J. Webster (ed.), *Wiley Encyclopedia of Electrical and Electronics Engineering*

ELLIPSOMETRY, VARIABLE ANGLE SPECTROSCOPIC

Variable angle spectroscopic ellipsometry (*VASE*) is an optical technique used to characterize film thickness, optical constants, and microstructural material properties (1 2 3–4). Samples of interest consist of bulk materials, thin films, and multilayers grown or deposited on flat substrates. Multilayered solids, liquids, liquids adjacent to solids, and gaseous plasmas in contact with solids can all be characterized with this technique. *VASE* is an attractive method because it is noncontact, is nondestructive, and does not require vacuum.

Many material properties can be characterized with *VASE*. Thin film thickness measurements are common. Optical constants for both thin films and bulk materials can be determined. Other material properties can be investigated with *VASE*, including surface and interfacial roughness, degree of crystallinity, alloy and dopant concentration, optical anisotropy, and other properties that affect the material optical constants.

VASE is commonly used wherever thin films are found. Applications include research and development of optical and protective coatings, polymer and lithography materials, flat panel displays, read–write computer head and media, and semiconductor integrated circuit manufacturing (3 4 5 6 7 8 9–10). Other applications occur in biology (11), medicine, chemistry, electrochemistry (12, 13) and basic materials research.

Early studies focused on the physics of polarized light and its interaction with materials, as well as the optics of instrumentation (5 6 7–8). However, personal computing advances have helped introduce many innovations in ellipsometry. Hardware automation and sophisticated software have taken advantage of increased computing speeds. Sophisticated analysis software allows for new approaches to problem solving and extremely fast solutions to new material analysis problems. Technological advances have permitted many new applications. Consequently, ellipsometry is now used extensively in research, development, and manufacturing.

In the early years, ellipsometry was performed at one or a few individual wavelengths, typically using a laser or a filtered strong emission line from an arc lamp. Most ellipsometry is now performed with light of many wavelengths (often hundreds, in a near-continuum) over a broad spectral range. *Spectroscopic* ellipsometry offers many advantages over single-wavelength ellipsometers: multilayer capabilities, depth profiling of properties, and solution of otherwise unsolvable materials problems. Light wavelengths ranging from 142 nm in the ultraviolet to 55 μm in the infrared are common, and the available spectral range is continually being extended. Many applications require measurement of optical properties at a desired wavelength. Spectral range may also optimize sensitivity to the material property of interest. For example, dopant concentrations will have a strong effect on the infrared optical properties of a material. Film thickness measurements require a transparent spectral range, where light can travel through the layer. Films as thin as a single layer of atoms on a surface can be measured. Furthermore, the long wavelengths used in infrared ellipsometry allow thickness measurement of films more than 50 μm thick.

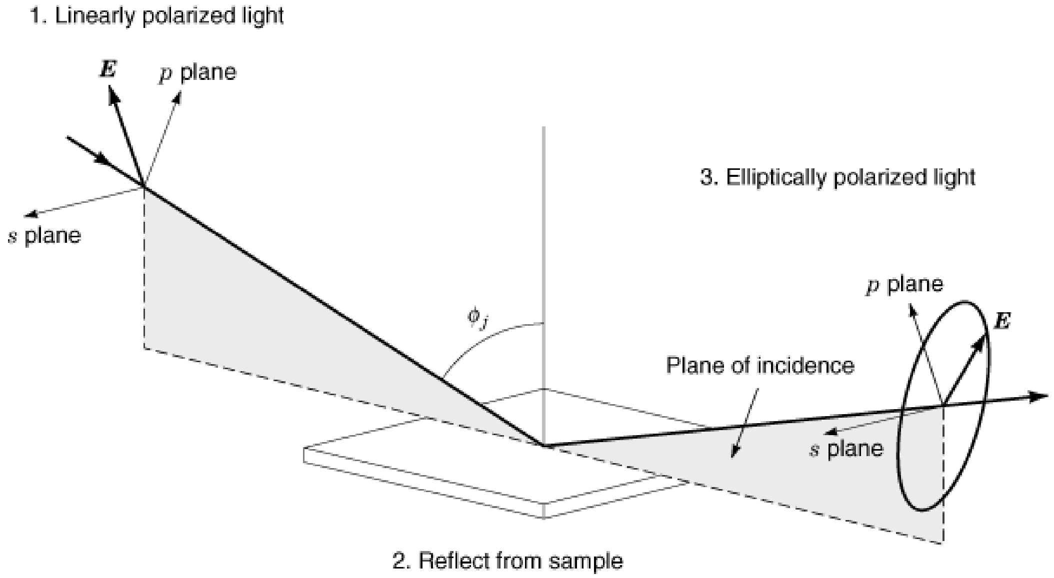


Fig. 1. Physical geometry of ellipsometry measurement.

BASIC CONCEPTS: HOW DO ELLIPSOMETERS WORK?

Figure 1 shows the essential optical physics of ellipsometry (1 2 3–4). A polarized light beam is reflected from a material of interest. The reflected light is analyzed for its polarization state. The measured change in polarization (both amplitude and phase) can be used to deduce properties of the material.

