

Characteristics of an Overstressed Discharge of Nanosecond Duration between Electrodes of Chalcopyrite in High Pressure Nitrogen

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Abstract

The electrical and optical characteristics of the overstressed nanosecond discharge in nitrogen at a pressure of 202 kPa, which was ignited between electrodes from chalcopyrite (CuInSe_2), are presented. Upon sputtering of chalcopyrite electrodes, CuInSe_2 compound vapors have been introduced into the discharge plasma. Chalcopyrite molecules were partially destroyed in the plasma and partially deposited in the form of thin films on a quartz substrate, which was placed near the system of discharge electrodes.

The main decomposition products of a chalcopyrite molecule in an overstressed nanosecond discharge were found, which were in excited and ionized states and which, in the plasma emission spectra, were mainly represented by atoms and singly charged copper and indium ions. The spectral lines of copper and indium are proposed, which can be used to control the deposition of thin films of chalcopyrite in real time.

On quartz substrates, gas-discharge method was used to synthesize thin films based on the CuInSe_2 compound, which effectively absorbed light in a wide spectral range (200-800 nm), which opens up prospects for their use in photovoltaic devices.

Introduction

Chalcopyrite of the $\text{CuInS}_2(\text{Se}_2)$ type is a promising material for use in photovoltaic devices, due to the large absorption coefficients of these materials in the visible and near infrared (IR) spectral regions, as well as their high light stability [1,2]. However, for the practical use of chalcopyrite in photovoltaic devices, it is important to obtain high-quality thin films of a significant area and the corresponding stoichiometric composition.

In the time-resolved emission spectroscopy method was used to study the characteristics and parameters of laser erosion plasma and was formed from a polycrystalline target based on the CuInSe_2 compound in vacuum [3, 4]. Laser deposition allows to obtain high-quality thin films of chalcopyrite with stoichiometric composition, but the yield of materials for solar energy is low, and the process of synthesis of such films is costly. Therefore, it is currently urgent to develop other less costly physical methods for the synthesis of thin films of chalcopyrite, which have the corresponding stoichiometric composition and are suitable for use in photovoltaic converters. It may turn out to be promising to use overstressed nanosecond discharges in gases where chalcopyrite and metal electrodes are sputtered [5, 6]. When working in air at atmospheric pressure,

there is no need to use cost vacuum and laser technology. Due to the generation of runaway electron beams and accompanying x-ray radiation under certain conditions, they are sufficiently uniform and suitable for depositing thin nanostructured films on various substrates. The most studied at present is sub nano second high-voltage discharges in air and nitrogen, which were ignited between metal electrodes at a distance between them in the range of 1-15 cm [7]. Less studied are discharges of nanosecond duration between the electrodes of polycrystalline semiconductors at inter electrode distances in the range of $d = 1-5$ mm.

This article presents the results of a study of the electrical and optical characteristics of nanosecond overstressed discharge in a high-pressure N_2 - CuInSe_2 vapor-gas mixture with injection of chalcopyrite-based electrode vapor into a gaseous medium due to their sputtering, as well as transmission spectra of probe radiation in the wavelength range of 200-500 nm films of chalcopyrite, which were synthesized in the process of experiment.

Technique and Experimental Conditions

The distance between the electrodes of chalcopyrite (CuInSe_2) was 1 mm, and the nitrogen pressure in the chamber was 202 kPa. The

experimental methods and technique, as well as the experimental setup are described in more detail in [5, 6].

When a quartz substrate was installed at a distance of 3 cm from the center of the discharge gap and a discharge burning time of 2–3 hours, film deposition from the sputtering products of the electrodes was recorded on the substrate. The resulting films were studied for transmitting light in a wide spectral region using radiation from deuterium and heat lamps (200–850 nm). These experiments were performed using a spectral complex based on an MDR-23 monochromator at room temperature according to the procedure described in [8].

In a system of chalcopyrite electrodes, at $p(N_2) = 200$ kPa and at a distance between the electrodes of $d = 1$ mm, a spatially uniform discharge in the form of a sphere was ignited, although this discharge was formed without using a separate system of pre ionization of the space between the electrodes.

