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# Study of Chemical Bonds and Element Composition of Silicon Oxycarbonitride Films by the Methods of XP-, IR-, and Energy-Dispersive Spectroscopy

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**Abstract**—The element composition and chemical bonds of nanocomposite films of hydrogenated silicon oxycarbonitride fabricated through high-frequency plasma-chemical deposition from initial gas mixtures of 1,1,3,3-tetramethyldisilazane with nitrogen and oxygen in the temperature range 373–973 K depending on the synthesis conditions is studied. The effect of changes in the temperature and chemical composition of the initial gas mixtures on the element composition and types of chemical bonds in $SiC_xN_yO_z$:H films is investigated.

**Keywords:** hydrogenated silicon oxycarbonitride, plasma-chemical synthesis, 1,1,3,3-tetramethyldisilazane, XPS, EDS

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## INTRODUCTION

Due to the development of oxygen-free dielectrics and solid protective and anticorrosion coatings, radiofrequency plasma-chemical deposition is used to form thin films of silicon carbonitride ($SiC_xN_y$) using organosilicon compounds with gases such as $H_2$, He, $N_2$, and $NH_3$ [1–5]. The properties of these films were comprehensively characterized through the application of a complex of analytical methods. Here, it was demonstrated that, in spite of their set of exceptional properties, $SiC_xN_y$ films contained impurities of the graphite-like carbon, which results in the degradation of the optic parameters and a reduction of the band gap, thus making them unsuitable for extensive application in optoelectronics and solar cells [6–10].

On the addition of $H_2$, $N_2$, and $N_2O$ gases to organosilicon substances, the optical properties of films are known to improve due to the decrease of the carbon content in them and their chemical composition changing into $SiC_xN_yO_z$:H [11–14].

To fabricate high-transparency $SiC_xN_yO_z$:H films in a broad spectral range from 180 to 3200 nm, we applied high-frequency plasma-chemical deposition from the gas phase using a mixture of 1,1,3,3-tetramethyldisilazane with oxygen and nitrogen, and extra thermal treatment in the temperature range 373–973 K [15].

This work is devoted to studying the chemical composition and the nature of the chemical bonds in compounds of variable compositions, such as silicon oxycarbonitride. To obtain qualitative and quantitative information on the films under study, a set of spectroscopic methods was used: FT IR, X-ray photoelectron, and X-ray energy-dispersive spectroscopy.

Besides, the effects of the temperature changes and chemical composition of the initial gas mixtures on the element composition and chemical bond types in $SiC_xN_yO_z$:H films were investigated.

## EXPERIMENTAL

In accordance with [15], thin $SiC_xN_yO_z$:H films were synthesized on substrates heated at 373–973 K in a quartz reactor of the tunnel type with the zones of plasma excitation and film growth separated over its length. Here, the inductive method of plasma excitation using an Ekran-2 high-frequency generator (frequency 40.68 MHz) was used. The input power of the high-frequency plasma was at a constant 50 W. Plates of monocrystalline silicon of (100) orientation and germanium of (111) orientation that underwent standard chemical treatment [8] were used as substrates. Flows of gaseous 1,1,3,3-tetramethyldisilazane (TMDS), nitrogen, and oxygen were fed into the reactor at a rate of 0–1 $cm^3$/min, which provided a constant working pressure in the reactor of $6 \times 10^{-2}$ Torr.

Three initial gas mixtures of TMDS with $N_2$ and $O_2$ were used. The first one was of the chemical composition TMDS + $O_2$ + $N_2$ with the following reagent ratios: $P_{O_2}/P_{TMDS} = 0.28$, $P_{N_2}/P_{O_2} = 1.2$, and $P_{N_2}/P_{TMDS} = 0.35$; the second one was of the chemical composition TMDS + $O_2$ + $3N_2$ with the following reagent ratios: $P_{O_2}/P_{TMDS} = 0.25$, $P_{N_2}/P_{O_2} = 3.0$, and $P_{N_2}/P_{TMDS} = 0.75$; and the third one was of the chemical composition TMDS + $O_2$ + $4N_2$ with the following reagent ratios: $P_{O_2}/P_{TMDS} = 0.21$, $P_{N_2}/P_{O_2} = 4.08$, and $P_{N_2}/P_{TMDS} = 0.82$.

