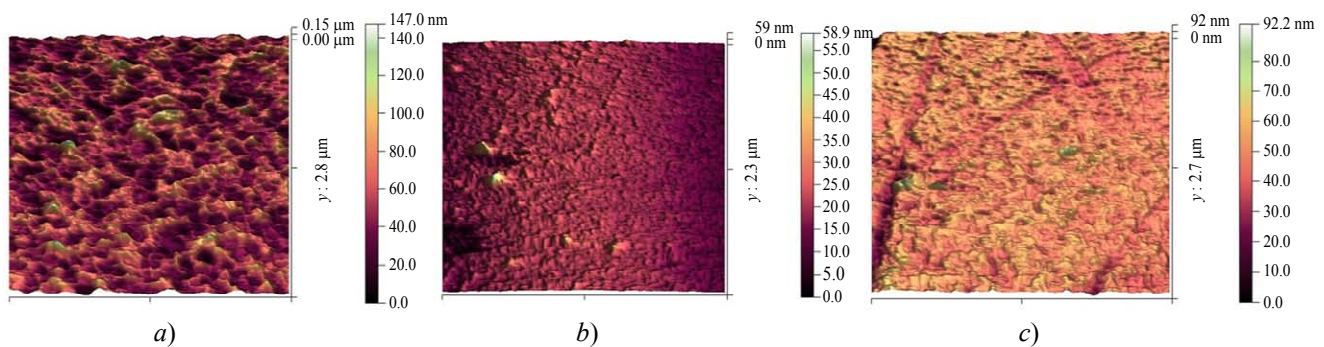


Fig. 1. AFM image of the film surface during sputtering of a tungsten target at different deposition rates. Voltage at the W target: a) – 600 V; b) – 900 V; c) – 1400 V

The images show that with an increasing deposition rate the tungsten surface becomes more uniform. The film surfaces are dense, without large particles, voids, or delaminations, which indicates good adhesion, even though the compressive stress in the tungsten film increases with thickness. At high deposition rates, microcracks appear on the film surface, and at thicknesses of $\sim 1 \mu\text{m}$ and above, the content of other phases increases, which leads to an increase in mechanical stresses due to the mismatch of the parameters of the unit cells of the different phases.

An increase in mechanical stresses leads to an increase in the electrical resistivity of tungsten films and exceeds the resistance of the bulk material by more than an order of magnitude. The films formed on the obtained samples are more uniform and contain mainly the α -phase – this leads to a decrease in electrical resistivity. Figure 2 shows surfaces where tungsten carbide films are

formed. According to literature data [1–5], the formation of WC occurs on the tungsten surface during film growth. Carbon atoms adsorbed on the surface and not having reacted with tungsten diffuse inward, resulting in the formation of W_2C crystals. With an increasing deposition rate and, as a consequence, local heating of the sample surface, the diffusion of C into W is accelerated, the WC content decreases, and W_2C becomes the dominant carbide compound. At low deposition rates, a more ordered but rougher surface is formed. With an increasing deposition rate, the roughness noticeably decreases – this probably occurs due to the lack of time for the formation of larger WC crystals.

It is assumed that under these deposition regimes the content of the pure α -phase of tungsten on the surface and inside the film is minimal, and the metallic tungsten in the near-surface region has completely reacted with carbon, which is confirmed by literature data [6–8].

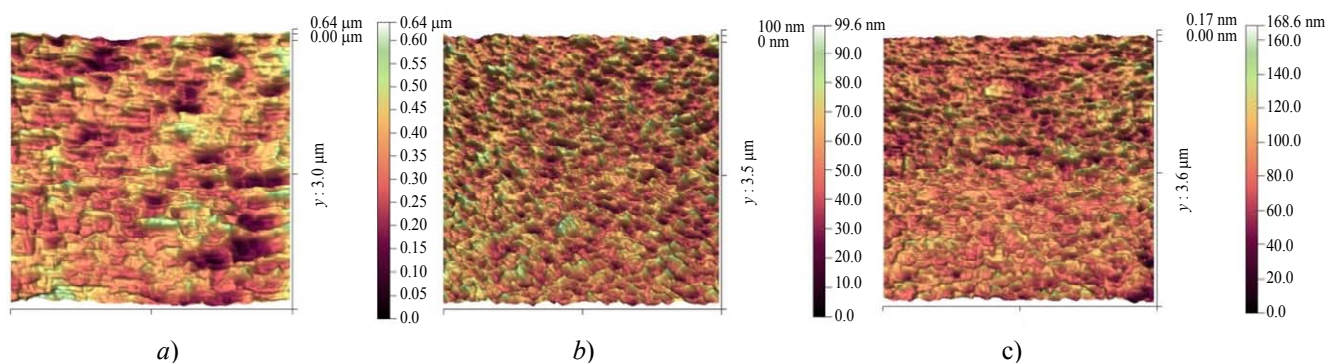


Fig. 2. AFM image of the film surface during sputtering of a tungsten target in a medium of hydrocarbons at different deposition rates. Voltage at the W target: a) – 600 V; b) – 900 V; c) – 1400 V

Findings and Discussion

The study of the film structure was carried out using a Solver P47-PRO scanning probe microscope from NT-MDT. Using atomic force microscopy, the surface morphology of tungsten-graphite films of the composition WC and W_2C was studied in the absence of hydrocarbons during their production. On the basis of the atomic force microscopy data and the particle size distribution plots, an assessment was made of the

characteristic features of the surface relief of WC and W_2C depending on the deposition rate according to key statistical parameters – such as average roughness, root-mean-square roughness, peak-to-valley height, and asymmetry. The results of these studies show a clear difference in the surface characteristics between W and WC films.

In Fig. 3, a plot of the height distribution on the tungsten surface is presented, that is, the dependence of the number of crystallites on their sizes per unit surface area.

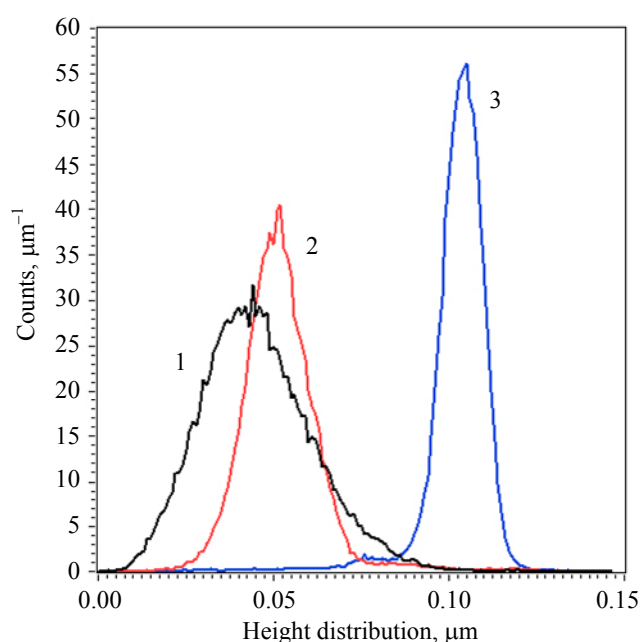


Fig. 3. Height distribution of crystallites on the surface of tungsten films at three deposition rates: 1 – low; 2 – medium; 3 – high

The W films after deposition had a relatively smooth and uniform surface with height variations from 40 to 120 nm. In the presented plot, the width of peak 1 indicates a somewhat greater surface roughness than in the other two peaks. The width of the second peak indicates an optimal and smooth surface. Peak 3 is shifted strongly to the right, since there are microcracks

on the surface that affect the average roughness parameter – the peak-to-valley height, which indicates a more complex surface morphology. On the other hand, WC films demonstrated significantly higher surface non-uniformity (Fig. 4). The differences in height have values from 100 to 500 nm. The broader and more symmetric distribution of the plots confirmed an

increase in the amount of the W₂C phase on the surface of the growing film. The change in surface morphology caused by the growth of the WC film showed that carbon alters the grain structure and increases the surface non-uniformity for all deposition rates.

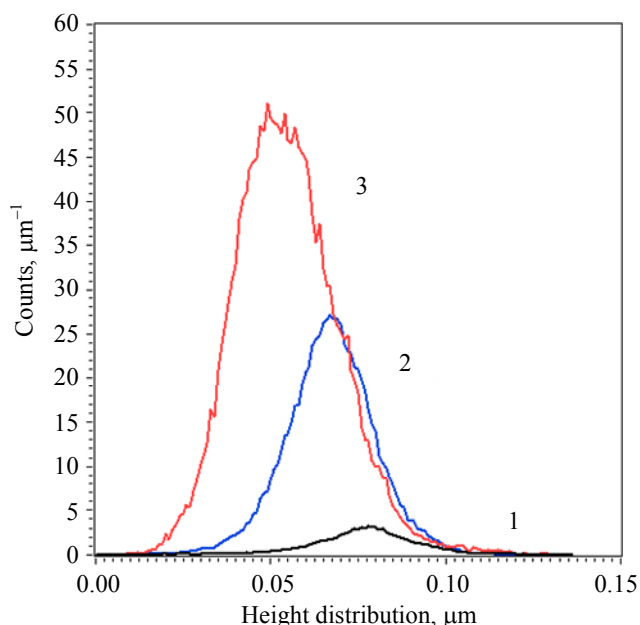


Fig. 4. Height distribution of crystallites on the surface of tungsten carbide films at three deposition rates: 1 – low; 2 – medium; 3 – high

These values additionally show differences in the symmetry and texture of the surface. WC films demonstrated more pronounced surface defects. W films are characterized by balanced

peaks and valleys, whereas for WC valleys predominate.

These results show that the addition of carbon increases the surface area and the relief of the structure of WC films, which makes them potentially more suitable for applications requiring high reactivity of surface-active substances, for example, in gas-sensitive materials or catalytic processes.

Conclusions

Thus, the work proposes and implements a method for obtaining thin polycrystalline WC films by introducing hydrocarbons with low pyrolysis energies into the plasma region and near the substrate. The method was tested on films with a thickness of 0.5 μm. The results of processing three-dimensional (3D) images obtained by AFM confirm the possibility of obtaining polycrystalline WC or WC + W₂C films in a 50:50 proportion, depending on the requirements of the experiment, by the indicated method. The possibility of controlling the "tungsten-carbon" phase composition by changing the discharge voltage at a constant direct current is shown. Further development of this method makes it possible to proceed to the study and manufacture of single-crystal films of smaller thicknesses, as well as metal-carbon films of other refractory metals.

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Features of the dynamics of an alternating current electric arc and its parameters in an air-blown plasma torch

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The paper deals with the results of an experimental study of a single-phase plasma torch with ring electrodes operating in the power range of up to 10 kW. The experiments were conducted with varying plasma gas (air) mass flow rates up to 3.2 g/s. The study methodology included synchronous recording of arc discharge current and voltage waveforms, coupled with high-speed video recording of the electric arc. Based on the obtained data, the arc attachment character was determined and a comprehensive analysis of its temporal and spatial characteristics was conducted. Plasma temperature was estimated using optical emission spectroscopy based on the relative intensity of copper Cu I lines. Calculations revealed that the temperature in the arc discharge zone reaches values of approximately 7000 K.

Keywords: plasma torch, alternating current, air, plasma temperature.

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Introduction

The intensive introduction of plasma torches into modern industrial technologies dictates the need for an in-depth study of the fundamental mechanisms underlying the initiation and maintenance of the stability of the arc discharge. For an adequate reproduction of the complex spatiotemporal dynamics of arc burning, which is especially critical under conditions of fast-occurring and nonstationary processes, the dynamics of arc motion and their attachment points were recorded using modern high-speed digital cameras with high spatial and temporal resolutions. This approach makes it possible to obtain comprehensive information about the morphology of the discharge by recording in detail the instantaneous transformations of its structure and allowing the

trajectories of motion to be traced with high accuracy. This method of comprehensive investigation finds confirmation in works studying the zone of arc attachment to the anode [1, 2], where high-speed imaging acts as a key diagnostic tool, as well as in studies of prototypes of technological synthesis installations in an open air environment, demonstrating similar approaches to recording electrophysical parameters [3, 4]. The methodology of the analysis was based on comparing video recording data with the measured values of the electrical characteristics of the arc discharge – oscillograms of the instantaneous currents and voltage values.

As a theoretical basis for analyzing the collected data, the mathematical apparatus of spectral analysis based on the Fourier transform is used. The Fourier transform makes it possible to represent a complex signal as a set of harmonic

oscillations, enabling the identification of the dominant frequencies. Such an approach allows for a transition from the analysis of the temporal parameters of the signal to the study of its frequency composition. By analyzing the radiation intensity of the plasma, it is possible to reveal the relationship between the spatiotemporal changes in the brightness of the glow and the dynamics of electric arc formation.

The integration of these two approaches – direct visualization and spectral analysis – creates the basis for the development of a new diagnostic protocol. It makes it possible to compare the arc power calculated from the oscillograms of current and voltage across the arc, with its integral luminosity obtained from optical measurements under various video recording geometries.

The aim of the present work is a comprehensive experimental study of the dynamics of arc discharge development in air in a single-housing alternating current plasma torch.

Experimental Setup and Measurement Methods

The studies were carried out on an experimental setup schematically presented in Figure 1. This work is a continuation of many years of research by the author team in the field of designing and creating low-temperature plasma generators [5]. The setup includes an alternating current plasma torch [6], a power supply consisting of a power transformer of the type OS-100/6, and a measurement system. The plasma torch is connected to a system for supplying the plasma-forming gas (gas mixture), a cooling liquid, and is equipped with a unit for regulating and measuring their flow rates. In contrast to previous prototypes, the presented modification is distinguished by an optimized geometry of the interelectrode gap and the use of a gas-dynamic stabilization scheme containing three gas supply circuits for more effective confinement of the arc column.

The power supply of the plasma torch was controlled by an Advantech industrial microcontroller, which provides monitoring of the current and voltage oscillograms recorded by sensors. The air supply system included a manifold rack with pressure regulators for

individual adjustment of the gas supply to each circuit.

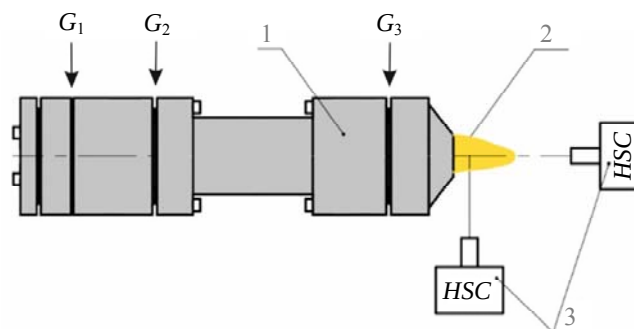


Fig. 1. Schematic view of the plasma torch and observation method using high-speed photo and video shooting: 1 – alternating current plasma torch; 2 – plasma torch plume; 3 – high-speed camera

The diagnostic technique included simultaneous recording of electrical parameters (current and voltage oscillograms) and optical diagnostics of the discharge dynamics. The recording of the electrical parameters of the arc was synchronized with high-speed video recording of the operation of the plasma torch in air (electrode gaps $D_1 = 1.9$ mm, $D_2 = 2.5$ mm) using a Citius imaging High Speed Video Camera.

The camera was positioned in two planes (Fig. 1): frontally to record the discharge inside the chamber and perpendicular to the axis to image the plasma plume at the exit cross-section. The recording was carried out at a frequency of 4000 fps with high resolution and data storage on a PC. This recording method made it possible to determine the location of the arc attachment and to carry out a detailed analysis of the temporal and spatial characteristics of the arc discharge from the time dependences of the plasma radiation intensity. The possibility of remote control of the camera ensured monitoring of the recording process in real time.

It was experimentally established that the studied type of plasma torch ensures stable arc burning and uninterrupted initiation of the arc discharge within the specified working gas flow rates. Analysis of the obtained data made it possible to obtain the electrical and energy characteristics of the setup over a wide range of operating parameters. The dynamics of arc burning is characterized by the corresponding oscillograms [6]. The power was also calculated

as the product of the instantaneous values of current and voltage, which depended on the mass flow rate of air and increased uniformly and linearly, in the range of 6.6–7.1 kW for flow rates of 2.5–3.2 g/s.

Findings and Discussion

The radiation of an arc discharge in an air atmosphere at normal pressure is predominantly continuous in nature, which is due to the high temperature of the arc plasma (6000–10000 K) and the dominance of bremsstrahlung and recombination mechanisms. This continuum, approximated by the Planck function, is superimposed by emission lines and bands of atoms and molecules, whose contribution to the total radiation energy does not exceed 30%. Analysis of the energy balance of radiation [7, 8] shows that the continuous (background) spectrum is the dominant component, while the contribution of the line-band spectrum is estimated at 10–30% of the total radiated energy. The energy distribution in the optical range at a voltage of 4 kV is characterized by the predominance of visible radiation, which accounts for up to 69% of the total energy of the optical spectrum. The energy emitted in the ultraviolet (UV) range is ~ 30%, whereas infrared (IR) radiation makes an insignificant contribution of ~ 1%.

Visualization and diagnostics of the arc plasma dynamics were carried out by the method of high-speed video recording. A camera was used whose photodetector array sensitivity was in the spectral range of 300–1050 nm, which made it possible to record the plasma radiation intensity and reflect oscillations of the arc column, extinction/restart, temperature fluctuations, and the arc shape. The recording was carried out at a frequency of 4000 fps, which, with a mains voltage period of 20 ms (50 Hz), gives a time resolution of 250 μ s per frame, i.e., 80 frames per period. These visual changes are related to changes in current and thermal power and, therefore, it can be assumed that, to a first approximation, they reflect the evolution of the plasma energy of the electric arc.

During high-speed video recording of the discharge process in the plasma torch, video files in .avi format with a resolution of 320×306 pixels

were obtained. For data analysis, preliminary filtering was carried out, after which the total brightness of all pixels in each frame was calculated. This made it possible to form a time dependence of the plasma glow intensity.

Figure 2 presents the obtained frame-by-frame images at different air pressures in the system: $P_1 = 0.4$ atm, $P_2 = 0.8$ atm, and $P_3 = 1.0$ atm, corresponding to the circuits for supplying the plasma-forming gas air, which are designated in Figure 1 as G_1 , G_2 , and G_3 . The horizontal axis (abscissa) shows the frame number, which is equivalent to time with a step of 250 μ s.

As can be seen from Figure 2, a gradual increase in radiation intensity is observed up to frame 421 and then a decrease to 436. Further in Figure 2: the second half-period (10–20 ms), during which a similar character of the radiation intensity dependence is observed. It is interesting to note the characteristic counterclockwise twisting of the arc from the attachment to the plasma torch housing. As can be seen from Figure 2, starting from frame 408 and up to and including 432, the arc attachment (the white point at the bottom of the frame) is stationary. However, for the second half-period, a different character of arc behavior is observed, which rotates quite intensively, and the attachment moves axially – frames 448 to the extinction of the arc at frame 481.

