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ELECTRON IMPACT EXCITATION AND IONIZATION OF SULFUR, SELENIUM, AND TELLURIUM VAPORS[1]

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Excitation and ionization processes of sulfur, selenium, and tellurium vapors by a low-energy (1–50 eV) electron impact have been investigated. The emission spectra are studied in the wavelength range 200–590 nm. The optical excitation functions for the most intense atomic and ionic spectral lines, as well as for molecular bands and emissions, are measured. The energy dependences of the total cross sections for the formation of positive and negative sulfur and selenium ions by the electron impact are also measured. The ionization energies of sulfur and selenium are determined, and the origin of the features observed in the measured curves is identified. It is found that, in the interval of temperatures, at which the experiments were carried out, the vapors of the researched objects, besides polyatomic molecules, also contain diatomic molecules of the studied elements at high concentrations. It is demonstrated that the atoms in excited states are mainly produced due to the dissociation of diatomic molecules.

Keywords: electron, atom, molecule, excitation, ionization, dissociation, fragmentation.

1. Introduction

Nowadays, a lot of experimental results have been accumulated concerning the elementary collision processes between low-energy electrons and atoms belonging to groups I, II, III, and VIII of the Periodic system. At the same time, there are almost no works devoted to the research of the excitation and ionization processes of group VI elements, in particular, S, Se, and Te. Most likely, this fact may be associated with a complicated character of experiments with the indicated elements that possess rather a high chemical activity. In addition, it is difficult to correctly determine the composition of their vapor. As was shown in works [1–6], depending on the evaporation temperature, the vapors of those elements contain atoms and two- and multiatomic molecules in various ratios.

Sulfur, selenium, and tellurium are widespread elements in the Nature. Moreover, sulfur can be found in the atmosphere of some space objects [7]. Those elements also have an important biological value for a human body. In particular, sulfur is a component of some amino acids (cysteine, methionine), vitamins (biotin, thiamin), and enzymes. In addition, redox reactions with the participation of sulfur are an energy source in chemosynthesis [8]. Selenium in a human body interacts with vitamins, enzymes, and biological membranes; it takes part in the regulation of the metabolism for fats, proteins, and carbohydrates.

Owing to unique properties of the examined elements, they are widely applied in various domains of science and industry. In particular, selenium and tellurium, being characterized by excellent photoelectric and photoconducting properties, are widely used in photo cells and solar batteries, as well as in various semiconductor devices. Sulfur is used in

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[1] The authors dedicate this work to M.G. Nakhodkin with the feeling of profound respect on the occasion of his 90th birthday.

the processes of plasma etching used in the precision treatment of surfaces of various metals.

All the aforesaid, from the viewpoint of fundamental knowledge, determines the urgency and the importance of researches dealing and elementary collision processes between low-energy electrons with the atoms of group VI elements in the gas phase. This work aimed at studying the processes of excitation and ionization of sulfur, selenium, and tellurium vapors by the electron impact in the energy interval from the threshold to 50 eV.

2. Experimental Part

2.1. Optical method

The processes of excitation of sulfur, selenium and tellurium are studied by means of the optical method. We use a high-transmission diffraction monochromator MDR-2 operating in the spectral interval 200–590 nm as a part of an automated experimental set-up. The latter, as well as the measurement technique and the procedure used to calibrate the energy scale of electrons in the exciting beam, was described in work [9] in detail.

An electron beam about 1.5 mm in diameter emitted by an oxide cathode was formed with the help of a four-electrode electron gun. It passed through a vapor-filled collision cell 12 mm in height and 10 mm in diameter and was detected using a deep Faraday cup. The electron current stability after the collision cell was not worse than 3% in the energy range 3–50 eV. The energy spread of electrons in the beam (full width at a half maximum of the differential current-voltage characteristic of the electron current at a collector) was equal to 0.4 and 0.6 eV at currents of 10 and 20 μA, respectively. Vapors of the studied elements were introduced into the collision cell from a separate reservoir loaded with the required substance (about 5 g). The reservoir was resistively heated through a thin-walled tube 4 mm in diameter and 60 mm in length made of stainless steel. This construction allowed the temperatures of the collision cell and the unit of electron-optical system, which had independent resistive heating, to be maintained by 20–30 K higher than the temperature of the tank with the substance. In such a way, we prevented the condensation of vapors of the studied elements on the units indicated above and provided the stability of the whole electron-optical system during long-term experiments. The vacuum chamber was evacuated with the help of an oil-vapor pump (a pumping rate of 500 l/s). The pressure of residual gases in the vacuum chamber did not exceed 10^{-4} Pa at measurements.

