Development of the Nitride Film Thickness Standard (NFTS)

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ABSTRACT

The semiconductor industry has been demanding film thickness reference material for films other than thermally grown silicon dioxide for sometime. To meet this challenge, Nitride Film Thickness Standard (NFTS) has been developed in four nominal thickness values, 20.0 nm, 90.0 nm, 120.0 nm and 200.0 nm. These are silicon nitride (Si_3N_4) films on silicon crystal substrate. Work is underway to develop a 9.0 nm standard. Thin nitride films are particularly needed for calibration of the thickness of nitride layers in capacitors and isolation masks for LOCOS (local oxidation of silicon). The reference material is certified for derived film thickness. The study consists of measurements made on four different sets of wafers that included patterned and unpatterned wafers. The measurements made on these wafer sets were used for answering issues related to film stability and cleaning. The stability study includes the search for a cleaning process that will restore a prior surface condition. On two sets of wafers two different types of cleaning procedures were used. Results indicate that a sulfuric acidmegasonic clean will etch the nitride film while an isopropyl alcohol clean followed by a deionized water rinse can be used over and over again. The third set of wafers was never cleaned and measurements were made on these over a period of two years. The last set of wafers is patterned. These are cleaned prior to measurement. Results show that LPCVD silicon nitride films are stable and can be used with confidence over a long period of time for calibrating optical metrology instruments.

Key words: thin dielectric films, reference material, calibration standard, ellipsometry, metrology, silicon nitride, thin films.

1. INTRODUCTION

The preferred approach to establishing and maintaining accuracy of measurement tool performance is through the use of reference materials. Reference materials are physical objects with one or more well established properties used to calibrate metrology instruments. Since they set the standard for comparison of data taken by different methods, by similar tools at different locations or between model and experiment, reference materials are critical part of metrology. For equipment manufacturers the cost of an incorrect decision can be in millions of dollars. The process engineer has to ascertain that the metrology equipment is making correct measurements. The manufacturing process is producing product and not scrap. Hence the need for reference materials is critical. The need for thin film reference materials is even more critical because thickness measurement is one of the first measurements made in IC fabrication.

The National Technology Roadmap for Semiconductors (NTRS)[1], 1997 has predicted 3 to 4 nm gate oxides for 1999 and 2-3 nm for the year 2001. Beyond that materials other than $SiO_2$are envisioned. Near term gate dielectric solution requires the fabrication and use of ultra thin silicon dioxide, oxynitride films or silicon nitride films. The latter film shows attractive boron diffusion penetration resistance and a moderately higher dielectric constant value of 7. Front end processing requires the growth and deposition of high quality, uniform and defect-free insulating films. These are critical for evolving gate dielectric materials, first for ultra thin oxides, oxynitrides and nitrides, and later for higherκ dielectrics. These requirements include reference materials for gate dielectrics for non-SiO_2 materials.

The demands on thin film metrology are numerous, but the payoffs from properly executed metrology are also substantial. Diebold and Monahan[2] contend that metrology reduces the cost of manufacturing by bringing in more robust processes, preventing scrap, and ramping and maintaining yield. They list the need for reference materials and standard methods for gate dielectrics and thin films among the most difficult challenges facing metrology . These reference materials and calibration standards have been a key factor in assuring the measurement quality of the instruments. These standards should be stable and yield accurate and repeatable certified values. In addition, cleaning and handling techniques that will not alter the certified values, are needed. In a nutshell, the standard, cleaning procedure, instrumentation factors and data analysis all need to be defined accurately and simultaneously[3,4,5].

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The thin film community desires that dielectric film standards should be available and certified for both thickness and refractive index. It should be possible to measure the standard on an ellipsometer or other film thickness evaluation equipment (e.g. reflectometer). The results from a recent survey[6] of optical characterization methods for materials indicate that for thickness evaluation an ellipsometer is the instrument of choice. This survey represents a broad view of the semiconductor industry and includes material suppliers, device manufacturers and makers of characterization equipment. Ellipsometers are optical instruments highly sensitive to the properties of thin films. They have been traditionally used in semiconductor metrology. The National Institute of Standards and Technology (NIST) has used a high accuracy single wave ellipsometer for certifying the SRM 2530 series of film thickness reference material. Over the last decade spectroscopic ellipsometry (SE) has become an important tool for the study[7-12] of thin films. The advent of computer control andmulti-channel detectors have made it possible to develop spectroscopic ellipsometers that can scan an entire range of wavelengths using an entirely numerical data acquisition and processing system. Most commercial spectroscopic ellipsometers measure a series of $\tan\Psi$ and $\cos\Delta$ (see theory) values which are then used to determine multiple useful parameters through data regression. This includes thickness and optical properties of multilayer thin film stacks. Coupled with effective medium theories spectroscopic ellipsometry enables the study of microstructures of deposited thin films. This is of importance for both semiconductor and optical coating technologies.

