

Investigation of optical properties of multilayer dielectric structures using prism-coupling technique

V.I. Sokolov, V.N. Glebov, A.M. Malyutin, S.I. Molchanova, E.V. Khaydukov, V.Ya. Panchenko

Abstract. A method based on resonant excitation of waveguide modes with a prism coupler is proposed for measuring the thickness and refractive index of thin-film layers in multilayer dielectric structures. The peculiarities of reflection of TE- and TM-polarised light beams from a structure comprising eleven alternating layers of zinc sulfide (ZnS) and magnesium barium fluoride (MgBaF₄), whose thicknesses are much less than the wavelength of light, are investigated. Using the mathematical model developed, we have calculated the coefficients of reflection of collimated TE and TM light beams from a multilayer structure and determined the optical constants and thicknesses of the structure layers. The refractive indices of the layers, obtained for TE and TM polarisation of incident light, are in good agreement. The thicknesses of ZnS and MgBaF₄ layers, found for different polarisations, coincide with an accuracy of $\pm 1\%$. Thus, we have demonstrated for the first time that the prism-coupling technique allows one to determine the optical properties of thin-film structures when the number of layers in the structure exceeds ten layers.

Keywords: multilayer dielectric structure, prism-coupling technique, measurement of thin-film parameters.

1. Introduction

The prism-coupling technique in frustrated total internal reflection (TIR) geometry has been successfully used to study optical properties of dielectric [1–3], polymer [4] and metal [5] films, as well as of transparent conducting coatings [6]. This technique allows one to determine the thickness of thin films with an accuracy of $\pm 1.5\%$ and their refractive index with an accuracy of $\pm 5 \times 10^{-4}$, which is comparable with the accuracy of TIR refractometers. This accuracy can be achieved at a large interaction length of excited waveguide modes having a layered structure, which can be several orders of magnitude greater than the film thickness. Recently, there have appeared a number of publications, greatly expanding the range of applicability of the technique. Thus, the possibility of measuring the parameters of ultrathin films using immersion liquids [7], determining the refractive index gradient in the thickness of the film [8] and measuring simultaneously thickness, refrac-

tive index and extinction coefficient of thin films [9] has been demonstrated. However, the method has been used hitherto only to study single-layer or two-layer structures.

There is presently a growing interest in the study of the optical properties of multilayer (including quantum-sized) dielectric and semiconducting structures containing up to several tens of layers. They are used in fabrication of interference mirrors and beam splitters for the UV, visible and near-IR wavelengths, photonic crystal fibres, etc. The number of alternating layers with high and low refractive indices in the multilayer structure is determined by the requirements to its spectral characteristics. For example, interference coatings consisting of 10–12 layers are optimal for the development of optical bandpass filters of width 20–30 nm, with a close-to-rectangular passband shape and transmittance of more than 90%.

The optical properties of such structures are traditionally studied by the methods of transmission spectroscopy and ellipsometry, each of which has its advantages and disadvantages. Thus, the use of spectroscopic ellipsometry allows one to accurately measure the thickness of thin films; however, accuracy of determining the refractive indices is rarely better than ± 0.005 [10].

In the present work we show that excitation of waveguide modes by a prism coupler makes it possible to determine optical parameters of thin-film structures, the number of layers in which is more than ten. At the heart of our proposed approach is the measurement of the reflection coefficient of a TE- or TM-polarised light beam from a layered structure in frustrated TIR geometry. The values of refractive indices, extinction coefficients and layer thicknesses are found by minimising the functional $\phi = \{N^{-1} \sum_{i=1}^N [R_{\text{exp}}(\theta_i) - R_{\text{thr}}(\theta_i)]^2\}^{1/2}$, where $R_{\text{exp}}(\theta_i)$ and $R_{\text{thr}}(\theta_i)$ are the experimentally measured and theoretically calculated reflection coefficients of a light beam from the boundary of a prism with a multilayer structure at an angle of incidence θ_i , and N is the number of experimental points. Estimates have shown that the accuracy of determining the refractive index of the layers by this method is $\pm 2 \times 10^{-3}$, the accuracy of measuring the layer thickness being $\pm 1\%$.