Emission was produced by the collisions of electrons with the vapor of the studied element. It was extracted through quartz windows of the collision cell and the vacuum chamber, focused with the use of a two-lens condenser on the input slit of a monochromator MDR-2 (a grating with 1200 lines/mm and an inverse dispersion of 2 nm/mm was used), and detected with the help of a photoelectron multiplier FEU-106. Pulses of single photoelectrons obtained from a photomultiplier were preliminarily amplified, formed by a broadband amplifier-discriminator, and it entered the pulse counter via an interface card in addition, they were accumulated in the memory of a personal computer. Depending on the produced emission intensity of spectral lines or bands, the signal was accumulated with an exposition of 10–60 s at every measurement point in order to ensure the measurement accuracy not worse than 5–10% at the maximum of optical radiation.

Original software programs developed by us to control the experiment allowed the radiation spectra of the analyzed elements to be registered in the automatic regime at fixed energies of bombarding electrons and at a given rotation increment for the drum of a monochromator diffraction grating. They were also capable of measuring the energy dependence of the radiation intensity for the selected spectral lines, bands, or spectral sections, i.e. the optical excitation functions (OEFs). While measuring the OEFs, the step of scanning over the energy of bombarding electrons can be established within the limits from 2.5 meV to 10 eV.

Radiation spectra in the interval 200–590 nm were registered in our experiments with a step varying from 0.548 to 1.2 nm, with a spectral resolution from 1 to 3 nm depending on the spectrum complexity, and at fixed energies of exciting electrons ranging from 8 to 50 eV. The illumination from the electron gun cathode that fell on a photomultiplier through the optical path was subtracted from the total signal by modulating the exciting electron beam. However, the changes in the photomultiplier sensitivity and the transmittance of a monochromator MDR-2 with the variation of the registered emission wavelength were not taken into account.

A special attention was paid to the calibration of the energy scale for the exciting electron beam. We used, in parallel, two techniques: the calibration by a shift of the current-voltage characteristic of the electron current at the electron collector and the calibration by the energy shift of a sharp maximum at an energy of 14.2 eV in the excitation cross-section of a spectral band in the second positive system of a nitrogen molecule (at $\lambda = 337.1$ nm, C3Πu → B3Πg) [10]. While implementing the latter technique, the OEF of this band was measured (the vacuum in the chamber was intentionally worsened by an order of magnitude; as a result, the required concentration of N_2 molecules was created in residual gases). It was the energy difference between the positions of the indicated maximum in the excitation cross-section in the OEFs measured by us and that determined in work [10] that gave us the contact potential difference. This procedure, which was carried out in every experiment, allowed us to calibrate the energy scale of an electron beam with an accuracy of ±50 meV.

2.2. Hypocycloidal electron spectrometer

In order to study the formation processes of negative and positive ions of the analyzed elements in the gas phase, we used a hypocycloidal electron spectrometer (HES) with a vapor-filled cell. A detailed description of the HES design and the principle of its operation was made in work [11].

The gas phases of the examined substances were obtained with the help of an effusive source with resistive heating (330 K for sulfur and 450 K for selenium). The energy spread of electron beam in those experiments was equal to about 0.2 eV. An extracting potential of 1.5 V with respect to the cathode was applied to the ion detector. This value was enough for the complete collection of ions in the collision chamber. In the course of measurements, vacuum not worse than 2×10^{-4} Pa was maintained in the working chamber. The HES was arranged in a homogeneous magnetic field created by a pair of Helmholtz rings 230 mm in diameter. The signal registration and the control over the measurement process were automated with the help of an original software program developed for a personal computer, which allowed the measured dependences to be visualized in the course of the experiment.

3. Discussion of Results

3.1. Excitation

We studied the emission spectra of the vapors of sulfur, selenium, and tellurium in the interval 200–590 nm at their excitation by monoenergetic electrons with fixed energies, E_3. In Fig. 1, the typical emission spectra of sulfur ($E_3 = 8$ eV), selenium ($E_3 = 30$ eV), and tellurium ($E_3 = 10$ eV) vapors are exhibited. The spectra were registered with a wavelength step of 1.1 nm and the spectral resolution $\Delta\lambda = 2$ nm. The temperature of the reservoir with the examined substance was maintained constant in vicinities of 330 (sulfur), 450 (selenium), or 570 K (tellurium) with an accuracy of ±3 K. We performed a series of experiments, in which the temperature of the reservoir was varied within the interval of ±30 K from the indicated values, but the emission spectra remained almost the same.

