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Multiangular and Spectral Ellipsometry for Semiconductor Nanostructures Classification

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The possibilities of multiparameter determination of semiconductor nanostructures based on spectral dependencies of polarized radiation reflection coefficient R_p, R_s on the incidence angle in the range of 200-800 nm are investigated. Experimental data have shown high sensitivity of reflection coefficients angular dependence to the type of polycrystalline structures at the same film thickness. The presence of additional extremums in spectral dependence of refraction and absorption indexes is detected; this could be connected with grain size of polycrystalline structure and type of grain boundaries. The possibility of multiparameter optical research of properties and thickness of semiconductor layers on Si substrate is shown.

Keywords: Polysilicon film, Spectral ellipsometry, Reflection coefficient, Refraction index, Absorption index.

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1. INTRODUCTION

Upon the development of nanotechnologies the methods of nondestructive, multiparameter, express, local determination of properties of semiconductor structures become the most essential. For most widespread layered film structures the significant properties are layer thickness and basic optical properties such as refractive index (n) and absorption index (κ). On the one hand, this is connected with informativeness of these parameters, and on the other hand – with significant dependence of layers properties on their thickness d (preparation conditions [1], thermal treatment [2], oxidation and exposure [3]). Dependencies $n(d)$ and $\kappa(d)$ for thin layers, obtained at the same wavelength, aren't constant and can have different shapes first of all as a result of differences in technological processes of structure preparation [1]. Therefore, use of any parameter value from tabulated or literature data can be erratic and lead to considerable inaccuracy in the diagnosis of other properties.

The way out of this situation is determination of set of related parameters within the same method and measurement system, which allows determining a set of properties during one act of measurement. However, currently the optical methods that allow simultaneous determining of refractive and absorption indexes, layer thickness for absorbing and non-absorbing materials aren't well developed. In recent investigations of local defects on the silicon surface [4] the wavefront measurements and its dependence from surface reflectance were used. In paper [5] the influence of local refraction and absorption indexes inhomogeneties on the reflection coefficient was mentioned. It should be noticed that higher precision of parameters estimation using data of wavefront measurement can be achieved by step-by-step sequential probing in two orthogonal polarizations [5, 6].

In this paper different possibilities of multiparameter determination of properties of semiconductor structures using data about spectral dependence of reflection coefficients of p- and s- polarized radiation (R_p and R_s) on the angle of incidence φ_{in} are investigated. Moreover, the possibility of reconstruction of spectral distribution of refraction and absorption indexes is studied.

2. POLYCRYSTALLINE SILICON FILMS

2.1 Preparation

Silicon films were prepared by low-pressure chemical vapour deposition from silan/argon mixture. Films were deposited on thermally oxidized (thickness of SiO_2 is 100 nm) single crystal silicon wafers with the thickness of about 2 μm. Polycrystalline Si samples were obtained by increasing deposition temperature (T_d) over 900 K, for amorphous films samples $T_d \approx 830$ K. Some samples were doped with phosphorus by ion implantation, thermodiffusion, under deposition (in situ) and annealed within the temperature range 1200-1500 K in nitrogen ambient. Thus the structure of poly-Si films depends on deposition conditions and temperature and forms three main structures: fibrous, dendritic or equiaxed [7].

The fibrous microstructure is formed in undoped poly-Si films (thickness $d \geq 70$ nm) under deposition temperature over 900 K. The dendritic structure is formed from amorphous or amorphous-crystalline states under annealing at temperature from 1100K to 1200K. Equiaxed structure results from recrystallization of high doped ($n_P \approx 10^{20} \div 10^{21}\ \mathrm{m}^{-3}$) poly-Si films under temperature over 1300 K. More details about different film structures obtaining and their typical structure charachteristics can be found in [7].

2.2 Basic Theory

For describing the characteristics of the light reflected from multilayer samples the model must have different layer thickness and boundaries with different refraction indices, which correspond to the following media: air, poly-Si film, SiO_2, massive substrate of Si (Fig. 1).