For the difference in the behavior of the electric arc attachment in different half-periods of the supply voltage of the plasma torch, the following explanation can be proposed. The ring-shaped electrodes used in this device are characterized by axial symmetry. As a consequence, the position of the electrodes corresponding to the anode and cathode relative to the plane of the exit nozzle changes vice versa every half-period. This leads to a change in the direction of arc ignition and, accordingly, the direction of the electric current in it. Obviously, the direction of the air flow remains constant, but in one half-period this flow coincides with the flow of electrons from the cathode (negative electrode) to the anode (positive electrode), while in the other, when the polarity changes, it is opposite. This serves as the reason for the difference in the behavior of the arc attachment and its rotation observed in different half-periods of the plasma torch operation. At the same time, such a design of the plasma torch has an

advantage over other devices, since it ensures reliable confinement of the arc on the surface of

the ring electrodes, preventing wear of the electrode edges.

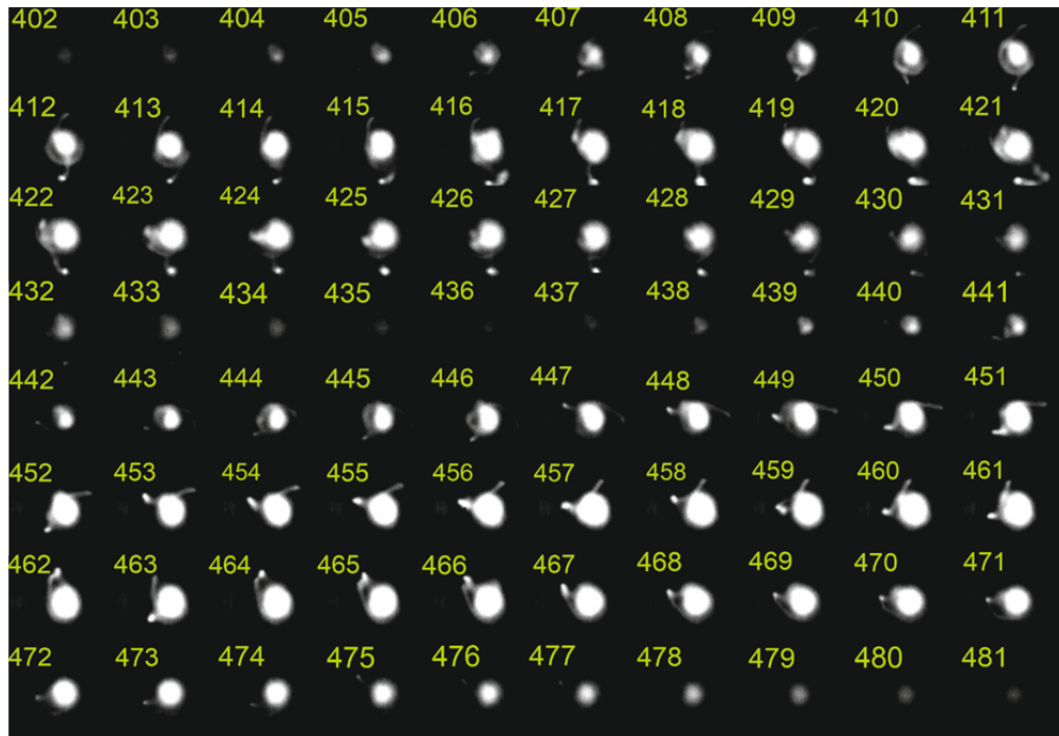


Fig. 2. Frame-by-frame images of the plasma plume over both half-periods: frames 402–441 – first half-period (positive); frames 442–481 – second half-period (negative)

Figure 3 presents the time-frequency characteristics of the arc plasma obtained from the radiation intensity using a high-speed video camera.

In the upper part of Figure 3, a difference in radiation intensity between the half-periods can be noted due to the change in the direction of arc ignition, i.e., the direction of the electric current. Accordingly, in the lower part of Figure 3 there is a frequency of 50 Hz reflecting the difference between the half-periods. The presence of a dominant frequency of 100 Hz confirms the operation of the plasma torch in each half-period. All the measured and processed time signals showed good correlation with the electrical power of the discharge.

The emission spectra in the range of 495–630 nm of the plasma jet at the exit cross-section of the plasma torch were obtained using an Andor Shamrock 303i spectrophotometer with the Andor Solis software. A diffraction grating with 600 lines/mm and a blaze wavelength of 500 nm was used. The slit width was 20 μm . From the intensity ratios of pairs of emission lines Cu I 510.554 nm transition $3d^9 4s^2 \rightarrow 3d^{10} 4p$ and 521.82 nm $3d^{10} 4p \rightarrow 3d^{10} 4d$ with statistical weights $g = 4$ and $g = 6$, respectively; 515.323 nm transition $3d^{10} 4p \rightarrow 3d^{10} 4d$ and 578.213 nm $3d^9 4s^2 \rightarrow 3d^{10} 4p$ with statistical weights $g = 4$ and $g = 4$, respectively, the temperature of the arc plasma was determined, which amounted to ~ 7000 K. Figure 4 shows the emission spectrum of the plasma jet at an air flow rate of 2.52 g/s.

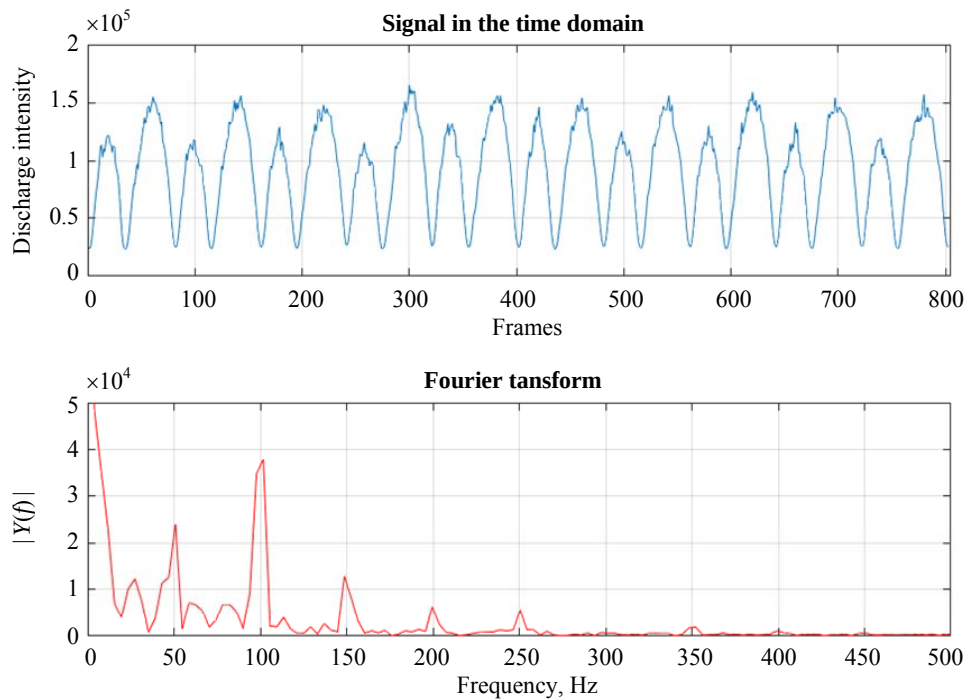


Fig. 3. Characteristics of the arc plasma: top – radiation intensity versus time over 10 full periods of the 50 Hz frequency (250 μ s per frame); bottom – frequency characteristic

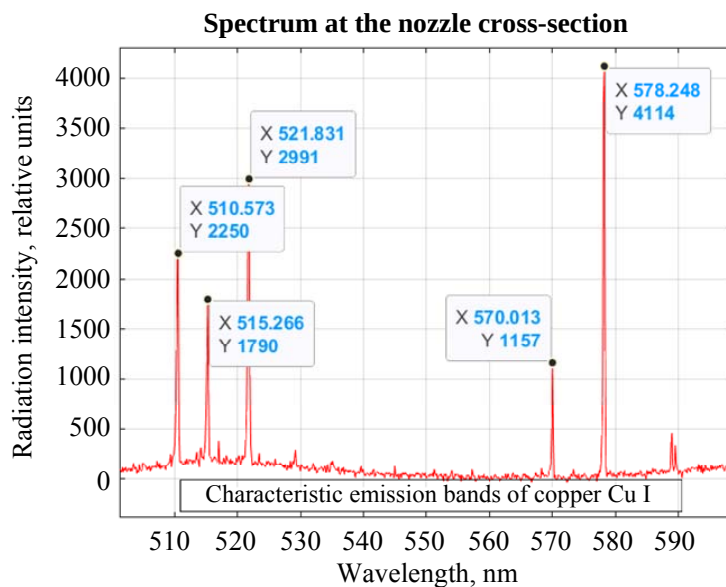


Fig. 4. Emission spectrum of the plasma jet at an air flow rate of 2.52 g/s

Conclusion

The work carried out a study of the discharge development processes in the plasma torch at two different positions of the high-speed camera. The characteristic counterclockwise twisting of the arc from the attachment to the plasma torch housing is noted, with the arc attachment being stationary.

However, for the second half-period, a different character of arc behavior is observed, in

which the attachment moves axially. During the analysis of measurements results we considered the dynamic characteristics related to changes in the process of arc burning in time, the value of arc power was calculated as product of instantaneous current and voltage values. The temperature of the arc plasma, which amounted to ~ 7000 K, was determined from the emission spectra of the copper lines Cu I.

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The influence of wire cutting and selective etching conditions on the structural perfection of CdZnTe 211(B) substrate wafers

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The slicing of CdZnTe ingots using diamond wire cutting was investigated. Optimal process conditions were determined, enabling an increase of up to 30% in the number of wafers obtained from a single ingot compared to the diamond saw cutting method. It is shown that the use of selective etching after slicing allows for the visualization of the dislocation pattern and serves as a reliable method for rejecting substrate material at the early stages of the manufacturing process.

Keywords: cadmium-zinc-telluride (CdZnTe), semiconductor ingot, diamond wire cutting, selective etching.

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The cadmium-zinc-tellurium (CdZnTe, CZT) solid solution is one of the most demanded materials in modern microelectronics. Device wafers based on it are used in the production of infrared (IR) array photodetectors as a substrate material for epitaxial structures of mercury-cadmium-tellurium (HgCdTe, MCT), as well as in X-ray detectors [1, 2]. The main disadvantage of this material is its high cost and limited production volumes, which stem from the specific features of grown CZT ingots, including a block structure, the presence of dislocations and other structural imperfections. Furthermore, the labor-intensive processing technology results in a low yield of acceptable wafers due to the low hardness and high brittleness of the material [3]. Increasing the yield of acceptable wafers at all

stages of production of CdZnTe substrate wafers is an urgent task aimed at improving the availability of IR devices based on HgCdTe/CdZnTe heterostructures.

One of the fundamental process operations is the slicing of a large-block CZT ingot into wafers. The structural features of the grown ingots do not allow for slicing parallel to their base, as is typical for other semiconductor materials, which imposes limitations on the process methods used [4]. The most well-established and commonly used method for separating CZT ingots into wafers remains slicing with a diamond inner cutting blade (DICB). The cutting tool is a metal disc with a central hole and a diamond-based abrasive material applied on its edge (Fig. 1a). This DICB, specially mounted in

a drum and rotating at a speed of 3000–5000 rpm, slices the ingot at an average speed of 40–50 mm/min (Fig. 1b). After slicing off each wafer, the mechanism returns the ingot to its original position, moves it by a specified step, and the cycle repeats.

When a large-block CdZnTe ingot is sliced using a DICB, the thickness of the cut blank wafer is about 2000 μm , which is necessitated by the brittleness of the CZT material. A reduced in thickness of the cut wafer increases the risk of mechanical fracture, which likely occurs due to micro-vibration of the DICB during slicing. After wafers are separated from the ingot by blade cutting, a typical pattern with a “rounded” morphology is observed on the surface (Fig. a),

and the depth of the damaged near-surface layer is about 100 μm on each side of the cut wafer, taking into account the physical and mechanical features of the CZT material [5]. Investigation of a typical surface pattern of a CZT wafer sliced with a DICB shows a height variation of 25–35 μm (Fig. 2b).

Considering the depth of the damaged layer after such slicing, at least 300 μm in total is removed from both sides of the blank wafer during further processing operations. Subsequent grinding/polishing operations reduce the wafer thickness to the required $900 \pm 100 \mu\text{m}$. Thus, material losses during the operations following the DICB slicing alone amount to more than half of the cut wafer thickness.

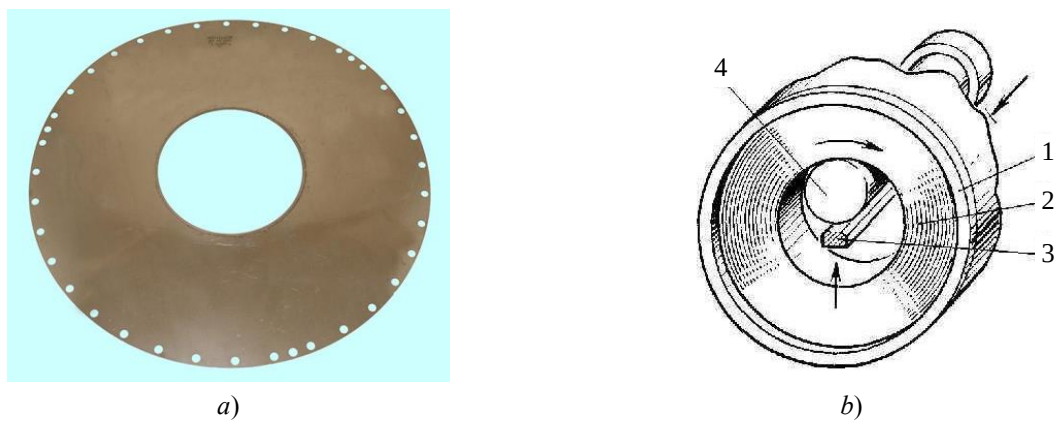


Fig. 1. Diamond inner cutting blade with peripheral mounting holes (a) and ingot slicing scheme (b), where 1 – cylindrical drum; 2 – DICB; 3 – mandrel holder; 4 – ingot

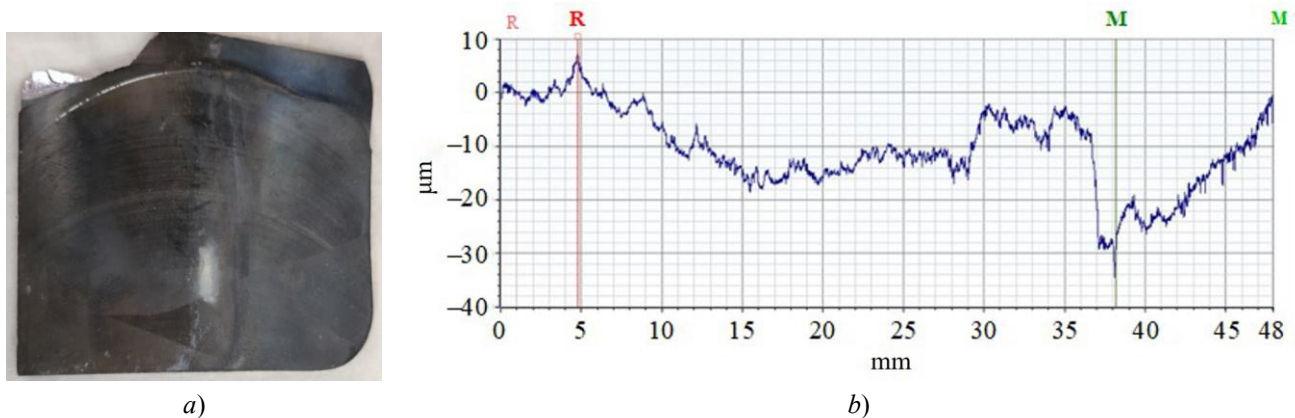


Fig. 2. Surface of a CdZnTe wafer after DICB slicing: a) – appearance of the wafer; b) – measurement of the surface pattern using a contact profilometer

The next generation in the evolution of methods for slicing semiconductor ingots into wafer blanks is the wire slicing method (with diamond-coated wire or with an additional supply of abrasive material into the cutting zone). The main advantages of this method (Fig. 3) are the use of a wire with a relatively smaller diameter compared to the thickness of a DICB, which allows for reduction in the kerf width and, consequently, minimizes material losses during slicing. Furthermore, the natural damping provided by the wire flexibility reduces thermodynamic loads, which in turn reduces the damaged layer thickness. This allows for the slicing of thinner wafers, thereby increasing the yield of wafers obtained from a single ingot.

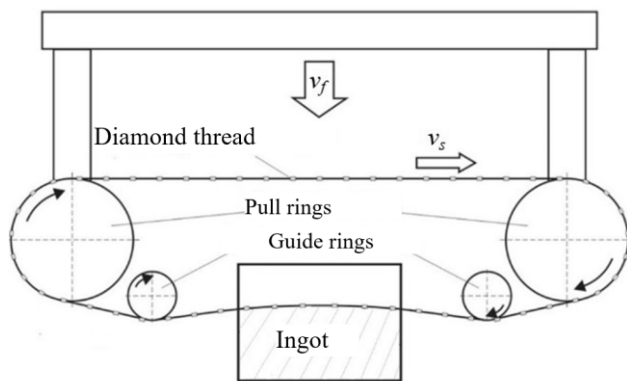


Fig. 3. Schematic diagram of the wire slicing setup

However, to exploit the above-mentioned advantages of the diamond wire slicing method, it is necessary to determine the optimal process conditions (e.g., ingot feed rate, wire drum rotation speed, cooling fluid supply, etc.), while considering the mechanical and structural features of the semiconductor material being processed.

Furthermore, one of the aspects affecting process productivity is the mounting of the ingot on the coordinate table of the slicing setup. The criteria for selecting a mounting material include rapid hardening, easy release from the wafer/ingot, and the ability to maintain shape during slicing despite minor vibrations and a continuous flow of water.

As a result of research and analysis of various mounting materials, the following combination was identified: gypsum is used for creating a mold and fixing a sample on the coordinate table of the slicing setup, and a cyanoacrylate adhesive is applied at the contact points between the semiconductor ingot and the gypsum. This combination of materials withstands the impact of water both as a flow and during immersion. This allowed mounting of the sample on the coordinate table of the slicing setup steadily and to control over the process of separating the ingot into wafers (Fig. 4).

