

Fixed-polarizer ellipsometry: a simple technique to measure the thickness of very thin films

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Abstract. The fixed-polarizer ellipsometer measures thickness of thin films. It is simple, inexpensive, and provides a linear response over a range of 800 Å. We develop a matrix formulation to describe the optical characteristics of the instrument and apply it to the case of a single thin film on a substrate. Excellent agreement is found between experimental and simulated results. Applying the instrument to optical immunoassay, we show that its sensitivity can extend to 4 pg/ml, depending upon the analyte. This compares favorably with commercially available manual and automated immunoassay systems. The fixed-polarizer ellipsometer appears to be well-suited for use in laboratory and production environments. © 1999 Society of Photo-Optical Instrumentation Engineers. [S0091-3286(99)02205-9]

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1 Introduction

Ellipsometry provides a powerful method to measure the thickness of thin transparent films. The measurement is based on the difference between reflection coefficients and phase delays of light having polarizations parallel versus perpendicular to the plane of incidence. Conventional ellipsometers^{1,2} incorporate two polarizers and a quarter-wave plate, at least one of which is rotating. These instruments can accurately measure film thicknesses in the angstrom range and below.

Another type of ellipsometer, the comparison ellipsometer,³ has no moving parts. It is designed such that light reflects first off a surface coated with a film of unknown thickness and then off a reference film. The reference film consists of a wedge of material having the same optical properties as those of the unknown film, but with varying thickness. A dark extinction line appears where the two thicknesses are the same. This approach provides the ability to view an area rather than a single point, but yields accurate thickness measurements only of films of known optical properties.

We present an even simpler instrument for accurately measuring the thickness of very thin films. The instrument incorporates one or two fixed-position polarizers in addition to the light source and detector. It is simpler and faster to use than a standard ellipsometer, requiring only one intensity measurement to determine the film thickness. Different substrates and films are easily accommodated by changing the polarizer and analyzer angles and the calibration curve. An instrument that has a similar configuration, but is restricted to operating close to the Brewster angle, was applied by Arwin and Lundström to quantify immunological reactions.⁴

In this paper we apply optical theory to describe the operation of the fixed-polarizer ellipsometer. The determination of single- or multi-layer transparent film thickness is

first modeled then compared to experimental results. Examples of the application of the fixed-polarizer ellipsometer to immunological reaction measurement are given and compared to other immunoassay techniques.

2 Optical Theory

2.1 Polarization States and Reflection Coefficients

When light is incident on a flat sample at a non-perpendicular angle, the directions of propagation for the incident and reflected beam together define a plane. This plane of incidence is the plane in which we analyze the problem. Our goal is to analyze the change in polarization state, including amplitude and phase, of the reflected beam to determine the thickness of a thin film on the reflecting substrate. There are two independent linear polarization states of the incident light. The electric field of the light can either be in the plane of incidence (*p*-polarized, also called TM-polarized) or it can be perpendicular to it (*s*-polarized, also called TE-polarized). Any linear state of polarization can be expressed as a combination of these two states, provided they are in phase. If there is a difference between the phases of these two states, the light is described as being elliptically or circularly polarized, because the electric field vector follows an elliptical or circular spiral as the light ray propagates.⁵

Due to differences in the coupling of the two polarizations to the medium of the sample, the amplitude and phase of reflected light is different for light in the two polarization states. The reflection coefficients for the amplitude are given in Eqs. (1a) and (1b) for waves respectively parallel and perpendicular to the plane of incidence.

$$r_{12}^p = \frac{n_2 \cos \theta_1 - n_1 \cos \theta_2}{n_2 \cos \theta_1 + n_1 \cos \theta_2} \quad (1a)$$

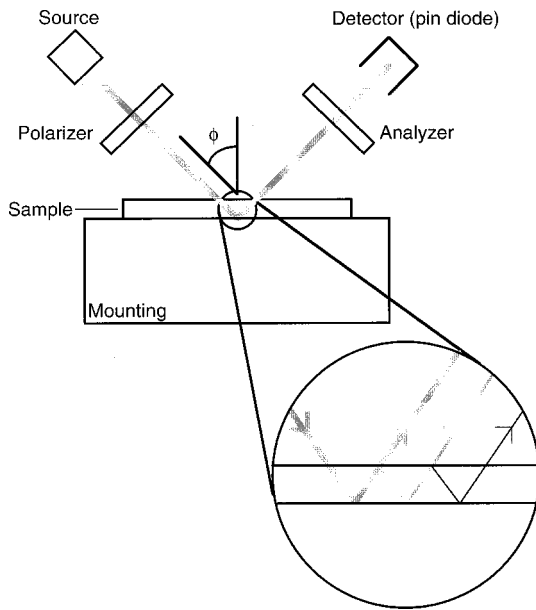


Fig. 1 Fixed-position ellipsometer configuration. The source can be a laser, laser diode, or filtered white light source. The instrument arm angle is ϕ . Inset: multiple reflections for a single film.