The optical wave reflection and refraction is possible on each edge between different layers due to the different refraction indices. The polycrystalline film had to be characterized by effective refraction index, which corresponds to the poly-Si film morphology, and differs from the refraction index for single crystal Si. Each layer of such multi-layered structure has refraction index and a certain thickness, passing which the

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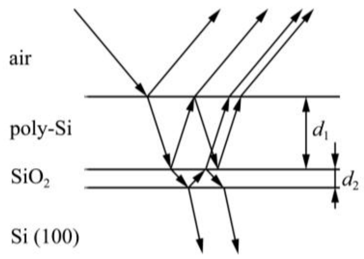

Fig. 1 – Scheme of the light reflection from the multilayer structure

wave reflects and refracts on each next edge. So, the final result will depend on the coefficients of refraction and reflection, optical path length and phase shifts between p- and s- components. The reflection coefficient for three-layer system: poly Si-SiO$_2$-Si(100) one can write as:

$$\mathbf{R} = Re^{i\delta} = \frac{r_{01} + \dfrac{r_{12} + r_{23}\mathrm{e}^{-i2\beta_2}}{1 + r_{12}r_{23}\mathrm{e}^{-i2\beta_2}}\mathrm{e}^{-i2\beta_1}}{1 + r_{01}\dfrac{r_{12} + r_{23}\mathrm{e}^{-i2\beta_2}}{1 + r_{12}r_{23}\mathrm{e}^{-i2\beta_2}}\mathrm{e}^{-i2\beta_1}}, \qquad (1)$$

where r_{01}, r_{12}, r_{23} are Fresnel reflection coefficients [8] at the interface air- poly Si, poly Si-SiO$_2$, SiO$_2$-Si(100) correspondingly; β- phase thickness of the film:

$$\beta_i = 2\pi\frac{d_i}{\lambda}\sqrt{(n + i\kappa)^2 - \sin^2\varphi_{in}}, \qquad (2)$$

where d_i is i-th layer thickness, λ is probe radiation wavelength, φ_{in} is incidence angle of radiation. Thus, the ellipsometry equation for such system is:

$$\mathrm{tg}\psi e^{i\Delta} = \frac{r_{p01} + \dfrac{r_{p12} + r_{p23}\mathrm{e}^{-i2\beta_2}}{1 + r_{p12}r_{p23}\mathrm{e}^{-i2\beta_2}}\mathrm{e}^{-i2\beta_1}}{r_{s01} + \dfrac{r_{s12} + r_{s23}\mathrm{e}^{-i2\beta_2}}{1 + r_{s12}r_{s23}\mathrm{e}^{-i2\beta_2}}\mathrm{e}^{-i2\beta_1}} \times \frac{1 + r_{s01}\dfrac{r_{s12} + r_{s23}\mathrm{e}^{-i2\beta_2}}{1 + r_{s12}r_{s23}\mathrm{e}^{-i2\beta_2}}\mathrm{e}^{-i2\beta_1}}{1 + r_{p01}\dfrac{r_{p12} + r_{p23}\mathrm{e}^{-i2\beta_2}}{1 + r_{p12}r_{p23}\mathrm{e}^{-i2\beta_2}}\mathrm{e}^{-i2\beta_1}}, \qquad (3)$$

where ψ and Δ stand for relative amplitude and phase change [8]. It's not difficult to notice, that reflection coefficient contains phase factor, which corresponds to phase incursion at wave propagation in multilayered medium. This, in turn, proves the probability of interference phenomenon appearance and reveals as oscillations of angular and spectral dependencies of reflection coefficient [9].

It's necessary to solve direct ellipsometry problem, when ellipsometric parameters (ψ and Δ) of reflected wave are calculated using known characteristics of reflection systems, and inverse problem, when parameters of test system can be found using measured values of polarization characteristics of reflected wave, while interpretating ellipsometric measurements. It's important to mention, that it's necessary to solve transcendental equation (3) for solving the inverse problem of obtaining refractive and absorption indexes of film. For its solution the numerical analog of diagrams method was used [10]. Calculation of ellipsometric parameters was performed for fixed angle of incidence or wavelength (eq. 3), varying layer thickness and its refraction index, with adding filling factors changing [11]. A set of curves converging at one value was build using the obtained results.

3. EXPERIMENTAL SET-UP

The photoelectric ellipsometer is the most suitable for spectral ellipsometry measurements, which in contradistinction from compensated ellipsometer doesn't require any compensator and has less measurement time. The operation principle of such ellipsometer is based on photoelectric Beattie method [10]. The essence of method is following: linearly polarized light beam falls on the surface of investigated object at arbitrary angle, and azimuth of polarizer (angle between electric vector of light wave and plane of incidence) equals 45°.

Elliptical polarization of reflected light is analyzed using linear polarizer, which is sequentially set at 0°, 45° and 90° azimuth, measuring corresponding intensity of reflected light I_0, I_{45}, I_{90}. Unknown ellipsometric parameters are computed by formulas:

$$\mathrm{tg}\psi = \sqrt{\frac{I_0}{I_{90}}}, \qquad (4)$$

$$\cos\Delta = \frac{2I_{45} - I_{90} - I_0}{2\sqrt{I_{90}I_0}}. \qquad (5)$$

The scheme of such device is shown at Fig. 2.

