CONDENSED-MATTER SPECTROSCOPY

The Influence of Low-Temperature Silver-Ion Exchange on the Spectral-Luminescent Properties of Fluorophosphate Glasses Doped with PbSe

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Abstract—Changes in the absorption and luminescence spectra of fluorophosphate glasses doped with PbSe caused by low-temperature Ag^+-Na^+ ion exchange are considered. It is found that the silver distribution gradient in a near-surface layer about 16 μm thick leads to two different processes of interaction between metal and semiconductor nanoparticles. PbSe molecular clusters and quantum dots more efficiently grow in deep layers with a low silver concentration. The near-surface glass layers with a high silver concentration exhibit formation of Ag metal nanoparticles, on the surface of which interaction with PbSe molecular clusters leads to the formation of $Ag-Se-Pb$ bonds, which transform into Ag_2Se layers in the process of heat treatment. The appearance of the new phase is confirmed by X-ray diffraction.

INTRODUCTION

The optical properties of quantum dots (QDs) formed in various media have been extensively studied for the last two decades [1–3]. It was shown that PbSe(S) can be used for photodetectors, convectors for solar-cell arrays, broadband optical amplifiers, and near-IR luminophores. Owing to the large Bohr exciton radius (46 nm), the narrow-band-gap PbSe semiconductor exhibits a strong size quantization effect in a wide spectral range (1000–3000 nm) [4–11]. In recent years, analogously to silver molecular clusters (MCs), interest has been shown in semiconductor MCs consisting of 1–8 molecular units, as well as to large clusters consisting of a great number of molecular units and called magic-sized clusters [12–15], which form from small clusters before the QD-formation stage. Possible structures of $(PbSe)_n$ ($n = 1–8$) MCs were calculated by computer simulation in [11]. PbSe magic-sized clusters with a high luminescence quantum efficiency were synthesized by colloidal-chemistry methods in [14]. In [15, 21], intense luminescence in the visible spectral region was recorded upon UV excitation of glasses doped with Pb, Se, and S, which made it possible to conclude that $(PbSe)_n$ and $(PbS)_n$ MCs exist in glasses directly after synthesis.

Great interest has recently been shown in nanoparticles (NPs) that have a complex structure and consist of a core and a shell of different chemical composi-

tions. Heterostructural materials in the metal–metal, metal–semiconductor, and semiconductor–semiconductor systems were synthesized by colloidal-chemistry methods [24–29]. These systems attract attention as materials for optoelectronics. However, there exist only occasional studies on the formation of composite NPs in glasses [16–21].

The aim of the present work was to study the influence of silver introduced into glass by low-temperature ion exchange (LIE) on the growth of QDs in the ion-exchange region of glass, as well as the influence on each other of metal and semiconductor NPs. We also consider the possibility of growth of complex composite NPs in the metal–semiconductor system.

Special interest in the formation of composite NPs is related to the fact that their nonlinear properties considerably differ from the properties of one-component NPs. Theoretical and experimental studies showed that the nonlinear Kerr effect can be enhanced by seven orders of magnitude due to the Mie resonance [16].

The formation of lead chalcogenide QDs in glasses occurs as a result of heat treatment (HT) of initial glasses containing lead and selenium ions at temperatures exceeding the glass transition temperature (T_g). It should be noted that the initial glass contains small molecular clusters [15, 20], which increase in the HT process to sizes of nanocrystals (QDs).

The clusters have characteristic luminescence bands in the visible region [15] in contrast to the QD luminescence that is observed in the IR range for PbSe. By changing the HT temperature and time, one can obtain QDs in a wide size range. It was shown in [18−21] that introduction of silver in low concentrations stimulates growth of PbS QDs. The sizes of PbS QDs in ion-exchange regions after $Ag^+−Na^+$ exchange turned out to be larger than in the regions without ion exchange. The introduction of silver led to an increase in the absorption and luminescence intensity. Since the absorption coefficient for QDs of identical sizes is proportional to their concentration, it was suggested that the number of QDs increases with increasing silver concentration, which testifies to the catalytic action of silver on the QD crystallization. This means that an increase in silver concentration leads to an increase in the number of NPs serving as nuclei for PbS crystallization, which, in turn, results in an increase in the number of QDs. Thus, Ag NPs are additional nucleators, which leads to an increase in the total number of QDs, as well as in the luminescence and absorption intensity [18, 19].