As one can see from Fig. 1, the registered spectra are rather complicated. However, it should be noted that the spectra of selenium and tellurium vapors are very similar to each other. In those two, the following atomic spectral lines can be observed in the ultra-violet region: at $\lambda = 207.5$ ($4p^{4\,3}P_2$–$4p^3 5s\,^5S_2^0$) and 216.4 nm ($4p^{4\,3}P_1$–$4p^3 5s\,^5S_2^0$) in the selenium radiation spectrum and at $\lambda = 214.2$ ($5s^2 5p^{4\,3}P_2$–$5s^2 5p^3 6s\,^3S_1^0$), 225.9 ($5s^2 5p^{4\,3}P_2$–$5s^2 5p^3 6s\,^5S_2$), 238.6 ($5s^2 5p^{4\,3}P_1$–$5s^2 5p^3 6s\,^3S_1^0$), and 253.0 nm ($5s^2 5p^{4\,3}P_1$–$5s^2 5p^3 6s\,^5S_2$) in the tellurium one. In addition, a broad continuous band in the interval 300–550 nm for selenium and 380–530 nm for tellurium is ob-

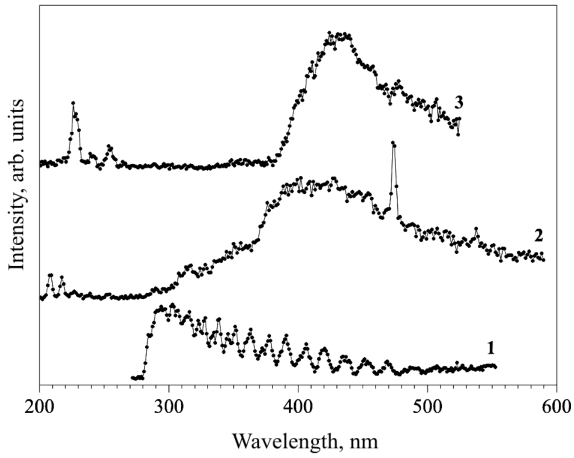

Fig. 1. Emission spectra of S (*1*, $E_3 = 8$ eV), Se (*2*, $E_3 = 30$ eV), and Te (*3*, $E_3 = 10$ eV) vapors

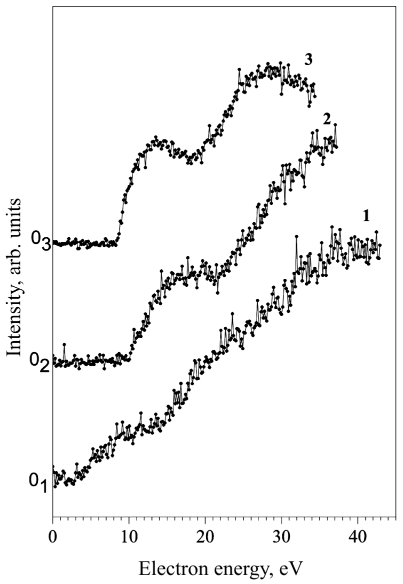

Fig. 2. OEFs of atomic spectral lines: sulfur (*1*, λ = 469.4/.5/.6 nm), selenium (*2*, λ = 207.5 nm), and tellurium (*3*, λ = = 225.9 nm)

served. However, only the radiation spectrum of selenium vapor contains a pronounced atomic spectral line at λ = 473.1/473.9/474.2 nm ($4p^35s^5S$–$4p^36p^5P_{3,2,1}$) located within the interval of the broad band. The emission spectrum of sulfur vapor is quite different. Here, a continuous emission band in the interval 280–480 nm is observed already at an electron excitation energy of 8 eV. This band plays the role of the background for a considerable number of well-distinguished lines. Taking into account that the excitation thresholds for the atomic spectral lines of sulfur in this spectral region exceed 8 eV, we may assert that this spectrum is associated with the excitation of sulfur molecules. Moreover, in view of a large enough distance between separate maxima in the spectrum, we may assume that, most probably, this spectrum is caused by the excitation of S_2 molecules.