Currently the National Institute of Standards and Technology (NIST) supplies reference materials[13] for thermally grown silicon dioxide film on silicon crystal substrate for six nominal thickness values. The lowest nominal thickness value in this set is a 10 nm oxide film and the highest is 200 nm. Recently, a Cooperative Research and Development Agreement (CRADA)[14] CN-1364 between the Semiconductor Electronics Division at NIST and VLSI Standards, Inc. was completed. One of the objectives of this collaboration was to develop and test artifacts with SiO_2 film thickness values less than 10 nm and devise a method by which transferability and traceability to NIST could be established in a straightforward way. The procedure tested during the collaboration was to serve as an initial evaluation of a method to provide a NIST Traceable Reference Material (NTRM). In an effort to meet the demands of the industry and to supplement the silicon dioxide reference material, it was decided to come up with a silicon nitride film thickness standard. After silicon dioxide, silicon nitride is the next most well understood dielectric material. Additional reasons for selecting silicon nitride are:
(1) Silicon nitride films act as an extremely good barrier to the diffusion of water and sodium. These impurities cause device metallization to corrode or devices to become unstable. For this reason silicon nitride films are used inpassivating silicon devices to increase the electrical stability.
(2) Silicon nitride is used as a mask for the selective oxidation of silicon. The silicon nitride is patterned and the exposed silicon substrate is oxidized. The silicon nitride oxidizes slowly and prevents the underlying silicon from oxidizing. This process of selective oxidation produces nearly planar device structures
(3) The reduced-pressure technique (LPCVD), used for the production of the nitride standard has the advantage of good uniformity and high wafer throughput.

2. SAMPLE PREPARATION

The nominal thickness values selected are 20.0 nm, 90.0 nm, 120.0 nm and 200.0 nm LPCVD silicon nitride on silicon crystal substrate. The nominal thickness values were determined after a market survey. For instance, we selected the nominal thickness of 20.0 nm after learning that memory makers need this thin nitride film for calibration of the thickness of the nitride layer in capacitors and isolation masks for LOCOS. The silicon wafers used meet the SEMI Standard Specifications according to M1, *Specifications for Polished Monocrystalline Silicon Wafers*. The silicon material is boron doped, p-type 100 surface (P<100>) prime silicon. The resistivity range is 5 - 20 ohm-cm. The wafers are polished on the front side.

The deposition of silicon nitride is carried out by reactingdichlorosilane and ammonia at a reduced pressure of 150milli torr, at a temperature of 800 ° C. The low pressure technique has the advantage of producing good film uniformity. Both gases used are top grade Scott Specialty gases (99.9997 VLSI grade). The chemical reaction is,

$$3SiCl_2H_2 + 4NH_3 \rightarrow Si_3N_4 + 6HCl + 6H_2$$

The activation energy for silicon nitride deposition is 1.8 eV (41 kcal/mole). A slow deposition rate was maintained by keeping the ammonia to dichlorosilane ratio fairly high (3:1). This leads to a uniform deposition of the nitride film. The time of deposition is adjusted to yield different film thickness values.

3. DESIGN VERIFICATION ACTIVITIES

The standards were developed over a period of two years. Development and certification of reference materials involves several generic challenges[15]. These include, (1) the standard should remain stable during use (variations in the certified value should lie within the stated calibration uncertainty), (2) measurement and certification of the standard must be carried out using standardized and well documented test procedures, and, (3) uncertainties in the certified value of the standard must be less than ¼ of the variability of the manufacturing process to be evaluated or controlled by the instrument calibrated using the standard. In the case of dielectric thin films an additional challenge is to come up with a cleaning procedure that will not alter the characteristics of the standard. Over an extended period of time, an organic film may develop over the dielectric film. This film needs to be removed from the surface without affecting the properties of the standard. The nitride standard reported in this paper was developed keeping these guidelines in mind.