The electric field of the incident light beam (linearly polarized light) can be decomposed into vector components in two planes, as shown in Fig. 1. The p plane contains the incoming and outgoing beams, and the s -plane is perpendicular to this plane. The electric field of the reflected (or transmitted) beam is likewise decomposed into p -plane and s -plane components. The reflected (or transmitted) light is typically elliptically polarized, whence the name “ellipsometry.” Detailed descriptions of polarized light are well documented elsewhere (14). Mathematically, the resulting change in polarization is expressed as ρ, a complex number (having both real and imaginary parts):

$$\rho = \tan\psi e^{j\Delta} = \frac{r_p}{r_s} \qquad (1)$$

where ψ and Δ are the amplitude and phase components, respectively. The complex *Fresnel reflection coefficients* are represented as r_p and r_s for the p -plane and s -plane components of the light wave. The mathematical expressions for r_p and r_s are derived from Maxwell’s equations for electromagnetic radiation applied to the boundary between dissimilar materials:

$$r_s = \frac{N_0\cos\phi_0 - N_1\cos\phi_1}{N_0\cos\phi_0 + N_1\cos\phi_1} \qquad (2a)$$

$$r_p = \frac{N_1 \cos\phi_0 - N_0 \cos\phi_1}{N_1 \cos\phi_0 + N_0 \cos\phi_1} \qquad (2b)$$

where ϕ_0 is the incident angle and ϕ_1 is the refracted angle. The incident angle is that between the incoming light beam and the surface normal under study, as shown in Fig. 1. Typically, oblique angles ranging from 55° to 80° are used for ellipsometry, as they provide the best sensitivity to material properties. They complex index of refraction for each media can be expressed as

$$N = n + jk \qquad (3)$$

where n is usually called the *index of refraction* and k the *extinction coefficient*. These two quantities are used to describe how light interacts with the material. They are referred to as *optical constants*, although in reality they do, in fact, vary with wavelength, temperature, and so on. When the medium surrounding the sample under study is air or vacuum, $_0$ is equal to 1.000. Details of the mathematical derivation of the Fresnel equations are too lengthy to present here; however, they are well known and well-documented (14).

Ellipsometers typically measure ρ [often expressed as $\psi$and Δ from Eq. (1), or a related quantity] as a function of wavelength and angle of incidence. After a measurement is made, the data need to be analyzed to determine optical constants, layer thickness, and other properties of interest. The analysis consists of multiple steps, as illustrated in Fig. 2.

A model is constructed to describe the measured sample. It consists of planar layers for each material, including the substrate. A thickness and a set of optical constants (n and k) describe each layer over the measured spectral range. An initial guess is made for any unknown properties. The simplest model is for a bulk homogeneous solid with no surface roughness or oxide. For this case, the complex index of refraction is directly obtained:

$$N_1 = N_0 \tan\phi_0 \left(1 - \frac{4\rho}{(1+\rho)^2} \sin^2\phi_0\right) \qquad (4)$$

However, most materials have a rough or oxidized surface, which invalidates Eq. (4).

In the next step of Fig. 2, the model is used to generate expected values of ρ for the same conditions (angle, wavelengths) as the measured data. The equations resulting from a theoretical description of the model are algebraically very complicated for even a single film on a bulk substrate. Consequently, they cannot in general be inverted to give an expression similar to Eq. (4) for optical constants or layer thickness in terms of the measured quantities (ψ Δ, and angle of incidence ϕ_0). Such problems, known generically as *the inverse problem*, occur frequently and include analysis from optical data such as transmission, reflection, or ellipsometry. Another example is in coding theory for communications. Although the solution to the inverse problem in mathematics is outside the scope of this article, much has been written about this large topic.

The most common solution to the inverse problem in ellipsometry uses the Levenberg–Marquardt algorithm in regression analysis (15). The experimental data are compared with the generated values using a comparator function (16), often the mean squared error (*MSE*), defined by

$$\mathrm{MSE} = \frac{1}{2N-M} \sum_{i=1}^{N} \left[\left(\frac{\psi_i^{\mathrm{mod}} - \psi_i^{\mathrm{exp}}}{\sigma_{\psi,i}^{\mathrm{exp}}} \right)^2 + \left(\frac{\Delta_i^{\mathrm{mod}} - \Delta_i^{\mathrm{exp}}}{\sigma_i^{\mathrm{exp}}} \right)^2 \right] = \frac{1}{2N-M} \chi^2 \qquad (5)$$

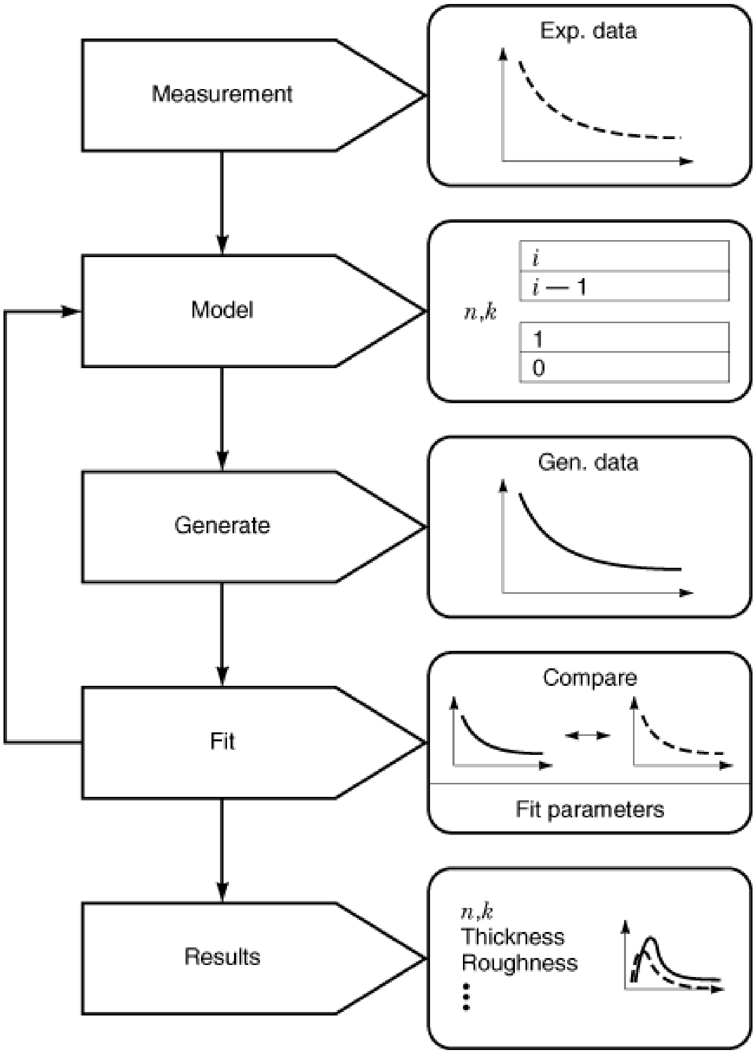


Fig. 2. Flow chart of *VASE* data analysis procedure.