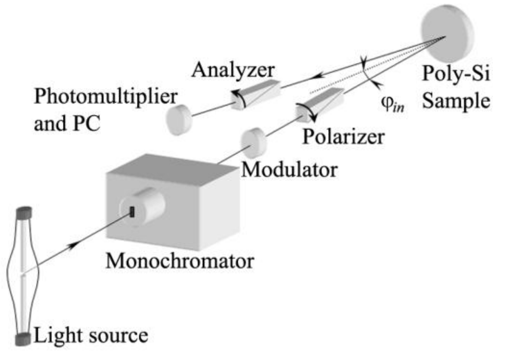

Fig. 2 – Block-diagram of spectral photoelectric ellipsometer

A source of light in this device is halogen lamp IEC 60061-1 (7004-73) in range of 450-800 nm and ultrahigh pressure arc xenon ball lamp DKsH-150 with regulated power supply in range of 240-578 nm. Polarizer (Glan prism) provides linear polarization of the light wave. It is mounted in frame, fitted with device for polarizer rotation around the direction of light beam propagation and the system of azimuth φ_{in} measurement. The unit for analyzing the polarization ellipse of reflected light wave also comprises Glan prism with positioner mechanism.

The system is placed on the goniometer with angular resolution up to 0,5' with two arms, where polarizer, analyzer units and light source are mounted. The sample with mechanism that allows rotating in vertical and horizontal plane and mechanism of translational displacement of platform in perpendicular direction to the surface is placed on the goniometer axis.

Measurement error of refraction and absorption indexes (estimated from the set of photovoltage measurements) doesn't exceed 1.5 % in UV and visible range and 2.5 % in IR range of electromagnetic spectrum.

4. RESULTS AND DISCUSSION

Ellipsometric measurements were performed for mirror reflection of optical radiation with illuminating beam aperture 5 mm at different angle of incidence on the sample. The silicon wafers Si (100) and amorphous silicon film Si (100) + Si (a) (film width was about 700 nm) were tested as the reference samples. The value of calculated spectral distribution of refraction (*n*) and absorption (κ) indexes for bulk silicon is shown at Fig. 3. Such spectrum corresponds to known data from literature [12].

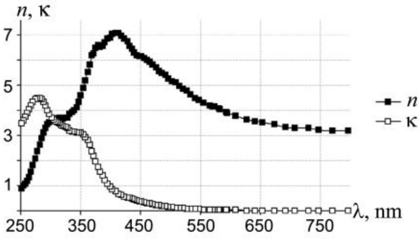

Fig. 3 – Spectral dependence of refraction and absorption indexes for Si (100)

The amorphous silicon as well as the polycrystalline silicon is the isotropic matter, thus the films of amorphous or polycrystalline Si on the same substrate have no significant polarization differences. As one can see from Fig. 4 the dependence of reflection coefficients for the amorphous silicon film don't differ from the dependence, obtained for continuous media, but the amorphous silicon film has other reflection coefficients (R_p and R_s) and the position of the main (pseudo-Brewster) angle.

Fig. 5 shows the similar dependencies for polysilicon films with fibrous, dendritic and equiaxed structures. As one can see, the nature of dependencies is substantially different for all three cases. It can be noticed that normal reflection coefficient *R* increases for investigated structures in direction – dendritic, fibrous, equiaxed. At the same time the size of surface inhomogeneities change in other sequence, i.e. it's logical to assume, that obtained optical features are connected with morphological peculiarities of polysilicon films structure, not with the size of surface inhomogeneities. Results, obtained for films with different structural modifications, are in good correspondence with presence of special grain boundaries and grain boundary junctions in the films. Speaking about grain boundary junctions, in [13] was shown the existence of different types of triple junctions and also the presence of multiple junctions of four and five grains, that vary in terms of quantity and interlayer spacing of boundaries for polysilicon films with mentioned structural modifications. Such influence of grain boundary junctions on the optical properties stipulates angular dependencies.