For the development of the nitride standard, a spectroscopic ellipsometer was used. There are numerous advantages in using a spectroscopic ellipsometer instead of a single wavelength ellipsometer. It does not suffer from thickness order ambiguity, it is more precise and offers more flexibility during data analysis due to the larger amount of data acquired over a large wavelength range. However, unlike the situation for SWE measurements at 632.8 nm, there is some disagreement in the literature regarding the dielectric function of silicon over the visible spectral range used for SE measurements. Recent studies[4,16] have shown that various choices for the silicon dielectric function and for parametrically modeling the optical index of the dielectric film (oxide) can lead to an unacceptably large variation in the derived thickness and index of the oxide with all modeling choices having comparable goodness of fit to the measured parameters. Improvements in the values for silicon substrate dielectric function are possible through measurements on specially prepared hydrogen-terminated samples. Unfortunately, while there are established chemical treatments for achieving this for (111) silicon, no comparable treatments have been identified for (100) silicon.

3.1 Theory

Ellipsometry is based on the principle of polarization of light[17], and relates film thickness and other optical parameters to the change in the state of known polarization on reflection. Polarized light consists of two components: one oscillating perpendicular (s component) and one oscillating parallel (p component) to the plane of incidence. The equation,

$$\rho = \frac{R_p}{R_s} = \tan \Psi e^{i\Delta} \qquad (1)$$

relates the measured ellipsometric parameters Δ and Ψ to the ratio of the amplitude reflectivities for p and s polarized light. This in general, is a complex number since there is a relative phase shift between the p and s reflected waves, and so ellipsometry at one angle of incidence yields two known quantities The advantage of the method is that by taking a ratio of reflectivities the method does not need intensity normalization which direct measurements of the absolutereflectivities do. In general, the ratio ρ is a function of several parameters,

$$\rho = \rho\ (d, N_0, N_1, N_2, \phi, \lambda). \qquad (2)$$

where d is the thickness of the film, N_0, N_1 and N_2 are the refractive indices of the ambient, film and the substrate respectively, ϕ is the angle of incidence and λ is the wavelength of the source. If the refractive indices of the ambient and the substrate, the angle of incidence and the wavelength of the source are known, then upon measuringΔ and Ψ, the film thickness and film refractive index, may be deduced. Spectroscopic ellipsometry (SE) is amonolayer sensitive optical characterization technique used widely to study thin films and properties of semiconductor materials. The SE employed for the measurements is a rotating polarizer type ellipsometer and is equipped with a photodetector. The intensity varies as a

function of the polarizer angle and is given by[12],

$$I = I_0 \frac{\cos^2 A}{\tan^2 \psi + \tan^2 A}[1 + \alpha \cos 2P + \beta \sin 2P] \tag{3}$$

where the parameters α and β are obtained from the detector signal by a Hadamart transform and are given by

$$\alpha = \frac{\tan^2 \psi - \tan^2 A}{\tan^2 \psi + \tan^2 A}, \quad \text{and} \quad \beta = \frac{2 \tan \psi \tan P \cos \Delta}{\tan^2 \psi + \tan^2 P}. \tag{4}$$

The ellipsometric parameters are obtained directly from the detector signal as $\tan\psi$ and $\cos\Delta$,

$$\tan \psi = \tan A \sqrt{\frac{1+\alpha}{1-\alpha}}, \qquad \cos \Delta = \frac{\beta}{\sqrt{1-\alpha^2}}. \tag{5}$$

SE measurements yield about a hundred values of $\cos\Delta$ and $\tan\Psi$ as a function of wavelength. The measured parameters $\cos\Delta$ and $\tan\Psi$ do not convey much information about the surface. In order to characterize the layered structure, it is necessary to solve the inverse problem, with the objective to find those values of the model parameters (thickness and refractive index of the film) that best match the computed and experimentally measured values of $\cos\Delta$ and $\tan\Psi$. It is these model parameters obtained by solving the inverse problem that are of more practical interest than $\cos\Delta$ and $\tan\Psi$. A regression technique based on the Levenberg-Marquardt-Fletcher algorithm is used for the calculation of thickness and refractive index[18]. A Cauchy dispersion model is used for the refractive index of the nitride film. The spectrum of refractive indices for silicon is taken from Ref. (19). The ellipsometric measurement and mathematical modeling techniques have been discussed in details by a number of authors[7,8,16,17,20,21].

3.2 Instrument Calibration

The spectroscopic ellipsometer was calibrated using the NIST SRM 2530 series. These Standard Reference Materials (SRM's) consist of thermally grown silicon dioxide film on silicon with nominal thickness values 10 nm, 14 nm, 25 nm, 50 nm, 100 nm and 200 nm. These SRM's are measured on the ellipsometer at regular intervals. Figures 1 and 2 illustrate the monitored thickness values for two of them over a period of two years. The NIST certified values for SRM 2536 # 1016-6 is 11.6 ± 0.5 nm and for SRM 2532 # 706-B-27 it is 100.2 ± 0.5 nm. GA refers to the grand average of thickness for all the