## METHODS OF STUDY

To identify the chemical composition and chemical bonds types, the following methods were used: X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectroscopy (FTIR), and X-ray energy-dispersive spectroscopy (EDS). These methods enable us to reveal specific functional groups from the characteristic vibration frequencies, making it possible to obtain information on the chemical bonding.

The thickness of the $SiC_xN_yO_z$:H films was determined from the ellipsometry measurements using the LEF-2 and LEF-3 devices with additional measurements using an SF-18 spectrophotometer ($\lambda$ = 400–750 nm). The films were studied by the method of IR spectroscopy using an FTIR Shimadzu 8300 spectrometer (300–6000 $cm^{-1}$, resolution 2 $cm^{-1}$).

The element composition of the silicon oxycarbonitride films was studied by the method of X-ray energy-dispersive spectroscopy (EDS) using an EX-23000BU accessory to a JSM-6700F microscope with a resolution of 0.18 nm.

The chemical composition of the film surface was studied using a SPECS Phoibos 150 X-ray photoelectron spectrometer with monochromatized Al$K_\alpha$ radiation ($h\nu$ = 1486.74 eV). The transmission energy of the electron analyzer was 20 eV. To remove the surface contaminants, ionic etching by a beam of $Ar^+$ ions with the energy of 1 keV for 1 min at an angle of 45° was used.

## RESULTS AND DISCUSSION

**XPS method.** The XPS method enables us to register the energy position of the atomic core and valence levels determined by the properties of the external valence bonds. The changes in the configuration of the valence bond (hybridization, length, and spatial distribution of the charge density) are related to the surrounding of atoms yield shifts in the energies of the lower atom levels. The important features of the XPS method include their high surface sensitivity related to the small escape depth of the signal electrons (just some interatomic distances), their rather insignificant dependence on matrix effects, and the possibility of obtaining concentration profiles in thin films and at the boundaries.

The features of the method mentioned above create certain difficulties in preparing the studied samples, since it is necessary to exclude the uncontrolled chemical impact on the surface (surface contamination through adsorption of foreign atoms, oxidation, etc.) emerging as a result of the contact with the atmosphere. The use of ionic bombardment could result in the preferential sputtering of individual sample components and the disruption of the chemical bonds (decrease of the concentration of the nitrogen chemical bonds in the SiCN samples), which requires taking into consideration these factors in the study process [16, 17].

In the case of studying nonconducting (dielectric) samples (e.g., layers of silicon oxycarbonitride), there emerges the problem of surface charging under the effect of the primary probing radiation yielding uncontrolled shifts of the photoelectron lines over the energy scale. In this case, we have to correct the energy scale on the external standard, for which purpose the C1*s* peak with the binding energy of 284.5 eV from the surface carbon is conventionally used.

The samples of hydrogenated silicon oxycarbonitride grown on silicon substrates of the (100) orientation by plasma-chemical decomposition of the systems below were studied by the XPS method: initial gas mixture TMDS + $O_2$ + $4N_2$ at 373, 773, and 973 K, and TMDS + $O_2$ + $N_2$ and TMDS + $O_2$ + $3N_2$ mixtures at 773 K. The Si, C, N, and O elements were determined on the surface of the $SiC_xN_yO_z$:H films by the XPS method, and information on the chemical state of silicon atoms was obtained. Table 1 shows the relative contents of the elements in the films of hydrogenated silicon oxycarbonitride. We can observe a dramatic decrease of the carbon and oxygen concentrations and, in contrast, an increase of the concentration of silicon and nitrogen, along with an increase of the synthesis temperature from 373 to 973 K. To obtain the information on the chemical state of the atoms in $SiC_xN_yO_z$:H films, the spectra (Fig. 1) were deconvoluted into symmetric components using the Gaussian and Lorentzian forms to identify the chemical bonds.

