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REGULAR ARTICLE

Synthesis of Surface Nanostructures of Silver Sulfide in Argon at Atmospheric Pressure in a Gas Discharge

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The study explores the specifications of a nanosecond overvoltage discharge in argon occurring between electrodes crafted from a superionic conductor, silver sulfide (Ag_2S). The discharge was initiated under argon pressures ranging from 13.3 to 101 kPa, with a 2 mm separation between the polycrystalline Ag_2S electrodes. Electrode material degradation within the discharge, alongside the introduction of Ag_2S vapor into the interelectrode region, stemmed from microexplosions triggered by inherent surface irregularities on the electrodes. This discharge configuration holds potential as a plasma-chemical reactor for synthesizing thin films predicated on silver sulfide. Additionally, the study presents findings from analyzing Raman scattering spectra of laser radiation interacting with films synthesized from the Ag_2S compound.

Keywords: Nanosecond discharge, Silver sulfide, Argon, Thin films, Radiation spectrum, Raman scattering spectra, Plasma.

1. INTRODUCTION

The modern development of nanotechnology requires an increasing number of different nanostructured elements for use in photovoltaics, sensor, laser and optoelectronic technology, photobiology and medicine, the synthesis of which is hindered by the lack of data on the basic conditions and physical mechanisms of the synthesis of surface nanostructures from gas discharge plasma products. Currently, there is no data on the relationship between the characteristics of nanostructures of superionic conductors with the characteristics of overvoltage nanosecond discharges in gas media of different pressure and composition and the characteristics of the corresponding nanostructures.

The development of new gas-discharge UV lamps based on the destruction products of superionic conductors (Ag_2S, Ag_8GeS_6), and the development of a technique for the synthesis of thin nanostructured films with the properties of superionic conductors, which are promising in the creation of supercapacitors, photovoltaic devices, sensitive gas sensors, catalysts, sorbents, biomedical engineering, medicine, biology, agriculture, food industry and which are implemented in non-equilibrium gas-discharge plasma and the study of the effect of various physical factors on the formed surface nanostructures are actual problems of experimental physics.

The work [1] reported on the synthesis of a thin film of a superionic superconductor – Ag_2S in a magnetron discharge with a closed magnetic field in hydrogen or an Ar–H_2 mixture. When using working gas mixtures

($p \approx 10^{-15}$ Pa) and at a voltage between the electrodes of 250 V and a discharge power of 25 W, a film of Ag_2S compound with a thickness of 40 nm was synthesized in 20 minutes.

The results of the synthesis of a coating based on the Ag_2S compound from the gas phase, which was formed by an electron beam with an energy of 800-1600 eV, are given in [2]. As a source of UV illumination, a laser was used that generated radiation with a wavelength of λ = 266 nm (laser pulse duration − 6 ns; energy − 117 mJ). The main disadvantage of this method of synthesizing silver sulfate films is the need to maintain a high vacuum in the reactor and use an expensive UV laser.

Chemical methods of synthesis of superionic conductor – Ag_2S were used in [3]. About the results of the hydrochemical deposition of Ag_2S/Ag heteronanostructures and the study of the phase transformation "acanthite – Ag_2S – argentite – Ag_2S".

The properties of an overvoltage nanosecond discharge occurring in atmospheric pressure argon between aluminum electrodes [4] have been effectively utilized to produce thin aluminum films within the UV radiation field of the discharge's plasma emission, operating automatically. Similar findings were reported in [5] for an overvoltage nanosecond discharge in argon between electrodes composed of chalcopyrite ($CuInSe_2$), and in [6] for a discharge between an aluminum electrode and chalcopyrite ($CuInSe_2$), leading to the determination of conditions for synthesizing quaternary chalcopyrite $CuAlInSe_2$. These

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investigations' outcomes are consolidated in review [7].

Presently, there is a gap in research regarding the characteristics of an overvoltage nanosecond discharge between silver sulfide electrodes in atmospheric pressure argon. Consequently, essential conditions for synthesizing surface micro-nanostructures based on silver sulfide from this discharge's plasma remain undiscovered, hindering the advancement of suitable plasma chemical reactors.

This article presents experimental findings concerning the characteristics of an overvoltage nanosecond discharge in argon between polycrystalline silver sulfide electrodes. It details the synthesis of thin films based on silver sulfide and their analysis through Raman scattering spectra of laser radiation.