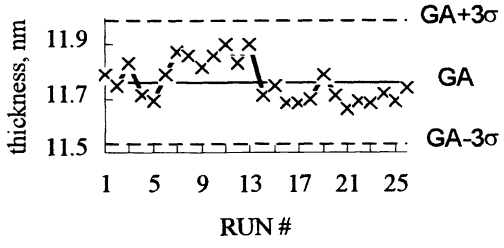

FIGURE 1: Precision and stability data for NIST SRM 2536 # 1016-6

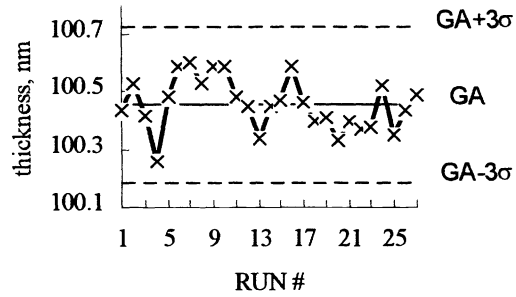

FIGURE 2: Precision and stability data for NIST SRM 2532 # 706-B-27

runs and σ is the standard deviation for the same. In addition to the SRM 2530 series, one set of wafers from NIST Cooperative Research and Development Agreement[14] (CRADA) CN-1364, is also routinely measured on the instrument to check its calibration. These include nominal thickness values of 4.5 nm, 7.5 nm, 10 nm, 50 nm, 100 nm and 1000 nm thick thermally grown oxide films.

3.3 Repeatability study

The initial set of wafers contained two wafers of nominal thickness value 9 nm, and four each of nominal thickness values 90 nm, 120 nm and 200 nm LPCVD (low pressure chemical vapor deposition) silicon nitride on silicon crystal substrate. None of these wafers are patterned. All samples selected were free of any contamination, microscopic defects and cosmetic flaws. The samples were then inspected for flatness, uniformity and manufacturing tolerances. Each wafer is measured at five locations, center, top, bottom, left and right. The off-center points are 10 mm from the center. In order to achieve high sensitivity, all spectra were recorded at an incident angle in the vicinity of 75°, which is close to both theBrewster angle and the principal angle for the silicon substrate. At the principal angle the ellipsometric parameter Δ equals 90° and Ψ equals the polarizer azimuth angle. Under these conditions, the reflected light is circularly polarized, there is greater sensitivity to small changes in the sample and highly accurate data are obtained. A wavelength window ranging from 250 to 650 nm is chosen for the 9 nm sample and 300 - 800 nm for the rest of the samples. Measurements were made at 100 spectral points. All thick samples (90 nm, 120 nm and 200 nm) were measured seven times over a period of 16 months. To determine the Si_3N_4 film thickness, the three-phase model consisting of Silicon substrate, Si_3N_4 film, and ambient was used in fitting the experimental data. For each wafer, Si_3N_4 film was modeled as a single homogeneous transparent layer. For the thick samples both thickness and refractive index are obtained simultaneously using Cauchy dispersion law. For the 9 nm and 20 nm, samples the spectrum of refractive index is taken from (Ref. 22) and only the thickness value is iterated. Figures 3 and 4 illustrate the results for two of the thickest standards developed. These results indicate that the repeatability of the sample thickness is

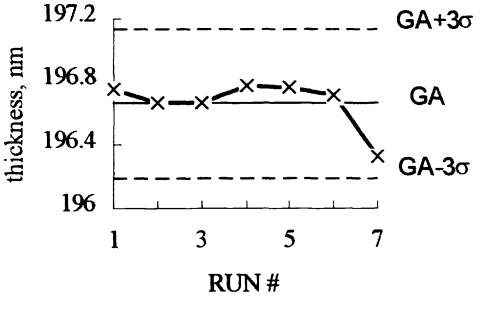

FIGURE 3: Repeatability data for SN 3726-003.

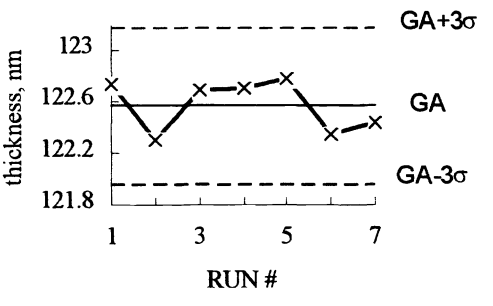

FIGURE 4: Repeatability data for SN 3727-003.

very good. Since the completion of this study one set of wafers are being measured regularly and are being maintained as control chart wafers. These include one of each thickness values. The results for the 90 nm sample is shown in figure 5. With silicon nitride there is always the concern that the characteristics of the film, change over time leading to a change in the refractive index. The refractive index plotted in figure 6 is for the sample shown in Fig.5. The first run was made in

