

Optical and photoelectrical properties of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ films obtained by high-frequency laser deposition in vacuum

Nikolay A. Bosak¹, Maxim V. Bushinsky², Liudmila V. Baran³, Victoria V. Malyutina-Bronskaya⁴, Olga S. Mantytskaya², Hanna S. Kuzmitskaya⁴

¹ B.I. Stepanov Institute of Physics, National Academy of Sciences of Belarus, 68-2 Nezavisimosti Ave., Minsk 220072, Republic of Belarus

² Scientific-Practical Materials Research Centre of the NAS of Belarus, 19 P.Brovka Str., Minsk 220072, Republic of Belarus

³ Belarusian State University, 4 Nezavisimosti Ave., Minsk 220030, Republic of Belarus

⁴ SSPA "Optics, Optoelectronics and Laser Technology", 68 Nezavisimosti Ave., Minsk 220072, Republic of Belarus

Corresponding author: Nikolay A. Bosak (n.bosak@ifanbel.bas-net.by)

Received 4 March 2025 ♦ Accepted 23 September 2025 ♦ Published 30 December 2025

Citation: Bosak NA, Bushinsky MV, Baran LV, Malyutina-Bronskaya V, Mantytskaya OS, Kuzmitskaya H (2025) Optical and photoelectrical properties of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ films obtained by high-frequency laser deposition in vacuum. *Modern Electronic Materials* 11(4): 243–248. <https://doi.org/10.3897/j.moem.11.4.152006>

Abstract

Nanostructured thin films on silicon and glass substrates have been synthesized by high-frequency pulse-periodic action of laser radiation with a wavelength of 1.064 μm . The morphology of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ thin films investigated using atomic force microscopy showed that films with a developed surface structure have been obtained. Transmission spectra of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ films were obtained in the visible, near and mid-IR regions. The maximum transmittance of the laser-deposited $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on silicon is observed in the near IR region of the spectrum and reaches the value of 59 % at a wavelength of $\lambda = 1182$ nm. Electrophysical characteristics of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structures were analyzed. When measuring the longitudinal current-voltage characteristic of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structure under the influence of laser radiation with a wavelength from 405 nm to 1064 nm (at a voltage of more than 1.5 V), a photoelectric effect have been observed. The measured capacitance-voltage characteristics of the studied structure have demonstrated the photo-ferroelectric properties.

Keywords

electrophysical characteristics, high-frequency laser irradiation, the structure of thin films, transmission and reflection spectra

1. Introduction

Currently, materials based on complex cobalt oxides with a perovskite (cobaltite) structure are gaining great popularity in the field of materials science due to their exceptional optical, magnetic and electrical properties. Among the numerous works devoted to the research

of the magnetic and transport properties of cobaltites, it is worth highlighting a number of investigations aimed at obtaining and studying films of rare-earth cobaltites and their interfaces, as well as studying the differences in the properties of bulk samples and their films [1–6].

Thus, the investigation of LaCoO_3 epitaxial films has shown that below a temperature of 85 K they are ferromagnetically ordered, whereas bulk samples of the same

composition are diamagnetic at temperatures below 30 K [7]. In another paper [8], the magnetic properties of a bulk sample and a film of rare-earth cobalt $\text{Pr}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$ were compared, which showed the absence of a second magnetic transition in the film upon cooling. The works devoted to the study of the properties of cobaltites of the $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$ system should be noted separately. Bulk samples (ceramics) of this system have been well studied [9–11], which cannot be said about film samples. However, in recent years, the situation has begun to change and works have appeared on the study of cobaltites of this system in the form of films. For example, in [12], the possibility of controlling the ordering of oxygen vacancies in $\text{La}_{1-x}\text{Sr}_x\text{CoO}_{3-\delta}$ epitaxial films has been investigated, which makes it possible to control the oxygen anisotropy. The same authors have shown the presence of gigantic anisotropic magnetoresistance in epitaxial films of this system [13].

The physical properties of thin films (up to 1 μm thick) differ significantly from those of the bulk samples. Previously, the authors of [14, 15] obtained and investigated the crystal structure, magnetic and electrical properties of bulk ceramic samples of a system containing anion-deficient cobaltites with a double substitution of $\text{La}_{1-x}\text{Sr}_x\text{Co}_{1-y}\text{Ni}_y\text{O}_{3-\delta}$. In this paper, we present the results of obtaining and comprehensively studying the structural, optical, and electrical properties of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ thin films laser-deposited in vacuum onto glass and silicon substrates under multi-pulse high-frequency laser exposure [16].

