Combined use of X-ray reflectometry and spectroscopic ellipsometry for characterization of thin film optical properties

Jason P. Cain[*a], Stephen Robie[b], Qiaolin Zhang[a], Bhanwar Singh[a], Iraj Emami[a]
[a]Advanced Micro Devices, Sunnyvale, CA, USA 94088
[b]Spansion, Sunnyvale, CA, USA 94088

ABSTRACT

Accurate characterization of the optical properties of thin film materials used in semiconductor manufacturing is essential for many metrology applications in the fab, including film thickness measurement and scatterometry. The most common method for measuring these optical properties is spectroscopic ellipsometry. In this work X-ray reflectometry is used as a means of independently determining the thickness of a film to be characterized. This information is then used in the conventional analysis of spectroscopic ellipsometry data to extract the optical properties. In addition, the use of a Cauchy dispersion model fitted to the transparent region of the spectrum (if it exists) was used to determine the film thickness. Once the thickness was determined, a point-by-point regression was performed on the ellipsometry data to extract the optical properties. The results from these techniques were compared with each other and with conventional analysis of the ellipsometry data using common dispersion models.

Keywords: X-ray reflectometry, spectroscopic ellipsometry, thin film optical properties

1. INTRODUCTION

Optical metrology for film thickness measurement has been a fixture in the semiconductor industry for decades, and more recently scatterometry[1] has emerged as a valuable tool for critical dimension (CD) and feature shape metrology. In order to achieve high accuracy using these methods, accurate characterization of the optical properties of the thin film layers being measured is critical. . In addition, process modeling applications (such as design of anti-reflective coatings using lithography simulation software) which also depend on accurate thin film optical property measurements are taking on an increasingly important role in process development and manufacturing. The key optical properties are index of refraction (n), and coefficient of extinction (k). Both the index of refraction and the extinction coefficient are functions of the wavelength of light interacting with the thin film, and therefore these properties may need to be characterized over a relatively wide band of the electromagnetic spectrum ranging from the deep ultraviolet to the near-infrared.

The most common method for measuring thin film optical properties has been spectroscopic ellipsometry[2,3]. These instruments have been available in various forms for nearly a century, and have provided excellent results. However, as the dimensions in semiconductor devices have continued to decrease as manufacturers struggled to keep up with Moore's Law, the thicknesses of some important thin film layers have decreased to levels which create difficulties in measuring the optical properties using ellipsometry alone. The primary difficulty is in finding unique solutions for both film thickness and index of refraction for very thin films (e.g., less than 100Å). In order to get around this problem, we propose the use of two data sources for optical property characterization: X-ray reflectometry (XRR) and spectroscopic ellipsometry (SE).

X-ray reflectometry[4,5] measures the reflected intensity of X-rays striking a surface at glancing incidence as a function of incident angle. This technique yields high-precision measurements of the thickness and density of thin films on the sample surface. In addition, it offers the advantage that this measurement does not depend upon the n and k values of the thin film. This method may therefore be used to measure the thickness of a thin film independently of knowledge of n and k.

In this work, we describe a method for characterizing the optical properties of thin films by measuring the film thickness using XRR and then performing a point-by-point regression to the SE data in order to determine the n & k. This method is compared with two other techniques: one technique involves fitting a simple dispersion model to a

[*] email: jason.cain@amd.com; phone: 1 408 749 2609

Data Analysis and Modeling for Process Control III, edited by Iraj Emami, Kenneth W. Tobin, Jr.,
Proc. of SPIE Vol. 6155, 61550P, (2006) · 0277-786X/06/$15 · doi: 10.1117/12.660088

restricted portion of the spectrum to determine the thickness followed by point-to-point regression, and the other is conventional analysis of the SE data alone. Section 2 describes the measurement procedure, while Section 3 presents the data collected. The optical property extraction methodology is outlined in Section 4, and results of the analysis are given in Section 5. Finally, conclusions are presented in Section 6.

2. MEASUREMENT PROCEDURE

2.1 Spectroscopic ellipsometry data collection

The spectroscopic ellipsometry (SE) data was collected using a J. A. Woollam vacuum-ultraviolet variable angle spectroscopic ellipsometer (VUV-VASE). The data was collected over a wavelength range of 145-1550 nm (photon energy of 0.8 to 8.5 eV). In addition, the measurements were made across the full spectrum for three different angles of incidence: near the Brewster angle at a chosen wavelength, and at ±5° away from this angle. The Brewster angle was measured at a wavelength of 248 nm (5 eV), as this value falls in the DUV part of the spectrum where optical property characterization is often difficult. The 'auto-retarder' option was employed combined with dynamic averaging for optimum precision in the SE measurements.

2.2 X-ray reflectometry data collection

After the SE data collection, the film thickness of each layer was measured using X-ray reflectometry (XRR). A PANanlytical X'Pert PRO MRD X-ray reflectometer was used for data collection. The range of incident angles scanned was 0-3.5° (measured from the sample surface). Modeling of the data was then performed to determine the density and thickness of films on the wafer.

3. DATA

Three sample wafers were used in this experiment. The first wafer is a silicon-rich nitride (SiN) with a nominal thickness of 100 nm deposited directly on a silicon substrate. The SE data for this wafer is shown in Figure 1, while the XRR data is shown in Figure 2. The second wafer is a titanium nitride (TiN) with a nominal thickness of 30 nm deposited on a silicon dioxide (SiO_2) layer with a nominal thickness of 100 nm on a silicon substrate. The SE data for this wafer is shown in Figure 3, and the XRR data is shown in Figure 4. The final sample is a silicon oxynitride (SiON) with a nominal thickness of 10 nm deposited on a silicon substrate. The SE data for the SiON sample is shown in Figure 5, and the XRR data is shown in Figure 6.