The characteristic dependences of the electric pulsed power of a nanosecond discharge in nitrogen are presented in Fig. 1. The pulsed power was obtained by graphically multiplying the waveforms of the voltage across the gap and the discharge current. In the experiment, voltage oscillations were recorded in the discharge gap with a half pulse period equal to 10 ns. These oscillations are caused by mismatching of the output resistance of the high voltage pulse modulator with the load. At a nitrogen pressure of 202 kPa, the maximum amplitude of positive and negative polarity voltage peaks was in the range of 30–50 kV. The current pulses of the nanosecond discharge looked like oscillations damped in time with an amplitude of up to 150 A.

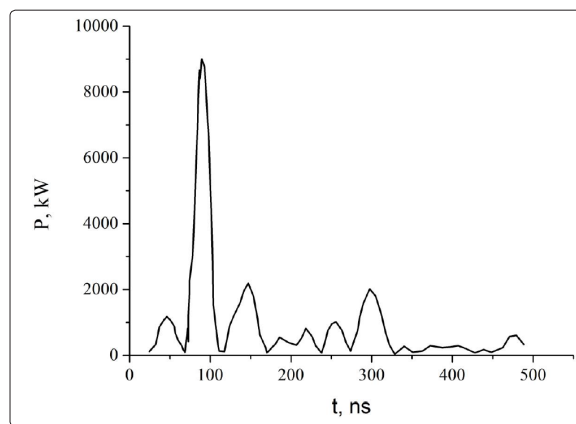


Figure 1: Pulsed power of an overstressed nanosecond discharge at a nitrogen pressure of 202 (2) kPa.

The main fraction of the electric pulsed power was introduced into the plasma of the overstressed nanosecond discharge in the first 100–150 ns. The maximum pulsed electric discharge power in a nitrogen-based mixture reached 9 MW (Fig. 1). With a decrease in the nitrogen pressure of the media to 5 kPa, the pulsed power decreased to 5 MW, and the energy contribution to the plasma was in the range 350–375 mJ [9].

Optical Characteristics of Plasma

Fig. 2 shows the emission spectrum of a nanosecond overstressed discharge plasma between the electrodes of the $CuInSe_2$ compound at a nitrogen pressure of 202 kPa.

The table shows the results of identification of the most intense spectral lines and molecular bands in the emission spectrum of the discharge plasma, which is shown in Fig. 2.

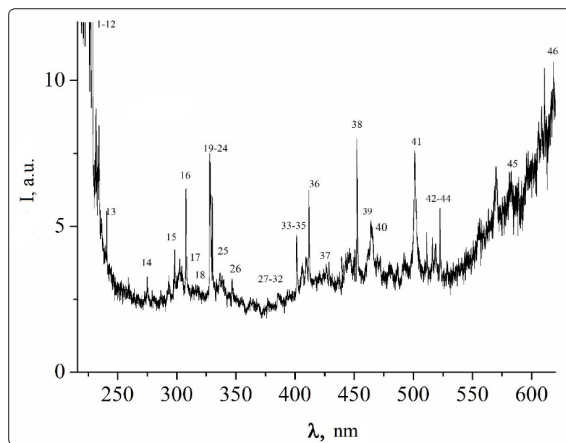


Figure 2: The emission spectrum of nanosecond overstressed discharge at $p(N_2) = 202$ kPa

Table 1: The results of the identification of the spectrum of the discharge radiation at a nitrogen pressure of 202 kPa

