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Polarization conversion effect in obliquely deposited SiO_x films

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Abstract. Structural anisotropy of the SiO_x films and $nc-Si-SiO_x$ light emitting nanostructures, prepared by oblique deposition of silicon monoxide in vacuum, has been studied using the polarization conversion (PC) effect. For this purpose, a simple method of PC investigation with usage of a standard null-ellipsometer is proposed and tested. This method is based on the analysis of the azimuthal angle dependence of the offdiagonal elements of the Jones matrix. The electron microscopy study shows that obliquely deposited SiO_x films have a porous (column-like) structure with the column diameter and inclination depending on the deposition angle. Polarimetric investigations revealed that both in-plane and out-of-plane anisotropy was present, which is associated with the columnar growth. The correlation between the PC manifestations and the scanning electron microscopy results is analyzed. It was found that the tilt angle of columns in obliquely deposited SiO_x is smaller than that predicted by the "tangent rule" and "cosine rule" models, and depends on the crystallographic orientation of Si substrate. It is concluded that the proposed method is effective non-destructive express technique for the structural characterization of obliquely deposited films.

Keywords: polarization conversion, ellipsometry, anisotropy, oblique deposition, silicon oxide, nanostructure, microstructure.

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1. Introduction

The microstructure of thin films is of great interest both from scientific and practical points of view because of many physical and chemical properties of films are controlled by their microstructure [1, 2]. Very often thin films exhibit columnar microstructure when deposited by vapor condensation processes in vacuum [3-5]. It is known [3-9] that formation of the columnar structures depends on the material being evaporated, substrate type and temperature, vapor pressure as well as the vapor incidence angle α , i.e. the angle between film normal and vapor deposition direction (obliqueness angle). The columns tend to lean toward the incident flux with an angle β relative to the surface normal ($\beta < \alpha$).

 SiO_x films obtained by the oblique deposition of the evaporated silicon monoxide (SiO) in vacuum also form a structure with inclined columns [10]. Recently, the method of porous light-emitting structure formation using oblique deposition of SiO in vacuum was proposed

[11]. During high-temperature annealing of these films, the thermally stimulated formation of Si nanoinclusions (nc-Si) occurs in a restricted volume of the SiO_x columns thus forming the silicon-based light emitting nc-Si-SiO_x structures, in which Si nanoparticles (nc-Si) are surrounded by porous oxide matrix (SiO_x) .

Study of specific features of both as-deposited and thermally and/or chemically treated SiO_x film is crucial goal-directed control of luminescent for the characteristics of the structures. Solution of this problem requires the methods of a columnar microstructure characterization (simple, express and non-destructive ones are especially desirable). Structural anisotropy (the shape anisotropy and the packing anisotropy) in the films with oblique columnar structure seems to induce strong anisotropy in their optical properties. This makes it possible to consider polarimetric methods as feasible effective instrument for characterization of these films.

In this paper, on the example of SiO_x films, a simple polarimetric method for the study of structural

anisotropy in obliquely deposited layers is proposed and tested. To determine the characteristics of the possible structural anisotropy, it was suggested to measure the so-called cross-polarization or p-s polarization conversion (PC) effect. The results obtained using the proposed method are compared with the scanning electron microscopy (SEM) results.

2. Experimental

The studied samples were fabricated by thermal evaporation of 99.9% pure silicon monoxide SiO (Cerac Inc.) in vacuum $(1-2) \times 10^{-3}$ Pa onto polished c-Si (111) and c-Si(100) substrates oriented at the angles $\alpha = 0^{\circ}$, 60°, 75° between the normal to the substrate surface and the direction to the evaporator. The substrate temperature during deposition was 150 °C. Because of additional oxidation by residual gases during evaporation SiO, the compositionally of nonstoichiometric SiO_x (x > 1) films were deposited in the vacuum chamber. The deposition rate and the thickness of the films being monitored in situ using the КИТ-1 quartz microbalance system.