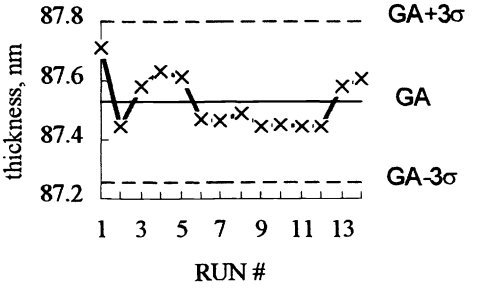

FIGURE 5: Repeatability data for SN 3706-003.

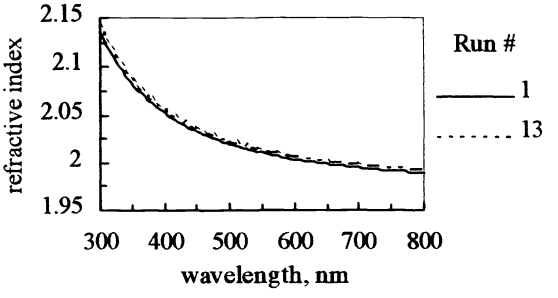

FIGURE 6: Comparison of refractive index for RUN # 1 and 13 for SN 3706-003.

December 1995 while the thirteenth run was made in March 1998. These wafers have never been cleaned and are stored in Fluoroware® containers in a class 1000 cleanroom. There is no discernible change in the refractive index of the nitride film. Similar observations are made for other samples used in the study. At the wavelength 632.8 nm, the refractive index changes from 2.00035 to 2.0042 between the two runs shown in Fig. 6. In order to verify that the manufacturer makes consistently stable product, another set was manufactured in October 1996. The new set of wafers consisted of three sets of Si_3N_4 film thickness products (nominal thickness values 90, 120 and 200 nm). Each set consisted of twenty five wafers. Three wafers were selected from each thickness value for verification purposes. Three sets of measurements were made on each sample in this set. It was determined that the repeatability of the products was extremely good.

For the 20 nm and 9 nm samples a different time table was followed. The 20 nm samples were included in the study only after August 1997. The 9 nm samples were initially included in the study but then it was decided that first the thicker samples should be studied. Once we are satisfied with the quality and characteristics of thick nitride film we could restart work on the thin samples. For the two thinnest samples measurements were made in Dec. 95, Jan. 96 and then every month

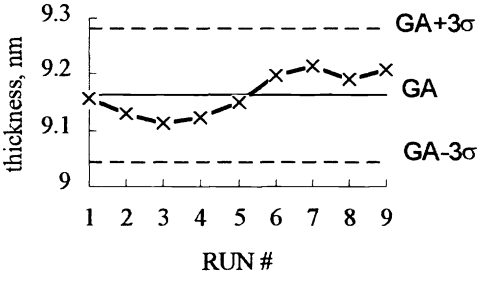

FIGURE 7: Repeatability data for SN EL-08.

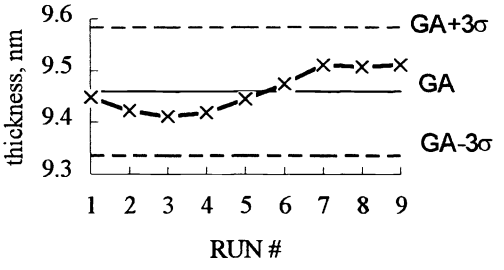

FIGURE 8: Repeatability data for SN EL-09.

since Aug. 97. Figures 7 and 8 show the repeatability data for these two samples. These samples were cleaned before making the first run but since then no cleaning has been performed. Both samples exhibit excellent precision and repeatability. There seems to be no major change between runs two and three that were taken in an interval of about 19 months! The charts exhibit that there is no apparent film growth and the standard deviations produced indicate that we can have uncertainties less than 0.1 nm for 9.0 nm silicon nitride films.

4. CLEANING PROCEDURES AND FILM STABILITY

Aside from repeatability and precision, another characteristic that should be exhibited by a dielectric film thickness reference material, is stability. In some quarters, more weight is given to film stability than accurate knowledge of film thickness. Three sets of wafers were used for this study. Two sets were not patterned and the third set was patterned. Each set consists of three wafers with nominal thickness values 90 nm, 120 nm and 200 nm. All wafers were measured prior to cleaning.