Another view on the interaction between silver NPs and semiconductor QDs was formulated in [17]. In this work, hybrid Ag/CdS NPs in a glass matrix were formed for the first time by introducing silver and semiconductor directly into the melt. As a result, the authors obtained glasses with silver and semiconductor NPs, which was proved by characteristic absorption spectra. The band corresponding to the surface plasmon resonance of silver NPs was shifted to longer wavelengths, which unambiguously indicated formation of a semiconductor shell on the silver NP surface.

In the present work, we study fluorophosphate glasses, which have numerous advantages compared to traditional silicate glasses. These advantages include low temperatures of glass synthesis and heat treatment needed for the formation of QDs, as well as a high concentration of the semiconductor phase with a homogeneous distribution of QDs over the volume in the process of growth [8]. In our previous works, we studied the growth of PbS QDs [8] and $(PbSe)_n$ MCs [15] in this matrix. It was shown that a two-stage HT makes it possible to form QDs with a narrow size distribution. This work considers specific features of the formation of NPs and QDs after LIE in glass of the $0.25Na_2O−0.5P_2O_5−0.05ZnF_2−0.1Ga_2O_3−0.02PbF_2−0.08AlF_3$ ZnSe composition.

EXPERIMENTAL

Samples of fluorophosphate glasses of the $0.25Na_2O−0.5P_2O_5−0.05ZnF_2−0.1Ga_2O_3−0.02PbF_2−0.08AlF_3$ composition without doping and doped with 0.5% of ZnSe in excess of 100% were synthesized in a way similar to that in work [8]. The glass was annealed at $T = 320°C$ for 2 h to remove thermal stresses. For measurements, we prepared polished plane-parallel samples with dimensions of $1.0 × 1.0$ cm and a thickness of $0.5−1.0$ mm. The LIE in the samples was performed for 30 min in melt of the $0.05 AgNO_3−0.95 NaNO_3$ composition at $T = 320°C$. After the LIE, the samples were washed with acetone. The prepared samples were heat-treated in a muffle furnace (Nabertherm) at $T = 410°C$ for $10−70$ min. The samples were placed in the furnace preliminarily heated to the required temperature. The glass-transition ($T_g = 400°C$) and crystallization temperatures were determined using an STA 449F1 Jupiter (Nietzsche) differential scanning calorimeter.

Silver was introduced into the glass by the LIE method. The depth of Ag^+ penetration into the glass was determined based on the refractive-index profile using the procedure of ion-distribution calculation described in [22].

X-ray diffraction analysis was performed using a Rigaku X-ray diffractometer with $CuK\alpha$ radiation and a D/teX Ultra detector.

The absorption spectra were measured on a Cary-500 (Varian) spectrophotometer within the range of $300−3000$ nm. Luminescence was measured on an LS55 PerkinElmer spectrofluorimeter and an EPP2000-UVN-SR (StellarNet) fiber-optic spectrometer under excitation by semiconductor lasers ($\lambda = 408$ and 808 nm). All measurements were made at room temperature.

RESULTS

The effect of silver on the growth of PbSe QDs was studied by comparing the experimental data for three glasses. We compared the absorption and luminescence spectra of glass without a semiconductor phase after 30-min $Ag^+−Na^+$ LIE (glass 1), glass containing lead and selenium but without silver (glass 2), and glass containing lead and selenium subjected to 30-min $Ag^+−Na^+$ LIE (glass 3).

First, we studied the initial glass matrix after LIE. The penetration depth of Ag^+ ions and their concentration were determined based on the calculated refractive-index profile using the ion-distribution calculation method described in [22]. The refractive-index profile was calculated by the inverse Wentzel−Kramers−Brillouin method. The validity of using this approach to determine the silver-concentration distribution in glass is based on the well-known fact that LIE (Ag^+-melt $\leftrightarrow$ Na^+-glass) leads to an increase in the refractive index due to a difference in the polarizabilities of exchanging ions [22]. In this case, the refractive-index profile coincides with the distribution profile of silver ions in the near-surface layer. Figure 1 demonstrates the penetration depth and concentration profile of silver in glass after LIE.