The spectra of the Si2*p* level are deconvoluted into 3 components with energies at 103.0–103.3, 102.0–102.7, and 101.0–101.6 eV corresponding to Si–O, Si–N, and Si–C–N bonds, respectively. In the spectra of the C1*s* level, we observe 4 components at 283.8, 284.5, 285.5–286.5, and 287.8–288.2 eV corresponding to C—Si, C—C and C—H, C—N($sp^2$), and C—O bonds, respectively. The spectra of the N1*s* level are deconvoluted into components at ~398.0–398.5 and 398.8–400.3 eV from N–Si and N–C($sp^2$) bonds, respectively [18, 19]. Finally, in the O1*s* spectra of all the fabricated films, we observe the only component at 532.4 eV corresponding to the O–Si bonds. The

**Table 1.** Dependence of chemical composition of $SiC_xN_yO_z$:H on ratio of components in initial gas mixtures and synthesis temperature measured by methods of XPS and X-ray energy-dispersive spectroscopy (at %)

| Synthesis conditions | Elements atomic concentrations in $SiC_xN_yO_z$:H films determined by XPS method | | | | Elements atomic concentrations in $SiC_xN_yO_z$:H films determined by EDS method | | | |
|---|---|---|---|---|---|---|---|---|
| | Si | C | N | O | Si | C | N | O |
| TMDS + $O_2$ + $4N_2$, 373 K | 28 | 18 | 4 | 50 | 15.3 | 28.5 | 8.6 | 47.6 |
| TMDS + $O_2$ + $4N_2$, 773 K | 39 | 5 | 29 | 27 | 32.2 | 3.4 | 49.2 | 15.8 |
| TMDS + $O_2$ + $4N_2$, 973 K | 41 | 6 | 31 | 22 | 34.9 | 3.3 | 46.5 | 15.3 |
| TMDS + $O_2$ + $3N_2$, 773 K | 39 | 6 | 21 | 35 | 29.0 | 4.4 | 41.1 | 25.2 |
| TMDS + $O_2$ + $N_2$, 773 K | 34 | 8 | 10 | 48 | 27.5 | 29.2 | 27.6 | 3.0 |

**Table 2.** Position (in eV) and contribution of different components to XPS spectra (%, in parentheses)

| Synthesis conditions for $SiC_xN_yO_z$:H samples | Si–O | Si–N | Si–C–N | N–Si | N–C | C–C | C–Si | C–N |
|---|---|---|---|---|---|---|---|---|
| TMDS + $O_2$ + $4N_2$, 373 K | 103.0 (87) | 102.0 (13) | – | 398.5 (75) | 400.3 (25) | 284.5 (83) | – | 286.5 (13) |
| TMDS + $O_2$ + $4N_2$, 773 K | 103.0 (33) | 102.3 (64) | 101.1 (3) | 398.1 (93) | 399.3 (7) | 284.4 (55) | 283.8 (7) | 285.5 (30) |
| TMDS + $O_2$ + $4N_2$, 973 K | 103.0 (27) | 102.3 (71) | 101.1 (2) | 398.0 (90) | 398.8 (10) | 284.4 (54) | 283.8 (10) | 285.6 (29) |
| TMDS + $O_2$ + $3N_2$, 773 K | 103.0 (44) | 102.4 (53) | 101.3 (3) | 398.2 (92) | 399.2 (8) | 284.4 (56) | 283.8 (10) | 285.8 (25) |
| TMDS + $O_2$ + $N_2$, 773 K | 103.3 (69) | 102.7 (27) | 101.6 (4) | 398.2 (95) | 399.2 (5) | 284.4 (59) | 283.7 (15) | 285.5 (19) |

parameters of components are shown in Table 2. We can see that, along with the increase of the synthesis temperature, the contribution of the Si atoms bonded to oxygen decreases by more than a factor of three, while that of Si atoms bonded to nitrogen increases more than fivefold in accordance with the changes in the oxygen and nitrogen concentrations in the films. At 773 and 973 K, we observe the emergence of an insignificant amount (~2 at %) of Si atoms bonded to carbon.