4.1 Stability study

The measurements made on these wafer sets were used for answering issues related to film stability and cleaning. The stability study includes the search for a cleaning process that will restore a prior surface condition. On two sets of wafers two different types of cleaning procedures were used. The first set of wafers was cleaned using a sulfuric acidnegasonic clean. This set will be referred to as "cleaning set 1". The wafers were cleaned in a sulfuric acid bath for twenty minutes at about 130-140 ° C. This was followed by deionized water rinse, megasonic (approximate medium: one gallon hydrogen peroxide plus quarter gallon ammonium hydroxide in deionized water) for 300 seconds, de-ionized water rinse and then dried. This

method of cleaning proved to be hazardous to the integrity of the nitride film. Table I shows the reduction caused by cleaning the wafers twice. Each value of thickness shown is the average of the five measurements made on the wafer surface. After cleaning only once reduction in thickness as high as 6.4 nm were observed. Also the film surface was rendered

Table I: Effect of sulfuric acid megasonic clean on silicon nitride film.

Wafer	Before cleaning		After clean 1		After clean 2	
Serial Number	thickness, nm	σt	thickness, nm	σt	thickness, nm	σt
SN 3706-010	86.55	0.1233	80.162	0.2109	79.858	0.2372
SN 3727-010	119.786	0.3157	114.512	0.4992	113.65	0.6024
SN 3726-010	195.198	0.4615	191.662	0.8515	191.834	0.8859

non-uniform. Before cleaning, the variation of the film thickness over a circle of radius 10 mm was less than 0.2 nm for all the samples. It increased to as much as 1.59 nm after the first clean. This method of cleaning was abandoned following the second clean. The second set of wafers (cleaning set 2) were treated to a gentler cleaning procedure. The sequence included

Table II: Effect of IPA clean on silicon nitride film

Wafer	Before cleaning		After clean 1		After clean 2	
Serial Number	thickness, nm	σt	thickness, nm	σt	thickness, nm	σt
SN 4181-010	90.888	0.1498	90.768	0.1558	90.704	0.1128
SN 4183-010	118.79	0.3168	118.536	0.3103	118.832	0.2864
SN 4182-010	205.582	0.4228	205.17	0.4416	204.916	0.4162

deionized water bath, isopropyl alcohol (IPA) rinse, deionized water rinse, megasonic for 120 seconds followed by deionized water rinse and dry. Table II summarizes the results of the second cleaning procedure. In this case the results look much more encouraging. In this case cleaning did not cause variations in film thickness over the 10 mm radius measurement area. The maximum thickness variation was observed for the 200 nm sample which changed from 0.41 nm before cleaning

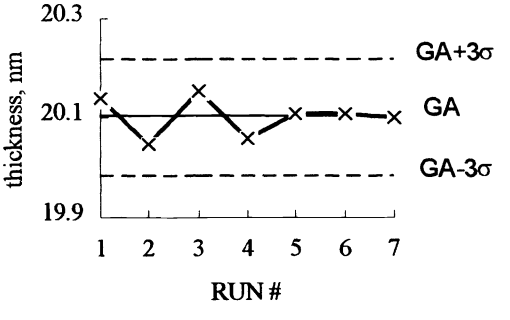

FIGURE 9: Stability of SN 4813-010. 4 cleans in IPA bath.

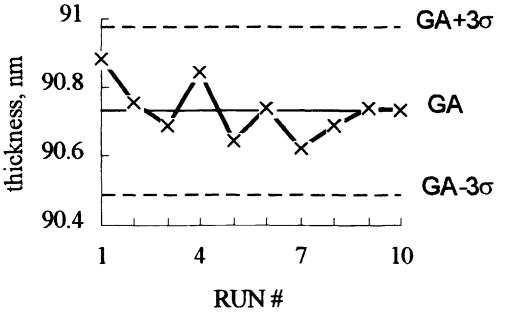

FIGURE 10: Stability of SN 4181-010. 7 cleans in IPA bath.

to 0.47 nm after two cleans. These three samples have been cleaned four more times since then. At this time a 20.0 nm sample was also included in the study. Figures 9 and 10 show the stability of the 20 nm and 90 nm sample after successive cleans performed in an IPA bath. The last three measurements were made on these samples without further

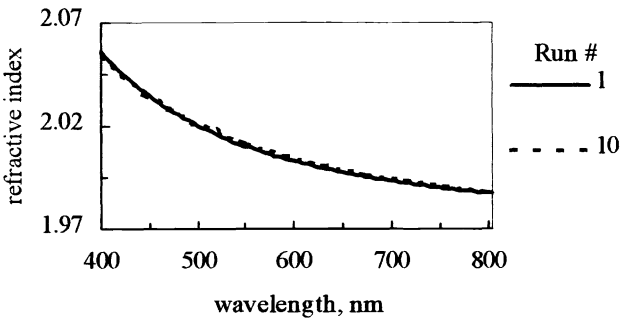

FIGURE 11: Comparison of refractive index of SN 4181-010 for RUN # 1 and 10.