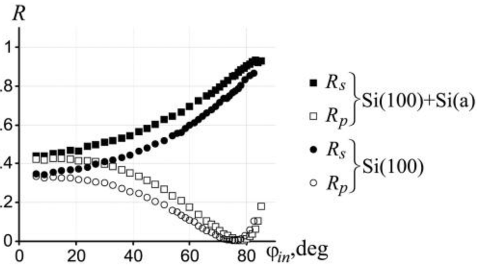

Fig. 4 – Angular dependencies of reflection coefficient for silicon Si (100) and for system of amorphous film on silicon substrate Si (100) + Si (a) for wavelength $\lambda = 633$ nm

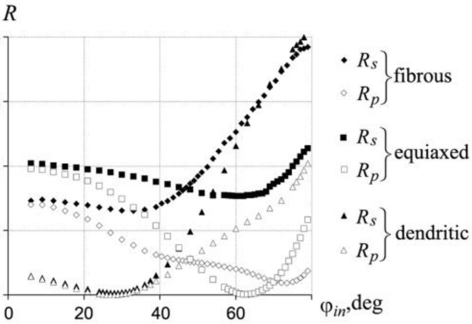

Fig. 5 – Angular dependencies for reflection coefficient of polycrystalline films with different structure at wavelength $\lambda = 633$ nm

The calculated spectral dependencies of silicon films refraction and absorption indexes are shown on Fig. 6. As it can be seen from given dependencies the dendritic, fibrous and equiaxed films (with the same thickness as amorphous one) show themselves on the background of amorphous film by the presence of oscillations of refraction and absorption indexes. This, obviously, is connected with presence of grain boundaries with different reflection coefficients, while presence of special boundaries [14] leads to the increase of amplitude of such oscillations.

Error in spectrum reconstruction of refraction and absorption indexes dependencies from equation (3) was controlled from Kramers-Kroning relation [15]:

$$\delta = \left\langle \left| n^2(\omega) + \kappa^2(\omega) - 1 - \frac{2}{\pi} P \int_{-\infty}^{\infty} \frac{n(w)\kappa(w)}{w-\omega} dw \right|^2 \right\rangle, \quad (6)$$

where ω and w are frequency, *P* is main integral value symbol, ⟨...⟩ is symbol of wavelength averaging. Let's mark, that value of spectral error, specified in this manner, doesn't exceed 0.1 at $(n^2(\omega) + \kappa^2(\omega) - 1)\,|_{\min} \approx 4.2$. Fig. 4 and Fig. 5 show the experimental angular dependencies of the reflection coefficient of investigated structures. Parameters of given structures, obtained from angular dependencies of reflection coefficient and from spectral dependencies of ellipsometric parameters, are listed in Table 1.

Small differences of parameters, defined by two different methods, indicate the adequacy of experimental techniques and theoretical model of the layered structure. Let's notice that experimental data demonstrated high sensitivity of reflection coefficient *R* angular dependence to values of optical constants and layered

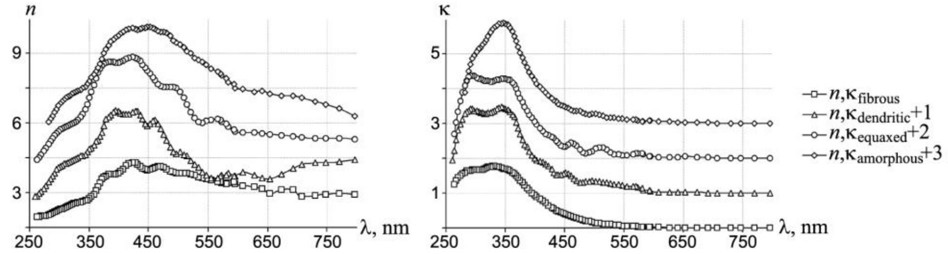

Fig. 6 – Spectral dependencies of refraction and absorption indexes for silicon films with different structural modifications

Table 1 – Values of characteristics of different structures, defined from angular dependencies of reflection coefficient and from spectral dependencies of ellipsometric parameters (λ = 633 nm)

Upper layer parameters		Angular dependencies	Spectral dependencies
amorphous silicon	n	3.38	3.34
	κ	0.025	0.029
	d, нм	732	721
fibrous	n	3.44	3.47
	κ	0.015	0.011
	d, нм	724	711
dendritic	n	2.79	2.81
	κ	0.007	0.005
	d, нм	697	703
equiaxed	n	3.51	3.49
	κ	0.035	0.031
	d, нм	754	749

structures thickness. The application of scheme of parallel angular dependencies measurement with fixed sample and time averaging of data can increase the accuracy of method [15].

Thereby, performed researches of different possibilities for multiparameter determination of structures properties, based on the analysis of angular dependencies of reflection coefficient and spectral dependencies of ellipsometric parameters, revealed the possibility of classification of polysilicon structures with different structural modifications.