2. TECHNIQUE AND CONDITIONS OF THE EXPERIMENT

A nanosecond discharge with overvoltage, featuring bipolar voltage pulses, was triggered within atmospheric pressure argon. The electrodes utilized were composed of a polycrystalline Ag_2S compound, re-nowned as a superionic conductor. The configuration of the discharge apparatus is depicted in Figure 1.
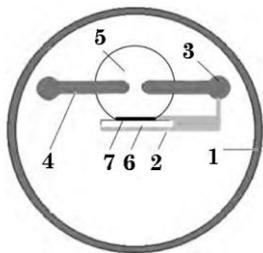

Fig. 1 – Configuration of the discharge chamber: 1 – enclosure of the dielectric discharge chamber; 2 – substrate fixation mechanism for sputtering thin films; 3 – the apparatus for adjusting the interelectrode gap; 4 – metallic electrodes; 5 – region for film deposition; 6 – glass substrate; 7 – film structure

The electrical power of a high-voltage nanosecond discharge was determined by analyzing voltage and current pulse oscillograms, with integration revealing the energy of each electric pulse delivered to the plasma. The discharge occurred between electrodes spaced 2 mm apart, each with a 10 mm radius and 5 mm diameter. To capture voltage and current pulses, a setup including a broadband capacitive voltage divider, a Rohovsky coil, and a 6LOR-04 broadband oscilloscope was employed, providing a time resolution of 2-3 ns. Plasma radiation spectra were recorded using a digital two-channel spectrometer with astigmatism compensation, the "SL-40-2-1024USB," operating in the range of 200-1200 nm. The absolute radiation power of a reference lamp was measured with a "TKA-PKM" ultraviolet absolute radiation power meter. A nanosecond discharge was triggered by an overvoltage across the discharge gap, coinciding with the formation of a runaway electron beam within it [8]. This discharge, occurring in atmospheric pressure argon, exhibited uniformity despite the uneven distribution of electric field strength between electrodes with rounded

hemispherical surfaces (~ 10 mm radius) (Fig. 2). The discharge orifice was approximately square-shaped, spanning about 4 mm^2. In the presence of a strong electric field on the surface of electrodes composed of the superionic conductor Ag_2S, microexplosions of nanowisters ensued [9], facilitating the release of vapors from the Ag_2S superionic conductor and their subsequent decomposition (Ag, Ag^+, $S...$) into the plasma. These vapors then deposited onto the glass substrate, forming a thin film based on the superionic conductor Ag_2S. The initial presence of Ag_2S molecules within the discharge gap, acting as easily ionizable impurities, also contributed to achieving a uniform discharge.

Fig. 2 – View of an overvoltage nanosecond discharge between Ag_2S electrodes in atmospheric pressure argon at a discharge pulse repetition rate f = 1000 Hz

When positioning a glass substrate 2-4 cm away from the discharge gap center (as shown in Figure 1) and allowing the discharge to burn for 30-60 minutes, we observed the deposition of a thin film composed of sputtered electrode material onto the substrate. The resulting thin film samples were analyzed using the "XploRA PLUS" Raman scattering spectrometer, enabling the examination of the film surface characteristics through imaging.

3. CHARACTERISTICS OF AN OVERVOLTAGE NANOSECOND DISCHARGE

Fig. 3 displays oscillograms illustrating the current and voltage pulses, the pulsed power of a nanosecond overvoltage discharge occurring between electrodes coated with the Ag_2S compound in atmospheric pressure argon. Additionally, it presents the energy contribution to the plasma per pulse.
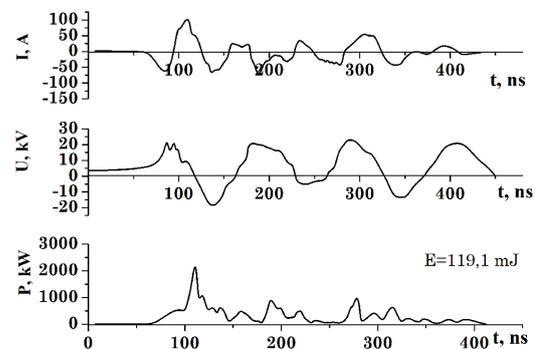

Fig. 3 – Oscillograms of current and voltage pulses, pulsed power and energy contribution to an overvoltage nanosecond discharge per pulse (p_{Ar} = 101 kPa, f = 1000 Hz, d = 2 mm)