The subscript i identifies terms for each unique wavelength and angle of incidence, σ is the standard deviation, N is the total number of (ψ, Δ) pairs, M is the number of *fit parameters*, and exp and mod signify experimental and calculated values. Another common estimator, chi-square (χ^2), is also defined in Eq. (5).

The computer calculates the MSE and then minimizes its value by adjusting each of the fit parameters. The latter consist of any unknown properties in the model. The process of finding the lowest MSE will depend on the complexity of the optical model. When finished, the results must be consistent with properties of the actual materials involved. For example, a negative thickness cannot be a correct result. The model must also be complex enough to describe all structure found in the experimental data. The fit results will indicate whether the model must be modified, as represented in Fig. 2.

There are subtle cases where the lowest MSE may generate unphysical or nonunique results. However, regression analysis has been applied successfully to hundreds of ellipsometry examples, resulting in trustworthy,

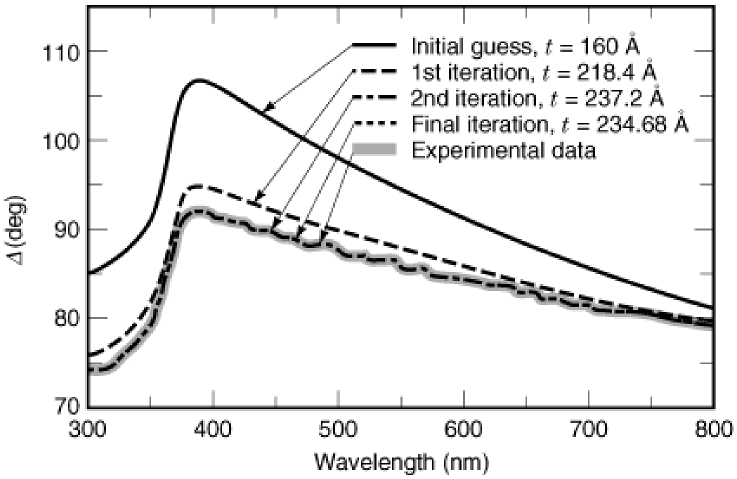


Fig. 3. Iterative fit to experimental data by varying the thickness.

physical, accurate, and precise results. Figure 3 shows an iterative fit to the thickness of a thermally grown oxide layer on crystalline silicon. The initial guess was 16 nm, and the final value of 23.47 nm closely matches the experimental data. Several other common examples of ellipsometric data analysis are presented later in this article.

INSTRUMENTATION

Different hardware configurations are utilized for spectroscopic ellipsometry measurements. Each must be capable of producing a light beam of known polarization and measuring the polarization state after the light has been reflected from or transmitted through the material of interest. This will allow the instrument to quantify the change in polarization, ρ, due to the sample. Some instruments measure ρ by rotating a polarizer used to define the incoming polarization, with a second fixed-position polarizer (called an analyzer) intercepting the outgoing beam. Other instruments use a fixed polarizer on the input and rotate a polarizer (analyzer) on the output. Still others use fixed polarizers on both input and output, combined with an intermediate means to modulate the polarization state (at high frequency) using, for example, acousto-optic crystals. Other configurations have also been used (1, 17). The final result for any configuration is a measure of the complex ρ as a function of wavelength (equivalently, as a function of photon energy) and often as a function of angle of incidence.

Spectral range and speed of measurement are frequently important considerations when choosing a spectroscopic ellipsometer. An extensive variety of spectral range options is available—from 142 nm in the ultraviolet to 55 μm in the infrared. The choice of spectral range is generally determined by the application, as different spectral ranges can provide different information regarding materials. Suitable instrumentation must be found to match the desired spectral range.

The measurement speed is often dominated by the choice of spectrometer (used to separate wavelengths). Monochromators are used to select a single, narrow band of wavelengths. By moving the internal optics in the monochromator (typically computer-automated), the monochromator can move through the wavelengths of interest. This method is precise, but sometimes slow, as one wavelength is measured at a time. When a monochromator is used before the sample, it has the added advantage of significantly reducing the amount of light reaching the sample (avoiding changes in photosensitive materials). Another approach is to measure the entire spectral range simultaneously and divide the light beam into its spectral components by dispersing it over a detector array. This method is commonly used when fast measurements are required. Fourier transform spectrometers can also acquire the entire spectral range simultaneously, but utilize a single detector rather than an array. This method is most commonly applied to the infrared spectral range.

APPLICATIONS

VASE can characterize many different material properties, as will be demonstrated in this section. It can be used for metals, semiconductors, dielectrics, polymers, and multilayers of these materials. Because of the diverse capabilities of *VASE*, it has been adopted for many industrial and research applications.

The semiconductor industry utilizes many different thin films and multilayers to fabricate microelectronic devices. Ellipsometry measurements include gate oxide thickness, photoresist optical constants, alloy ratios in compound semiconductors, crystallinity in polysilicon layers, and complex grading profiles of a photomask.

Ellipsometry has also been applied to the many different coatings used for data storage applications, including disk media [CD-R, DVD, and magnetic and magneto-optic disks], magnetic tape, and magnetoresistive (*MR*) or giant magnetoresistive (*GMR*) read head structures. Flat panel displays may contain liquid crystal layers, color filters, transparent conductor films, and the multilayered transistor structures required for thin film transistor (*TFT*) displays. *VASE* is also used extensively for research. It can play a vital role in the study of new materials.