2. Experimental

The experimental set up based on a neodymium laser ($\lambda = 1.06 \mu\text{m}$) included an optical system for transporting laser radiation to a sprayed target, a vacuum chamber, and a measuring and diagnostic complex. To obtain a multi-pulse laser generation mode with a high pulse repetition rate, a passive optical shutter made of radiation-irradiated lithium fluoride LiF with F_2 color centers was installed inside the resonator. The repetition rate of the laser pulses was varied by varying the pump level of the laser and the optical density of the shutter, and the duration of the laser pulses at half-height was ~ 85 ns. Effective deposition of thin films was achieved at a laser power density $q = 36 \text{ MW/cm}^2$ and a pulse repetition rate of $f \sim 9\text{--}10 \text{ kHz}$. The films were deposited at a pressure of 2.6 Pa. Ceramic targets $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ were obtained by molding and subsequent sintering at a temperature of $T = 1250^\circ\text{C}$ for 5 h in air. In our case, the initial target $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ and bulk samples of this material are a crystalline substance as well, namely ceramics. The $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ films obtained by the laser deposition method in vacuum are amorphous and this is the main reason for the difference in their physical properties from the bulk ones. Also, the physical properties of thin films are strongly influenced by the heterogeneity of the

surface and the presence of a non-stoichiometric layer at the interface of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ and $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{glass}$ structures.

The morphology of the surface of the samples was investigated using a scanning probe microscope Solver P47-Pro (NT-MDT, Russia). Non-contact silicon cantilevers with a radius of curvature of the needle tip less than 10 nm have been used. The investigation of the surface morphology was carried out in the mode of amplitude-frequency modulation by the constant force method.

The C–V characteristics have been measured in the voltage range from -20 V to $+20 \text{ V}$ on the imittance meter E7-20 (Belarus) at a signal frequency of 500 kHz and 1 MHz. The I–V characteristics have been measured using 4-probe method in the voltage range from 0 V to $+10 \text{ V}$ on the Source Meter 2450, Keithley. The current-voltage (I–V) and capacitance-voltage (C–V) characteristics have been measured in the dark and under the influence of laser radiation. A multispectral source based on a set of laser semiconductor diodes with wavelengths of 405, 450, 520, 660, 780, 808, 905, 980 and 1064 nm and a calibrated radiation power of 2 mW, having a common fiber-optic output, was used as a laser radiation source.

The transmission of optical radiation by thin films in the near infrared (IR) range of the spectrum was measured using a Carry 500 Scan spectrophotometer in the wavelength range of 200 nm – 3000 nm. Reflectivity measurements were carried out using an MS122 “PROSCAN” spectrophotometer in the wavelength range of 200 nm – 1100 nm at an incidence angle of 10° . Transmission spectra in the far infrared region were recorded using a NEXUS infrared Fourier spectrometer (Thermo Nicolet) in the range of 400–4000 cm^{-1} with a resolution of 2 cm^{-1} after 128 scans.

3. Results and discussion

The nanocrystalline structure of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ thin films on a silicon substrate has been established by the atomic force microscopy (Fig. 1): the average height of the surface relief of the films is 23 nm with a RMS roughness of 9.1 nm. The thickness of the obtained films on glass was 200 nm, and on a silicon substrate it reached 250 nm. This is caused by the uneven distribution of the intensity of laser radiation in the impact spot (and, accordingly, by the density of deposited particles along the radius). Large particles with a lateral size of 0.5–1 μm and a height of 50 nm to 400 nm were observed on the surface of the films (Fig. 1c).

The average height of the surface relief of films on a glass substrate is 17 nm with a RMS roughness of 10.3 nm. Large particles with a lateral size of 0.3–2 μm and a height from 50 nm to 300 nm are observed on the surface of the films (Fig. 2c).

