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Surface polariton excitation in ZnO films deposited using ALD

E.F. Venger[1], L.Yu. Melnichuk[2], A.V. Melnichuk[2], T.V. Semikina[1]
[1]*V. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine,*
41, prospect Nauky, 03680 Kyiv, Ukraine,
[2]*Mykola Gogol State Pedagogic University,*
2, Kropyv'yanskogo str., 16600 Nizhyn, Ukraine
E-mail: mov310@mail.ru

Abstract. The conductive ZnO films deposited using atomic layer deposition (ALD) on the optical glass substrates were studied using the modified method of the disturbed total internal reflection within the range 400…1400 cm^{-1} for the first time. The frequency "windows" with the obtained excited surface phonon and plasmon-phonon polaritons have been found in the measured infrared reflectance spectra. The dispersion response of high and low frequency branches of the IR spectra have been presented.

Keywords: disturbed total internal reflection, surface phonon, plasmon-phonon polariton, conductive ZnO film.

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1. Introduction

Thin zinc oxide films deposited on the dielectric and semiconductor substrates are attractive material for researchers in the whole world due to their unique properties such as high photosensitivity, high yield of photo and cathodoluminescence, availability of pyro- and piezoelectric effects, *etc.* These films are the base for different optical and acoustelectronic devices that are constructed on the ground of surface wave usage [1]. The advantages of these devices based on high work efficiency in a wide frequency range, diminutiveness and possibility of integration with other microelectronics elements [2-4].

Among the numerous technological methods used to deposit ZnO (pulse laser deposition, radio-frequency magnetron sputtering, *etc*.), the atomic layer deposition method (ALD) demonstrates several advantages (see, e.g., [5-7]). For example, the ALD technology permits the deposition of ZnO films very homogeneous in thickness and with good structural properties on the very big substrates. The films are highly transparent and conductive. One from the main advantages of this technological method is providing the process under low temperature (100…200 °C) using cheap precursors, which results in reduction of costs for microelectronic devices. Moreover, it is possible to get conductive films even without doping. That is why ZnO films obtained by ALD are chosen for further experimental researches.

The structures of air – ZnO film – optical glass were investigated using the method of infrared reflection spectroscopy by us in the works [8, 9], and we demonstrated ability of good simulation of reflection coefficient in the range of "residual" rays under usage of bulk mutually agreed parameters for single oscillatory mathematical model that was presented for single crystal of ZnO in [10, 11].

The fulfilled research corroborated suitability of the IR-spectroscopy method for determination of ZnO electrophysical features as well as the degree of surface texturization. On the base of dispersion analyzes of studied structures, it was shown that non-destructive methods of IR-spectroscopy enable to obtain electron concentration, mobility and specific conductivity of the studied samples within experimental errors without destroying them. Moreover, the obtained results allowed calculation the spectrum of dielectric permittivity in the whole IR spectrum range and identify gaps with negative permittivity. As is known [11, 12] exactly in the frequency range where the permittivity of one from the bordering medium has the negative value, the propagation of surface waves of TM-type is possible along the plane boundary of media. The field amplitude of TM-type waves decrease exponentially along the normal direction from the border separation environment. The corresponding excitation for these waves is called as the surface polaritons (SP) [11, 12].

However, despite the huge number of publications devoted to ZnO films, there are no data in literature concerning the study of interaction between electromagnetic radiation and different types of oscillations (for example phonons, plasmons, *etc*.) and conditions of excitation and propagation of the surface polaritons in thin polycrystalline transparent conductive ZnO films deposited on optical glass.

This paper presents experimental and theoretical studies of the ranges of possible existence, stimulation and propagation of surface polaritons in thin polycrystalline transparent conductive ZnO films deposited on optical glass substrates. The series of ZnO films with different concentration of free charge carriers (electrons) and film thickness were investigated.