Concerning the broad continuous bands at 300–550 nm in the emission spectrum of selenium vapor and at 380–530 nm in tellurium one, we may suppose that they result from the excitation of electron-vibrational states in Se_n ($n = 2\div8$) and Te_n ($n = = 2\div8$), respectively, molecules, because, as was said above, at our experimental temperatures, selenium and tellurium vapors contain Se_n ($n = 2\div8$) and Te_n ($n = 2\div8$), respectively, molecules in various ratios [3–6]. Our assumption is confirmed by a series of experiments dealing with the research of the spectra of radiation emission by tellurium vapor registered at a high spectral resolution ($\Delta\lambda$ = 1 nm) and a small scanning step (0.411 nm) [12].

Now, let us consider the optical excitation functions. In Fig. 2, the OEFs of atomic spectral lines are shown for sulfur (λ = 469.4/.5/.6 nm; $3s^23p^35p^5P_{1,2,3}$–$3s^23p^34s^5S_2^0$), selenium (λ = = 207.5 nm; $4p^{43}P_2$–$4p^35s^5S_2^0$), and tellurium (λ = = 225.9 nm; $6s^25p^45p^3P_2$–$5s^25p^36s^5S_2$). The threshold energies of their excitation equal 9.16, 5.97, and 5.49 eV, respectively. As one can see from Fig. 2, the OEFs of spectral lines at 207.5 and 225.9 nm have a similar character: a sharp excitation threshold at energies of 9.6±0.5 and 8.2±0.5 eV, respectively; a considerable growth of the excitation cross-section with maxima at about 17 and 13 eV, respectively; and the following growth of the excitation cross-section. The values found by us for the excitation thresholds of those lines are shifted with respect to the real spectroscopic energies of excitation of the initial levels by about 3.6 and 2.7 eV, respectively. Therefore, we may assert that the formation of excited states of selenium and tellurium atoms near the thresholds occurs following the scheme

$$\mathrm{M} + e \to \mathrm{A}^* + \mathrm{A} + e.$$

The threshold of this reaction is determined by the excitation energy of the initial level of the spectral line (5.97 and 5.49 eV for selenium and tellurium, respectively), and the molecule dissociation energy (3.1 eV for Se_2 and 2.3 eV for Te_2) [13].

Excited states of sulfur atoms are formed following a scheme similar to that given above. In the OEF of the spectral line at 469.4/.5/.6 nm (Fig. 2), we observe a sharp threshold at an energy of about 4.3 eV. This is the excitation threshold for electron-vibrational levels of sulfur molecules. As a result, we obtain a wide band at 280–480 nm in the emission spectrum of sulfur vapor (Fig. 1). A less sharp threshold in the discussed OEF, which is located at an energy of 13.6 eV, is shifted with respect to the real excitation threshold of this line by about 4.4 eV, i.e. practically by the magnitude of the dissociation energy of S_2 molecule (4.4 eV). Therefore, we obtained the sound experimental confirmation of the fact that, at temperatures used to evaporate the analyzed elements in our experiments, the corresponding vapors contained significant amounts of two-atomic molecules. The excitation of atoms of those elements occurs following the scheme given above. Some features (maxima and breaks) in the OEFs of the spectral lines of sulfur, selenium, and tellurium atoms testify to the presence of additional excitation mechanisms, such as resonance phenomena, cascade transitions from higher energy levels, and the formation and the decay of highly excited molecular ionic states.

In Fig. 3, the OEFs of the spectral lines of single-charged sulfur ions at $\lambda = 393.3$ and 542.8/543.2/545.3 nm are observed. The excitation threshold energies for the indicated lines equal 19.4 and 15.94/15.89/15.86 eV, respectively. The first line is located in the continuous band of the emission spectrum, which is attributed to the excitation of electron-vibrational levels of molecules. Accordingly, in this OEF, as was in the case of the spectral line of atomic sulfur (Fig. 2), a sharp excitation threshold followed by a significant growth in the emission intensity to about 5 eV is observed at an energy of about 4.3 eV, as well as a complicated character of the excitation function and some peculiarities in the form of maxima and breaks at energies of 7.9 and 20 eV. The complicated character of the excitation function and the weakly expressed real excitation threshold for the initial level of the line are also a result of the superposition of this function with the emission of the intense molecular band at $\lambda = 393.8$ nm.