4. OPTICAL PROPERTY CHARACTERIZATION METHODOLOGY

Conventional analysis of ellipsometry data involves fitting a model to describe the dependence of the index of refraction (*n*) and the coefficient of extinction (*k*) on wavelength (a dispersion model) and tuning the model parameters and film thicknesses until the simulated ellipsometry response matches the measured data. The film thickness and the *n* & *k* values which minimize the error between the simulated and experimental ellipsometry data are taken to be the true properties of the film. Only one film at a time may be characterized, as it is otherwise difficult or impossible to find stable models which can separate two uncharacterized films in the same stack.

4.1 Cauchy dispersion model

Several different dispersion models exist. One of the simplest models is the Cauchy expansion[2]:

$$n(\lambda) = B_0 + \sum_j \frac{B_j}{\lambda^{2j}} \tag{1}$$

where B_j are fitting parameters and λ is the wavelength of illumination. Typically the series is truncated to two or three terms. The Cauchy model is most often used to describe thin films that are transparent. In these cases, the absorption term may be neglected in the model (*i.e.*, *k* is taken to be zero).

4.2 Lorentz oscillator dispersion model

A second common dispersion model is the Lorentz oscillator model[2], which assumes that the interaction between light and a solid material generates time-dependent internal polarization of the material by oscillating the position of individual electrons with respect to the stable nuclei. Therefore, the equation that models this interaction is given by the classical expression for a damped oscillator (where the driving force is proportional to the local electric field of the illuminating beam). It is most often expressed in terms of the dielectric function ε, which is related to n and k according to the following relation:

$$\varepsilon = \varepsilon_1 - i\varepsilon_2 = \left(n^2 - k^2\right) + 2ink \ . \tag{2}$$

The dielectric function for the Lorentz oscillator model is given by

$$\varepsilon(\omega) = 1 + \frac{4\pi e^2}{m} \sum_j \frac{N_j}{\left(\omega_{0j}^2 - \omega^2\right) - i\Gamma_j \omega} \ , \tag{3}$$

where ω_{0j} is the natural frequency of the jth oscillator, ω is the driving field frequency ($\omega = c/2\pi\lambda$, where c is the speed of light and λ is the wavelength), Γ_j is the resonance width, and N_j is the number of electrons per unit volume associated with the jth resonance. The values of e and m represent the electronic charge and mass, respectively. Typically the number of oscillators is on the order of 1-5. The Lorentz oscillator model is only valid for photon energies well below the bandgap of the material. However, unlike the Cauchy model, it can still be valid in regions of the spectrum where there is significant absorption.

4.3 Tauc-Lorentz dispersion model

If the spectral region of interest falls close to or above the bandgap of an amorphous material, the Lorentz oscillator model will produce poor results. In such cases the Tauc-Lorentz dispersion model may provide useful results[2,6]. Under the Tauc-Lorentz model, the imaginary part of the dielectric function (for a single transition) is specified by

$$\varepsilon_2(E) = 2n(E)k(E) = \frac{A\left(E - E_g\right)^2}{\left(E^2 - E_0^2\right)^2 + \Gamma^2} \frac{\Theta\left(E - E_g\right)}{E} \ , \tag{4}$$

where $\Theta(E-E_g)$ is the Heaviside function [$\Theta(E) = 1$ for $E \geq 0$, $\Theta(E) = 0$ for $E < 0$], E is the photon energy of the illuminating beam, E_g is the bandgap of the amorphous material, E_0 is the energy of the Lorentz peak, A is a magnitude parameter, and Γ is a broadening parameter. The real part of the dielectric function may then be obtained by Kramers-Kronig integration[2,7,8]:

$$\varepsilon_1(E) = \varepsilon_1(\infty) + \frac{2}{\pi} P \int_{E_g}^{\infty} \frac{\xi \varepsilon_2(\xi)}{\xi^2 - E^2} d\xi \ , \tag{5}$$

where $\varepsilon_1(\infty)$ is a fitting parameter and P is the Cauchy principle value of the integral.

In this work an attempt was first made to characterize the optical properties of the different samples using either a Lorentz oscillator model (in the case of TiN) or a Tauc-Lorentz model (in the case of SiN and SiON). This was done without using the film thickness iinformation from the XRR measurements, and is intended to represent the typical process for characterizing thin film optical properties. Once a good model fit was obtained, the film thickness was fixed at the value obtained from XRR measurements and the model was refitted for comparison.

4.4 Procedure for extracting optical properties

In addition to the conventional ellipsometry data analysis techniques, an automated approach was used which attempted to locate a transparent region of the spectrum and fit a Cauchy model over this region to determine the film thickness. The thickness was then fixed at this value and a point-by-point regression was run to extract the optical properties. The

point-by-point regression started at the longest wavelength and adjusted the *n* and *k* of the film being characterized until the results from simulations using these values matched the experimental data. The software then moves to the next-shortest wavelength and repeats the process until the *n* and *k* have been determined for all wavelengths. Although this technique is not novel, it represents an accessible initial attempt at automating the optical property extraction process. However, it is not without its problems. If the thin film being characterized does not have a transparent region within the range of the ellipsometer, the Cauchy model will not give a realistic fit and therefore the thickness value obtained will be unreliable. In addition, the point-by-point regression method does not ensure that the solution obtained will be consistent with the Kramers-Kronig relations, meaning that the result may be non-physical. Further development is required to find ways of dealing with these issues.

Finally, the XRR data was used to specify the film thickness and the point-by-point regression was run using this value instead of the value from the Cauchy fit. A flowchart for the automated approach is shown in Figure 7.

