Structural Characterization of CdSe-doped Sol-gel Silicophosphate Films

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SiO_2-P_2O_5 films doped with CdSe have been prepared and investigated for temperature sensors applications. The films have been synthesized by sol-gel method, spin coating technique, the deposition being made on glass substrates. The sols have been prepared from tetraethylorthosilicate (TEOS) and phosphoric acid as precursors for SiO_2 and P_2O_5, respectively, together with ethanol and water as reaction environment and a hydrolysis reagent, respectively. The films have been deposited at three rotation rates: 1000 rpm, 2000 rpm and 3000 rpm, starting on the first day after the sol preparation moment and going on, four days consecutively, and then on the eighth day. We have analyzed the influence of the rotation rate and the time elapsed since the sol preparation till the deposition moment on the structural properties of the doped films. The films deposited at each rotation rate have been annealed at 200°C, 300°C, 350°C, 400°C, and 550°C in order to investigate the structural modifications noticed during the formation process of silicophosphate network.

Keywords: FTIR spectroscopy, Raman spectroscopy, sol-gel method, CdSe.

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INTRODUCTION

CdSe quantum dots are extensively investigated due to their quantum confinement effect and size-dependent photoemission characteristics [1, 2].

The cadmium selenide films prepared by the sol-gel method were studied due to the their good chemical and physical properties, that results in their successful application in many areas, for instance, in gas sensors, photo-detectors, light amplifiers, laser micro cavities, solar cell, optoelectronic devices, temperature sensors, etc [3–5].

Starting from a solution, a further evolution into a gel requires successive reactions, which would ensure bonds between particles in a controlled way, similarly to the hydrolysis/condensation reactions in the silica polymerization.

The sol-gel process and the colloidal chemistry offer good opportunities for the synthesis of transparent materials embedding semiconductor nanocrystals. The work in this field has been carried out on group II-VI semiconductors, especially on CdSe for which the quantum size effect is well understood and the colloidal synthesis well controlled [6–8].

The sol-gel method offers certain advantages in comparison to classical solid-state synthetic routes, such as mild synthetic conditions, versatility, high homogeneity and purity of the final material, which are fundamental for the development of new advanced materials [9].

In the present paper, structural properties of CdSe-doped silicophosphate bulk gel material and films were investigated. The influence of the rotation rate and the time elapsed since the sol preparation till the deposition moment as well as the structural modifications that occurred after annealing at different temperatures were studied.

EXPERIMENTAL

Thin films with CdSe nanoparticles embedded in silicophosphate matrix were prepared by the sol-gel method, spin coating technique. As reagents we used tetraethylorthosilicate [$(C_2H_5O)_4Si$, 98 wt. %], phosphoric acid [H_3PO_4, 85 wt. %], distilled water, ethanol [C_2H_5OH, 98 wt. %] as reaction environment, and cadmium selenide [CdSe, 1 wt. %] as dopant powder. The molar ratio of precursors were TEOS/H_3PO_4 = 5/1; TEOS/C_2H_5OH = 1/2; TEOS/H_2O = 1/5. All reagents were used without any previous purification. Phosphoric acid was added to water and, separately, tetraethylorthosilicate [$(C_2H_5O)_4Si$ was mixed with ethanol, then the two resulted solution were mixed together. After checking the pH of the solution, CdSe was added followed by mechanical stirring to improve the homogeneity of the precursor mixture [10, 12].

In the following, some chemical reactions of hydrolysis and condensation of the reagents are presented:

$$(C_2H_5O)_4Si + H_2O \rightarrow SiOH\,(OC_2H_5)_3 + C_2H_5OH$$

$$SiOH(OC_2H_5)_3 + H_2O \rightarrow \\ Si(OH)_2(OC_2H_5)_2 + C_2H_5OH$$

$$Si(OH)_2(OC_2H_5)_2 + H_2O \rightarrow \\ Si(OH)_3(OC_2H_5) + C_2H_5OH$$

$$Si(OH)_3(OC_2H_5) + H_2O \rightarrow Si(OH)_4 + C_2H_5OH$$

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$$SiOH(OC_2H_5)_3 + SiOH(OC_2H_5)_3 \rightarrow$$
$$(OC_2H_5)_3Si\text{-}O\text{-}Si(OC_2H_5)_3 + H_2O$$
$$(C_2H_5O)_4Si + (OH)_3OP \rightarrow$$
$$(OC_2H_5)_3Si\ OPO(OH)_2 + C_2H_5OH$$

The CdSe – doped silicofosphate solution was kept at room temperature, the deposition being made on clean glass substrates, at three rotation rates: 1000 rpm, 2000 rpm and 3000 rpm, starting since the first day after the sol preparation moment and going on four days consecutively and also on the eighth day. The gelification occurred on the ninth day. The layers were deposited, dried at room temperature in air and then thermally treated at different temperatures: 200°C, 300°C, 350°C, 400°C, 550°C, for 2 h.

