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Ellipsometric characterization of VO_2, $VO_2:Mg$, $VO_2:Ge$ nanocrystalline films

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The spectra of the refractive index $n(\lambda)$ and the extinction coefficient $k(\lambda)$ of thin VO2, $VO_2:Mg$, $VO_2:Ge$ films were measured using the ellipsometric method. For an undoped VO_2 film at a wavelength $\lambda = 632.8$ nm, near the insulator-metal phase transition, the $n(T)$ and $k(T)$ thermal hysteresis loops were studied. An interpretation of the results is given on the base of the Moss relation, the idea of a change in $n(T)$ and $k(T)$ with an impurity variation of the material density, and also on the base of the ideology of the Coulomb transformation of the density of states function in strongly correlated materials.

Keywords: ellipsometry, vanadium dioxide, insulator-metal phase transition, strongly correlated materials.

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1. Introduction

Study of vanadium dioxide films is justified from both fundamental and application points of view [1–3]. Large attention to study of the fundamental mechanism of insulator–metal phase transition (IMPT) in monocrystals and vanadium oxide films is caused by unusualness of the results, obtained for vanadium dioxide (VO_2) films — the main oxide of Magneli's series [4]. Unusualness of the results appears due to presence of strong interelectronic correlations in vanadium oxides. Novelty of the obtained data is related to usage of high-information modern study methods: photonic crystals spectroscopy with VO_2-filling [5], pico- and femtosecond spectroscopy [6], near-field optical microscopy along with atomic force microscopy [7–9], as well as EXAFS methods [10].

Applicational significance of studies of VO_2 films is in properties uniqueness of this material: record-breaking (femtosecond) response time, that allows to use it as high-speed optical limiters [11]. Presence of unlimited number of „writing–erasing" cycles, if VO_2 is used as a recording medium for optical correlators [12,13], is also unique for VO_2 films.

Despite this, data on ellipsometry of the doped films of vanadium dioxide are poorly presented in literature. There is a work [14], in which films of $VO_2:Na$ and $VO_2:K$ are studied using ellipsometric method. But mechanism of optical constants change near T_C — IMPT point — remains unknown. There is also a work [15], where refraction index n and extinction coefficient k are studied in a very wide range of wavelengths (300 nm−30 μm). This work also does not give information on mechanism of optical constants change near T_C. Besides, lack of information on characteristics and nature of changes of optical parameters of VO_2 film at variation of free electrons concentration in conductivity band. These data are very important for understanding the physical properties of strongly correlated compounds, to which VO_2 belongs.

Due to insufficiency of data on ellipsometry of vanadium dioxide films near IMPT, the purpose of this work is to study the ellipsometric spectra of VO_2 films, undoped and doped with Mg and Ge at temperatures near T_C.

2. Samples and experimental procedure

Nanocrystalline films of vanadium dioxide with thickness of 1000−2100 Å were synthesized using laser ablation method. This method allows to obtain the high-quality films of VO_2 and films of $VO_2:Ge$, $VO_2:Mg$ on sitall (SiAl) substrate. The synthesis was performed using a method of oxidation of metallic vanadium, magnesium and germanium steams in oxygen flow. Oxygen at pressure of 10^3 Pa was blown near a substrate with area of 1.2 cm^2. Simultaneously with oxygen blowing the exposure of laser pulses with a wavelength of $\lambda = 1.06\,\mu$m, duration of 25 ns and recurrence frequency of 10 Hz to metal targets, made of V, Ge and Mg, was performed. This was performed for generation of metals plasma flames. Surface density of laser energy at radiation focusing to a spot with diameter of 0.1 mm was 10^8 J/(cm$^2 \cdot$ s).

Ellipsometric spectra of samples were studied at temperature of 293 K (20°C) using spectral ellipsometer with binary modulation of polarization state. Ellipsometer consisted of optical-mechanical part with integrated small-size grating monochromator controlled by computer. Ellipsometer design had electronic systems of ellipsometric parameters control and registration, equipped with required software package.

Principle of spectroellipsometer operation is based on method of periodic changeover of polarization state between S- and P-components. Monochromatic light beam in S- and P-states was alternatively directed to the examined sample at incidence angles, set by the computer program. Incident beam was transformed to a sequence of orthogonally polarized beams with linear polarization azimuths P and $P + 90°$. Computer analysis of photometric signals was performed at azimuths of analyzer A and $A + 90°$.

