

## SPECTROSCOPY OF CONDENSED STATES

# Optical Characteristics of Strontium Titanate Films Obtained by the Sol–Gel Method

N. I. Stas'kov<sup>a,\*</sup>, A. B. Sotskii<sup>a</sup>, L. I. Sotskaya<sup>b</sup>, I. V. Ivashkevich<sup>a</sup>, A. I. Kulak<sup>c</sup>,  
N. V. Gaponenko<sup>d,e</sup>, M. V. Rudenko<sup>d</sup>, and A. N. Petlitskii<sup>f</sup>

<sup>a</sup> Kuleshov State University, Mogilev, 212022 Belarus

<sup>b</sup> Belarusian–Russian University, Mogilev, 212000 Belarus

<sup>c</sup> Institute of General and Inorganic Chemistry, National Academy of Sciences of Belarus,  
Minsk, 220076 Belarus

<sup>d</sup> Belarusian State University of Informatics and Radioelectronics, Minsk, 220000 Belarus

<sup>e</sup> National Nuclear Research University Moscow Engineering and Physics Institute,  
Moscow, 115409 Russia

<sup>f</sup> Belmikrosistemy Research and Development Center, OAO Integral, Minsk, 220108 Belarus

\*e-mail: ni\_staskov@mail.ru

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**Abstract**—Using data of multiangle spectrophotometry and spectral ellipsometry in the UV and visible ranges, spectra of the refractive indices and absorption coefficients and width of the forbidden band of one- and five-layer strontium titanate films obtained by the sol–gel method have been calculated. Layer-by-layer deposition of sol on quartz substrates, from one to five layers, leads to an increase in film porosity from 4 to 33%. This causes a decrease in the refractive indices in the middle part of the visible spectrum from 2.33 to 1.87, which leads to a decrease in the reflectance and an increase in the transmittance or transparency of five-layer films. With an increase in the heating temperature of such films from 500 to 750°C, a shift of the absorption band maximum from 239 to 253 nm takes place and the optical width of the forbidden band decreases from 4.63 to 4.20 eV. The absorption band maximum of the single-layer film falls on the wavelength of 252 nm, and its optical width of the forbidden band is 3.96 eV.

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## INTRODUCTION

Strontium titanate SrTiO<sub>3</sub> is a wide-band perovskite oxide semiconductor characterized by a unique combination of high dielectric permittivity and optical transparency in the visible range, chemical and composition stability, and other physical properties, which make it prospective for opto- and microelectronics. Films based on SrTiO<sub>3</sub> are of interest for designing MIS structures and DRAM capacitors, monolithic microwave integrated circuits (MMICs), and IR radiation sensors [1]. The reversible transition of SrTiO<sub>3</sub> from the high-ohmic state to the low-ohmic state under the action of the UV and visible radiation opens a prospect of using this material as memristors—elements of nonvolatile memory [2–6]. Intense photo-, cathode-, and radio luminescence has been found in SrTiO<sub>3</sub> films doped with lanthanides [7, 8].

SrTiO<sub>3</sub> films are obtained both by physical (magnetron, pulsed laser deposition) and by chemical methods, among which the sol–gel technology is the most widespread [9–15]. Using this method, one can obtain at relatively low temperatures quality films with

a high degree of crystallinity and corresponding SrTiO<sub>3</sub> stoichiometry [16]. To achieve the necessary film thickness, subsequent multifold deposition is applied, which, however, leads to an increase in porosity of the films and changes in optical properties [17]. Besides, an increase in the heat treatment temperature from 500 to 750°C leads to a change in their thickness and a decrease in the forbidden band width  $E_g$ . It is well known that the value of  $E_g$  for direct optical transitions varies from 3.43 to 3.62 eV in SrTiO<sub>3</sub> crystalline films [18] and from 3.68 to 3.94 eV in amorphocrystalline structures [19]; in amorphous films, it increases to 4.07 eV [20].