The deposited films were characterized by Fourier Transform Infrared (FTIR) and Raman spectroscopy, fast, non-destructive and reproducible techniques which allow detecting and quantifying the changes that occur in the structure of those films.

The FTIR spectra were recorded with a Perkin Elmer Spectrophotometer-Spectrum 100, provided with a UATR accessory (Universal Attenuated Total Reflectance) in the range 550–4000 cm^{-1}. The measurement error was ± 0.1% and the number of scans 32.

Raman spectra were collected by means of the LabRAM HR 800 UV–VIS-NIR Horiba Jobin-Yvon system at room temperature. The samples were excited with the 514.5 nm line of an ion laser, focused on the surface sample with a confocal microscope, using an objective magnification X100, 1 μm^2 laser spot size, laser power on the surface sample 2.6 mW, 0.5 to 1 cm^{-1} resolution with a 1800g/mm grating.

The X-ray diffraction (XRD) spectra were collected from 10° up to 70° (2θ) at room temperature, with a step size of 0.02° and a counting rate of 2s/step in a Brucker-AXS type D8 ADVANCE diffractometer.

RESULTS AND DISCUSSIONS

The evolution of the FTIR spectra of the films deposited within days 1, 2, 3, 4 and 8, with the treatment temperature, is presented in Table.

The FTIR spectra of the films deposited on the first day (Fig. 1a) of sol preparation show the absorption bands at ~ 787, 907, 1051, 1446, 1639, 3200 cm^{-1}, corresponding to the vibration modes as presented in Table 1. The intensities of the absorption bands situated at ~ 787, 907, 1051 cm^{-1} decrease with the increase of temperature, due to the break of some Si-O- and P-O- linkages during the evaporation of water and volatile organic compounds. The intensities of bands at 1446, 1639 and 3200 cm^{-1} characteristic for δ (H_2O) and ν (OH) decrease with the increase of the treatment temperature and disappear after 350°C, indicating the loss of water. An interesting feature is the emergence of an absorption band at 575 cm^{-1}, attributed to 4-fold rings of SiO that were formed at 550°C by breaking the chains Si-O-Si. The intensity of the band at 787 cm^{-1} attributed to δ (Si–O–Si) decreases with the increase of temperature and splits into two bands, 732 and 783 cm^{-1}, confirming the broken linkages of Si-O-Si and the formation of P-O-P structures at the treatment temperature of 550°C. Weak Si-OH bands were noticed at about 2920 cm^{-1} starting from the thermal treatment at 300°C.

Analyzing the FTIR spectra of the films deposited on the second day (Table 1) and thermally treated at 200°, 300°, 350°, 400° and 550°C, it is necessary to specify that the absorption bands positions and intensities do not differ very much from the spectra recorded on the first day, except for the band at 2925 cm^{-1}. This is possibly due to Si-OH groups that disappear when the temperature is raised because of the condensation between Si-OH and P-OH units.

The films spin coated on the 3rd and 4th days exhibit spectra with noticeable different features as compared with first two days after sol preparation (Table 1). The shift to a higher energy of the absorption bands corresponding to ν_s and ν_{as} vibration modes is an indication of stronger P-O-P and Si-O-Si linkages. When the treatment temperature goes up, starting from 350°C, two bands at ~ 800 and ~ 850 cm^{-1} were noticed on days 3 and 4 and were attributed to ν_{sym} (P-O-P) and ν_{asym} (P-O-P), respectively. The intensities of the bands around 800, 960 and 1051 cm^{-1} corresponding to Si-O and P-O linkages in different compounds do not decrease with the increase of the treatment temperature as a consequence of stronger bonds that cannot be broken by evaporation of water and ethanol. The weak Si-OH band at ~ 2965 cm^{-1} present in the spectra of days 3 and 4 at all treatment temperatures give evidence for the Si-OH groups formed during the hydrolysis phase of the sol-gel process that are entrapped in the film pores. The intensity of the stretch vibration of OH groups from the adsorbed H_2O (1450 cm^{-1}), of deformation modes of OH groups and δ adsorbed H_2O (1635 cm^{-1}) along with δ (H_2O) (3200 cm^{-1}) decreases with the increase of the treatment temperature. These bands do not disappear at elevated temperatures meaning that OH linked by different compounds was entrapped in the pores of the film.