Silver concentration, mol %
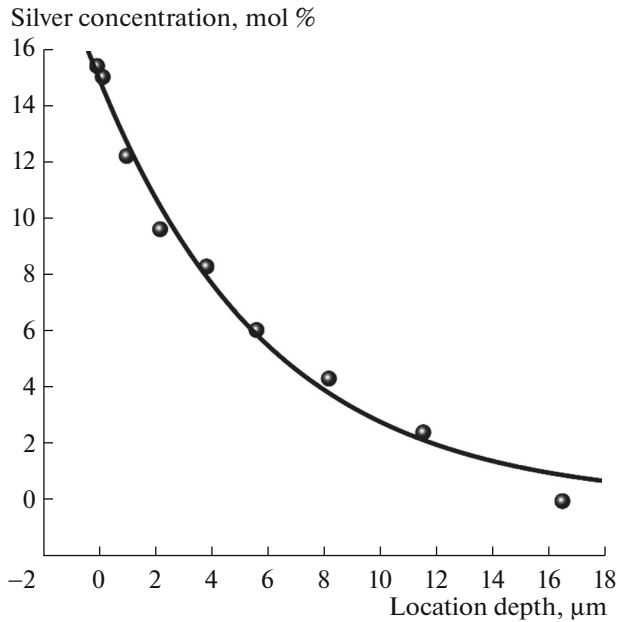
Fig. 1. Silver-concentration distribution in glass after 30-min LIE.

To form silver NPs, the glass after LIE was additionally heat-treated at a temperature exceeding T_g ($T = 410°C$) for 10, 20, and 30 min.

Figure 2 shows the absorption spectra of glass 1 after LIE and HT. LIE causes no noticeable changes in the absorption spectrum. The spectrum exhibits only some increase in absorption in the region above 4.5 eV. After HT at 410°C for 10−30 min, a surface plasmon resonance band appears at $\lambda_{max} = 410$ nm, which indicates formation of silver NPs [23].

To determine the average diameter D of Ag NPs by analyzing the surface plasmon resonance band [23], we used the formula

$$D = 2V_f/\Delta\omega_{1/2}, \qquad (1)$$

where $\Delta\omega_{1/2}$ is the halfwidth of the surface plasmon resonance band, $\Delta\omega_{1/2} = \omega_2 - \omega_1$, $\omega_i = 2\pi c/\lambda_i$, λ_i is the corresponding wavelength, c is the speed of light, and $V_f = 1.39 \times 10^8$ cm/s is the Fermi velocity for metal silver.

Figure 2 shows that the 10-min HT forms metal silver NPs with an average size of 4 nm ($\alpha = 7.5$ cm^{-1}), while an increase in the HT time to 30 min leads to an increase in the NP concentration ($\alpha = 35$ cm^{-1}) without changes in size.

Retaining all the treatment parameters, we performed LIE for glasses containing lead and selenium. Figure 3 shows the influence of the Na$^+$−Ag$^+$ ion exchange, which corresponds to the glass 2 → glass 3 transition, on luminescence (a) and absorption (b).

Absorption coefficient, cm^{-1}
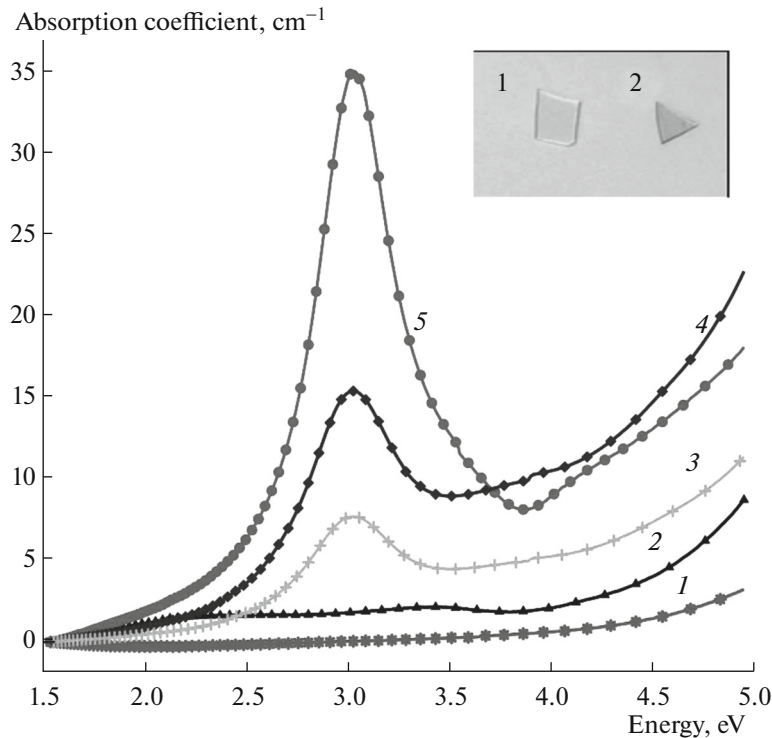
Fig. 2. Absorption spectra of glass 1 (*1*) before and (*2–5*) after LIE at $T_{LIE} = 320°C$ for $t_{LIE} = 30$ min with subsequent HT at $T = 410°C$ for (*2*) 0, (*3*) 10, (*4*) 20, and (*5*) 30 min. The inset shows a photograph of glass 1 (*1*) before and (*2*) after HT at $T = 410°C$ for 30 min.