2. Technology of ZnO films deposition and conditions of experiments

Thin transparent conductive ZnO films were grown on the optical glass substrates in the Savannah-100 ALD reactor (Cambridge NanoTech). ZnO films were grown from diethylzinc and water vapor as zinc and oxygen precursors, respectively. More detail information about technology of these films has been presented in [5-7]. In these works, technological regimes for deposition of the films with good structural properties were determined. For example, X-ray diffraction (XRD) measurements showed that the growth mode of the ZnO films (e.g., the c-axis perpendicular or parallel to the surface) and grain diameters depend on purging time [5]. In the case of samples deposited with short purging times, the main diffraction peak corresponds to the [10.0] direction, which indicates the growth mode with c-axis parallel to the substrate. In turn, for the layers prepared with an increased purging time, the c-axis is perpendicular to the surface and the [00.2] XRD peak dominates the patterns, especially at higher growth temperature (200 °C). The lattice parameters (*c* and *a*) approach the value of lattice parameters of the bulk ZnO crystal with an increase of a growth temperature [5]. Surface morphology was investigated using atomic force microscopy (AFM). The authors found that roughness clearly depends on a growth temperature (100 to 200 °C), thickness of the film and purging time. The range of roughness values was from 0.9 up to 3.9 nm. On the base of previously obtained technological regimes, we deposit two series of conductive ZnO films.

The ZnO films for both series (set III-1, III-2 and set 312-1, 312-2, 312-3) were grown at the temperature 200 °C, which results in film growth with lattice parameters of bulk ZnO crystal, c-axis is perpendicular to the surface and films have high conductivity. Inside each series, the films had the same purging time and pulse durability with different number of cycles. Varying the number of cycles supports the film growth with different thickness. The series differ from each other by durability of pulse and purging, which leads to some structural variations. The prepared films have different thicknesses that were measured by Mikropack Nanocalc 2000 reflectometer.

The electrical measurements (Hall effect studies) of our samples show that the electron concentration (n) varied between 10^{19} to 10^{20} cm^{-3}. The fact that even undoped films have very low resistivity, namely $4\cdot10^{-3}$ Ohm·cm, is explained by the presence of native defects, mainly zinc interstitial which can act as a very efficient donor in ZnO.

To investigate surface polaritons of phonon and plasmon-phonon types in the doped ZnO films, it was used the isolators from optical glass that is the most convenient among the substrates because doesn't have any oscillation in infrared spectral range [8, 9].

Spectral measurements of disturbed total internal reflection (DTIR) were fulfilled at room temperature by using the spectrophotometer ИКС-31 with the attachment for reflection НПВО-2 and reference mirror in the frequency range 400…1400 cm^{-1}. The spectra I(н)/I_0(н) were recorded with a polarizer, the degree of polarization was $P = 0.98$. The DTIR coefficient value was obtained with the accuracy of 1…2%. The I(н)/I_0(н) measurements were carried out at $T = 300$ K.

3. The theory and analysis of DTIR spectra

As known, one from the non-destructive research methods for studying the optical and electrophysical properties of thin ZnO films, which allows controlling their quality and structural perfection, is the polariton spectroscopy method [11]. This method gives information not only about the physical and chemical film properties, but also parameters of the substrates and their surface quality.

The DTIR method content is based on [11, 12]: under condition of total internal reflection the ray of IR radiation is able to penetrate from transparent in the IR-range more optical dense medium into less optical dense medium. The depth of penetration is commensurable

with the IR-range incident wavelength. Under these conditions, if the optically less dense medium has the absorption, the light intensity that penetrates into studied medium increases and reflectance is not total. Keep in mind that the studied medium has strong absorption in the given IR-range. Under this condition, the spectral instruments record the spectrum of disturbed total internal reflection.

The area of ZnO residual rays is placed in the range between frequencies of transverse and longitudinal optical phonon. Concerning the fact that ZnO films are polycrystalline, we use the obtained interconsistentive parameters of single oscillation model for the ZnO single crystal at orientation $E \perp C$ (Table 1).

Table 1. Mutually agreed parameters of single oscillation model for monocrystalline ZnO at orientation $E \perp C$ [2, 14].

e_0	e_∞	$н_T$, $см^{-1}$	$н_L$, $см^{-1}$
8.1	3.95	412	591

More detail methods for obtaining mutually agreed parameters for single oscillation model is described in [10, 11, 13]. The authors [10] showed that ZnO is characterized by considerable anisotropy of the phonon properties subsystem and weak anisotropy of the plasma subsystem. Due to this fact, the zinc oxide films are a good model object that is convenient to study anisotropy of optical and electrical properties in the IR-spectrum under the condition of presence of long-wave optical lattice oscillations with electron plasma.

