A Novel Scheme for the Deposition and Spectroscopic Ellipsometry Characterization of PECVD, Silicon-based, Dielectric Layers for Optoelectronics Applications

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Abstract

A novel scheme for the PECVD deposition and variable-angle spectroscopic ellipsometry (VASE) characterization of silicon-based dielectrics for both, electronic and optical device application, is described. Depending on the application, different sensitivity and accuracy requirements will be placed on the VASE measurements and subsequent data analysis. As example, we consider dielectric distributed-Bragg reflectors (DBRs) for 980-nm wavelength, vertical-cavity surface-emitting lasers. Pitfalls associated with the nonlinear least-squares method used to extract the physical parameters from the raw ellipsometry data are reviewed. Shortcomings of the widely used Cauchy model for film visible-near infrared dispersion are indicated. Suggestions for improving the confidence of the ellipsometer results are discussed.

INTRODUCTION

The dielectric materials amorphous silicon dioxide (a-SiO_2), hydrogenated amorphous silicon nitride (a-SiN_x:H) and oxynitride (a-SiO_xN_y:H) are of significant interest in the manufacture of microelectronic and optical devices. In recent years, a great deal of attention has been devoted to the growth of dielectric DBRs because of their potential application in optoelectronic devices. They have become attractive for vertical-cavity light emitters and resonant-cavity enhanced photodiodes where they replace the conventional Bragg reflectors based on semiconductor material.

The growth process-film property relationships for silicon-based dielectrics have been studied extensively. Deposition rate (DR) and refractive index (N) are probably the two most basic quantities of interest since they are routinely obtained from either ellipsometric or spectrophotometric methods. During the processing of devices in our laboratory, the ellipsometric determination of DR and N on witness silicon substrates have routinely been used as control parameters for these thin films. The success of this simplified qualification of the film deposition process depends on a reasonably good *a-priori* understanding of the material as well as the errors associated with the ellipsometric method.

A novel scheme for the PECVD deposition and VASE characterization of silicon-based dielectrics for both, electronic and optical device application, is described. Depending on the application, different sensitivity and accuracy requirements will be placed on the VASE measurements and subsequent data analysis. In microelectronic device fabrication, the ellipsometer is basically a metrology tool used to determine values of DR and N of primarily single-layer films with only enough precision to qualify film quality. For optical devices, these quantities have a much more fundamental impact on device design. As a result, the ellipsometer is much more of an analytical tool that is used to measure, not only single-layer thickness and refractive index with maximum precision, but also to extract microstructural and compositional information from complicated multi-layered films. These drastically different, application-driven requirements have significant impact on the ellipsometric approach.

EXPERIMENTAL

The materials grown in this work were accomplished with a production-level PECVD system. Table 1 lists our standard recipes. Noteworthy is the novel use of helium (He) plasma to control intrinsic film stress [1, 2].

TABLE I
STANDARD PECVD GROWTH PARAMETERS

	a-SiO_2	**a-SiN_x:H**
PECVD Conditions:		
Wafer Temperature (°C)	300	300
Pressure (mTorr)	900	800
RF Power (W @ 13.56 MHz)	25	40
Gases (sccm):		
5%-Silane (SiH_4)/Helium (He)	160	170
Nitrous Oxide (N_2O)	900	0
Ammonia (NH_3)	0	4
Nitrogen (N_2)	0	200
Helium (He)	0	600

The VASE measurements were performed with a commercially available ellipsometer of the phase-modulated type. The PME scheme features a photoelastic birefringence modulator with a novel three-element feedback controlled design that results in improved stability and control of the phase-shift modulation process throughout the entire spectral range of operation (450 -1100 nm). The angle of incidence can be continuously varied as well.

The nonlinear least-squares regression analysis was performed on the experimental data with commercially available thin film analysis software employing the Levenberg-Marquardt algorithm, modified to incorporate parameter constraints [3].

The information obtained on the single-layer characterization is applied to the fabrication and testing of a-SiO_2/a-SiN_x:H DBRs for 980-nm wavelength VCSELs.

RESULTS

The VASE characterization of our standard a-SiN_x:H recipe (Table 1) is summarized by the scatter diagram in Fig. 1. Each of the 108-samples which have been deposited to date is represented by a point in Refractive Index/Deposition Rate Space, (N, DR).