cleaning. Figure 11 shows the comparison of the film refractive index between the first and the tenth runs which were taken more than a year apart. It should be pointed out that after the third run measurements were made in the wavelength range 400-800 nm. The value of the refractive index changes from 2.0558 to 2.0530 at 400 nm source wavelength, and from 1.9992 to 2.0003 at 632.8 nm. The results in Figs. 9, 10 and 11 demonstrate that the nitride film is stable and the optical constants remain unchanged over time.

4.2 Patterned wafers

Three wafers with nominal thickness values 90.0 nm, 120.0 nm and 200.0 nm are chosen for this study. A total of seven sets of measurements were taken between September 1997 and March 1998. The first run was made before the wafers were cleaned or patterned. The rest of the measurements were made after the wafers were cleaned and patterned.

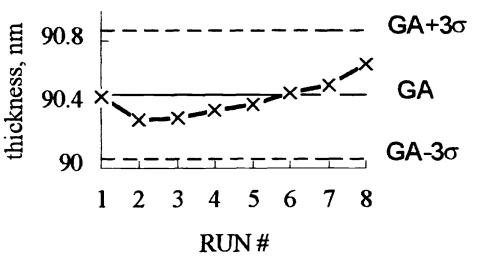

FIGURE 12: Stability of SN 4181-004, cleaned once in IPA bath.

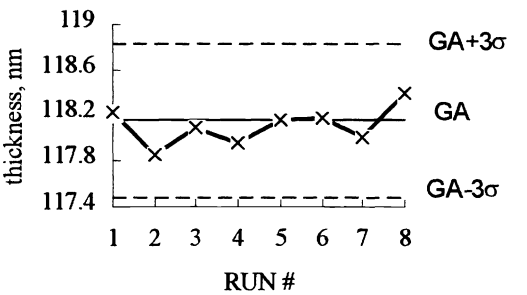

FIGURE 13: Stability of SN 4183-004, cleaned once in IPA bath.

Figures 12, 13 and 14 illustrate the film stability of these three wafers over time. In all three cases, it is observed that the variations in film thickness are within allowed uncertainties. In this case also, measurements were made in the

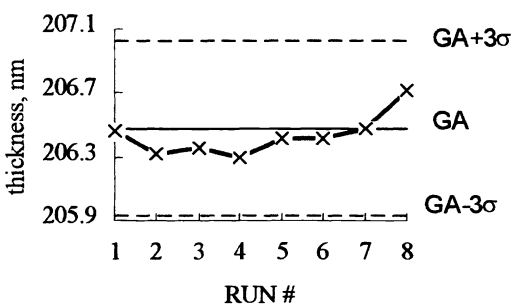

FIGURE 14: Stability of SN 4182-004, cleaned once in IPA bath.

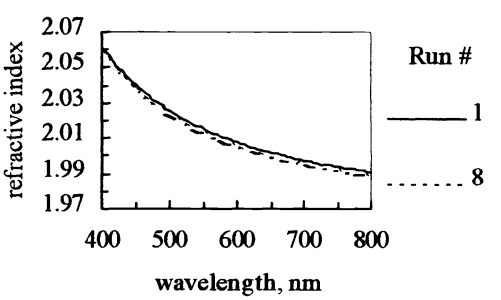

FIGURE 15: Comparison of refractive index for SN 4182-004, RUN # 1 and 8.

wavelength range 400-800 nm. Fig. 15 illustrates the stability of the refractive index of the 200 nm patterned sample shown in Fig. 14. This is done by comparing the refractive indices obtained from the first (prior to cleaning and patterning) and the eighth runs. Numerically quoting, the values of the refractive indices are 2.0616 and 2.0594 at 400 nm wavelength, and 2.0038 and 2.0014 at 632.8 nm respectively for the first and the eighth runs. Similar results are obtained for the other two patterned wafers also.