At present, the forbidden band width of semiconductor films is estimated using techniques [17, 21–26] based on the following assumptions.

(i) The substrate does not absorb light in the absorption band region of the film (its absorption coefficient  $k_s = 0$ ).

(ii) Absorption coefficient  $\alpha_f(\lambda)$  of the film depending on its absorption coefficient  $k_f(\lambda)$  [27] and wavelength  $\lambda$  as

$$\alpha_f(\lambda) = \frac{4\pi k_f(\lambda)}{\lambda} \quad (1)$$

can be determined based on approximate formulas with allowance for light losses only for absorption (Bouguer's law) [17, 21],

$$\alpha_f(\lambda) = \frac{1}{h_f} \ln \frac{1}{T_t(\lambda)}; \quad (2)$$

for absorption and single reflection [22, 23],

$$\alpha_f(\lambda) = \frac{1}{h_f} \ln \frac{1 - R_t(\lambda)}{T_t(\lambda)}; \quad (3)$$

and for absorption and double reflection [24],

$$\alpha_f(\lambda) = \frac{1}{h_f} \ln \frac{[1 - R_t(\lambda)]^2}{T_t(\lambda)}. \quad (4)$$

The absorption coefficient in the region of the edge of the intrinsic absorption band is also calculated [25] by a more complicated formula:

$$\alpha_f(\lambda) = \frac{1}{h_f} \ln \frac{[1 - R_t(\lambda)]^2}{2T_t(\lambda)} + \sqrt{[R_t(\lambda)]^2 + \frac{[1 - R_t(\lambda)]^4}{4[T_t(\lambda)]^2}}. \quad (5)$$

In formulas (2)–(5), parameter  $h_f$  is the film thickness measured in centimeters and  $T_t(\lambda)$  and  $R_t(\lambda)$  are the measured transmittance and reflectance of the film on the substrate upon normal ( $\varphi = 0$ ) incidence of non-polarized light on the sample. After the calculation of the absorption coefficient spectrum, there appears a possibility to determine the width of the SrTiO<sub>3</sub> forbidden energy band based on the Tauc extrapolation [28]:

$$[\alpha_f(\lambda)E]^2 = B(E - E_g). \quad (6)$$

In formula (6),  $B$  is a constant and  $E = 1240\lambda^{-1}$  is the photon energy in electron volts (the wavelength  $\lambda$  is taken in nanometers). The intersection of the linear range of the dependence  $[\alpha_f(\lambda)E]^2$  with the  $E$  axis yields an estimate of  $E_g$ .

Formulas (2)–(5) were obtained for thick layers without regard to light interference in them. To exclude in (2) the influence of reflected light for a thin film in the presence of interference, let us turn to the energy conservation law  $T_t(\lambda) + R_t(\lambda) + A_t(\lambda) = 1$ , where  $A_t(\lambda)$  is the absorptance of the film–substrate structure. In the region where the light absorption by the substrate is absent ( $k_s(\lambda) = 0$ ), one can assume based on the Bouguer law that  $A_t(\lambda) = h_f \alpha_f(\lambda)$ . Then, outside the intrinsic absorption band of the film,

where  $h_f \alpha_f(\lambda) \ll 1$ , we have  $T_t(\lambda) + R_t(\lambda) = \exp[h_f \alpha_f(\lambda)]$ . In this approximation,

$$\alpha_f(\lambda) = \frac{1}{h_f} \ln \frac{1}{T_t(\lambda) + R_t(\lambda)}. \quad (7)$$

In contrast to expression (1), where it is assumed that the absorption coefficient of the film  $k_f(\lambda)$  is determined from the rigorous solution of the inverse problem of spectrophotometry, approximations (2)–(5), and (7) contain an unknown parameter—the film thickness. The quantity  $h_f$  is determined using methods of surface profilometry [22, 23] and spectral ellipsometry [26]. All these methods are characterized by different accuracy of determining the quantity  $h_f$ . For nonabsorbing media,  $\alpha_s(\lambda) = 0$ ,  $k_f(\lambda) = 0$ , and  $T_t(\lambda) + R_t(\lambda) = 1$ ; from (3) and (7), we have  $\alpha_f(\lambda) = 0$ . However, relations (2), (4), and (5) yield  $\alpha_f(\lambda) \neq 0$ , which call into doubt the applicability of expressions (2), (4), and (5) for determining the absorption coefficient of SrTiO<sub>3</sub> films.