λ , nm	$I_{exp. a.u.}$	Object	$E_{low.}$ eV	$E_{up.}$ eV	Lower term	Upper term	
1	216.50	31.930	Cu I	0.00	5.72	4s ² S	4p' ² D ^o
2	218.17	19.179	Cu I	0.00	5.68	4s ² S	4p' ² P ^o
3	220.97	12.062	Cu II	8.78	14.39	4p ³ D ^o	4d ³ D
4	221.45	20.454	Cu I	1.39	6.98	4s ² ² D	4p'' ² P ^o
5	222.56	25.788	Cu I	0.00	5.57	4s ² S	4p' ⁴ D ^o
6	224.26	13.028	Cu II	3.26	8.78	4s ¹ D	4p ³ D ^o
7	224.70	17.438	Cu II	2.72	8.23	4s ³ D	4p ³ P ^o
8	226.37	17.517	Cu II	8.92	14.39	4p ¹ F ^o	4d ³ D
9	227.62	14.472	Cu II	2.98	8.42	4s ³ D	4p ³ P ^o
10	231.32	9.0328	In II	12.10	17.46	5s5d ¹ D	5s9p ¹ P ^o
11	233.45	7.7654	In II	12.68	17.99	5s5d ³ D	5s8f ³ F ^o
12	234.34	8.4608	In II	13.37	18.65	5s6p ³ P ^o	5s18d ¹ D
13	240.66	5.5221	Cu I	1.64	6.79	4s ² ² D	6p ² P ^o
14	274.97	3.2812	In II	12.10	16.61	5s5d ¹ D	5s5f ¹ F ^o
15	298.63	3.9352	Cu II	14.20	18.35	4s ² ³ P	5p ¹ P ^o
16	307.38	6.3182	Cu I	1.39	5.42	4s ² ² D	4p' ² F ^o
17	314.27	2.91573	In II	12.66	16.60	5s5d ³ D	5s9f ³ F ^o
18	315.93	3.01321	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (1; 0)			
19	324.75	2.67602	Cu I	0	3.82	4s ² S	4p ² P ^o
20	327.39	2.6437	Cu I	0	3.39	4s ² S	4p ² P ^o
21	328.27	7.5029	Cu I	5.15	8.93	4p' ⁴ F ^o	4d' ² G
22	329.05	7.1779	Cu I	5.07	8.84	4p' ⁴ F ^o	4d' ⁴ F
23	330.79	6.0058	Cu I	5.07	8.82	4p' ⁴ F ^o	4d' ⁴ G
24	333.78	3.2064	Cu I	1.39	5.10	4s ² ² D	4p' ⁴ F ^o
25	337.13	3.16835	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (0; 0)			
26	348.37	2.47326	Cu I	5.51	9.06	4p' ⁴ D ^o	4d' ⁴ G
27	350.05	2.46826	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (2;3)			
28	357.69	2.27036	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (0;1)			
29	367.19	2.28779	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (3;5)			
30	371.05	2.02989	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (2;4)			
31	375.54	2.25408	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (1;3)			
32	394.30	2.51954	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (2;5)			
33	402.26	4.4826	Cu I	3.79	6.87	4p ² P ^o	5d ² D
34	405.94	3.6360	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (0;3)			
35	409.48	3.9541	N ₂	Second positive system C ³ Π _u ⁺ -B ³ Π _g ⁺ (4; 8)			
36	410.17	6.2437	In I	-	3.02	5s ² 5p ² P ^o	5s ² 6s ² S _{1/2}
37	427.99	3.7876	Cu II	15.07	17.96	5p ³ D ^o	7s ³ D
38	451.13	8.0094	In I	0.27	3.02	5s ² 5p ² P ^o	5s ² 6s ² S _{1/2}
39	459.97	3.7377	N ₂ ⁺	² Σ→ ² Σ			
40	462.07	4.0571	In II	15.33	18.01	5s4f ³ F ^o	5s _{1/2} F=4 8g
41	500.67	7.5981	Cu II	14.65	17.13	4d ¹ D	4f ¹ F ^o
	500.52	7.5981	N II	25.50	27.97	3s ⁵ P	3p ⁵ P ^o
42	510.55	4.5436	Cu I	1.39	3.82	4s ² ² D	4p ² P ^o
43	515.83	4.6149	Cu I	5.69	8.09	4p' ² P ^o	5s' ² D
44	521.82	5.5957	Cu I	3.82	6.19	4p ² P ^o	4d ² D

45	570.02	7.0079	Cu I	1.64	3.82	4s ² 2D	4p 2P ^o
46	609.59	9.5381	In II	13.44	15.47	5s6p 3P ^o	5s6d 3D

The line part of the emission spectrum was observed against the background of continuous radiation (continuum) of the plasma, which may be due to thermal radiation of the plasma or its recombination radiation. As shown in copper and indium atoms are less bound in the chalcopyrite molecule, which is a component of massive electrodes [10]. Therefore, the line part of the plasma radiation spectrum is mainly due to individual spectral lines of atoms and singly charged copper and indium ions, as for a laser plasma, which was formed on the target surface from the compound under vacuum, as for a gas-discharge air plasma [11-14].

The emission spectrum of the gaseous component manifested itself at high nitrogen pressures and consisted mainly of intense bands of the second positive system of the nitrogen molecule in the spectral range of 280-390 nm.