At the temperature increasing from 373 to 773 K, the binding energy of the Si2*p* level from Si–O bonds remains constant, whereas in the case of Si–N bonds, it increases from 102 to 102.3 eV. In the N1*s* spectra, at the temperature increasing from 373 to 973 K, the contribution of the component related to the formation of the N–Si bond increases from 75 to 94%—here, the position shifts to lower binding energies from 398.5 to 398.0 eV. As reported in [19], at the formation of Si–N–C bonds, we observed the increase of the N1*s* energy in comparison with that for silicon nitride because of the higher electronegativity of the carbon atoms in comparison to that of the silicon atoms. Thus, the increase of the binding energy of the Si–N

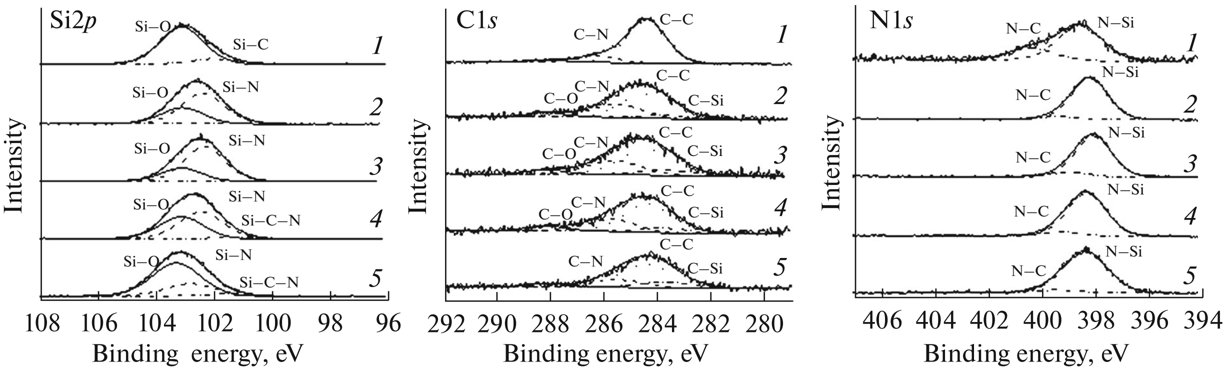

**Fig. 1.** Deconvolution of Si2*p*, C1*s*, and N1*s* XPS spectra obtained for $SiC_xN_yO_z$:H films grown by plasma-chemical decomposition of initial TMDS mixtures at different temperatures into components: (*1*) TMDS + $O_2$ + $4N_2$, 373 K; (*2*) TMDS + $O_2$ + $4N_2$, 773 K; (*3*) TMDS + $O_2$ + $4N_2$, 973 K; (*4*) TMDS + $O_2$ + $3N_2$, 773 K; (*5*) TMDS + $O_2$ + $N_2$, 773 K.

component on the silicon atom and its decrease on the nitrogen atom can be related to the decrease of the concentration of carbon atoms. Besides, for the sample synthesized at the lowest temperature (373 K) having the highest carbon content, the N1*s* spectrum contains the component at 400.3 eV, which originated from the N–C($sp^2$) bond. The spectra of the other samples contain a small component at 399.0–399.3 eV from the N–C($sp^2$) bond. The width of the lines of the N1*s* level decreases from 1.9 to 1.6 eV, which points to the increasingly ordered surrounding of the nitrogen atoms on the increase of the nitrogen concentration in the gas mixture. In the C1*s* spectra at the synthesis temperature, the main contribution (~80%) is provided by the component from the C–C bond, while it is approximately halved along with the increase of the synthesis temperature. However, at the increase of the synthesis temperature, the contribution of the C($sp^2$)–N component approximately doubles, and there emerges a new component from the C–Si bond at 283.8 eV, whose intensity increases along with the temperature increase. It is worth mentioning the significant decrease of the binding energy of the C1*s* level of the C($sp^2$)–N component, along with the temperature increase. Similarly to the N1*s* lines, such behavior of the C($sp^2$)–N component is related to the formation of Si–N–C bonds.

