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Research Article

Application of Ellipsometry to Control the Plasmachemical Synthesis of Thin TiONx Layers

F. N. Dultsev[1,2] and E. A. Kolosovsky[1]

[1] *Institute of Semiconductor Physics SB RAS, 13 Lavrentyev Avenue, Novosibirsk 630090, Russia*
[2] *Novosibirsk State University, Novosibirsk 630090, Russia*

Correspondence should be addressed to F. N. Dultsev; fdultsev@isp.nsc.ru

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Ellipsometry is often used to determine the characteristics of films. Ellipsometric studies may turn out to be ineffective because several solutions correspond to the same polarization angles. It is demonstrated that the ambiguity is not due to the physical limitations of the method but it has a purely mathematical character. So, additional information about the film is necessary to determine the absolute values of refractive index, attenuation, and thickness.

1. Introduction

Preparation of thin layers with the given stoichiometry and minimal size of granules is important for the development of nanostructures. For example, ultrathin layers of titanium nitride (TiN) are widely used in microelectronics as adhesion layers or diffusion barriers [1, 2], as metal-gate materials for metal oxide semiconductor devices. They possess good mechanical properties and thermal stability [3, 4]. In the present work, we used ellipsometry to study the mechanism of nitridation of the titanium matrix deposited on silicon surface [5]. Thus we obtained the layers of titanium nitride or oxynitride at the substrate temperature of 4–6°C. The advantage of this method in comparison with that described in [6–9] is, first of all, low temperature at which nitridation is carried out; second, this method allows us to obtain fine-structured layers of required thickness.

On the other hand, reliable control devices are needed to prepare such layers. It is necessary to apply high-precision measuring means intended for efficient diagnostics and tests of promising materials and structures under development. Ellipsometers belong to the measuring means of this kind [10–13]. A broad range of the applications of ellipsometry (from nanoelectronics to biotechnologies), as well as the availability of up-to-date ellipsometric instrumentation, make optical ellipsometry a very attractive and available method. However, it is the high sensitivity of this technique to small changes in the parameters of the medium that can cause in some cases incorrect interpretation of measurement results. This is especially so if the formed layers are not transparent. In our work we use ellipsometry to observe the formation of thin films having different compositions and thickness from several monolayers to $d_j \sim$ 1–100 nm. For the case when a film grows without cluster formation, in order to understand and reject incorrect interpretations of ellipsometric measurements, we will consider the reasons of misinterpretation. We will demonstrate below that misinterpretation of thin film parameters and error recovery are not connected with the physical nature of measuring technique but they have purely mathematical character.

It is known that the solution of inverse ellipsometric problem is ambiguous in the case of discrete light angles. For the ellipsometric examination of the growing TiONx films, we asked ourselves the question whether the ambiguity of the determination of film parameters is conserved if the number of measurements is increased *many times* embracing the whole possible range of light angles θ_a, or an increase in the number of measurements will cause the *statistical adjustment* of the same parameters. The second question is as follows: is this situation conserved for spectral ellipsometry?

And the last question is as follows: is it possible to escape from ambiguity or decrease its extent in the ellipsometric monitoring of growing TiONx?

2. Experimental Section

The metal Ti layer was deposited onto the 4-inch epiready Si (001) wafers in high vacuum (10^{-5}–10^{-7} Pa), using the e-beam sputtering technique. The Ti layers were deposited with the molecular beam epitaxy unit. The rate of metal layer deposition was 10 nm/min. Layer thickness was 5 to 50 nm.

After deposition, we used plasmachemical nitridation of Ti nanolayers to obtain TiN nanolayers. Plasmachemical treatment was performed in nitrogen gas plasma at a pressure of 0.8 Torr, with RF power 150 Watt and radio frequency 13.56 MHz ("MATRIX" unit) at a temperature of 4–6°C.

The metal-to-dielectric transition, that is, the formation of the dielectric film, was studied with the ellipsometer. Ellipsometric measurements were carried out according to multiangle technique with a LEF-4M ellipsometer at the wavelength of 632.8 nm. Measurements were carried out at the light angle within the range 45–80 angular degrees. The ellipsometric setup was assembled according to the PCSA scheme (polarizer-compensator-film-analyzer); beam blanking method (null ellipsometry) was used. Ellipsometer allows carrying out measurements over 4 angle zones. In addition to the ellipsometric measurements, UV absorption spectra of the films were recorded with the Shimadzu UV-2401PC spectrometer within the wavelength region 190–500 nm. Spectroscan ellipsometer manufactured at the ISP SB RAS was used for spectral ellipsometry.