Mathematic analysis of the results was performed based on the following considerations.

For systems with „polarizer–sample–analyzer" configuration the light intensity at photodetector is defined with expression

$$I = I_0\Big(\sin^2 A \sin^2 P + \cos^2 A \cos^2 P \tan^2 \psi + \frac{1}{2}\sin^2 A \sin^2 P \cos\Delta \tan\psi\Big),$$

where P and A — azimuths of polarizer and analyzer; I_0 — coefficient, independent from P and A; Ψ and Δ — ellipsometric angles, defining relation of complex amplitude reflection coefficients R_S and R_P for S- and P-polarizations as per expression

$$\rho = \frac{R_P}{R_S} = \tan\psi \exp(i\Delta).$$

Determination of numerical values of optical constants of vanadium dioxide films was performed using Calc spectrum software, integrated into the spectroellipsometer. This program performed calculation of film layers parameters as per measured values of Ψ and Δ. Calculation was performed based on standard ellipsometric equations [14].

Beside the study of ellipsometric spectra at room temperature, we performed ellipsometric measurements at the fixed wavelength of $\lambda = 632.8$ nm and at various temperatures in a range of 20–100°C. Temperature measurements were performed with temperature step of 5°C. Peltier element, that was used as a heater–cooler, was put onto a specimen stool. Film sample of vanadium dioxide and thermocouple for temperature control were mounted on the Peltier element.

3. Experimental results

Figure 1 shows the spectra of refraction index $n(\lambda)$ of undoped films of VO_2 and films, doped with magnesium and germanium. Presence of regions of normal $(dn(\lambda)/d\lambda < 0)$ and anomalous $(dn(\lambda)/d\lambda > 0)$ dispersion of refraction index is well seen. Also, change of a shape of spectral distribution $n(\lambda)$ curve at VO_2 films doping with Mg and Ge ions is observed. In case of Mg doping the $n(\lambda)$ curve generally shifts towards short-wave side of the spectrum, while at Ge doping the $n(\lambda)$ curve shifts towards long-wave side of the spectrum. The same shift in spectra of the doped samples is made by the points of $n(\lambda)$ curves intersection with anomalous dispersion with „zero" levels of normal dispersion [15] (Fig. 1). Zero levels of normal dispersion are presented in Fig. 1 with dotted lines.

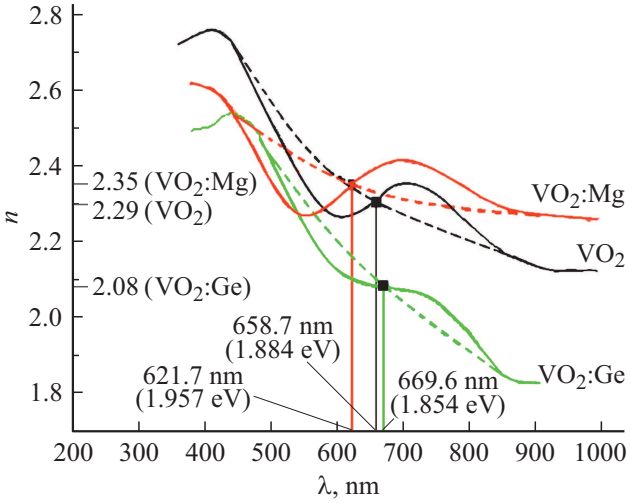

Figure 1. Spectra of VO_2 films refraction index. The dotted lines indicate „zero lines" of normal dispersion curves, made as per principle of equality of peak and drop areas of dispersion curve of anomalous dispersion on both sides of the dotted line.

In Fig. 1 the point of intersection of „zero" line with anomalous dispersion curve is shifted relative to undoped VO_2 film towards higher energy (1.957 eV) for VO_2 : Mg (Mg — donor impurity) and towards lower energy (1.854 eV) for VO_2 : Ge (Ge — acceptor impurity). Growth of refraction index at Mg doping and decrease of refraction index at Ge doping are well seen. This indicates the dominance of the first term in Moss formula

$$n(n_0, N) = n_0 - (e^2 N)/(n_0 \varepsilon_0 m_{\text{eff}} \omega^2), \qquad (1)$$

where n_0 — refraction index in the lack of free carriers, N — concentration of free carriers, e — electron charge, ε_0 — dielectric constant, m_{eff} — effective electron mass, $\omega = 2\pi\nu$ — cyclical frequency of light oscillations, ν — frequency of light oscillations.

