

ELLIPSO-METRIC CHARACTERIZATION OF OXIDE LAYERS ON SILICON SUBSTRATE¹

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Method of measuring ellipsometric parameters of polarised light from the surface of examined material enable to specify the optical parameters of thin films. A simple model assumes a nonabsorbing homogeneous layer lying on an absorbing substrate of defined parameters. The growth of oxide layers on Si doped by P and Sb in a low thermal rf generated oxygen plasma was studied. The validity of the assumed model by a selection of measuring parameters - two wavelengths and two incidence angles was verified.

1 Introduction

Ellipsometric measurements on surfaces of multilayer structures rank among the important nondestructive optical methods. The condition of their use is that the layers may be described as a continual medium, characterised by a complex refractive index $\tilde{n} = n - ik$, where n is the index of refraction and k is the extinction coefficient.

The reflection at the planar interface between two isotropic media is described by Fresnel's complex coefficients (given by ratios of the reflected and incident amplitudes of the electric field, polarised parallel and perpendicular to the plane of incidence). These coefficients are functions of \tilde{n}_1 and \tilde{n}_2 being the complex refractive indices of the first and second media respectively, $\tilde{\varphi}_1$ and $\tilde{\varphi}_2$ being the complex angles of incidence and refraction respectively [1].

$$\tilde{r}_p = r_p e^{i\delta_p} = f_1(\tilde{n}_1, \tilde{n}_2, \tilde{\varphi}_1, \tilde{\varphi}_2), \tilde{r}_s = r_s e^{i\delta_s} = f_2(\tilde{n}_1, \tilde{n}_2, \tilde{\varphi}_1, \tilde{\varphi}_2). \quad (1)$$

The ellipsometric parameters, azimuth Ψ and the differential of the phase shifts Δ are determined

$$\tan \Psi e^{i\Delta} = \frac{\tilde{r}_p}{\tilde{r}_s}. \quad (2)$$

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For a clean surface the optical constants of the material can be determined from ellipsometric parameters by direct calculation

$$n_2^2 - k_2^2 = n_1^2 \sin^2 \varphi_1 (1 + \tan^2 \varphi_1 \frac{\cos^2 2\Psi - \sin^2 2\Psi \sin^2 \Delta}{(1 + \sin 2\Psi \cos \Delta)^2}) \quad (3)$$

$$2n_2 k_2 = n_1^2 \sin^2 \varphi_1 \tan^2 \varphi_1 \frac{\sin 4\Psi \sin \Delta}{(1 + \sin 2\Psi \cos \Delta)^2} \quad (4)$$

The first medium is usually air i. e. nonabsorbing one.

In the case, that the substrate is covered by a thin layer, interferences arise in this layer as a consequence of the multiple reflections on the interfaces of the layer. Then the ellipsometric parameters depend on the Fresnel's coefficients and this dependence is expressed in the relation:

$$\tan \Psi e^{i\Delta} = \frac{\bar{r}_{12p} + \bar{r}_{23p} e^{-2i\delta}}{1 + \bar{r}_{12p} \bar{r}_{23p} e^{-2i\delta}} \frac{1 + \bar{r}_{12s} \bar{r}_{23s} e^{-2i\delta}}{\bar{r}_{12s} + \bar{r}_{23s} e^{-2i\delta}}, \quad (5)$$

where \bar{r}_{12} , \bar{r}_{23} are the Fresnel's coefficients for the boundaries and δ is the phase shift of the beam across the layer.

In general, the ellipsometric parameters depend on the refractive index of the surrounding n_1 , on the complex refractive indices \bar{n}_2 , \bar{n}_3 of the layer and the substrate respectively, on the thickness of the layer d , on the angle of incidence φ and many other parameters which include deviations from the ideal model of a thin homogeneous layer. Only two unknowns can be determined from (5), but their analytical expression is not possible. It is possible to find for graphical expressions of the functions $\Delta = f_1(n_1, \bar{n}_2, \bar{n}_3, d, \varphi)$ and $\Psi = f_2(n_1, \bar{n}_2, \bar{n}_3, d, \varphi)$ such a suitable angle of incidence that the sensitivity of the given method is optimal.

This paper deals with the formation of dielectric layers on silicon in a rf generated oxygen plasma. The temperature of the substrate was especially low, the exposure ran by the temperature about 350°C. This temperature was lower about 300 K as the usually used temperature [2]. Therefore the basic optical properties of the layers were necessary to determine.