On day 8 of the deposition (Fig. 1b), the absorption bands at 800 and 850 cm^{-1} attributed to δ (Si–O–Si), ν_{as} (PO_4^{3-}), Si-O-P and ν_{sym} (POP) were present at all the treatment temperatures with no noticeable modifications in intensities. The intensity of the band at about 1039 cm^{-1} attributed to ν_s(Si–O–Si)

Table 1. Bands assignment in FTIR spectra of CdSe – doped silicophosphate films

Annealing temperature 200^0C				Annealing temperature 300^0C						Annealing temperature 350^0C					Annealing temperature 400^0C					Annealing temperature 550^0C					
Day since sol preparation				Day since sol preparation						Day since sol preparation					Day since sol preparation					Day since sol preparation					
1	2	3	4	8	1	2	3	4	8	1	2	3	4	8	1	2	3	4	8	1	2	3	4	8	
																				575		576	576		4 fold-rings SiO [14]
787	787	795	799	802, 849	787	783	803	804 851	803 849	787	795	804 852,	803 851	803 850	791	787	803, 852,	803 852	800 850	732, 783	733, 783	804, 850	803	800 851	δ (Si–O–Si) [13, 14] νas (PO_4^{3-}) [16, 17] νsym (POP) [16, 17] Si-O-P [18, 19]
907	903	931	943	948	907	903	943	951	951	911	927	955	952	951	907	907	955	951	951	904, 985	904,	959	956	952	νs PO_4^{3-}- νs Si–OH [16] νas (POP) [16, 17]
1051	1048	1051	1051	1035	1048	1047	1051	1051	1036	1048	1048	1051	1051	1039	1047	1047	1051	1051	1039	1051	1051	1051	1051	1036	νs (Si–O–Si) [14] νs (PO_4^{3-}) [14] νas (SiO_4) [13] νs (PO_3^{2-}) (chain end groups) [16] ν (P–O) stretching modes [16]
1446	1440	1443	1446	1446	1456	1446	1456	1446	1446	–	1446	1456	1446	1446	1456	1457	1453	1446	1446	–		1453	1450	1446	Stretch vibration of OH from adsorbed H_2O [14, 15]
1639	1642	1639	1639	1633	1639	1642	1635	1635	1632	1646	1642	1635	1635	1633	1635	1646	1635	1635	1632	1620	1643	1635	1635	1632	Deformation modes of OH groups δ adsorbed H_2O [14, 15]
2200						2300				2323						2300									
	2400																								
			2965	2966	2920		2968	2966	2966	–		2968		2966	2970		2968		2966	2925	–	2967		2966	weak Si-OH band [15]
3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	3200	–	–	3200	3200	3200	δ (H_2O) [15]

δ = bending vibration mode
ν = stretching vibration mode

was not modified with the increase of temperature. At all temperatures the bands at 1446, 1632, 2966 and 3200 cm^{-1}, corresponding to stretch vibration of OH from the adsorbed H_2O, deformation modes of OH groups, δ adsorbed H_2O, the weak Si-OH band and δ (H_2O) were present. Since the water could not be evaporated at temperatures above 200°C, we concluded that the water was entrapped in the pores of the films.