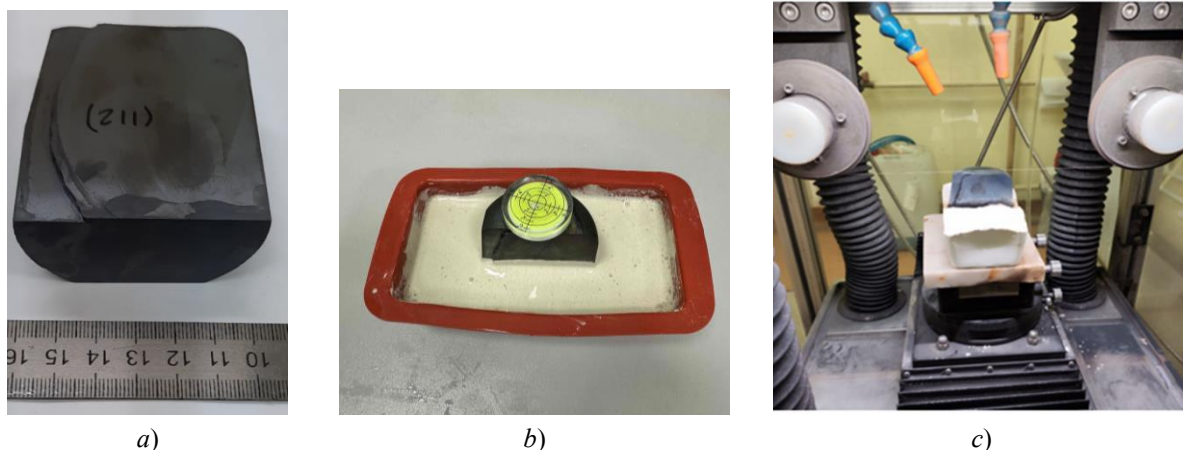


Fig. 4. Large-block CdZnTe ingot: a) – oriented in direction (211); b) – in a special retaining mold; c) – secured on the coordinate table of the slicing setup and ready for separation into wafers

The use of diamond wire for slicing a large-block CZT ingot allowed reducing the thickness of the processed wafers down to 1400 μm without loss of mechanical strength (Fig. 5). The suitability of such wafers for further

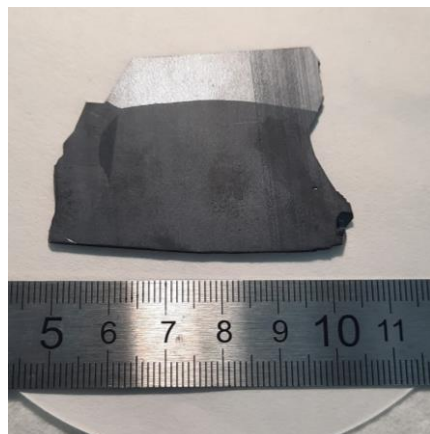
grinding/polishing operations was confirmed at subsequent processing stages.

The surface morphology of CZT wafers sliced by the diamond wire slicing method was also studied using the contact profilometry

(Fig. 6). The height variation is less than $20\text{ }\mu\text{m}$, which does not exceed the same parameter for wafers sliced with a DICD.

To assess the depth of the near-surface damaged layer on a CZT wafer after diamond

wire slicing using various process approaches to optimize slicing conditions, the lateral cleavage edge of the wafer was studied using the scanning electron microscopy (Fig. 7).



a)



b)

Fig. 5. CdZnTe wafer cut by diamond wire slicing method: a) – appearance of the wafer; b) – thickness measurement using a digital micrometer

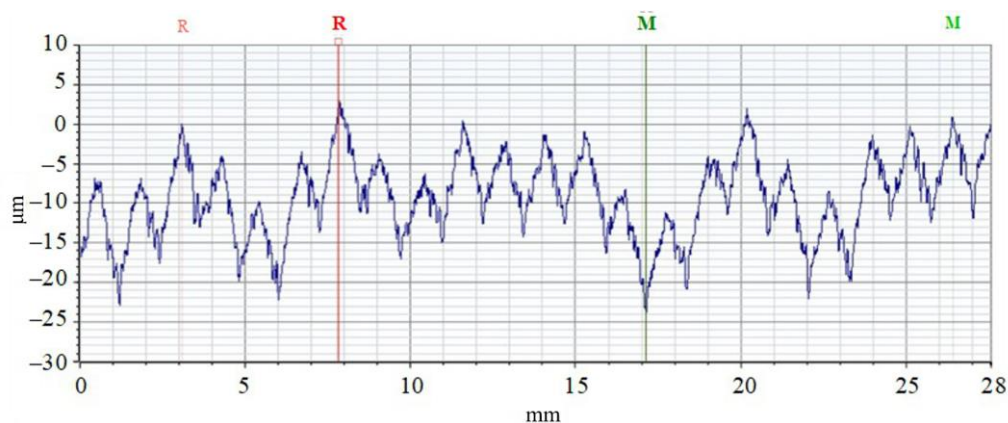


Fig. 6. Surface pattern of a wafer sliced using diamond wire

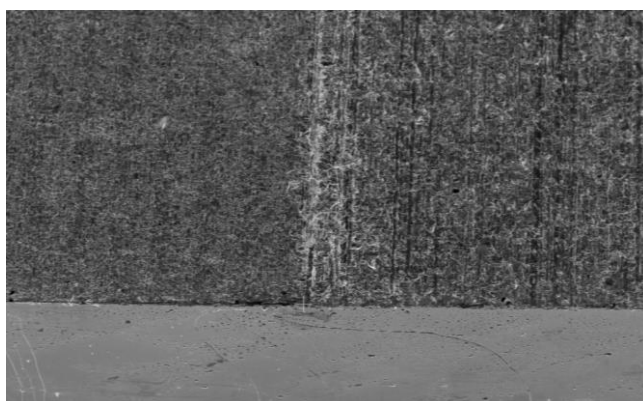


Fig. 7. General view of the investigated wafer fragment at an angle of 40°

In Fig. 7, the interface between the processed areas in a more optimal (left) and non-optimal (right) mode can be clearly seen.

More detailed examination of the near-surface damaged area assessed the damaged layer depth

at the level of $10\text{--}15\text{ }\mu\text{m}$ under optimal slicing conditions (Fig. 8a) and of about $40\text{--}50\text{ }\mu\text{m}$ under non-optimal conditions (Fig. 8b), which confirms the importance of properly selecting the process parameters for this operation.

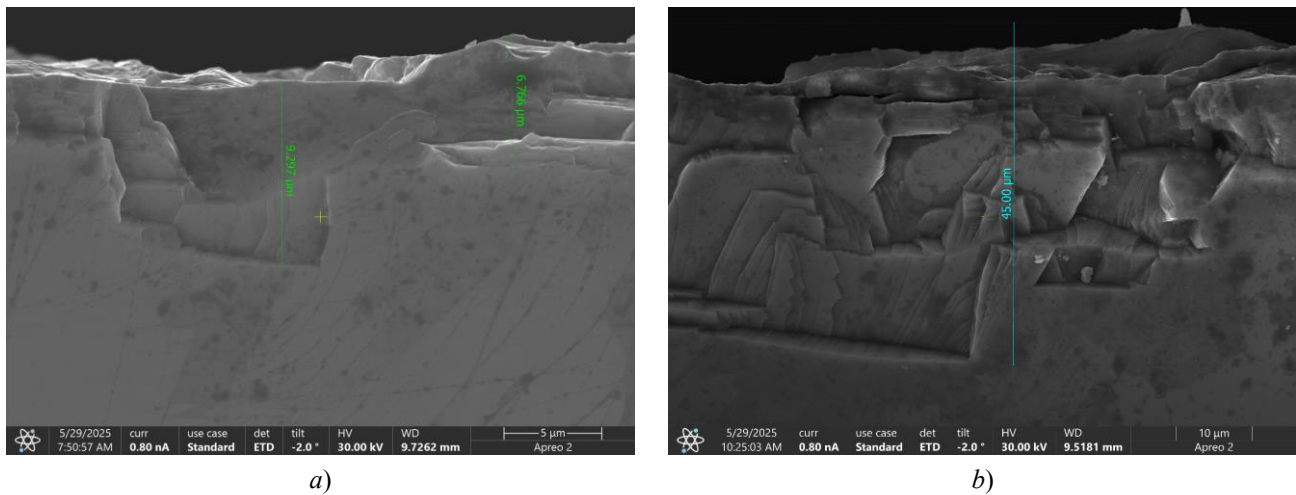


Fig. 8. Investigation of the near-surface damaged layer depth of a CZT wafer after diamond wire slicing:
a) – area under optimal slicing conditions; b) – area under non-optimal slicing conditions

Thus, the study of possible applications of diamond wire slicing for separating large-block CZT ingot and determination of process conditions allowed for a reduction in the thickness of the cut wafers, the height variation of the wafer surface pattern, and the depth of the resulting near-surface damaged layer. Combined, these improvements reduce overall material losses and increase the yield of usable wafers at this stage as compared to DICB slicing.

Then, the sliced wafers undergo defect structure analysis to determine the single-crystalline region, block structure, and/or twinning over the wafer area, identify the A/B sides and count the dislocation density. Specific features of the material under consideration include its pronounced softness, low critical shear stress, and low stacking fault energy, which result in relatively easy dislocation generation during crystallization in the ingot growth process [6]. Dislocations in the crystal lattice of the material have a direct impact on the quality of epitaxial structures grown thereon. While defects caused by mechanical processing at the slicing and grinding stages can be minimized by subsequent polishing, growth defects extend throughout the entire depth of the material. Detection of a wafer's non-compliance with structural requirements at this production stage allows for its rejecting it in due time, thus preventing obviously unsuitable samples from proceeding to

the labor-intensive processes of subsequent mechanical and chemical-mechanical processing, as well as epitaxial growth, thereby reducing costs and increasing the yield of usable devices [7].

One of the optimal methods for revealing the dislocation pattern in substrates is selective chemical etching. In addition to dislocations, this technique allows for the visualization of twins and block structures, thereby helping to identify the single-crystalline region and determine the polarity of the wafer sides, which is important for future growth processes. Since the anisotropy of the crystal properties need accounting for during the development of selective etching, the etchant is selected based on the crystallographic orientation of the wafers under study. To reveal the dislocation pattern and determine the polarity of wafers with a (211)B orientation, Everson's solution was used [8]. Prior to the selective etching process, it is necessary to remove the damaged layer formed after ingot separation into wafers, which is achieved by chemical-mechanical polishing of the wafer on both sides.

A specific pattern becomes visible on the wafer after selective etching, allowing for the determination of the polarity (sides A/B) of the single-crystalline region, blocks, and twins (Fig. 9). Side A acquires a matte-dark color due to cadmium (Cd) enrichment, whereas side B has a metallic luster due to tellurium (Te).



Fig. 9. Appearance of a CdZnTe wafer (211)B after selective etching. The contour marks the single-crystalline region on side B of the cut wafer section

After selective etching, optical microscopy is used to analyze the wafer surface in the marked region on side B to determine the dislocation density. Dislocation pits have an asymmetric shape with a clearly defined pointed apex (Fig. 10) due to the deviation of the plane of the crystallographic orientation under consideration (211) from the reference plane (111) by 19.5° , which induces anisotropy in the pit projection on the surface. Pits with round or irregular contours lacking a pronounced apex do not originate from dislocations (for example, they represent tellurium precipitates or artifacts of mechanical processing) and are not counted in the calculation. If they significantly distort the overall pattern, they can be studied separately.

To estimate the dislocation density value correctly, measurements are taken over several equally spaced fields, followed by calculation of the average value. The optimal number of fields is selected depending on the size of the marked region. The edge zone with a width of 2-3 mm is excluded from consideration, since the defect density in it is systematically higher due to the contact of the melt with the crucible walls during crystal growth, while peripheral areas of the substrate are generally not involved in formation of active elements during manufacture of array photodetectors.

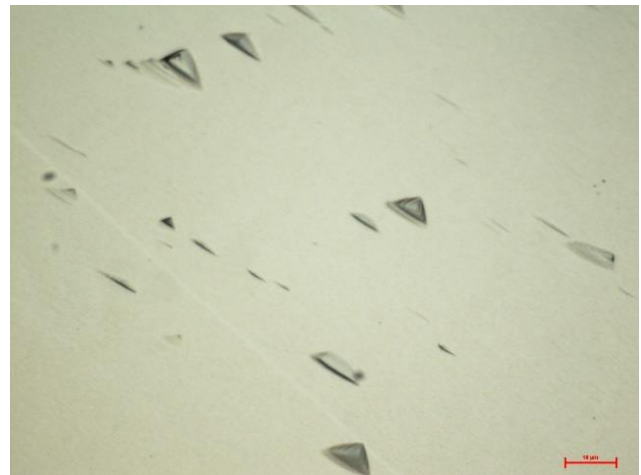


Fig. 10. Typical view of the dislocation pattern of the single-crystalline region of a CdZnTe wafer (211)B after selective etching

The criterion of suitability for the epitaxial growth of HgCdTe during the fabrication of IR devices is a value of not more than $3 \times 10^4 \text{ cm}^{-2}$ [9, 10]. During the study, the dislocation density was calculated on the substrate wafers sliced by the method considered herein. It was established that the samples meet this criterion, which allowed to conclude that they are suitable for further processing operations.

Conclusion

Thus, the application of the considered process solutions optimizes the manufacturing process of CdZnTe wafers (211)B. Compared to slicing with a DICB, the separation of large-block CZT ingots into wafers using the diamond wire slicing method allows for the slicing of thinner ($1400 \mu\text{m}$ vs $2000 \mu\text{m}$) blank wafers with a shallower damaged layer, while maintaining wafer integrity. This increases the number of wafers obtained from a single ingot by up to 30% and also reduces the subsequent processing time.

Selective etching to reveal the spatial non-uniformity of defect distribution, followed by the dislocation density calculation, allows for assessing the suitability of the wafer for subsequent epitaxial growth operations, thereby facilitating the early rejection of unsuitable samples and establishing a basis for reproducible incoming qualification of substrates.

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Modeling the parameters of the multi-wire cutting mode, ensuring high surface quality of InSb (100) wafers

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The surface preparation processes for InSb plates of the "Epi-ready" class include a number of machining processes that significantly affect the quality of the final products. One of these processes is wire cutting of single crystals. In this study, the issue of calculating the conditions of wire cutting and determining the limiting parameters for the movement of the desktop, as one of the factors affecting the BOW and WARP plates, was studied. The experimental work was carried out to determine the cross-sectional sections of the InSb single crystal required for segmentation, and the cutting speed was calculated for each of them. The practical confirmation of theoretical calculations with an effective change in surface parameters is shown.

Keywords: A^{III}B^V, InSb, wire cutting, optical profilometry, wire cutting parameters, calculation of worktable speed.

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Introduction

A^{III}B^V compounds are currently the main materials for the manufacture of focal plane arrays (FPAs) [1], which are created using the technology of epitaxial growth of layers, which requires high-quality substrate surfaces. Obtaining "epi-ready" class A^{III}B^V wafers is one of the main challenges for the development of microelectronics [2]. The technology for producing FPAs consists of many stages, including the mechanical processing of single crystals, which involves main operations such as wire sawing into wafers, subsequent grinding, and chemical-mechanical polishing. Therefore, improving the quality of the wafers requires optimization of the processing regimes at each

stage of their manufacture. The separation of single crystals into wafers is performed by wire sawing. Although this process receives limited attention, it exerts a substantial influence on the surface parameters on the bow (BOW) and warp (WARP) of finished "epi-ready" wafers. This technological operation largely determines the quality of future products. For this reason, multi-wire sawing requires a more detailed analysis regarding its impact on the qualitative characteristics of wafers.

In a previous work [3], the main wafer surface parameters determined during the multi-wire sawing stage – specifically, wafer bow (BOW) and wafer warp (WARP) – were identified. These parameters can either increase or decrease depending on the specified processing

conditions. To identify the degree of significance of this surface parameters, wafers were selected after sawing, grinding, and polishing, allowing the trends in their evolution to be tracked across each technological step.

The main feature in the processing of InSb is that these single crystals exhibit significant bulk stresses. During the mechanical processing of InSb, the depth of the subsurface damaged layer is larger than in Si and GaAs. This requires the removal of a significant layer during grinding and subsequent polishing. "Gentle" sawing regimes can reduce the depth of this structurally damaged layer, leading to material savings in obtaining "epi-ready" wafers.

The main technology for the mechanical processing of InSb relies on multi-wire sawing with a constant stage feed rate. However, based on the analysis in the article [4], it can be hypothesized that varying the cut width of the sample alters both the wire runout and the force exerted by the wire on the sample. A uniform distribution of this force it can be hypothesized would favorably affect the surface parameters. This hypothesis arises from findings indicating

that the surface roughness of a wafer cut by wire sawing is usually nonuniform, which can create problems during subsequent grinding and polishing. Also, the parametric studies presented in the article [4] show that the vibrational displacement of the wire decreases with increasing distance of contact between the wire and the crystal.