$$r_{12}^s = \frac{n_1 \cos \theta_1 - n_2 \cos \theta_2}{n_1 \cos \theta_1 + n_2 \cos \theta_2}. \quad (1b)$$

The indices of refraction of the media above and below the interface are n_1 and n_2 , respectively, and θ_1 and θ_2 are the respective angles between a normal to the interface and the light beam for the incident and transmitted light. These angles are found using Snell's law: $n_1 \sin(\theta_1) = n_2 \sin(\theta_2)$.

2.2 Layered Films

The fixed-polarizer ellipsometer provides a measurement of the thickness of a film on a substrate that has a different refractive index from that of the film. While the formulas given above work for a single reflection, one must also take into account multiple reflections among the interfaces of the layer. This multiple reflection condition is depicted in the inset of Fig. 1.

The multiple reflections from this film are accounted for using a matrix formulation of the reflection equations. For multi-layer films, the matrix formulation models the multiple interfaces, with each layer and each interface between layers corresponding to a matrix. Interfaces which are rough can be modeled using an effective medium approximation.⁶⁻⁸ These matrices are then multiplied together to find the overall reflection coefficient. Each of the two polarization states of the system requires a separate matrix which is used to find the reflection coefficient for that polarization. The matrices and examples of their use can be found in the literature.⁹

In this paper we analyze a single film on an opaque substrate. When dealing with such a system, the matrix technique simplifies to the following equation

$$r_{tot} = \frac{r_{12} + r_{23}e^{-2i\beta}}{1 + r_{12}r_{23}e^{-2i\beta}}, \quad (2a)$$

$$\beta = \frac{2\pi n_2 d \cos \theta_2}{\lambda}, \quad (2b)$$

where medium 1 is the air, 2 is the film, and 3 is the substrate; r_{12} and r_{23} are the amplitude reflection coefficients at each interface; β is the phase delay that results from the film; d is the thickness of the film; and θ_2 is the angle of the light beam in the film with respect to the surface normal. There are two values for r_{tot} , corresponding to the two polarization states and two sets of reflection coefficients.

These reflection coefficients are complex numbers. This results from the wave nature of light and is a shorthand notation for both the magnitude and the phase of the electro-magnetic wave. The instrument measures light intensity, and we are therefore concerned with the reflection coefficient for intensity. The intensity reflection coefficient, also called the reflectivity, is the square of the amplitude reflection coefficient, $|r|^2$.

2.3 Fixed-Polarizer Ellipsometer

The fixed-polarizer ellipsometer uses the variation in reflection coefficient with film thickness [affecting β in Eq. (2b)] and the difference between reflection coefficients for the two polarizations [r_s vs. r_p from Eqs. (1a), (1b), and (2a)] to determine the thickness of a thin film. This is done in a manner similar to conventional ellipsometry with two key differences: (1) the fixed-polarizer ellipsometer uses no quarter-wave plate and thus can be used with monochromatic light sources of any wavelength without changing components; and (2) the polarizers in the fixed-polarizer ellipsometer are fixed, as opposed to the rotating polarizers found in conventional ellipsometers. (For more on conventional ellipsometers, see the excellent book by Tompkins,¹ and the classic work on ellipsometry by Azzam and Bashara.²)

Figure 1 is a schematic of the fixed-polarizer ellipsometer. The input polarizer is referred to as the "polarizer," while the output polarizer is called the "analyzer." Both are linear polarizers which can be rotated about the beam axis. Once adjusted, they remain fixed throughout the measurement. The instrument arm angle is ϕ , which is equal to θ_1 in Eqs. (1a) and (1b).