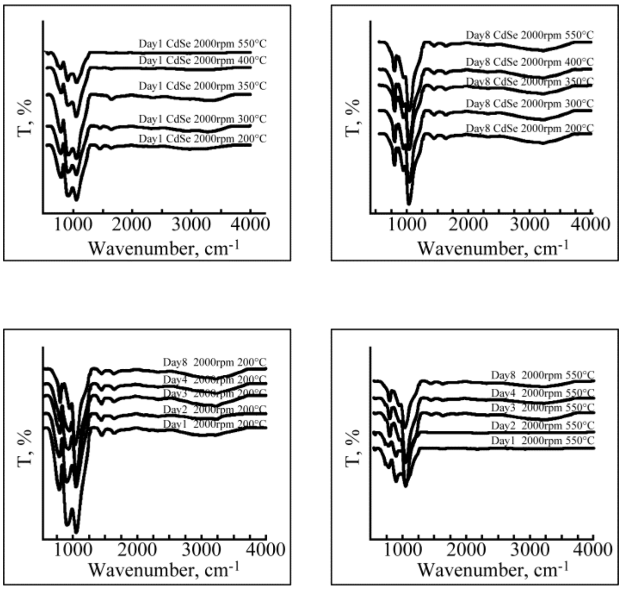

Fig. 1. FTIR spectra of CdSe-doped silicophosphate films: (a) Day 1, annealing at different temperatures; (b) Day 8, annealing at different temperatures; (c) Different days of deposition, annealing at 200°C; (d) Different days of deposition, annealing at 550°C.

When looking at the recorded spectra at 200°C (Fig. 1c), on days 1 up to 8, the bands corresponding to v_{sym} (P-O-P) and δ (Si–O–Si) shifted from 787 cm^{-1} (day 1) to higher wave numbers − 795 cm^{-1} (day 3) and 799 cm^{-1} (day 4), and split into 802 cm^{-1} and 850 cm^{-1} (day 8) due to the formation of stronger P-O-P and Si–O–Si bonds. The bands corresponding to v_{as} (POP) and v_s Si–OH at 907 cm^{-1} (day 1) shifted to 931 cm^{-1} (day 3), 943 cm^{-1} (day 4), and 948 cm^{-1} (day 8). These shifts were due to increased Si-O- and P-O- linkages in the films. The intensity of the band at 1051 cm^{-1} was decreasing from day 1 to day 8 due to broken Si-O-Si chains. The bands at 1446, 1632, 2966 and 3200 cm^{-1}, corresponding to the stretch vibration of OH from the adsorbed H_2O, deformation modes of OH groups, δ adsorbed H_2O, weak Si-OH band and δ (H_2O) were present on all days, without any significant modification in intensities.

The FTIR spectra of the films deposited on different days and treated at 300°C, 350°C and 400°C (Table 1) give evidence for the progress of the hydrolysis and condensation processes. Thus the bands corresponding to v_{sym} (P-O-P) and δ (Si–O–Si) shifted to higher wave numbers due to the formation of stronger bonds P-O-P and Si–O–Si. The intensities of 1051 cm^{-1} band increase on days 3 and 4 and decrease on day 8. Also on day 8, a shift towards lower wave numbers (energies) was noticed giving evidence of weaker Si-O-Si bonds. The bands corresponding to OH, Si-OH and H_2O vibration modes at 1446, 1632, 2966 and 3200 cm^{-1} were present on all days, though their intensities were slightly decreasing on day 8.

The FTIR spectra of the films deposited on different days and treated at 550^{0}C (Fig. 1d) exhibit the bands corresponding to v_{sym} (P-O-P) and δ (Si–O–Si) shifted to higher wave numbers due to the formation of stronger bonds P-O-P and Si–O–Si. The intensities of adsorption bands at 1051 cm^{-1} increase on days 3 and 4 and decrease on day 8. Also on day 8, a shift towards lower wave numbers (energies) was noticed giving evidence of weaker Si-O-Si bonds in long chains. The bands corresponding to OH, Si-OH and H_2O vibration modes at 1446, 1632, 2966 and 3200 cm^{-1} were present only in the FTIR spectra of the films deposited on days 3, 4 and 8.

To conclude, it is necessary to say that during the first two days the hydrolysis phase of the process was more pregnant, and due to the condensation process Si-O-Si, Si-O-P and P-O-P compounds were formed in smaller quantities. When the treatment temperature increased, the Si-OH and P-OH and H-OH bonds are broken and completely evaporated from the films, thus providing new Si-O-P bonds. Further, on days 3 and 4, the condensation process became pregnant and the increase of temperature allowed to OH groups to be entrapped in the pores of the films. As the gelification process is running, especially from the 3rd day of the film deposition, at 300°C and higher treatment temperatures, a band around 800 cm^{-1} is observed, assigned to bending/stretching modes of P-O-P, Si-O-P and O-P-O units. Thus, we can say that the structure of the deposited layers consists of a mixed framework built of [SiO_4] and [PO_4] tetrahedra.