The high-resolution transmission electron microscopy (HRTEM) images of TiN-SiO_2-Si interfaces were taken with JEM 4000 EX. Measurements were carried out at 300 kV. The surface roughness of films was determined with AFM Solver P47H atomic force microscope (AFM) in the resonant mode operating at 350 kHz and 38 nm amplitude. The size of the tip was 10 nm.

Equations used in ellipsometric calculations are simple and readily testable in the case of the direct problem (by means of the forward substitution). To solve these problems, one can use any mathematical program package: MATLAB, Mathcad, Excel, and so forth. To solve the inverse problem, we used our own original algorithm within the framework of the same packages; the system of equations was solved over the complex numbers field, similarly to the method described elsewhere [14].

3. Results and Discussion

The kinetics of the transformation of metal to dielectric during plasma treatment is shown in Figure 1. These results were obtained from ellipsometric data. Measurements were carried out with the help of ellipsometer at different light angles, as well as at different wavelengths (Spectroscan). Absorption was measured by means of UV spectroscopy. The figure shows how the film thickness changes actually versus nitridation time; during nitridation, we observe total increase in film thickness.

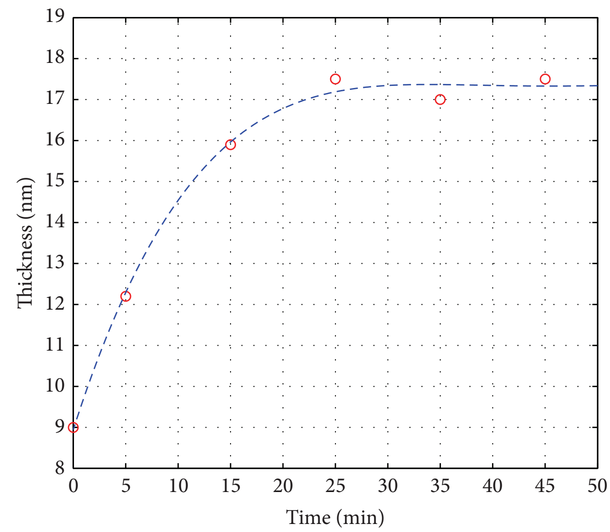

Figure 1: Kinetics of the formation of titanium nitride: nitridation of titanium layer 100 Å thick.

The initial film thickness was 90 Å. Thickness was calculated according to the procedure proposed by us.

We determined the thickness of Ti film before nitridation with the help of AFM. Using AFM, we also observed the change of film relief as a result of nitridation. One can see that nitridation is accompanied by surface smoothing. We demonstrated previously [15] with the help of high-resolution transmission electron microscopy that nitridation proceeds layer by layer, so we used the multilayer model. The ellipsometric data show that layer thickness increases during nitridation almost by a factor of 2 (Figure 1). It was shown previously that the resulting layers are uniform, and a model for ellipsometric calculations was proposed [15]. Now we will consider the solution of the inverse problem and demonstrate why a set of solutions arises.

3.1. General Solution of the Ellipsometric Equations. The fundamental equation of ellipsometry connecting ellipsometric parameters delta (Δ) and psi (Ψ) with the complex absolute reflectance coefficients R_p and R_s for p- and s-polarized light (see Figure 3) is written as [16–19]

$$\rho = \frac{R_p}{R_s} = \tan(\Psi) \cdot e^{i\Delta}. \tag{1}$$

In the case of a multilayered structure, the absolute reflectance coefficients R_s, R_p coincide with coefficient r_N calculated in the recurring manner using (2) and (3) for the corresponding polarizations [14–18]:

$$r_j = \frac{\rho_j + r_{j-1} \cdot \exp(2i\varphi_j)}{1 + \rho_j \cdot r_{j-1} \cdot \exp(2i\varphi_j)}; \qquad r_0 = \frac{P_1 - P_{\text{sub}}}{P_1 + P_{\text{sub}}}; \qquad j = 1, \ldots, N, \tag{2}$$

where r_j are the coefficients of reflection from the system of the first j layers; ρ_j are the coefficients of reflection from the interfaces between the layers; φ_j are phase incursions in layers.