In this experiment, the total duration of voltage pulses ranged from 400 to 450 ns. The voltage pulse itself exhibited oscillations that gradually dampened over a period of about 40 to 50 ns. Across the discharge gap, the maximum voltage drop of one polarity reached 22 kV, while the maximum amplitude of the current pulse of one polarity peaked at 100 F. The highest pulsed power of the discharge was achieved within the first 120 ns from ignition, reaching approximately 3 megawatts (at t = 110-120 ns). Subsequently, the second and third peaks of pulsed power, each around 1 MW, were observed between t = 175-300 ns from the start of discharge ignition. The energy of an individual electrical pulse measured approximately 119 mJ.

The dependencies of the radiation intensity of a nanosecond overvoltage discharge in argon at different pressures across the UV-C, UV-B, and UV-A spectral ranges are illustrated in Figures 4 and 5. These are shown in relation to the repetition frequency of voltage or current pulses, while keeping a constant voltage at the anode of a hydrogenous thyratron, as well as the voltage pulse modulator with a stable repetition rate.
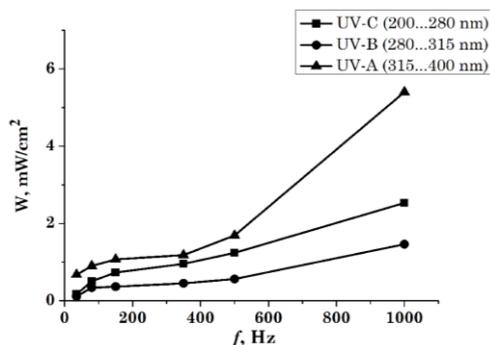

Fig. 4 – Dependences of UV intensity – UV-C, UV-B, UV-A radiation – discharge ranges on the frequency of repetition of current pulses at a charging voltage of U = 13 kV in argon between Ag_2S electrodes at a pressure of 101 kPa (d = 2 mm)
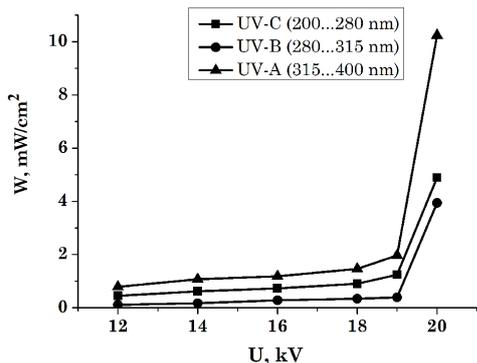

Fig. 5 – Dependences of UV intensity – UV-C, UV-B, UV-A – discharge ranges on the voltage on the electrodes at f = 80 Hz in argon between Ag_2S electrodes at a pressure of 101 kPa (d = 2 mm)

The maximum total average power of UV radiation in atmospheric argon, encompassing UV-C (200-280 nm) and UV-B (280-315 nm) ranges, reached 8.82 mW/cm² at a voltage pulse repetition rate of 1000 Hz. Elevating the frequency from 350 to 1000 Hz resulted in a notable augmentation in UV radiation

intensity across all ranges. The most substantial increase occurred within the spectral range $\Delta\lambda$ = 315-400 nm (refer to Fig. 4).

As the voltage at the anode of the hydrogenous thyratron from 12 to 20 kV (see Fig. 5), a rise in the discharge UV radiation intensity was observable across all ranges. However, compared to frequency escalation, voltage increment yielded a comparatively smaller boost in UV radiation intensity across all UV spectral ranges.

These findings underscore that even at a minimal voltage of 12 kV, the intensity of "soft" UV radiation predominated the overall plasma radiation intensity within the spectral range of 200-315 nm. This dominance holds significance for applications in biomedical engineering and medicine. Such a lamp presents a alternative to exciplex lamps (λ = 308 nm XeCl*) for treating skin ailments.

The peak UV radiation power from a nanosecond discharge in atmospheric argon, spanning UV-C and UV-B ranges, was 8.82 mW/cm². Reducing the argon pressure to 13.3 kPa decreased it to 4.1 mW/cm².