Experiment

In the work [5], a model was proposed into which some corrections were introduced considering the conditions of InSb sawing. During the sawing, a wire with a tension T acts on the InSb crystal (Fig. 1). As sawing progresses, the wire deflects by a certain angle α and applies a total force F_{total} to the sample. In the Y -direction, the force of action F_{total} is equal to the reaction force F_{total}^K , since the force with which the wire acts on the InSb crystal equals to the force with which the crystal acts on the wire [6]. Thus:

$$F_{\text{total}}^K = F_{\text{total}} = 2T \sin \alpha. \quad (1)$$

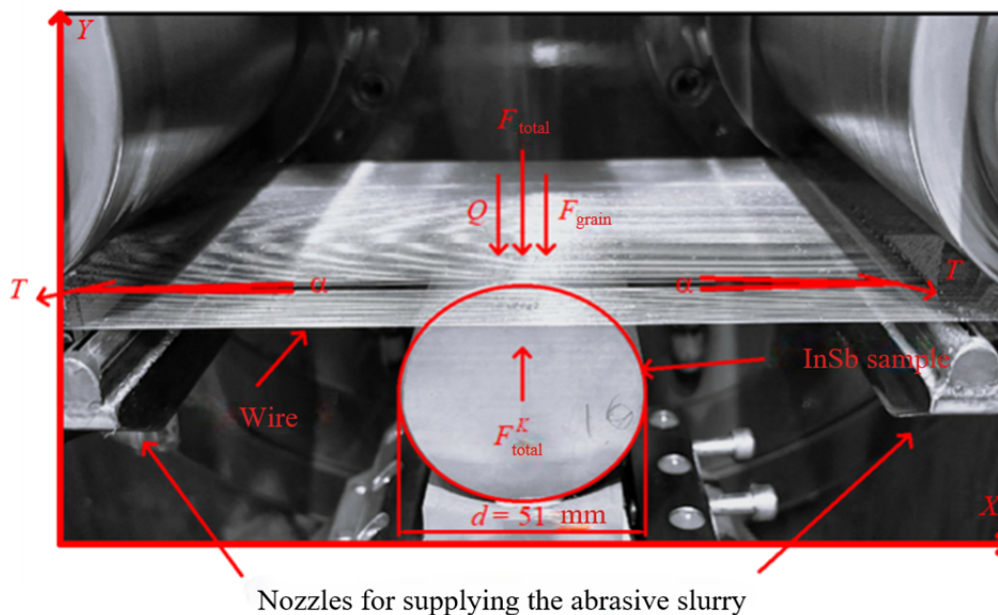


Fig. 1. General view of the InSb sample being cut

The force of the wire's action on the crystal F_{total}^K is not applied to the crystal at a single point, but is distributed over the entire cut width – L' . Let us denote this distribution as – Q . Thus, we obtain the expression:

$$Q = \frac{F_{total}}{L'} = \frac{2T \sin \alpha}{L'}. \quad (2)$$

In addition, a distinct force is applied to each abrasive grain entering the cutting zone. This force can be calculated by dividing the total applied force F_{total} by the number of abrasive grains in the cutting zone. To calculate the number of abrasive particles (N), it is necessary to divide the cut width of the InSb crystal (L') by the distance between the abrasive particles (L_{part}).

$$N = L_o / L_g. N = \frac{L'}{L_{part}}. \quad (3)$$

The force applied to a single abrasive grain, F_{grain} , is obtained by dividing the total applied force F_{total} by the number of abrasive grains (N) located along the entire length of the cut:

$$F_{grain} = \frac{F_{total}}{N} = \frac{2T \sin \alpha}{L_{part}} \times L'. \quad (4)$$

The configuration of the applied forces (F_{total}^K , F_{total} , F_{grain}) in the real process (Fig. 1) is also presented as a vector diagram in Figure 2.

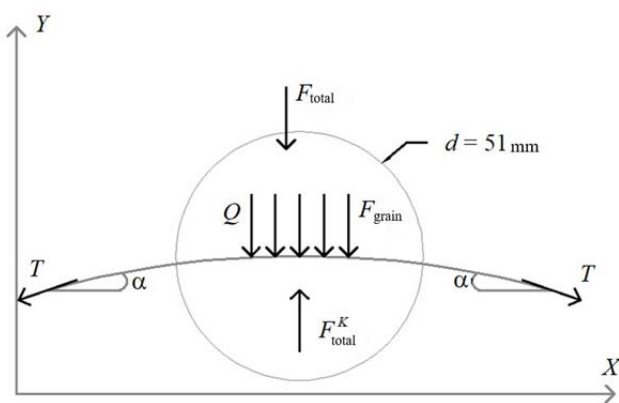


Fig. 2. Vector representation of the forces acting during wire sawing on the InSb sample being cut

Due to the circular cross-section of the InSb single crystal, the cut width L' is not constant and varies as the cutting progresses. The supplied flow of slurry into the cutting zone remains constant; consequently, the same number of abrasive particles enters the wire sawing region, allowing the distance between the abrasive particles L_{part} to be considered constant (const). In such a case, the distribution of the total force acting on the crystal F_{total} directly depends on the cut width of the InSb single crystal. Under conventional sawing conditions with a constant feed rate (table feed rate V_{table}) the variable cut width of the circular InSb crystal causes the force exerted by the wire to also vary. This leads to qualitative variations in the surface topology of the cut wafer (Fig. 3a). These differences manifest themselves as five surface non-uniformity regions when measuring its topology using the Microprof optical profilometry system (Fig. 4). This system enables non-contact topography imaging and measurement of wafer geometric characteristics, such as total thickness variation of the wafer (TTV), bow (BOW), warp (WARP), and total indicator reading (TIR).

To equalize the force exerted by the wire on the single crystal and eliminate these five non-uniformity regions, it is proposed to implement a blockwise sawing method by calculating and adjusting a specific feed rate for each of the blocks. For this purpose, the cross-section of the 51-mm-diameter InSb crystal was divided into 20 blocks. Thus, the height of each block is 2.55 mm (Fig. 3b).

Each block has a cut width limited by its sector, located above and below the block. The cut width for the beginning of each block is indicated in Table 1. Analyzing the surface topology of the wafer after sawing revealed that the five non-uniformity regions on the 51-mm-diameter InSb wafer strictly correlate with the sector indices presented in the table (Table 2).

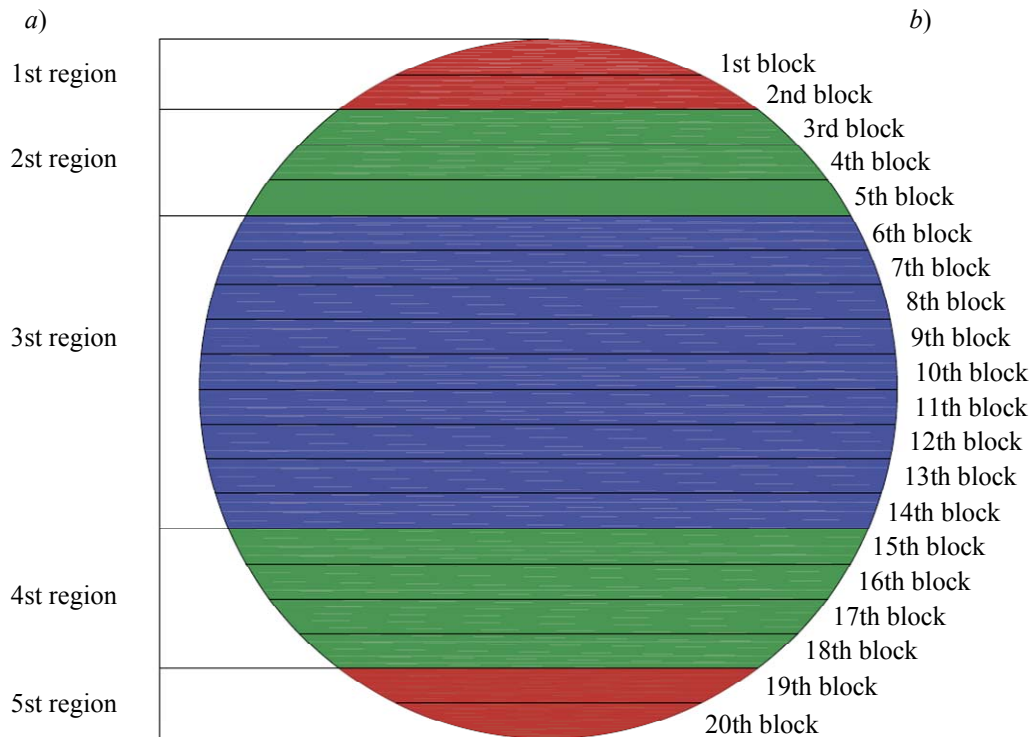


Fig. 3. Schematic representation of the InSb 2'' wafer being cut with division: a) into regions of nonuniformity obtained as a result of measuring the surface of the cut wafer by means of optical profilometry, cut at a constant sawing speed $V_{table} = 8$ mm/h; b) into 20 blocks, for each of which it is necessary to calculate its own value of cut width and sawing speed. Height of each block: 2.55 mm

Table 1

Main variables in the calculation and their values

$V_{table} = 8$ mm/h	–	standard constant table feed rate (mm/h)
V_{block}	–	sawing speed calculated for each of the 20 blocks (mm/h). For blocks No. 1, 20, the sawing speed is taken equal to 8 mm/h
Q	–	value of wire tension (N)
α	–	wire deflection angle (degrees)
F_{total}	–	total force applied by the wire to the sample being cut (N)
F_{total}^K	–	force with which the crystal acts on the wire (N)
L'	–	cut width of the single crystal (total width of contact between the wire and the InSb crystal) (mm)
Q	–	distribution of the force of the wire's action on the crystal per unit cut width (N/mm)
F_{grain}	–	force applied to one abrasive grain (N)
N	–	number of abrasive particles (pcs.)
L_{part}	–	distance between abrasive particles (mm)
n	–	ordinal number of the block; in the calculation formula n varies successively from 1 to 18
l_n	–	n -value of the width of the beginning of the block
k	–	coefficient of the ratio of the average value of the width of block n to the average value of the width of block $n + 1$

Table 2**Results of measuring the width of the beginning of each block**

No. of the region of nonuniformity	No. of the sawing block (n)	Width of the beginning of the block (l_n) (mm)
1	1	0.00
1	2	22.23
2	3	30.60
2	4	36.42
2	5	40.80
3	6	44.17
3	7	46.74
3	8	48.65
3	9	49.97
3	10	50.74
3	11	51.00
3	12	50.74
3	13	49.97
3	14	48.65
4	15	46.74
4	16	44.17
4	17	40.80
4	18	36.42
5	19	30.60
5	20	22.23

For each block, it is necessary to determine the parameters of the sawing process, namely the stage feed rates for a uniform distribution of the total force F_{total} that the wire applies to the InSb single crystal being cut. This can be achieved by calculating the average cut width for each block, then determining the ratio coefficient of the average width of block n to that of block $n + 1$, in order to determine the ratio of the blocks to each other. The resulting coefficient is then successively multiplied by each successive feed rate value. Then blockwise feed rate V_{block} is determined as:

$$V_{\text{bl}} = V_{\text{lb-1}} \times \frac{(l_n + l_{n+1})}{\frac{(l_{n+1} + l_{n+2})}{2}}, \quad (5)$$

where n takes a value from 1 to 18, and $V_{\text{block-1}}$ begins with 8 mm/h for the first calculation value. This parameter was chosen as the initial feed rate, since it corresponds to the constant sawing speed (table feed rate) in the initial process, against which the analytically modified process is compared (Table 3).

Table 3**Calculation of the sawing speed for each block in the process modified on the basis of the calculations**

No. of the sawing block (n)	Width of the beginning of the block (l_n) (mm)	Average value of the cut width for each block (mm)	Coefficient k	Calculated sawing speed for each block V_{block} (mm/h)
1	0.00	11.12	1.00	8.00
2	22.23	26.42	0.79	7.98
3	30.60	33.51	0.87	6.31
4	36.42	38.61	0.91	5.47
5	40.80	42.48	0.93	4.97
6	44.17	45.45	0.95	4.65
7	46.74	47.70	0.97	4.43
8	48.65	49.31	0.98	4.29
9	49.97	50.36	0.99	4.20
10	50.74	50.87	1.00	4.15
11	51.00	50.87	1.01	4.15
12	50.74	50.36	1.02	4.20
13	49.97	49.31	1.03	4.29
14	48.65	47.70	1.05	4.43
15	46.74	45.45	1.07	4.65
16	44.17	42.48	1.10	4.97
17	40.80	38.61	1.15	5.47
18	36.42	33.51	1.27	6.31

End of table 3

No. of the sawing block (n)	Width of the beginning of the block (l_n) (mm)	Average value of the cut width for each block (mm)	Coefficient k	Calculated sawing speed for each block V_{block} (mm/h)
19	30.60	26.42	1.27	7.98
20	22.23	11.12	1.00	8.00

Note: Here k is the coefficient of the ratio of the average value of the width of block n to the average value of the width of block $n + 1$, where n is the block index. Coefficient $k = 1$ for blocks No. 1, and 20, as their sawing speed is initially defined as 8 mm/h. For block No. 10, the coefficient is also $k = 1$, since the ratio of the average value of the cut width for block No. 10 to that of block No. 11 is equal to 1. The coefficient k gradually increases toward 1 from block No. 2 to block No. 10, since the width of block n is smaller than that of block $n + 1$ and successively increases in the indicated range. Conversely, the coefficient k is greater than 1 from block No. 11 to block No. 19, since the width of block n is larger than that of block $n + 1$ and successively decreases in the indicated range.

Results and Their Discussion

To carry out the experiment, a Takatori wire sawing machine equipped with a free abrasive cutting system (an abrasive slurry consisting of oil and silicon carbide powder SiC) was used. Two wire sawing processes were performed on 51-mm-diameter InSb single crystals grown via the Czochralski method under identical technological conditions. The first was conducted at a constant feed rate [7] ($V_{\text{table}} = 8$ mm/h), while

the second utilized the blockwise feed rates (V_{block}) calculated for each block (Table 3).

The first and second sawing processes yielded 50 and 47 wafers, respectively. The thickness of the wafer being cut was ≈ 1 mm. After sawing, the surface topography [8] of all InSb wafers was imaged. The scatter of the surface parameters across the measured wafers remained within a 3–5% range. Figure 4 presents a typical surface topography image for each of the performed processes.

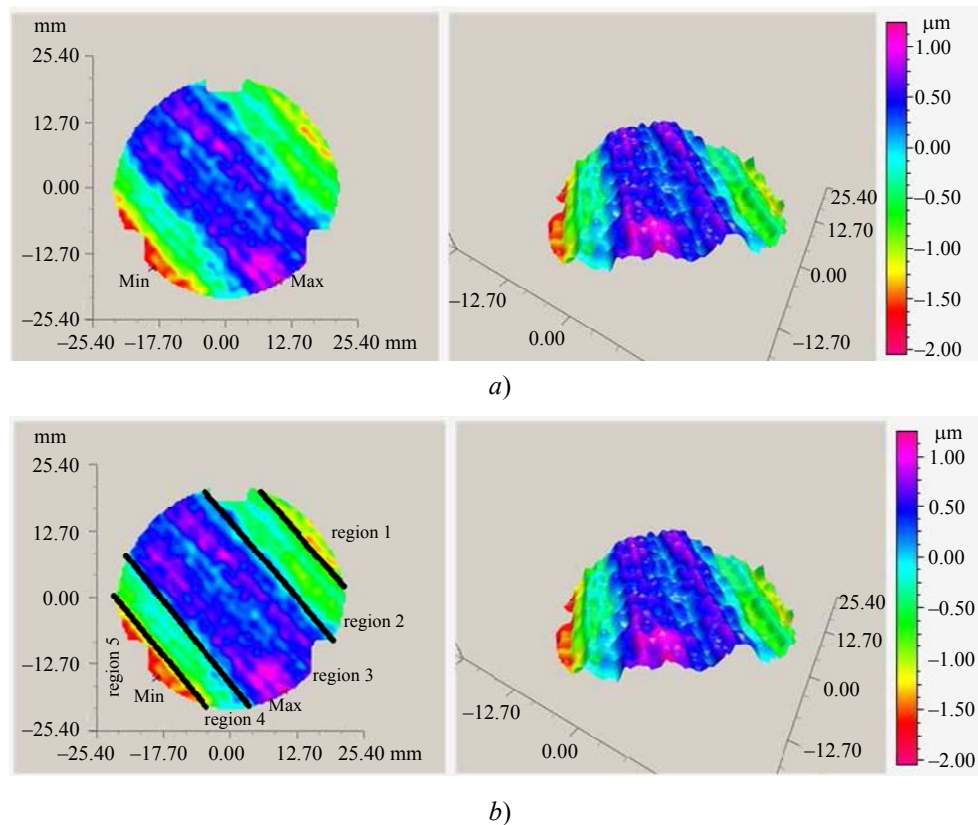


Fig. 4. Surface topography image of an InSb wafer cut at a constant sawing speed $V_{\text{table}} = 8$ mm/h: a) initial surface topography image; b) schematic representation of the division of the wafer into sectors with differing surface quality

This image reveals a distinct bow of the wafer. From the surface topography, the wafer can be divided into five non-uniformity regions, the boundaries of which are marked in the figure with different colors. The measured boundaries of the regions agree well with the scheme proposed in Figure 3.

The calculated wire sawing speeds (Table 3) were entered into the control program for each of the 20 blocks, after which the InSb single crystal

was sawed with subsequent surface topography imaging of the obtained wafer. The result of this measurement is presented in Figure 5.

In this topographic image, the wafer bow is less pronounced. Unlike the results shown in Figure 4, the five previously distinct non-uniformity regions were not observed in this topography. Measurements of the surface parameters [9] for both sawing processes are presented in Table 4.

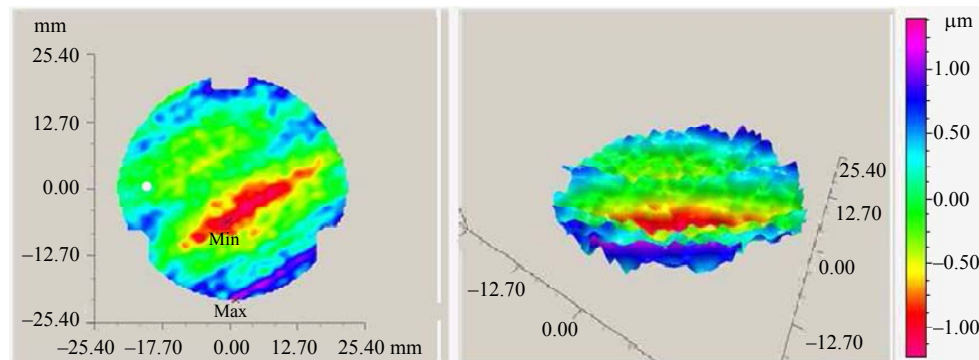


Fig. 5. Surface topography image of an InSb wafer cut at a calculated variable sawing speed

Table 4

Measurement of surface parameters [10] for the sawing process performed at a constant speed $V_{\text{table}} = 8 \text{ mm/h}$ and for the sawing process performed at a feed rate calculated for each of the 20 blocks

	Magnitude of measurement of surface parameters (μm)				
	TTV	TIR	FPD	WARP	BOW
InSb with constant speed $V_{\text{table}} = 8 \text{ mm/h}$	3.189	2.196	1.409	3.284	0.85
InSb with calculated speed	2.942	2.618	1.378	2.605	0.642

The abbreviations in the table designate: total thickness variation of the wafer (TTV), wafer bow (BOW), wafer warp (WARP), total indicator reading (TIR), maximum distance from the surface to the reference plane (FPD). The values of the measured parameters are given in μm .

The surface parameter measurements demonstrate (Table 4), that the transition from a constant sawing speed to a blockwise wire sawing process, divided into 20 blocks, with optimized feed rates significantly affects the resulting surface quality. According to significance, the obtained surface parameters can be classified as high-priority or low-priority. The prioritization is based on whether specific parameters can be substantially changed in subsequent processing operations (grinding and polishing) or if they are inherently determined during the wire sawing stage and change negligibly at later fabrication steps. Thus, the high-priority parameters include wafer bow (BOW) and wafer warp (WARP). As a result of the experiment, the BOW and WARP

parameters decreased by 25% and 21%, respectively. At the same time, the changes in the remaining parameters were the following: total thickness variation of the wafer (TTV): -8% – total indicator reading (TIR): $+19\%$ – maximum distance from the surface to the reference plane (FPD): -2% .