In the OEF of the spectral line at $\lambda = 542.8$/543.2/545.3 nm, which is located at the edge of the continuous spectral component, the “background” is practically absent. Therefore, its excitation threshold at an energy of about 15.8 eV is more pronounced. Above the excitation threshold, a smooth growth of the intensity is observed to an energy of about 28 eV, with no peculiarities in the excitation function being observed.

In Fig. 4, the OEFs for the spectra of molecular emission for sulfur ($\lambda = 376$ nm), selenium ($\lambda = 383$ nm), and tellurium ($\lambda = 430$ nm) are shown. One can see that all emission OEFs demonstrate a sharp excitation threshold at energies of about 4 (sulfur), 3.2 (selenium), or 2.9 eV (tellurium); a sharp growth of excitation cross-sections to an energy of 5–6 eV, where features in the form of a narrow maximum are observed; a further growth of the cross-sections with the maxima observed at energies of about 8 and 10 eV; a small drop; and a further insignificant growth of the excitation cross-sections. The features typical of all OEFs (Fig. 4) testify to the resonance excitation mechanism of sulfur, selenium, and tellurium molecules near the excitation threshold. The behavior of OEFs for those emissions is characteris-

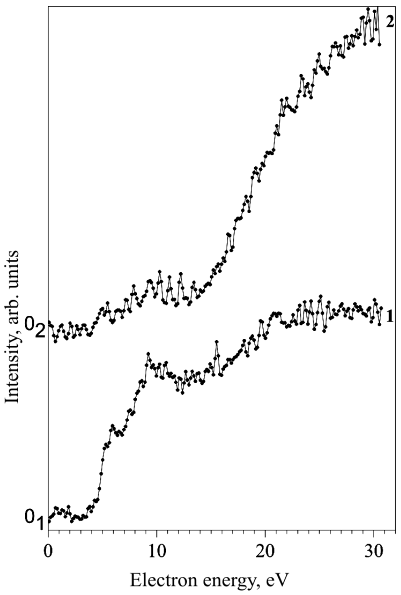

Fig. 3. OEFs of spectral lines of single-charged sulfur ions at $\lambda = 393.3$ (*1*) and 542.8/543.2/545.3 nm (*2*)

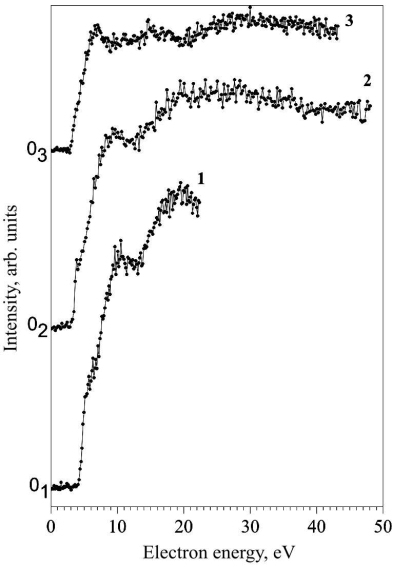

Fig. 4. OEFs of spectral molecular emissions: sulfur (*1*, $\lambda = 376$ nm), selenium (*2*, $\lambda = 383$ nm), and tellurium (*3*, $\lambda = 430$ nm)

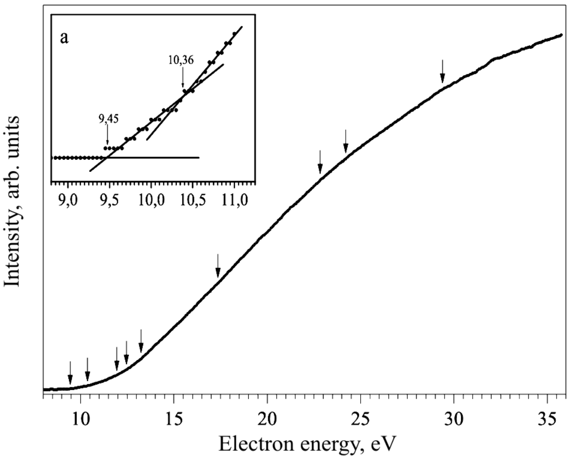

Fig. 5. Energy dependence of the total cross-section for the formation of positive ions in sulfur vapor. An example of the approximation of linear sections is shown in the inset