2. Interaction of a light beam with a multilayer dielectric structure

The scheme of excitation of waveguide modes by a prism coupler in a multilayer dielectric structure in frustrated TIR geometry is shown in Fig. 1. A plane electromagnetic wave E_{in} is incident at angle θ at the interface between a measuring prism and a layered structure from the side of the prism having a high refractive index N_p . Below we assume that absorp-

V.I. Sokolov, V.N. Glebov, A.M. Malyutin, S.I. Molchanova, E.V. Khaydukov, V.Ya. Panchenko Institute on Laser and Information Technologies, Russian Academy of Sciences, ul. Svyatoozerskaya 1, 140700 Shatura, Moscow region, Russia; e-mail: cbeta34@ya.ru, panch@laser.ru

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tion in the prism material is absent, and its permittivity is given by the expression $\varepsilon_p = N_p^2$. The multilayer structure is located on a substrate with a refractive index n_s . The gap between the structure and the prism has thickness h_{im} and can be filled with air or immersion liquid with a refractive index n_{im} . The permittivity of the immersion layer and the substrate are, generally speaking, complex quantities, which are given by the expressions $\varepsilon_{im} = (n_{im} + im_{im})^2$ and $\varepsilon_s = (n_s + im_s)^2$, where m_{im} and m_s are the extinction coefficients of the respective media.

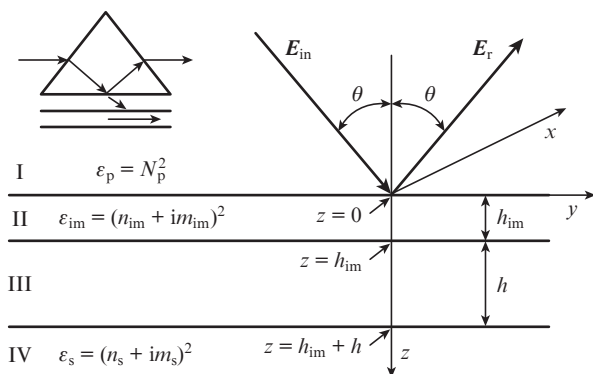


Figure 1. Scheme of excitation of waveguide modes in a multilayer structure using a prism coupler [ε_p , ε_{im} and ε_s are the permittivities of measuring prism I, immersion liquid (air) II and substrate IV, respectively; h_{im} is the thickness of the gap between structure III and the prism; h is the total thickness of the multilayer structure; and θ is the angle of incidence]. The inset shows the path of the laser beam in the measuring prism and its penetration into the film.

If the angle of incidence θ is larger than the critical one, we deal with the TIR of the beam from the prism–layered structure interface. However, when the thickness of the air gap between the prism and the structure is small (typically, 100–200 nm), at certain angles of incidence θ_m for which the synchronism condition $-N_p \sin \theta_m = \beta_m$ ($m = 0, 1, 2, 3, \dots$; β_m is the effective refractive index of the waveguide mode with the number m) – is fulfilled, the TIR condition is violated and light can penetrate into the structure, which leads to the excitation of an appropriate waveguide mode in it. Therefore, the angular dependence of the reflection coefficient $R(\theta)$ at $\theta = \theta_m$ has sharp and narrow dips (so-called dark m -lines). Note that in the case of TE and TM polarisations of the incident beam, different systems of waveguide modes, which are characterised by a different set of mode angles θ_m , are excited.

A dielectric structure comprising eleven alternating layers of two different materials is shown in Fig. 2. Layers I, III, V, VII, IX and XI have a thickness h_1 , permittivity $\varepsilon_1 = (n_1 + im_1)^2$, refractive index n_1 and extinction coefficient m_1 , and layers II, IV, VI, VIII and X – h_2 , $\varepsilon_2 = (n_2 + im_2)^2$, n_2 and m_2 ; h is the total thickness of the structure.