In this work, to study the causes of changes in optical transparency and forbidden band width of SrTiO<sub>3</sub> in the process of subsequent multilayer sol deposition onto quartz plates, inverse problems of multiangle spectrophotometry for homogeneous films on plane-parallel quartz substrates with a finite thickness are solved using a developed algorithm. The thicknesses and spectra of refractive indices  $n_f(\lambda)$  and absorption coefficients  $k_f(\lambda)$  of single- and five-layer films have been determined. Based on this, possibilities of using known approximate formulas for estimating the forbidden band width and absorption coefficient have been analyzed.

To calculate spectra  $T_t(\lambda)$  and  $R_t(\lambda)$  of a homogeneous film on a plane-parallel substrate with a finite thickness (the direct problem of spectrophotometry), we used the solution of the electrodynamic problem on transmission and reflection of partially coherent light from this structure. Two computer programs have been developed. In the first of them, the Gaussian instrument function of the monochromator was integrated in a convolution with coherent reflectance and transmittance of the structure. In the second program, functions  $T_t(\lambda)$  and  $R_t(\lambda)$  were calculated by summation of ray series. The first program served for estimating the applicability limits of the second one, less accurate but with a significantly lesser amount of computations. The algorithm of the latter formed the basis of constructing objective functions for the numerical search of substrate spectra  $n_s(\lambda)$  and  $k_s(\lambda)$  and ensuing reconstruction of functions  $n_f(\lambda)$  and  $k_f(\lambda)$  and film thickness  $h_f$  by the least squares method. The developed programs [29] allow one to take into account the finiteness of the substrate thickness, light absorption in the whole structure, and the increase in the light path length in the film and substrate upon oblique incidence. Any restrictions on the film and substrate

thickness are not imposed. For the numerical determination of five main characteristics,  $h_f$ ,  $n_f(\lambda)$ ,  $k_f(\lambda)$ ,  $n_s(\lambda)$ , and  $k_s(\lambda)$  (the inverse problem of spectrophotometry), one should first measure spectra  $T_s(\lambda)$  and  $R_s(\lambda)$  of the substrate without a film and, then, spectra  $T_f(\lambda)$  and  $R_f(\lambda)$  of the substrate with a film. Control over functions  $n_s(\lambda)$  and  $k_s(\lambda)$  is necessary due to their noticeable deviation from dispersion tables known in the literature under conditions of a real experiment.

## EXPERIMENTAL

Strontium titanate films were obtained using a sol prepared by mixing of solutions of strontium acetate in acetic acid and titanium tetraisopropoxide  $\text{Ti}(\text{OCH}(\text{CH}_3)_2)_4$  in ethylene glycol monomethyl ether in the presence of acetone as a stabilizer [6]. The films were deposited on quartz substrates by centrifuging at 2700 rpm. Each  $\text{SrTiO}_3$  layer was dried and submitted to a preliminary heat treatment at  $200^\circ\text{C}$ . After deposition of five layers, the final heat treatment was implemented at temperatures of  $500\text{--}750^\circ\text{C}$  in air for 60 min.