The SiO_x oblique deposited film (ODF) growth morphology was visualized by ZEISS EVO 50XVP scanning electron microscope. The geometrical thickness of the films was also measured with MИИ-4 interferometric microscope. Multiangle ellipsometric measurements have been performed to determine the thickness and refractive index of the films. The measurements were done using the PCSA null-type ellipsometer $JI \Im \Phi - 3M - 1$ (Feodosia, Ukraine) equipped with He-Ne laser as a light source ($\lambda =$ 632.8 nm). The same ellipsometer was used for the investigation of PC in the films.

3. Foundations of the proposed method

It is well known that for light reflection from an isotropic film the *p*-*s* polarization uncoupling condition is valid [12]:

$$E_p' = r_p E_p, \quad E_s' = r_s E_s, \tag{1}$$

where E_p and E_s are the components of incident wave (one is parallel to the incidence plane and another is perpendicular to it). The corresponding components of the waves reflected from the film are denoted as E_p' and E_s' , respectively. In general, for anisotropic film the *p*-*s* polarization uncoupling condition is broken and the incident and reflected waves are related by 2×2 reflection matrix as follows [12-16]:

$$E_p' = r_{pp} E_p + r_{ps} E_s ,$$

$$E_s' = r_{sp} E_p + r_{ss} E_s ,$$
(2)

where the subscripts "*ps*" and "*sp*" denote *s*-wave converted to *p*-wave and *p*-wave converted to *s*-wave, respectively (polarization conversion), whereas "*pp*" and "*ss*" represent *p*-wave reflected as *p*-wave and *s*-wave reflected as *s*-wave.

To obtain full information about optical (and thus structural) anisotropy of ODFs from polarimetric investigations, it needs, in general, to solve inverse problem of ellipsometry in the framework at least "biaxially anisotropic uniform layer" model with the use of spectral and/or angular dependences of ellipsometric parameters Δ and Ψ measured at various relative orientation of light incidence plane and vapor deposition plane (the plane containing both the substrate normal and the vapor incidence vector is referred to as the deposition plane). It is rather difficult task that can be solved by numerical fitting the measured Δ and Ψ angles data with an appropriate model [15, 16].

Based on above described general principles of PC effect, we have realized a more simple method for the analysis of optical anisotropy in ODFs which makes it possible to obtain information about the structure of the films, specifically, on such important parameter as columns tilt angle β . This method explores the fact that off-diagonal elements r_{ps} and r_{ss} [12-16]. As a result, in general, in case of s(p)-linear polarized incident wave the reflected wave will be elliptically polarized, but the ellipse of polarization will be strongly elongated and its major axis will have some small angle with the s(p)-direction. The absolute value of this angle will be proportional to $|r_{ps}/r_{ss}|$ for s- or $|r_{sp}/r_{pp}|$ for p-polarized incident wave.

At the transition to the isotropic situation, r_{ps} and r_{sp} diminish, and the relationship (1) is valid. The *p*-s polarization uncoupling condition (1) is also valid for some special cases of anisotropic media, precisely, when one of the main axes of dielectric function tensor ε_{ik} is perpendicular to the incident plane. This is possible when this axis is parallel to the plane of substrate surface. If two principal axes of ε_{ik} are parallel to the sample surface and mutually perpendicular, and the third axis is perpendicular to the first two axes, then r_{ps} and r_{ps} will be zero precisely through the every 90° when the substrate is rotated around the surface normal axis. For example, for the Lengmuir-Blodgett films with a weak in-plane anisotropy of the sample and with the third principal axis being slightly (several degrees) tilted from the surface normal, experimental dependences of $\operatorname{Re}(r_{ps}/r_{pp})$ and $\operatorname{Re}(r_{ps}/r_{ss})$ versus the azimuth angle Ω are close to the cosine (sinusoidal) dependence with the 180° period [13].