Spectroscopic ellipsometry has many ex situ applications; however, in situ measurements offer additional advantages. Ellipsometry is fast enough to keep up with many processes to provide real-time monitoring and control. The following sections describe a few of the different applications of ex situ and in situ ellipsometry.

Ex Situ Examples.

Layer Thickness: Single Layers. Single-layer thickness measurements are probably the most common application of ellipsometry. Films as thin as a single layer of atoms on a surface, or even fractional coverage by an atomic layer, can be measured. At the other extreme, data from thick layers are characterized by a large number of interference oscillations. The wavelength range and bandwidth of the instrument determine the maximum measurable thickness. Infrared ellipsometers have measured thicknesses greater than 50 μm . The use of longer wavelengths results in wider interference oscillations that can be easily resolved.

Film thickness measurements utilize the interference between multiple light beams. The light traveling through the film rejoins the light reflected from the film surface, as shown in Fig. 4. The various components of light will have different phases, depending on the additional optical distances they travel. The interference between multiple light beams will depend on the wavelength (due to different phase velocities) and angle of incidence (due to different path lengths). The film thickness is determined by analysis of the resulting interference oscillations.

A transparent spectral range is necessary to determine film thickness. Thickness information is not available unless a portion of light can propagate to the bottom of the film and back to the surface. If these components are absorbed or scattered, then the thickness information is lost. Metal thicknesses are usually not available, except for very thin layers (typically less than 100 nm).

Layer Thickness: Multilayer stack. Multiple layer thicknesses can often be determined in a stack. In that case, it is often beneficial (and sometimes required) to measure at multiple angles of incidence. The different angles change the path length through the films, which may provide new information about the individual layers. Figure 5 shows ψ and Δ data taken at four angles of incidence, and the optical model for a five-layer stack. In this example, five layer thicknesses, plus the optical properties of the buried poly-Si layer, were determined by combining a wide spectral range and multiple angles of incidence.

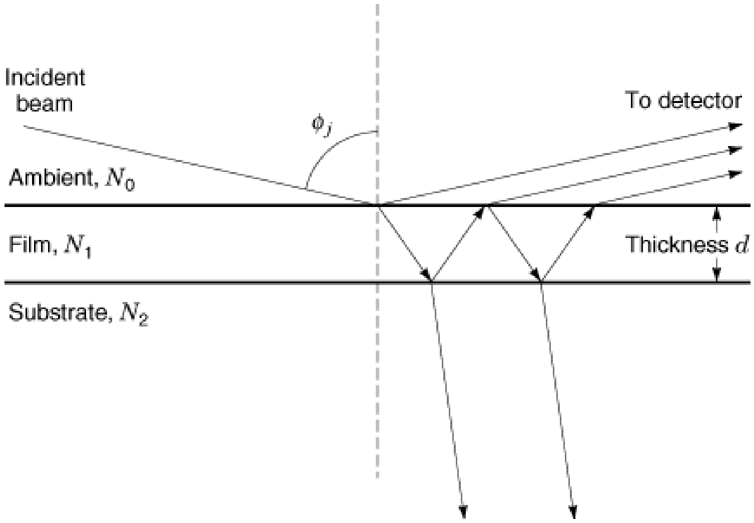


Fig. 4. Reflection of light from a thin film. The multiple reflections that occur within the film result in interference oscillations.

Optical Constants. The optical properties of an isotropic material can be described using two parameters. These parameters characterize how a material will respond to light of a given wavelength. One representation of the optical constants is the complex index of refraction, N, given in Eq. (3). The real index n describes the phase velocity of light propagating through the material compared to propagation in vacuum. The extinction coefficient k describes the exponential decay of light traversing the material and is related to the absorption coefficient α by

$$\alpha = \frac{4\pi k}{\lambda} \tag{6}$$

where λ is the wavelength of the light (1).

Alternatively, the optical constants can be described using the complex dielectric function, given by

$$\tilde{\epsilon} = \epsilon_1 + j\epsilon_2 \tag{7}$$

The complex dielectric function is related to the complex index of refraction of a material by

$$\tilde{\epsilon} = N^2$$

Both optical-constant representations are encountered in the literature, and usage is generally determined by the application.

The optical properties are often dependent on other microstructural properties of the material—degree of crystallinity, alloy concentration, doping concentration, surface roughness, interfacial roughness, and any other property that affects the optical constants. Hence, an understanding of the relationship between other material properties and optical properties can facilitate the measurement of these other material properties.

Commercial ellipsometers are now able to take data from 142 nm through the infrared. An understanding of material optical properties is important for proper interpretation of results (18 19–20). Proper choice of

Layer	Thickness (nm)
SiO_2	12.73
Si_3N_4	10.50
SiO_2	17.66
Poly-Si	262.2
SiO_2	8.84
Si substrate	0.5