Conclusions

1. A positive trend in the surface parameter evolution of the sawed wafers has been established. It is shown that the force exerted by the wire on the sample varies with changes in the cut width. Ensuring a uniform force distribution

relative to variations in the sample width favorably affects the surface parameters, which is confirmed by both the analytical calculations and experimental results.

2. The positive trends in the surface parameter evolution of the wafer consists mainly in a substantial change in wafer bow (BOW) and

wafer warp (WARP). In contrast, the change in total thickness variation of the wafer (TTV) and focal plane deviation (FPD) are negligible. The increase in the total indicator reading (TIR) can also be considered insignificant, since this parameter can be corrected during subsequent stages of mechanical processing.

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Hybrid structures of integrated photonics for directional micro-light sources based on dielectric waveguides and photoluminescent organic microcrystals

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The development of the element base of modern photonics is a relevant and promising area of research. This paper proposes an approach to creating active structures that enable the implementation of directional light microsources by combining a waveguide with an input/output adapter fabricated using two-photon laser printing and an emitting microcrystal made of an organic dye. It is demonstrated that the proposed device enables both the excitation of the crystal's photoluminescence (PL) by radiation coupled through the waveguide structure and, conversely, the extraction of the crystal's PL radiation through the waveguide and adapter. These capabilities also apply to resonant Fabry-Perot modes, the excitation of which is demonstrated for organic dye crystals with a shape similar to a parallelepiped.

Keywords: photonics, two-photon laser printing, organic microcrystals.

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Introduction

The trend toward miniaturization of photonic elements and photonic integrated circuits stimulates the search for new approaches to their manufacture and testing. In this aspect, of great interest is the method of two-photon laser printing (TPL), or direct laser writing, which makes it possible to manufacture both planar and three-dimensional dielectric structures of arbitrary shape and, moreover, not requiring the use of lithographic masks [1, 2]. At the same time, the spatial resolution of TPL can reach down to a few hundred nanometers. As noted in the review [3], the advantages of TPL include the relative simplicity, the operation under ambient conditions (without vacuum requirements), single-cycle manufacturing, and, most importantly, printing arbitrary three-dimensional objects. The latter feature makes it possible, in particular, (a) to print three-dimensional input-

output couplers that allow optical radiation routing normal to a planar photonic structure; and also (b) to produce "suspended" structures located above the substrate [4, 5]. Finally, at the stage of preparing the polymer composition for printing, it is possible to introduce various functional additives into it – such as dye solutions, photoluminescent compounds, nanoparticle suspensions [6, 7], etc., as a result forming qualitatively new properties of materials. Thus, in the work [8], a single-photon light source based on a Fabry-Perot resonator with nanodiamonds was implemented; the structure was manufactured by the TPL method.

Despite the fact that the TPL technology needs further improvement and development, new methods can already be proposed for implementing active photonic devices, one of which is investigated in this work. This method is based on combining a photoluminescence (PL) source – a microcrystal of an organic dye – and a

system for launching and extracting radiation in the form of an adapter with a waveguide, manufactured by the TPL method in a single planar structure. The presented results demonstrate that the positioning a PL microcrystal on a waveguide structure makes it possible to realize a localized efficient source of broadband PL as part of a planar photonic circuit and, conversely, to extract PL radiation from the microcrystal directly into the waveguide while preserving the resonant part of the spectrum. The proposed approach is promising for testing microresonators without the use of precision devices for input and output of radiation required to ensure optical coupling of photonic elements.

Experimental section

For the study, a series of short waveguides with an adapter for vertical input/output of radiation were manufactured; for this, the TPL method described in detail in [9] was used. Printing was carried out using the radiation from a femtosecond laser with a central wavelength of 780 nm and a pulse duration of 380 fs; the voxel diameter during printing was approximately $0.4\ \mu\text{m}$ with a height of less than $0.8\ \mu\text{m}$. The material for TPL was the hybrid negative

photoresist OrmoComp, and crystalline magnesium fluoride plates were used as the substrates.

An image of one of the manufactured structures, obtained with an optical microscope is shown in Fig. 1; the inset shows the three-dimensional model of the structure implemented during printing. The radiation input/output adapter consisted of a lens with a spherical surface with a radius of $9\ \mu\text{m}$, ensuring focusing of radiation in the direction along the normal to the substrate surface into a waveguide with a length of about $42\ \mu\text{m}$, the cross-section of which was $4\times 4\ \mu\text{m}^2$. The flat base of the lens, inclined at an angle of 48° to the plane of the substrate, directed the incident light into the end face of the waveguide due to the effect of total internal reflection. The value of this angle was chosen to optimize the conditions for launching a converging laser beam into the waveguide. Thanks to the presence of a contrast in refractive indices between the substrate (1.38) and the polymer (1.51), such a waveguide supports a multimode regime of the light propagation in the visible spectral range. To give the adapter greater strength, an annular base was used, increasing the area of contact between the structure and the substrate.

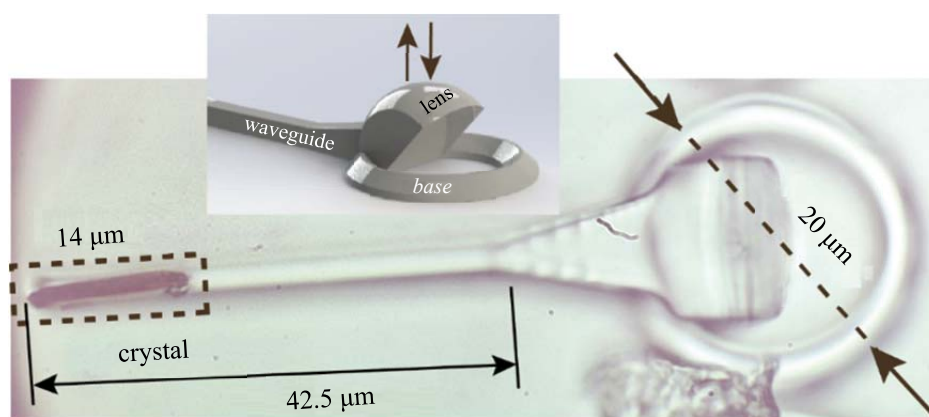


Fig. 1. Image in an optical microscope of the manufactured waveguide structure with a radiation input-output adapter. Inset: the model used during printing by the TPL method; arrows indicate the direction of input/output of radiation

Microcrystals of organic dyes DCM or HDMAC were synthesized on separate glass substrates by the method of sublimation from the gas phase, as described in detail in [10, 11]. The dyes have a band of bright PL in the visible range of the spectrum. Then the selected

microcrystal was mechanically separated from the substrate using a chemically sharpened optical fiber [12] and moved in front of the end face of the waveguide or onto its surface. Fig. 1 shows an image of one of the studied structures, in which a microcrystal of the HDMAC dye in the form of a

strip with dimensions of $1.2 \times 7 \times 14.2 \text{ } \mu\text{m}^3$ is located at the end of the manufactured polymer waveguide; its long side is almost parallel to the waveguide, and its plane is nearly perpendicular to the substrate.

The experiments were carried out using the method of one-photon-excited PL on a confocal microscopy setup (see the inset to Fig. 2); the radiation from a continuous semiconductor laser with an emission wavelength of 405 nm, focused by a Plan Apo 50 $\times$ /0.55NA objective into a spot with a diameter of about 0.9 μm , was used as optical pumping. The PL radiation was collected by a similar objective, passed through a system of filters cutting off the pump radiation, and then the PL signal from the microcrystal was directed into a spectrometer with subnanometer spectral resolution. The experimental setup made it possible to measure PL spectra at different points of the structure, spatially separating the regions of excitation and collection of the PL signal.

First, the PL spectrum of the microcrystal was measured with the regions of irradiation and collection of the PL signal coinciding; the corresponding results are presented in Fig. 2. The black curve shows the measured spectral dependence of PL, modulated by a set of resonant features; the PL spectrum of the microcrystal after extraction of the nonresonant PL signal is shown by the violet line in Fig. 2. The inset to the figure shows a part of the spectrum on an enlarged scale, the wavelengths corresponding to the most intense PL intensity maxima, I , are given. Let us note that in this experimental scheme, the excitation of microresonator modes corresponds to PL maxima arising due to light scattering on inhomogeneities in the near-surface region of the structure. This applies, among other things, to Fabry–Perot modes in the case of their excitation in the microcrystal under consideration; the growth of PL intensity in this case is a consequence of an increase in the density of states of the electromagnetic field under resonant conditions [13].

For the spectrum shown in the inset to Fig. 2, the average intermode distance for the most pronounced maxima is about 9 nm, which corresponds to Fabry–Perot modes excited along the long side of the microcrystal ($L = 14.2 \text{ } \mu\text{m}$) with a refractive index $n = 1.7$. In this case, estimates of the intermode distance for a

wavelength of $\lambda = 670 \text{ nm}$ using the expression $\Delta\lambda \approx \lambda^2/(2nL)$ give a value of about 9.3 nm, which is close to that obtained in the experiment. Considering the spectral width of the PL maxima $\delta\lambda \approx 5 \text{ nm}$, the mode quality factor $Q = \lambda/\delta\lambda$ is about 130. It should be noted that the complex structure of the PL spectrum reflects the presence of Fabry–Perot modes of different orders and different polarization states, as well as other families of resonator modes that can be excited along all three directions in the microcrystal having the shape of a parallelepiped.

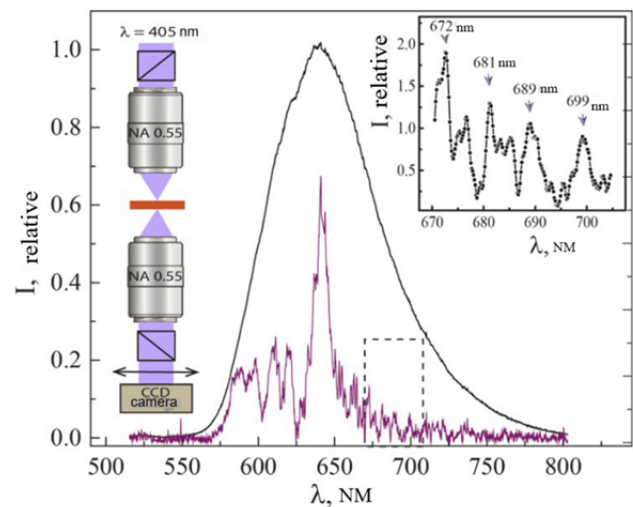


Fig. 2. Spectrum of PL intensity of the HDMAC microcrystal placed on top of the waveguide, measured with coincidence of the regions of PL excitation and its registration (black curve), and the spectrum of microresonator modes isolated from it (violet curve). Inset on the left – schematic diagram of the confocal optical microscopy setup; on the right – the spectrum of the isolated resonant modes (the corresponding region is outlined by a rectangle) on an enlarged scale

Fig. 3 presents the results demonstrating the ability of the structure under consideration to extract PL radiation from a microcrystal located at the end face of the waveguide. In the experiment, a laser beam focused on the microcrystal excites PL in it, and the spectrum of the signal at the output of the adapter is recorded; in this case, the sample was positioned with its substrate toward the pump beam, and the output of the adapter was directed toward the collecting objective. The shape of the PL intensity spectrum obtained in this way is analogous to the initial PL spectrum of the microcrystal; at the same time, the parameters of the isolated resonant modes differ from those presented in Fig. 2, and the

average intermode distance turned out to be significantly larger and amounted to about 18 nm for the most clearly manifested modes (see the inset to Fig. 3). An estimate of the intermode distance for the vertically oriented side of the microcrystal with $L \approx 7 \mu\text{m}$ for $\lambda = 625 \text{ nm}$ gives the value of $\approx 16.4 \text{ nm}$, which is close to the experimental one, while the quality factor $Q \approx 60$. Thus, we assume that in this case scattering of a different type of Fabry–Perot modes is observed compared with those presented in Fig. 2 – namely the modes, oscillating along the side of the microcrystal about $7 \mu\text{m}$ long, located vertically relative to the substrate.

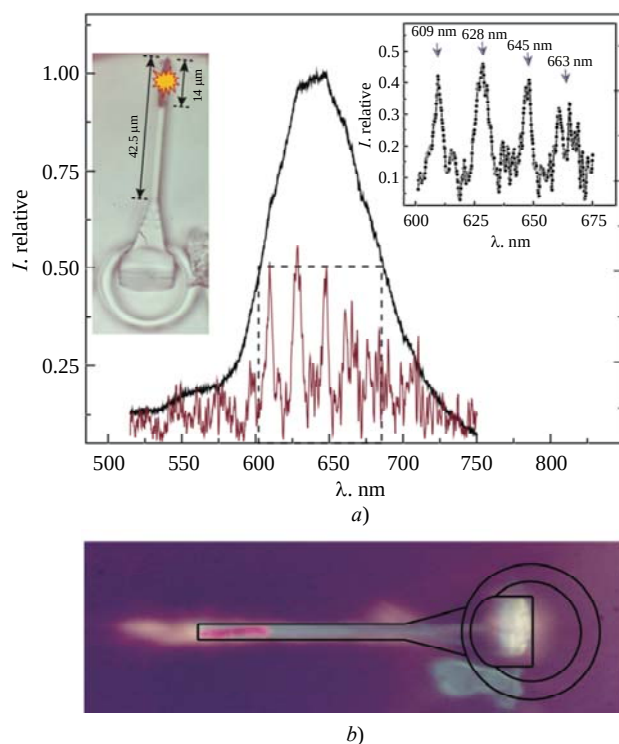


Fig. 3. (a) Radiation spectrum (black curve) at the output of the radiation input-output adapter upon excitation of PL in the microcrystal and the Fabry–Perot mode spectrum isolated from it. Inset on the right – on an enlarged scale, the spectrum of the isolated resonant modes (the corresponding region is outlined by a rectangle). (b) Optical image of the structure upon launching radiation with $\lambda = 405 \text{ nm}$ into the adapter (on the right in the figure)

Fig. 3b shows an optical image of the structure upon pumping the microcrystal with laser radiation with $\lambda = 405 \text{ nm}$, focused into the adapter located on the right in the figure (a pump-cutting light filter is used). Based on the PL signal of the substrate (orange color), a relatively small scattering of the pump light is visible in the regions of the adapter and the junction of the

cone-shaped part of the radiation launching system with the waveguide, which is associated with the non-uniformity of the structure in these regions. The intensity of the scattered radiation is the highest at the output from the waveguide through the adapter. According to the performed calculations [14], the transmittance of the structure is at least 50%, whereas the experimental value of the transmittance for a similar structure with two radiation input/output adapters was about 14%.

The brightest luminescence in the studied structure corresponds to the PL of the dye microcrystal, which indicates its efficient optical excitation by radiation propagating through the waveguide. For such an experimental geometry, the PL spectrum of the microcrystal, recorded directly from the region of its location, is analogous to that presented in Fig. 2. It should be noted that at a pump radiation power of up to $0.5 \mu\text{W}$, no photodegradation of the microcrystal was observed for at least tens of minutes, which corresponds to the results of previous studies [11].

Conclusion

Thus, in the present work, an approach to creating active photonic structures is proposed that combines the potential of the two-photon lithography method for creating polymer waveguides and devices for input and output of optical radiation, with microresonators exhibiting a high PL yield. The possibility of efficient optical excitation of PL of a dye microcrystal (for example, HDMAC) located on the surface of the waveguide has been shown, depending on the method of recording the PL response, different families of resonant modes are observed. Namely, in the PL signal scattered on the microcrystal, Fabry–Perot modes propagating without contact with the waveguide (parallel to the substrate) are predominantly detected, whereas through the waveguide the signal from the Fabry–Perot modes maximally coupled with the polymer structure effectively passes through the waveguide and is recorded.

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Determination and comparison of hydrogen content in industrial graphite materials

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The study of hydrogen content in graphite materials is relevant due to its potential impact on the physical and mechanical properties of these structural materials operating at extremely high temperatures. The objective of this study was to directly measure hydrogen content in carbon materials (samples up to 1.0 g) and develop a simple method for its determination in industrial graphite materials. The hydrogen content in graphite, pyrolytic graphite, and carbon-carbon composite material (CCCM) was determined using a setup including a vacuum chamber, laser, and gas dehydrator (silica gel). It was found that the materials contain 3–5 at.% (0.3–0.45 wt.%) hydrogen.

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Introduction

Graphite and carbon-carbon composite materials are widely used as part of refractory structures [1, 2], structures and parts resistant to the action of high-temperature gas [3, 4] and plasma flows [5, 6, 7].

The problem of studying the hydrogen content in graphite materials is relevant due to the possible influence of hydrogen on the physicomaterial properties of such multifunctional structural materials, which generally operate under conditions of extremely high temperatures.

In recent years, the greatest number of works devoted to the study of the influence of hydrogen on the properties of graphite have had a purely practical orientation associated with the use of graphite for protecting the first wall of thermonuclear reactors [5–8]. In most studies, it is usually assumed that the process of interaction of hydrogen atoms with graphite is purely surface-related and limited to the temperature region below 1500 K, while the possible

hydrogen content in the bulk of graphite materials is most often neglected [5].

The objective of this work is to develop a simple methodology for determining the hydrogen content in various industrial types of graphite materials, averaged over the sample volume and to compare the obtained results.

At the initial stage of the work, an elemental composition analyzer CHNS "Eurovector" EA-3000 was used to determine the hydrogen content, making it possible to determine the content of nitrogen, carbon, hydrogen, nitrogen, and sulfur contents in materials.

The principle of operation of the analyzer is based on the combustion of a sub-milligram or few-milligram sample of the analyzed substance in pure oxygen.

Three series of measurements were performed. Their results are presented in Table 1. The probable error of the results is about 0.1–0.2% according to the data of the developer of the elemental composition analyzer CHNS "Eurovector" EA-3000.

Table 1

Hydrogen content (wt.%) in samples of different graphite materials according to three series of measurements

Material	1st series		2nd series		3rd series		Average
Graphite MPG-7	0.51	0.58	0.39	0.32	0.39	0.33	0.42
Pyrolytic graphite	0.37	0.41	0.41	0.35	0.36	0.45	0.39
UUKM	0.46	0.36	0.57	0.51	0.42	0.45	0.46

As can be seen, the measurements confirmed the presence of hydrogen in graphite materials and gave its average content of about 0.42 wt.% with a scatter of values of about 0.1%. At the same time, no significant change in the hydrogen content in different materials was revealed. The disadvantage of the performed measurements is the small mass of the studied materials (several mg), which under conditions of their significant spatial non-uniformity (especially in UUKM), can cause an increased scatter of results.