Monochromatic electromagnetic fields $\mathbf{E}(y, z, t) = \mathbf{E}(y, z) \exp(-i\omega t)$, $\mathbf{H}(y, z, t) = \mathbf{H}(y, z) \exp(-i\omega t)$ (ω is the frequency, t is the time) in measuring prism I (see Fig. 1) in the case of a TE-polarised incident plane wave can be written in the form

$$\mathbf{E}(y, z) = \mathbf{x}[E_{in}^s \exp(ik_y y + ik_z z) + R_a^s \exp(ik_y y - ik_z z)],$$

$$\mathbf{H}(y, z) = \mathbf{y} \frac{k_z}{k} [E_{in}^s \exp(ik_y y + ik_z z) - R_a^s \exp(ik_y y - ik_z z)] \quad (1)$$

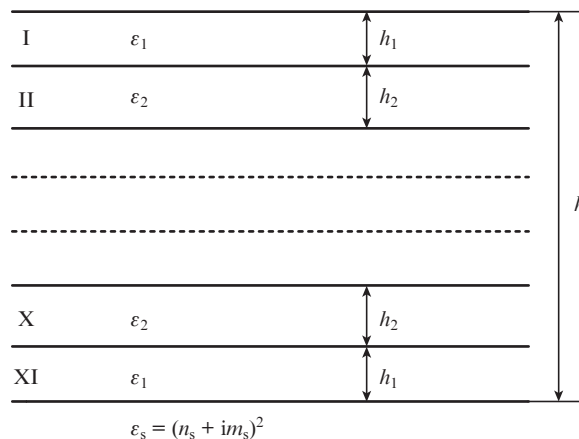


Figure 2. Thin-layer structure on a substrate with permittivity $\varepsilon_s = (n_s + im_s)^2$, comprising eleven (I, II, ..., X, XI) alternating layers of two different materials with permittivities ε_1 and ε_2 .

$$-z \frac{k_y}{k} [E_{in}^s \exp(ik_y y + ik_z z) + R_a^s \exp(ik_y y - ik_z z)],$$

where E_{in}^s is the amplitude of the incident wave; R_a^s is the amplitude of the reflected wave; $k_y = k N_p \sin \theta$; $k_z = k N_p \cos \theta$; $k = 2\pi/\lambda$; and λ is the wavelength of light in vacuum. In the case of a TM-polarised wave

$$\mathbf{E}(y, z) = -\mathbf{y} \frac{k_z}{k \varepsilon_p} [H_{in}^p \exp(ik_y y + ik_z z) - R_a^p \exp(ik_y y - ik_z z)]$$

$$+ z \frac{k_y}{k \varepsilon_p} [H_{in}^p \exp(ik_y y + ik_z z) + R_a^p \exp(ik_y y - ik_z z)], \quad (2)$$

$$\mathbf{H}(y, z) = \mathbf{x}[H_{in}^p \exp(ik_y y + ik_z z) + R_a^p \exp(ik_y y - ik_z z)].$$

Using the Fresnel formulas [11] we can easily derive expressions for the coefficients of specular reflection $R^s(\theta) = |R_a^s/E_{in}^s|^2$, $R^p(\theta) = |R_a^p/H_{in}^p|^2$ for an arbitrary number of layers [12, 13]. In the particular case of a structure of eleven layers (Fig. 2)

$$R^s(\theta) = \left| \left[\frac{k_z - i\gamma_i}{k_z + i\gamma_i} + A_s \exp(-2\gamma_i h_i) \right] \times \left[1 + \frac{k_z - i\gamma_i}{k_z + i\gamma_i} A_s \exp(-2\gamma_i h_i) \right]^{-1} \right|^2, \quad (3)$$

$$R^p(\theta) = \left| \left[\frac{\varepsilon_i k_z - i\varepsilon_p \gamma_i}{\varepsilon_i k_z + i\varepsilon_p \gamma_i} + A_p \exp(-2\gamma_i h_i) \right] \times \left[1 + \frac{\varepsilon_i k_z - i\varepsilon_p \gamma_i}{\varepsilon_i k_z + i\varepsilon_p \gamma_i} A_p \exp(-2\gamma_i h_i) \right]^{-1} \right|^2,$$

where $\gamma_i = (k_y^2 - k^2 \varepsilon_{im})^{1/2}$, and $\text{Re} \gamma_i \geq 0$. The explicit expression for the terms A_s and A_p , describing the resonant excitation of waveguide modes in a layered structure near the angles θ_m , are given in the Appendix. As can be seen from these formulas, $R^s(\theta)$ and $R^p(\theta)$ depend on the permittivities and thicknesses of the layers constituting a multilayer structure.