The transmission (T) of the silicon substrate (Fig. 3a) increases sharply from $T = 0.5\%$ at a wavelength of $\lambda = 975 \text{ nm}$ to a transmission value of $T = 53\%$ at a wavelength

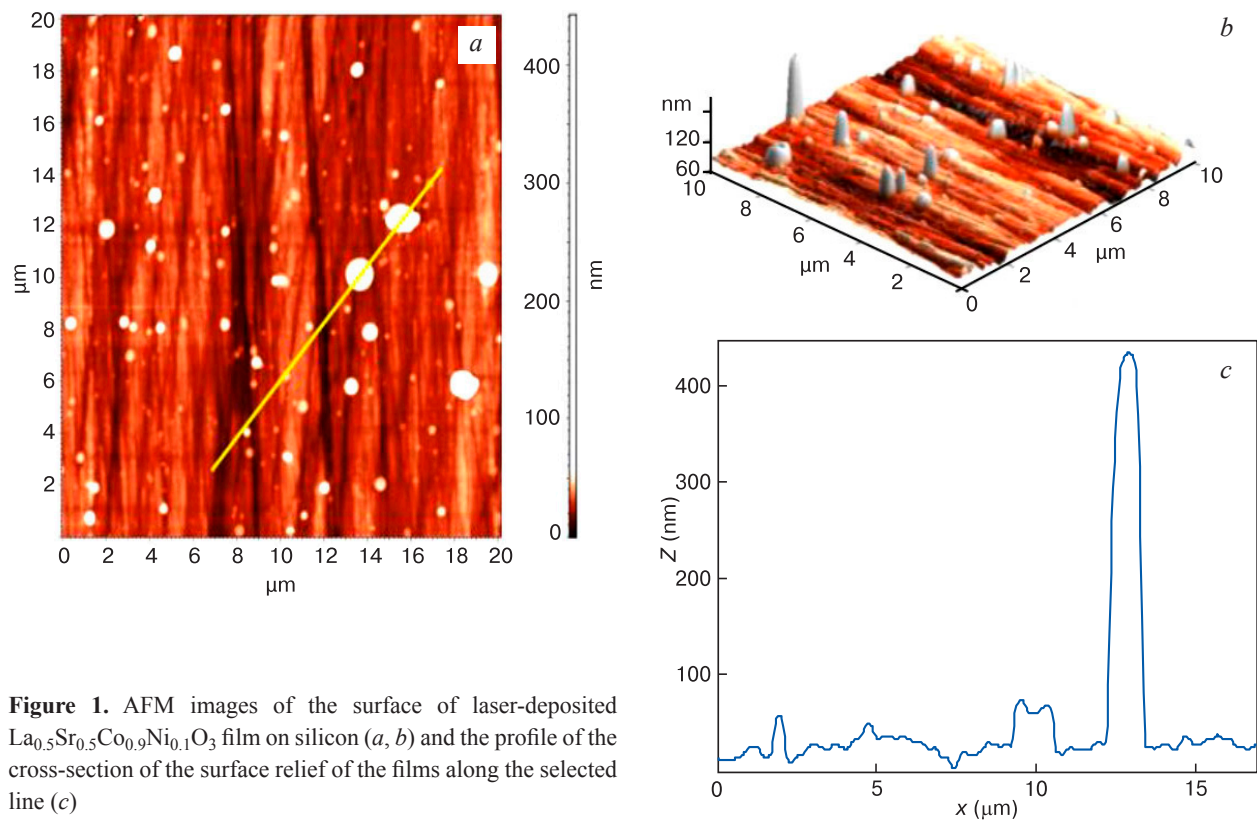


Figure 1. AFM images of the surface of laser-deposited $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on silicon (*a*, *b*) and the profile of the cross-section of the surface relief of the films along the selected line (*c*)

of $\lambda = 1186 \text{ nm}$, remaining almost constant up to $\lambda = 2526 \text{ nm}$. The transmission of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on a silicon substrate increases rapidly from $T = 0.2\%$ at a wavelength of $\lambda = 962 \text{ nm}$ to $T = 59\%$ at a wavelength

of $\lambda = 1182 \text{ nm}$, gradually decreasing to $T = 51\%$ at a wavelength of $\lambda = 2994 \text{ nm}$. The higher transmittance of the film $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ compared to silicon Si in the wavelength range from 1144 nm to 2434 nm