The obtained data are qualitatively consistent with the results of analogous measurements performed at NIIgrafit [9], where for a material of the UUKM type after impregnation and carbonization a value of 1.1 wt.% was obtained, and after high-temperature treatment (HTT) at 2500 K – 0.3 wt.%.

The aim of the present work is the direct measurement of the hydrogen content in carbon materials with sample masses up to 1.0 gram.

Methodology of conducting the experiments

The determination of the hydrogen content in graphite materials consists in burning the sample in an oxygen atmosphere with the subsequent determination of the amount of water vapor in the combustion products.

Fig. 1 presents a diagram of the experimental setup for measuring the hydrogen content in carbon materials.

To absorb water vapor (to dry the gas mixture formed after combustion of the sample in oxygen), silica gel according to GOST 3956-76 in the form of granules with a diameter of 2–4 mm, previously subjected to heat treatment at a temperature of 150–200 °C, was used. Silica gel is capable of drying various gases (air, oxygen, carbon dioxide, nitrogen, hydrogen, argon, etc.) at atmospheric pressure. It was preliminarily

verified that 1 g of silica gel is capable of absorbing up to 50–60 mg of vapor to saturation, that the time of its saturation in open air with a humidity of 40% is 4–6 hours, and also that all the additional water specially introduced into the working chamber (5–15 mg) upon reproduction of the working cycle gave a corresponding increase in the mass of the silica gel.

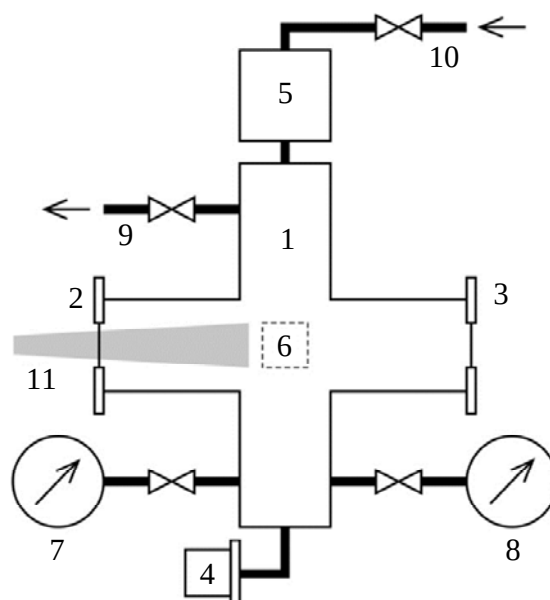


Fig. 1. Experimental setup diagram: 1 – vacuum chamber; 2, 3 – optical windows; 4 – tube with a flange for silica gel; 5 – tube with a flange for a weighed portion of water; 6 – sample; 7 – valve and manometer; 8 – valve and vacuum gauge; 9 – valve for connection to the pump; 10 – valve for oxygen supply; 11 – laser radiation

The sequence of conducting the experimental studies was as follows.

1. A sample of graphite material with dimensions of 40×6×2 mm and a weight from 0.3 to 1.0 g was installed in the vacuum chamber.

2. The stainless steel vacuum chamber with a volume of about 250 cm³, after evacuation, was filled with oxygen to a pressure of 0.3–0.7 MPa.

3. Silica gel with a mass of 1–2 g in the form of granules was placed in a bag made of metal mesh, which was located in a tube (with a

diameter of 30 mm and a length of about 50 mm) docked to the vacuum chamber.

4. The process of burning the graphite material was ensured by the action of laser radiation with a power of 400 W for 10 seconds.

5. After combustion of part of the sample (until the oxygen was burned out), the chamber was dried for the required time (usually 12–48 hours), after which the pressure in the chamber was reduced to atmospheric. For each pressure value, the required drying time must be determined experimentally by ensuring complete absorption of the additionally introduced known amount of water.

6. The chamber is opened, and the bag with silica gel is quickly removed.

7. The silica gel is weighed every minute for 15–30 minutes in order to determine as accurately as possible the initial mass of the silica gel at the moment of its removal by extrapolating the obtained growing dependence (due to absorption of vapor from the air) of mass on the

time spent in air to the time of removal of the bag.

8. The remainder of the partially burned sample of material is removed from the chamber and weighed.

As direct control measurements have shown, a correction must be introduced into the obtained results for the absorption of moisture from the air by the silica gel during the time between its first weighing and installation in the sealed volume of the chamber, and then between removal from the sealed volume and weighing. This time is about one minute, during which the silica gel gains about 1 mg of moisture from the ambient air of the room (with a relative humidity of about 40%).

Characteristic quantities: the burned mass of carbon is 0.35–0.45 g, and the increase in the mass of silica gel is 8–13 mg.

The general view of the setup for measuring hydrogen in graphite materials is presented in Fig. 2.



a)



b)



c)

Fig. 2. Setup for measuring hydrogen in graphite materials: a) – initial state; b) – moment of laser ignition; c) – combustion of the sample

Discussion of the results

The obtained results are presented in Table 2.

It can be seen that the hydrogen content in different samples obtained by cutting from different places of one block of material is substantially different.

Table 2

Results of measurements of hydrogen content in wt. %

Material	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Mean
MPG-7	0.32	0.36	0.26	0.31	–	0.31 ± 0.03
UUKM	0.36	0.45	0.41	0.38	0.38	0.4 ± 0.03

These results are consistent with those obtained earlier and for the first time indicate a possibly higher hydrogen content in UUKM, which undergoes multiple impregnation with pitch with subsequent carbonizing firings and, what is especially important, multiple high-temperature graphitizing firings, which apparently contribute not to the removal of hydrogen from the material, but, on the contrary, to its saturation with it.

The mass-spectrometric studies of the composition of graphite materials performed by the authors of [10], in our opinion, also indicate a significant (about 0.5 wt.%) hydrogen content in graphite and its presence in vapor phase in a bound state (as part of hydrocarbons).

It is significant that not once was a hydrogen content greater than 0.51 wt.% recorded.

Conclusion

Thus, according to the data of the work [9], the measurements on the "Eurovector" EC-3000, mass spectroscopy data [10], and the results obtained in the present work, industrial graphite materials always contain about 3–5 at % (0.3–0.45 wt.%) hydrogen. This is obviously due to the technology of their production (either upon saturation with pitch, or in a hydrogen atmosphere during gas-phase deposition).

The mastering of operational monitoring of the hydrogen content in industrial graphite materials opens up the possibility of a comprehensive study of the influence of this element on the physicomachanical characteristics of materials, as well as on their behavior at high temperatures.

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Degradation of the optical properties of ZnO micropowder modified with SiO₂ nanoparticles under sequential irradiation with electrons, protons, and solar spectrum quanta

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The degradation of the integrated emissivity coefficient $\Delta\alpha_s$ of $m\text{ZnO}$ micropowder modified with $n\text{SiO}_2$ nanoparticles ($m\text{ZnO}/n\text{SiO}_2$) was studied under sequential irradiation with 30 keV electrons (e^-), 5 keV protons (p^+), and solar spectrum quanta (SSP). The additivity coefficient (Cadd) was calculated to quantify the mutual influence of different types of radiation. It was found that, depending on the type of radiation and the irradiation sequence, Cadd can be less than, equal to, or greater than unity.

Keywords: zinc oxide, silicon dioxide, modification, irradiation, electrons, protons, solar spectrum quanta, additivity coefficient.

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Introduction

Thermal control coatings (TCC) based on zinc oxide pigments represent one of the most common classes of materials for spacecraft thermal control systems. Their widespread application is attributed to the combination of a low integrated solar absorptance ($\alpha_s = 0.15\text{--}0.25$) and a high infrared emittance $\varepsilon > 0.85$, which provides efficient radiative heat exchange with the environment [1, 2]. The stability of these characteristics throughout the spacecraft's active lifetime is a key requirement in the design of thermal control systems [3].

In actual space flight conditions, TCCs are exposed to a combination of space environmental factors. The main destabilizing agents in the geostationary orbit are fluxes of electrons and protons from the Earth's outer radiation belt with energies ranging from several to hundreds of keV, as well as solar ultraviolet radiation [4, 5]. In low Earth orbit, the impact of atomic oxygen is added

to the above factors. All these factors induce an increase in α_s , which causes heating of the spacecraft surface and disruption of the thermal balance. Predicting such degradation is a mandatory stage in spacecraft design [3].

Modification of $m\text{ZnO}$ micropowder with $n\text{SiO}_2$ nanoparticles is an effective method for increasing the radiation resistance of TCCs [6, 7]. It has been established that the deposition of $n\text{SiO}_2$ onto the surface of $m\text{ZnO}$ particles reduces the density of intrinsic defects in zinc oxide, which are centers of radiation-induced absorption, and forms a protective layer that partially shields corpuscular radiation [8]. As a result, TCCs based on $m\text{ZnO}/n\text{SiO}_2$ exhibit a significantly smaller increase in α_s under electron and proton irradiation compared to the unmodified zinc oxide powder [9, 10].

However, ground-based simulation of flight conditions is traditionally performed using separate exposure to individual factors, and the resulting degradation is estimated as the sum of

separate contributions. This approach fails to consider the possible mutual influence of factors on each other. For a quantitative assessment of the deviation of the combined effect from simple additivity, the additivity coefficient (K_{add}) is used.

The aim of this paper is to investigate K_{add} for modified $m\text{ZnO}/n\text{SiO}_2$ powders under sequential exposure to electrons, protons, and solar ultraviolet radiation, and to establish the physical mechanisms of the observed effects.

Study Object and Experimental Procedure

The object of study was a zinc oxide micropowder. Silicon dioxide nanopowder was used as the modifying additive. The $m\text{ZnO}$ micropowder was mixed with the $n\text{SiO}_2$ nanoparticles in a 100:3 ratio.

For the modification process, the $n\text{SiO}_2$ was dissolved in distilled water under ultrasonic agitation, then $m\text{ZnO}$ was added and the mixture was stirred with a magnetic stirrer. The mixture was dried, ground, and heated in air at the optimal temperature [11].

The integrated solar absorptance (a_s) was calculated from the diffuse reflectance spectra according to equation (1) [12–14]:

$$a_s = 1 - R_s = \frac{\int_{\lambda_1}^{\lambda_2} \rho_{\lambda} \cdot I_{\lambda} \cdot d_{\lambda}}{\int_{\lambda_1}^{\lambda_2} I_{\lambda} \cdot d_{\lambda}}, \quad (1)$$

where R_s is the integrated reflectance of solar radiation, I_{λ} is the solar spectrum, and $(\lambda_1 - \lambda_2)$ is the wavelength range of the solar spectrum used for integration, equal to 0.2–2.5 μm .

The change in the absorption coefficient a_s was calculated from its values before and after irradiation. For each irradiation condition, the additivity coefficient K_{add} was calculated by equation (2) [15, 16]:

$$K_{\text{add}} = \frac{\sum(\Delta a_{s1} + \Delta a_{s2})}{\Delta a_{s(\text{COBM})}}, \quad (2)$$

where $\sum(\Delta a_{s1} + \Delta a_{s2})$ is the sum of changes under separate irradiation; $\Delta a_{s(\text{COBM})}$ is the change in value under simultaneous irradiation.

Experimental Results and Their Discussion

Table 1 contains the experimental values of K_{add} for the $m\text{ZnO}/n\text{SiO}_2$ powder under sequential irradiation by protons and solar spectrum quanta: $\text{p}^+ \rightarrow \text{solar spectrum quanta}$.

Table 1

Values of K_{add} for the $m\text{ZnO}/n\text{SiO}_2$ powder after irradiation by protons and solar spectrum quanta

Sequential irradiation $\text{p}^+ \rightarrow \text{solar spectrum quanta}$ (Φp^+ , cm^{-2} ; t_{SSQ} , h)					
$\Phi\text{p}^+ \rightarrow t_{\text{SSQ}}$	0.63×10^{16}	1.08×10^{16}	2.09×10^{16}	3×10^{16}	4.07×10^{16}
Δa_s	0.039	0.060	0.084	0.107	0.124
$\Sigma \Delta a_{s(\text{sep.})}$	0.028	0.043	0.068	0.093	0.113
K_{add}	0.71	0.72	0.82	0.87	0.91
$\Phi\text{p}^+ \rightarrow t_{\text{SSQ}}$	$4.07 \times 10^{16} \rightarrow 2$	$4.07 \times 10^{16} \rightarrow 4$	$4.07 \times 10^{16} \rightarrow 6$	$4.07 \times 10^{16} \rightarrow 8$	-
Δa_s	0.138	0.145	0.152	0.158	-
$\Sigma \Delta a_{s(\text{sep.})}$	0.129	0.133	0.138	0.145	
K_{add}	1.05	1.06	1.07	1.10	

1. Sequential irradiation $\text{p}^+ \rightarrow \text{solar spectrum quanta}$ was carried out in the following mode:

– First, separate proton irradiation was performed at fluences from $0.63 \times 10^{16} \text{ cm}^{-2}$ to $4.07 \times 10^{16} \text{ cm}^{-2}$, and the values of Δa_s were recorded;

– then, after the proton irradiation, the samples were sequentially exposed to solar spectrum quanta, and Δa_s values were recorded after each irradiation period;

– then, separate irradiation by solar spectrum quanta was performed for 2, 4, 6, and 8 hours;

– next, the sum of changes in the absorption coefficient under separate irradiation by protons and solar spectrum quanta was calculated as $\Sigma\Delta a_{s(\text{sep.})}$;

2. finally, K_{add} was calculated from values $\Sigma\Delta a_{s(\text{sep.})}$ and values Δa_s obtained under sequential irradiation $\Delta a_{s(\text{seq.})}$, which were close to unity.

Sequential irradiation solar spectrum quanta $\rightarrow p^+$ was carried out in the following mode:

– first, separate irradiation by solar spectrum quanta was performed for 6 hours, yielding $\Delta a_s = 0.069$;

– then, sequential irradiation by protons at fluences from $0.52 \times 10^{16} \text{ cm}^{-2}$ to $3.93 \times 10^{16} \text{ cm}^{-2}$ was performed, yielding Δa_s from 0.094 to 0.164;

– the sum of changes of Δa_s after separate irradiation by solar spectrum quanta and protons in the same conditions is significantly lower than the values obtained under sequential irradiation: with proton fluence increasing to $3.93 \times 10^{16} \text{ cm}^{-2}$ $\Sigma\Delta a_{s(\text{sep.})}$ increases from 0.049 to 0.135, but remains significantly lower than $\Sigma\Delta a_{s(\text{seq.})}$.

As a result, K_{add} has values of 0.8 over the entire studied range from $\Phi_1 = 0.52 \times 10^{16} \text{ cm}^{-2}$ to $\Phi_2 = 3.93 \times 10^{16} \text{ cm}^{-2}$; its values are less than unity, which indicates the presence of synergistic effects.

3. Sequential irradiation $e^- \rightarrow$ solar spectrum quanta was carried out in the following mode:

– first, irradiation by electrons at a fluence of $7 \times 10^{16} \text{ cm}^{-2}$ was performed, yielding $\Delta a_s = 0.04$;

– then, irradiation by solar spectrum quanta was performed sequentially after electrons for 2 and 4 hours, yielding Δa_s values equaling to 0.086 and 0.103, respectively;

– then, separate irradiation by solar spectrum quanta was performed for 2 and 4 hours, yielding Δa_s values equaling to 0.044 and 0.057, respectively.

The sum of changes of Δa_s under separate irradiation by electrons and solar spectrum quanta was 0.084 and 0.097, while under sequential irradiation $e^- \rightarrow$ solar spectrum quanta it was 0.086 and 0.103, respectively, and K_{add} was **0.977 and 0.941**. Its values are close to unity; no synergistic effects are observed, and in ground testing of this pigment, sequential irradiation can be replaced by separate irradiation by electrons and solar spectrum quanta.

Sequential irradiation solar spectrum quanta $\rightarrow e^-$ was carried out in the following mode:

– first, irradiation by solar spectrum quanta was performed for 6 hours, yielding $\Delta a_s = 0.067$;

– then, sequential irradiation by electrons at fluences of 1×10^{16} and $2 \times 10^{16} \text{ cm}^{-2}$ was performed, yielding $\Delta a_s = 0.059$ и 0.064, respectively;

– finally, separate irradiation by electrons at the same fluences of 1×10^{16} and $2 \times 10^{16} \text{ cm}^{-2}$ was performed, yielding $\Delta a_s = 0.017$ и 0.024, respectively.

The sum of changes of Δa_s under the separate irradiation by solar spectrum quanta and electrons was 0.084 and 0.091, and K_{add} was 1.43 and 1.42. These values are significantly higher than unity, indicating the presence of synergistic effects under this sequential irradiation mode. Solar spectrum quanta $\rightarrow e^-$, and in ground-based testing of this pigment, sequential irradiation cannot be substituted by separate irradiation.

Conclusion

The degradation of the integrated solar absorbance Δa_s of the mZnO micropowder modified with nSiO₂ nanoparticles (mZnO/nSiO₂) was studied under sequential irradiation by electrons (e^-) at 30 keV, protons (p^+) at 5 keV, and solar spectrum quanta (SSQ). The additivity coefficient (K_{add}) was calculated to quantitatively assess the mutual influence of these types of radiation on the formation and accumulation of defects and absorption centers.

It was established that for the sequential and separate irradiation of the mZnO micropowder modified with nSiO₂ nanoparticles, the additivity coefficient in two of the four sequential irradiation modes ($p^+ \rightarrow$ SSQ and $e^- \rightarrow$

SSQ) is close to unity; in the $\text{SSQ} \rightarrow \text{p}^+$ mode it equals 0.8, whereas in the $\text{SSQ} \rightarrow \text{e}^-$ mode it reaches 1.42–1.43.

To clarify the mechanisms of the processes occurring in the powders during irradiation, further studies are required, which are planned to be carried out by decomposing the induced absorption spectra under these types and modes of irradiation and analyzing the parameters of the elementary absorption bands. Such studies can be performed using the “Spectrum” setup, a unique apparatus in the Russian Federation, that allows for the in situ registration of diffuse reflectance spectra and induced absorption

spectra after each irradiation period directly within the irradiation chamber.

The practical significance of this study lies in the fact that the further advancement of space exploration relies on the development of technologies for producing spacecraft materials that ensure high reliability over long-term orbital missions (15–20 years).

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Deposition rate and substrate temperature during deposition of silicon-carbon coatings by electron beam evaporation of silicon carbide

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Silicon-carbon coatings are prepared by electron beam evaporation of silicon carbide. The heating of the substrates occurred due to the thermal radiation of the target. The deposition rate and the substrate temperature are inversely proportional to the square of the distance to the target. It is shown that the deposition rate is uniquely related to the substrate temperature.