5. CONCLUSIONS

The possibility of non-destructive determination of thickness, refraction and absorption indexes of surface layers with different structural modifications of polysilicon is realized on the base of the measurements of reflection coefficient dependencies on the incidence angle of monochromatic radiation and ellipsometric parameters dependencies on the wavelength of scanning radiation. Let's notice that carrying out of such measurements is possible for determination of film and substrates properties in structures with different combinations of absorbing and slightly absorbing materials. Besides, the measurement errors in angular dependencies of reflected radiation analysis (which is simpler in hardware realization), are close to the errors of simple ellipsometric method in fixed conditions, though angular dependencies are significantly less informative.

Класифікація мультикутової та спектральної еліпсометрії для напівпровідникових наноструктур

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Досліджені можливості багатопараметричного визначення напівпровідникових наноструктур на основі спектральних залежностей поляризованого випромінювання коефіцієнта відбиття R_p, R_s від кута падіння в діапазоні 200-800 нм. Експериментальні дані показали високі коефіцієнти чутливості відбивання кутової залежності від типу полікристалічних структур. Наявність додаткових спектральних екстремумів в залежності від заломлення і поглинання може бути пов'язане з розміром зерен полікристалічної структури і типу меж зерен. Показана можливість багатопараметричного дослідження оптичних властивостей і товщини напівпровідникових шарів на кремнієвій підкладці.

Ключові слова: Полікремнієва плівка, Спектральна еліпсометрія, Коефіцієнт відбиття, Показник заломлення, Показник поглинання.

Классификация мультиугловой и спектральной эллипсометрии для полупроводниковых наноструктур

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Исследованы возможности многопараметрического определения полупроводниковых наноструктур на основе спектральных зависимостей поляризованного излучения коэффициента отражения R_p, R_s от угла падения в диапазоне 200-800 нм. Экспериментальные данные показали высокие коэффициенты чувствительности отражения угловой зависимости от типа поликристаллических структур. Наличие дополнительных спектральных экстремумов в зависимости от преломления и поглощения может быть связано с размером зерен поликристаллической структуры и типа границ зерен. Показана возможность многопараметрического исследования оптических свойств и толщины полупроводниковых слоев на кремниевой подложке.

Ключевые слова: Поликремниевая пленка, Спектральная эллипсометрия, Коэффициент отражения, Показатель преломления, показатель поглощения.

REFERENCES

1. B. Poqany, *Annalen der Physik* **49**, 537 (1916).
2. S.W. Xuea, X.T. Zu, et al., *J. Alloy. Compd.* **448**, 21 (2008).
3. S.A. Kovalenko, M.P. Lisitsa, *Semiconductors Physics, Quantum Electronics and Optoelectronics.* **4**, 352 (2001).
4. A.A. Goloborodko, V.I. Grygoruk, et al., *Ukr. J. Phys.* **53**, 946 (2008).
5. N.S. Goloborodko, V.I. Grygoruk, et al., *Semiconductors Physics, Quantum Electronics and Optoelectronics* **13**, 65 (2010).
6. A.A. Goloborodko, V.N. Kurashov, D.V. Podanchuk, N.S. Sutyagina, M.M. Kotov, *Proc. of SPIE* **7388**, 73880P (2009).
7. N.G. Nakhodkin, T.V. Rodionova, *phys. status solidi a* **123**, 431 (1991).
8. H. Fujiwara, *Spectroscopic Ellipsometry. Principles and Applications* (John Wiley & Sons Ltd: The Atrium, Southern Gate, Chichester, West Sussex PO19 8SQ, England: 2007).
9. O.I. Barchuk, K.S. Bilenko, et al., *Nanosystems, Nanomaterials, Nanotechnologies.* **7**, 421 (2009).
10. R.J. Archer, *J. Opt. Soc. Am.* **52**, 970 (1962).
11. A.A. Goloborodko, N.S. Goloborodko, et al., *Semiconductors Physics, Quantum Electronics and Optoelectronics* **16**, 128 (2013).
12. J.R. Beattie, G.K.T. Conn, *Philos. Magazine* **43**, 222 (1955).
13. N.G. Nakhodkin, N.P. Kulish, et al., *Nanosystems, Nanomaterials, Nanotechnologies* **2**, 793 (2004).
14. N.G. Nakhodkin, N.P. Kulish, et al., *Bulletin of University of Kyiv. Series: Physics and Mathematics.* 201, (2010).
15. Yu.E. Zabiyakin, *Optika i Spektroskopiya.* **24**, 828 (1968).
16. D.I. Bilenko, A.A. Sagaidachnyi, V.V. Galushka, and V.P. Polyanskaya, *Tech. Phys.* **55**, 1478 (2010).