Intensity, rel. units
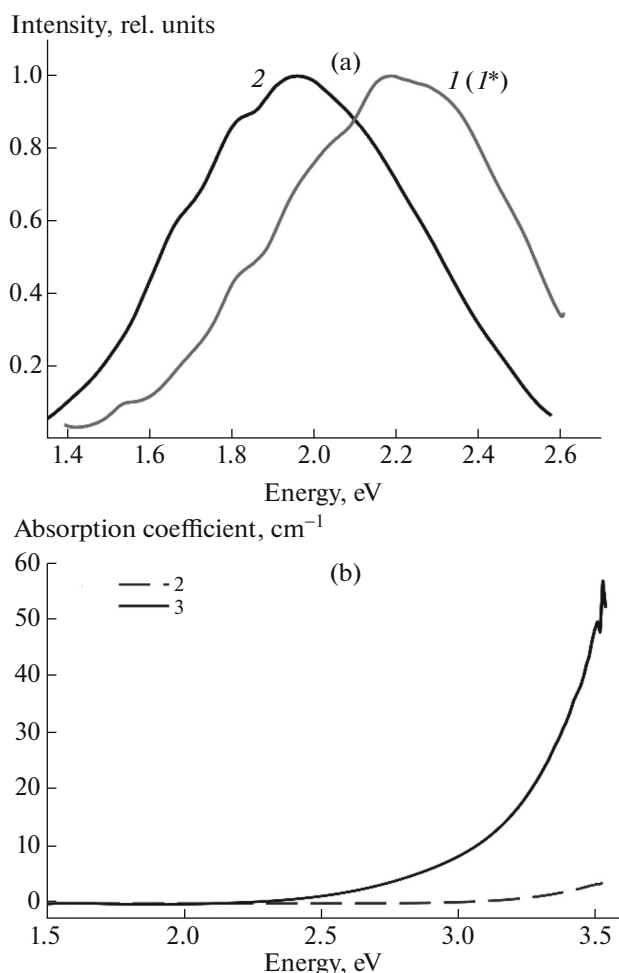
Fig. 3. (a) Normalized luminescence of glass 2 (*1*) without HT and (*I**) after 30-min HT at $T = 320°C$, as well as of (*2*) glass 3 (2 + LIE) ($\lambda_{exc} = 409$ nm). (b) Absorption spectra of initial glasses 2 and 3.

Figure 3a shows the shift of the luminescence band occurring after LIE. The luminescence in the initial glasses with lead and selenium is caused by the formation of $(PbSe)_n$ MCs [15]. The luminescence band of $(PbSe)_n$ in glass 2 with $E_{max} = 2.25$ eV (550 nm) shifts to $E_{max} = 1.97$ eV (630 nm) after LIE ($T = 320°C$, 30 min) (glass 3), while identical HT without LIE does not change the luminescence spectrum, which corresponds to stability of the ensemble of emitters.

Figure 4a shows how the absorption spectra of glass 2 change after HT.

Figure 4b demonstrates changes in the luminescence spectrum of glass 2 due to similar HT. It is obvious that heating at $T \geq T_g$ for 10 min causes no shift of the luminescence band of initial glass 2 with $\lambda_{max} = 560$ nm (2.21 eV). An increase in the HT duration to 20 and 30 min leads to successive shifts of the band to $\lambda_{max} = 590$ (2.1 eV) and 670 nm (1.85 eV), respectively. The long-wavelength shifts of the absorption and

luminescence bands indicate a growth in size of $(PbSe)_n$ clusters [13, 14]. Heating for 40 min leads to the appearance of two bands: a broad luminescence band of $(PbSe)_n$ clusters and a narrow band with $\lambda_{max} = 1000$ nm (1.24 eV) corresponding to the luminescence of small PbSe QDs [13].

An increase in HT time to 50−70 min leads to the appearance of a discrete absorption spectrum characteristic for QDs. The size of PbSe QDs was determined using the formula [5]

$$E_g(D) = E_g(\infty) + \frac{1}{0.0105D^2 + 0.2655D + 0.0667}, \quad (2)$$

where D is the effective QD diameter (nm), E_g is the energy of the first excited state of PbSe QDs, and $E_g(\infty)$ is the bandgap energy of bulk PbSe crystal (0.29 eV).

