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Optical Constant and Conformality Analysis of SiO_2 Thin Films Deposited on Linear Array Microstructure Substrate by PECVD

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Abstract: SiO_2 thin films are deposited by radio frequency (RF) plasma-enhanced chemical vapor deposition (PECVD) technique using SiH_4 and N_2O as precursor gases. The stoichiometry of SiO_2 thin films is determined by the X-ray photoelectron spectroscopy (XPS), and the optical constant n and k are obtained by using variable angle spectroscopic ellipsometer (VASE) in the spectral range 380–1600 nm. The refractive index and extinction coefficient of the deposited SiO_2 thin films at 500 nm are 1.464 and 0.0069, respectively. The deposition rate of SiO_2 thin films is controlled by changing the reaction pressure. The effects of deposition rate, film thickness, and microstructure size on the conformality of SiO_2 thin films are studied. The conformality of SiO_2 thin films increases from 0.68 to 0.91, with the increase of deposition rate of the SiO_2 thin film from 20.84 to 41.92 nm/min. The conformality of SiO_2 thin films decreases with the increase of film thickness, and the higher the step height, the smaller the conformality of SiO_2 thin films.

Keywords: conformality; plasma-enhanced chemical vapor deposition (PECVD); silicon dioxide (SiO_2); optical thin films

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1. Introduction

SiO_2 is a commonly used optical thin film material with low refractive index. SiO_2 thin films have many advantages, such as high light transmittance, good insulation, good dielectric properties, and strong corrosion resistance. At present, SiO_2 thin films have been widely used in optical film devices, electronic devices, integrated devices, sensors, and other fields [1–6]. However, several new applications for SiO_2 thin films will require conformal coverage (good conformality) of micro- and nano-scale features in the substrate. Conformality, conformal coverage, or step coverage are important for many applications such as array optical filters, microelectronics [7–9], integrated circuit technologies [10,11], and nano-imprint lithography [12,13]. The main methods of preparing SiO_2 thin films are physical vapor deposition (PVD), chemical vapor deposition (CVD), Sol-Gel method, and liquid precipitation deposition (LPD) [14–17]. Generally, when the thin film is deposited on a patterned substrate with CVD technology, it is easy to obtain good conformality, while it is difficult with PVD [18,19].

The conformality or step coverage has been previously reported to depend on deposition parameters, including deposition temperature, total gas flow, and substrate material. Levin et al. studied the relationship between deposition pressure and step coverage of SiO_2 thin films prepared by CVD [20]. Gao et al. studied the effects of precursors and substrate materials on step covering in metal organic chemical vapour deposition (MOCVD) [21]. Machida improved the film step covering by adding bias [22]. Bierner et al. studied the effect of the ratio of gaseous reactants on step coverage in the PECVD of silicon nitride [23]. Özkol et al. deposited hydrogenated amorphous silicon (a-Si:H) films by PECVD and studied the relationship between the conformality of a-Si:H films and deposition temperature [24]. However, the effect of deposition rate, especially substrate size characteristics on

the conformality of SiO_2 thin films, has rarely been addressed. Furthermore, Siriwongrungson, et al. [25] proposed a quantitative measure of the conformality of the thin film deposit over any shape of feature. Conformality is calculated based on a statistical analysis of a number of film thickness measurements of a fracture surface over the features of interest. However, in this method, film thickness is measured by a field emission scanning electron microscope (FESEM). Moreover, it is required to give the thickness of the film at many points on a fracture surface.

In this work, the new method to quantitatively measure the conformality of thin films on patterned substrate is proposed. The optical properties of silicon dioxide thin films prepared by PECVD were investigated, and the influence of deposition rate and the size of substrate steps on the conformality of the thin films were emphatically analyzed.