From geometrical consideration one may assume that in ODFs two principal axes of ε_{ik} are located in the vapor deposition plane (one of the axes coincides with the longitudinal axis of the columns and the second one is perpendicular to the first) and the third axis is perpendicular to this plane. That is why, one could expect that at low angles of column inclination r_{ps} and r_{sp} will be zero for the cases when the light incidence plane will coincide with or perpendicular to the vapor incidence plane, which is through, approximately, every 90°. For sufficiently inclined columns, r_{ps} and r_{sp} will be zero only for the case when the light incidence plane will

coincide with the vapor incidence plane, which is through every 180°. So, the simplest way to determine the essential features of the anisotropy in ODFs is to measure the angle between the main axis of polarization ellipsoid for the reflected wave and s-direction of the incident wave as a function of the azimuth angle Ω (Ω is the angle between the laser beam incidence plane and the vapor deposition plane). For this purpose, the polarizer and the compensator of laser ellipsometer $\Pi \Theta \Phi - 3M - 1$ are adjusted to obtain the s-polarization of the laser beam projected on the sample (incidence angle of the laser beam was $\varphi_0 = 45^\circ$). Samples were mounted on the rotating stage of the ellipsometer, thus allowing a complete 0°-360° variation of Ω (the value $\Omega \equiv 0^\circ$ corresponds to the situation when the laser beam incidence plane coincides with the vapor deposition plane, and the laser beam falls on the sample surface from the same side as the vapor flux does). The sample surface was accurately set normal to the rotation axis of the stage.

Let us direct the *s*-polarized at isotropic or uniaxially anisotropic sample with distinguished optical axis perpendicular to its surface and pass the reflected from such sample light through analyzer of ellipsometer. At setting the analyzer in position at which it transmits light of *p*-polarization (designate this position as A_s), the light intensity, transmitted by the analyzer, will be zero at any Ω . In other words, for these two types of samples the reflected light will remain *s*-polarized at any Ω .

This result was obtained for reflection from the monocrystalline silicon substrates and the substrates with normally deposited SiO_x films. But at the reflection from the substrates with SiO_x ODFs the *p*-*s* polarization uncoupling was not observed, and as a result of PC effect minimal intensity of the light transmitted by the analyzer was at other position of analyzer (designate as A_{\min}). For small values of r_{ps}/r_{ss} the angle of the rotation of the analyzer ($A_{\min}-A_s$) is proportional to the r_{ps}/r_{ss} and approximately equal to the angle θ between the main axis of polarization ellipsoid for reflected wave and *s*-direction of the incident wave ($\theta = A_{\min}-A_s$). The angular resolution of the instrument is 1' for θ and 6' for Ω .

4. Results and discussion

To estimate the thickness and refractive index of the films, the inverse problem of ellipsometry was solved for deposited SiO_x films. By fitting the measured $\Psi(\varphi_0)$ and $\Delta(\varphi_0)$ angles data with "isotropic uniform layer" and "uniaxial uniform layer" models, the thickness and optical constants of the films were determined. The Nelder-Meed method was used to minimize the difference between experimental ellipsometric angles $\Psi(\varphi_0)$ and $\Delta(\varphi_0)$ and theoretical ones.

The solution of inverse problem of ellipsometry for SiO_x film deposited at $\alpha = 60^\circ$ in the frameworks of both isotropic and uniaxial anisotropic layer models gave the values of the film thicknesses that are close to one another and to the interferometric value. Within the

former n = 1.545, and within the latter the anisotropy is rather small ($n_o = 1.559$, $n_e = 1.565$). Results obtained using uniaxial anisotropic layer model are indicative of column-like porous microstructure in films with preferential orientation of columns and pores along the normal to substrate [17]. It is indirect evidence that inplane anisotropy of the sample is rather weak and the third principal axis is slightly tilted from the surface normal. The values of refractive index for this SiO_x ODF are sufficiently smaller than refractive index values of normally deposited dense SiO_x films ($n \approx 2.0$). Obviously, it is caused by higher porosity of obliquely deposited SiO_x films [11].

