Physics of the Solid State, 2022, Vol. 64, No. 7

09,08

Luminescence of $Hf_xZr_{1-x}O_2$ thin films upon excitation with vacuum ultraviolet synchrotron radiation

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Received March 10, 2022
Revised March 10, 2022
Accepted March 11, 2022

Using the methods of low-temperature luminescence spectroscopy and vacuum ultraviolet synchrotron radiation (MAX IV synchrotron, Lund, Sweden), charge transfer processes and the nature of charge carrier traps responsible for leakage currents in nanometer dielectric films of hafnium-zirconium-oxygen $Hf_xZr_{1-x}O_2$ solid solutions on silicon substrate, as well as in films doped with La-ions and annealed in argon atmosphere. The photoluminescence spectra, photoluminescence excitation spectra of the 2.7 eV emission band and previous simulation data within the framework of density functional theory confirm the presence of an oxygen vacancy in the studied films. Based on the study of competing channels for the relaxation of electronic excitations due to the radiative decay of self-trapped excitons (4.35 eV emission band) and the luminescence of defects (2.7 and 3.5 eV emission bands), a conclusion was made about the efficiency of transport and capture of excitons and separate charge carriers in films with varying degrees of defectiveness and films doped with lanthanum. The obtained data confirm the conclusion that oxygen vacancies are traps responsible for leakage currents in the films under study

Keywords: Luminescence, synchrotron radiation, defects, energy transfer.
DOI: 10.21883/PSS.2022.07.54587.305

1. Introduction

This article is aimed at solving the fundamental problem associated with the development of materials for nanotechnologies, with the development of the element base of nanoelectronic devices, components of non-volatile storage devices for information systems of a new generation. The purpose of this article is to study the localization, capture, and relaxation of charge carriers arising under photon irradiation, as well as the excited states of defects and their radiation and thermochemical modification in dielectric nanometer oxide films.

Study objects are nanometer films of solid solutions of hafnium-zirconium-oxygen $Hf_xZr_{1-x}O_2$ on a silicon basis, having a high permittivity (so called high-κ-dielectrics). Films of high-κ-dielectrics have real prospects for practical application in cells of RAM and flash memory. Such films have a ferroelectric effect, the ability to polarize in an electric field and subsequently retain electric polarization in the absence of an external applied voltage (non-volatile memory) [1–4]. The results of initial research show real opportunities for creating new technologies in the field of controlled formation of nanostructures with desired properties. Understanding the mechanisms of electron and hole transport and determining the parameters of their trapping centers will enable to create a physical basis for monitoring and controlling the parameters of non-volatile flash memory elements.

HfO_2 has three allotropic centrosymmetric modifications: monoclinic, cubic, tetragonal. The ferroelectric effect is not observed in these phases. However, recently a ferroelectric effect has been discovered on thin nanometer (15−20 nm) films of hafnium oxide in a non-centrosymmetric orthorhombic phase [4–6]. Such a crystalline phase of HfO_2 can be obtained by adding 3−8% impurities, such as La, Si, Al, to the chemical composition. The $Hf_xZr_{1-x}O_2$ solid solution also has a ferroelectric effect. These silicon-based films are considered as one of the options for the formation of thin dielectric layers in metal-dielectric-semiconductor (MIS) [5] structures. In this case, one of the problems with the use of high-κ-dielectrics is the presence of large leakage currents. Conductivity in such films is directly determined by the transport and capture of charge carriers and is due to defects formed during their synthesis. Oxygen vacancies, which have the highest concentration, and impurity centers can lead to a charge drain and loss of information. Point defects (vacancies, interstitial and substituting atoms) can be luminescence centers, charge carrier trapping centers, non-radiative recombination centers and determine the efficiency of charge carrier transport. The processes of transport, conversion of absorbed energy, oxygen vacancies, their various charge states in these oxide films can be successfully studied by highly sensitive and, what is important, nondestructive luminescence methods.