The average size of synthesized QDs estimated based on formula (2) for HT durations of 50 and 70 min was 4.1 and 5.2 nm, respectively. The size of 5.2 nm coincides with the result obtained by X-ray diffraction with calculation by the Debye−Scherrer formula (Fig. 6b). The luminescence spectra for the same glasses excited at $\lambda_{exc} = 808$ nm are presented in Fig. 4d.

Figure 5 shows the changes in the absorption and luminescence spectra of glass 3 after HT under the same conditions as in the case of glass 2. Already after 10-min HT, the spectra exhibit an absorption band at $\lambda = 850$ nm (1.46 eV) and two luminescence bands, namely, a band of $(PbSe)_n$ MCs ($\lambda = 600−650$ nm) and a low-intense QD band with $\lambda_{max} = 950−1000$ nm (1.3−1.24 eV). The first HT of Se- and Ag-containing glass 3 at $T = 410°C$ leads to an anomalous short-wavelength shift of the luminescence band from 633 to 608 nm (from 1.93 to 2.04 eV). The absorption bands after this HT naturally shift to longer wavelengths. Heating for 20 min results in the appearance of two identically intense luminescence bands belonging to clusters and QDs.

The luminescence spectra after HT for 30 and 40 min are caused by the centers formed in the regions subjected to LIE. The broad QD size distribution due to the silver concentration gradient and different QD growth rates leads to a shift of absorption and to the absence of a discrete spectral structure.

The luminescence spectra of glasses 2 and 3 after HT for 50 and 70 min completely coincide in intensity and positions of bands, which is explained by the geometry of the experiment, in which we measured the total luminescence of the samples. An increase in the HT time obviously leads to a sharp increase in the QD concentration in the entire glass volume, which makes the contributions of ion-exchange layers (16 μm) unresolved.
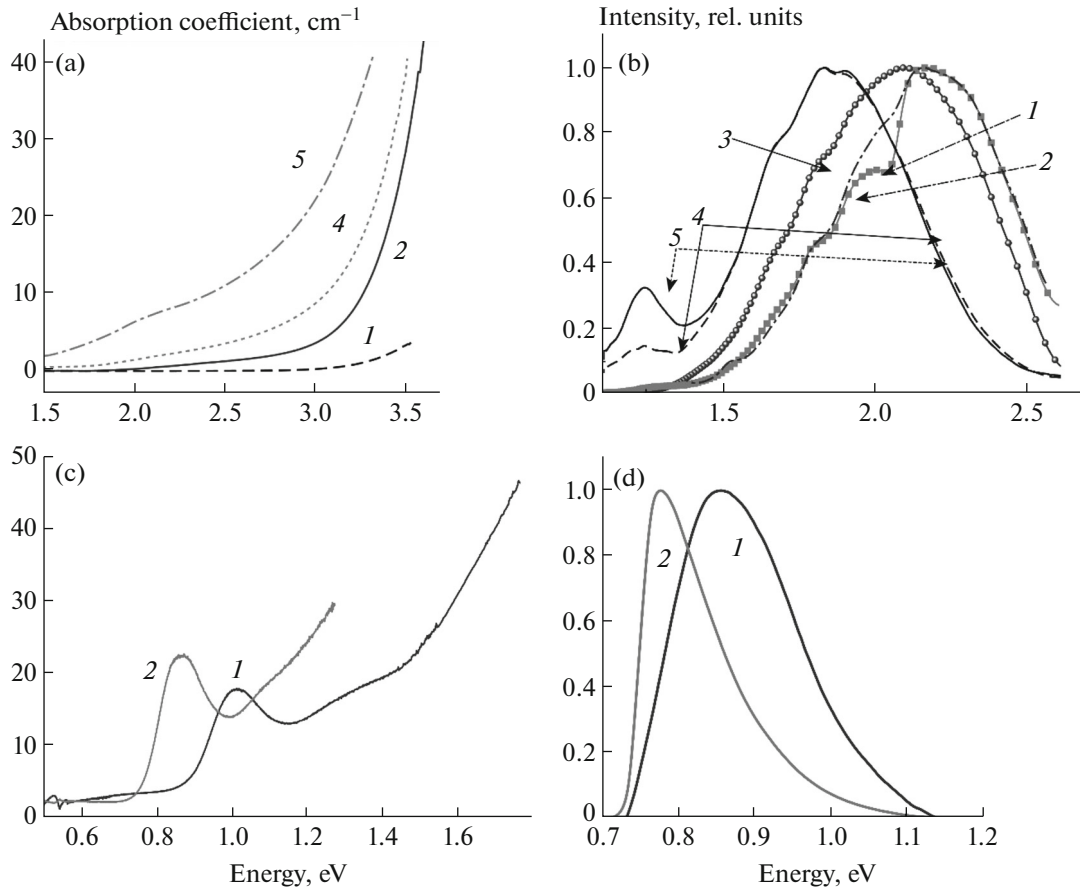
Fig. 4. (a) Absorption and (b) normalized luminescence (λ_{exc} = 409 nm) spectra of glass 2 (*1*) before and (*2–5*) after HT at *T* = 410°C for (*2*) 10, (*3*) 20, (*4*) 30, and (*5*) 40 min. (c) Absorption of glass 2 after HT at *T* = 410°C for (*1*) 50 and (*2*) 70 min. (d) Normalized luminescence (λ_{exc} = 808 nm) of glass 2 after HT for (*1*) 50 and (*2*) 70 min.