Light incident on the sample is linearly polarized. After reflection it has, in the general case, some degree of ellipticity. For a bare silicon substrate this ellipticity is very small, and the light is very close to being linearly polarized. The analyzer is oriented such that most of the light is blocked. The remaining light enters the detector and produces a photocurrent which is proportional to the intensity. The photocurrent is usually transformed into a voltage by a transimpedance amplifier.

When a film is first grown on the silicon substrate the ellipticity increases with thickness, the axes of the ellipticity rotate, and the fraction of reflected light passing through the analyzer increases. This increase in output intensity continues until an axis of the ellipse passes the analyzer angle or until the film reaches a thickness of

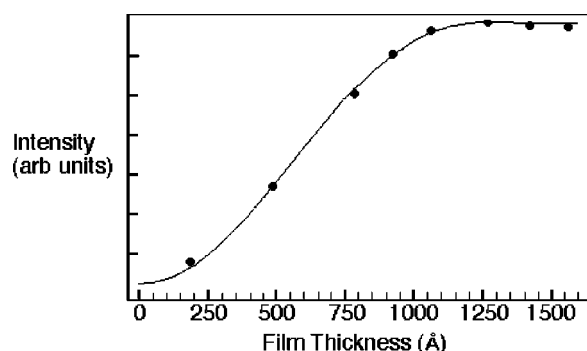


Fig. 2 Comparison of simulated (line) and experimental (points) results for a siloxane film. The wavelength is 670 nm, the refractive index of the film is 1.41, the polarizer angle (θ_p) is 10° , the analyzer angle (θ_a) is 50° , and the instrument arm angle (ϕ) is 65° .

$(\lambda \cos(\theta_2))/(4n_2)$. In the former case, the transmitted intensity falls slightly because less of the elliptically polarized light goes through the polarizer, i.e., the component of polarization along the polarizer is falling as the ellipse axis rotates away. In the latter case, the effect of the film starts to cancel itself and the ellipticity of the light returns towards its original state. By the time the thickness has reached $(\lambda \cos(\theta_2))/(2n_2)$, the film is optically invisible and the sample looks like a clean substrate. Thus the transmitted intensity is periodic with thickness.

We now describe mathematically this transmitted light intensity. The intensity at the detector can be expressed as

$$I_{\text{out}} = I_{\text{in}} |r_{\text{tot},p} \cos(\theta_p) \cos(\theta_a) + r_{\text{tot},s} \sin(\theta_p) \sin(\theta_a)|^2 \quad (3)$$

where I_{in} is the source intensity and θ_p and θ_a are the respective polarizer and analyzer angles with respect to the p -polarization axis.

In a conventional ellipsometer the polarizer and analyzer must be rotated to determine a film thickness. The fixed-polarizer ellipsometer, however, is constrained such that a single intensity measurement with fixed polarizers is sufficient to provide the thickness of a deposited film to a high degree of accuracy. A simulation of the fixed-polarizer ellipsometer was made using the software program Mathematica.¹⁰ The results are shown in Fig. 2. This simulation incorporated values for our particular experimental system, described below. (Numerical values for a given experiment are given in the corresponding figure caption.) The measurement of other common materials, e.g., organic films, silicon nitride, and indium tin oxide, can be measured using this technique. The magnitude and period of the curve change with a change of the instrument angles (ϕ , θ_p and θ_a) and the refractive index of the film.

2.4 Region of Sensitivity

The variation of ellipticity with film thickness is not linear, and hence the output of our instrument exhibits regions of high and of low sensitivity, consistent with the discussion preceding Eq. (3). The measurement is most accurate where the variation of intensity with film thickness is the greatest. This corresponds to the region where the slope is highest in Fig. 2. From the figure, we can see that the measurement is most accurate in the range of 150 to 950 Å. While this

range of accuracy works well for our current immunoassay technology, future work in this or other areas may require accuracy in a different range.

Changing this region of sensitivity can be accomplished, but this requires complicating the system. The lack of sensitivity outside of this thickness region arises due to the elliptical polarization of the reflected light, which cannot be readily blocked by a linear polarizer. This ellipticity reduces the contrast between different thickness levels and thus reduces the sensitivity of the reading. If a quarter-wave plate is added to remove this ellipticity, however, the region of sensitivity can be moved to any thickness of interest. Addition of a quarter wave plate changes the physical setup to that used in most conventional ellipsometers which incorporate a rotating polarizer.