3. Fabrication of a multilayer structure and measurement of reflection coefficients $R^s(\theta)$ and $R^p(\theta)$ using a prism coupler

The dielectric structure consisting of eleven alternating layers of zinc sulfide (ZnS) and magnesium fluoride barium (MgBaF_4) was produced on a substrate of quartz glass. The structure was prepared using a VTC-900 PO vacuum facility by the method of resistive evaporation at a substrate temperature of 150°C and a chamber pressure of 7×10^{-6} mbar. The layers were deposited in automatic mode with a quartz control system of the condensation rate and thickness of the layers, the condensation rate being maintained with an accuracy better than 1% and the accuracy of fixing each layer thickness being better than 2%. This allowed us to minimise differences in the permittivities and thicknesses of ZnS and MgBaF_4 layers.

The coefficients of reflection of light from the multilayer structure were measured using a Metrikon 2010/M prism coupler [14], intended for determining the refractive index and thickness of thin films by the method of resonant excitation of waveguide modes. The coupler has a measuring prism made of zirconium oxide [ZrO_2 ; $N_p(\lambda = 632.8 \text{ nm}) = 2.15675$], which allows one to record waveguide modes with effective refractive indices β_m from 1.12 to 1.99. Moreover, the coupler makes it possible to measure the angular dependences of $R^s(\theta)$ and $R^p(\theta)$ with an increment $\Delta\theta = 0.001^\circ$ by scanning the sample with a collimated beam of a 632.8-nm helium–neon laser. According to the manufacturer's specifications, the accuracy of the measurement of the refractive index and thickness of single-layer films is ± 0.001 and $\pm 1.5\%$, respectively. For each polarisation we measured reflection spectra when the sample was in optical contact with the prism (in this case, one can observe dark m -lines in the angular dependence of the reflection coefficients) and when the sample was removed (m -lines are absent). To improve the accuracy, averaging was performed over several scans. The resulting reflection coefficients $R^s(\theta)$ and $R^p(\theta)$ were determined from the ratio of these spectra (Fig. 3). In the case of TE and TM polarisations of the incident beam we found in the structure three m -lines ($m = 0, 1, 2$) and two m -lines ($m = 0, 1$), respectively. Thus, at $\lambda = 632.8 \text{ nm}$, this multilayer structure supports three TE and two TM waveguide modes.

For the numerical simulation of $R^s(\theta)$ and $R^p(\theta)$ we developed computation programmes describing the reflection of plane TE and TM electromagnetic waves from a multilayer structure [see expressions (1)–(3)]. The optical parameters of thin film layers were found by minimising the functional

$$\phi = \sqrt{\frac{1}{N} \sum_{i=1}^N [R_{\text{exp}}(\theta_i) - R_{\text{thr}}(\theta_i)]^2}, \quad (4)$$

where $R_{\text{exp}}(\theta_i)$ and $R_{\text{thr}}(\theta_i)$ are experimentally measured and theoretically calculated reflection coefficients of a light beam from the interface of the prism with a multilayer structure at an angle of incidence θ_i . The adopted model assumes that the permittivities and thicknesses of all ZnS layers are equal; the same applies to MgBaF_4 layers. Therefore functional (4) was minimised over seven parameters: refractive indices n_1 and n_2 , extinction coefficients m_1 and m_2 and thicknesses h_1 and h_2 of zinc sulfide layers and magnesium fluoride barium layers, respectively, and the thickness h_{im} of the gap between the sample and the measuring prism. As an illustration, Fig. 4 shows the dependence of the residual functional ϕ on n_1 at