The first studies of such films by the photoluminescence (PL) method using laboratory excitation sources showed that, depending on the degree of defectiveness of the films, the low-temperature PL spectra show a broad band peaking at 2.7−2.9 eV with a large Stokes shift [5,6]. It is most efficiently excited in the film transparency region at energy ~ 5.2 eV and further with increasing energy in the region of the long-wavelength edge of fundamental absorption and interband transitions [5]. However, it is well known that the possibilities of laboratory sources of photons in this far UV region are significantly limited. The band gap E_g in HfO_2, ZrO_2, according to different authors [4,7,8], is 5.4−6.1 eV (depends on temperature). This assumes that the selective creation of exciton states or band charge carriers with different kinetic energies requires the use of vacuum ultraviolet (VUV) radiation. As practice shows, the greatest success here can be achieved only with the use of synchrotron radiation (SR), which has a high intensity and a continuous spectrum in the UV−VUV spectral regions. Indeed, as was shown even earlier, under such an SRexcitation in the PL spectra of the most perfect films based on HfO_2, ZrO_2 obtained by the plasma atomic layer deposition (ALD), a band in the region 4.2−4.4 eV, corresponding to the emission of self-trapped excitons (STE) [7,8], is clearly manifested. It is noted that as the defect concentration increases, the STE PL yield decreases, but the emission band increases in the region 2.7−3.1 eV, which is associated with defects in the crystal structure [9].

In this article, using the methods of low-temperature luminescence spectroscopy and synchrotron radiation in the vacuum ultraviolet range, we study the processes of charge transfer and the nature of charge carrier traps responsible for leakage currents in nanometer dielectric films of solid solutions of hafnium-zirconium-oxygen Hf_x $Zr_{1-x}O_2$ on a silicon substrate, as well as in films doped with La ions. A conclusion is made about the efficiency of transport and trapping of excitons and separate charge carriers in films with different degrees of defectiveness and films doped with lanthanum.

2. Experimental procedure

The study by luminescence spectroscopy methods during SR excitation in the UV−VUV spectral ranges was carried out at the FinEstLUMI [10,11] station of the Finnish-Estonian SR channel (FinEstBeAMS) of the MAX IV synchrotron complex (Lund, Sweden) [12]. The SR source was an undulator, the excitation photon energy range was 4.5−45 eV, the photon flux on the sample — $10^{11}-10^{12}\,s^{-1}$ [11]. The PL excitation spectra were corrected for an equal number of exciting photons using AXUV-100G photodiode. Luminescence detection in the UV and visible regions of the spectrum (200−800 nm) was carried out using a quartz fiber optic cable, Andor Shamrock SR-303i monochromator with diffraction gratings optimized for spectral regions of 300 and 500 nm, and Hamamatsu H8259-01 photodetector. The presented PL spectra are corrected for the spectral sensitivity of the optical detection system. The samples were placed in a helium vacuum cryostat of a closed cycle, measurements were carried out at a temperature of 10 K.

3. Study objects

The studied films were synthesized in the laboratory of NaMLab gGmbH (Dresden, Germany) by plasma atomic layer deposition (ALD) of ferroelectric $Hf_xZr_{1-x}O_2$ layers on silicon substrate (substrate resistance $7.5\,\Omega\cdot\mathrm{cm}$) [4]. The $Hf_{0.5}Zr_{0.5}O_2$ layer was deposited on (n,p)-Si (100) substrates using the precursors tris(dimethylamino)cyclopentadienyl hafnium ($(C_5H_5)Hf[N(CH_5)_2]_3$, $HfCp(NMe_2)_3$, HyALD^{TM}), tris(dimethylamino)cyclopentadienyl zirconium ($(C_5H_5)Zr[N(CH_5)_2]_3$ $Zr(NMe_2)_3$, ZyALD^{TM}) and tris(isopropyl)cyclopentadienyl lanthanum ($[iPrCp]_3La$) at temperature of 280°C. Ozone-oxygen plasma was used as an oxidizing agent for all three precursors. Next, fast (for 20 s) thermal annealing was carried out at 450°C in atmosphere N_2. The La content was calculated based on the growth rates and the cycle ratio. The thickness and stoichiometry of the films were controlled by laser ellipsometry and Rutherford backscattering spectroscopy. The final thickness of the films was determined by the ellipsometric method according to the technique described in the article [13] and varied in the region of 20 nm. The films were certified by X-ray photoelectron spectroscopy (XPS) [4]. The characteristic XPS spectra in the region of the core levels and the valence band indicate the quality of the prepared films. In particular, it is shown that the studied films doped with lanthanum have the electronic structure and band gap almost identical to those of the initial ones; its estimate gives the value $E_g = 5.4 \pm 0.2$ eV at room temperature. For additional generation of oxygen vacancies, reductive annealing in argon atmosphere at 900°C for an hour was used. The concentrations of oxygen vacancies in the films annealed in argon were estimated by comparing the experimental XPS spectra of the valence band with the calculated ones obtained on the basis of the density functional theory. A detailed analysis of the XPS spectra and other results of the certification of the films under study are given in [4].