Keywords: Electron beam evaporation of silicon carbide, silicon-carbon coatings, deposition rate, thermal radiation, substrate temperature.

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Introduction

The properties of silicon-carbon films deposited by electron-beam evaporation allow for their use as protective and hardening coatings [1]. One of the main advantages of electron-beam evaporation is the higher deposition rate compared to the plasma-enhanced chemical method [2] and magnetron sputtering [3]. The problem of substrate heating by all types of radiation during magnetron sputtering was addressed in [4, 5]. At the same time, substrate heating is considered [6] insignificant during electron-beam evaporation, which allows depositing coatings on heat-sensitive substrates. However, as for silicon carbide evaporation, deposition rates of the order of 1 $\mu\text{m}/\text{min}$ and higher are achieved by heating the target to temperatures over 2500 K. This is accompanied by intense thermal radiation, which leads to noticeable heating of substrates. As for substrates

made of low-melting materials, for example, aluminum, such heating can result in fatal consequences. The aim of this paper is to analyze the relationship between the coating deposition rate and the substrate heating temperature.

Experimental technique and methodology

To establish the relationship between the substrate temperature and the coating deposition rate, experiments were conducted using a setup equipped with a fore-vacuum electron source with a plasma cathode [7]. The working chamber accommodated the tooling, which included a graphite crucible with a target placed inside it. The target consisted of a pellet 12 mm in diameter and 3 mm thick, sintered from silicon carbide powder with a grit size of F 60 (Fig. 1). Five substrate holders, placed on steel supports at various distances from the target, shared the same platform with the crucible.

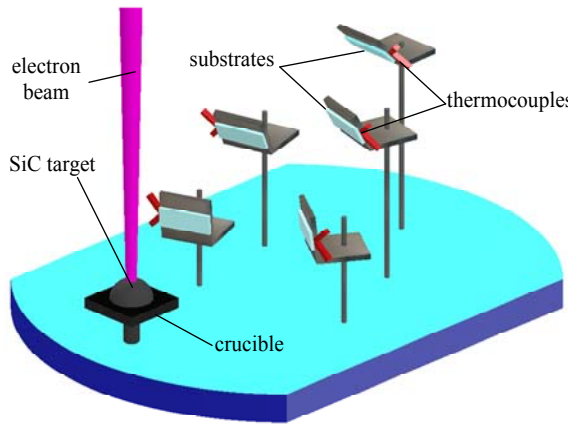


Fig. 1. Schematic of silicon-carbon coating deposition

Borosilicate glass plates sized $10 \times 20 \times 2$ mm were used as substrates. The solid angle within which the substrates were located did not exceed 0.2 steradians. A chromel-alumel thermocouple was attached to each substrate.

Deposition was carried out in a helium atmosphere continuously for one hour. The thickness of the deposited films was measured using an MII-4 microinterferometer.

Measurement results

The experimental dependencies shown in Fig. 2 demonstrate that, within the margin of error, the deposition rate ν and the substrate temperature T_s are inversely proportional of the square of distance r from the target to the substrate at a constant electron beam power. Moreover, as the electron beam power increases, the slope of the ν versus $(1/r^2)$ dependences increases, while the slope of the $T_s(1/r^2)$ straight lines changes less significantly. The dependence of the deposition rate on the substrate temperature (Fig. 3) is of particular interest. Despite the fact that the measurements were performed for substrates located at different distances from the target, the experimental points fit reasonably well onto a single curve. For a low-melting substrate material, such as aluminum, this dependence means that in the absence of forced substrate cooling, the deposition rate has a limit set by the substrate temperature.

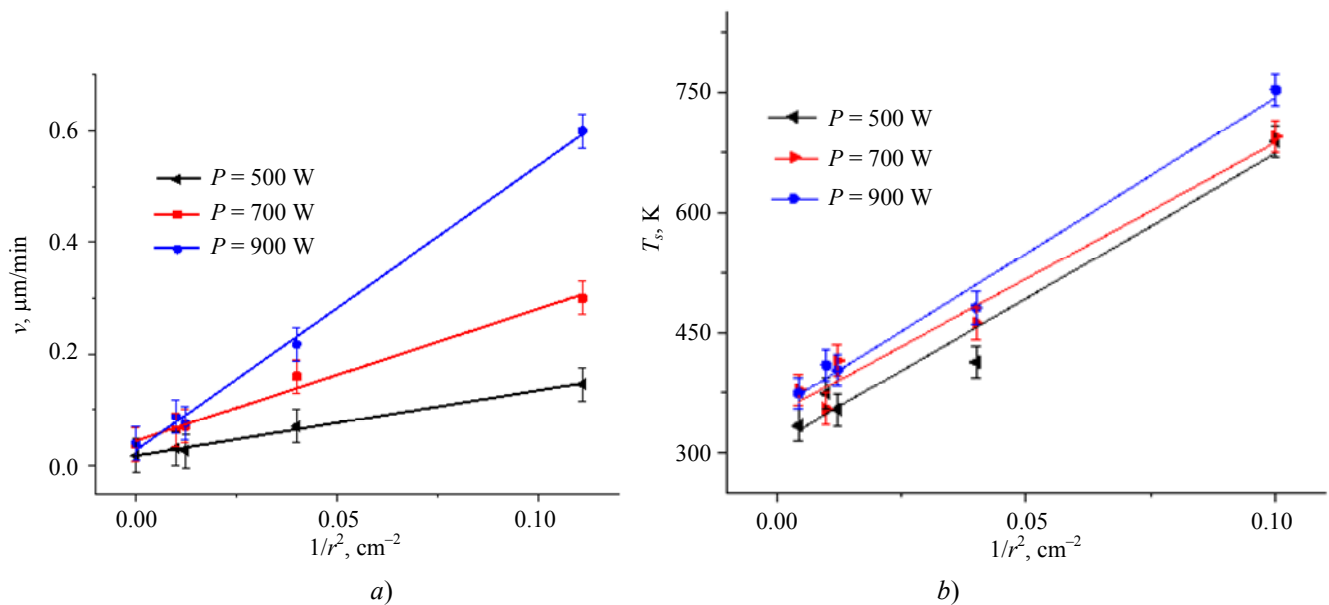


Fig. 2. Deposition rate ν (a) and substrate temperature T_s (b) as a function of the inverse square of distance $1/r^2$ for different electron beam powers

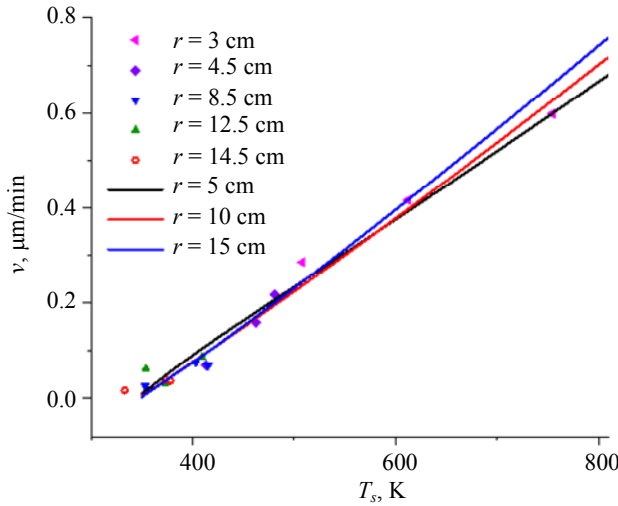


Fig. 3. Experimental (points) and calculated (lines) dependences of deposition rate v on substrate temperature T_s

Analysis of thermal load and deposition rate

To explain the obtained dependences, we adopt the following assumptions. Evaporated particles travel without collisions with gas molecules and are deposited on the surfaces they reach. The target is a black body. Silicon carbide evaporation follows the Clausius-Clapeyron law with the Antoine correction [8]. Let's define evaporation rate V_{ev} as the product of the saturated vapor density, average molecular velocity and evaporation area S_t :

$$\begin{aligned} V_{ev} &= S_t \sqrt{\frac{kT_t}{m}} \frac{mp_0}{kT_t} \cdot e^{-\frac{\Delta H}{R(T_t+C)}} = \\ &= S_t \sqrt{\frac{\mu}{RT_t}} p_0 \cdot \exp\left[-\frac{\Delta H}{R(T_t+C)}\right] \end{aligned} \quad (1)$$

where T_t is the target temperature; m is the mass of SiC molecule; μ and ΔH are the molar mass and heat of evaporation of silicon carbide; R is the gas constant; C is Antoine correction; p_0 and C are constants determined by comparison with experiment. Considering the assumptions listed above, we obtain $V_{dep} = V_{ev}/2\pi r^2$ for deposition rate V_{dep} . The target temperature is related to power P_t delivered to the target by electron beam $T_t = (P_t/S_t\sigma)^{1/4}$, where σ is the Stefan-Boltzmann constant. To establish the relationship between substrate temperature T_s , substrate area S_s , and target temperature T_t , it is necessary to use the setting mechanism of substrate thermal balance. If the energy loss by the substrate

occurs via thermal radiation, then from the energy balance equation:

$$P_t \frac{S_s}{4\pi r^2} = 2S_s\sigma T_s^4, \quad (2)$$

$$\text{it follows that } T_s = \left(\frac{P_t}{8\pi\sigma r^2}\right)^{1/4} \quad (3)$$

That is, substrate temperature T_s depends on distance as $1/r^{1/2}$. This does not correspond to the experimental dependence shown in Fig. 2. Another heat removal path is thermal conductivity. Under the condition that P_t is on the order of one kilowatt, heat flux P_s to the substrate estimated from expression $P_s = \frac{P_t}{4\pi r^2} S_s$ amounts to a few watts. At the pressure of several pascals, the thermal conductivity of gas cannot provide such heat removal. Consequently, the main heat flow path is the thermal conductivity through the substrate holder elements. Since precise calculations are hardly appropriate, the heat flux from the substrate can be accounted for by expression $\alpha(T_s - T_b)$ [4], where T_b is the temperature of the platform on which the substrate holder is mounted, and coefficient α is to be determined from comparison with experiment. Estimates can be made to confirm the possibility of heat removal from the substrate via thermal conductivity. Heat flow Q through a steel rod of diameter d and length l , on which the substrate holder is placed, can be written as:

$$Q = \frac{\lambda \pi d^2}{4l} (T_s - T_b), \quad (4)$$

where $\lambda = 45$ W/m·K is thermal conductivity of steel. For $d = 3$ mm, $l = 5$ cm, $T_s - T_b = 400$ K we have $Q = 2.5$ W, which is consistent with the radiant energy flux to the substrate. Considering the assumptions made, the energy balance equation on the substrate now takes the form:

$$S_t \sigma T_t^4 \frac{S_s}{2\pi r^2} = \alpha (T_s - T_b), \quad (5)$$

That is, the substrate temperature is inversely proportional to the square of the distance. Expressions (1) and (5) reflect the relationship between the coating deposition rate and substrate temperature with the target temperature, and hence with the electron beam power. By expressing T_t from (5) and substituting it into (1), and also considering the relationship between the rates, we obtain an expression convenient for analyzing the dependence of deposition rate V_{dep} on substrate temperature T_s :

$$V_{dep} = \frac{A}{r^{9/4} (T_s - T_b)^{1/8}} \times \exp \left\{ -\Delta H / R \left[B \sqrt{r} (T_s - T_b)^{1/4} + C \right] \right\}, \quad (6)$$

$$\text{where } A = \frac{S_t}{2\pi\sqrt{B}} p_0 \sqrt{\frac{\mu}{R}}, \quad B = \left(\frac{2\pi\alpha}{\sigma S_t S_s} \right)^{1/4}.$$

At first glance, the dependence of the deposition rate on distance that follows from expression (6) appears as $V_{dep} \sim r^{-9/4}$, which

differs from $V_{dep} \sim r^{-2}$ found experimentally. However, one should consider the presence of r in the exponent of (6), according to which V_{dep} weakly increases with increasing r . The calculated curves plotted in relative units (Fig. 3) match the experimental points. Moreover, the curves obtained for different distances slightly differ from each other, which also corresponds to the experimental results. The slight change in the slope of the lines in Fig. 2b cannot be satisfactorily explained within the framework of the proposed model. However it can be noted that in the same range of distance r variation, deposition rate V_{dep} changes by an order of magnitude, while substrate temperature T_s changes by only a factor of two.

Conclusion

The performed measurements and analysis have shown that substrate heating due to thermal radiation from the target can be significant during deposition of silicon-carbon coatings by electron-beam evaporation of silicon carbide. In the case of a low-melting substrate material and absence of forced cooling, this heating imposes limitations on the electron beam power and, consequently, on the coating deposition rate. Moreover, the deposition rate is unambiguously determined by the substrate temperature. This limitation should be expected during deposition of coatings from other materials with a high evaporation temperature.

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Determination of the optimal volume of synthetic data for training object detection neural networks (using YOLOv8 as an example)

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The problem of determining the minimum sufficient volume of synthetic data for training YOLO-format object detection neural networks is considered. An approach to generating labeled synthetic images using the developed Yolo Synthetic Data Generator software module is proposed. Three variants of the YOLOv8 model (nano, small, medium) were trained on datasets of various sizes (from 100 to 10,000 frames). Quality assessment was performed on five test videos based on the number of detected objects and frames with detections. It is shown that for the nano and small models, a dataset of ~1,000 frames is optimal, while for the medium model, ~500 frames are sufficient. A further increase in the dataset does not lead to a significant improvement in quality but increases computational costs. The obtained results can be used in the development of machine vision systems, including for ROI-based autofocusing.

Keywords: synthetic data, YOLO, object detection, neural network training, dataset size, Yolo Synthetic Data Generator, autofocus.

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Introduction

Modern computer vision systems have been using deep neural networks for object detection with increasing frequency. One of the key factors determining the accuracy and efficiency of such systems is the availability of an annotated training dataset. However, manual image annotation is labor-intensive and costly, especially for specialized tasks where objects are rare or difficult to access. An alternative approach is to use synthetic data generated by software tools with automatic annotation [1].

Previously, the author developed a software Yolo Synthetic Data Generator, which allows for the creation of annotated synthetic images using three-dimensional models of objects on static backgrounds, incorporated controlled randomization of position, rotation, exposure, and gamma parameters. The program is registered in

the Register of Computer Programs (certificate No. 2026667324). The previous paper [2] examined the use of neural network detection for extracting a region of interest (ROI) during the autofocusing of an optoelectronic system. The goal of this paper is to determine the optimal volume of synthetic data required to train a detection model for a given object (a sponge) on various backgrounds, aiming to ensure high accuracy with minimal computational costs.

1. Synthetic Data Generation Method

The *Yolo Synthetic Data Generator* program is implemented in C++14 using the Vulkan graphics API and the tinyGLTF, GLM, and ImGui libraries. It allows for loading 3D models in glTF/GLB formats, overlaying them onto background images (JPG) while preserving perspective and lighting based on Physically

Based Rendering (PBR), and automatically generating annotations in the YOLO format comprising normalized coordinates of bounding boxes.

Randomization of the following parameters is provided during dataset generation:

- object position within the visible area of the scene;
- rotation around three axes (range 360°);
- exposure (0.1–10.0) and gamma correction (0.1–4.0).

Seven datasets were prepared for the training purposes. They consisted of 75 different background images, for each of which 100, 500, 1000, 1500, 3000, 5000, or 10000 frames were generated, respectively. Thus, the total volume of each dataset amounted to 7500, 37500, 75000, 112500, 225000, 375000, and 750000 images. In all cases, the same 3D model of the object was used, specifically a sponge (sized 160×55 mm).

Generation was performed at a resolution of 1280×1280 pixels, subsequently followed by conversion to the format adopted for YOLOv8 training (640×640). Annotations were saved in text files containing normalized coordinates.

2. Experimental Procedure

Three YOLOv8 architecture variants were chosen for training: **nano** (the smallest, 3.2 million parameters), **small** (11.2 million), and **medium** (25.9 million). Each model was trained on each of the seven datasets with the same hyperparameters: 100 epochs, a batch size of 16, the SGD optimizer with a momentum of 0.937, and an initial learning rate of 0.01 with cosine decay. Training was performed on an NVIDIA GeForce RTX 4080 GPU (16 GB).

Quality evaluation was carried out for five test videos with a duration of approximately ~2883 frames each (about 14415 frames in total).

The videos contained the same sponge model placed under various lighting conditions and angles, which differed from the training backgrounds. The following metrics were calculated for each trained model on each video:

- total number of detected objects (total_detections);
- number of frames with at least one detection (det_frames);
- proportion of frames with detections relative to the total number of frames;
- processing time per video (time_sec).

A confidence threshold of 0.7 was used, and the IoU threshold for non-maximum suppression was set to 0.35. All measurements were performed on the same computing device.

3. Findings and Discussion

The table shows the average metrics for each model and each dataset size, averaged over the five videos. For clarity, data for video No. 4, which recorded the maximum detection values, are also presented.

Analysis of the table shows that the maximum number of detections for the **nano** and **small** models is achieved with a dataset size of **1000 frames**. With a further increase in data volume, a decrease in detections is observed, which may be attributed to overfitting on synthetic data or insufficient background variability. Regarding the **medium** model, the peak also occurs at 1000 frames; however, the values at 500 frames are close to the maximum, and at 3000 frames a secondary rise is observed (possibly due to random factors). Considering that the medium model requires more computational resources, a dataset of 500 frames can be considered optimal for it, as it provides good quality with lower training costs.

Table

Average values for five videos (with values for video No. 4 in brackets)

Dataset size (number of frames)	Model			Time, s
100	nano	442 (596)	442 (596)	48.6
	small	485 (653)	485 (653)	59.1
	medium	602 (771)	602 (771)	67.5
500	nano	476 (641)	476 (641)	48.3
	small	549 (763)	549 (763)	59.0
	medium	695 (866)	695 (866)	67.4

End of table

1000	nano	532 (760)	529 (759)	48.0
	small	561 (793)	560 (793)	58.9
	medium	618 (776)	617 (776)	67.1
1500	nano	511 (644)	506 (643)	47.9
	small	533 (658)	531 (658)	58.8
	medium	626 (744)	624 (744)	67.2
3000	nano	399 (480)	395 (480)	48.1
	small	469 (570)	467 (570)	59.0
	medium	620 (790)	617 (790)	67.2
5000	nano	306 (281)	304 (281)	48.0
	small	395 (460)	393 (460)	58.9
	medium	532 (621)	530 (621)	67.3
10000	nano	358 (441)	355 (441)	48.1
	small	448 (547)	446 (546)	59.2
	medium	550 (584)	548 (584)	66.9

The video processing time (inference) is practically independent of the dataset size and is determined only by the model architecture: nano – ~48 s, small – ~59 s, medium – ~67 s. This implies that a model should be chosen based not only on quality but also on performance requirements. For example, the nano model

provides acceptable quality (760 detections on video No. 4) with minimal time consumption, making it preferable for real-time systems, such as ROI-based autofocusing.