The emission spectrum of overstressed nanosecond discharges in the spectral range 200-230 nm consisted of a group of closely spaced spectral lines of an atom and a singly charged copper ion and low-intensity lines of a singly charged indium ion (spectral lines 1-13; Table 1). The spectral lines of copper were similar to those that we observed in the emission spectra of plasma overstressed nanosecond discharge between copper electrodes in atmospheric pressure air at a distance between copper electrodes of $d = 2$ mm [14,15]. As for the discharge between copper electrodes, the intensity of this group of spectral lines decreased with decreasing nitrogen pressure from 202 to 101 kPa. An essential role in introducing the material of electrodes, both metal and semiconductor, into the interelectrode gap is played by the ectonic mechanism of their sputtering in a strong electric field of a nanosecond discharge [16].

The intensive group of spectral lines and bands of the nitrogen molecule is visible in the spectral range of 290-410 nm (lines and bands 14-35; Table 1). For the largest part of the spectrum, the most characteristic was the spectral lines of copper atom (lines 16, 19-24, 26, 33), as well as the intense bands of the second positive system of nitrogen molecule (bands 14, 18, 25, 27-32, 34, 35). The atomic and single-charged ions in the visible region in the visible spectral range are represented by spectral lines 410.17; 451.13 nm In I and 462.07; 609 nm In II. The radiation spectra of plasma also showed a characteristic spectral line with the wavelength of 500.5 nm N II, which is often observed in the radiation spectra of nanosecond discharges in an atmospheric pressure air [17].

In the yellow-red regions of plasma spectrum (Fig. 3), the continuous spectrum in the form of the continuum was recorded, against the background of which, separate lines of small intensity, as well as molecular bands have been located, which could appear due to the radiation of selenium molecules.

With an increased pressure of nitrogen from 101 to 202 kPa, the main features of the spectrum of an overstressed nanosecond discharge plasma were caused by significant magnitude of the intensity of the group of spectral lines of Cu I, Cu II, and In II in the range of wavelength 200-250 nm. The intensities of the emission bands of the nitrogen molecule, in this case, are sharply reduced. Instead of bands of a nitrogen molecule in the spectral range 290 - 460 nm,

only separate intense spectral lines of Cu I, In I, In II (lines 15-39) appear in the radiation spectrum.

Radiation in the spectral range of 550–630 nm was observed in the form of molecular bands, against the background of which individual low-intensity spectral lines of atoms or ions were also recorded. Accurate identification of this part of the spectrum requires the use of a spectrophotometer with a higher spectral resolution.

To diagnose the process of deposition of thin films of chalcopyrite on solid substrates of glass or quartz in real time, it is possible to use the following intense spectral lines of copper and indium atoms in the spectral range 300-460 nm 307.38 CuI, 329.05 Cu I, 410.17 In I and 451.13 nm In I.

Plasma Parameters

The plasma parameters were calculated under the assumption that it is possible to replace the chalcopyrite molecule with a copper atom [18-20].

The parameters of the discharge plasma in a mixture of nitrogen with chalcopyrite vapor at a partial pressure ratio of 202 kPa: 30 Pa were determined numerically by solving the Boltzmann kinetic equation in the two-term approximation using the program, which also includes the effective cross sections for the interaction of electrons with copper atoms and nitrogen molecules [21, 22].

Table 2: Transport characteristics of electrons on a mixture of nitrogen and copper vapor with a component ratio of 202 kPa: 30 Pa

E/N, Td	ϵ , eV	T0, K	V, m/s	N, cm ⁻³
614	2.352	27283,2	9.6·105	4.95·1019
1025	4.422	51295,2	12·105	3.96·1019

Table 3: The rate constants of excitation (k) and ionization (k_i) by the electrons of the resonant (k_r) and metastable (k_m) levels of copper atoms and nitrogen molecules in a mixture of nitrogen with copper vapor. The ratio of the components is 202 kPa: 30 Pa.

E/N, Td	$k_r \cdot 10^{13}$, m ³ /s	$k_m \cdot 10^{15}$, m ³ /s	$k_i \cdot 10^{15}$, m ³ /s	$k_r \cdot 10^{16}$, m ³ /s	$k_m \cdot 10^{17}$, m ³ /s	$k_i \cdot 10^{17}$, m ³ /s
Cu			N2			
614	0.66	5.1	1.1	0.31	0.82	0.31
1025	2.04	13	4.8	1.29	3.24	8.1

Numerical simulation of the transport characteristics of electrons on a mixture of nitrogen and copper vapor with a component ratio of 202 kPa: 30 Pa (Table 2) revealed that in plasma with increasing values of the reduced field strength (E/N) at which the experiments were carried out, their growth was observed (mean electron energy (ϵ), electron temperature (T^0K), as well as the electron drift velocity (V) in the electric field. The electron concentration (N) decreased with a higher value of the reduced electric field intensity. The rate constants of excitation and ionization by electrons of copper atoms and air molecules (Table 3) also increase with increasing the parameter E/N. The maximum values are observed for the excitation constant of the resonance level of copper atoms.