5. RESULTS

5.1 Silicon-rich nitride (SiN)

The results of the optical property extraction for the silicon-rich nitride (SiN) are shown in Figure 8. This includes a Tauc-Lorentz model fit without the film thickness value from XRR, a Tauc-Lorentz fit with the film thickness fixed at the XRR value, a point-by-point regression with the film thickness determined by a Cauchy fit to wavelengths above 600 nm, and a point-by-point regression with the film thickness determined by XRR. The film thickness values and the root-mean-square error (RMSE) between simulated ellipsometry data (using the extracted *n* and *k* values) and the experimental ellipsometry data are summarized in Table 1 below. In general all four techniques performed very well. The fully automated method of using a Cauchy model in the transparent region to determine the film thickness and feeding this information into the point-by-point regression algorithm is a good candidate for this material because it offers results of a comparable quality to the other methods without the need for any user interaction.

Table 1. Summary of optical property extraction from silicon-rich nitride (SiN) film.

	Tauc-Lorentz	**Tauc-Lorentz with XRR**	**Pt.-by-Pt. with Cauchy**	**Pt.-by-Pt. with XRR**
Film thickness	98.92 nm	98.33	98.82	98.33
RMSE to ellipsometry data	1.747	4.668	0.146	3.810

5.2 Titanium nitride (TiN)

The results of the optical property extraction for the titanium nitride (TiN) are shown in Figure 9. This includes a Lorentz oscillator model fit without the film thickness value from XRR, a Lorentz oscillator model fit with the film thickness fixed at the XRR value, and a point-by-point regression with the film thickness determined by XRR. The Cauchy model is not applicable for this material as there is no transparent region in the spectrum covered by the ellipsometer. The film thickness values and the root-mean-square error (RMSE) between simulated ellipsometry data (using the extracted *n* and *k* values) and the experimental ellipsometry data are summarized in Table 2 below. In this case the point-by-point regression algorithm appears to get off-track in the ultraviolet region of the spectrum between about 250-350 nm. The Lorentz oscillator model with the film thickness fixed at the XRR value appears to be the best choice in this case judging by the RMSE.

Table 2. Summary of optical property extraction from titanium nitride (TiN) film.

	Lorentz oscillator	**Lorentz oscillator with XRR**	**Pt.-by-Pt. with XRR**
Film thickness	29.83 nm	28.44	28.44
RMSE to ellipsometry data	7.187	1.8723	7.491

5.3 Silicon onynitride (SiON)

The results of the optical property extraction for the silicon-rich nitride (SiN) are shown in Figure 10. This includes a Tauc-Lorentz model fit without the film thickness value from XRR, a Tauc-Lorentz fit with the film thickness fixed at the XRR value, a point-by-point regression with the film thickness determined by a Cauchy fit to wavelengths above 600 nm, and a point-by-point regression with the film thickness determined by XRR. The film thickness values and the root-mean-square error (RMSE) between simulated ellipsometry data (using the extracted *n* and *k* values) and the experimental ellipsometry data are summarized in Table 3 below. In this case the different techniques disagree significantly in both the film thickness and the *n* and *k*. It is likely that the reason for this is the very small thickness of the SiON film (only 9.18 nm according to XRR). Based on the RMSE values, it appears that the Tauc-Lorentz model (without the film thickness fixed at the XRR value) is the best. However, given that the film thickness from this model disagrees significantly from the XRR value, use of this model seems questionable. A definitive answer to this question is still being sought.

Table 3. Summary of optical property extraction from silicon oxynitride (SiON) film.

	Tauc-Lorentz	Tauc-Lorentz with XRR	Pt.-by-Pt. with Cauchy	Pt.-by-Pt. with XRR
Film thickness	15.834	9.18	15.891	9.18
RMSE to ellipsometry data	2.333	15.332	6.60	30.272

6. CONCLUSIONS

Spectroscopic ellipsometry (SE) and X-ray reflectometry (XRR) data was collected for three different samples: a silicon-rich nitride (SiN) with a nominal thickness of 100 nm, a titanium nitride (TiN) with a nominal thickness of 30 nm on a silicon dioxide (SiO_2) with a nominal thickness of 100 nm, and a silicon oxynitride (SiON) with a nominal thickness of 10 nm. The data was modeled to extract the optical properties (*n* and *k*) using conventional techniques involving Lorentz oscillator and Tauc-Lorentz models. In addition, a point-by-point regression algorithm was used to extract the *n* and *k* with the film thickness determined by either the XRR measurements or by a preliminary Cauchy dispersion model fit to the transparent region of the spectrum (if applicable). In cases where a transparent region is available (the SiN and SiON), the point-by-point regression with the Cauchy-determined thickness performs very well. This is encouraging because this process is completely automated. However, in the case of very thin films (~10 nm or less), the thickness is difficult to determine using only ellipsometry data. For materials in which there is no transparent region in the spectrum covered by common ellipsometry tools, the model fit may be improved by using XRR to determine the film thickness, and point-by-point regression with the film thickness fixed at this value may be a viable approach. A great deal of further development is required in order to refine the automated optical property extraction process.

ACKNOWLEDGEMENTS

The authors wish to thank Harish Bolla of Spansion for his help in acquiring sample wafers for use in this work.