Calculation were performed using the formulae that take into account interaction of infrared radiation with plasma and phonon subsystems in the ZnO film for the case $E \perp C$ on the "semi-infinite" optical glass substrates by using the mathematical processor MATCAD:

$$R(\nu,\phi) := \left(\left| \frac{1 + i \cdot P(\nu,\phi)}{1 - i \cdot P(\nu,\phi)} \right| \right)^2$$

$$P(\nu,\phi) := \frac{\beta 2(\nu,\phi)}{\beta 1(\nu,\phi)} \times$$

$$\times \frac{\beta 3(\nu,\phi) \cdot A(\nu,\phi) + \beta 2(\nu,\phi) \cdot \tanh(\kappa 2(\nu,\phi) \cdot 2 \cdot \pi \cdot d)}{\beta 2(\nu,\phi) + \beta 3(\nu,\phi) \cdot A(\nu,\phi) \cdot \tanh(\kappa 2(\nu,\phi) \cdot 2 \cdot \pi \cdot \nu \cdot d)}$$

$$A(\nu,\phi) := \frac{\beta 4(\nu,\phi) + \beta 3(\nu,\phi) \cdot \tanh(\kappa 3(\nu,\phi) \cdot 2 \cdot \pi \cdot \nu \cdot 1)}{\beta 2(\nu,\phi) + \beta 4(\nu,\phi) \cdot \tanh(\kappa 3(\nu,\phi) \cdot 2 \cdot \pi \cdot 1 \cdot \nu)}$$

$$\beta 1(\nu,\phi) := \frac{\varepsilon 1}{\kappa 1(\nu,\phi)} \qquad \beta 2(\nu,\phi) := \frac{\varepsilon 2}{\kappa 2(\nu,\phi)}$$

$$\beta 3(\nu,\phi) := \frac{\varepsilon 3(\nu,\phi)}{\kappa 3(\nu,\phi)} \qquad \beta 4(\nu,\phi) := \frac{\varepsilon 4(\nu,\phi}{\kappa 4(\nu,\phi)}$$

$$\kappa 1(\nu,\phi) = \sqrt{\varepsilon 1}\cos(\phi)$$

$$\kappa 2(\nu,\phi) = \sqrt{(\kappa x(\nu,\phi))^2 - \varepsilon 2}$$

$$\kappa 3(\nu,\phi) = \sqrt{(\kappa x(\nu,\phi))^2 - \varepsilon 3(\nu,\phi)}$$

$$\kappa 4(\nu,\phi) = \sqrt{(\kappa x(\nu,\phi))^2 - \varepsilon 4(\nu,\phi)}$$

$$\kappa x(\nu,\phi) = \sqrt{\varepsilon 1}\sin(\phi)$$

The signs 1 to 4 designate, respectively, the DTIR prism, vacuum gap thickness d_g, ZnO semiconductor film with the thickness d_f and "semi-infinite" optical glass substrate; $\varepsilon_3(\nu)$ – film dielectric constant, which takes into account the additive contribution of active optical phonons ν_T and plasmons ν_p in the ZnO thin films:

$$\varepsilon_3(\nu) = \varepsilon_{\infty 3}\left(1 + \frac{\nu_{т3}^2(\varepsilon_{03} - \varepsilon_{\infty 3})}{\nu_{т3}^2 - \nu^2 - i\nu\gamma_{\phi 3}} - \frac{\nu_{p3}^2}{\nu(\nu + i\gamma_{p3})}\right);$$

$\varepsilon_4(\nu)$ is the dielectric constant of optical glass substrate; all the other symbols are generally accepted.

The mutually agreed ZnO parameters presented in Table 1 are used at DTIR spectrum calculation. The calculations were carried out using the data on the thickness and concentration of free charge carriers and presented in Table 2. Regarding the plasmon damping coefficient in ZnO films, the calculation of all the curves were performed under the condition $\nu_p = \gamma_p$. This

Table 2. Electrophysical parameters of conductive ZnO films obtained by ALD.