Figure 6 illustrates the ultraviolet spectr emitted by a plasma generated from silver sulfide under atmospheric argon pressure, with electrodes positioned 2 mm apart. Results of the identification of spectral lines in these spectra are shown in Tables 1. Reference [10] was instrumental in identifying the spectral lines observed in the radiation emitted by the discharge plasma.
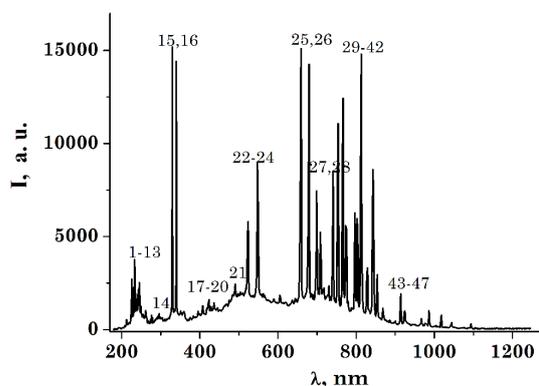

Fig. 6 – UV-radiation spectrum of the discharge plasma between the electrodes from the Ag_2S compound at: p_{Ar} = 101 kPa; f = 1000 Hz, d = 2 mm

The plasma under study exhibits strong emission within the spectral range of 200-340 nm. Emission in the 200-300 nm range primarily originates from singly charged silver ions, while emission in the 300-340 nm range is predominantly from silver atoms. The most prominent spectral lines spanning from 200 to 400 nm correspond to silver atoms, with their ground state serving as the lower energy level.

Upon reducing the frequency to 80 Hz and argon pressure to 13.3 kPa, a notable decrease in the intensity of silver spectral lines was noted. This reduction can be attributed to the diminished concentration of electrode material and its decomposition products within the plasma. Consequently, the spectral characteristics observed in

the discharge between electrodes composed of silver sulfide indicate that the optimal operational conditions for synthesizing surface structures from electrode material are achieved at an argon atmospheric pressure of 101 kPa and a pulse repetition frequency of 1000 Hz.

Table 1 – The results of the identification of the plasma radiation spectrum of an overvoltage nanosecond discharge between silver sulfide electrodes at $p_{Ar} = 101$ kPa and $d = 2$ mm; $f = 1000$ Hz

№	λ_{tab}, nm	I_{exp} a.u.	Object	$E_{low.}$, eV	$E_{up.}$, eV	Lower term	Upper term
1	211.38	340	Ag II	4.85	10.71	$4d^9(^2D_{5/2})5s\ ^2[5/2]_3$	$4d^9(^2D_{5/2})5p\ ^2[5/2]^\circ_3$
2	224.64	1363	Ag II	4.85	10.37	$4d^9(^2D_{5/2})5s\ ^2[5/2]_3$	$4d^9(^2D_{5/2})\ 5p\ ^2[7/2]^\circ_4$
3	227.99	992	Ag II	5.70	11.05	$4d^9(^2D_{3/2})5s\ ^2[3/2]_2$	$4d^9(^2D_{3/2})5p\ ^2[5/2]^\circ_3$
4	232.02	1418	Ag II	5.05	10.36	$4d9(^2D_{5/2})\ 5s2[5/2]_2$	$4d^9(^2D_{5/2})\ 5p\ ^2[3/2]^\circ_1$
5	233.13	2473	Ag II	5.05	10.36	$4d9(^2D_{5/2})\ 5s2[5/2]_2$	$4d^9(^2D_{5/2})\ 5p\ ^2[3/2]^\circ_1$
6	241.13	1005	Ag II	5.42	10.56	$4d^9(^2D_{3/2})5s\ ^2[3/2]_1$	$4d^9(^2D_{5/2})5p\ ^2[5/2]^\circ_2$
7	243.77	1901	Ag II	4.85	9.94	$4d^9(^2D_{5/2})5s\ ^2[5/2]_3$	$4d^9(^2D_{5/2})\ 5p\ ^2[3/2]^\circ_2$
8	244.78	1413	Ag II	5.70	10.77	$4d^9(^2D_{3/2})5s\ ^2[3/2]_2$	$4d^9(^2D_{3/2})5p\ ^2[5/2]^\circ_2$
9	260.59	553	Ag II	10.18	14.94	$4d^9(^2D_{5/2})\ 5p\ ^2[7/2]^\circ_3$	$4d^9(^2D_{5/2})6s\ ^2[5/2]_3$
10	261.43	644	Ag II	10.77	15.51	$4d^9(^2D_{3/2})5p\ ^2[5/2]^\circ_2$	$4d^9(^2D_{3/2})6s\ ^2[3/2]_1$
11	266.04	245	Ag II	12.14	16.78	$4d^85s^2\ ^3F_3$	$4d^8(^3F)5s5p(^3P^\circ)\ ^5G^\circ_2$
12	271.17	276	Ag II	10.37	14.94	$4d^9(^2D_{5/2})\ 5p\ ^2[7/2]^\circ_4$	$4d^9(^2D_{5/2})\ 6s\ 2[5/2]_3$
13	276.75	480	Ag II	5.70	10.18	$4d^9(^2D_{3/2})5s\ ^2[3/2]_2$	$4d^9(^2D_{5/2})5p\ ^2[7/2]^\circ_3$
14	293.83	619	Ag II	10.77	14.99	$4d^9(^2D_{3/2})5p\ ^2[5/2]^\circ_2$	$4d^9(^2D_{5/2})6s\ ^2[5/2]_2$
15	328.06	14166	Ag I	0.00	3.77	$4d^{10}5s\ ^2S_{1/2}$	$4d^{10}5p\ ^2P^\circ_{3/2}$
16	338.28	12136	Ag I	0.00	3.66	$4d^{10}5s\ ^2S_{1/2}$	$4d^{10}5p\ ^2P^\circ_{1/2}$