2.5 Calibration

The instrument angles (ϕ , θ_p and θ_a) are chosen through a trade-off of competing criteria, including sensitivity, dynamic range, and background signal. For a given polarizer angle there is a range of analyzer angles that yield close-to-optimal results. Our instrument angles were chosen experimentally to minimize the background signal while maintaining the dynamic range.

Before use, the fixed-polarizer ellipsometer must be calibrated to determine the thickness as a function of detector output. In principle, this could be accomplished by careful measurement of polarizer transmissivity and input beam intensity, taking into account the photodetector and amplifier characteristics. We have taken the simpler approach of running a series of thickness standards through the instrument to calibrate it. To measure films of unknown thickness, first the substrate (which can include thin films onto which the coating of unknown thickness is deposited) is pre-read. Then the film which coats the pre-read substrate is measured. Comparing the pre-reading to the film measurement provides a very accurate measurement of thickness change.

We consider the effects of variations in the instrument angles on the measured thickness. For a 750 Å thick film having a refractive index of 1.4, the measured thickness changes by $\sim 0.3\%$ per degree variation in θ_p , $\sim 3\%$ per degree variation in θ_a and 6% per degree variation in ϕ . This assumes no pre-reading. When there is a pre-reading, the closer the thickness of the pre-read substrate is to that of the coated substrate, the smaller the error. We have found that it is not difficult to achieve sufficient precision in the instrument angles to produce highly repeatable measurements. We note that the measurement is least tolerant to variations in ϕ , and therefore the instrument arms should be fixed rigidly.

3 Examples

3.1 Siloxane Polymer Film

We have made a series of thickness standards in order to calibrate the fixed-polarizer ellipsometer. These standards consist of a siloxane polymer film spun onto a silicon wafer and baked. These films vary in thickness from 23 Å to 1570 Å and have an index of 1.41 as determined by a Gaertner ellipsometer.¹¹ The silicon wafer has an index of 3.821

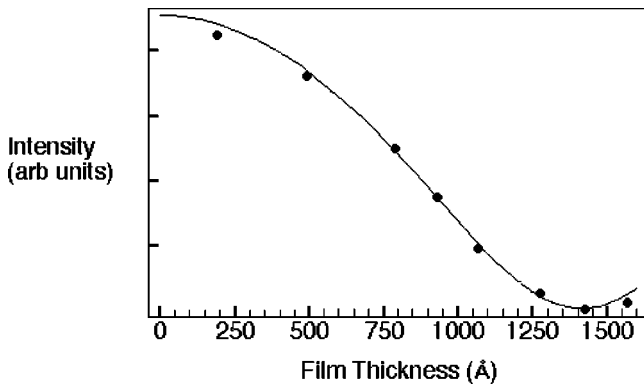


Fig. 3 Comparison of simulated (line) and experimental (points) results, for a siloxane film. The wavelength is 670 nm, the refractive index is 1.41, the polarizer angle (θ_p) is -25° , the analyzer angle (θ_a) is 80° , and the instrument arm angle (ϕ) is 55° .

$+0.014i$ for $\lambda = 670$ nm.¹² The index of silicon is complex because it is absorptive at this measurement wavelength.

3.2 Thickness Standards

The thickness standards described above have been measured using both our fixed-polarizer ellipsometer and a standard ellipsometer.¹¹ In the current working version of the fixed-polarizer ellipsometer, the intensity-readout scale is arbitrary but the response of the detector is linear with intensity in the thickness region of interest. To scale the normalized intensities found in the theory to the arbitrary units of the fixed-polarizer ellipsometer, we normalize around a given point.

We have modeled the expected response of the fixed-polarizer ellipsometer for two different sets of polarizer orientations. The first case involved setting θ_p and then θ_a manually so as to minimize the output intensity for a film-free substrate. The results, shown in Fig. 2, show close agreement between theory and experiment. For the second case, θ_p and θ_a are chosen to maximize background intensity for a film-free substrate. As can be seen in Fig. 3, this arrangement allows us to reverse the trend in our readings such that increasing thickness yields decreasing intensity.

4 Application to Immunoassay

Two examples of the application of the fixed-polarizer to immunoassay are described below. In both examples, specific antibody molecules are coated onto the silicon wafer. The surface is then pre-read, as described above, to determine a baseline reading. When a sample containing the specific analyte is placed onto the test surface, binding occurs between the analyte (antigen) and the immobilized antibody, causing an increase in thickness of the molecular thin film. The thickness increase over the baseline reading is measured by the fixed-polarizer ellipsometer. If antigen is not present in the sample, no binding takes place and the baseline reading is maintained.