Parameters ρ_j and φ_j are determined on the basis of Fresnel's formulas:

$$\rho_j = \frac{P_{j+1} - P_j}{P_{j+1} + P_j}; \qquad \varphi_j = k_0 \cdot n_j \cos(\theta_j) \cdot d_j. \tag{3}$$

Here $P_j = n_j \cdot \cos\theta_j$ for s-polarized waver, and $P_j = n_j / \cos\theta_j$ for p-polarized wave, while angles θ_j (the angles at which the wave spreads over the layers) are determined from Snellius law: $n_j \sin\theta_j = n_{\text{sub}} \sin\theta_{\text{sub}} = n_a \sin\theta_a$, k_0 is the wavenumber of light in vacuum. For absorbing media, a refractive index of each medium is also represented as a complex factor: $n_j - ik_j$. The direct problem of ellipsometry involves determination of parameters delta (Δ) and psi (Ψ); it is easily solved for the geometrically flat interface. In a multilayered structure with a set of layers $\{n_j - ik_j, d_j\}$ this determination, according to (1), holds true unambiguously. It should be noted that (1)–(3) imply isotropy of layers.

When solving the inverse ellipsometric problem, the optical parameters of sample surface are calculated on the basis of measured Δ and Ψ values relying on the chosen model. The coincidence of the calculated Δ and Ψ with the experimental values is the evidence in favour of the correctness of the chosen optical model. As a rule, it is necessary to solve the inverse ellipsometric problem, that is, to determine the optical characteristics of the reflecting system on the basis of the measured set of Δ and Ψ values under different conditions: different light angles θ_a at which the light falls on the surface from different media and different light frequencies (so-called spectral ellipsometry) [14, 17, 19, 20].

3.2. Multiangle Measurements at One Wavelength. At first, we will consider a one-layer structure shown in Figure 2: a model metal film with refractive index $n_1 = 2.5 - i \cdot 2.0$ and thickness $d_1 = 100$ Å on silicon substrate (refractive index for silicon at the light wavelength of $\lambda = 6328$ Å is $n_{\text{sub}} = 3.865 - i \cdot 0.023$). Assigning the angles at which the light falls from the air ($n_a = 1$) within the range from 45 to 80 with a step of 1 degree, we will obtain the solution of the direct ellipsometric problem in the form of a plot in Figure 3, where the numbers above points correspond to the light angle θ_a. The side dash lines indicate the area of plot shift with a 10% change of film thickness d_1. One can see that the change of film thickness by only ±1 nm leads to a visible substantial shift at the plot, which confirms the high sensitivity of the ellipsometric technique to film parameters during film formation.

Now we will pass to the inverse problem of restoration of film parameters. Experimental (Δ) and (Ψ) values measured for one specific light angle θ_a allow us to calculate one of the film parameters—thickness d_1, or its complex refractive index $n_1 - ik_1$ following a general rule: one equation—one unknown quantity. Usually not two but several measurements for different light angles θ_a are used, so that matching of the results with the help of the least squares method could help us to eliminate the errors in angle measurement.

It is known that the inverse problem when solving (1) in a one-layer structure can give several solutions [17, 19]

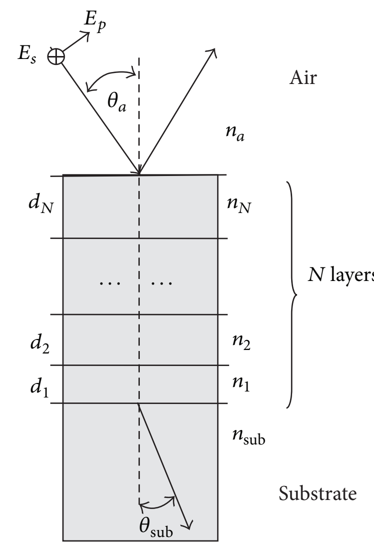

Figure 2: Light polarized as E_p and E_s falls onto the layered structure composed of N layers $\{d_j, n_j\}$.