Thickness of the quartz substrate  $h_s = 3.50$  mm was measured on an IKG 1 horizontal optimizer. Reflection and transmission spectra of the substrate  $T_s(\lambda)$  and  $R_s(\lambda)$  and of the substrate with a single-layer film  $T_f(\lambda)$  and  $R_f(\lambda)$  for TE- and TM-polarized light (the electric field vectors of TE- and TM-polarized light lie in the plane of incidence and perpendicularly to it, respectively) were recorded at angles of incidence of  $8^\circ$ ,  $24^\circ$ , and  $40^\circ$  on a Photon RT spectrophotometer in the spectrum range from 250 to 700 nm. Spectra  $T_f(\lambda)$  of five-layer films on a quartz substrate were obtained on a Cary-500 spectrophotometer in the spectrum range from 200 to 700 nm at  $\varphi = 0$  [17]. The spectra of ellipsometric angles of the substrate  $\psi_s(\lambda)$  and  $\Delta_s(\lambda)$  and of the substrate with a single-layer film  $\psi_f(\lambda)$  and  $\Delta_f(\lambda)$  were measured at angles of incidence of  $60^\circ$  and  $65^\circ$ , respectively, on a Horiba UVISEL spectroscopic ellipsometer in the range of 200–700 nm.

## RESULTS

Figure 1 presents the (a) transmittance and (b) reflectance spectra of three films under study on quartz substrates. Spectra  $n_s(\lambda)$  and  $k_s(\lambda)$  of the substrate were reconstructed by spectra  $T_s(\lambda)$  and  $R_s(\lambda)$  measured at three abovementioned angles of incidence and two light polarizations. Figure 2 presents the spectra of (a) refractive indices and (b) absorption coefficients of three films under study. Spectra  $n_f(\lambda)$  and  $k_f(\lambda)$  (curves 1) of a single-layer film were determined using the homogeneous film–substrate model in which eight spectra  $T_f(\lambda)$  and  $R_f(\lambda)$  measured with the use of TE- and TM-polarized light at angles of incidence of  $8^\circ$  and  $24^\circ$  were treated. The film thick-

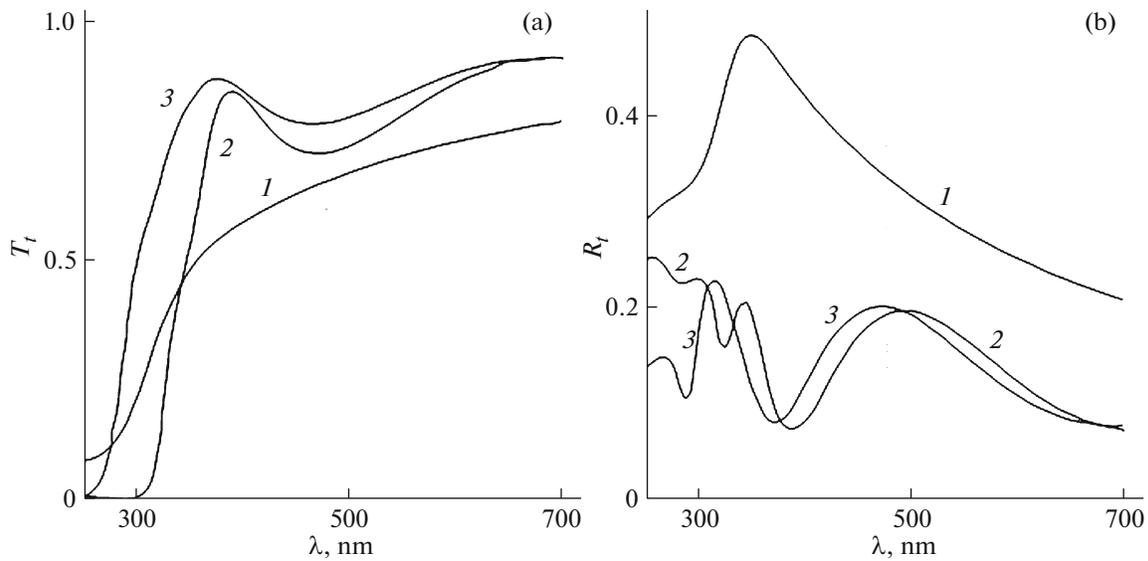
ness calculated in this case turned out to be equal to 33.6 nm. Thicknesses and optical functions  $n_f(\lambda)$  and  $k_f(\lambda)$  of two five-layer films were calculated by substitution of spectra 2 and 3 for  $\varphi = 0$  into formulas for  $T_f(\lambda)$  from [30] (Fig. 1a). The thicknesses of five-layer  $\text{SrTiO}_3$  films thermally treated at 500 and  $750^\circ\text{C}$  turned out to be equal to 186 and 194 nm, respectively. The value obtained from rigorous calculations for the thickness of a film treated at  $750^\circ\text{C}$  is close to that determined by electron microscopy,  $\sim 190$  nm [17]. The calculated spectra of the refractive indices and absorption coefficients of two thermally treated films are presented by curves 2 and 3 in Fig. 2. Solutions of the inverse problem of spectrophotometry for five-layer films made it possible to calculate their reflectances by formulas for  $R_f(\lambda)$  from [30] at  $\varphi = 0$  (curves 2, 3 in Fig. 1b).