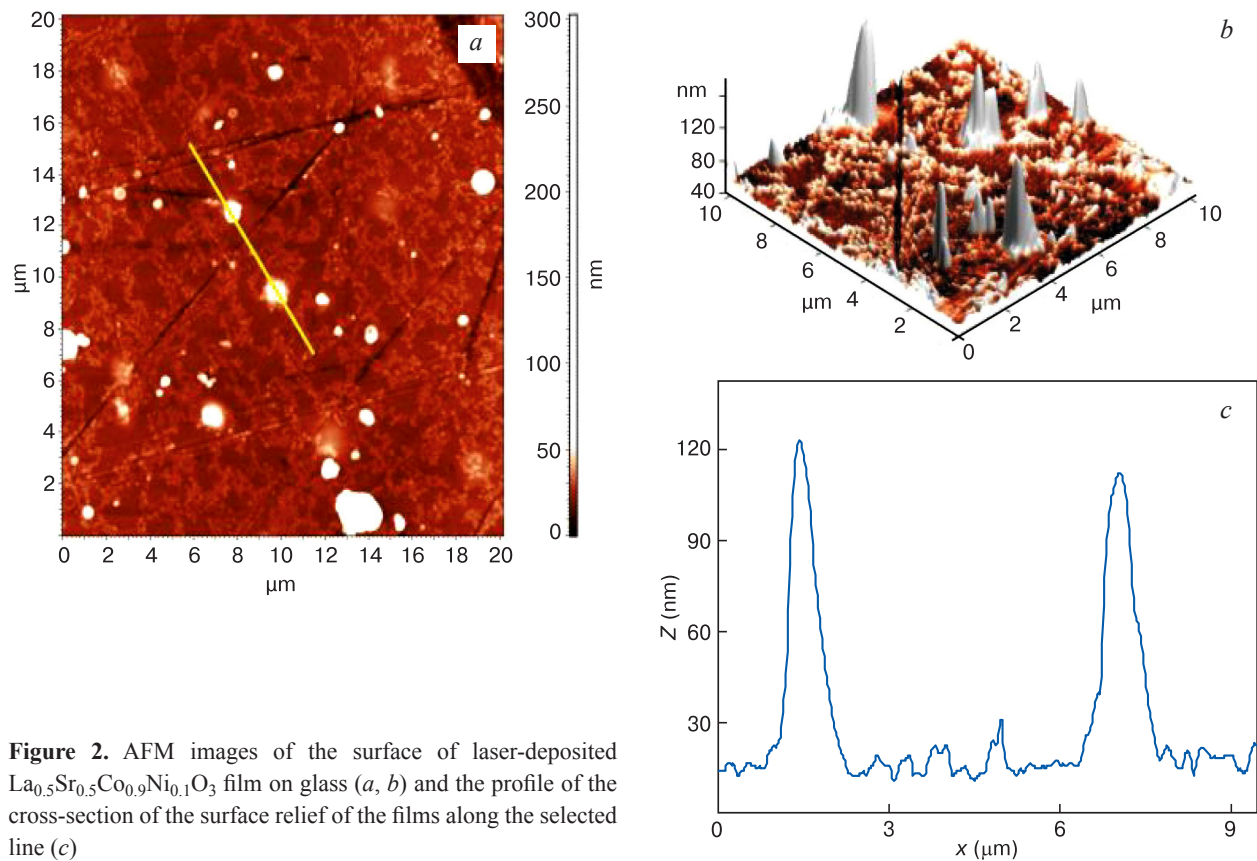


Figure 2. AFM images of the surface of laser-deposited $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on glass (*a*, *b*) and the profile of the cross-section of the surface relief of the films along the selected line (*c*)