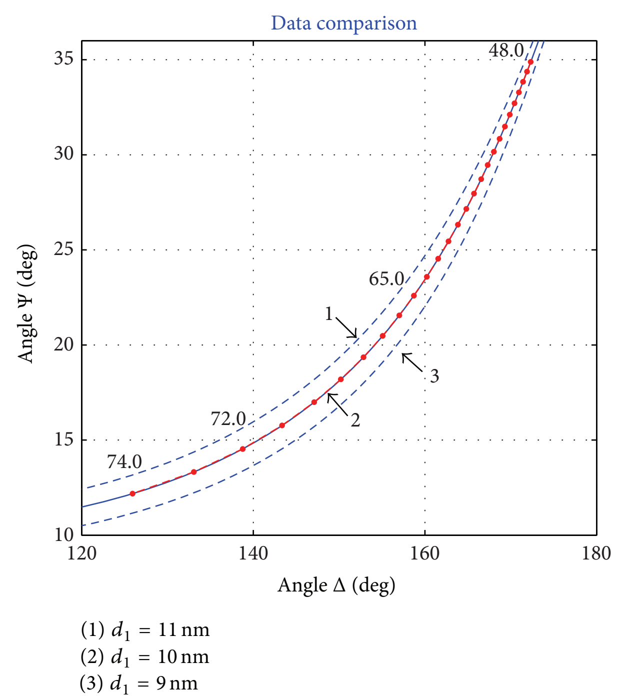

Figure 3: Ellipsometric parameters (Δ) and (Ψ). Dash line shows the boundaries of the area with thin film d_1 changed by ±1 nm. Light angle θ_a varies from 48 to 74 deg.

(this fact is not connected with the harmonic exponent in (2) because $d_1 \ll \lambda_0$). It was demonstrated that the total number of possible solutions can be determined beforehand applying the theory of the functions of complex variables [14]. To confirm this statement, the model data are listed in Table 1 depicting several solutions: different values of refractive index for the films of different thickness values. All of them satisfy (1) and go well into the same central curve of Figure 3 within

TABLE 1: Calculation results for one-layer structure.

d_1, Å	n_1 $(n + ik)$
50	$1.6275 - 3.6481i$
90	$2.1491 - 2.1822$
100	**2.5 − 2i**
110	$2.6164 - 1.8500i$
150	$2.9908 - 1.4533i$
300	$3.7628 - 1.0180i$
1000	$4.0495 - 1.4444i$

TABLE 2: Calculation results for two-layered structure.

d_1, Å	d_2, Å	n_1	n_2
50	**50**	**2.5 − 2i**	**2.5 − 2i**
50	20	$2.5 - 2i$	$1.0271 - 4.1815i$
50	20	$2.2776 - 2.7318i$	$1.5291 - 2.5012i$
50	20	$1.7903 - 3.1159i$	$2.9627 - 2.0428i$
50	150	$2.5 - 2i$	$3.6502 - 1.2975i$
50	150	$1.9293 - 0.2837i$	$3.3544 - 1.5333i$
50	100	$2.9989 - 1.1472i$	$2.7523 - 1.8887i$

the whole range of θ_a angles and not only at some values of this light angle.

Now it is necessary to answer the question whether the ambiguity of parameter determination in two- and more layered films remains if we increase the number of measurements embracing the whole possible range of light angles θ_a, or an increase in the number of measurements would cause statistical refinement of the same parameters.

We specially carried out a thorough investigation of this question concerning the solution of the inverse problem. For two- and three-layered structures, at first we chose the same total film thickness equal to $d_1 + d_2 + d_3 = \mathbf{100}$ Å and the same attenuation $n_1 - ik_1 = \mathbf{2 - 2i}$ as those for the one-layer structure (shown in bold characters in Tables 1, 2, and 3). Thus, the starting dependencies of parameters (Δ) and (Ψ) were reduced to one plot coinciding with curve 2 in Figure 3. Then thickness d_j was varied within rather broad range, and a special algorithm was used to reveal the new meanings of refractive index to give coincidence with the starting one by solving the direct problem within the whole range of angles θ_a. The mathematical precision of this coincidence exceeded the possible errors of experimental angle measurements by several orders of magnitude. As a result, all the one-, two-, and three-layered structures marked in Tables 1–3, in spite of essential differences in their parameters, belong to the same ellipsometric curve in Figure 3. We see that an increase in the number of measurements made with a single-wavelength ellipsometric data (in multiangle ellipsometry) conserves the ambiguity of film parameter determination. The experimental values for ellipsometric angles delta and psi are shown in Table 4. These values were used to perform verification.