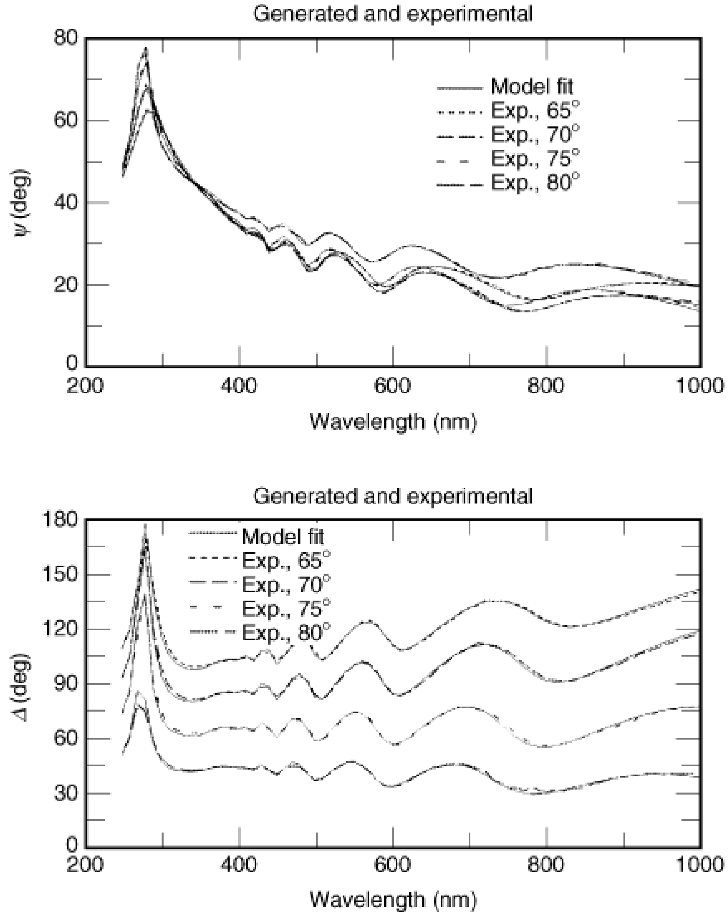


Fig. 5. Model and fit results for a multilayer measurement. A fit was achieved by varying five layer thicknesses and the optical constants of the poly-Si layer.

spectral range is important. Optical constants for two different polymers are shown in Fig. 6. Note that the index of refraction of these layers is similar through most of the visible. Only at very short wavelengths is there good optical contrast between these two polymer films. However, the films are also absorbing in this region. This example requires measurement in both the visible and the ultraviolet. The ultraviolet region will allow

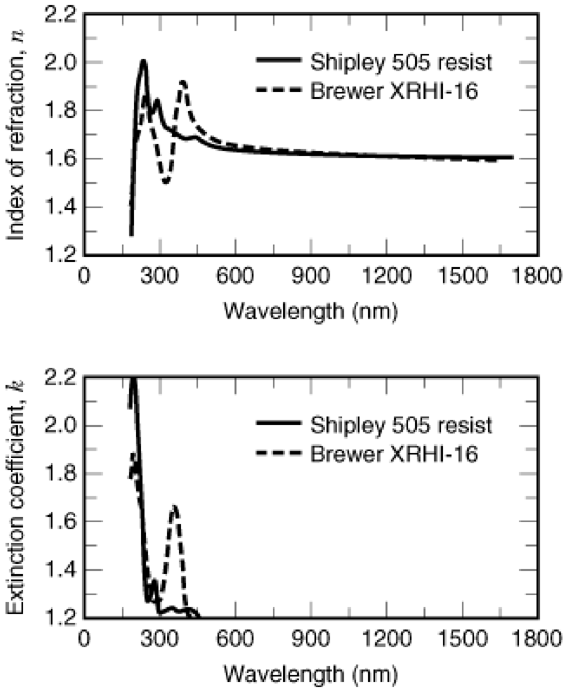


Fig. 6. Optical constants for two polymer films, illustrating that optical contrast is only available in the ultraviolet (*UV*) spectral range. A multilayer of these materials was measured with *VASE*, and both thicknesses could be uniquely determined because of their UV properties.

the two layers to be separated (optical contrast), while the visible will allow the thicknesses of each layer to be determined (little light absorption).

In subsequent sub-subsections, the relationship between optical constants and other material properties is demonstrated. Examples of crystallinity, alloy and doping concentration, surface roughness, and anisotropy are presented.

Degree of Crystallinity. Silicon is a widely used material, and its properties depend strongly on the degree of crystallinity. Crystalline silicon is important in microelectronics. Amorphous silicon is important in solar cells and transistors.

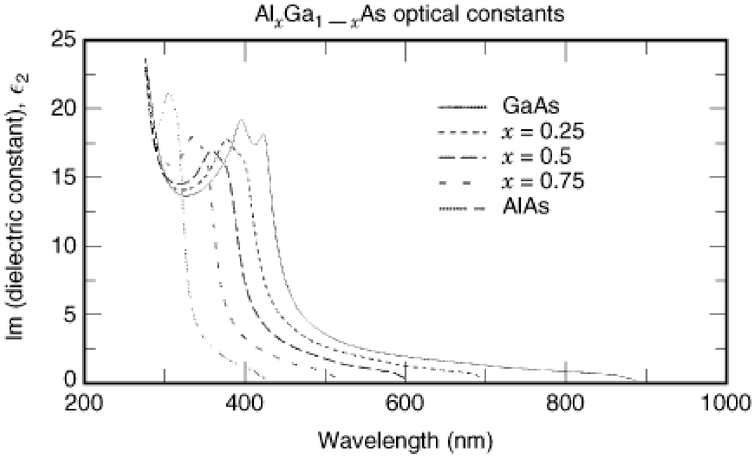
Polycrystalline silicon (poly-Si) is used for circuit conductors. In contrast with amorphous material, its optical properties depend strongly on the crystallite size. Ellipsometric measurements of optical properties are sensitive to the degree of crystallization of poly-Si (21, 22).

Optical constants of the poly-Si layer from Fig. 5 were determined using effective-medium theory for atomic-scale mixing of amorphous and crystalline silicon. Effective-medium theory is beyond the scope of this article; it is used to represent optical constants of multiconstituent materials mixed on an atomic scale (23, 24). The optical constants of each constituent must be known. The mixing is in regions smaller than the measured wavelengths. The advantage of effective-medium theory is determination of spectrally dependent optical properties using few parameters (constituent fractions).