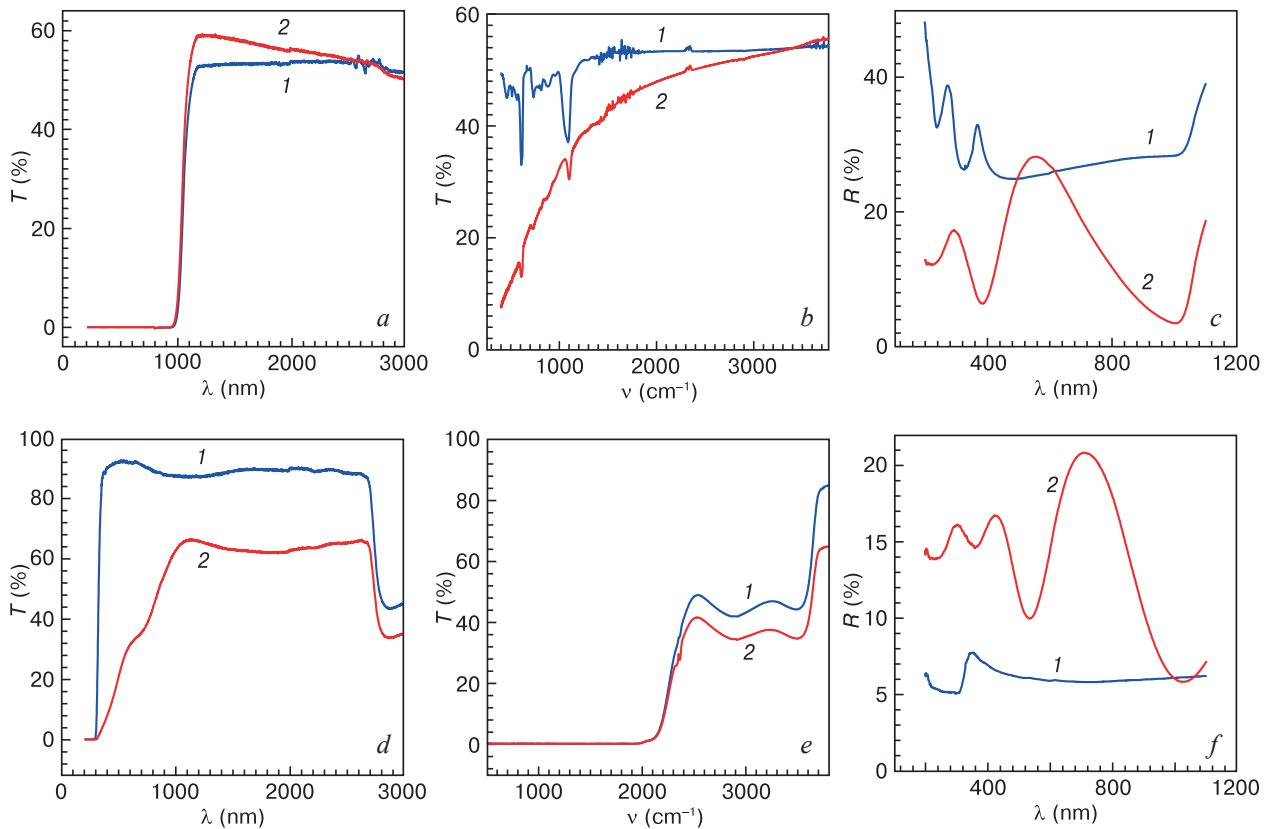


Figure 3. Transmission spectra in the visible and near-IR (*a*, *g*) and mid-IR (*b*, *d*) regions and reflection (*c*, *e*) spectra of laser-deposited $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on silicon (*a*, *b*, *c*: 1 – Si, 2 – $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$) and glass (*d*, *e*, *f*: 1 – glass, 2 – $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{glass}$) substrates

can be explained by the phenomenon of “brightening”. In the middle IR region of the transmission spectrum of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on a silicon substrate, the transmission coefficient increases smoothly from $T = 7.3\%$ at a frequency of $\nu = 407\text{ cm}^{-1}$ to $T = 55\%$ at a frequency of $\nu = 3772\text{ cm}^{-1}$ with pronounced absorption bands of silicon at frequencies of $\nu_1 = 614\text{ cm}^{-1}$ and $\nu_2 = 1099\text{ cm}^{-1}$ (Fig. 3b). The reflection spectrum of silicon (Fig. 3c) has an oscillatory character with two maxima $R_1 = 39\%$ at $\lambda_1 = 275\text{ nm}$ and $R_2 = 33\%$ at $\lambda_2 = 370\text{ nm}$. Two maxima are also observed in the reflection spectrum of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on a silicon substrate: $R_1 = 17\%$ at $\lambda_1 = 294\text{ nm}$ and $R_2 = 28\%$ at $\lambda_2 = 555\text{ nm}$.

The transmission coefficient of the glass substrate in the visible and near-infrared regions of the spectrum (Fig. 3d) increases sharply from $T = 0.2\%$ at a wavelength of $\lambda = 293\text{ nm}$ to a transmission value of $T = 88\%$ at a wavelength of $\lambda = 362\text{ nm}$, and up to a wavelength of $\lambda = 2637\text{ nm}$, the transmission coefficient remains almost unchanged. In the transmission spectrum of the laser-deposited $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on a glass substrate, an increase region is observed from $T = 0.9\%$ at a wavelength of $\lambda = 312\text{ nm}$ to a transmission value of $T = 66\%$ at a wavelength of $\lambda = 1090\text{ nm}$, remaining constant to a wavelength of $\lambda = 2672\text{ nm}$.