4.1 Hepatitis B Surface Antigen (HBsAg)

The HBsAg assay is a 30-minute, room-temperature immunoassay. Dilutions of HBsAg in plasma ranging from 0.1

ng/ml to 10 ng/ml were incubated on the antibody coated surfaces for 15 minutes. The surface was washed and monoclonal antibody to HBsAg labeled with horse-radish peroxidase (HRP) was incubated on the surface for 5 minutes. The surface was washed and a precipitating HRP substrate was incubated on the surface for 10 minutes. The surface was washed and air dried, and the results were measured by the fixed-polarizer ellipsometer. The 0.1 ng/ml specimen gave a signal that was 10 fold above that of the negative control. The detection limit calculated as 3 standard deviations above the mean negative control reading results in a theoretical cutoff for this assay of 4 pg/ml. However, the clinical sensitivity of this assay must be validated by analyzing negative samples and a range of low-level positive clinical samples.

4.2 Group A *Streptococcus* (GAS) Antigen

The performance of the fixed-polarizer ellipsometer was compared to the manual STREP A optical immunoassay (OIA[®]) for identification of GAS antigen from patient throat swabs. The manual STREP A OIA test has consistently been shown to be more sensitive than other rapid test formats. The assay is also more sensitive than a routine agar throat culture.^{13,14} For this comparison, pharyngeal swabs were collected from symptomatic patients and acid extracted to release the group A specific antigen. After an initial reading to establish the baseline value, HRP-labeled antibody was added to the extraction and the sample was incubated on the antibody coated surface. After a 3 minute incubation, the surface was washed and the precipitating substrate was added for 4 minutes, washed, and then the surface thickness was measured. Total assay time was 10 minutes.

The results of this study, comprising 107 samples, is presented in Fig. 4. The purpose of this figure is to show a comparison of results using the fixed-polarizer ellipsometer and the manual STREP A optical immunoassay (OIA[®]) and to show the spread of intensity readings and establish a clinical cutoff for positive/negative sample discrimination. The cutoff was set at 60 mV for this assay because this level gave 99% correlation with the manual assay. The data show that a clear discrimination can be made between the positive and negative patient population.

5 Discussion

A wide range of different methods to quantify immunoassay results have been developed, from spectrophotometric determination of colorimetric substrates to sophisticated biosensors.¹⁵ We compare three immunoassay systems, the Abbott IMx[®], electrochemiluminescence, and surface plasmon resonance to the fixed-polarizer ellipsometer. Several immunoassays are used as a basis for comparison of sensitivity among the different detection platforms.

The IMx system is widely used for commercial immunoassays. Assays conducted on the IMx instrument to quantify high molecular weight analytes are based on the Microparticle capture Enzyme ImmunoAssay (MEIA).¹⁶ MEIA can be formatted in a "sandwich" assay in which

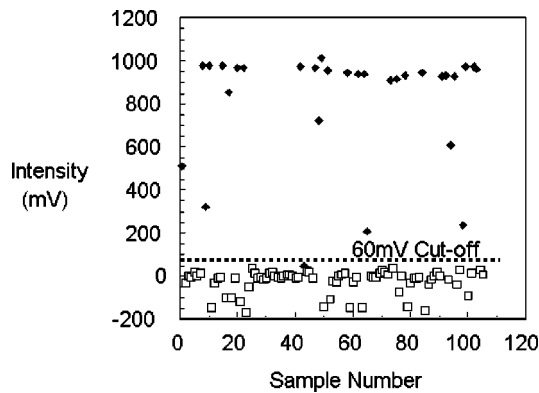


Fig. 4 Comparison of the fixed-polarizer ellipsometer and the manual STREP A optical immunoassay (OIA®) for identification of group A streptococcal antigen in 107 different samples from patient throat swabs. The open squares were identified as negative by the manual assay and the filled diamonds as positive. The light intensity, measured using the fixed-polarizer ellipsometer (in mV), is plotted as a function of sample number. The discriminating value between positive and negative samples was chosen to be 60 mV because this level gave 99% correlation with the manual assay (with only sample #43 in disagreement).

latex microparticles are covalently coated with capture antibody. A capture complex is formed on the microparticle in the presence of analyte. An enzyme-labeled antibody conjugate then forms the second half of the sandwich complex. Microparticles are captured on a glass fiber matrix and unbound conjugate is washed through. The remaining conjugate fluoresces in response to excitation from a filtered mercury vapor lamp. The fluorescence intensity is detected by a photomultiplier tube. The detection limit of the MEIA technology for measurement of HBsAg is 0.31 ng/ml.¹⁷ In comparison, the fixed-polarizer ellipsometer limit of detection for this analyte is 0.004 ng/ml.