Figure 2 shows the spectra of refraction index $n(\lambda)$, combined with spectra of extinction coefficient $k(\lambda)$ of undoped VO_2 films, as well as VO_2 films, doped with Mg and Ge. Figure 2 shows that HeNe laser light with a wavelength of $\lambda = 632.8$ nm falls onto region of anomalous dispersion $n(\lambda)$ and region of extinction coefficient $k(\lambda)$ sharp change. This fact is principally important for establishing a mechanism of spectra changes at doping

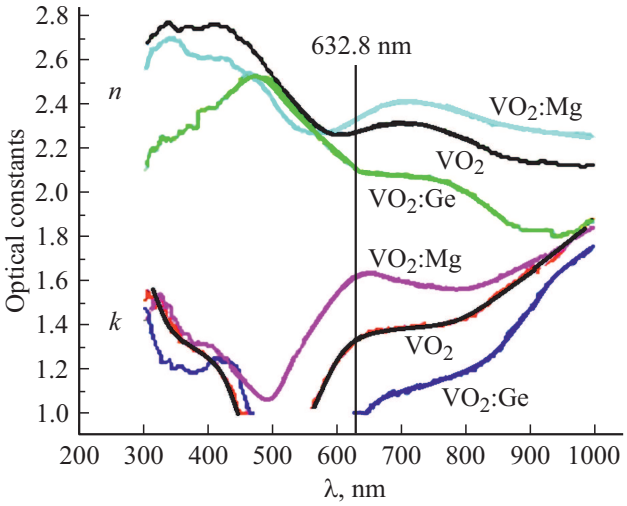

Figure 2. Comparison of ellipsometric data on $n(\lambda)$ and $k(\lambda)$ for VO_2 films.

of VO_2 films and at undoped VO_2 films temperature change.

Figure 3 shows the temperature hysteresis loops of refraction index $n(\lambda)$ (a) and extinction coefficient $k(\lambda)$ (b) for undoped VO_2 films at wavelength of $\lambda = 632.8$ nm in temperature interval of 20−100°C. This temperature interval corresponds to IMPT temperature region. It is seen, that with temperature growth the refraction index decreases with simultaneous increase of extinction coefficient. Besides, heating and cooling branches of the loops non-monotonously depend on temperature. These circumstances require consistent explanation, based on mutual dependence of refraction index and extinction coefficient [16]. The explanation is based on statement on dependence of refraction index of strongly correlated semiconductor on free electrons concentration and presented in i. 4.2.

4. Discussion of results

4.1. Forbidden band width

As per [17], electric width of the forbidden band $E_{g_{\text{electr}}}$ of undoped VO_2 is 0.7 eV. From here it follows, that thermal throwing of electrons into conductivity band starts at energy level Start, located by 0.2 eV below Fermi level. This is showed in Fig. 4, where function density of states (FDoS) is presented near Fermi level, calculated from the first principles [18].

Since the experimental determination of position of optical absorption spectrum maximum is a less sensitive method of $E_{g_{\text{opt}}}$ measurement compared to registration of low conductivity values, the optical width of the forbidden band $E_{g_{\text{opt}}}$ is larger than electrical width of the forbidden band $E_{g_{\text{electr}}}$ in value. Such statement corresponds with a form of a state density energy dependence function of VO_2. Figure 4 shows, that $E_{g_{\text{opt}}}$ should be in interval of 1.1−1.3 eV. This conclusion is confirmed with results of the work [19], according to which the experimental value is $E_{g_{\text{opt}}} = 1.173$ eV.

It was established, that point 1.884 eV (Fig. 1) of intersection of „zero“ line of normal dispersion $n(\lambda)$ and a curve of anomalous dispersion $n(\lambda)$ coincides, within the limits of experimental error, with energy position of minimum 1.832 eV in absorption spectrum [19] and position of minimum 1.844 eV of FDoS (designated in Fig. 4 with L letter, „L-drop“). Such result is natural, since it corresponds to Kundt's law [20]. According to Kundt's law, the region of anomalous dispersion $n(\lambda)$ coincides with a feature of insulator absorption spectrum, since the absorption of anomalous dispersion $n(\lambda)$ is caused