2. Analysis Methods

The step coverage is generally defined as the ratio of the film thickness at the trench bottom to the thickness on the flat at the trench mouth opening, as shown in Figure 1a. Krumdieck, et al. [25,26] proposed a quantitative step coverage assessment method to describe the conformality of a sample in the dimension of interest. This method of measuring conformality is a statistical method, so it requires measuring the thickness of a large number of films at different locations. Conformality is calculated based on a statistical analysis of a number of film thickness measurements of a fracture surface over the features of interest, as shown in Figure 1b. Conformality (C_1) can be written as shown in Equation (1) [26].

$$C_1 = 1 - \frac{\sum_{i=1}^{M} \left| \delta_i - \overline{\delta} \right|}{\sum_{i=1}^{M} \delta_i} \quad (1)$$

where M is the number of film thickness measurements, δ_i is the film thickness measured at point i, and $\overline{\delta}$ is the average film thickness calculated from all of the measurements. We propose another method to quantitatively measure the conformality of thin films deposited on the surface of steps, as shown in Figure 1c. Conformality (C_2) can be written as

$$C_2 = 1 - \frac{|\alpha - \beta|}{\beta} \quad (2)$$

where β is the angle between the bottom edge of the step and the side of the step on the cross section of the basement step before coating and α is the angle between the side and the bottom of the step on the cross section after coating. d and l are can been measured on a cross section using Hitachi SU1510 scanning electron microscope (SEM, Hitachi, Ibaraki, Japan), or can been measured by Surface Profiler (Taylor Hobson, Leicester, UK), $\alpha = \arctan(d/l)$.

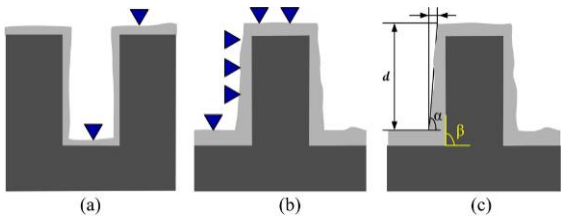

Figure 1. Description of the measurement of step coverage and conformality. (**a**) Step coverage, (**b**) conformality proposed by S. P. Krumdieck, and (**c**) conformality proposed in this work.

3. Experimental Details

SiO_2 thin films are deposited using PECVD technique, from appropriated gaseous mixtures of silane (SiH_4, purity, 99.999%) and nitrous oxide (N_2O, purity, 99.99%). The film deposition equipment is a PD-220 PECVD system produced by SAMCO (Kyoto, Japan), as

shown in Figure 2 schematically. The reaction system is a parallel planar discharge system. The upper electrode is connected to a 13.56 MHz rf power supply, and the lower electrode supports the substrate and is connected to a 13.56 MHz RF bias power supply. The substrate is arranged on a tray and the heating system under the tray heats the substrate. Before SiO_2 thin films deposition, the substrate is bombarded for about 5 min with N_2 discharge (discharge pressure: 5×10^{-2} Pa) of 80 W RF power to improve adhesion between the film and the substrate [27]. The substrate is a single crystal silicon sheet patterned through lithography and etching to obtain step shaped profile. SiO_2 thin films are deposited by reaction of silane and nitrous oxide, in which the flow rate of SiH_4 is 50 sccm, the flow rate of N_2O is 70 sccm, the RF power is 150 W, and the operating temperature is 250 °C. In order to study the effect of the deposition rate of the SiO_2 thin film on the conformality, the gas flow ratio SiH_4:N_2O (50:70), reaction temperature (250 °C), and RF power are kept constant, and the reaction pressure is only changed between 80 and 120 Pa.

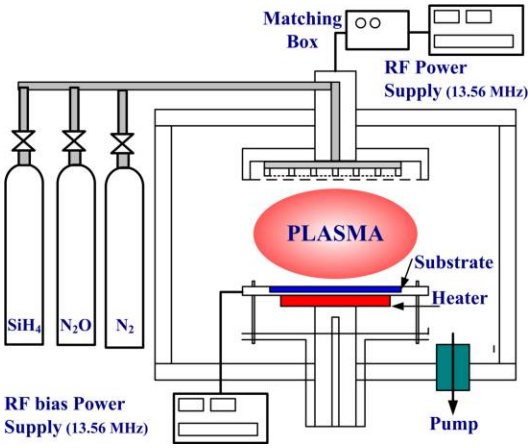

Figure 2. A schematic diagram of the RF PECVD equipment.