2 Method and Results

A visual half-shadow ellipsometer was utilised for the ellipsometric measurements. It was constructed by adaptation of a goniometer [3]. Polarizers and a quarter-wave plate in turnable holders with the azimuth with the precision of one hundredth degree were placed into the optical path. The polarizers and the quarter-wave plate are simply changeable which enables measurements at various wave lengths. The adjustment sensitivity of the azimuth is increased by smoothing the half-shadow fields. Two Nakamura's half-shadow plates in front and behind the sample and an objective representing their half-shadow boundary were placed into the optical path.

An optimum angle of incidence φ can be chosen by the goniometer. The optical system enables to adjust the angle of incidence on the sample exactly and to keep the plane of incidence in the field of view for the adjustment of the polarizers.

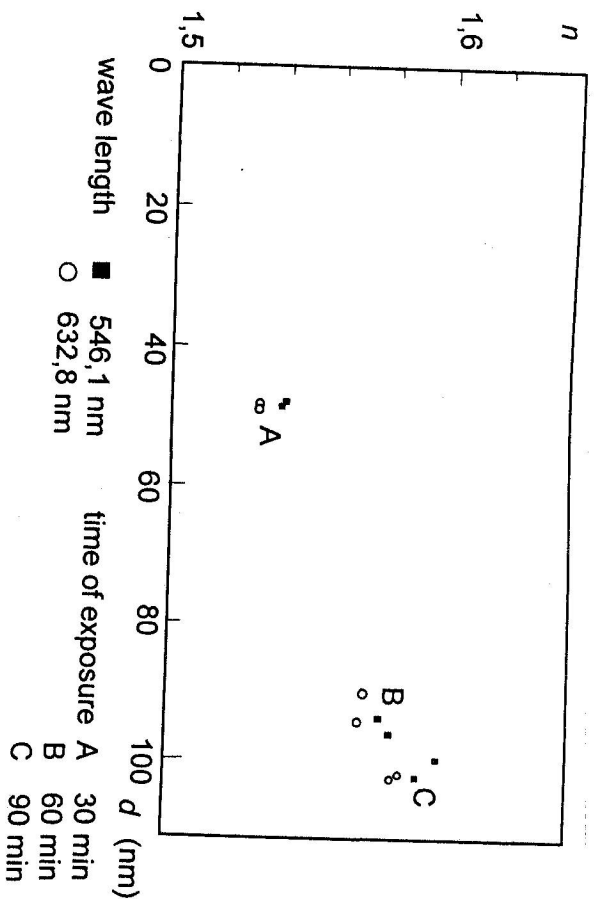


Fig. 1. Dependence of refractive index n on layer thickness d . Si n-type

The changeable quarter-wave plates are designed for two wave-lengths 546.1 nm and 632.8 nm. Their phase-shift for the given wave-lengths are 84.44° and 83.70°, respectively. The rotation of the plane of incidence arising by an incorrect setting of the sample and the inaccuracy of the quarter-wave plates were eliminated by measurements at all possible settings of the polarizer and analyzer and at two positions of the quarter-wave plates with the azimuths $\pm 45^\circ$ [4].

The measurements were made on the samples prepared at the Institute of Physics of the Slovak Academy of Sciences by exposure to O_2 rf plasma. The equipment SECON 200 XPD was used. The vacuum chamber is made from aluminium. Its inner surface is covered by the electrochemically prepared thick ($\sim 1 \mu m$) oxide layer. The frequency and the power of the rf generator was 100 kHz and 100 W respectively. The pressure of oxygen was 50 Pa. The temperature of the sample during the exposure was 350°C. The substrate was (111) oriented silicon, doping level was 10^{18} cm^{-3} , the p-type was doped by B and the n-type by Sb. The thin films were prepared with increasing time of exposure: 30 min, 60 min, 90 min. The prepared films are relatively simple and homogeneous. Their interpretation is based on the idea of one nonabsorbing layer on a substrate. The fabrication used at so low temperature as 350°C lead to nonstoichiometric oxide layers. The growth of the dielectric can be determined by the deposition of both the discharge wall and electrode material due to their sputtering in the rf plasma.