The figure shows the average number of detections vs. the dataset size for each model.

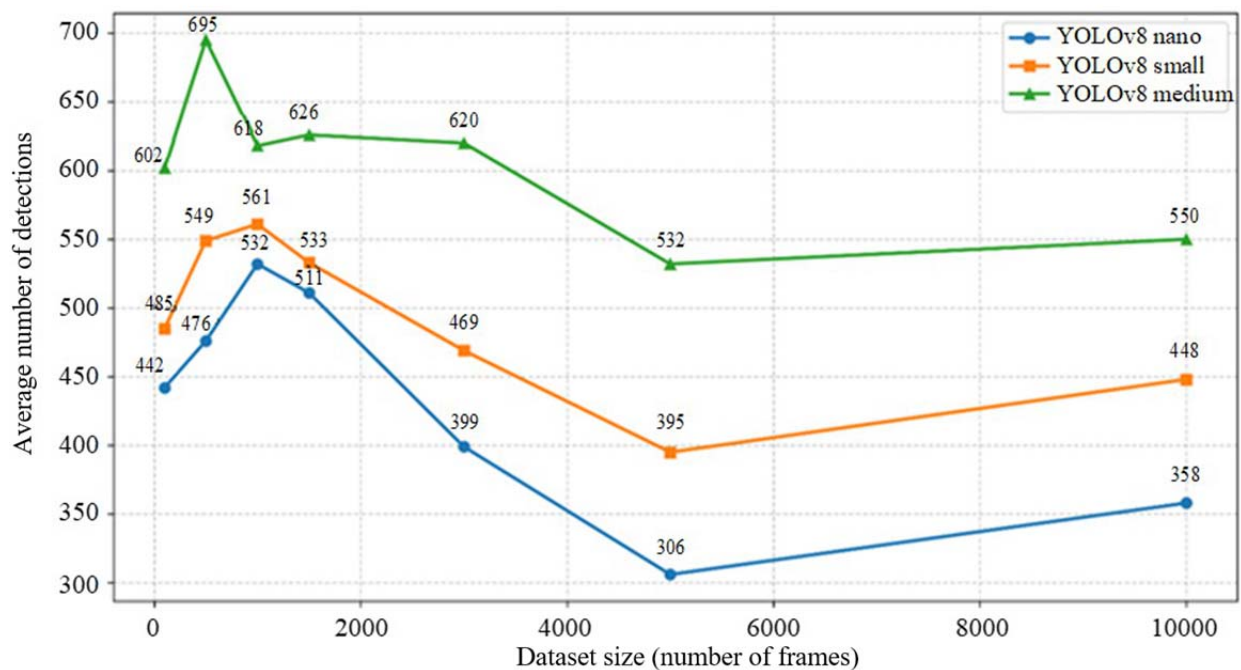


Fig. Average number of detections vs. the dataset size (averages on five videos)

The figure shows that the curves for all models have a similar pattern: rapid growth in the initial segment (up to 500–1000 frames) followed by saturation or even a decline. This

confirms the hypothesis that there is an optimal data volume, beyond which the model's generalization ability does not improve and may even deteriorate.

Discussion

The obtained results are consistent with prior studies on the use of synthetic data [3, 4], which also note that model quality ceases to grow after a certain threshold, and a further increase in the dataset may lead to overfitting on synthesis artifacts. In our case, the decline in quality at large datasets (3000–10000 frames) is likely due to the model starting to memorize specific features of backgrounds and angles that are not present in the test videos, thereby reducing its generalization ability.

It should be noted that the synthetic data were generated with limited diversity (75 backgrounds, one object). It is possible that increasing the number of backgrounds or object variations would increase the optimal data volume. Nevertheless, the obtained recommendations are justified for practical tasks similar to ours (specifically, detection of a single object on a limited set of backgrounds).

The application of the results of this paper in autofocusing systems [2] makes it possible to reduce manual annotation time and accelerate the development process. Using a synthetic dataset of 1000 frames and the YOLOv8 nano model allows for achieving high accuracy in object detection in real time, thus ensuring correct ROI extraction for focus adjustment.

Conclusion

This paper investigated the impact of synthetic data volume on the quality of training neural networks for object detection. Experiments with the YOLOv8 nano, small, and medium models using datasets of various sizes established that:

- the optimal dataset size for the nano and small models is ~ 1000 frames (with 75 backgrounds), which provides the maximum number of detections on test videos;
- the medium model shows close results already at 500 frames; however, its use is justified only when higher accuracy is required and sufficient computational resources are available;
- a further increase in data volume (above 1000–1500 frames) does not lead to quality improvement, and in some cases deteriorates it, which is a symptom of overfitting.

The practical significance of the paper lies in providing recommendations for selecting the synthetic dataset volume for single-object detection tasks on static backgrounds. In combination with the developed synthetic data generator, this allows for a significant reduction in the labor costs for the preparation of annotated samples and accelerates the development of machine vision systems, including ROI-based autofocusing systems.

Further research is aimed at expanding the variability of synthetic data (increasing the number of backgrounds, adding various objects, changing lighting conditions) and studying the influence of these factors on the optimal dataset volume.

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Determining of the longitudinal and transverse displacements of a coldfinger in a cryocooler manufactured

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The results of a study examining the impact of an integrated cryogenic system on cooled IR photodetectors are presented. It is shown that longitudinal displacements are below the measurement accuracy threshold, while transverse displacements depend on the rotor speed and range from 4.3 to 11.9 μm . This must be taken into account when developing algorithms for compensating for image shift during operation of the photodetector.

Keywords: gas cryogenic machine, cryocooler, Stirling's cycle, microcryogenic machine, cold tip temperature.

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Introduction

It is important during the operation of cooled array photodetectors to minimize the influence of longitudinal and transverse displacements of the cooling system on the image. However, no values directly regulating this parameter are established in the regulatory documents of the current standards of the Russian Federation and CIS countries [1].

Western standards also do not prescribe specific linear displacement values, but rather test the device under conditions simulating real operation. The functionality of an array photodetector is confirmed by measuring its primary photoelectric parameters (noise equivalent temperature difference, voltage sensitivity, defect rate), among which there is no control of matrix displacement [2].

There are scientific papers that determine the linear displacement of the photosensor array during the operation of cryosystems, specifically

in [3, 4]. Methods are described for measuring the orientation of the photosensor array of array photodetectors, controlling the longitudinal and transverse displacements of the cooled finger of the microcryogenic system (hereinafter referred to as MCS), and the tilt angle of the MCS cooled finger.

Research Method

The schematic diagram of the experimental setup for measuring linear displacements of the reticle plane is shown in Figure 1.

The setup is arranged as follows: the investigated vacuum cryostated housing (6) integrated with an MCS of the Kryo 2000 model is placed in front of the microscope objective (2). The objective used has a numerical aperture of $A = 0.64$ and a working distance of 8 mm. Prior research [3] mentions the interest towards the tests using an input window made of leucosapphire to enhance measurement accuracy.

To study linear displacements, a vacuum cryostated housing with a leucosapphire input window was fabricated. The distance between the outer end face of the input window and the plane where the array photosensitive elements are installed is equal to 3 mm on the reticle (see

Figure 2). Linear displacements were recorded along gold paths deposited on the reticle. As a reference on the reticle for determining the maximum displacement amplitude of the plane where the photosensor array was installed, a corner of the gold path was chosen.

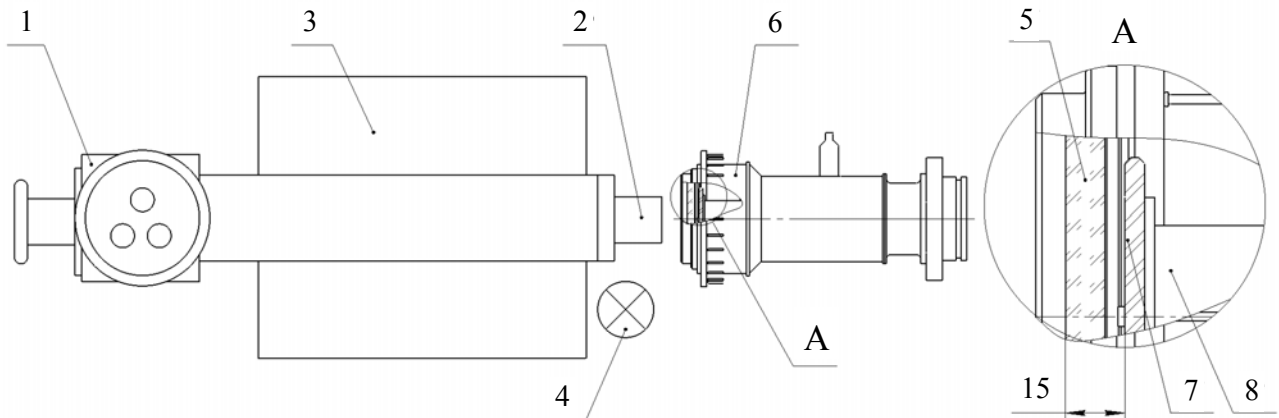


Fig. 1. Setup for monitoring displacements of a photosensor using a microscope: 1 – eyepiece, 2 – microscope objective, 3 – microscope rider, 4 – light source, 5 – input window, 6 – vacuum cryostated housing, 7 – reticle, 8 – cooled finger of the MCS

When applied to monitoring the displacement of the position of the photosensor array plane on the reticle, the measurement accuracy is determined by the sensitivity of transverse and longitudinal focusing. Specifically, the transverse focusing sensitivity governs the measurement accuracy in the direction transverse to the optical axis, whereas the longitudinal focusing determines it along the optical axis [4].

The transverse focusing sensitivity is determined by the formula:

$$T = \frac{1}{6} \cdot \varepsilon_p, \mu\text{m}$$

$$\varepsilon_p = \frac{\lambda}{2A}$$

where ε_p is the linear resolution limit of the instrument, λ is the wavelength, and A is the numerical aperture.

The longitudinal focusing sensitivity is determined by the following expression:

$$\Delta z = \frac{0.2}{A^2}, \mu\text{m}.$$

When monitoring in the visible range, the ultimate measurement accuracy achieved with the selected microscope objective is $0.06 \mu\text{m}$ for measurements in the direction perpendicular to the optical axis and $0.39 \mu\text{m}$ for measurements along the optical axis. Any displacement induced by various reasons will be visually imperceptible if it falls below these values.

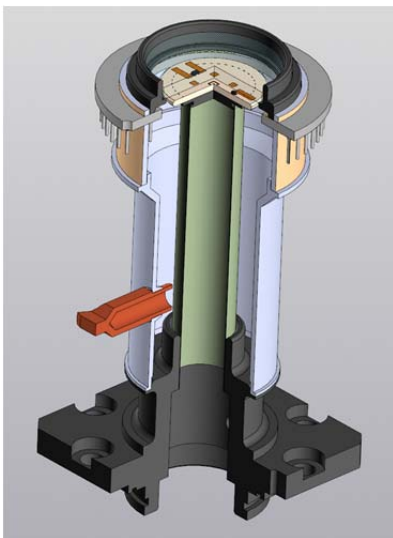


Fig. 2. Three-dimensional model of the vacuum cryostated housing

Measurement Results

Figure 3 shows a gold path with a width of $350\text{ }\mu\text{m}$ in the steady state. When the MCS is activated, vibrations of the photosensor array plane are observed; furthermore, the magnitude of displacements varies depending on the MCS rotor speed. Figures 4a and 4b show the extreme positions describing the displacement amplitude.

Figure 5 shows the obtained experimental dependence of transverse displacements of the

photosensor array plane across the entire range of rotation frequencies under the steady-state mode used in array photodetectors of various configurations. The values on the ordinate axis correspond to the root-mean-square deviation of the controlled point in two directions. The minimum displacement amplitude of $4.3\text{ }\mu\text{m}$ was recorded at the rotation speed of 20 Hz, whereas the maximum amplitude of to $11.9\text{ }\mu\text{m}$ was observed at 33.3 Hz.

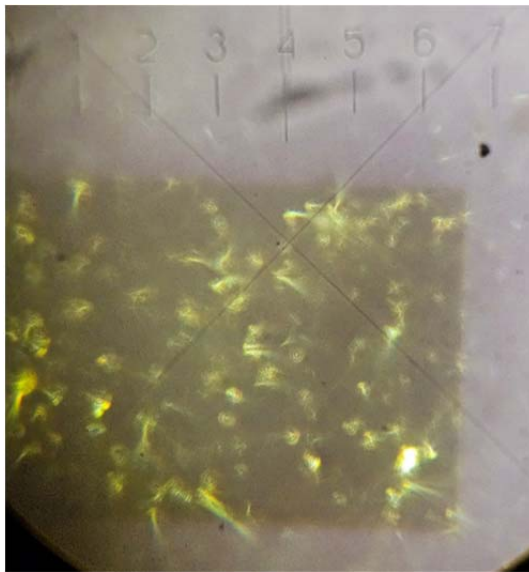
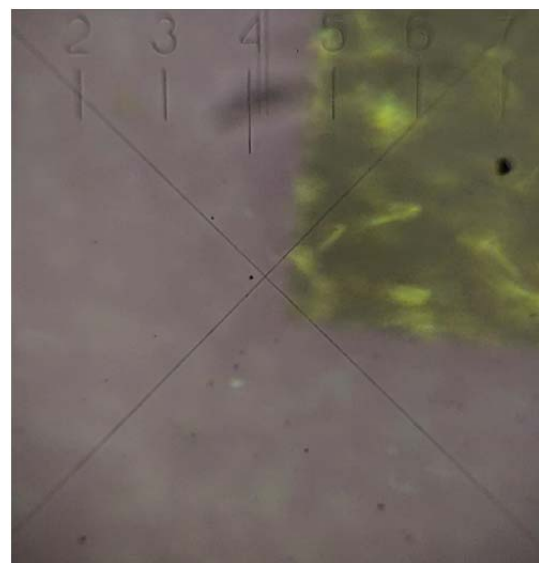


Fig. 3. Gold path on the reticle



a)



b)

**Fig. 4. Displacement of the photosensor array plane of installed on the reticle:
a) – extreme left position, b) – extreme right position**

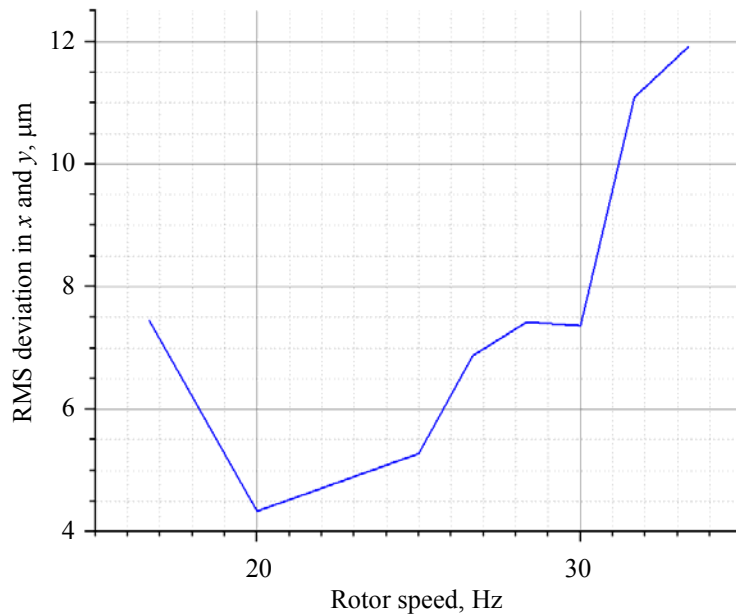


Fig. 5. Displacement of the photosensor array plane at different speeds of the MCS rotor

No displacements along the optical axis were visually observed during the MCS operation, i.e., the maximum amplitude remained below $0.39 \mu\text{m}$.

As can be seen from the cooling capacity graph in Figure 6, the cooling capacity increases as the speed of the MCS rotor speed rises. With

an increase in wear, the efficiency of a single MCS cycle declines; thus, the MCS increases its speed to maintain the cryostating temperature of the array photodetector. Consequently, this implies that as the MCS operating time increases, the displacement magnitude will be higher.

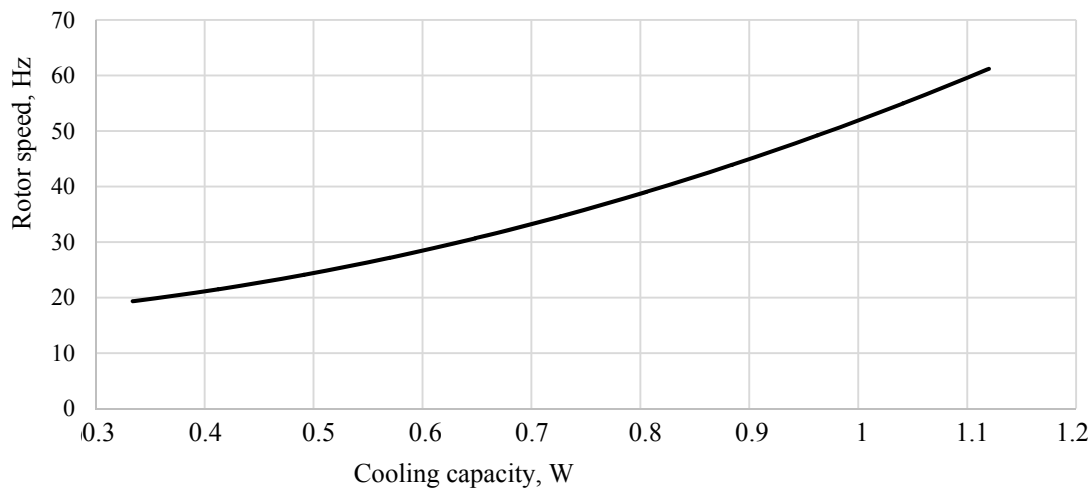


Fig. 6. Graph of the cooling capacity of experimental MCS

Conclusion

The study of the linear displacements of the photosensor array plane during the operation of experimental MCS samples manufactured by AO NPO Orion revealed that the magnitude of longitudinal displacements is less than the measurement accuracy, while the magnitude of

transverse displacements depends on the rotor speed. Depending on the MCS operating mode, the displacement magnitude varies from $4.3 \mu\text{m}$ to $11.9 \mu\text{m}$. Therefore, the increase in displacement amplitude with increasing MCS operating time must be accounted for when developing displacement compensation algorithms for cooled array photodetectors.

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Conference proceedings:

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