For the SiO_x film deposited at $\alpha = 75^{\circ}$, the considerable discordance between the models of isotropic and uniaxially anisotropic layer takes place: within the former n = 1.545, h = 668 nm and within the latter $n_{av} = 1.423$, h = 755 nm values are obtained. As seen, the isotropic layer gives close n values for the films deposited at both 60° and 75°. "Uniaxial anisotropic layer" model gives much less n values for the film deposited at 75°. It is more consistent with the general expected trends of increased porosity with vapor deposition angle (this tendency is also valid for SiO_x ODFs [10, 11]). Moreover, the *h* value obtained in uniaxial anisotropic layer model is closer to the interferometrically determined h value. Nevertheless the use of this model is still inadequate approximation because the sign of anisotropy depends on the orientation of light incidence plane. These results of ellipsometric modeling indicate that higher anisotropy (connected, obviously, with more inclined columns) takes place in the SiO_x ODF deposited at $\alpha = 75^{\circ}$ as compared to the films deposited at $\alpha = 60^{\circ}$.

As was stated above, on the contrary to normally deposited films, in oblique deposited films the polarization conversion takes place. The θ value depends on the orientation of light incidence plane relative to the plane of vapor deposition (Ω). The dependences θ on Ω obtained at rotation of the ellipsometer stage with SiO_x ODFs are shown in Figs 1 and 2.

Fig. 1 demonstrates this dependence for the SiO_x films deposited at $\alpha = 60^{\circ}$ on c-Si substrate with (111) orientation of its surface. For this film, the reflected light remains s-polarized when Ω is close to 0°(360°), 90°, 180°, 270° (Fig. 1). For other values of Ω , the light is elliptically polarized. The $\theta(\Omega)$ dependence for this case is close to the dependence $\theta(\Omega) = B\sin(\Omega/2)$, but there are some deviations from the latest one – the absolute value of $\theta(\Omega)$ extrema in the first and fourth quadrants is somewhat smaller as compared to the second and third ones. Such character of the $\theta(\Omega)$ dependence (with 180°-period and some asymmetry in the extrema values) confirms that two principal axes of ε_{ik} tensor are in proximity to the plane of film surface - one of them is nearly normal and other nearly parallel to the direction of the projection of the vapor-beam direction on the substrate surface. And the third principal axis of ε_{ik} is perpendicular to the substrate surface or is somewhat deflected from the surface normal.

The experimental $\theta(\Omega)$ dependence from Fig. 1 agrees in general with the results of papers [13] as well as [18] where the 180° azimuth period has been observed for optical parameters characterizing PC in TiO₂ films with slightly inclined columns ($\beta \le 20^\circ$). So, one may expect that for the SiO_x film deposited at $\alpha = 60^\circ$ the β value is close to 20°. This estimation don't coincide with the values predicted by the most used and recognized models describing the relation between β and α [4, 19, 20]. The tangent rule tan $\beta = 0.5$ tan α [4, 19] for $\alpha = 60^\circ$ gives $\beta \approx 41^\circ$, and the cosine rule sin ($\alpha - \beta$) = (1 - cos $\alpha/2$) [17] for $\alpha = 60^\circ$ gives $\beta \approx 45^\circ$.

Fig. 2 shows the dependence of θ on Ω for the SiO_x film deposited at $\alpha = 75^{\circ}$. The condition $\theta = 0$ ($A_{\min} = A_s$) is valid when Ω is close to 0°(360°), 25°, 180° and 335°. Despite much more complicated form of the $\theta(\Omega)$ dependence for this sample there is some similarity with the previous one. The transformation of $\theta(\Omega)$ dependence from Fig. 1 to Fig. 2 includes further decrease of $|\theta_{max(min)}|$ in the first and fourth quadrants as well as narrowing these quadrants from $\Omega = [0^{\circ} - 90^{\circ}]$ and $\Omega = [270^\circ - 360^\circ]$ to $\Omega = [0^\circ - 25^\circ]$ and $\Omega =$ $[335^{\circ} - 360^{\circ}]$, correspondingly. The second and third quadrants have broadened at the expense of the first and fourth quadrants, correspondingly. As a result, the period of the $\theta(\Omega)$ dependence in Fig. 2 is equal to 360°, and in outline the dependence is close to the $\theta(\Omega) = B\sin(\Omega)$ dependence which is presented by solid line in Fig. 2. In this case, one or two of the principal axes of ε_{ik} were sufficiently deflected from the substrate surface and/or substrate surface normal. It directly shows that more inclined columns are formed in this film.