DISCUSSION OF RESULTS

To clarify the influence of Ag NPs on the growth of PbSe MCs and QDs in glass, we compared the results of measurements of three glasses, i.e., of the initial glass after ion exchange and PbSe-doped glasses before and after ion exchange.

The study of changes in the absorption and luminescence spectra of glass 2 indicates that HT with durations up to 40 min leads to the growth only of $(PbSe)_n$ MCs, and the clusters of the maximum size exhibit an absorption band with λ_{max} = 2.0 eV and a luminescence band at 1.8 eV (Figs. 3a, 3b).

The spectra of $(PbSe)_n$ ($n = 1-8$) MCs as functions of *n* were calculated in [11]. The calculations revealed a red shift in the spectra from 4.0 to 1.9 eV with increasing number of formula units. It was shown that the cubic $(PbSe)_4$ clusters are stable and serve as structural blocks for large clusters. According to calculations, the $(PbSe)_8$ clusters have sizes of about 1 nm and are characterized by the first absorption peak near 1.90 eV. Comparison of our results with calculated

data allows us to suggest the formation of $(PbSe)_8$ cluster in glasses (Fig. 3a).

Stable clusters with sizes from 1.0 to 1.8 nm synthesized in [13] by colloidal-chemistry methods exhibited intense luminescence in the spectral range from 780 to 1000 nm. This size range corresponds to the transition from MC to QD. It was supposed that the first absorption peaks at 625, 690, 750, 820, and 880 nm (1.98, 1.80, 1.65, 1.51, and 1.41 eV) correspond to NPs with diameters of 1.0, 1.2, 1.4, 1.6, and 1.8 nm, respectively. The luminescence spectra of these NPs contain bands with maxima at 1.58, 1.53, 1.45, 1,35, and 1.23 eV (780, 810, 850, 920, and 1000 nm). Taking into account these results, we may suggest that the absence of luminescence bands with maxima in the range of 700–900 nm in these glasses and the appearance of the 1000-nm band belonging to small QDs indicate the absence of clusters with $n > 8$ and 10 in glasses 2 and 3 and the formation of QDs by aggregation of $(PbSe)_8$ clusters ($D \sim$ 1 nm).