The dependence of the transmittance coefficient of a glass substrate in the middle IR region of the spectrum has a two-stage character (Fig. 3e). The transmission of

the glass substrate increases sharply from $T = 2.8\%$ at a wavenumber of $\nu = 2134\text{ cm}^{-1}$ to a transmission value of $T = 48\%$ at $\nu = 2504\text{ cm}^{-1}$, and also increases from a transmission value of $T = 46\%$ at $\nu = 3557\text{ cm}^{-1}$ to a transmission value of $T = 83\%$ at $\nu = 3708\text{ cm}^{-1}$. The transmission of a perovskite film on a glass substrate repeats the shape of the transmission spectrum of the glass substrate with a parallel shift towards lower transmission coefficients due to absorption in the film itself.

In the spectrum of the reflectance coefficient of the glass substrate (Fig. 3f), two maxima are observed: $R = 6.3\%$ at a wavelength of $\lambda = 204\text{ nm}$ and $R = 7.8\%$ at a wavelength of $\lambda = 350\text{ nm}$. In the reflectance spectrum R of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ thin film on the glass substrate, four pronounced maxima are observed: $R_{1\text{max}} = 14\%$ at a wavelength of $\lambda = 205\text{ nm}$, $R_{2\text{max}} = 16\%$ at a wavelength of $\lambda = 304\text{ nm}$, $R_{3\text{max}} = 16.7\%$ at a wavelength of $\lambda = 425\text{ nm}$ and $R_{4\text{max}} = 21\%$ at a wavelength of $\lambda = 711\text{ nm}$.

Figure 4a shows the longitudinal I–V characteristic of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structure at positive voltages applied to the film under the action of laser radiation with different wavelengths. The ratio of the forward to reverse current at a voltage of 18 V was about 2. As it can be seen, under the action of laser radiation at voltages higher than 1.5 V, a photoelectric effect is observed for all wavelengths. Similar behavior of the I–V characteristic under the action of radiation is observed at negative voltages, as

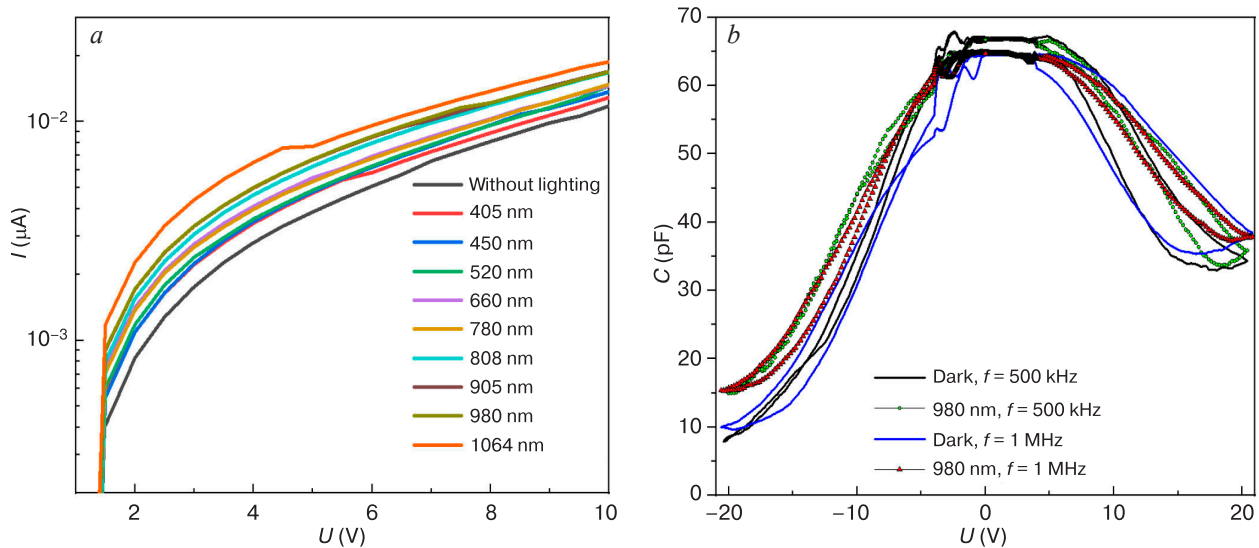


Figure 4. Current-voltage characteristic (a) and capacitance-voltage characteristic (b) of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structure