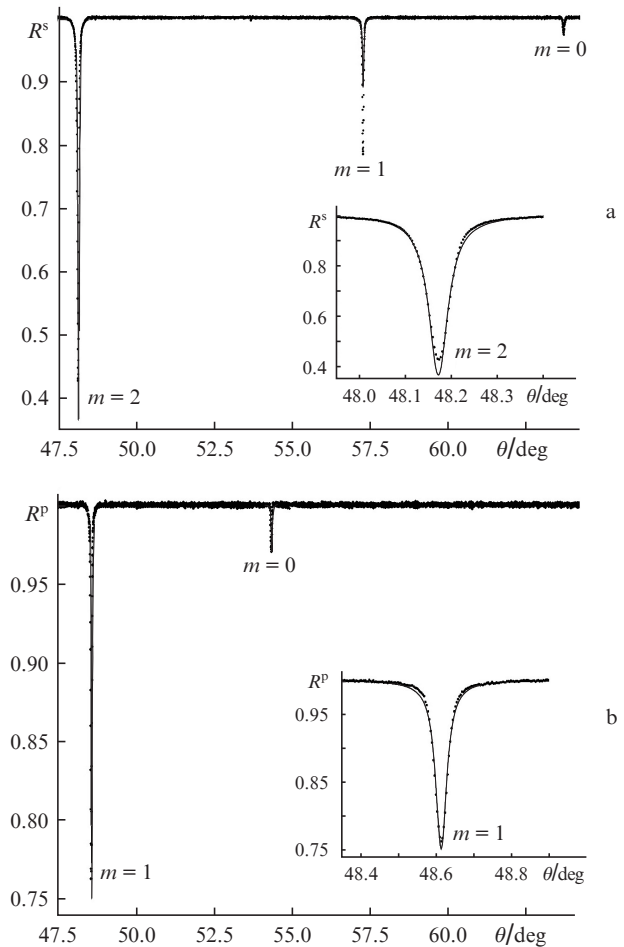


Figure 3. Measured (dots) and calculated (lines) dependences of specular reflection coefficients $R^s(\theta)$ and $R^p(\theta)$ (3) in the case of incidence of (a) a TE- and (b) TM-polarised laser beam at $\lambda = 632.8 \text{ nm}$ on a structure consisting of eleven alternating ZnS and MgBaF_4 layers; $m = 0, 1, 2$ are the numbers of m -lines in the order of decreasing the effective refractive index of waveguide modes. The insets show in an enlarged scale the dependences $R^s(\theta)$ and $R^p(\theta)$ near the m -lines with $m = 2$ (TE) and $m = 1$ (TM).

optimal values of the other parameters, the dependence being obtained for the case of TE polarisation. One can see that the

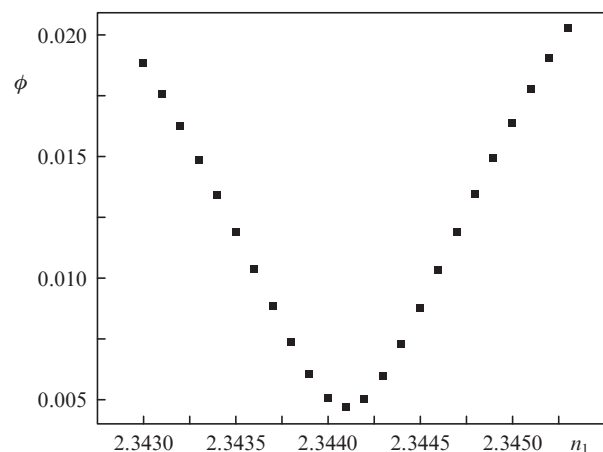


Figure 4. Dependence of the functional ϕ on n_1 at optimum values of the parameters m_1, h_1, n_2, m_2, h_2 and h_{im} , obtained for TE polarisation.

functional has a pronounced minimum, whose position can be determined with an accuracy of ± 0.0001 .

The values of n_1 , m_1 , h_1 , n_2 , m_2 and h_2 , at which the ‘residual’ ϕ between $R_{\text{thr}}(\theta)$ and $R_{\text{exp}}(\theta)$ is minimal, are the sought-for parameters for the given multilayer structure. The results of the calculation performed using expressions (3) are shown in Fig. 3 and summarized in Table 1.

Table 1. Refractive indices n_i , extinction coefficients m_i and layer thicknesses h_i ($i = 1, 2$) in a structure of eleven alternating ZnS and MgBaF₄ layers (see Fig. 2), calculated for the case of TE and TM polarisations of an incident light beam with a wavelength of 632.8 nm.