Comparison of the absorption and luminescence spectra of glasses before and after ion exchange allows
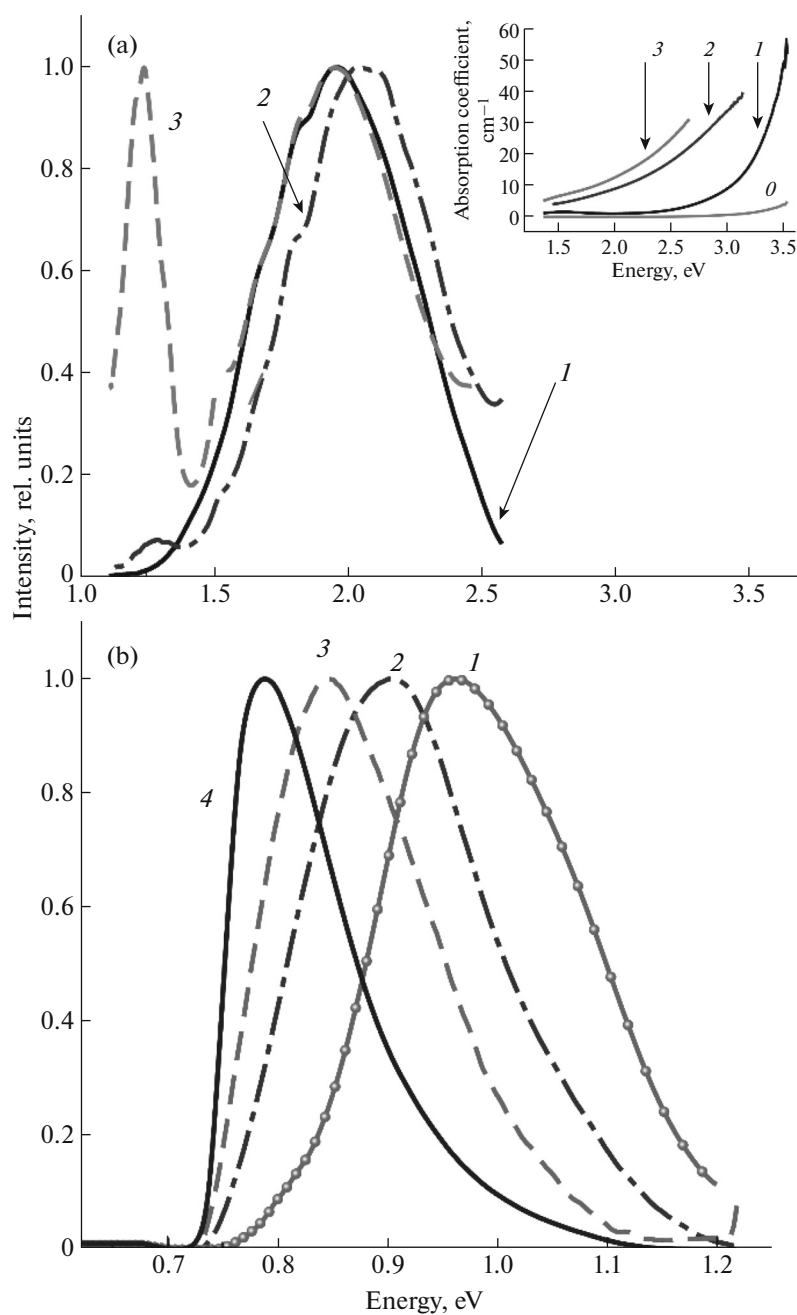
Fig. 5. (a) Normalized luminescence spectra of glass 3 (*1*) before and (*2, 3*) after HT at *T* = 410°C for (*2*) 10 and (*3*) 20 min (λ_exc = 409 nm). The inset shows the absorption spectra of (*0*) glass 2 and (*1–3*) glass 3 (*1*) before HT, (*2*) after HT for 10 min, and (*3*) after HT for 20 min. (b) Normalized luminescence (λ_exc = 808 nm) of glass 3 after HT at *T* = 410°C for (*1*) 30, (*2*) 40, (*3*) 50, and (*4*) 70 min.

us to make some suggestions concerning the mechanism of interaction of PbSe MCs and QDs with silver in the form of MCs and metal NPs. Analysis of the optical spectra of glasses 1 and 2 makes it possible to separate the contributions made by the near-surface ion-exchange layer (which is about 16.0 μm thick and contains on average 8.0 mol % of Ag_2O and 1.0 mol %

of PbF_2) and by the glass volume and to formulate the following specific features.

—The first HT of glass 3 causes an anomalous blue shift of the 633-nm luminescence band to 608 nm, while the corresponding absorption spectrum shifts to longer wavelengths. Similar HT of glass 2 naturally shifts its luminescence and absorption bands to longer

Intensity, arb. units
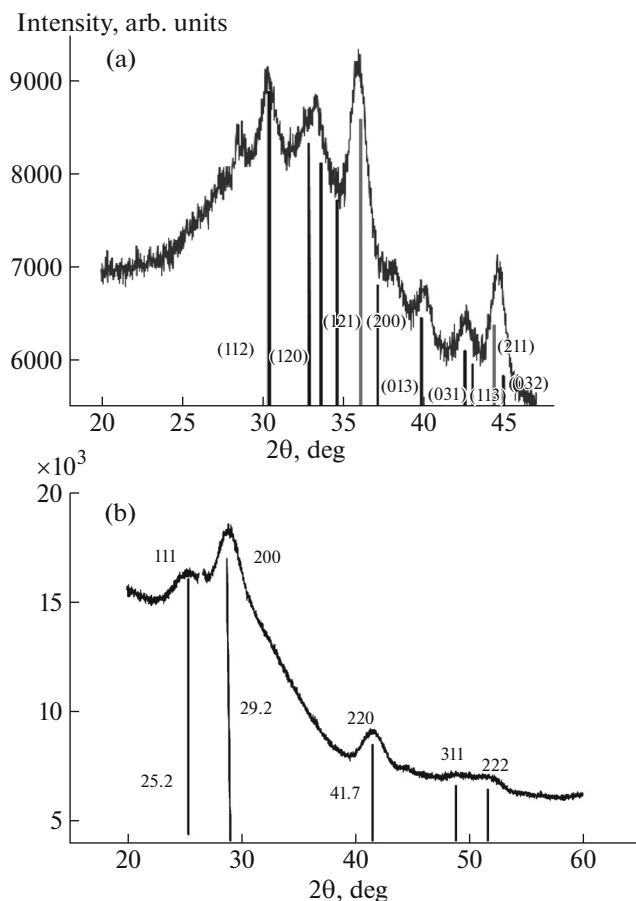
Fig. 6. (a) X-ray pattern of the surface of glass 3 after LIE for 90 min and HT at $T = 410°C$ for 360 min: α-Ag_2Se crystal phase (JCPDS card, no. 00-027-0619) and orthorhombic Ag_2Se (JCPDS card, no. 00-024-1041). (b) X-ray pattern of the volume of glass 2: PbSe crystal phase (JCPDS card, no. 00-024-1042).