4. Results and discussion

Below are the typical PL spectra and the PL excitation spectra of the films measured at $T = 10$ K and reflecting the characteristic features of the radiative relaxation of excitons and separate electron-hole pairs. The results show both the effect of additional annealing in argon atmosphere and the results of film doping with lanthanum.

Figures 1 and 2 show the PL spectra of the $Hf_{0.5}Zr_{0.5}O_2$ film (thickness $d = 19.5$ nm) measured at different energies of exciting photons E_{exc}, and PL excitation spectra measured for different emission bands E_{em} at $T = 10$ K. Depending

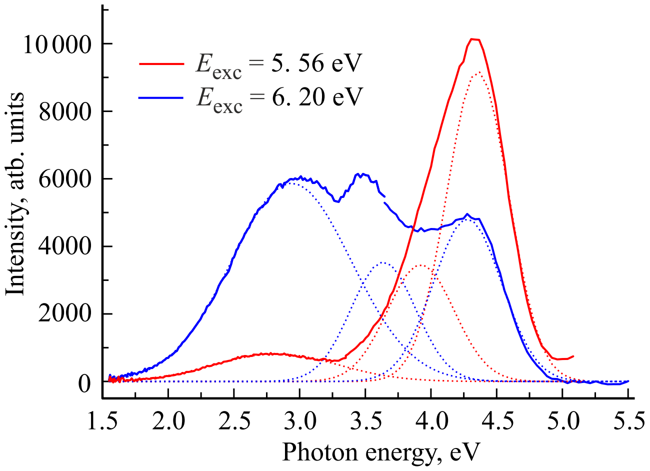

Figure 1. PL spectra of the $Hf_{0.5}Zr_{0.5}O_2$ ($d = 19.5$ nm) film, measured at different excitation photon energies, $T = 10$ K. The dashed lines show the Gaussian components obtained by approximation of the spectra.

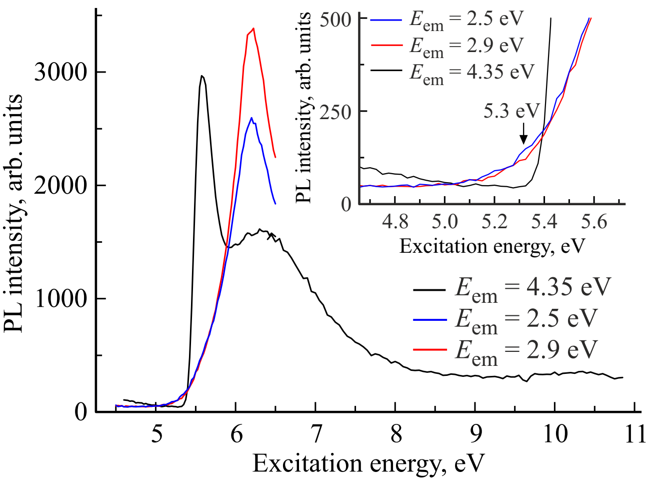

Figure 2. PL excitation spectra of the $Hf_{0.5}Zr_{0.5}O_2$ ($d = 19.5$ nm) film, measured for different E_{em} emission bands, $T = 10$ K.