SEM results confirm the conclusions made on the basis of polarimetric results. The scanning electron imaging of the SiO_x film profiles demonstrates that the films grow in columnar manner, the individual columns run through the entire volume from the substrate plane to the film surface. Besides, the oblique deposition leads to the column inclination toward the substrate plane, the columns being elongated in the vapour beam direction. Fig. 3 shows the cross-sections of the obliquely deposited at $\alpha = 60^{\circ}$ (in the top panel) and $\alpha = 75^{\circ}$ (in the bottom panel) SiO_x films. The β values for the SiO_x ODFs determined by scanning electron microscopy are 26–29° for $\alpha = 60^{\circ}$. These β values are significantly smaller than those predicted by the tangent rule and the cosine rule, but close to β values obtained in [18]. SEM reveals more inclined nanocolumn structure of the SiO_x ODFs deposited at $\alpha = 75^{\circ}$ as compared to the case $\alpha = 60^{\circ}$ (Fig. 3a and 3b). Determined by SEM β values for the SiO_x ODFs deposited at $\alpha = 75^{\circ}$ were 34°–41° in various points of the sample. So, they are also significantly smaller than those predicted by the tangent rule (gives $\beta \approx 62^{\circ}$) and the cosine rule (gives $\beta \approx 53^{\circ}$).

Results of Figs 1 to 3 indicate that the deviation of the $\theta(\Omega)$ experimental dependence from equation $\theta(\Omega) = B\sin(\Omega/2)$ is more pronounced for the films deposited at higher angles and, consequently, with more inclined columns.



Fig. 1. Measured values of θ (crosses) as a function of the sample azimuth Ω for the SiO_x films deposited at $\alpha = 60^{\circ}$ on c-Si substrate with (111) crystallographic orientation. Solid curve is the dependence $\theta(\Omega) = B \sin(\Omega / 2)$ with B = -50'.



Fig. 2. $\theta(\Omega)$ dependence for the SiO_x films deposited at $\alpha = 75^{\circ}$ on c-Si substrate with (111) crystallographic orientation of surface (crosses). Solid line – the dependence $\theta(\Omega) = B \sin(\Omega)$ with B = +65'.

Recently, a semi-empirical analytical model for the β versus α relationship has been proposed [8]. It uses one input parameter, the fan angle, and fits experimental data better than the tangent and cosine rules. Using this model, we have estimated the fan angle in our physical vapor condensation of SiO_x films. The obtained value of the fan angle is 62-68° and may be partially caused by collisions of the evaporated species in vapor flux. Such collisions will cause the loss of directionality of the evaporated particles in their way to the substrate. In recent theoretical investigation [7] by solving the Monte Carlo ballistic model, using diverse calculated incident angle distribution functions in various conditions, it has been shown that the loss of directionality leads to the column tilt angle decrease (it corresponds to the increase of fan angle in terms of the model [8]).

The above described polarimetric technique for the analysis of the ODFs structure has been used to

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investigate SiO_x films deposited on the monocrystalline silicon substrates with different crystalline orientation of the surface (Fig. 4). These samples were annealed in vacuum at 975 °C. Fig. 4 demonstrates that deviation of the $\theta(\Omega)$ experimental dependence from $\theta(\Omega) =$ $Bsin(\Omega/2)$ is a little more pronounced for the film on c-Si(111) substrate. It means that the column tilt angle β is greater in the film on c-Si(111) substrate. A greater tilt angle of columns resulted in greater porosity of these films, but the difference in column tilt angle and, consequently, in porosity is very small. Nevertheless, it was found that the nc-Si-SiO_x films, deposited on c-Si(111), are dissolved by HF vapors more rapidly than the films on c-Si(100).