N	Sample	Film thickness, (nm)	Concentration of carriers, ($см^{-3}$)	Plasma frequency, ($см^{-1}$)	Mobility, ($см^2/V \cdot s$)	Conductivity, ($Ohm^{-1}см^{-1}$)
1	ZnO/glass (S312-1)	220	$1.5 \cdot 10^{20}$	1140	24	$1.73 \cdot 10^3$
2	ZnO/glass (S312-2)	220	$7.35 \cdot 10^{19}$	2530	23.3	$3.65 \cdot 10^2$
3	ZnO (III-2)	203.2	$1.3 \cdot 10^{20}$	3370	24.8	$1.94 \cdot 10^3$
4	ZnO (III-1)	380.8	$1.42 \cdot 10^{20}$	3520	26.5	$1.66 \cdot 10^3$
5	ZnO/glass (S312-3)	720	$4.33 \cdot 10^{20}$	6145	25.7	$5.6 \cdot 10^3$

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coefficient for the phonon subsystem was taken as 15 cm^{-1} for all the samples. As it was shown in [8, 9], the best matching the theory and experimental data could be obtained under the noted parameters. Inaccuracy is not more than 3%.

Fig. 1 (a, b, c (lines *1*–*3*)) shows the calculated frequency dependence of DTIR coefficient for the ZnO film on an optically isotropic optical glass substrate. The film parameters, under which the calculation is done, are presented in Tables 1 and 2 under the stipulation that the used orientation is $E \perp C$. The curves *1*–*3* corresponds to the film depth d = 22 мм at the free charge carrier concentration (electrons) $1.5 \cdot 10^{19}$ cm^{-3} (curve *1*), $7.35 \cdot 10^{19}$ cm^{-3} (curve *2*), $1.3 \cdot 10^{20}$ cm^{-3} (curve *3*).

The theoretical calculation were carried out under the condition that the value of damping coefficient for the phonon subsystem is r_{ph} = 15 cm^{-1}, gap thickness in DTIR prism d_g = 5 μm and the IR-radiation incident angle in the prism $\phi = 40°$ is constant for curves *1*–*3* (Fig. 1a). As shown in Fig. 1, the electron concentration altering in ZnO films under constant film thickness and phonon damping coefficient is accompanied by increasing the "halfwidth" of DTIR spectrum. This tendency is observed for all the samples. Thus, in Fig. 1a, the "halfwidth" of spectrum increases from 33 (curve *1*) up to 75 cm^{-1} (curve *3*). According to the data of [13], this fact indicates the increase of surface polaritons damping coefficient in ZnO films, if the film is doped and the doping level increases. A similar tendency is observed also for the curves *1*–*3* that are presented in Figs. 1b and 1c, which are given for abovementioned parameters, but at r_{ph} = 30 cm^{-1} (Fig. 1b) and r_{ph} = 45 cm^{-1} (Fig. 1c). In Fig. 1 (curve *1*), it is clearly visible increasing of the "halfwidth" inherent to DTIR spectrum from 33 to 69 cm^{-1}, when the phonon subsystem damping coefficient increases from 15 up to 45 cm^{-1}.

Validation of the fact that surface polariton is excited in the system ZnO – optical glass is the minimum seen in the DTIR spectrum at only *p*-polarized IR-radiation. The second validation belongs to the range of negative values of dielectric permittivity (the range between frequency of transverse and longitudinal optical phonon). Moreover, the DTIR minimum spectrum shifts to the high frequency range when increasing the incident light angle in the DTIR prism and decreasing the absorption intensity under the same distance between the DTIR prism and ZnO – optical glass system [11, 12].

Fig. 2 shows the DTIR spectrum inherent to the structure ZnO – optical glass with the different film thickness values (from 0.22 up to 0.72 μm). Theoretical spectra for the curves *1* to *3* were obtained at an angle of incidence light $\phi = 40°$ in the DTIR element. The minima in these spectra were observed at the frequencies 496 (curve *1*), 504 (curve *2*), 513 cm^{-1} (curve *3*). As it is seen from the figure, under condition of the film thickness increase the surface polariton frequency shift to the high frequency range and "halfwidth" expansion are observed. This fact could be explained by the influence of ZnO film phonon subsystem on SP spectrum within the range of zinc oxide "residual rays". In case of ZnO films with larger thickness, we observe that minima in DTIR spectra, which belong to ZnO film surface polariton, are shifted from 73 down to 64 cm^{-1} (see Fig. 2).