3.3. Spectral Ellipsometry. As far as spectral ellipsometry is concerned, with the measurements performed at different light wavelengths, a similar situation is still actual. Similarly to the previous case, Figure 4 depicts the solution of the direct problem of spectral ellipsometry for three layers 100, 200, and 300 nm thick, deposited on silicon (line). Then the thickness of two films was varied by ~50 nm, and the new film parameters were determined by solving the inverse problem. One can see in this figure that the new solution (points) graphically coincides with the primary solution. Figures on the plot mark the positions of measurements depending on the light wavelength λ, which was varied from 250 nm to 850 nm with a step of 1-2 nm, which corresponds to several hundred measured data values. The light angle θ_a is equal to 70 angular degrees. Film parameters for both solutions are indicated in Table 5. One can see that the refractive index for layer d_3 changed insignificantly and refractive index and attenuation in layer d_2 decreased much more substantially, while the refractive index of the very first layer increased from $n = 1.5$ to 1.8983, and losses for light extinction disappeared completely for this layer.

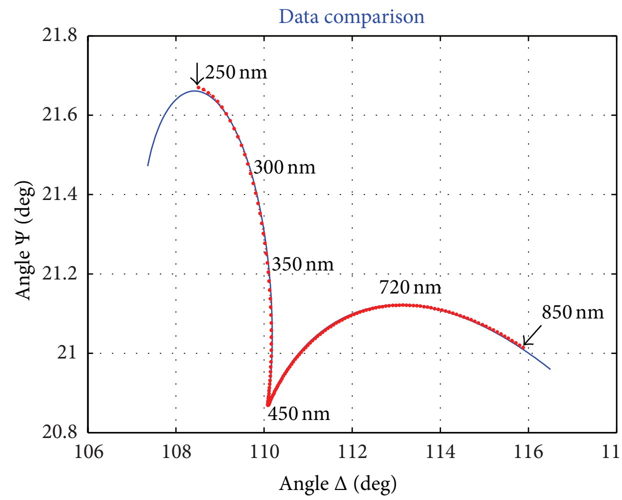

FIGURE 4: Coincidence of parameters (Δ) and (Ψ) for two different three-layered structures in spectral ellipsometry with the variation of light wavelength λ from 250 nm to 850 nm. (Line: the first structure from Table 5, points: the second structure.)

As the objects for investigation, we prepared three samples of TiONx films with thickness $d \sim 16$–22 nm on silicon according to the technology described in [15]. The data for titanium layer deposited on silicon substrate for different nitridation times are shown in Figure 5. In view of the fact that the TiOx films were deposited onto silicon, one should take into account the changes of its optical properties versus the wavelength. The data shown in Figure 6 demonstrate that the refractive index changes by a factor of 7, while absorption changes by a factor of several hundreds. This change is not monotonous but, unlike for quartz [21], it has a complicated character due to differences in the mechanism of light absorption in Si (electron-hole and lattice-related) [22]. Because of this, the restoration of the parameters of TiONx films was carried out as follows. On each plot of measured data in Figure 5, we could choose any three successive points at a small distance from each other. Because measurements

Table 3: Calculation results for three-layered structure.

d_1, Å	d_2, Å	d_3, Å	n_1	n_2	n_3
30	**30**	**40**	**2.5 − 2i**	**2.5 − 2i**	**2.5 − 2i**
30	20	20	$2.5 - 2i$	$1.8058 - 4.0108i$	$1.7848 - 2.1868i$
30	50	20	$2.4034 - 3.9591i$	$3.2192 - 1.2237i$	$2.7586 - 1.1836i$
30	20	50	$2.6243 - 3.7342i$	$3.2267 - 1.2572i$	$2.7594 - 1.1830i$
30	100	100	$1.8556 - 2.4132i$	$2.4437 - 0.2124i$	$3.9365 - 2.1136i$

Table 4: Precise values of ellipsometric angles (for test).

(θ_a)	(Δ)	(Ψ), deg
50	171.4310	33.8416
55	168.6927	30.8415
60	164.8167	27.1589
65	158.7096	22.5939
70	147.0962	16.9903