Fig. 3. Scanning electron micrograph images for SiO_x samples prepared by oblique deposition at 60° (a) and 75° (b) offnormal on c-Si substrate with (111) surface orientation.



Fig. 4. $\theta(\Omega)$ dependence for the SiO_x films deposited at $\alpha = 75^{\circ}$ on c-Si substrate with (100) (circles) and (111) (crosses) orientation of substrate surface, then annealed at 975 °C in vacuum.



Fig. 5. Spectral dependence of PL for the SiO_x films deposited at $\alpha = 75^{\circ}$ on c-Si substrate with (100) (circles) and (111) (crosses) orientation of substrate surface, then annealed at 975 °C in vacuum and HF vapor treated for 1 min.

To control the photoluminescence (PL) intensity and PL peak position of light emitting nc-Si-SiO_x structures, the annealed SiO_x films are treated by HF vapor [21]. As a result of HF vapor treatment, considerable PL intensity growth (near 200 times of magnitude) and blueshift of PL peak position are observed. It is suggested that the evolution of the PL spectra in HF vapor-treated samples can be attributed to selective-etching-induced decrease in Si nanoparticle dimensions and to passivation of Si dangling bonds (that are nonradiative recombination trap states) by hydrogen and oxygen.

Fig. 5 shows the difference in PL spectra of HF treated nc-Si-SiO_x structures deposited on c-Si substrates with different orientation. We suppose that this difference is caused by the difference in column tilt angle in the annealed SiO_x films. Since the films with more inclined columns are more porous (structure deposited on c-Si (111) substrate), they are more susceptible to chemical vapor treatments because the dissolving vapors more easily penetrate into these films. It produces more complete passivation of nonradiative traps and higher PL intensity. More effective vapor treatment causes higher oxidation of the nc-Si surface, more expressed decrease in nc-Si sizes, and some blueshift of the PL spectrum.

These results show an important role of substrate surface in formation of the nanocolumn structure of ODFs. Further comprehensive study of this effect may make significant contribution in elucidation of a structure formation mechanism in SiO_x ODFs and nc-Si-SiO_x structures formed on their base.

From Fig. 4, it could be concluded that a difference in column tilt angle for the films on c-Si(100) and c-Si(111) substrates is not significant. But the difference in the dissolution rate in HF vapors is large. That is why, fixation of small structural differences in SiO_x ODFs is very important, and the proposed polarimetric method makes it with high sensitivity. At the same time, fixation

of this small difference can be problematic with SEM. The reason of that - destructive nature of SEM as technique for analysis of column inclination in ODFs. In order to measure the actual column inclination, one must fracture the sample in the deposition plane. But, SEM is sensitive to misalignment between the deposition plane and the fracture plane, as well as polar misalignment. Obviously, just these errors are the reason of the some scattering of β value at the SEM measurement in various points of our samples. Moreover, the columns located near the fracture site have slightly different inclinations due to edge irregularities, introducing a new source of error [16]. On the contrary, the proposed simple polarimetric technique is very sensitive to the differences in the microstructure of ODFs and, in addition, poses the advantage of non-destructivity.

5. Conclusions

In summary, we have presented a specific simple optical approach for the investigation of obliquely deposited films. This technique is based on the modulation of the normalized off-diagonal terms of the Jones reflection matrix, induced by the rotation of the anisotropic sample. The study of polarization conversion is realized by means of conventional null-ellipsometer, and it reveals the type of optical anisotropy caused by columnar structure of the films and makes it possible to estimate the tilt angle of columns. We apply this method to the characterization of SiO_x films and nc-Si-SiO_x light emitting nanostructures, deposited on silicon substrates. Structural difference between films simultaneously deposited on Si substrates with (100) and (111) crystallographic orientations of surface has been established. The technique may be utilized for characterization of obliquely deposited thin films during their fabrication and processing.

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