wavelengths, which corresponds to an increase in the size of clusters.

—HT of glass 3 for 30 and 40 min leads to the appearance of bands with $E_{max} = 0.9$ and 0.95 eV. These bands are absent in glass 2.

Comparison of the spectra of glasses 2 and 3 shows that $(PbSe)_n$ clusters and QDs formed in glass 3 are larger than those formed in glass 2 at the same HT duration. Already the first HT of glass 3 leads to the appearance of a band with $\lambda_{max} = 950–1000$ nm in addition to the band of $(PbSe)_n$ MCs at $\lambda = 600–650$ nm.

The shortest HT also causes a blue shift of the luminescence band of $(PbSe)_n$ clusters from 1.95 to 2.03 eV (from 633 to 608 nm). We can suppose that the observed changes occur due to the appearance of metal NPs, which were detected under similar HT conditions in glass 1 (Fig. 2). The presence of metal Ag may enhance the luminescence of $(PbSe)_n$ MCs local-

ized near the surface of Ag NPs. The competition between lead and silver for the formation of (Ag–Se) or (Pb–Se) bonds may result in a decrease of the size of $(PbSe)_n$ MCs near metal NPs. When an Ag NP interacts with a $(PbSe)_n$ MC, selenium forms the (Ag–Se) chemical bonds on the Ag NP surface. An increase in the HT duration increases the number of these bonds, which results in the formation of layers of the new Ag_2Se compound. Thus, we believe that the anomalous luminescence shift relates to the formation of Ag_2Se monolayers on the surface of Ag NPs (Fig. 5). This agrees with the results obtained for similar objects by colloidal-chemistry methods [24–29]. Further changes in the luminescence spectra and the appearance of the band with $\lambda_{max} = 1000$ nm demonstrate an acceleration of the growth of PbSe and, probably, Ag_2Se QDs, which also luminesce in this range [24]. The simultaneous presence of the MC and QD luminescence bands testifies to the formation of QDs by aggregation of MCs rather than to a successive increase in the number of structural units in the process of growth (Fig. 5a, curve *3*).

Taking into account the difference in the concentrations of lead and silver in the near-surface (ion-exchange) regions of glass (Fig. 1), we can conclude that Ag_2Se may appear near the glass surface. With increasing HT time, Ag NPs may transform into Ag_2Se QDs due to interaction with selenium. To confirm the proposed mechanism of interaction between PbSe and Ag, we performed an additional experiment. The glass containing Pb and Se was subjected to Ag LIE at $T = 320°C$ for 90 min and to HT at $T = 410°C$ for 6 h. The X-ray pattern of the glass surface (Fig. 6a) shows the formation Ag_2Se QDs about 8 nm in size, while the volume, in contrast, contains only PbSe QDs (Fig. 6b). Comparison with X-ray patterns of Ag_2Se QDs obtained by colloidal-chemistry methods [24–29] completely confirms our results.

CONCLUSIONS

The use of the LIE method to introduce silver in fluorophosphate glasses leads to a high silver concentration in the near-surface regions and to formation of Ag NPs at minimal HT duration. HT of glasses with lead, selenium, and silver results in the appearance of Ag–Se–Pb bonds on the surface of metal particles and in the formation of Ag_2Se layers. The appearance of the new phase is confirmed by X-ray diffraction. In deeper layers with a low silver concentration, the growth of PbSe MCs and QDs occurs faster than in glasses without silver.

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