The core levels of Si 2p, O 1s, and C 1s are analyzed and the structure of the SiO_2 thin films is characterized by X-ray photoelectron spectroscopy (XPS). XPS analysis is performed on a vacuum generators (Fisons Instruments, Loughborough, UK) MT-500 with a non-monochromatic Al X-ray source (Kα 1486.6 keV) and a CLAM-2 hemispherical analyzer for electron detection. The samples are supported on carbon adhesive tape. The refractive index n, extinction coefficient k, and thickness d of SiO_2 thin films are characterized using a J.A. Woollam M-2000UI (J.A.Woollam, Lincoln, USA) variable angle spectroscopic ellipsometer (VASE) in the wavelength region between 380 and 1600 nm. The cross-section of SiO_2 thin films is observed by a scanning electron microscopy (SEM). Substrate microstructure and SiO_2 thin film surface profile are measured by Taylor Hobson TalySurf CCI-2000 (Taylor Hobson, Leicester, UK) surface profilometer.

4. Results and Discussion

The stoichiometry of silicon oxide thin film is determined by the XPS using the Si 2p and O 1s binding energy. Figure 3 shows the XPS spectrum of the silicon oxide thin film deposited at a substrate temperature of 250 °C. The XPS results showed that (Figure 3a) Si 2p has a peak at 103.3 eV and (Figure 3b) O 1s has a peak value at 533.1 eV, indicating that the deposited silicon oxide thin films are SiO_2 thin films [28]. In case of stoichiometry SiO_2 thin film, the silicon atom surrounded by four oxygen atoms has a characteristic binding energy (103.3 eV).

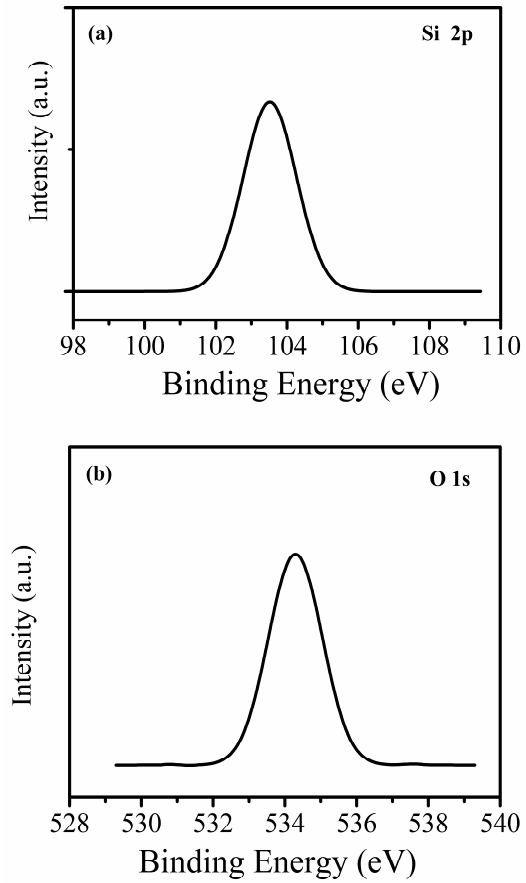

Figure 3. The XPS spectra of SiO_2 thin films deposited by PECVD. (**a**) Si 2p spectra; (**b**) O 1s spectra.

The optical constants *n* and *k* are determined by fitting the ellipsometer parameters in the wavelength region from 380 to 1600 nm. Figure 4 shows the refractive index *n* and extinction coefficient *k* of SiO_2 thin films deposited at a substrate temperature of 250 °C, discharge power 150 W, and working pressure 100 Pa. As can be seen from Figure 4, both the refractive index and extinction coefficient of SiO_2 film change with the wavelength, which indicates that the SiO_2 film has a certain dispersion. The refractive index and extinction coefficient of the deposited SiO_2 thin films at 500 nm are 1.464 and 0.0069, respectively. The results show that the deposited SiO_2 thin films can be used in optical films with low refractive index.