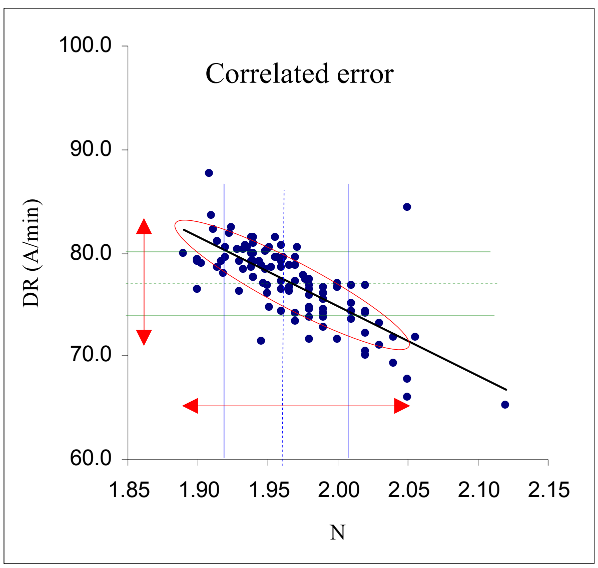


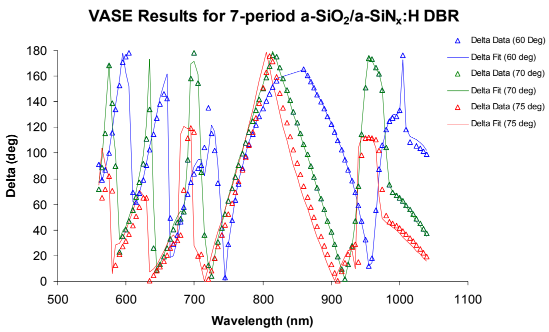
Figure 1 Scatter diagram showing the results of VASE characterization of a-SiN_x:H samples. Each of the 108 samples was deposited by the same PECVD recipe. The solid lines indicate confidence regions in one- and two- dimensions. The same fraction of points (here 68%) lies - (i) between the two (blue) vertical lines, (ii) between the two (green) horizontal lines, and (iii) within the (red) ellipse. The (red) two-headed arrows show the magnitudes of the correlated errors.

Due to time constraints, we typically utilize a single spectroscopic scan from 550 – 900 nm at a 70° angle of incidence. A simple optical model of a single isotropic, non-absorbing layer on an absorbing substrate is assumed. The optical constants of the substrate are taken from tables; whereas the Cauchy model is used for the thin film layer. The software computes the best values for the unknown parameters (N, DR) as well as their associated statistical uncertainties.

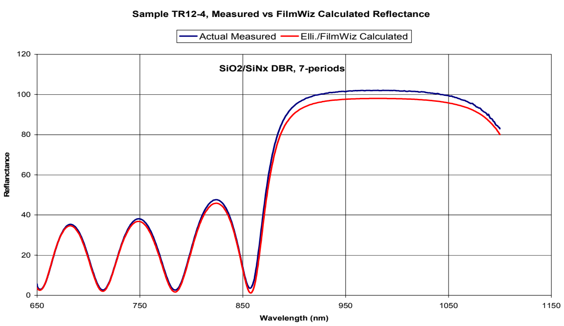
In the ideal case, there is no sensible physical relationship between these variables for a given set of PECVD growth conditions. Therefore, we would normally expect deviations from the mean values to be independent and Gaussian-distributed as a result of run-to-run fluctuations and random measurement errors normally associated with the growth and characterization processes. In the usual way, calculation of the mean and standard deviation could then be used to identify the most probable value and its associated error. Correlation (between parameters) is an effect that is an inherent weakness of the nonlinear least-squares method used to extract the physical parameters (N, DR) from the raw ellipsometry data. In our case, it is manifested by the tendency for higher DR films to have lower N, and vice versa. As a result, we should more properly consider confidence regions, rather than mean and standard deviation, to characterize this film deposition process [4].