5. UNCERTAINTY AND TRACEABILITY

5.1 Uncertainty

The calibration of the Film Thickness Standards is done ellipsometrically and like any othermetrological measurement, it is subject to various errors which can distort the results in a more or less critical way. Despite the complicated nature of ellipsometric expressions the uncertainties in the model parameters such as thickness and refractive index can be calculated[23]. It has been pointed out earlier that the parameters measured by the spectroscopic ellipsometer aretanΨ and cosΔ as a function of wavelength, and the thickness and index of the film are obtained by using theoretical models. The absolute accuracy of the measurements is limited by the model used to calculate thickness and refractive index. For the present analysis, ideal models have been used, i.e., smooth surface, discrete interfaces and perfectly isotropic homogeneous layers! This choice adds to the uncertainty in characterizing the film structure. Additional contributions to the uncertainty come from systematic errors that include errors not known and which cannot be reduced by statistical methods, such as equipment imperfection and alignment, optical components, mechanical tolerances, setup and alignment, stray light, parasitic beams, noise and residual polarization of the light source, polarization dependent detector sensitivity and alignment procedures. Uncertainty is computed using the procedure described in the ISO Guide to the Expression of Uncertainty in Measurement[24], and satisfies the requirements of NIST guide to uncertainty[25]. Expanded uncertainty at 95% confidence level is provided.

5.2 Traceability

Most quality management systems mandate that the measurement be traceable to a national or international authority. Most often traceability is achieved through the use of standards. A key concept is the"Unbroken chain of traceability" in instrument calibration, utilization of common and reproducible measurement methods and data analysis. When standards from a national or international authority are unavailable then one has to look to other methods of traceability. There are no NIST SRM's or other international standards available for the silicon nitride film on silicon substrate. The traceability of this standard (see figure 16) is based upon physical first-principles analysis according to the tenets of intrinsic standards set forward in ANSI/NCSL Z540-1-1994[26] and ISO 10012-1[27]. Calibration of the equipment is achieved by measuring the

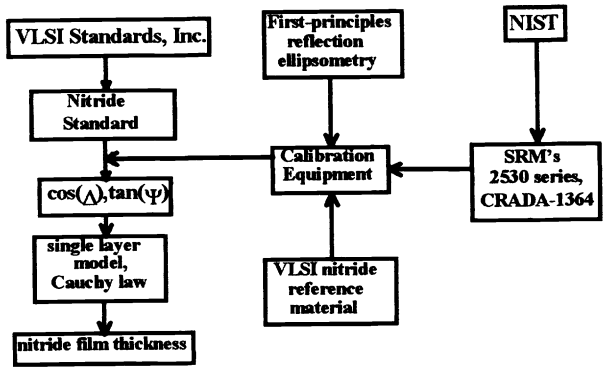

Figure 16: NFTS Traceability Path

NIST SRM's 2531, 2532, 2533, 2534, 2535 and 2536 on the instrument. The thickness value is calculated using a regression technique. The derived thickness values of the NIST SRM's are found to be consistent with those stated on the NIST certificates. Aside from NIST SRM's (the only SRM's for film thickness available from NIST are for silicon dioxide) a set of in-house Nitride Film Thickness Reference Material (one for each thickness value, 9 nm, 20 nm, 90 nm, 120 nm and 200 nm) is maintained. After calibrating the ellipsometer using silicon dioxide SRM's from NIST, the intrinsic nitride film thickness reference materials are measured.

6. RESULTS AND DISCUSSIONS

A set of calibration standards of LPCVD silicon nitride film on silicon wafers have been developed to supplement the currently existing silicon dioxide reference materials. The nominal thickness values are 20.0 nm, 90.0 nm, 120.0 nm and 200.0 nm. This product can be used for the calibration of ellipsometers or other film thickness measurement equipment (e.g. reflectometer). The derived thickness of the nitride film is certified and traceable. Expanded uncertainty at 95% confidence level is calculated in accordance with the ISO"Guide to the Expression of Uncertainty in Measurement"[24]. Aside from the thickness of the nitride film Cauchy coefficients are provided to compute the refractive index of the nitride film as a function of wavelength. This is done for the three thick nitride films. For the 20 nm film a fixed spectrum for the refractive index[22] of silicon nitride is used. The accuracy and precision of the test samples was found to be extremely good. Several studies were conducted to examine the stability and reproducibility of the nitride film after cleaning. Results indicate that a sulfuric acid megasonic clean will etch the nitride film while an isopropyl alcohol clean followed by a deionized water rinse can be used over and over again.

With ellipsometry the desired parameters of interest, thickness and refractive index are not directly measured. They are derived from measured data by using theoretical models. The factors affecting data analysis include, using the correct model of the material structure, using the correct goodness of fit criteria, and examining the extent of correlation of the material parameters extracted from the fit to the data. These considerations lead to limitations in achievable accuracy. Further, several choices for the dielectric function of silicon are available in the literature. There is no consistent data base established for universal use in thin film metrology. Keeping these factors in mind and also to reduce the final uncertainty an idealized single layer model was used for data analysis.

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