At the increase of the nitrogen content in the gas mixture from TMDS + $O_2$ + $N_2$ to TMDS + $O_2$ + $4N_2$ at 773 K, its concentration in $SiC_xN_yO_z$ films increases, while that of oxygen decreases. The deconvolution of spectra into components shows that the increase of the nitrogen content in the used mixtures of the TMDS + $O_2$ + $xN_2$ compositions leads to a decrease of the number of Si–O (or Si–O–C) bonds and a increase of the content of the Si–N bonds in high-temperature films. At the increase of the nitrogen concentration in the gas mixture, the binding energies of the components decrease: Si–O, from 103.3 to 103.0 eV; and Si–N, from 102.7 to 102.3 eV. The width of the Si2*p* lines also decreases from 2 to 1.6 eV, which points to the equalization of the charge state of the silicon atoms due to the formation of a more homogeneous surrounding. The charge state of the carbon, oxygen, and nitrogen atoms remains virtually constant at changes in the compositions of the gas mixture.

**FTIR spectroscopy.** The FTIR spectroscopy method was used to study the nature of the chemical bonds of $SiC_xN_yO_z$:H films fabricated through the plasma-chemical decomposition of mixtures of a general composition of TMDS + $O_2$ + $xN_2$. For comparison, all the IR spectra were normalized on the film thickness. The obtained spectra absorption bands were assigned to specific chemical bond vibrations using the available published data [20–25].

Figure 2 shows the typical IR spectra of the same five $SiC_xN_yO_z$:H films grown from the three initial gas mixtures at different synthesis temperatures: TMDS + $O_2$ + $N_2$, TMDS + $O_2$ + $3N_2$, and TMDS + $O_2$ + $4N_2$. All the IR spectra of the $SiC_xN_yO_z$:H films have the main absorption band, whose width increases (from ~614 to ~701 $cm^{-1}$) with an increase in the synthesis temperature and decreases (from ~701 to ~689 $cm^{-1}$) with the increase of the nitrogen content in the initial gas mixture. The main absorption bands were deconvoluted into several components using the Gaussian function (for deconvolution curves). The main band of the IR spectra of the low-temperature films comprises the superposition of the contributions from the valence vibrations of the Si–C (800 $cm^{-1}$), Si–N (950 $cm^{-1}$), Si–O (1035 $cm^{-1}$), and Si–O–C (1145 $cm^{-1}$) bonds. Besides, the spectra include bonds corresponding to hydrogen-containing bonds, namely, bending vibrations of the Si—$CH_3$ bond (~1250 $cm^{-1}$), bending vibrations of the C—H bond (~1345 and 1463 $cm^{-1}$), stretching vibrations of the Si—H bond (~2200 $cm^{-1}$), characteristic stretching vibrations of the C—H bond (~2900 and ~2960 $cm^{-1}$) in the $CH_3$ group, and stretching vibrations of the N–H bonds (~3390 $cm^{-1}$). There emerges a characteristic peak at 1550–1600 $cm^{-1}$ corresponding to the skeletal stretching vibrations of C—C bonds of the aromatic ring.

The main absorption band of the high-temperature $SiC_xN_yO_z$:H films synthesized at 773 K (Figs. 2a–2c) from the three initial gas mixtures (TMDS + $O_2$ + $N_2$, TMDS + $O_2$ + $3N_2$, and TMDS + $O_2$ + $4N_2$) consists of three components attributed to the Si–N, Si–C–N, and Si—O bonds. The increase of the nitrogen amount in the initial mixtures changes ratios between these bonds. In the first case, the Si–N and Si–C–N bonds are equal, in the second and the third case, the Si—N bonds are predominant, while in the last case, the concentration of the Si—O bond decreases dramatically. Thus, the chemical composition of films at very high temperatures (above 773 K) is always similar to that of silicon oxynitride. The high-temperature $SiC_xN_yO_z$:H films, unlike those of silicon carbonitride ($SiC_xN_y$), contain small numbers of hydrogen-containing bonds, mainly Si–H and N–H.