on the excitation energy, dominant selective bands 4.35 eV are observed, then in the region 3.5–3.7 and ~ 2.7–2.8 eV. The 4.35 eV band corresponds to the emission of autolocalized excitons (STE), it is typical for hafnium or zirconium oxide crystals, and is also observed in the most perfect films based on them [7,8]. The purer the crystal or the less defective film, the higher the PL yield is observed in this band [8]. Note that the PL spectrum also exhibits a band in the region 3.5–3.7 eV, which indicates the presence of defects centers of radiative recombination of charge carriers, the nature of which is still unknown. The broad band in the region 2.7–2.8 eV, according to [5,6], corresponds to the PL of anion vacancies. STE fluorescence does not have selective excitation bands in the transparency region, it is effectively excited in the region of the fundamental absorption edge, forming a band at 5.6 eV, and then in the region of interband transitions, Fig. 2. The defect emission band 2.72 eV is excited in the film transparency region at the energy $E_{exc} = 5.3$ eV (Fig. 2, see inset) and in the region of interband transitions ($E_{exc} \geq 5.9$ eV). However, in the excitation spectrum of the 2.7 eV band, there is no singularity in the exciton production region ($E_{exc} = 5.6$ eV). Nevertheless, at the excitation energies E_{exc} indicated in Fig. 1, the PL spectrum clearly manifests itself and even the bands dominate corresponding to the emission of point defects-vacancies. The energy transfer of excitons and electron-hole pairs to anion vacancies — emission centers is observed. Under interband VUV-photoexcitation (or X-ray excitation), luminescence arises due to energy transfer from the matrix to defects. The separated electron-hole pairs formed under such excitations can be sequentially captured by a defect — oxygen vacancy Vo as a result of a reaction

$$(\mathrm{Vo} + \mathrm{e}^- + h^+ \rightarrow \mathrm{Vo}^- + h^+ \rightarrow \mathrm{Vo}^* \rightarrow \mathrm{Vo} + h\nu).$$

On the other hand, separated electron-hole pairs can bind into excitons e^0 with subsequent energy transfer to the oxygen vacancy as a result of the reaction

$$(\mathrm{Vo} + \mathrm{e}^- + h^+ \rightarrow \mathrm{Vo} + \mathrm{e}^0 \rightarrow \mathrm{Vo}^* \rightarrow \mathrm{Vo} + h\nu).$$

Such electron-hole and exciton energy transfer mechanisms are effective for many binary or complex oxides based on tungstates, molybdates, vanadates, see, for example, [14].

The shape of the PL band excitation spectrum of STE 4.35 eV of the film correlates well with the excitation spectra of hafnium or zirconium oxide films presented in the articles [7,8], for which, based on these PL excitation spectra and absorption spectra, an estimate of the minimum energy of interband transitions E_g equal to 5.8 and 6.15 eV at $T = 10$ K for ZrO_2 and HfO_2, respectively, is given. Using a similar ideology, for the $Hf_{0.5}Zr_{0.5}O_2$ films under study, we can estimate the value $E_g = 5.9$ eV at $T = 10$ K. This value is somewhat higher than the above estimate $E_g = 5.4 \pm 0.2$ eV at room temperature according to XPS data [4], even taking into account the temperature shift of the fundamental absorption edge. However, this discrepancy in this context is not fundamental and depends on the method of determining E_g.

Figures 3 and 4 show the PL spectra of the $Hf_{0.5}Zr_{0.5}O_2$ film additionally annealed in argon, measured at different excitation photon energies E_{exc}, and PL excitation spectra measured both for the STE emission band and at different points of the low-energy defect emission band. Etching with argon ions, as shown by the XPS data [4], creates an additional concentration of anion vacancies in the film. After annealing in argon, the STE band at 4.35 eV dominates in the PL spectrum, the 3.5 eV defect bands are almost invisible, and the defect emission band at 2.72 eV clearly manifests itself and is selectively excited in the 5.3 eV region (Fig. 4 is indicated by an arrow). Under such excitation, point defects-oxygen vacancies are clearly manifested. Note that in the PL excitation spectra there is no sharp drop in the PL yield with increasing excitation energy from the beginning of the fundamental absorption region (~ 6.2 eV) up to the energy 11 eV — the high-energy boundary of the measured spectra. The shape of the PL excitation spectra in this region is determined both by the features of the band structure and by the energy dependence of the absorption coefficient. A high value of the absorption coefficient in the region of interband transitions ($10^5 - 10^6$ cm^{-1}) leads to a decrease in the depth of light penetration into the crystal and an increase in the number of acts non-radiative decay of electronic excitations (band electrons, holes, excitons) near the surface [15]. In our case, the shape of the PL excitation spectrum indicates that the non-radiative near-surface energy losses of electronic excitations are small, and there is an efficient recombination of charge carriers at defects or the formation of excitons followed by their self-trapping. A comparison of the PL excitation spectra of defects forming the 2.7 eV emission band in films before and after annealing in argon is shown in Fig. 5.