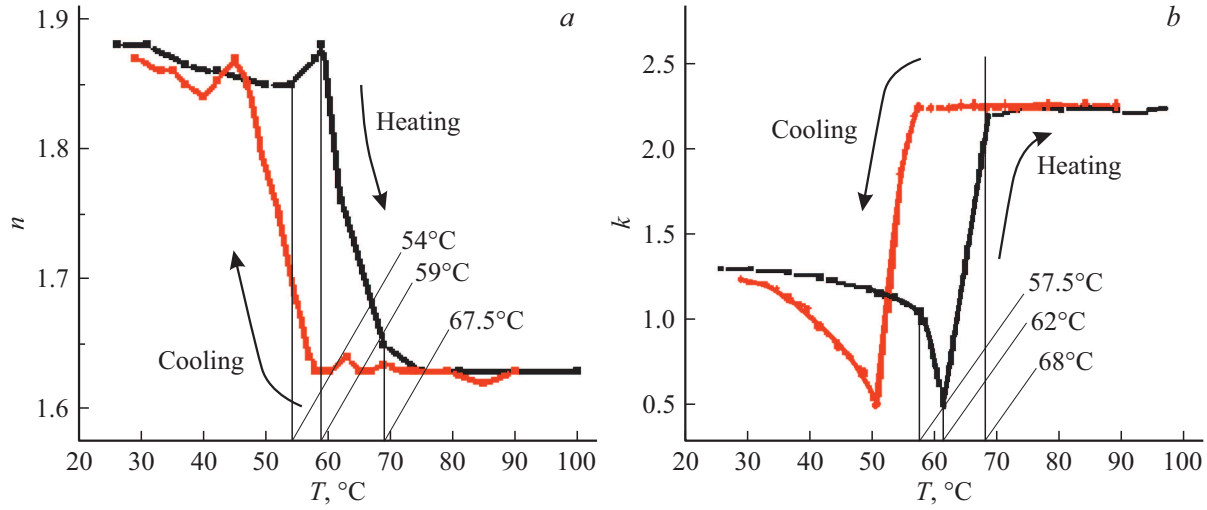

Figure 3. Temperature hysteresis loops: a — refraction index and b — extinction coefficient of undoped VO_2 film at radiation wavelength of He−Ne laser of 632.8 nm. Reduction of refraction index at simultaneous growth of extinction coefficient in the temperature region of 50−60°C indicates the dominance of the second term in the formula (1).

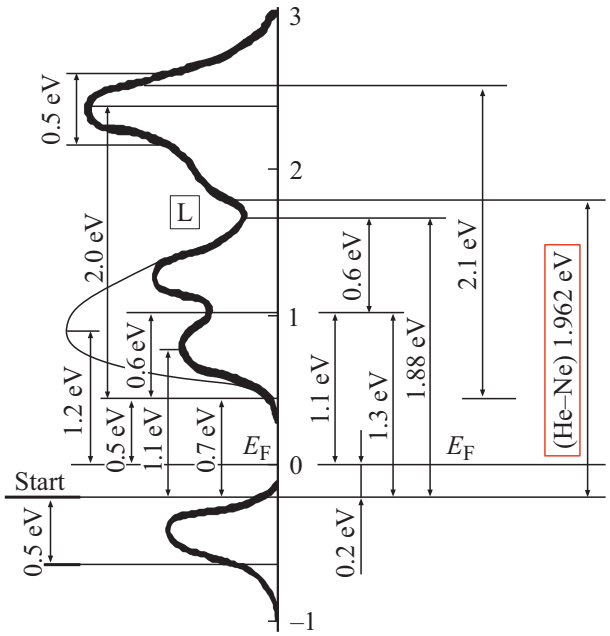

Figure 4. Energy dependence of FDoS of insulating phase of vanadium dioxide [18]. In the region of 1.9 eV the L-drop in state density energy dependence function is explicit.

by extinction coefficient sudden change near absorption spectrum lines. Thus, the energy positions of all features of ellipsometric spectra of undoped VO_2 are defined with a type of energy dependence of material FDoS.

4.2. Dispersion of refraction index $n(\lambda)$ and dependence of refraction index $n(T)$ on temperature

Changes of refraction index characteristics are caused, in our opinion, by three reasons.

1. The first one is that dependence (1) $n(\lambda)$ of a crystal on its physical parameters at presence of free charge carriers, interacting with a light (for instance, in case of strongly correlated materials), contains two terms [21].