**X-ray energy-dispersive spectoscopy.** The element composition of $SiC_xN_yO_z$:H films was studied by the method of energy-dispersive spectroscopy using a JSM-6700F scanning electron microscope equipped with an EX-23000BU accessory. The device enables us to analyze elements from beryllium to uranium with a locality of a few cubic micrometers. Earlier [26], the effect of the thickness of the silicon carbonitride, substrate material, and electron beam energy on the energy-dispersive analysis results was investigated. The EDS method is characterized with some extra complications when applied to the analysis of hydrogenated silicon oxycarbonitride films. First of all, all

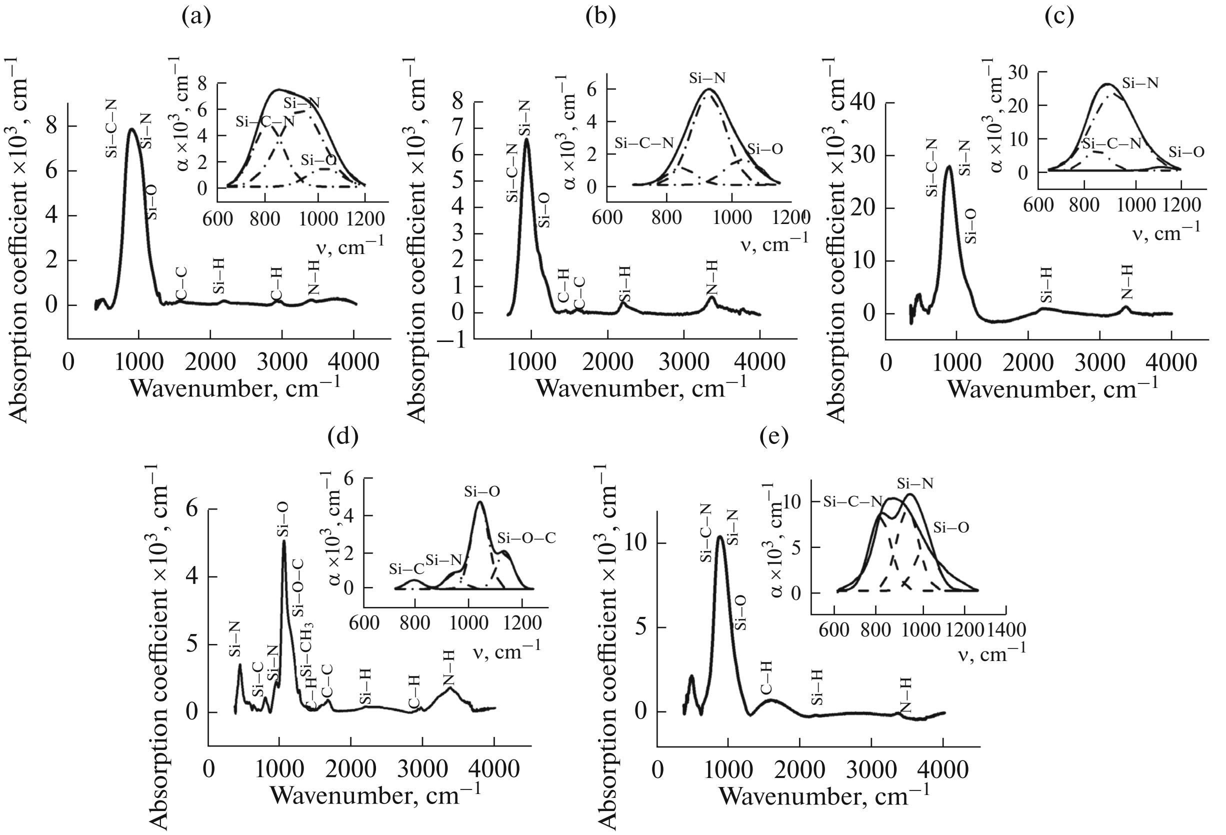

**Fig. 2.** Dependence of IR spectra of $SiC_xN_yO_z$:H films grown by plasma-chemical deposition on changes in chemical composition of initial gas mixtures and synthesis temperatures. Synthesis conditions (gas mixture and temperature $T$, K): TMDS + $O_2$ + $N_2$, 773 (a); TMDS + $O_2$ + $3N_2$, 773 (b); TMDS + $O_2$ + $4N_2$, 773 (c); TMDS + $O_2$ + $4N_2$, 373 (d); TMDS + $O_2$ + $4N_2$, 973 (e). Inserts contain deconvolution of main IR peak into components.

the elements to be determined are light and similar to each other with respect to the atomic number. Hydrogen cannot be determined at all by this method; however, since its concentration in films grown at the intermediate and high temperatures from mixtures with 1,1,3,3-tetramethyldisilazane is low [15], we can compare the data of the element composition over the whole temperature range. The concentrations of the Si, C, N, and O elements were measured at the electron energy of 10 keV, which ensures the reliable excitation of the characteristic radiation of all the elements under study. A licensed program is provided in the device for processing the X-ray spectra. Besides, to increase the reliability of the results, germanium with the (111) orientation was used as the substrate material, as its atoms are not contained in $SiC_xN_yO_z$:H films.

Table 1 shows atomic concentrations of the main elements in $SiC_xN_yO_z$:H films synthesized from different initial gas mixtures of a general composition TMDS + $O_2$ + $xN_2$ on germanium substrates in the temperature range 373–973 K. The results were obtained by the EDS method and are of a semiquantitative character. From the table, we can see a substantial