The specific losses of discharge power in a mixture of copper vapor with nitrogen at a component ratio of 30Pa: 202 kPa for inelastic collisions of electrons with mixture components are maximal for nitrogen molecules (FIG. 3) And reaches about 30% for excitation of vibrational levels of N_2 ($V1_{res}$ nitrogen molecules with the reduced electric field strength equal to 46 Td, and for copper atoms they did not exceed 0.39% (to excite the resonance state $2P_{3/2^o, 1/2}$ at $E/N = 718$ Td). For the reduced field strength 614 Td, they were at the level of 0.36%. With the increase of for the parameter E/N up to 1000 Td, specific losses of the discharge power in the mixture reach a maximum of 21% for the N_2 electronic (C3) process of nitrogen molecules collision with electrons. The rate of increase and decrease of the discharge power losses on the processes of excitation of electronic states and ionization and its value are related to the nature of the dependence of the effective cross sections of inelastic processes of electron collisions with the components of the mixture on energies of electrons, their absolute values, as well as with the dependence of the electron distribution function on the magnitude of the reduced field strength and threshold energy of the process. The losses in the discharge power due to the excitation and ionization of copper atoms are small, due to the low content of copper vapor in the mixture. Based on the obtained distribution of power losses in the discharge, one can expect a significant role from the processes of energy transfer from metastable nitrogen molecules to copper atoms in a plasma on a mixture of atmospheric pressure copper and nitrogen. This assumption was experimentally confirmed in [23].

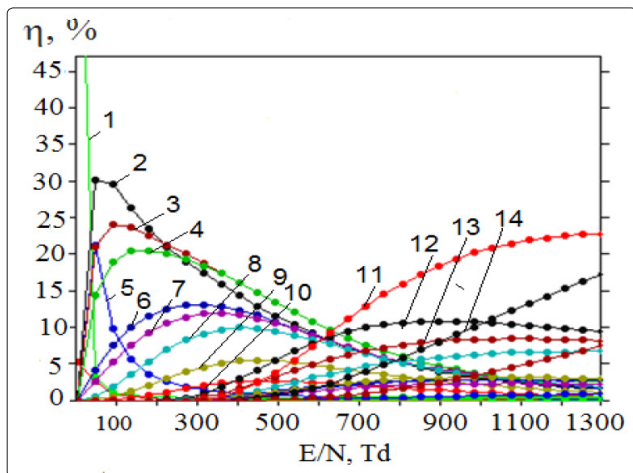


Figure 3: The dependence of the specific power losses of the discharge power on the processes of collisions of electrons with nitrogen molecules on the E/N parameter in a plasma for Cu: $N_2=30.3$ Pa: 202 kPa at a total pressure of $P=101.030$ 7

kPa: 1 - N_2 rotational excitation, 2 - N_2 vibrational ($V1_{res}$), 3- N_2 vibrational ($V=2$), 4- N_2 vibrational ($V=3$), 5- N_2 vibrational ($V=1$), 6- N_2 vibrational ($V=4$), 7- N_2 vibrational ($V=5$), 8- N_2 vibrational ($V=6$), 9- N_2 vibrational ($V=7$), 10- N_2 vibrational ($V=8$), 11- N_2 electronic (C3), 12- N_2 electronic (B3), 13- N_2 electronic (W3), 14- N_2 sum of singlet states.

Radiation Transmission Spectra of Chalcopyrite Films

Typical radiation transmission spectra of thin films based on the $CuInSe_2$ compound in the spectral region of 200-800 nm, which were synthesized in nitrogen at various pressures, are shown in FIG. 4. The film was probed by radiation from a deuterium lamp in the spectral range 200–500 nm.