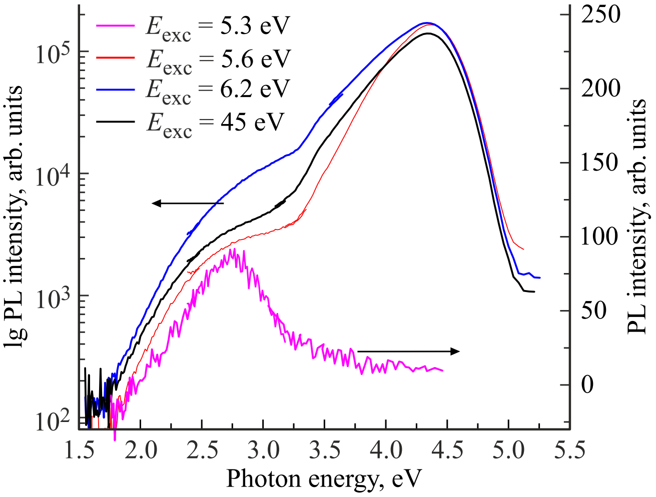

Figure 3. PL spectra of the $Hf_{0.5}Zr_{0.5}O_2$ ($d = 19.5$ nm) film annealed in argon, measured at different excitation photon energies E_{exc}. Note that the logarithmic scale is indicated along the y-axis for the upper three spectra, and linear for the lower spectrum.

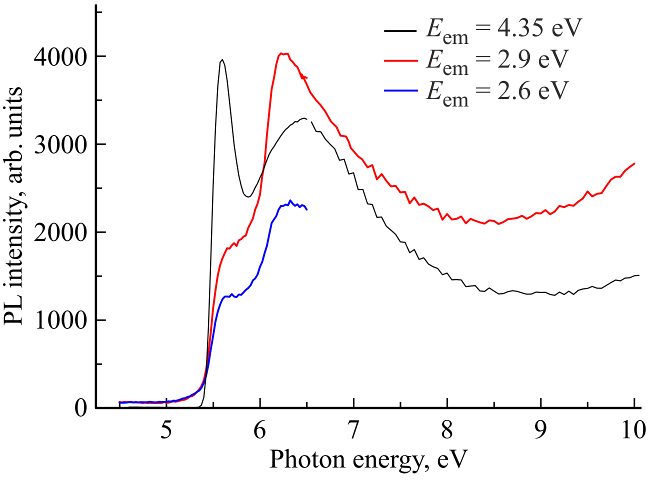

Figure 4. PL excitation spectra of the $Hf_{0.5}Zr_{0.5}O_2$ ($d = 19.5$ nm) film annealed in argon, measured for different emission bands at $T = 10$ K.

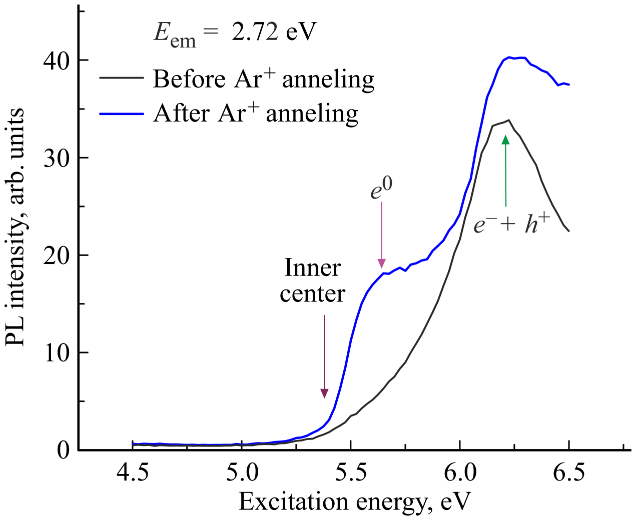

Figure 5. PL excitation spectra for the 2.72 eV emission band in the $Hf_{0.5}Zr_{0.5}O_2$ film before and after annealing in argon.