difference in the element composition of the $SiC_xN_yO_z$:H films fabricated at different temperatures from TMDS mixtures with nitrogen and oxygen. The high-temperature films grown from the TMDS + $O_2$ + $N_2$ gas mixture are characterized with the lowest oxygen content and the highest carbon content in comparison to those grown from gas mixtures with the increased nitrogen content. The relatively high carbon content in them is corroborated by the Raman spectroscopy method—the respective spectra contain the $D$ (1360 cm$^{-1}$) and $G$ (1540 cm$^{-1}$) modes attributed to the presence of graphite-like carbon in the mixture [28]. The use of TMDS + $O_2$ + $3N_2$ and TMDS + $O_2$ + $4N_2$ gas mixtures yields the formation of $SiC_xN_yO_z$:H films with the increased nitrogen and silicon contents and very low carbon content, which is corroborated by the absence of Raman scattering [27]. Thus, the chemical composition of films, which is similar to silicon oxynitride, is realized in the high-temperature range of the synthesis.

## CONCLUSIONS

As was found on analyzing the dependence of the element composition of $SiC_xN_yO_z$:H films on the conditions of the synthesis, the used XPS and EDS methods provide the same qualitative changes in the atomic contents of Si, C, N, and O in films, namely, along with the increase of the nitrogen content in the initial TMDS + $O_2$ + $xN_2$ gas mixtures and the increase of the synthesis temperature, the atomic contents of nitrogen and silicon increase, while the concentrations of carbon and oxygen decrease. However, the exact values are unequal, which can be justified to a certain extent. Since XPS is a method for surface studies and the depth of penetration of the X-ray beam is ~4 nm, then, in spite of the preliminary surface etching by an argon beam, it still contains a higher oxygen concentration. In addition, the interaction of the argon beam with the nitrogen-containing bonds yields their partial destruction and removal from the films, which decreases the atomic concentration of the nitrogen found by the XPS method.

It has been determined by the FTIR and XPS spectroscopy methods that the high-temperature films contain mainly Si–N, Si–C–N, and Si–O bonds with a predominance of Si–N bonds; i.e., high-temperature films are similar to silicon oxynitride in chemical composition.

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