Shown in Fig. 1 are three different confidence regions, all at the same confidence level. The two (blue) vertical solid-lines enclose an interval which represents the 68% confidence interval for N without regard to the value of DR. Similarly the (green) horizontal solid-lines enclose a 68% interval for DR. The ellipse shown in the diagram defines the 68% confidence limits for N and DR jointly. Notice that to enclose the same probability as the two independent intervals, the ellipse must necessarily extend outside of both of them. The tangent projections of the elliptical confidence region onto the respective axes, defines the correlated error [4]. These are indicated by the (red) arrows in Fig. 1. Besides the obvious increase in uncertainty, an interesting consequence of this is that there exist values of (N, DR) that are located outside the ellipse, yet are closer to the mean (intersection of dotted lines) than some of the interior points. Despite being closer to the mean, these exterior points are less probable than any of the interior ones. Sometimes it is difficult to realize that a suggested point lies outside a region in a multi-dimensional space.

The information obtained on the single-layer characterization is applied to the fabrication of SiO_2/SiN_x-Bragg reflectors for 980-nm wavelength, vertical-cavity surface-emitting lasers. Fig. 2 illustrates the characterization for the case of a seven period (14-layer) DBR. Fig. 2(A) shows a good quality of fit to the VASE data. Fig. 2(B) compares a direct measurement of reflectance to that calculated from the VASE results. Almost perfect agreement between the two is observed.



(A)



(B)

Figure 2 Characterization of a seven period DBR. (2A) VASE results showing a good quality of calculated fit (lines) to the raw data (points). (2B) The comparison of direct reflectance measurement to calculation using the VASE results.

DISCUSSION

From a production standpoint, especially for optoelectronic devices like lasers and filters which can be extremely sensitive to material refractive index and/or absorption values, the correlated error can be the difference between a successful run and wasted material.

We have had some success reducing this correlated error on a limited number of samples utilizing some of the common techniques [5, 6]. The basic approach involves taking more and/or better data. More data in the sense several angle of incidence can be utilized for measurement. Better data in the sense that an optimal VASE configuration which maximizes sensitivity of the data with respect to the parameter of most interest could be sought. Unfortunately these steps are fairly heroic, in terms of both time and computational resources, especially for casual ellipsometry users.

A key reason the correlation effect is so large in our case is the ellipsometry measurements are restricted to the visible and very near-IR spectral region, about 560 – 860 nm. As a result, the refractive index of this dielectric (Si_3N_4) is modeled using the well-known Cauchy approximation

$$n(\lambda) = A_n + \frac{B_n}{\lambda^2}$$

The nonlinear least-squares method compares the model to the data, minimizes the root-mean-square deviations, and in our case, extracts the parameters (thickness, A_n, B_n). Since thickness is on the order of 10s or 100s (of nanometers), A_n is on the order of 1 - 2, and B_n can be about 10^{-3} – 10^{-5}, we have widely varying magnitudes of the parameters to be determined. In mathematical terms, the system of difference equations generated by our least-squares method suffers from what is referred to as ill-conditioning [6]. The impact is that the accuracy and precision of B_n is difficult to determine and the correlation effect described above is exacerbated. An ellipsometer operating from the UV through the NIR spectral range solves this problem, not by simply generating additional data; but more importantly, by enabling the use of more realistic (Kramers-Kronig consistent) dispersion models for the optical constants.

CONCLUSIONS

Main conclusions of this work are as follows:

(1) A novel scheme for the low-stress, PECVD deposition and PME/VASE characterization of silicon-based dielectrics is described.

(2) Correlated error is approaching 2-times larger than the uncorrelated error for both DR and N in the case of our standard Si_3N_4 recipe. This degree of correlation is sufficiently small for electronics applications; however, it is too large for optical applications.

(3) Shortcomings of the widely used Cauchy model for film visible-near infrared dispersion were indicated. Suggestions for improving the confidence of the ellipsometer results were discussed.

(4) The method has proven to be a useful technique for the deposition and characterization of complicated, multi-layer dielectric structures for optoelectronics applications.

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ACRONYMS

PECVD: Plasma-Enhanced Chemical Vapor Deposition
VASE: Variable-Angle Spectroscopic Ellipsometry
DBR: Distributed-Bragg Reflector
VCSEL: Vertical-Cavity Surface-Emitting Laser
a-SiO_2: Amorphous Silicon Dioxide
a-SiN_x:H: Hydrogenated Amorphous Silicon Nitride
a-SiON:H: Hydrogenated Amorphous Silicon Oxynitride
DR: Deposition Rate
N: Index of Refraction
PME: Polarization-Modulated Ellipsometry