The transmission of thin chalcopyrite films, compared to the transmission of the substrate, decreased by about 2-2.5 times and for the film, was synthesized using a discharge in nitrogen, was minimal at $p(N_2) = 101$ kPa. The forms of the transmission spectra for chalcopyrite films at nitrogen pressures of 13.3 and 101 kPa were close. The decrease in the transmission of the film that was synthesized at $p(N_2) = 101$ kPa compared to the transmission of the film synthesized at $p(N_2) = 5.5$ kPa may be due to the smaller thickness of the film that was synthesized at low nitrogen pressure.

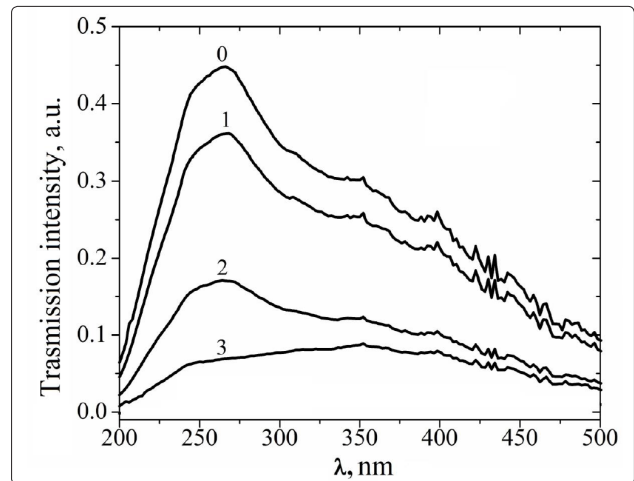


Figure 4: Light transmission spectra of chalcopyrite films deposited on quartz substrates, depending on the pressure and type of gas medium in the discharge, when they are probed with radiation from a deuterium lamp: 0 - without sample; 1 - pure quartz glass; 2 - electrodes from $CuInSe_2$ compound at $p(N_2) = 13.3$ kPa; 3 - $CuInSe_2$ at $p(N_2) = 101$ kPa

The strong absorption of deuterium lamp radiation by chalcopyrite films in the spectral range (200-500) nm is due to the fact that when thin chalcopyrite films are deposited by the gas-discharge method and using electrodes based on the $CuInSe_2$ compound, they repeat the stoichiometry of the electrode material. This is important when using synthesized films in photovoltaic devices.

Conclusion

Thus, it was found that at a nitrogen pressure of 202 kPa between the electrodes based on the $CuInSe_2$ compound and an interelectrode distance of 1 mm, a uniform nanosecond discharge is ignited with a pulsed electric power of 5–9 MW and an energy contribution to the plasma of 0.35 J per pulse. Investigation of spectral plasma characteristics based on « $N_2 - CuInSe_2$ » mixture showed that the most intense are the spectral lines of the copper atom in the wavelength range 200-250 nm and the spectral lines of the indium atom and copper and indium ions in the longer wavelength part of the spectrum. The nature of the plasma emission spectra allows the presence of selective mechanisms of the formation of excited atoms and copper ions, indium in the plasma, which are determined by the transfer of energy from metastable nitrogen atoms and molecules. Based on the measured relative intensities of the spectral lines of atoms and ions of copper, indium, it is possible to carry out estimates of the temperature and density of electrons in the plasma that was studied. The following separately placed and most intense lines in the spectral range of 300-460 nm can be used to diagnose the deposition of chalcopyrite films in real time: 307.38 Cu I, 329.05 Cu I, 410.17 In I, 451.13 nm In I.

Plasma parameters are determined for the reduced electric field strength at which experimental studies were carried out. It has been established that there is an increase in the average energy and temperature of electrons, as well as the electron drift velocity in an electric field. The electron concentration (N) at a higher value of the reduced intensity decreased. The rate constants of excitation and ionization by electrons of copper atoms and air molecules (also increase with increasing the parameter of the reduced electric field strength). The maximum values are observed for the excitation constant of the resonance level of copper atoms. The powers that are introduced in inelastic collisions of electrons with the components of the plasma mixture are maximal for excitation of vibrational energy levels of nitrogen. Based on the obtained distribution of power losses in the discharge, one can expect a significant role of the processes of energy transfer from metastable nitrogen molecules to copper atoms in a plasma on a mixture of atmospheric pressure copper and nitrogen.

The study of the spectra of the relative transmission of probe radiation in the wavelength range of 200-500 nm by chalcopyrite films synthesized in nitrogen showed that the transmission is the least for films synthesized at elevated gas pressures, which is due to more intense atomization of the electrodes and an increase in the thickness of chalcopyrite films.

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