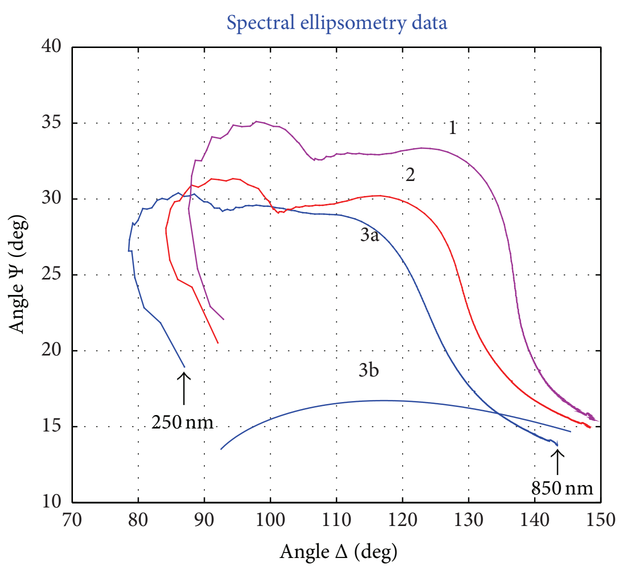

Figure 5: Experimental data for three samples of TiONx films in ellipsometric measurements with the light wavelength λ_0 varied from 250 to 850 nm with 2 nm resolution. Lower curve 3b shows the result of calculation without taking dispersion into account.

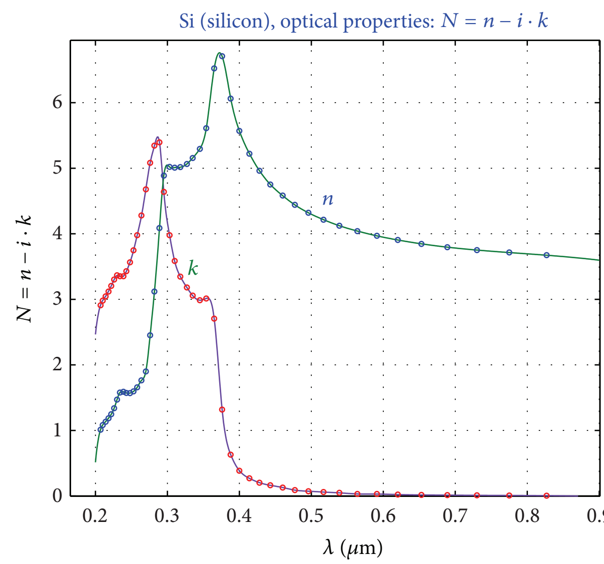

Figure 6: The spectral dependence of refractive index in silicon $N = n - i \cdot k$ on the light wavelength λ, according to the data of [17].

are performed with a small step over wavelength λ_0, we may accept that the optical parameters of the films and the substrate are constant within the limit of these three measurements and correspond to the second (average) value of λ_0. Taking the thickness of TiONx film from additional measurements (e.g., by means of AFM) and choosing the refractive index of the silicon substrate according to the data shown in Figure 6, we may solve the direct and inverse problems of ellipsometry and determine film parameters.

Repeating this procedure many times with other groups of three points λ_i, we obtain all the optical parameters of TiOx films including dispersion, the spectral dependence of refractive index, and absorption of light on wavelength λ_0.

For comparison, at the bottom of Figure 5 we show curve 1 calculated without taking dispersion into account. It depicts the dependence of ellipsometric parameters Δ, Ψ for curve 3a related to the film with thickness $d = 22$ nm and refractive index $N = n - i \cdot k = 2.8 - 1 \cdot i$ corresponding to the red light with wavelength $\lambda_0 = 0.63\,\mu$m (see Figure 7). Comparing the two curves 3a and 3b in Figure 5, one can see that the account of the effect of dispersion in precise ellipsometric measurements does not bear only a character of correction but it holds the defining importance. Actually, the account of this fact does not hinder processing and interpretation of changes in film structures but even allows one, relying on the optical dispersion of the silicon substrate, to extract the unknown spectral characteristics of TiONx and similar films themselves. That is, it allows us to know the behaviour of both the refractive index and extinction coefficient versus the wavelength λ_0 of light in any thin films on silicon. If the thickness and the optical parameters of the films are determined correctly, curves 3a and 3b in Figure 5 should coincide. We carried out this work, and the results for three samples when two curves 3a and 3b (in Figure 5) completely coincide are presented in Figure 7. Of course, due to the mentioned mathematical ambiguity of the solution of the inverse problem of ellipsometry, thickness d should be determined also using an independent method.