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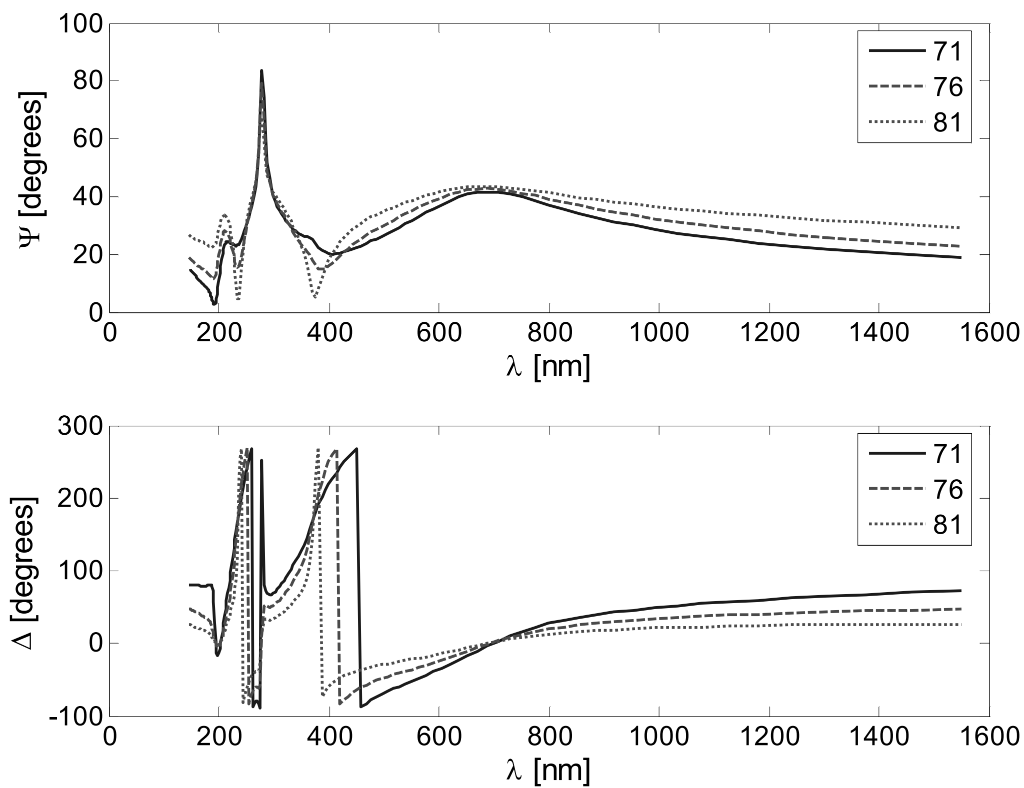


Figure 1. Spectroscopic ellipsometry data collected from silicon-rich nitride (SiN) sample at angles of incidence of 71°, 76°, and 81°.

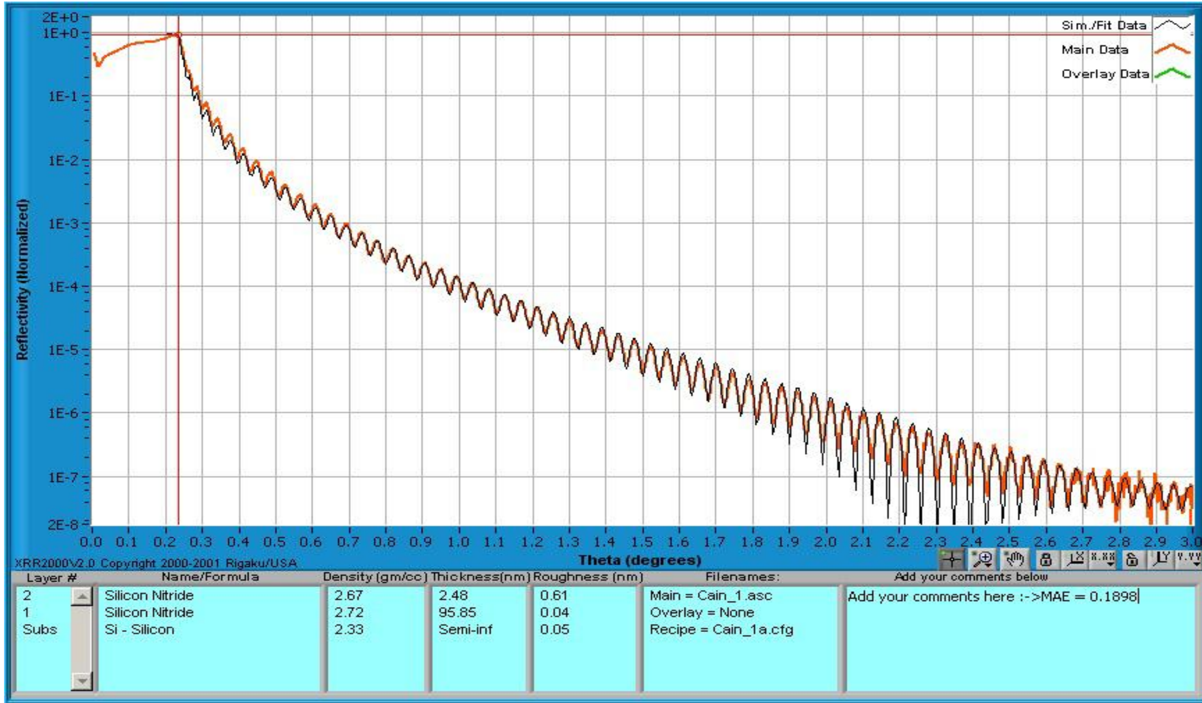


Figure 2. X-ray reflectometry data collected for silicon-rich nitride (SiN) sample with a nominal thickness of 100 nm. Measured thickness is 98.33 nm.