One of criteria of correctness of the numerical solution of the inverse optical problem considered above is the correspondence of solutions obtained by different methods, e.g., by spectrophotometry and spectroscopic ellipsometry. Using the DeltaPsi2 software for the Horiba UVISEL ellipsometer, we have calculated parameters of the substrate and single-layer  $\text{SrTiO}_3$  film by measured spectra  $\psi_s(\lambda)$ ,  $\Delta_s(\lambda)$  and  $\psi_f(\lambda)$ ,  $\Delta_f(\lambda)$ . The dispersion dependence of the refractive index and absorption coefficient of  $\text{SrTiO}_3$  on photon energy  $E$  in the DeltaPsi2 software was determined by the formulas [31]

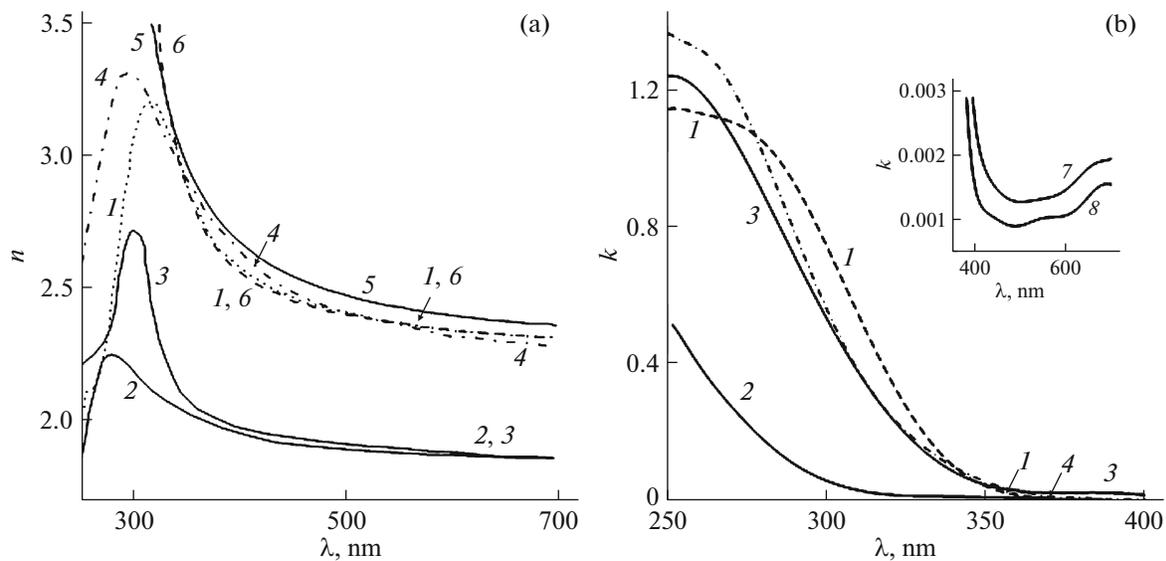
$$n_f(E) = n_\infty + [B(E - E_j) + C][(E - E_j)^2 + \Gamma_j^2]^{-1}, \quad (8)$$