Table 5: The data of spectral ellipsometry for three-layered structure.

d_1, Å	d_2, Å	d_3, Å	n_1	n_2	n_3
100	200	300	$1.5 - 0.2i$	$2.0 - 0.8i$	$2.5 - 2i$
133	144	320	1.8983	$1.6534 - 0.6166i$	$2.5041 - 2.0016i$

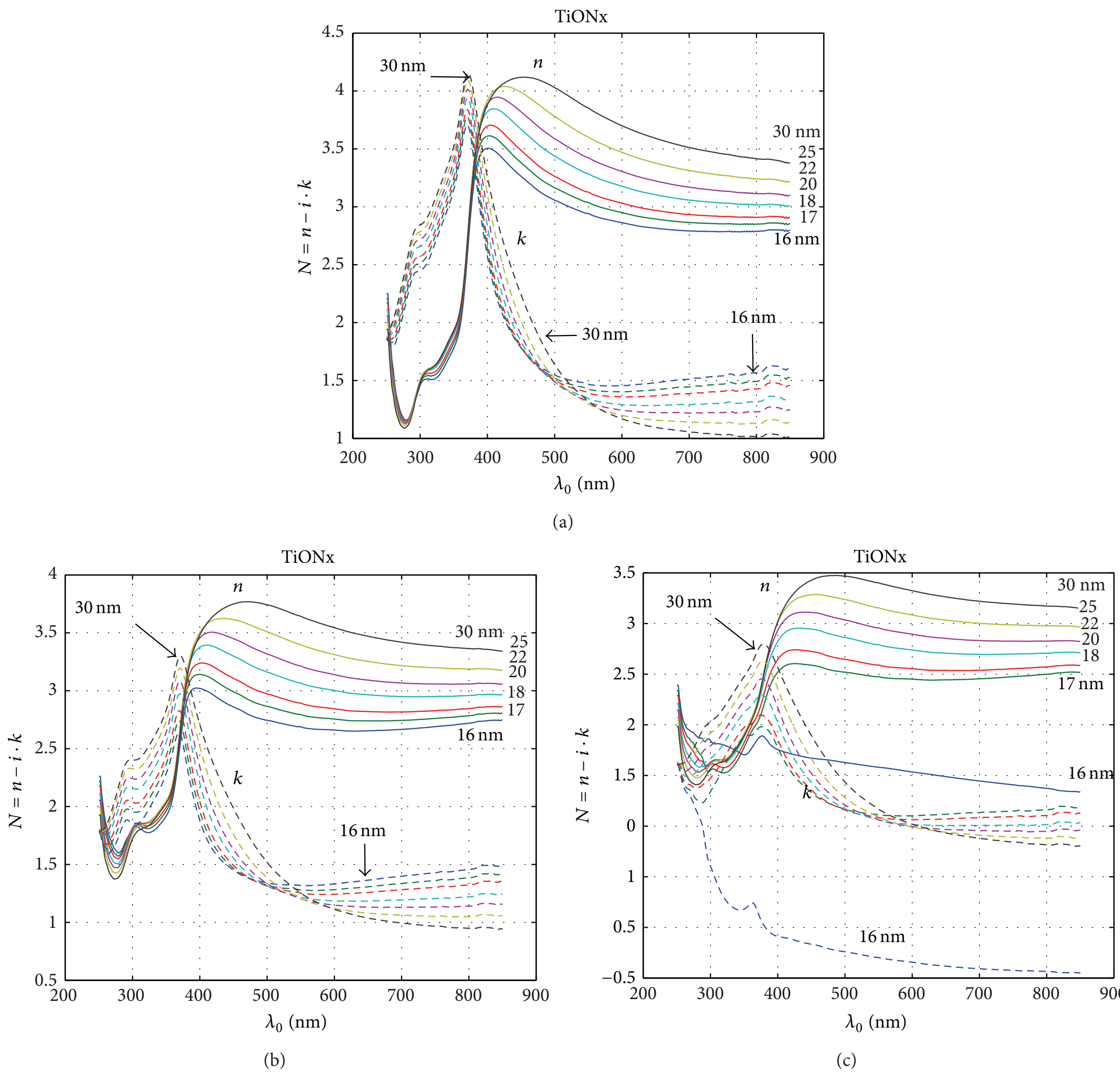

Figure 7: Optical dispersion of refractive index (lines) and light absorption (dash line) in TiONx films of different thickness (different nitridation time) according to the data of Figure 5 for (a) sample number 1, (b) sample number 2, and (c) sample number 3a.

The real thickness of TiONx films in all the three samples was $d = 16$ to 22 nm. In order to demonstrate how film thickness may affect its optical characteristics, we processed seven successive thickness values. All the results with different thickness values d shown in Figure 7 correspond to the experimental curves shown in Figure 5. It may be concluded from the behaviour of the absorption curve (Figure 7(c)) that the films with thickness $d = 16$ nm and less cannot exist. We conclude this because starting from this thickness a "passive" film should possess amplifying properties because for $\lambda_0 > 380$ nm the absorption coefficient of the film material changes its sign (see the lower dash line in Figure 7(c)).