The oscillograms from the emission of spectral lines from both an atom and a singly charged silver ion, alongside the oscillogram of the current during an overvoltage nanosecond discharge in argon between silver sulfide electrodes, reveal significant insights

The peak radiation intensity of 89 rel. units was observed for the spectral line at 293.83 nm of Ag II, occurring precisely at $t = 80$ ns.

Furthermore, a peak radiation intensity of 52.52 rel. units was recorded for the spectral line at 424.06 nm of Ag II, reaching its maximum at $t = 126$ ns. Additionally, a peak radiation intensity of 63 rel. units was noted for the spectral line at 328.06 nm of Ag I, occurring at $t = 103$ ns. Similarly, a peak radiation intensity of 45 rel. units was observed for the spectral line at 338.28 nm of Ag I, reaching its maximum at $t = 92$ ns.Moreover, the peak radiation intensity of the spectral line at 405.54 nm of Ag II was observed at a level of 47 rel. units, occurring at $t = 125$ ns. Simultaneously, the maximum current of 92 A was reached at $t = 62$ ns.

All the oscillograms of the spectral lines of the silver atom and ion had a pick structure of radiation caused by the pick form of the voltage on the discharge gap (the duration of individual picks is 5-10 ns). The total maximum of the radiation intensity of the silver lines was in the afterglow of the main current maximum ($t = 62$ ns). This may be due to the recombination nature of the radiation of silver atoms and ions in this experiment.

In powerful pulsed discharges of atmospheric pressure with electron density at the level of 10^{15}-10^{17} cm^{-3}, the formation of excited metal ions occurs mainly in the processes of excitation of a singly charged metal ion by electrons, as was typical for copper and aluminum atoms [11].

4. SPECTRA OF RAMAN LIGHT SCATTERING

Thin films composed of argon-silver sulfide plasma were fabricated using the technique outlined in [12, 13]. By positioning a glass substrate approximately 2-3 cm away from the discharge center and operating at pulse frequencies ranging from 500 to 1000 Hz, homogeneous thin films were produced from electrode erosion products under the influence of a nanosecond discharge with overvoltage in heavy inert gases like argon. The inert gas pressure was maintained at 101 kPa throughout the synthesis process, while the films were continually exposed to ultraviolet radiation emitted by the discharge plasma.
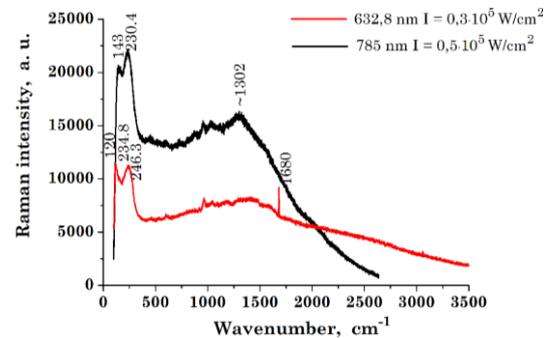

Fig. 7 – Raman scattering spectrum of laser radiation at a wavelength of 785 nm by a thin film synthesized from electrode erosion products of an overvoltage nanosecond discharge between electrodes from the superionic conductor Ag_2S ($p_{Ar} = 101$ kPa)

Figure 7 presents the characteristic spectrum of Raman scattering from a semiconductor laser emitting at a wavelength of 785 nm.