In expression (1) for function of $n(n_0, N)$ the value of the second term depends on value of the first one (its denominator contains n_0). Besides, the value of the second term depends on m_{eff}. m_{eff}, in its turn, depends on curving of the conductivity band in Γ-point of Brillouin zone. And conductivity band curving can change at bands reconstruction near T_C of Mott phase transition, as well as at lattice structure sudden change after Peierls phase transition [4]. The abovementioned should be considered during analysis of the experimental results.

For this reason the case of influence of free electrons concentration N only on value of total refraction index $n(T)$ change with sample temperature growth is examined in i. 4.2. Reduction of refraction index $n(T)$ in temperature interval of 54−67.5°C, as shown in Fig. 3, *a*, is naturally explained by influence of the second term of Moss formula in expression (1) at thermal throwing of electrons into conductivity band. This result indicates the presence of effective interaction of a probe light and free electrons in conductivity band of VO_2. Such interaction happens with strict observance of the momentum conservation law due to strong electron-electron correlations. Specifically, it should be noted, that changes of N and, as a result, $n(T)$ with temperature are caused by electrons concentration growth in conductivity band due to temperature-extensive Mott part of a complex thermal IMPT [4], characteristic for VO_2. It is assumed, that n_0 in zero approximation does not depend on temperature.

Branches of temperature hysteresis loop of $k(T)$ dependence, obtained using ellipsometric method, are non-monotonous (Fig. 3, *b*), that is indicated with features in regions of 54−59°C (heating branch) and 40−45°C (cooling branch), close in temperature position to features of hysteresis loop of refraction index $n(T)$ (Fig. 3, *a*). Features of dependence of extinction coefficient $k(\lambda)$, defining light absorption, thus correlate with features of dependence of $n(\lambda)$. Indeed, coefficient $k(T)$ with T growth also changes non-monotonously (Fig. 3, *b*). Particularly, at the heating branch of hysteresis loop the light absorption in interval of 18−62°C $k(T)$ reduces and than increases only in interval of 62−68°C.

Such complex behavior of optical constants $k(T)$ and $n(T)$ we also associate with free electrons concentration growth, particularly with two reasons, additional to (1).

2. The second reason is a shift during heating towards short-wave side of minimum in absorption spectrum of insulating phase, located at 658.8 nm (1.884 eV) [19]. The minimum itself is caused by L-drop (Fig. 4) in energy dependence of the state density function [18]. This drop shifts upwards in energy due to growth of Coulomb interaction between electrons at thermal growth of their concentration, as shown in the work [23].

3. The third reason is that simultaneously with L-drop shift upwards in energy the width of the forbidden band reduces in energy due to correlation effects [18]. Thus, the growth of free electrons concentration results in expansion of energy range, occupied by state density function. At temperature growth of free electrons concentration the movement of L-drop upwards (Fig. 4) through a probe laser line of 632.8 nm (1.96 eV) initially results in reduction of extinction coefficient $k(T)$ (Fig. 3, *b*). The further increase of $k(T)$ with temperature is related to „L-drop ending“ as a result of its further shift upwards. Such movement of L-drop also results in non-monotonous change of branches of hysteresis loop of refraction index $n(T)$ (Fig. 3, *a*). Non-monotonicity of $n(T)$ is caused by relation between $n(T)$ and $k(T)$ [16].

4.3. Vanadium dioxide films doping with Mg and Ge ions

Process of vanadium dioxide doping with donor or acceptor impurities results in changes of optical characteristics

of VO_2 films, which may be even more complex than temperature variations.

4.3.1. Doping of VO_2 film with Mg ions

Mg impurity in VO_2 film crystallites is an electrons donor [24]. According to our measurements (Fig. 1), Mg doping moves the intersection point of „zero" level (dotted line) of anomalous dispersion curve $n(\lambda)$ towards higher energies from position at 1.884 eV for undoped VO_2 to 1.957 eV for $VO_2:Mg$. Energy position of this point coincides with position of absorption spectra feature (and position of L-drop of FDoS). Therefore it can be expected, that VO_2 film crystallites doping with donor impurities will also result in movement towards higher energies of minimum position in absorption spectrum of doped VO_2 film — of course under condition, that the shift reason is the donor electrons concentration increase in conductivity band. Experimental confirmation of this conclusion was obtained in the work [19], where absorption spectrum minimum shift is observed in a film of $VO_2:W(3\%)$ to 1.901 eV from energy of 1.83 eV for undoped VO_2 film. However, it is shown in [25], that phase transition temperature in a film of $VO_2:W(1.1\%)$ reduces by 30°C. This corresponds with $E_{g_{\text{opt}}}$ narrowing due to correlation properties of VO_2 crystal. Experimental confirmation of $E_{g_{\text{opt}}}$ narrowing in $VO_2:W(3\%)$ film was also observed in [19] as a movement in spectrum of optical absorption edge of $VO_2:W(3\%)$ film towards low energies to energy of 1.081 eV from value of 1.173 eV for undoped VO_2 film.