well. When the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structure is exposed to laser radiation with a wavelength from 405 nm to 1064 nm (at a voltage of +4 V), a photoelectric effect is observed on the longitudinal I–V characteristic and the spectral sensitivity is growing with the increasing wavelength from 0.45 A/W at $\lambda = 450$ nm to 1.81 A/W at $\lambda = 1064$ nm. At a wavelength of $\lambda = 405$ nm, the spectral sensitivity is 1.08 A/W, which is due to the optical band-gap width of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film being equal to 3.81 eV. This has been calculated using the Tauc plot. Wide-gap materials are characterized by a high sensitivity to UV radiation, which is observed in our case at a wavelength of 405 nm. Then, due to the recombination of minority charge carriers at impurity levels both in the film and at the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ interface, the spectral sensitivity drops to 0.45 eV at $\lambda = 450$ nm. Thereafter, with an increase in wavelength up to IR radiation with $\lambda = 1064$ nm, the photosensitivity rises, since the greatest contribution to the photoconductivity starts to be provided by the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ interface and silicon with its impurity levels being formed during the formation of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film.

Figure 4b shows the longitudinal C–V characteristics of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structure without illumination and with illumination by laser radiation with a wavelength of 980 nm at frequencies of 500 kHz and 1 MHz. The C–V characteristic has hysteresis at both

voltage polarities and an asymmetrical appearance, and an insignificant photoelectric effect is observed, as well.

For the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structure, a typical type of C–V characteristic is observed – a “butterfly” loop due to the ferroelectric properties of the film [17]. The hysteresis values ΔU for all measured C–V characteristics are presented in the Table 1. With increasing frequency at different voltage polarities, the hysteresis value for unilluminated structures increases by approximately 2 times, and under illumination with a wavelength of 980 nm, ΔU drops by approximately 1.3 times. The nature of the hysteresis loops indicates nonlinearity with the applied electric field of the dielectric properties of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film.

The photoconductivity of the structure and the change in capacitance under the influence of laser radiation are caused by the mechanisms of capture and recombination of minority charge carriers at impurity levels in the film and at the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ interface [18, 19].

Conclusions

Thin films of $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ on silicon and glass substrates with a developed surface structure have been obtained. The average height of the surface relief of the films on silicon is 23 nm with a root-mean-square roughness of 9.1 nm. A significant number of formations in the form of drops with a diameter of up to 2 μm and a height of up to 400 nm are observed. The maximum transmittance of the laser-deposited $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film on silicon is observed in the near IR region of the spectrum and reaches $T = 59\%$ at a wavelength of $\lambda = 1182$ nm. The spectrum of the reflection coefficient R of the nanostructured film on a silicon substrate has an oscillating character. When the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structure is exposed to laser radiation with a wavelength from 405 nm to 1064 nm (at a voltage of more than 1.5 V), a photoelectric effect is observed on the longitudinal I–V character-

Table 1. The hysteresis value of the C–V characteristics of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3/\text{Si}$ structure without and under illumination by laser radiation

Signal frequency	ΔU (V)			
	$U > 0$		$U < 0$	
	without lighting	light at $\lambda = 980$ nm	without lighting	light at $\lambda = 980$ nm
500 kHz	2.75	2.26	0.74	1.52
1 MHz	5.75	1.73	1.62	1.10

istic and the spectral sensitivity increases with increasing wavelength from 0.32 A/W at $\lambda = 405$ nm to 1.28 A/W at $\lambda = 1064$ nm. Analysis of the C–V characteristics showed that the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ film exhibits ferroelectric properties. With increasing frequency from 500 kHz to 1 MHz, at both voltage polarities, the hysteresis value for unilluminated structures increases by approximately 2 times, and under illumination with a wavelength of 980 nm, ΔU drops by approximately 1.3 times. The photoelectric properties of the $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.9}\text{Ni}_{0.1}\text{O}_3$ structure are determined by the mechanisms of capture and recom-

bination of minority charge carriers at impurity levels both in the film and at the interface.

Acknowledgments

The authors are grateful to the Belarusian Republican Foundation for Fundamental Research for the support of the research work in frames of the Project No. F23ME-029.

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