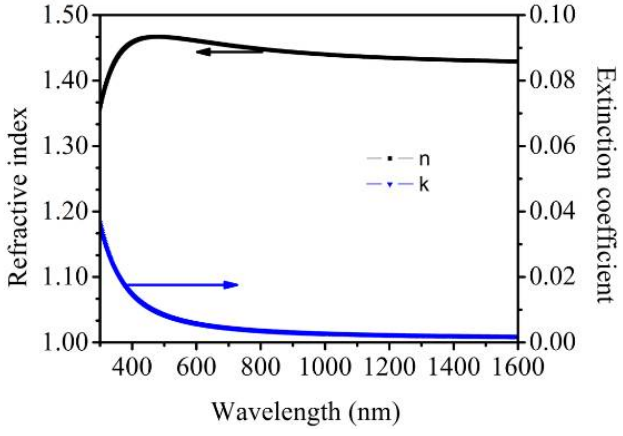

Figure 4. The refractive index (*n*) and extinction coefficient (*k*) of SiO_2 thin film as a function of wavelength.

SiO_2 thin films are deposited at the flow ratio of 50/70 of SiH_4 and N_2O; the substrate temperature is 250 °C; the RF discharge power is 150 W; and the reaction pressure is 80, 90, 100, 110, and 120 Pa, respectively. The deposition rates of SiO_2 thin films are 20.84, 25.76, 30.91, 33.04, and 41.92 nm/min, respectively. SiO_2 thin films of 600 nm thickness are deposited at different deposition rates on linear array micro-structural substrate with a height of 1000 nm and widths of 3, 5, and 10 μm, respectively. The deposition rates of SiO_2 thin films are 20.84, 25.76, 30.91, 33.04, and 41.92 nm/min, respectively. Based on this, the effects of deposition rates on the complex properties are studied.

The SEM images are used to estimate the film thickness at the top surface, side walls, and bottom of 15 different positions on the substrate step, and the conformality (C_1) is calculated by Equation (1). The surface contour of SiO_2 thin films is measured by surface profilometer, and the conformality (C_2) is obtained by Equation (2). Figure 5 shows the image measured by surface profilometer of 600 nm SiO_2 thin films deposited on an array substrate with a height of 1000 nm and width of 5 μm and deposition rate of 41.92 nm/min. As can be seen from Figure 5, the SiO_2 thin films with good conformality can be grown on linear array microstructure substrate at a deposition rate of 41.92 nm/min. Figure 6 shows the conformality results as a function of deposition rate. As can be seen from the Figure 6, under the same conditions, the conformality (C_1) calculated by Equation (1) is slightly less than the value of the conformality (C_2) calculated by Equation (2). The conformality (C_1) and (C_2) of the SiO_2 thin films deposited on the substrate with a step height of 1000 nm and a step width of 3 μm is approximately 0.86 and 0.89, respectively, when the deposition rate of SiO_2 thin film is 41.92 nm/min. Moreover, the conformality of the SiO_2 thin films increases with the increase of deposition rate. The conformality of SiO_2 thin films is proportional to the deposition rate. As expected, at higher deposition rate, the surface reaction is the rate limiting step which produces conformal coverage of the step shapes.

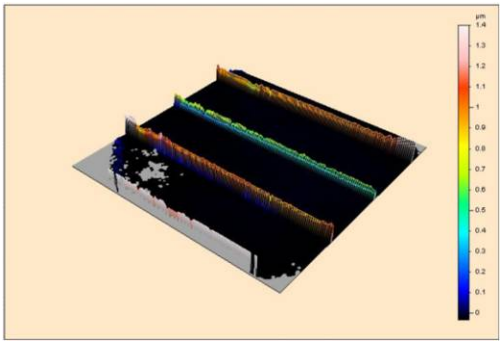

Figure 5. The images of 600 nm SiO_2 thin films deposited on an array substrate measured by surface profilometer.