One can see in the plot that the monotonous decrease in the refractive index of the TiONx film, so typical for quartz glass, is present only in the second (longer wavelength) half of the optical range, for radiation with the wavelengths longer than 500 nm. In the first (shorter wavelength) part of the optical range, the refractive index of the film material changes substantially, from 1.5 to 3.5. One can notice that light extinction attenuation in the film is very large. The absorption coefficient changes in the nonmonotonic manner and is large, within the range from 1 to 3. Because of this, the described procedure based on the data of spectral ellipsometry is applicable mostly to the thin films, that is, for TiONx films with thickness 2 to 50 nm (20–500 Å), and the films composed of any other material, if they are transparent enough (their thickness should be small enough to allow us to see the substrate). The procedure proposed by us for the determination of the behaviour of the optical characteristics of films uses the known dispersion in silicon as the calibration standard, the radiation must "sense" the material of the silicon substrate, and strongly absorbing films should not be too thick. In other words, in order to reduce the extent of ambiguity, we used the effect of dispersion of the refractive index, that is, its dependence on wavelength in Si. Usually even a small change of the refractive index versus wavelength within the visible light range is considered as a parasitic (undesirable) phenomenon, because it causes errors in the measurement of film parameters. However, it is this effect (dispersion of light in silicon) that turned out to be essential when thoroughly measured within a broadened wavelength range, and we used it as the basic information for calibration. In the case of thin films on silicon, either TiONx or other compositions, when the incident light wave partially penetrates and thus "senses" the Si substrate, this is possible.

So, we see that an increase in the number of ellipsometric measurements and broadening of the range of light angles for one-, two-, and more layered structures conserve ambiguity of the determination of film parameters. This ambiguity is not due to the physical limitations of the method but it bears the mathematical character of so-called ill-conditioned problem [23–25]. Because of this, in spite of the high sensitivity of the method, determination of refractive index and thickness of absorbing thin film requires additional data. For example, additional measurements of absorption or thickness of the film are to be carried out using other methods.

Because of this, the most promising approach is to combine ellipsometry with any other methods of surface studies, for example, Auger spectroscopy, UV and X-ray spectroscopy, electron diffraction and ion scattering, and so forth. The advantages of ellipsometry are simplicity and rapidity of measurements (automatic ellipsometers are available), the possibility to carry out measurements in course of the process (*in situ*), in vacuum, at high temperatures, and in aggressive media; in addition, the surface is not contaminated or destroyed during measurements.

4. Conclusion

In spite of the high differential sensitivity of ellipsometric method, for titanium nitride films obtained by means of nitridation (metal-to-dielectric transition), determination of the absolute values of refractive index, attenuation, and film thickness requires independent additional information.

For one-layer, two-layer structures and the structures with larger number of layers, an increase in the number of ellipsometric measurements and broadening of the light angle range or wavelength range (for spectral ellipsometry) conserve the ambiguity of film parameter determination. Ambiguity is not due to the physical limitations of the method but has the mathematical character.

Solution of the inverse problem for two- and more layered structures showed that the measurement of the same films within different angle ranges, as well as an increase in the number of measurements, does not allow one to obtain new information on film composition, and no adjustment of film parameters occurs in principle. Because of the mathematical specificity of equations, coincidence of solutions for different models is possible not only in discrete points Δ and Ψ but also along the entire curve.

We considered one-, two-, three-, and more layered structures that belong completely to the same ellipsometric curve, in spite of the substantial differences between their parameters.

This situation is conserved also for spectral ellipsometry. We demonstrated quite different structures that gave coincidence (within the measurement error) of the Δ, Ψ-curves within their entire continuous range under continuous change of the wavelength of light.

In order to eliminate ambiguity or decrease its extent in the ellipsometric control of growing TiONx or other films, we used dispersion of refractive index, that is, its dependence on the light wavelength in Si. Thoroughly measured within the whole wavelength range, this effect turned out to be essential. We used it as the major calibration information. This is possible in the case of absorbing thin films on silicon, when the incident light wave partially penetrates and "senses" the Si substrate.

Thus, confirming the necessity of additional information for the determination of film parameters using optical methods that are convenient, highly precise, and nondestructive, for TiONx-Si films as example we demonstrated the whole set of the reasons of ambiguity inherent to ellipsometry, either discrete or continuous, either angular or spectroscopic.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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