The Si-O-Si band at about 1051 cm^{-1} is shifted from lower wave numbers on days 1 and 2 to higher ones on days 3 and 4 and decreases on day 8. That means that stronger bonds are formed during days 3 and 4. The maximum intensities of this vibration band are for the films deposited on days 3 and 4, which means that the Si-O-Si is pregnant in the deposited films.

Structural changes appeared as a result of the gelification process and the influence of the thermal treatment temperatures and different rotation rates on the hydrolysis/condensation process was investigated by Raman spectroscopy.

The gelification process was found to be performed, to a great extent, on day 8, taking into account that after this moment the gel could not be deposited on the glass substrate due to the increase of viscosity.

Figure 2a displays Raman spectra of bulk gel and films deposited on days 1 and 8, annealed at 200°C and 550°C.

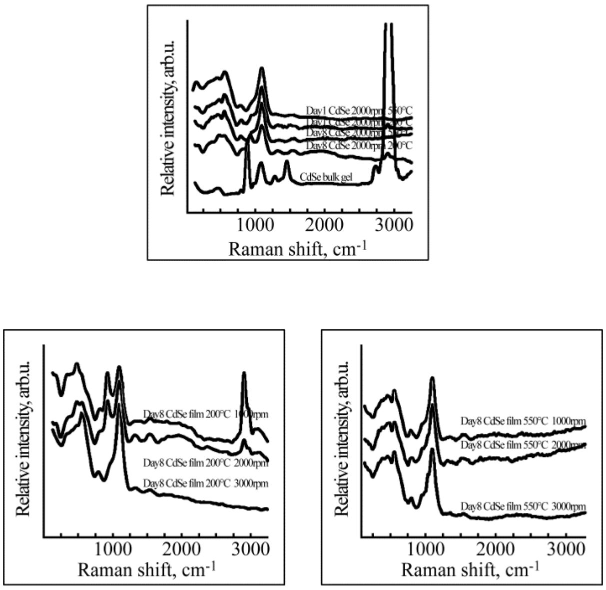

Fig. 2. Raman spectra of CdSe-doped silicophosphate films: (a) Day 1 and Day 8, annealing at 200°C and 550°C; (b) Day 8, annealing at 200°C, different rotation rates; (c) Day 8, annealing at 550°C, different rotation rates.

The presence of the following main peaks was noticed at 475, 555, 800, 925, 980 and 1098 $\mathrm{cm^{-1}}$. Thus, we attributed, in agreement with the literature data [14, 20, 21], the peak placed at 478 $\mathrm{cm^{-1}}$ to the transverse-optical (TO) Raman vibration mode of Si-O-Si bonds, the peak at 555 $\mathrm{cm^{-1}}$ to longitudinal optic (LO) rocking vibration mode of Si-O-Si, and the peak located around 1098 $\mathrm{cm^{-1}}$ to stretching asymmetric vibration mode for Si-O-Si. The peak attributed to Si-OH, located at 925 $\mathrm{cm^{-1}}$ in bulk gel, was shifted to 980 $\mathrm{cm^{-1}}$ in films. The peak at about 800 $\mathrm{cm^{-1}}$ is assigned to P-O-Si vibration mode.

The films deposited on days 2, 3 and 4 show similar Raman behavior and therefore we chose to present only films deposited on days 1 and 8 (see Fig. 2a).

The vibration modes specific to Si-OH (980 $\mathrm{cm^{-1}}$) and H-O-H (2900 $\mathrm{cm^{-1}}$) were noticed for the bulk gel and film at 200°C and their intensity decreased with the increase of annealing temperature, as expected.

A thorough analysis of the Raman spectra shows that the positions of the Si–O–Si (475, 555, 1098 $\mathrm{cm^{-1}}$) and Si-O-P bands (800 $\mathrm{cm^{-1}}$) remain invariable, irrespective of the heat treatment temperature.

The spectra of the films deposited on day 8 and annealed at 200°C at 3 different rotation speeds (Fig. 2b) are dominated by the peaks 475, 555, 800, 1098 $\mathrm{cm^{-1}}$ without any significant modification with the rotation speed (thickness of the film). The peak at 980 $\mathrm{cm^{-1}}$ corresponding to the vibration mode of Si-OH was noticed, only for 1000 and 2000 rpm. We suppose that at 3000 rpm (reduced thickness of the film) Si-OH groups are easily decomposed even at 200°C.