Micro-Raman spectroscopy was employed to probe the synthesized film at both 785 nm and 632.8 nm wavelengths, with the laser beam focusing on a film surface area measuring 1-2 μm in diameter. The semiconductor laser produced an average power of 32 mW at 785 nm and 9.9 mW at 632.8 nm. Raman scattering spectra were identified with reference to sources [14, 15].

Thin films were studied using a Renishaw InVia™ confocal Raman microscope from the UK, with an exposure time of 10 seconds.

The Raman light scattering spectrum of the film (Fig. 7) exhibits distinctive bands spanning from 100 to 300 cm^{-1}, notably at 143 and 230.4 cm^{-1}. The peak at 143 cm^{-1} corresponds to the vibrations of the silver lattice [14], while the broader band between 210 and 250 cm^{-1}, particularly the peak at 230.4 cm^{-1}, arises from Ag_2S nanoparticles, representing symmetric longitudinal vibrational modes of Ag–S–Ag bonds.

Furthermore, a broad band within the range of 1200-1300 cm^{-1} is attributed to silver vibrations, released during the photoinduced decomposition of Ag_2S. Additionally, a broader band ranging from 1500 to 1650 cm^{-1} is observed, associated with the products generated from the photodecomposition of Ag_2S [14].

5. CONCLUSIONS

Investigating the specifications of the plasma generated by an overvoltage nanosecond discharge in argon, occurring between electrodes comprised of the Ag_2S compound, uncovered the following observations:

– at a gap distance of 2 mm between electrodes, a uniformly distributed discharge was observed, taking on a square-like shape with a side length of 2 mm;

– the voltage pulses lasted up to 450-500 ns, with the maximum amplitude of one polarity reaching 22 kV across the discharge gap. Short-lived oscillations within the voltage pulses lasted 5-10 ns. The maximum amplitude of current pulses reached 100 A, resulting in a pulse electric power of 10 MW and a single-pulse energy of 119 mJ;

– under argon pressure of 101 kPa and a pulse repetition frequency of 1000 Hz, the maximum absolute intensity of radiation in the spectral range of 200-315 nm was 8.82 mW/cm^2, and 10.23 mW/cm^2 in the range of 315-400 nm. Lowering the pulse repetition frequency to 80 Hz and argon pressure led to a notable reduction in radiation intensity;

– the ultraviolet radiation spectrum of the discharge predominantly consisted of emissions from singly charged silver ions in the 200-300 nm range and silver atoms in the 300-340 nm range. This holds promise for the development of spot UV lamps utilizing Ag_2S compound vapors, applicable in nanotechnology, medicine, and biology. Maximum UV radiation intensity from silver atoms and ions was also achieved at 101 kPa argon pressure and a pulse frequency of 1000 Hz;

– Raman spectra analysis of laser radiation scattered by thin films synthesized from plasma utilizing the gas-vapor mixture of "argon - silver sulfide" revealed compositions primarily consisting of silver sulfide. This discovery suggests potential applications in various devices leveraging superionic conductivity.

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Синтез поверхневих наноструктур сульфіду срібла в аргоні атмосферного тиску в газовому розряді

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У роботі досліджено характеристики наносекундного перенапруженого розряду в аргоні, що виникає між електродами, виготовленими з суперіонного провідника – сульфіду срібла (Ag_2S). Розряд ініціювався під тиском аргону в діапазоні від 13,3 до 101 кПа з відстанню між полікристалічними електродами Ag_2S 2 мм. Деградація електродного матеріалу в розряді, поряд з проникненням парів Ag_2S в міжелектродний простір, відбувалася внаслідок мікровибухів, спричинених нерівностями поверхні електродів. Така конфігурація розряду має потенціал як плазмохімічний реактор для синтезу тонких плівок на основі сульфіду срібла. Крім того, в роботі представлено результати аналізу спектрів комбінаційного розсіяння лазерного випромінювання, що взаємодіє з плівками, синтезованими на основі сполуки Ag_2S.

Ключові слова: Наносекундний розряд, Сульфід срібла, Аргон, Тонкі плівки, Спектр випромінювання, Спектри комбінаційного розсіювання, Плазма.