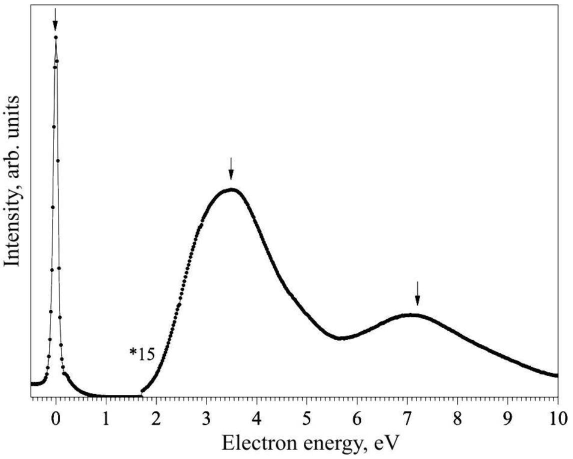

Fig. 6. Energy dependence of the total cross-section for the formation of negative sulfur ions

tic of the excitation of triplet states in a two-atomic molecule [14].

The analysis of the results obtained also testifies that the spectral lines (Fig. 1) are caused by the decay of the excited states of two-atomic molecules. This conclusion is also confirmed, in particular, by the results of mass-spectrometric researches carried out at our laboratory [3], which showed that S_2 molecules dominate in sulfur vapor at temperatures of 300–700 K, and by the control measurements performed within the electron spectroscopy method, when the excitation threshold energy for one of the lower states of a sulfur molecule was found to equal 4 eV. The low excitation thresholds of selenium (3.2 eV at $\lambda = 383$ nm) and tellurium (2.9 eV and the molecular emission at $\lambda = 430$ nm) testify to the excitation of transitions between the ground state and the first excited electron-vibrational level in those molecules.

3.2. Formation of positive and negative ions

We also studied thoroughly the ionization and the dissociative ionization of sulfur, tellurium, and selenium vapors on two experimental techniques: with a monopole mass-spectrometer (the crossed beam method) and with a hypocycloidal electron spectrometer equipped with a vapor-filled cell. In connection with certain difficulties that arose at studying the ionization of tellurium vapor, we discuss only the results obtained for sulfur and selenium.

In work [3], we studied the mass-spectrum of sulfur vapor at a temperature of 450 K and an energy of ionizing electrons of 70 eV. It was found that, at this temperature, the most intense are the peaks produced by the ions of sulfur molecules S_2 ($m/z = 64$) and S_8 ($m/z = 256$) and by the ions of atomic sulfur ($m/z = 32$). This fact testifies that sulfur vapor at a temperature of 450 K mainly consists of molecules, which are formed both as a result of the dissociative ionization and in the course of sulfur evaporation. The influence of the temperature variation on the formation of sulfur ions was also researched. It turned out that the concentrations of sulfur vapor components substantially change as the temperature grows, which was explained by their thermally induced intense decomposition.

In Fig. 5, the energy dependence of the total cross-section of positive sulfur ion formation in the energy interval 8–36 eV is plotted. The analysis of this dependence shows that its slope changes in the form of breaks. With the help of a special procedure consisting in the approximation of linear sections in the measured dependence, we determined the points of their intersections, which gave the energies, at which the new channels of ionization appeared. This procedure is illustrated in the inset in Fig. 5. In such a way, we determined the first ionization potential of sulfur vapor equal to 9.45 eV and the energy positions of detected breaks at 10.36, 11.91, 12.48, 13.23, 17.37, 22.84, 24.20, and 29.40 eV. The first ionization potential of sulfur vapor corresponds to the energy of appearance of an ionized sulfur molecule S_2^+, and the energy position of the break at an energy of 10.36 eV coincides with the energy of appearance of an ionized sulfur atom S^+ [15]. This fact demonstrates that the main components of sulfur vapor are sulfur atoms and sulfur molecules S_2, as was shown in the experiments carried out by the mass-spectrometric method [3]. It is worth noticing that the features at energies of 17.37 and 29.40 eV correspond to the double and triple ionizations, respectively, of the S_2 molecule [16]. A number of features were observed at energies above 31 eV. Their appearance may probably be connected with the excitation and the following decay of the autoionization states of S_2^+ and S^+ ions.