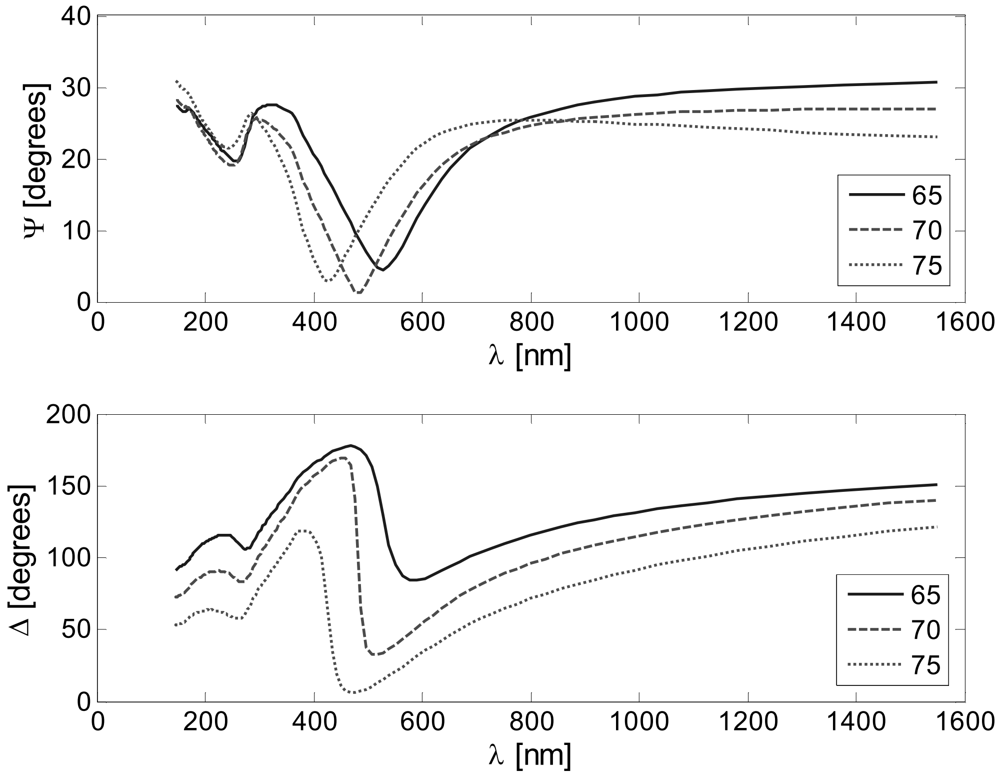


Figure 3. Spectroscopic ellipsometry data collected from titanium nitride (TiN) sample at angles of incidence of 65°, 70°, and 75°.

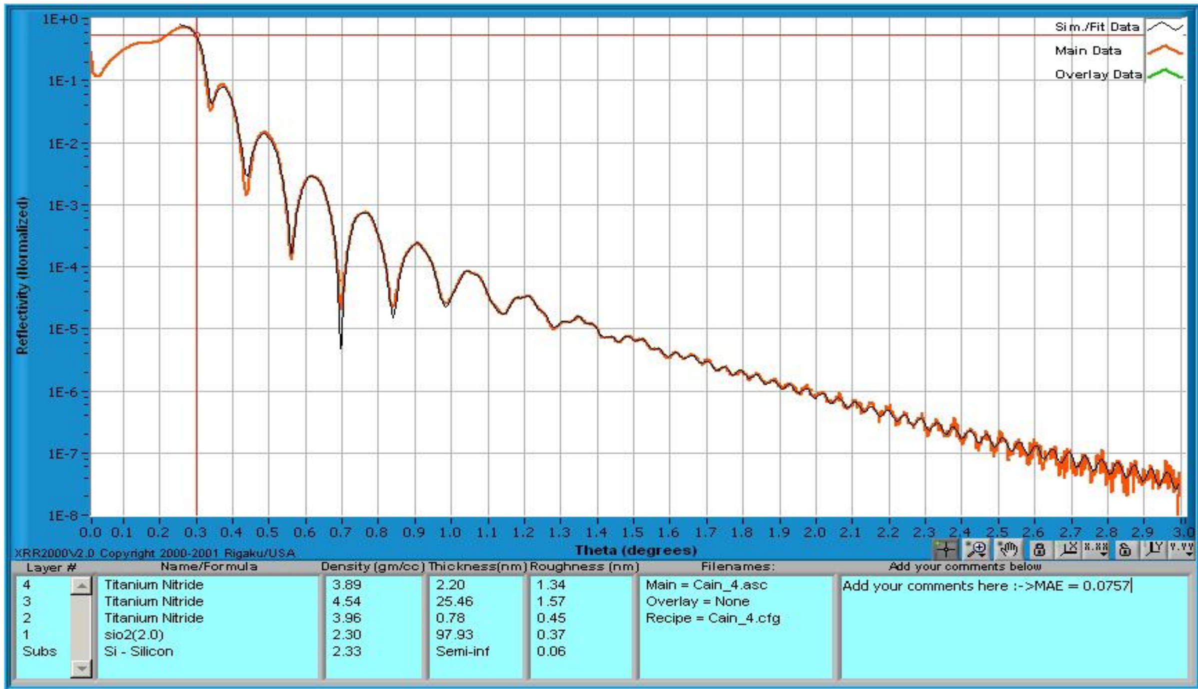


Figure 4. X-ray reflectometry data collected for titanium nitride (TiN) sample with a nominal thickness of 30 nm on oxide layer with a nominal thickness of 100 nm. Measured TiN thickness is 28.44 nm, while measured oxide thickness is 97.93 nm.