Thus, after annealing in argon atmosphere, the following features of PL spectra should be noted:

– the defect concentration — centers of radiative recombination of charge carriers, which form the emission band in the region of 3.5 eV, decreased. This is evidenced by an increase in STE fluorescence yield, the absence of a sharp drop in the STE fluorescence yield, as well as a sharp drop in the vacancy fluorescence band (2.7–2.8 eV) at high energies in the fundamental absorption region ($E > 6.1$ eV);

– the intensity of the band 2.72 eV, due to defects such as oxygen vacancies, increased, which can be associated with an increase in their concentration. The excitation spectrum of the 2.72 eV band has changed significantly — fluorescence of vacancies is excited in the region of 5.3 eV (intracenter excitation, indicated by an arrow in Fig. 4), then in the region of 5.6 eV (due to energy transfer through exciton, which was absent in the original unannealed sample) and at energies $E_{exc} > 6.1$ eV, i.e. when creating separate charge carriers, both thermalized and having a certain kinetic energy. Thus, the exciton mechanism of energy transport to defects becomes more efficient.

Next, we consider the results of doping with lanthanum ions. A priori, the introduction of trivalent substitutional ions should lead to an increase in the concentration of anion vacancies, based on the local charge compensation condition. XPS data [4] confirm this. Figure 6 shows the PL spectra of the La : HfZrO film measured at different energies of exciting photons, and Figure 7 shows the PL excitation spectra in the same films measured for different emission bands.

The PL spectra of the La: HfZrO film are dominated by a broad non-elementary band in the region of 3.7–4.3 eV,

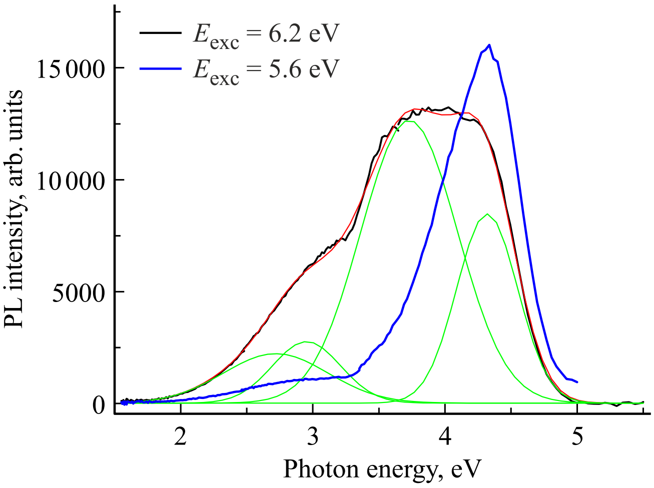

Figure 6. PL spectra of the La:HfZrO (2.0 mol.% La, $d = 21.2$ nm) film measured at different excitation photon energies and the result of approximation of the PL spectrum by Gaussian components.

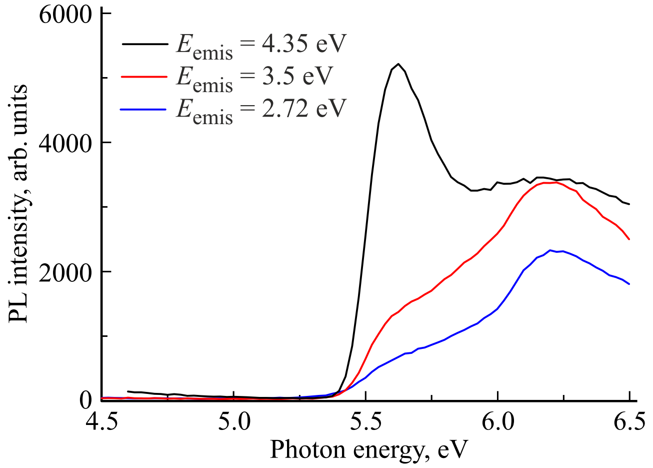

Figure 7. PL excitation spectra of La:HfZrO (2.0 mol.% La, $d = 21.2$ nm) film measured for different emission bands.