A second detection technology for immunoassay reactions is electrochemiluminescence, developed by IGEN.¹⁸ In this system magnetic beads coated with capture antibody are incubated with the target and conjugate antibody labeled with ruthenium to produce luminescence. The immune complex sandwich is captured onto an electrode surface by a magnetic force, and unbound reagents are washed away. A voltage is applied between the electrode and a counterelectrode in the solution, causing an electron transfer reaction which produces light emission from the labeled molecules. The resulting electrochemiluminescence intensity is measured with an adjacent photomultiplier tube. The detection limit of this system ranges from 10 pg/ml to 5 ng/ml, depending on the analyte.

Surface plasmon resonance is generated when a light beam is directed through a medium of sufficiently high refractive index onto a thin film of metal at a critical angle, which is dependent upon the wavelength of the light and the characteristics of the thin film. Usually the high refractive index medium is a glass prism. At resonance the light is absorbed, but for other conditions the light is reflected. The resonance condition may be shifted by biomolecules captured close to the metal surface.^{19,20} Thus the reflected light intensity varies with the mass and concentration of adsorbed target molecules in the sample. The detection limit of surface plasmon resonance is in the ng/ml range

when a labeled secondary antibody is used to amplify the signal.

Based on the HBsAg test described above, the theoretical sensitivity of the fixed-polarizer ellipsometer, which is 4 pg/ml for this analyte, is significantly higher than that of commercially available assay systems. The sensitivity of fluorescence or chemiluminescence systems ranges from 0.15 to 0.3 ng/ml with a time to result of 25 to 60 minutes.^{17,21} In our GAS test, the high agreement between the fixed-polarizer ellipsometer and the manual assay implies that this automated assay for identification of GAS antigen is also more sensitive than routine agar culture methods. In addition, this assay is complete in approximately 10 minutes while culture results are not available for 24-48 hours. None of the other technologies appears to be as sensitive as the fixed-polarizer ellipsometer which has demonstrated sensitivity in the pg/ml range depending upon the analyte.

The three alternative technologies described above measure a signal which is generated over a brief period of time and subsequently decays or disappears. Generation of a thin film as the positive signal for an optical immunoassay using the fixed-polarizer ellipsometer creates a permanent result which can be measured at any time.

6 Conclusions

The main limitation of this measurement technique is that a prior knowledge of the sample optical constants is required. The substrate (including any non-changing layers) must be well-characterized and the index of the film being grown must be known. Therefore, the fixed-polarizer ellipsometer is not useful where these quantities are unknown or cannot be found through separate means. Fortunately, these values are often known or measurable even in a research environment.

A more practical application of the fixed-polarizer ellipsometer, however, is in a production environment. As this system requires none of the rotating components or complex calculations of a standard ellipsometer, it is far less expensive and easier and faster to use. In a production setting, the substrates and film indices are generally well known and are within the tolerances imposed by the fixed-polarizer ellipsometer. Different substrates and films can be accommodated by changing the polarizer and analyzer angles and calculating, or looking up, a new calibration curve. This new curve is calculated using the mathematics above. As the fixed-polarizer ellipsometer must be recalibrated only once for each system being measured, the time required to retool is minimal.

For biological applications, assays that require sensitivity for a low-concentration range of analyte are suitable candidates for analysis with the fixed-polarizer ellipsometer. It is well adapted for measuring thin films generated by molecular binding events, such as immunological reactions, nucleic acid probe detection, and receptor-analyte binding.

In conclusion, we have demonstrated a working instrument with which to measure thickness and thickness changes. This fixed-polarizer ellipsometer has a linear response over 800 Å, is simple to calibrate and operate, and can accommodate different substrates and films easily. It is inexpensive to manufacture and compact, so that it fits in diagnostic laboratory or physician office environments.

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