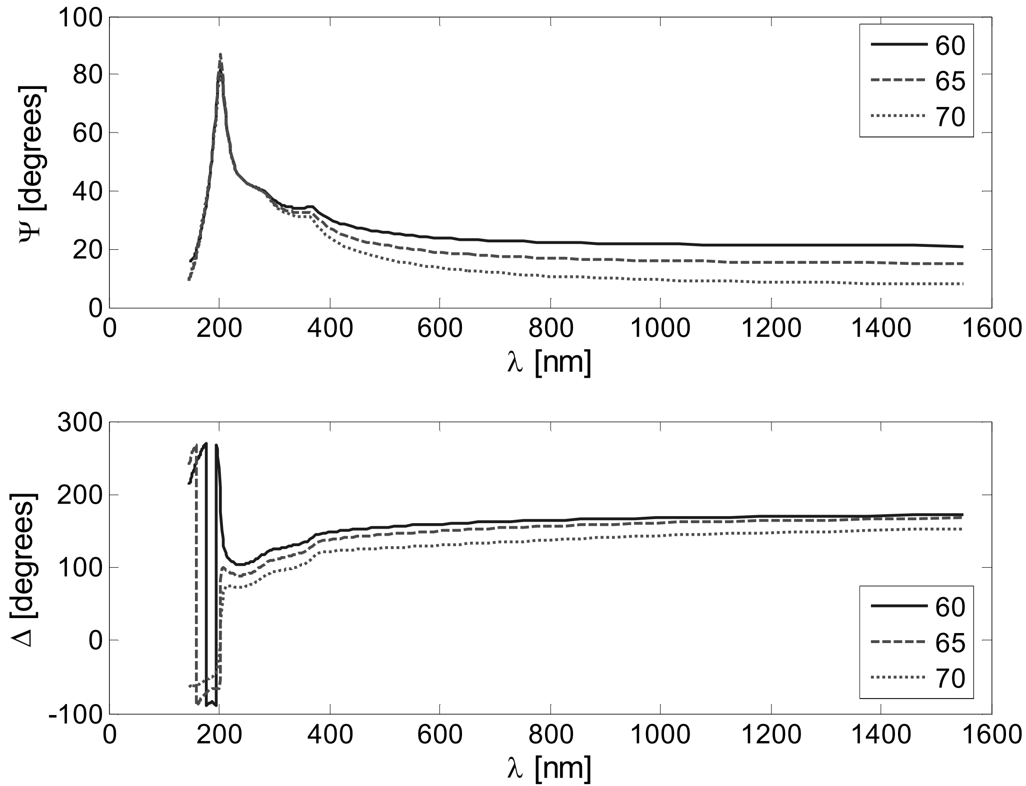


Figure 5. Spectroscopic ellipsometry data collected from silicon oxynitride (SiON) sample at angles of incidence of 60°, 65°, and 70°.

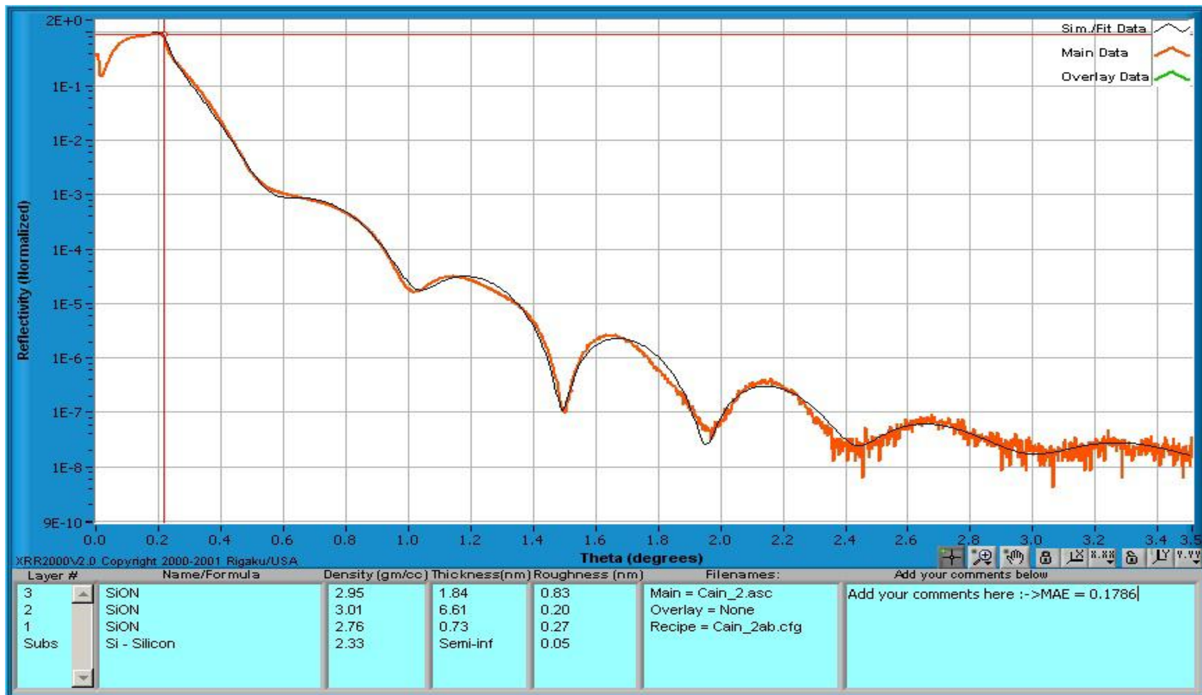


Figure 6. X-ray reflectometry data collected for silicon oxynitride (SiON) sample with a nominal thickness of 10 nm. Measured thickness is 9.18 nm.