$$k_f(E) = \begin{cases} f_j(E - E_g)^2[(E - E_j)^2 + \Gamma_j^2]^{-1}, & E > E_g \\ 0, & E < E_g, \end{cases} \quad (9)$$

where  $B = f_j\Gamma_j^{-1}[\Gamma_j^2 - (E_j - E_g)^2]$ ,  $C = 2f_j\Gamma_j(E_j - E_g)$ ,  $n_\infty$  is the refractive index corresponding to the zero photon energy,  $E_j$  is the photon energy corresponding to the absorption band maximum,  $f_j$  is the elastic constant of the oscillator,  $\Gamma_j$  is the absorption band half-width, and  $E_g$  is the photon energy corresponding to the edge of the absorption band. The least residual between the calculated and measured spectra  $\psi_f(\lambda)$  and  $\Delta_f(\lambda)$  was reached for the rough layer–film–substrate model. The thickness of the rough layer was  $(4.1 \pm 0.4)$  nm. The layer was modeled by the Bruggeman effective medium [32] consisting of air (50%) and  $\text{SrTiO}_3$  (50%). Parameters of the dispersion formulas (8) and (9) presented above for the film material turned out to be equal:  $n_\infty = 2.00 \pm 0.04$ ,  $E_g = (3.1 \pm 0.1)$  eV,  $f_j = 0.41 \pm 0.02$ ,  $E_j = (4.39 \pm 0.05)$  eV, and  $\Gamma_j = (0.84 \pm 0.05)$  eV. The thickness of the single-layer film without the rough layer is  $31.4 \pm 0.6$  nm, which satisfactorily corroborates the value determined above by spectrophotometry. Figure 2 presents the corresponding spectra of the refractive indices and absorption coefficients calculated for this film by formulas (8) and (9)



**Fig. 1.** (a) Transmission and (b) reflection spectra of (1) a single-layer SrTiO<sub>3</sub> film on quartz substrate and five-layer SrTiO<sub>3</sub> films thermally treated at (2) 750 and (3) 500°C.



**Fig. 2.** Spectra of the (a) refractive index and (b) absorption coefficient of a SrTiO<sub>3</sub> film: (1, 4, 7, 8) a single-layer film; five-layer films treated at (2) 500 and (3) 750°C, (5) a model film, and (6) an epitaxial film [30].

(curves 4). In the visible range, where  $E < E_g$ , the spectra  $n_j(\lambda)$  and  $k_j(\lambda)$  calculated by data of two optical methods are in a satisfactory accordance (curves 1, 4).

In [33], optical properties of a thin crystalline epitaxial SrTiO<sub>3</sub> film in the visible spectrum range were described using the Lorentz dispersion model:

$$n(\lambda) - ik(\lambda) = \sqrt{\epsilon_\infty + \frac{A}{E_g^2 - E^2 + i\Gamma_0 E}} \quad (10)$$

Curve 5 in Fig. 2a was constructed by parameters  $\epsilon_\infty = 3.077$ ,  $E_g = 4.466$  eV,  $A = 41.874$  eV<sup>2</sup>, and  $\Gamma_0 = 0$  presented in [33]. We numerically calculated parameters of the Lorentz model  $\epsilon_\infty = 3.98 \pm 0.02$ ,  $E_g = 4.14 \pm 0.06$  eV,  $A = 19.4 \pm 0.1$  eV<sup>2</sup>, and  $\Gamma_0 = 0$  for the interpolation in the range from 400 to 700 nm by function (10) (curve 6, Fig. 2a) of the experimental refractive index (curve 1) of a single-layer SrTiO<sub>3</sub> film. Let us pay attention, first, to the fact that parameters  $\Gamma_0$  in (10) and  $k(E)$  in (9) are zero. This is a consequence of

the fact that the single-layer SrTiO<sub>3</sub> film under study slightly absorbs visible light. Second, the value of the parameter  $n_\infty = 1.994$  for model (10) almost coincides with the value calculated for model (8). The position of curves 1–4 and 5 in Fig. 2a in the visible spectrum range can be explained by the fact that the density of the epitaxial film is higher than the density of films obtained by the sol–gel method.