So, at VO_2 films doping with donors there is an experimental fact of movement in energy into the opposite sides of intrinsic absorption edge and position of intersection point of „zero" level (dotted line) of dispersion curve $n(\lambda)$. Particularly, „zero" point shifts towards high energies, while the forbidden band width reduces, i.e. the absorption edge shifts towards low energies. The physical reason for such phenomenon is in expansion of the conductivity band in FDoS energy, when electrons concentration increases in the conductivity band. Electrons concentration can grow at doping with donors or at thermal generation of electrons (see i. 4.2). According to literature data, this property is characteristic for compounds with strong electron correlations. In [23] it is shown, that energy region above Fermi level, occupied by FDoS, strongly expands with increase of Coulomb interaction between free electrons. It is obvious, that Coulomb interaction energy increases with free carriers concentration growth in conductivity band. At the same time, as shown in [23], FDoS does not intersect Fermi level lines. High-energy region of FDoS sharply moves upwards in energy. Considering the results of the work [18], it can be concluded, that low-energy part of FDoS in conductivity band of VO_2 lowers in energy at doping with donors, while high-energy part of FDoS simultaneously moves upwards in energy according to the work [23]. For this reason the FDoS energy expansion happens in strongly correlated materials.

This conclusion is in good agreement with experimental data of the works [19,25] on VO_2 films doping with tungsten. It is worth reminding that W, while being a donor, also lowers temperature T_C of phase transition of insulator–metal type. It means, that doping with W reduced the forbidden band width, since reduction of E_g is caused in $VO_2:W$ by correlation parameters [17]. At the same time, doping with W increases transmission of VO_2 film in maximum of transmission spectrum, located at 1.901 eV. Besides, doping with W, according to the experiment [19,25], shifts maximum in transmission spectrum towards higher energies from value of 1.83 eV for undoped VO_2 to 1.90 eV for $VO_2:W$. It should be noted, that at the same time the maximum of translucence and maximum of absorption move in energy into opposite sides relative to their position in undoped VO_2.

Conclusion on shift to the opposite sides of spectrum features also with electrons concentration growth is confirmed for $VO_2:Mg$ film with the results of experiments from our work on shift of „zero" point of anomalous dispersion $n(\lambda)$ towards high energies (Fig. 1). Simultaneously with this shift the reduction of T_C of phase transition happens. Reduction of T_C, as mentioned, is caused by narrowing of the forbidden band $E_{g_{\text{opt}}}$ with increase of free electrons concentration. And narrowing happens due to Mott phase transition in VO_2 film, extensive in free electrons concentration [17].

But the described process is not the only result of donors impact on optical properties of $VO_2:Mg$ films. The thing is that in $VO_2:Mg$ an ionic radius of Mg^{2+}, equal to 0.71 Å [27], is larger than ionic radius of vanadium ion (0.58 Å). Therefore, doping with Mg increases the average material density and results in increase of refraction index n_0 [28]. At the same time, Mg^{2+} ion in VO_2 has poorly expressed donor properties [24], resulting in growth of extinction coefficient in $VO_2:Mg$ film, that we observed experimentally (Fig. 2).

Particularly, our studies of $VO_2:Mg$ films showed increase of refraction index from 2.29 (VO_2) to 2.35 ($VO_2:Mg$) in intersection point of dispersion curve of „zero" level (dotted line) (Fig. 1). According to relation (1), this is related to increase of n_0 due to material density increase. Poorly expressed donor properties of Mg ion result in increase of extinction coefficient and narrowing of the forbidden band (Fig. 2) due to correlation effects. But Mg ions can not significantly increase the second term in expression (1) for refraction index $n = f(n_0, N)$ and override increase of n_0 value. The reason is that in the second term of expression (1) the value of n_0 is in denominator. This value growth due to crystal density increase compensates the growth of N, caused by electrons concentration growth. Therefore, the second term of expression (1) remains constant.