however, the position of its maximum and the shape depend significantly on the excitation energy. Thus, when creating thermalized electron-hole pairs ($E_{exc} = 6.2$ eV), the maximum of this non-elementary band falls on the energy of ~ 3.9 eV. However, the shape of the PL spectrum is complex and can be satisfactorily approximated by a set of four Gaussian components with maxima of 2.72, 2.95, 3.72, and 4.32 eV (Fig. 6). The first three of them clearly correspond to the defects emission, the last one — to the STE fluorescence. When excitons are created ($E_{exc} = 5.6$ eV), the maximum of the PL spectrum is at energy of 4.35 eV, which corresponds to STE emission, and low-energy PL bands associated with exciton relaxation on defects appear. The PL excitation spectra of these bands (Fig. 7) show an arm of 5.6 eV in the exciton absorption region and a maximum of 6.2 eV, the position of which corresponds to the formation of separate thermalized charge carriers. That is, in a film doped with lanthanum, excitation of PL defects proceeds both by the recombination path (predominantly) and due to the exciton mechanism of energy transfer. The PL spectroscopy data obtained on films annealed in argon atmosphere and films doped with lanthanum show very similar results. This indicates that defects, including anion vacancies, are effective centers for capture and recombination of charge carriers and centers for localization of exciton states.

5. Conclusion

The method of low-temperature luminescent VUV spectroscopy using synchrotron radiation showed high information content in the study of defects and processes of electronic excitation energy transfer in nanometer dielectric films of hafnium-zirconium-oxygen solid solutions. Based on the study of competing channels for relaxation of electronic excitations due to the radiative decay of STE (low-temperature emission band 4.35 eV) and defect luminescence (emission bands 2.7 and 3.5 eV), a conclusion was made about the efficiency of transport and capture of excitons or separate charge carriers in films with different degrees of defectiveness (initial and annealed in argon) and in films doped with lanthanum. Introduction of lanthanum changes the efficiency of the radiative relaxation channels of created excitons or band charge carriers with different kinetic energies, and leads to appearance of relaxed excitons bound to defects. The data of low-temperature PL spectroscopy and literature simulation data within the framework of the density functional theory confirm the correctness of the Nasyrov–Gritsenko multiphonon model proposed earlier for describing the charge transfer mechanism [16] and the conclusion that oxygen vacancies (band 2.7 eV in the PL spectra) are traps responsible for leakage currents in the films under study. At the same time, the charge state of anion vacancies, as well as the nature of the defect luminescence band in the region of 3.5 eV, are still unknown and require model calculations. From an experimental point of view, low-temperature VUV experiments with subnanosecond time resolution using synchrotron radiation are needed.

Acknowledgments

V.A.P. thanks the MAX IV laboratory for providing beam time on the FinEstBeAMS synchrotron radiation channel (MAX-Lab project No. 20200211) and Dr. Kirill Chernenko (MAX-Lab) for assistance in taking measurements. The research carried out in the MAX IV laboratory was supported by the Swedish Research Council (contract 2018-07152), the State Agency for Innovation Systems of Sweden (contract 2018-04969) and Formas (contract

2019-02496). The co-authors from the Institute of Semiconductor Physics SB RAS thank Uwe Schroeder and Prof. Thomas Mikolajick (NaMLab gGmbH (Dresden, Germany) for providing lanthanum-doped $Hf_{0.5}Zr_{0.5}O_2$ films.

Funding

This work was supported in part by the Ministry of Science and Higher Education of the Russian Federation (basic part of the state assignment, project No. FEUZ-2020-0060), the Russian Foundation for Basic Research (grant No. 20-57-12003 NNIO_a), and the Russian Scientific Fund (project No. 21-12-00392).

Conflict of interest

The authors declare that they have no conflict of interest.

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