Alloy Fraction. Several years ago Snyder et al. developed an *alloy optical constant shifting* method of analyzing data on alloys such as Al_xGa_{1-x} As to determine the alloy fraction x (25). This method has been widely adopted. In the example below, spectroscopic ellipsometry was used to determine two layer thicknesses and the alloy ratio of each layer, along with the surface oxide thickness. An increase in aluminum

Layer	Thickness (nm)
GaAs oxide	6.03 nm
$Al_xGa_{1-x}As$, $x = 0.213$	1.674 μm
$Al_xGa_{1-x}As$, $x = 0.252$	2.546 μm
GaAs substrate	1 mm

(a)



(b)

Fig. 7. The model (a) is shown for a bilayer of AlGaAs with different alloy concentrations. The alloy concentration provides optical contrast because an increase in the aluminum concentration increases the bandgap of the material. This is illustrated in (b) as a shift in the ε_2 band edge toward shorter wavelengths.

concentration increases the bandgap. This is seen optically as a shift in the absorption edge to shorter wavelengths, allowing ellipsometry to accurately determine the alloy concentration. Figure 7 shows the optical model and the imaginary part of the dielectric function, ε_2 (the real part, ε_1, likewise depends on the alloy fraction) versus alloy ratio for a semiconductor example. In this example, the alloy fractions of two layers were determined simultaneously with three layer thicknesses.

Surface Roughness. Spectroscopic ellipsometry is very sensitive to the surface of thin films and bulk materials. The surface often contains an ultrathin (subnanometer) oxide or roughness layer. Roughness can be modeled using the effective-medium approximation.

An opaque mercury cadmium telluride (*MCT*) film was measured with *VASE* to determine its alloy ratio. A poor fit was obtained from a simple model, assuming the MCT was a bulk. However, addition of a 1.9 nm surface roughness layer, modeled using effective-medium theory, improved the fit by an order of magnitude. The extracted surface roughness value from the ellipsometer correlated well with roughness measurements by other techniques, such as atomic force microscopy (*AFM*). The analysis included simultaneous determination of surface roughness and alloy fraction ($x = 0.222$), as shown in Fig. 8.

Doping Concentration. Dopants in a material will introduce absorption in the infrared spectral range due to free carriers. Spectroscopic ellipsometry measurements at long wavelengths can characterize this optical absorption, thus characterizing the dopant concentration (26). Figure 9 shows the expected Drude free-carrier effects on extinction coefficient *k* for three different doping levels in silicon. Such differences in optical properties

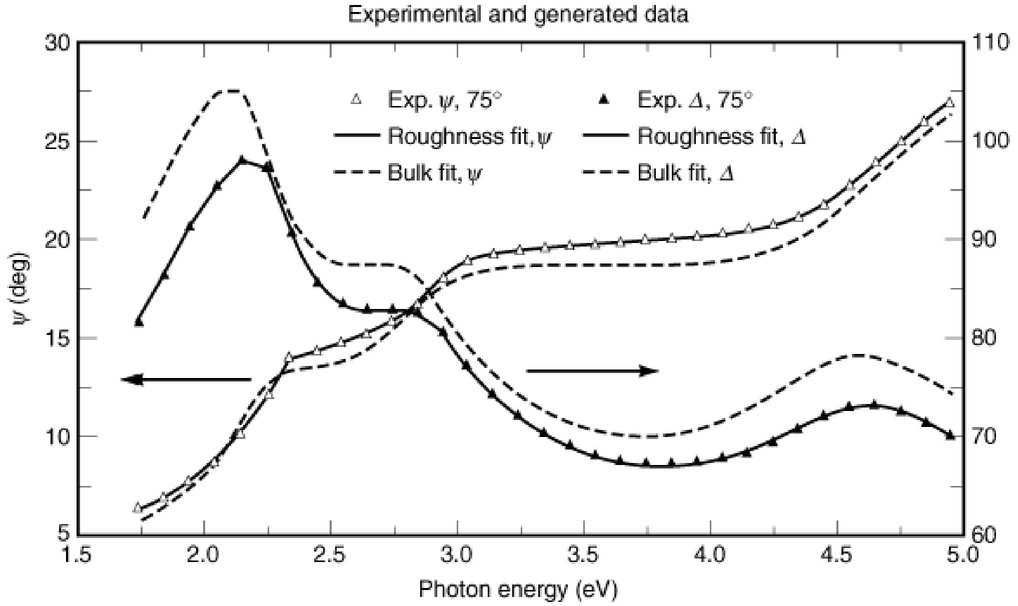


Fig. 8. The experimental data for an opaque MCT film are shown along with the resulting fit curves from two distinct models. The first model, represented by dashed lines, treated the material as a bulk film without surface films. The second model, represented by solid lines, allows for the surface roughness and is in good agreement with the experimental data.

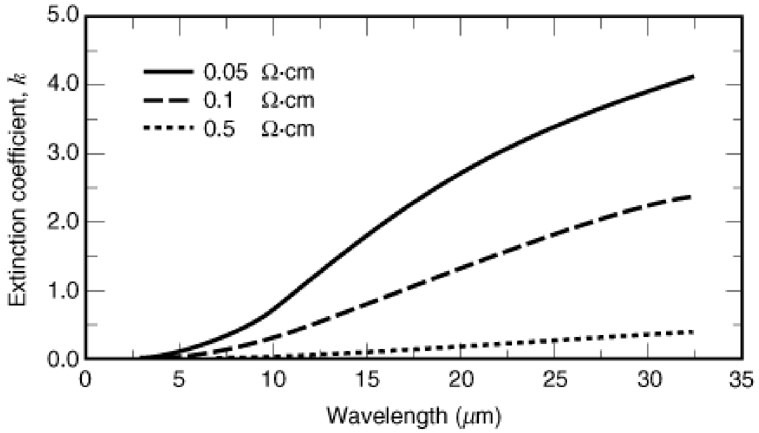


Fig. 9. Extinction coefficient in the infrared for three different silicon doping concentrations, illustrating the Drude absorption edge, that increases as the resistivity decreases (doping concentration increases).

allow use of infrared optical measurements to determine the doping density, doping density profiles, and epitaxial layer thicknesses in semiconductors. Infrared spectroscopic ellipsometry is also useful for quantitative analysis studies of resonant chemical vibrational effects in ultrathin films (27) and phonon effects (28).