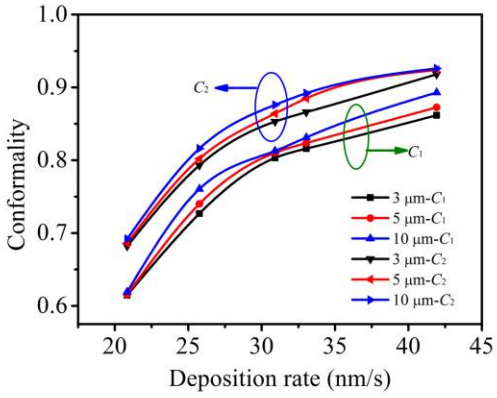

Figure 6. Conformality as functions of the deposition rate.

When the deposition rate of SiO_2 thin films is 30.91 nm/min, SiO_2 thin films of different thicknesses are deposited on 30 substrates of 6 different surface structures with step widths of 3, 5, and 10 μm and step heights of 500 and 1000 nm, respectively. Figure 7 shows the conformality (C_2) as a function of SiO_2 thin film thickness. In Figure 7, the conformality of all SiO_2 thin film samples are calculated by Equation (2). The conformality is inversely dependent on SiO_2 thin film thicknesses. Within a certain range, the thinner the film, the better the conformality; the increase of film thickness, the shadow effect, and the inherent characteristics of film growth will make the conformality decrease. Moreover, the conformality of SiO_2 thin films on the substrate with a step height of 1000 nm is lower than that on the substrate with a step height of 500 nm. At the same step height, the step width has little effect on the conformality of SiO_2 thin films. When the height of the step is 500 nm and the width of the step is 5 μm, the conformality of SiO_2 thin films with thickness of 104 nm is 0.92 and that of the SiO_2 thin films with thickness of 521 nm is 0.71.

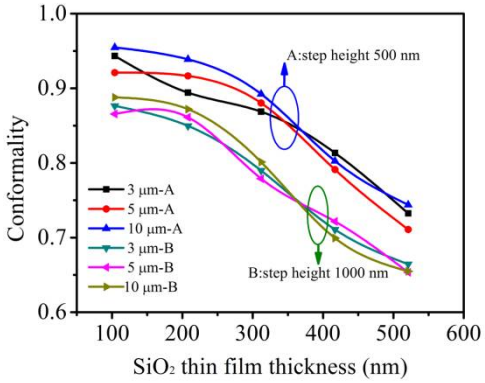

Figure 7. Conformality as functions of the SiO_2 thin film thickness.

5. Conclusions

A method for quantitative evaluation of the conformality of thin films by measuring the angles of step before and after thin films deposition is proposed. SiO_2 thin films are deposited by RF PECVD technique using SiH_4 and N_2O as precursor gases. The experimental results show that the minimum and maximum deposition rates of SiO_2 thin films are 20.84 and 41.92 nm/ min, respectively, when the gas flow of SiH_4 and N_2O is 50 and 70 sccm, the RF power is 150 W, the reaction temperature is 250 °C, and the gas reaction pressure vary between 80 and 120 Pa. The XPS spectrum indicates that the deposited thin film is SiO_2 thin films with a refractive index of about 1.464 at the wavelength of 500 nm.

The results of the film conformality evaluation method proposed in this paper are consistent with the results of the evaluation method given in literature [25], with a slight difference in value. The relation between the deposition rate and the conformality of SiO_2 thin films with the same step height and different step widths is obtained. The relationship between the conformality of SiO_2 thin films and the film thickness is given under different step size characteristics on the substrate. In general, the conformality of SiO_2 thin films prepared by PECVD is proportional to the deposition rate and inversely related to the film thickness.

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