The parameters of clean doped surfaces were measured and the constants of the substrate were determined from the relations (3), (4) for both wave lengths. The results

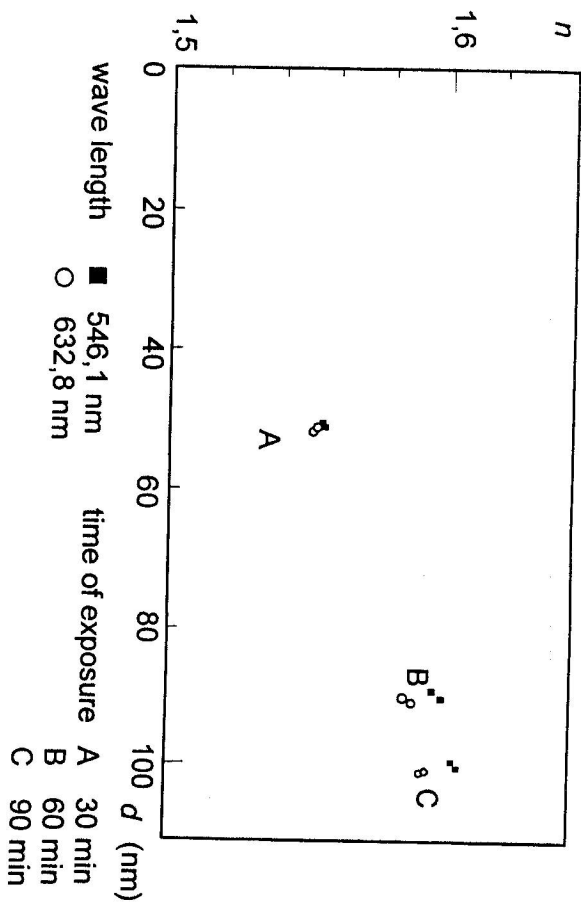


Fig. 2. Dependence of refractive index n on layer thickness d . Si p-type

are in good agreement with the literature [5].

The complex equation (5) enables to determine two real constants. If the optical constants of the substrate are known, the equation can be numerically solved and the index of refraction n and the thickness d of the layer is calculated. The equation (5) can be transformed to the form of a complex quadratic equation with constants, which are trigonometric functions of entrance data and of unknown parameters. The solution have to give a real thickness and a real index of refraction. This solution was found by a numerical method [3].

The measurement has the largest sensitivity for the angles of incidence near the Brewster's angle. To compare the results and to determine the thickness unambiguously, the angles of incidence were 70°C and 75°C .

The ellipsometric parameters have been measured for two wave lengths. The light source has been a He-Ne laser and a mercury lamp with an interference filter for the green spectral line. The calculated thickness of the layer has to be equal for both wave lengths. The thickness is usually determined up to a unknown multiplier of the phase-shift rising in the thin layer. The combination of two measurements enables to determine this constant.

The results of measurements are shown in fig. 1 and 2 for the silicon substrate doped by Sb and B respectively.

3 Conclusions

The calculated constants of the oxide layer show that a model of one homogenous layer is real. That follows from:

1. agreement of the layer thickness measured for two different wave lengths,
2. agreement of the refractive index for two different angles of incidence.

The time dependence of layer growth was obtained without finding an essential difference for differently doped substrates.

The refractive index is in agreement with the refractive index of the oxide layer made by rf plasma. The layers fabricated consist of both silicon and aluminium nonstoichiometric oxides, therefore the measurement by ellipsometry can give higher indices values. The refractive indices of Al_2O_3 measured on anodised layers and on layers prepared by chemical vapour deposition (CVD) with density 2.1 g/cm^3 and 2.9 g/cm^3 , respectively, were determined to be 1.45 and 1.76, respectively [6]. The refractive index of crystalline corundum is 1.76, and the refractive index of Al_2O_3 in antireflective layers is 1.69 [7]. The sputtering of SiO_2 and SiO from the inner wall of a silica reaction chamber was proved, the SiO_2 and SiO reacted with oxygen and created a deposited SiO_2 layer on the Si surface [2]. The relative good agreement of results on both differently doped substrates confirms the deposition of both silicon and aluminium oxides. The refractive index increases with the time of exposure. It is lower for the wave length 632.8 nm as for the wave length 546.1 nm. That is in agreement with the normal dispersion of glass. The growth of the layer slows down exponentially with time of exposure. Ellipsometric measurements enable to choose the parameters of exposure for a required layer.

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