Vertical Grading. Many thin film properties change with vertical position throughout the film (along the direction perpendicular to the surface). This is most often induced by processing conditions, whether intentional or unintentional. Indium tin oxide (*ITO*) and TiO_2 are examples where this commonly occurs. In the example of

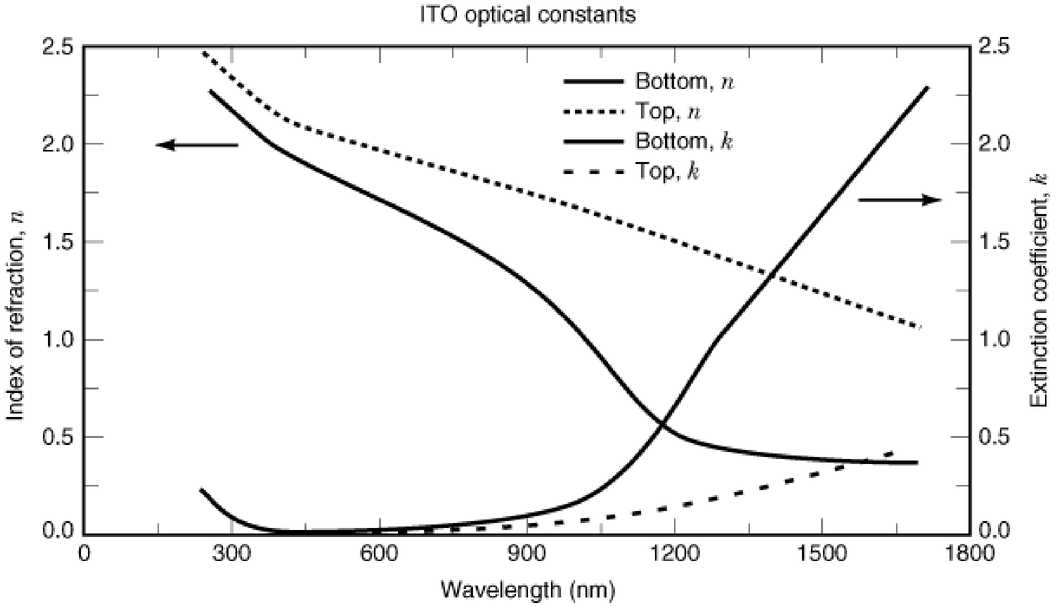


Fig. 10. Optical constants at the top and bottom of a thin ITO film that was modeled with vertical index grading. The variation in optical properties from the substrate to the surface is due to the process conditions.

an ITO film, the refractive index grading is determined along with the layer thickness and a surface roughness thickness. Figure 10 shows the optical constants at the top and bottom extremes of the graded ITO layer.

Optical Anisotropy. Many materials are optically anisotropic; that is, their optical properties vary in different film directions. An example is shown below for a 995.2 nm thick organic film. *VASE* data at multiple angles are very sensitive to optical anisotropy, because different amounts of light interact with the different film directions as the angle changes. The dramatic difference between an isotropic and an anisotropic model fit is illustrated in Fig. 11. Obviously, anisotropy must be included in the model. The resulting optical constants in the plane of the film and perpendicular to the film are shown in Fig. 12. Modeling anisotropic materials has been previously discussed (29, 30), and a detailed mathematical description of “generalized” ellipsometry has been presented by 31.

Uniformity over a Surface. Industrial applications often require tight control over uniformity, which implies tight tolerances on the thin film properties. This is important for optimum performance in microelectronic devices over an entire wafer (currently up to 300 mm) and for uniform display quality on a flat panel. Any parameter can be mapped with *VASE*.

In Situ: How Ellipsometers Monitor or Control Processes. Ellipsometers have been used to monitor processes inside vacuum and other chambers for many years. At first, single-wavelength ellipsometers were used, but lately multiwavelength ellipsometers have provided greatly increased information, and indeed have been able to control processes in a closed-loop feedback mode (32 33–34). Some of this is proprietary to the companies using this technique, but there is extensive literature on the academic progress in this field. A few key references on this important topic are provided for the interested reader. An example chamber that might use an in situ ellipsometer is shown in Fig. 13, with input and output optical beams visible in this cross-section view (35). This particular chamber is used for sputtering, but in situ ellipsometry has been used on molecular-beam-epitaxy and chemical-vapor-deposition crystal growth chambers, as well as on evaporation, ion-beam,