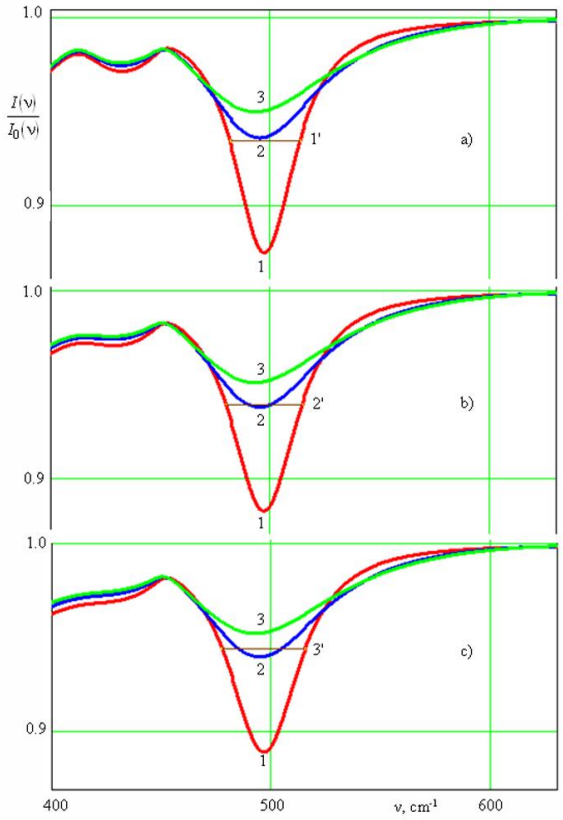

Fig. 1. DTIR theoretical spectra of conductive ZnO films deposited on optical glass within the range of zinc oxide "residual rays".

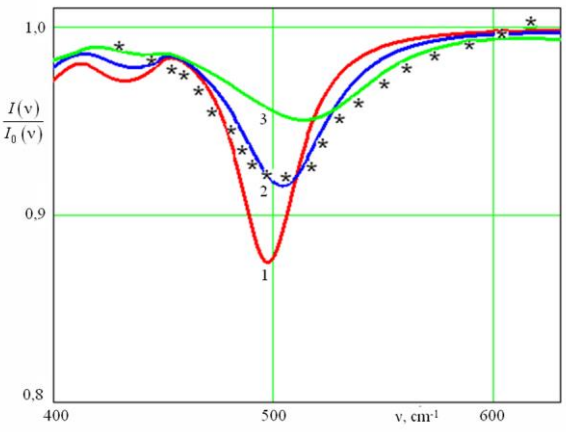

Fig. 2. DTIR spectra of ZnO – optical glass structure at d_f = 0.22 μm (*1*); d_f = 0.38 μm (*2*); d_f = 0.72 μm (*3*); * – experiment (d_f = 0.38 μm).

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The points depicts the DTIR experimental spectrum of ZnO – optical glass system for the sample with the thickness d_f = 0.38 μm of type of semiconductor film on isolator "semi-infinite" at orientation $K \perp C$, $xy \parallel C$. The spectrum is recorded at air space between the DTIR element with KRS-5 and the sample with the thickness d = 5 μm and angle ϕ = 40° in the DTIR element. In this spectrum, we observe a minimum at the frequency 508 cm^{-1}, that is matched with calculated data within the measurement error (504 cm^{-1}, curve *2*).

Obviously, the most accurate data could be obtained via construction of the so-called DTIR reflection surface $I(\nu)/I_0(\nu)$, which is tree-dimensional representation of the transmission coefficient for the abovementioned system. This reflection surface depends on radiation frequency and incident angle. In the case of absence of interaction with surface of the structure, the value $I(\nu)/I_0(\nu) = 1$ and the surface is field in this area. But if the excitation of surface polariton in ZnO film or optical glass substrate exists, then a number of "cavities" appears on the mentioned surface (Fig. 3).

The depth of "cavities" depends on system parameters: value of gap d_g between DTIR semi-cylinder and sample, radiation frequency н and so on. To confirm the existence of surface polariton in the structure of ZnO – optical glass be the fact that at increasing of incident angle we observe shifting of frequency minimum in TDIR spectrum (see Fig. 2) into direction of higher frequency range. We also observe decrease in the spectrum "halfwidth".

From the above presented theoretical calculations and analysis of monograph data [11] for ZnO/Al_2O_3 structure, we coul conclude that the pattern of change in the dispersion of polariton branches does not qualitatively change at ZnO film thickness altering. But we observe the quantitative change. It could be suggest that these quantitative changes are caused by increasing the values of permittivity effective part in the infrared spectral range.