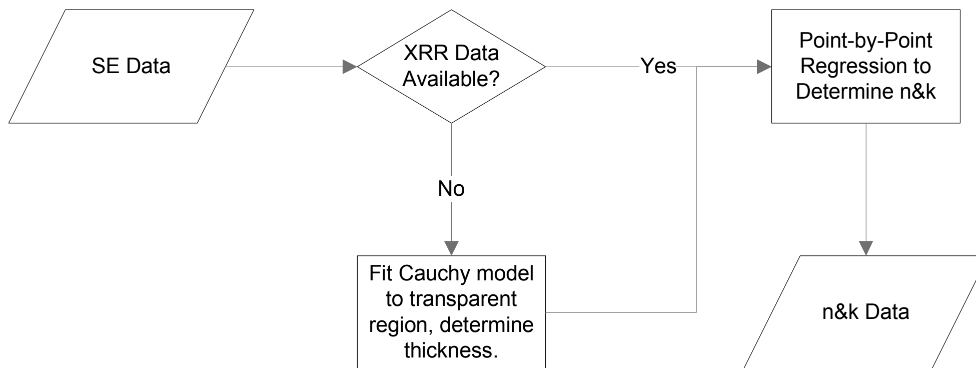


Figure 7. Flowchart showing procedure for extracting optical properties (*n* & *k*) from spectroscopic ellipsometry and X-ray reflectometry (if available) data.

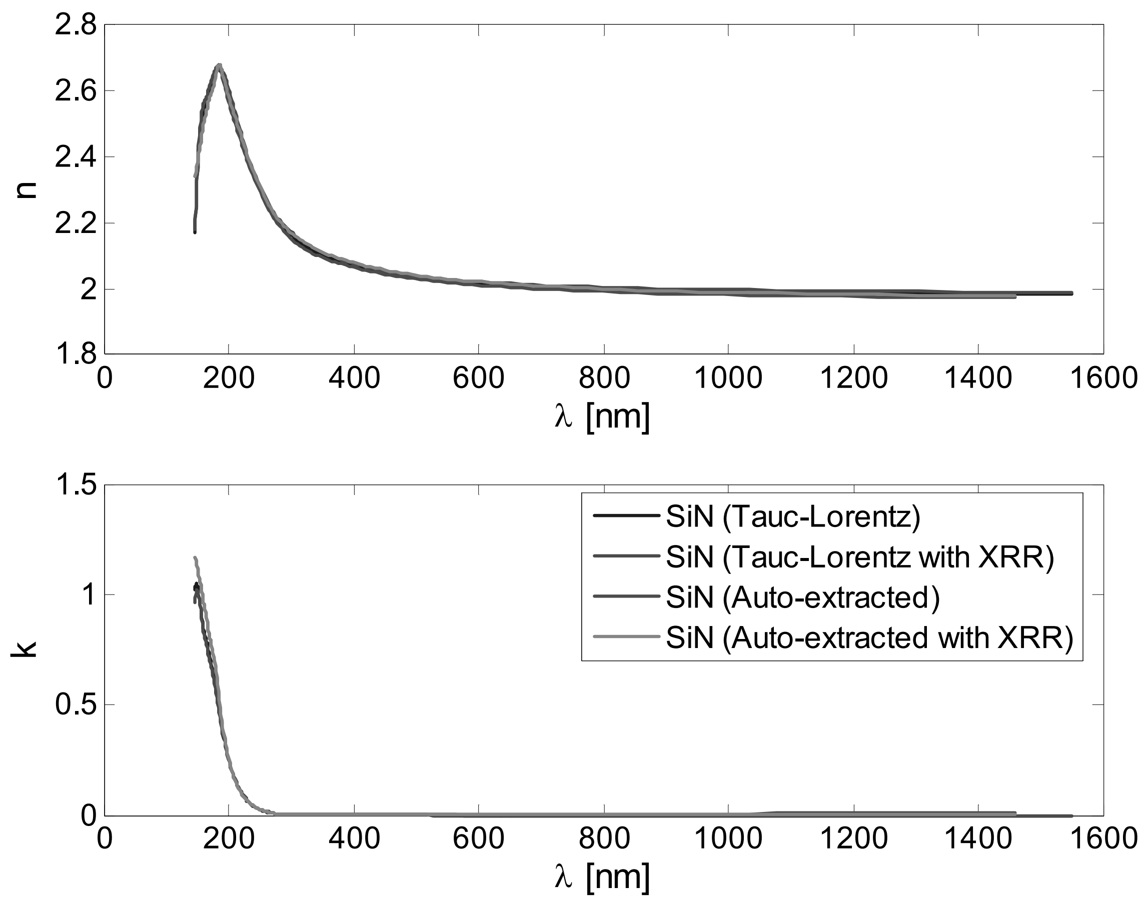
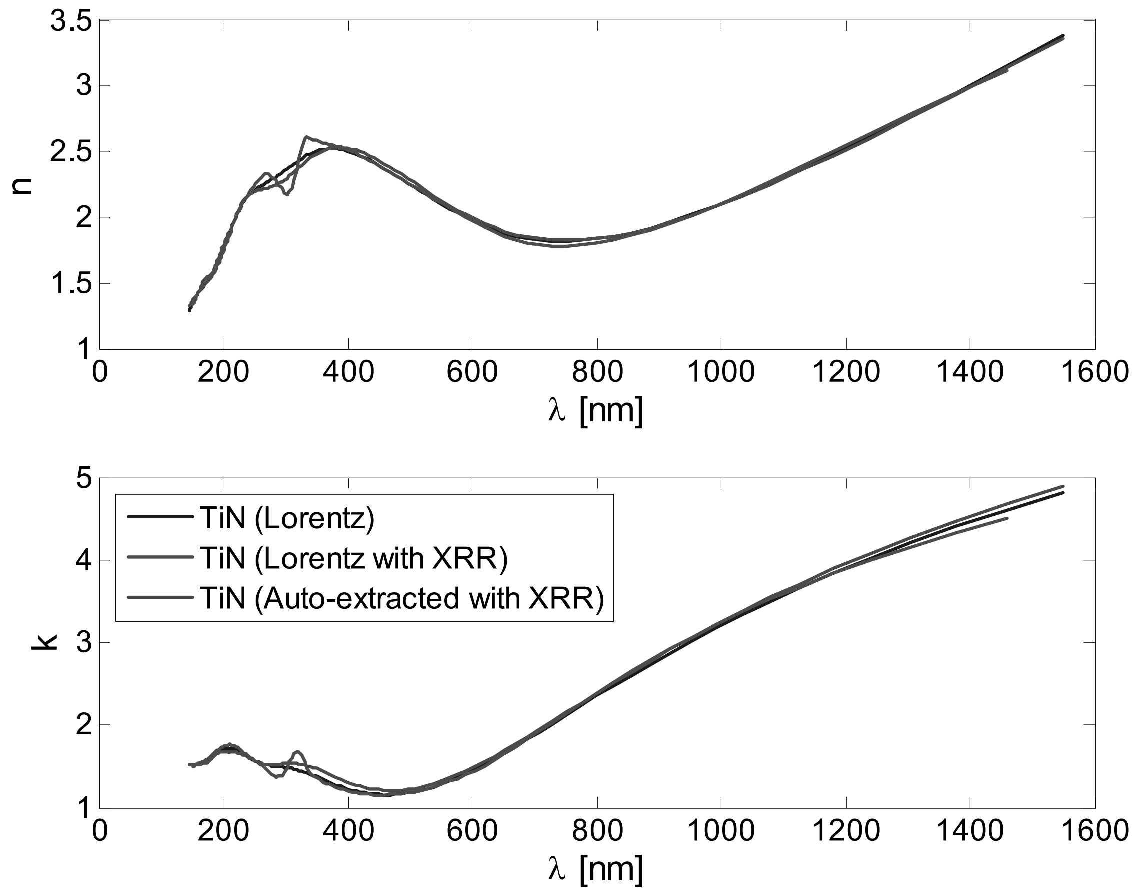