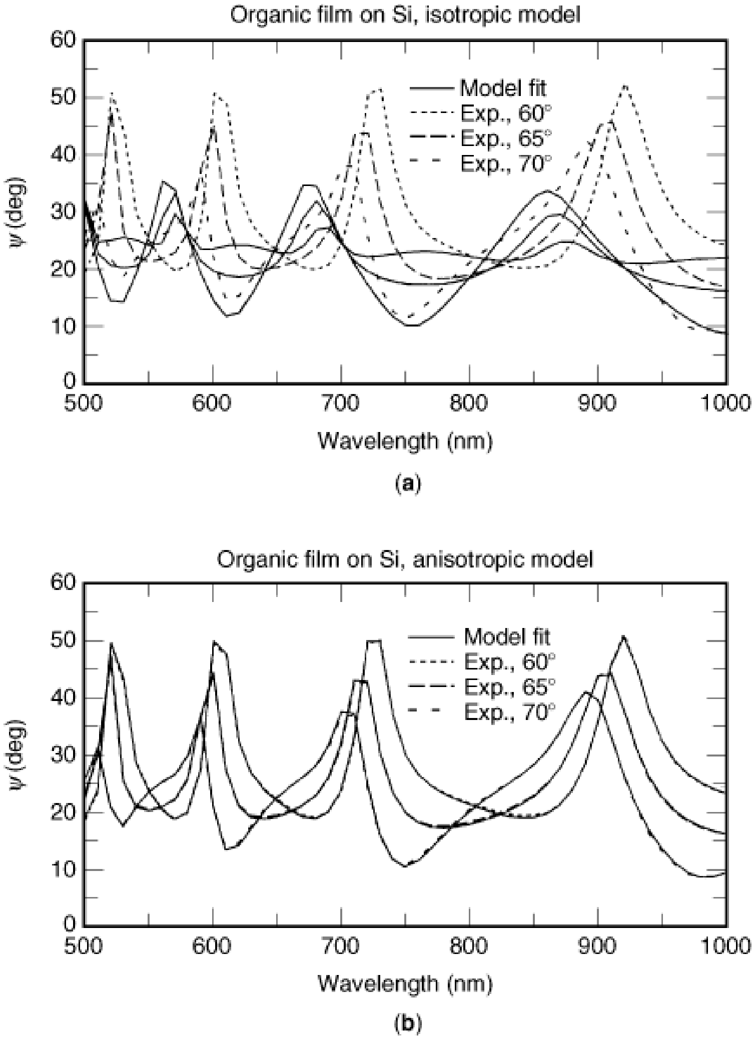


Fig. 11. Data fits for an organic film on silicon. The isotropic model (a) was not in agreement with the experimental data. The anisotropic model (b) was able to fit all the features of the data. This illustrates the importance of having a correct representation of the measured materials and that *VASE* can measure the optical properties of anisotropic films.

and plasma etching chambers. Other examples of in situ applications are in electrochemical reactions and electrodeposition.

FUTURE DIRECTIONS OF SPECTROSCOPIC ELLIPSOMETRY

There are several new areas, in both technology and applications of ellipsometry, that deserve mention. These are being developed both by manufacturers and in universities and national laboratories. We mention here only a few examples.

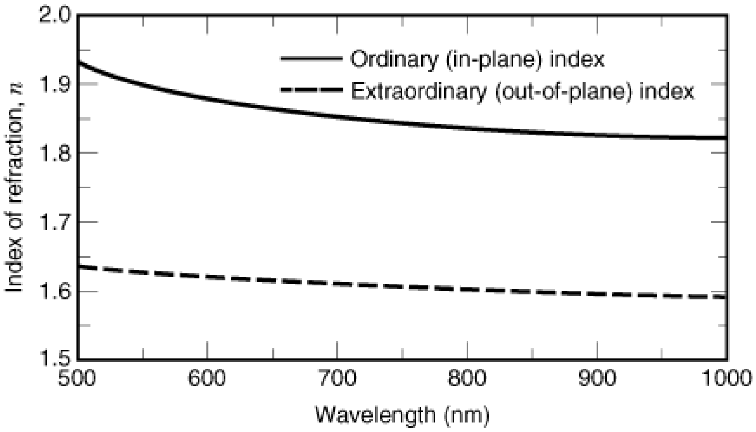


Fig. 12. The resulting index of refraction for the in-plane and out-of-plane directions of the film based on the best fit to the data in Fig. 11.

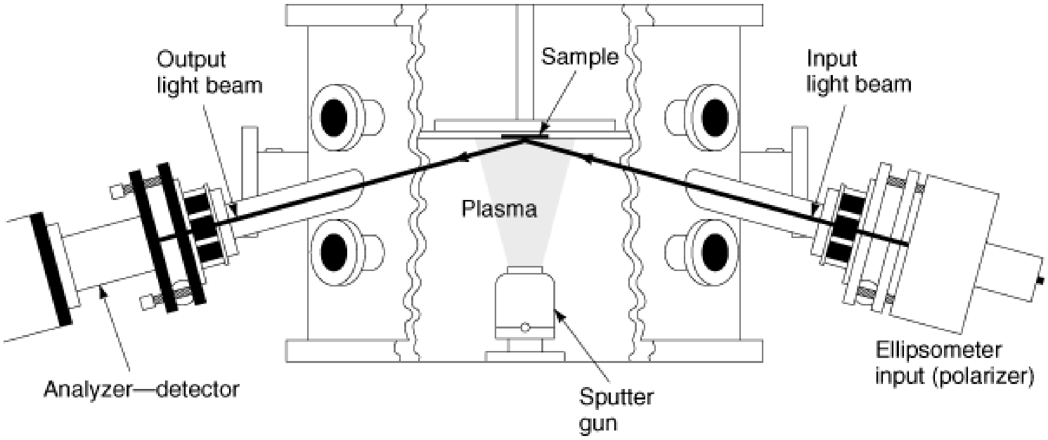


Fig. 13. Geometry of an in situ ellipsometer attached to a vacuum chamber.

Developments of in situ applications for industry are rapidly taking place, and process control is utilized in a few manufacturing examples. Continued progress in this area is expected.

Present commercial ellipsometers cover 142 nm to 35 μm . We believe that extension at both ends of this spectrum will take place in the very near future. Both these developments will open up large new industrial and basic research applications. For example, 157 nm is a laser wavelength for use in ultrafine lithography. For progress in this technology, the optical constants of the optical components (such as lenses) as well as the photoresist and antireflection coatings on them must be repeatedly measured. In the infrared, applications include determining doping density, anisotropy, layer thicknesses of thick layers, and other properties.

Another actively advancing area is in variable-temperature optical spectroscopy. This is currently an area of intense interest to experimental physicists and other basic research scientists. Industrial applications may soon develop in the determination of doping density in semiconductor processing.

Imaging ellipsometers are now commercially available. These use single-wavelength laser sources and image using charge-coupled devices. These instruments have been used to monitor biological motions on a surface as a function of time.

Finally, there has been a small effort to use ellipsometers on patterned materials. This is an application of enormous industrial potential. For example, ellipsometers aid in processing and manufacture of read–write heads for computers and in the manufacture of integrated circuits. However, these patterns are usually created using short-wavelength optical lithography, which results in pattern dimensions only a bit smaller than the light wavelength. Ellipsometer beams using light of comparable wavelength experience diffraction effects that are very difficult to model.

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