The spectra of films deposited in the 8th day annealed at 550°C at 3 different rotation speeds (Fig. 2c) reveal the same peaks as in Fig. 2b. No significant changes with the rotation speed were noticed.

The Raman characteristic band of CdSe could not be noticed in any samples due to small dimensions of the embedded microcrystals [22].

As it can be seen from Fig. 3, XRD pattern for CdSe 99,999% purity powder used as a dopant in silicophosphate films exhibits sharp lines characteristic for the semiconductor crystalline compound. The XRD lines are in accordance with data presented in [23, 24].

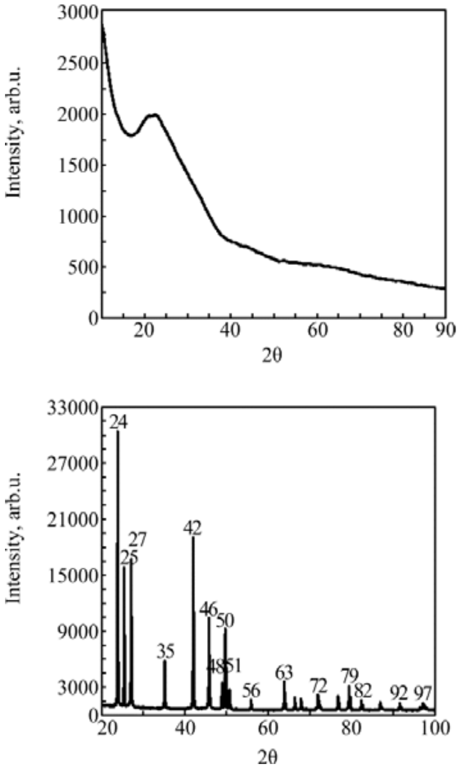

Fig. 3. XRD spectra of: (a) CdSe-doped silicophosphate film, deposited on Day 8, annealing at 550°C; (b) CdSe powder used as dopant.

As concerns the XRD pattern for the CdSe-doped silicophosphate film, day 8, annealed at 550°C for 2 *h*, (see Fig. 3), the non-crystalline feature of the graph is observed, which is due to the major vitreous silicophosphate network. It is to be taken into consideration that CdSe dopant lines do not appear due to a low concentration, bellow 5 wt. %, being under the detection limit of the measurement device and possibly due to the much reduced dimensions of the dopant particles.

CONCLUSIONS

The FTIR absorption spectra (550–4000 $\mathrm{cm^{-1}}$) as well as the Raman spectra (150–3500 $\mathrm{cm^{-1}}$), taken by 514.5 nm laser excitation), revealed structural units specific to the silicophosphate matrix. These features revealed the role of vitreous network formers of SiO_2 and P_2O_5.

The time elapsed since the sol preparation till the deposition moment, as well as the thermal treatment at different temperatures, brought about changes of the structural peculiarities of the deposited films.

Different rotation speeds of the substrates used in the deposition process did not influence significantly the structure of the films.

The low concentration of CdSe dopant embedded in a predominant vitreous silicophosphate network did not allow giving evidence for the crystalline degree of semiconductor particles.

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Реферат

Изготовлены пленки SiO_2-P_2O_5, легированные селенидом кадмия (CdSe), и исследованы их свойства с целью применения в качестве температурных датчиков (сенсоров). Пленки синтезированы золь-гель методом с использованием техники напыления вращением (spin coating technique); нанесение пленок проводилось на подложках из стекла. При изготовлении золя в качестве прекурсоров для SiO_2 и P_2O_5 использовались, соответственно, тетраэтилортосиликат (TEOS) и фосфорная кислота. Этанол служил растворителем, а вода использовалась как реагент для гидролиза. Пленки наносились при трех скоростях вращения: 1000, 2000 и 3000 рот/мин, начиная с одного дня после изготовления золя до четвертого дня, соответственно, затем после восьмого дня. Проанализировано влияние скорости ротации и времени, пройденного с момента изготовления пленок до момента их нанесения, на структурные свойства легированных пленок. Нанесенные пленки при каждой скорости ротации были отожжены при температурах 200, 300, 350, 400 и 550°С с целью исследования структурных модификаций в процессе образования силико-фосфатной решетки.

Ключевые слова: инфракрасная спектроскопия, спектроскопия Раман, золь-гель метод, CdSe.