Figure 8. Extracted optical properties for silicon-rich nitride (SiN) using four different techniques.



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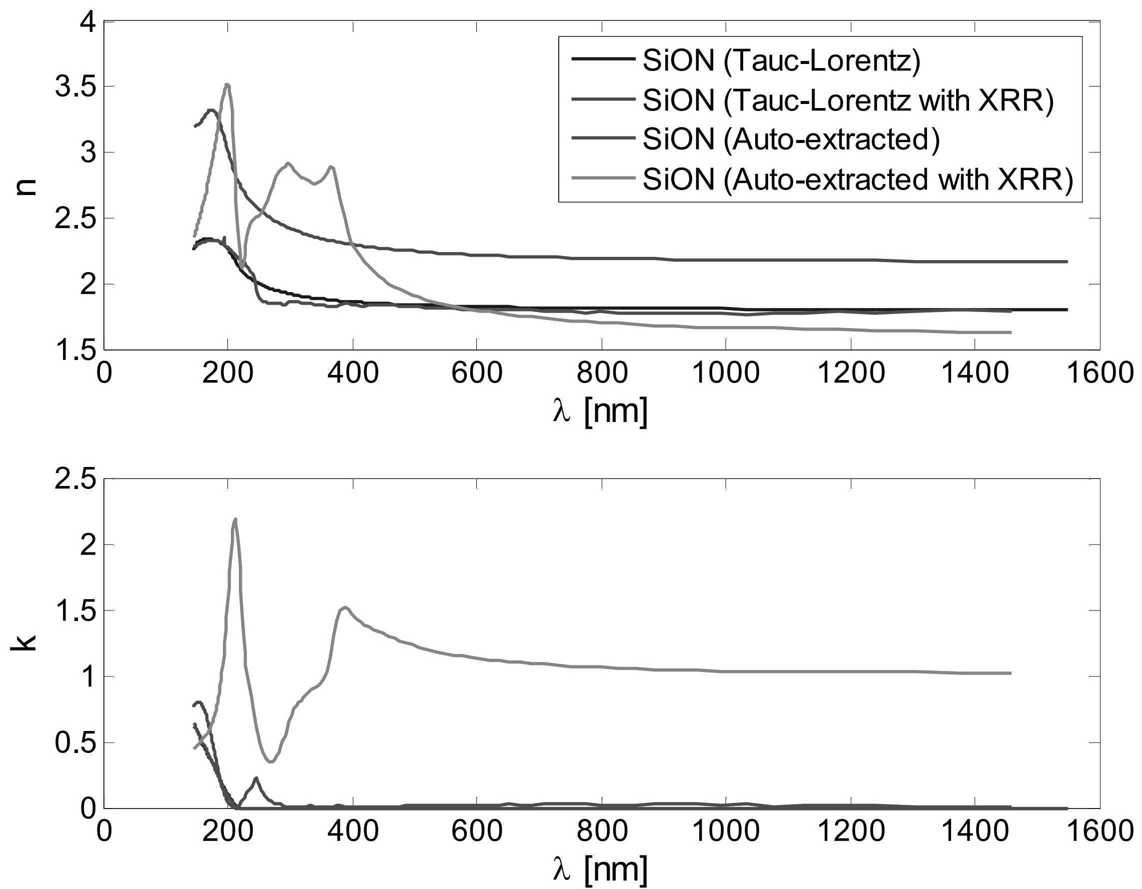

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