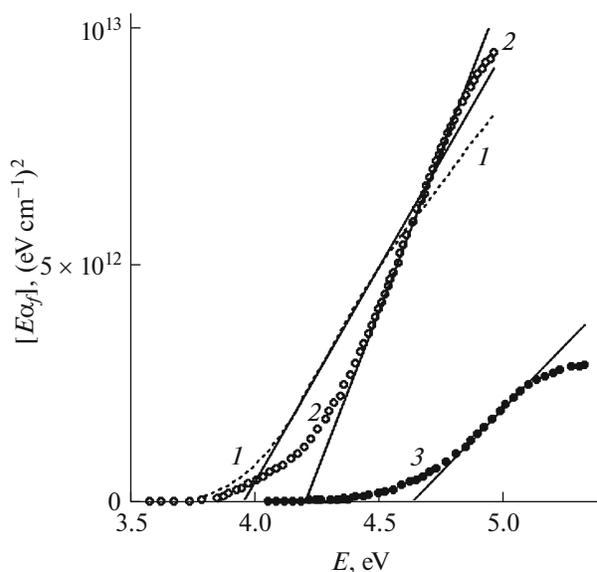
Let us determine the percentage of air and SrTiO<sub>3</sub> in films under study in the Bruggeman effective medium approximation. For this purpose, we assume that the epitaxial film does not contain air. Since the absorption coefficients of all the films under study in the visible range are close to zero, we find from their effective refractive indices (curves 1–3, Fig. 2a) that the single-layer film contains about 4% air (96% SrTiO<sub>3</sub>). Five-layer films obtained at different temperatures of thermal treatment contain about 33% air and 67% SrTiO<sub>3</sub>.

With an increase in the temperature of thermal treatment of five-layer films from 500 to 750°C, maximums of absorption bands those of the dispersion dependence  $n_f(\lambda)$  (curves 2, 3 in Fig. 2) increase and shift to the longwave region. The edges of the absorption bands also shift to the adequate extent.

To determine the forbidden band width of three SrTiO<sub>3</sub> films under study by absorption coefficients (curves 1–3, Fig. 2b), the absorption coefficients were calculated based on formula (1) and Tauc functions were constructed according to (6) (Fig. 3). It is seen from Fig. 3 that, for a single-layer film (curve 1),  $E_g = 3.96$  eV; for five-layer films thermally treated at temperatures of 750 and 500°C,  $E_g = 4.20$  and 4.63 eV, respectively (curves 2, 3).

Let us analyze possibilities of using the known approximate formulas (2)–(5) for estimating the forbidden band width of a single-layer strontium titanate film. At the angle of incidence of 8°, the increase in the light path length in the film and substrate can be neglected. For this reason,  $\alpha_f(\lambda)$  of a single-layer film is calculated with  $h_f = 33.6$  nm. Table 1 presents values of  $E_g$  (the first row of the table), which were calculated graphically based on approximate formulas (2)–(5) (the second row of the table) by the measured spectra  $T_f(\lambda)$  and  $R_f(\lambda)$  (curves 1, Fig. 1).

For a single-layer SrTiO<sub>3</sub> film, formula (3) allows one to obtain  $E_g$  value almost coinciding with that calculated based on expression (1). This can be explained by the fact that  $k_f(\lambda)$  is large in the region of the intrinsic absorption band of SrTiO<sub>3</sub> and, as a consequence, the light passes through the film not more than two times. Formula (2) allows one to estimate the minimum value of the forbidden band width, while formulas (4) and (5) allow one to determine its maximum value.