Polarisation	n_1	m_1	h_1/nm	n_2	m_2	h_2/nm
TE	2.3441	0.0007	55.0	1.4904	0.0001	57.4
TM	2.3496	0.0005	56.0	1.4948	0.0004	56.6

As seen from Table 1, the averaged refractive index $\langle n_1 \rangle$ of ZnS layers, found for the case of TE and TM polarisations of the incident beam, is 2.3469 ± 0.0027 and close to the refractive index n of bulk zinc sulfide (632.8 nm) = 2.3505 [15]. The averaged refractive index of MgBaF₄ layers is $\langle n_2 \rangle = 1.4926 \pm 0.0022$, which is close to the value 1.4715 obtained in paper [16]. Some difference in refractive indices, we measured for TE and TM polarisations, can be due to the anisotropy of thin film layers in the structure. The averaged thicknesses of ZnS and MgBaF₄ layers, measured for different polarisations, are $\langle h_1 \rangle = 55.5 \pm 0.5$ nm and $\langle h_2 \rangle = 57.0 \pm 0.4$ nm, i.e., the difference is less than $\pm 1\%$. This confirms the efficiency of the proposed method and the validity of the chosen model. Thus, we have demonstrated the possibility of determining the optical properties of multilayer thin-film structures using a prism coupler when the number of layers in the structure is more than ten.

Note good agreement of the extinction coefficients of ZnS and MgBaF₄ layers, obtained for TE and TM polarisations of the incident beam. Previously, we have demonstrated that the method of excitation of waveguide modes allows one to simultaneously measure the refractive index, extinction coefficient and thickness of single-layer films [9]. This possibility is due to high sensitivity of the angular width of the m -line to the extinction coefficient of the film material.

Let us estimate the accuracy of determining the optical constants and thicknesses of layers in thin-film structures by the prism-coupling technique. It depends both on the experimental accuracy of $R^s(\theta)$ and $R^p(\theta)$ measurements and on the structure model used in the inverse problem. The accuracy of $R^s(\theta)$ and $R^p(\theta)$ measurements using a prism coupler is $\pm 0.5\%$ or better, the accuracy of fixing the angle of incidence being equal to 0.001° . With regard to the models used, they can be simple (e.g., models of uniform and isotropic layers) or complex (models taking into account the thickness nonuniformity of the layers, the presence of transition layers at interfaces, etc.). In this case, additional information about the structure is often required, and the accuracy is evaluated by comparing the results obtained by independent methods. Thus, in the present study, we used TE and TM polarisation of the incident light, resulting in the excitation of two different systems of waveguide modes in a multilayer structure. The comparison of the results allows us to make a conclusion that the accuracy of determining the refractive index and extinction coefficient of the proposed method is $\pm 2 \times 10^{-3}$ and $\pm 2 \times 10^{-4}$, respectively, and the accuracy of determining the layer thickness is $\pm 1\%$. It should be noted that in the proposed approach

the optical parameters of thin-film structures are reconstructed by using a very large amount of data on $R^s(\theta)$ and $R^p(\theta)$, including reflection of near resonances (associated with the excitation of waveguide modes) and away from them.

Thus, we have proposed a method for measuring optical properties of multilayer dielectric structures, which is based on resonant excitation of waveguide modes in frustrated TIR geometry. We have determined refractive indices and thicknesses of layers in a structure consisting of eleven alternating ZnS and MgBaF₄ layers. Refractive indices and thicknesses of the layers, found in the case of TE and TM polarisations of the incident beam, are in good agreement. The method can be used to analyse structures with a greater number of layers if appropriate modifications are introduced to the mathematical model.

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Appendix

The expressions for the coefficients A_s and A_p in formula (3) have the form:

$$\begin{aligned}
 A_s &= \frac{a_s + B_s \exp(-2\gamma_1 h_1)}{1 + a_s B_s \exp(-2\gamma_1 h_1)}, & B_s &= \frac{b_s - C_s \exp(-2\gamma_2 h_2)}{1 - b_s C_s \exp(-2\gamma_2 h_2)}, \\
 C_s &= \frac{b_s - D_s \exp(-2\gamma_1 h_1)}{1 - b_s D_s \exp(-2\gamma_1 h_1)}, & D_s &= \frac{b_s - E_s \exp(-2\gamma_2 h_2)}{1 - b_s E_s \exp(-2\gamma_2 h_2)}, \\
 E_s &= \frac{b_s - F_s \exp(-2\gamma_1 h_1)}{1 - b_s F_s \exp(-2\gamma_1 h_1)}, & F_s &= \frac{b_s - G_s \exp(-2\gamma_2 h_2)}{1 - b_s G_s \exp(-2\gamma_2 h_2)}, \\
 G_s &= \frac{b_s - H_s \exp(-2\gamma_1 h_1)}{1 - b_s H_s \exp(-2\gamma_1 h_1)}, & H_s &= \frac{b_s - I_s \exp(-2\gamma_2 h_2)}{1 - b_s I_s \exp(-2\gamma_2 h_2)}, \\
 I_s &= \frac{b_s - J_s \exp(-2\gamma_1 h_1)}{1 - b_s J_s \exp(-2\gamma_1 h_1)}, & J_s &= \frac{b_s - K_s \exp(-2\gamma_2 h_2)}{1 - b_s K_s \exp(-2\gamma_2 h_2)}, \\
 K_s &= \frac{b_s - c_s \exp(-2\gamma_1 h_1)}{1 - b_s c_s \exp(-2\gamma_1 h_1)}, \\
 A_p &= \frac{a_p + B_p \exp(-2\gamma_1 h_1)}{1 + a_p B_p \exp(-2\gamma_1 h_1)}, & B_p &= \frac{b_p - C_p \exp(-2\gamma_2 h_2)}{1 - b_p C_p \exp(-2\gamma_2 h_2)}, \\
 C_p &= \frac{b_p - D_p \exp(-2\gamma_1 h_1)}{1 - b_p D_p \exp(-2\gamma_1 h_1)}, & D_p &= \frac{b_p - E_p \exp(-2\gamma_2 h_2)}{1 - b_p E_p \exp(-2\gamma_2 h_2)}, \\
 E_p &= \frac{b_p - F_p \exp(-2\gamma_1 h_1)}{1 - b_p F_p \exp(-2\gamma_1 h_1)}, & F_p &= \frac{b_p - G_p \exp(-2\gamma_2 h_2)}{1 - b_p G_p \exp(-2\gamma_2 h_2)}, \\
 G_p &= \frac{b_p - H_p \exp(-2\gamma_1 h_1)}{1 - b_p H_p \exp(-2\gamma_1 h_1)}, & H_p &= \frac{b_p - I_p \exp(-2\gamma_2 h_2)}{1 - b_p I_p \exp(-2\gamma_2 h_2)}, \\
 I_p &= \frac{b_p - J_p \exp(-2\gamma_1 h_1)}{1 - b_p J_p \exp(-2\gamma_1 h_1)}, & J_p &= \frac{b_p - K_p \exp(-2\gamma_2 h_2)}{1 - b_p K_p \exp(-2\gamma_2 h_2)},
 \end{aligned}$$

$$K_p = \frac{b_p - c_p \exp(-2\gamma_1 h_1)}{1 - b_p c_p \exp(-2\gamma_1 h_1)},$$

where

$$a_s = \frac{\gamma_i - \gamma_1}{\gamma_i + \gamma_1}, \quad b_s = \frac{\gamma_1 - \gamma_2}{\gamma_1 + \gamma_2}, \quad c_s = \frac{\gamma_1 - \gamma_s}{\gamma_1 + \gamma_s};$$

$$a_p = \frac{\varepsilon_1 \gamma_i - \varepsilon_i \gamma_1}{\varepsilon_1 \gamma_i + \varepsilon_i \gamma_1}, \quad b_p = \frac{\varepsilon_2 \gamma_1 - \varepsilon_1 \gamma_2}{\varepsilon_2 \gamma_1 + \varepsilon_1 \gamma_2}, \quad c_p = \frac{\varepsilon_s \gamma_1 - \varepsilon_1 \gamma_s}{\varepsilon_s \gamma_1 + \varepsilon_1 \gamma_s};$$

$$\gamma_1 = (k_y^2 - k^2 \varepsilon_1)^{1/2}; \quad \operatorname{Re} \gamma_1 \geq 0;$$

$$\gamma_2 = (k_y^2 - k^2 \varepsilon_2)^{1/2}; \quad \operatorname{Re} \gamma_2 \geq 0;$$

$$\gamma_s = (k_y^2 - k^2 \varepsilon_s)^{1/2}; \quad \operatorname{Re} \gamma_s \geq 0; \quad i = 1, 2.$$

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