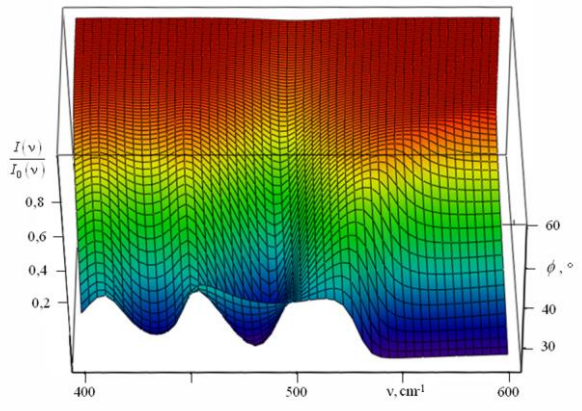

Fig. 3. The surface of DTIR modification system of ZnO film – optical glass at $E \perp C$.

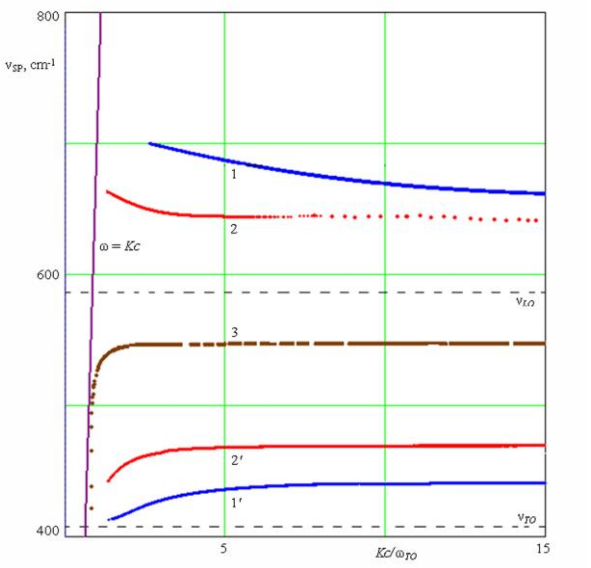

Fig. 4. The surface plasmon frequency dependence on the wave vector K of the ZnO film – optical glass system at $E \perp C$ under different values of film thickness d_f.

Fig. 4 shows the high-frequency (curves *1*, *2*) and low frequency (curves *1'*, *2'*) dispersed branches for ZnO film thickness 0.22 and 0.72 μm, respectively. Occurrence of dispersion curves is observed within the frequency range of "residual rays" for the ZnO film (412…591 cm^{-1}) on the optical glass substrate. It is in this range that the real part of the dielectric constant of ZnO film is negative, and the energy losses are infinitely small. Calculation of the dispersion branches in the system ZnO – optical glass are carried out using the formulae without account of anisotropy fluctuations in the film [14, 15]. We see that in such a system at $d_f \leq 5$ μm, two branches are observed. Similar results were obtained earlier in [14] for the system ZnO/Al_2O_3. The solution of equations can be represented by low-frequency (curves *1'*, *2'*) and high frequency (curves *1*, *2*) dispersed branches.

From the analysis of the DTIR spectrum (Figs. 1 to 3) and the dispersion branches (Fig. 4), plotted for different film thicknesses, it follows that the smaller the film thickness, the greater will be the distance between minima in the DTIR spectrum, and therefore between the dispersion branches.

As seen from the above graphs, the high-frequency branch at $d_f \leq 0.22$ μm for zinc oxide film with the high concentration of free electrons ($n_0 = 1.5 \cdot 10^{20}$ cm^{-3}) is located higher then the frequency of longitudinal optical phonons in zinc oxide. Calculations show that with increasing the ZnO film thickness both branches are close to each other. At the film thickness $d_f \geq 5$ μm, the curves are almost degenerate into one curve (Fig. 4, curve *3*), which coincides with the data of [11] for single crystal ZnO.

4. Conclusions

Thus, this paper experimentally and theoretically studies optical spectra of conductive ZnO films with a high concentration of free electrons, which were deposited on optically isotropic glass substrates. It has been shown the spectrum dependence on the frequency within the "residual rays" range of ZnO films and substrates, and presence of the frequency "windows" where excitation and propagation of surface polaritons are possible. For the first time, for a specified structure three-dimensional representation of DTIR spectra have been made under conditions of simultaneous scanning the incidence angle and frequency of infrared radiation. We obtain the dispersion relations for high- and low-frequency branches.

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