By performing the quantitative comparison of our and literature data for two possible mechanisms of influence of doping with magnesium ions on $n(\lambda)$ and $k(\lambda)$ spectra form, the conclusion is made, that $n(\lambda)$ spectra form change is mainly defined with material density increase, while $k(\lambda)$ — by free electrons concentration increase.

4.3.2. Doping of VO_2 films with Ge ions

Ge ions have not donor, but acceptor properties [24]. Doping with Ge should, as opposed to $VO_2:Mg$, move the value of „zero' point of $n(\lambda)$ (dotted line) towards lower energy. This is observed experimentally (Fig. 1). Doping with Ge not only decreases free electrons concentration in conductivity band of VO_2, but also reduces crystal density [27], thus reducing n_0 as per linear law [15]. The end result of doping influence on refraction index is defined with ratio of contributions of both terms of Moss formula (1) to $n(n_0, N)$ value. Let's analyze this topic in details.

Ionic radius of Ge^{4+} ion, equal to 0.5 Å [27], is less than ionic radius V^{4+} (0.58 .Å) by almost 11%. Such position inevitably results in significant internal stresses of a crystal lattice. For instance, at 3% doping with Ge each thirtieth crystal cell is in a state of internal tension stress. At the same time, the average crystal density decreases. At the same time, according to the experiment (Fig. 1), refraction index n_0 also decreases due to material density decrease [28,29]. But it should be noted again, that in formula (1) the value n_0 is in a denominator of the second term. Therefore, increase of n_0 results in the second term growth increase. This additionally reduces the total $n = f(n_0, N)$.

Our ellipsometric measurements of $VO_2:Ge$ films confirmed this conclusion and showed decrease (not increase, as for $VO_2:Mg$) of refraction index from 2.29 (VO_2) to 2.08 ($VO_2:Ge$) in intersection point of „zero" level (dotted line) of dispersion curve $n(\lambda)$ (Fig. 1). According to relation (1), this is related to decrease of n0 due to material density decrease.

Besides, measurements show shift of „zero" point $n(\lambda)$ towards low (not high, as for $VO_2:Mg$) energies (Fig. 1). This happens due to Coulomb transformation of FDoS conductivity band, caused by reduction of free electrons concentration. At the same time, the dielectric spectra of $VO_2:Ge$ film [24] indicate increase of temperature T_C of phase transition and, thus, expansion of the forbidden band (not narrowing, as for $VO_2:Mg$). Such expansion is caused by acceptor properties of Ge impurity. It should be noted, that extinction coefficient $k(\lambda)$ of $VO_2:Ge$ film is lower, while of $VO_2:Mg$ film — higher, than of undoped VO_2 film (Fig. 2). This corresponds to mechanism, that considers increase (or decrease) of free electrons concentration at VO_2 crystal doping with Mg (or Ge) impurity.

5. Conclusions

Based on comparative analysis of spectral and temperature dependencies of optical parameters of VO_2, $VO_2:Mg$ and $VO_2:Ge$ the following conclusions can be made.

1. Heating of undoped VO_2 film results in decrease of refraction index of insulating phase. I.e. the second term of formula (1) dominates for $n = f(n_o, N)$.

2. Refraction index $n(T)$ and extinction coefficient $k(T)$ of VO_2 films non-monotonously change with temperature growth in T_C region due to expansion of conductivity band in FDoS energy. Expansion is caused by increase of Coulomb interaction between free electrons at thermal growth of their concentration during IMPT.

3. Increase (decrease) of refraction index of insulating phase of $VO_2:Mg$ ($VO_2:Ge$) is defined with increase (decrease) of material density at doping with Mg(Ge) ions. Density change is defined with ratio between ionic radii of introduced impurities and radius of vanadium ion. It means, that in formula (1) the first term dominates for $n = f(n_0, N)$, regardless of the fact, whether impurity has donor or acceptor properties.

4. Doping with donor impurities, exceeding electrons concentration in conductivity band, results in extinction coefficient increase, while with acceptor impurities — to its decrease.

5. Optical forbidden band width of VO_2 insulating phase is elaborated, it is 1.173 eV. This is almost twice the value of electrical width of the forbidden band (0.68 eV).

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Conflict of interest

The authors declare that they have no conflict of interest.

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