We also studied the positive ionization of selenium in the gas phase by the electron impact [17]. The energy dependence of the total cross-section of positive ion formation in selenium vapor was measured in the energy interval from the threshold value to 16 eV. The ionization threshold energy for selenium vapor was determined to equal 8.05 eV, which corresponds to the energy of appearance of an Se_8^+ ion. In the region near the threshold, we found the breaks at energies of 8.32, 8.87, 9.10, 9.39, and 9.75 eV, which correspond to the energies of appearance of Se_6^+, Se_2^+, Se_4^+, Se_3^+, and Se^+ ions, respectively [18]. Unlike sulfur, in the case of selenium, the molecules with a larger number of atoms dominate, which is explained by the temperature dependence of the vapor composition. As was shown in mass-spectrometric researches with sulfur, the temperature change strongly affects the vapor composition of those elements. It is probable that the same situation takes place in the course of selenium evaporation.

In order to obtain a more complete picture for the formation of ions in sulfur and selenium vapors, the energy dependences of the total cross-sections of their negative ion formation at the interaction with electrons in the energy interval 0–10 eV were studied. In Fig. 6, the corresponding dependence for sulfur ions is plotted. Three maxima are observed at energies of 0, 3.5, and 7.2 eV. The intensity of the first maximum is an order of magnitude higher than those of the second and third maxima. In addition, the energy width of the first maximum (about 0.1 eV) coincides with the electron energy spread in the beam, which evidences the resonant attachment of low-energy electrons to sulfur molecules. Using the results of work [19], it was found that the largest contribution of the negative sulfur ion formation to the total cross-section was made by S_2^-, S_3^-, and S_4^- ions. A substantial width of the maxima at 3.5 and 7.2 eV can be explained by the simultaneous formation of negative ions of various sulfur molecules with the energies of appearance close to one another.

In the case of selenium, we studied the processes of formation of its negative ions as well. In the corresponding dependence, as was in the sulfur case, four maxima were found at energies of about 0, 2.17, 3.55, and 4. 75 eV [20]. Owing to the absence of any data concerning the negative ions of selenium and the difficulties in the determination of vapor composition, their identification will be done later.

4. Conclusions

With the help of the optical spectroscopy method and with the use of a vapor-filled cell, the processes of sulfur, selenium, and tellurium excitation in the gas phase by the electron impact are studied. The emission spectra of the vapors in the interval 200–590 nm at the fixed energies of exciting electrons in the interval 8–50 eV are analyzed. Two-atomic molecules of the corresponding elements are shown to prevail in their vapors at experimental temperatures.

The OEFs of the spectral lines of S, Se, and Te atoms and ions, the molecular bands and emissions of the vapors of those elements in the energy interval 2–50 eV are studied. The positions of the excitation thresholds and the features in the measured OEFs are determined with a high accuracy, which enables us to establish the mechanisms of excitation of atomic spectral transitions. It is found that the broad bands in the radiation spectra emitted by sulfur, selenium, and tellurium vapors are composed by a number of narrow lines, so that those bands are a result of the superposition of radiation from the excited electron-vibrational states of molecules.

The processes of formation of negative and positive sulfur and selenium ions at their interaction with low-energy electrons are studied. The first ionization potentials for sulfur and selenium vapors are found to equal 9.45 and 8.05 eV, respectively. The energy of triple ionization of an S_2 molecule (29.40 eV) is determined experimentally for the first time. It is found that, in the case of sulfur vapor, the negative ions of molecules S_2, S_3, and S_4 are formed with the highest probability owing to the resonant electron attachment to them.

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ЕЛЕКТРОННЕ ЗБУДЖЕННЯ ТА ІОНІЗАЦІЯ ПАРІВ СІРКИ, СЕЛЕНУ, ТЕЛУРУ

Резюме

Досліджено процеси збудження та іонізації парів сірки, селену, телуру при зіткненнях з електронами низьких енергій (1–50 еВ). Вивчено спектри випромінювання в діапазоні 200–590 нм, а також оптичні функції збудження найбільш інтенсивних атомних і іонних спектральних ліній та молекулярних смуг і емісій. Виміряно також енергетичні залежності повних перерізів утворення негативних і позитивних іонів сірки та селену при їх взаємодії з повільними електронами. Визначено енергії іонізації сірки і селену та ідентифіковано природу особливостей, які були виявлені на виміряних кривих іонізації. Встановлено, що при температурах проведення експериментів, у парах крім багатоатомних молекул у значних концентраціях присутні двоатомні молекули досліджених елементів. Показано, що атоми у збудженому стані утворюються, переважно, в процесі дисоціації двоатомних молекул.