**Fig. 3.** Tauc functions for (1) a single-layer SrTiO<sub>3</sub> film and five-layer SrTiO<sub>3</sub> films thermally treated at (2) 750 and (3) 500°C.

Solutions of inverse problems of spectrophotometry (Fig. 2b) show that the studied strontium titanate films do not absorb light ( $k_f(\lambda) = 0$ ) in the region of  $380 < \lambda < 700$  nm. We considered the possibility of determining  $k_f(\lambda)$  of a single-layer film by absorption coefficient spectra which were calculated in a distinguished spectral region by approximate formulas (2)–(5) and (7). For this purpose, we obtained  $k_f(\lambda) = \lambda\alpha_f(\lambda)(4\pi)^{-1}$  from expression (1). Formula (2) leads to overestimated values of the absorption coefficient ( $k_f(\lambda) > 0$ ). Formulas (4) and (5) lead to negative values of  $k_f(\lambda)$  on some portions of the spectrum. In contrast to formula (3), minimum values of absorption coefficients can be obtained using formula (7). The corresponding spectra  $k_f(\lambda)$  are represented by curves 7 and 8 in the inset of Fig. 2b. In the region of the absorption band, the absorption coefficient can be estimated using formula (3).

Therefore, the layer-by-layer sol deposition onto quartz substrates, from one to five layers, leads to an increase in porosity of strontium titanate by 4–33%. This causes a decrease in the refractive index of the films from 2.33 to 1.87 in the middle portion of the spectrum (curves 1–4 in Fig. 2a). A decrease in the refractive index of five-layer films as compared to the refractive index of a single-layer film leads to

**Table 1.** Forbidden band width of a single-layer SrTiO<sub>3</sub> film

$E_g$ , eV	3.89	3.98	4.08	4.08	3.96
Formula	(2)	(3)	(4)	(5)	(1)

a decrease in their reflectance (curves 1–3, Fig. 1b) and to an increase in their transmittance (curves 1–3, Fig. 1a), respectively. The transparency of SrTiO<sub>3</sub> films in the visible portion of the spectrum is determined by the dispersion of the refractive index.

## CONCLUSIONS

The porosity of thin single-layer SrTiO<sub>3</sub> films obtained by the sol–gel method is low ( $\approx 4\%$ ). Their absorption band with a maximum  $k(252\text{ nm}) = 1.148$  is caused by direct band gap transitions of electrons to exciton levels in the forbidden band. Porosity of five-layer films is low ( $\approx 33\%$ ). If the temperature at which the five-layer films are heated is increased from 500 to 750°C, the absorption band broadens and its maximum increases from  $k(239\text{ nm}) = 0.603$  to  $k(253\text{ nm}) = 1.237$  and shifts to the longwave region. The bands are in the region of anomalous dispersion of refractive indices.

With an increase in the temperature of thermal treatment of five-layer films, the refractive index corresponding to the wavelength of the absorption band maximum increases from  $n(239\text{ nm}) = 1.730$  to  $n(253\text{ nm}) = 2.204$ . A decrease in the refractive index of multilayer films ( $n = 1.872$ ) in comparison with that of a single-layer film ( $n = 2.331$ ) in the region without absorption ( $\lambda = 632.8\text{ nm}$ ) leads to an increase in their transmittance or transparency.

The forbidden band width calculated strictly by the spectrum  $k_f(\lambda)$  of the film is within limits specified by known approximate formulas. The lower limit of  $E_g$  can be estimated by a formula which does not take into account the reflection of light from the film–substrate structure. The upper limit of quantity  $E_g$  is determined by formulas in which the reflection of light is taken into account twice. In the absorption band region, the best approximation ( $\delta E_g/E_g \approx 1\%$ ) to spectrum  $\alpha_f(\lambda)$  of a thin SrTiO<sub>3</sub> is provided by empirical formula (3), which takes into account a one-time reflection of light from the film–substrate structure. After sol–gel deposition of SrTiO<sub>3</sub> films, the forbidden band width increases from 3.96 to 4.63 eV.

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