

Conf-9104187--1

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CONF-9104187--1

[Submitted to Journal of Applied Physics]

DE91 010131

Two-Channel Spectroscopic Polarization Modulation Ellipsometry: A New Technique for the Analysis Thin SiO₂ Films

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March 1991

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Two-Channel Spectroscopic Polarization Modulation Ellipsometry: A New Technique for the Analysis Thin SiO₂ Films

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Abstract

A new spectroscopic ellipsometer is described, where the incoming light is dynamically polarized using a photoelastic modulator, and the reflected light is separated into orthogonally polarized beams using a Wollaston prism. Both beams are detected using photomultiplier tubes whose bias voltage is dynamically controlled for constant dc. All three of the associated ellipsometry parameters ($N = \cos 2\psi$, $S = \sin 2\psi \sin \Delta$, $C = \sin 2\psi \cos \Delta$) can be determined simultaneously in a single scan (240–840 nm or 5.16–1.47 eV).

This instrument has been used to study the optical properties of thin SiO₂ films from 3 to 325 nm in thickness. Using a biased estimator fitting technique, the raw ellipsometric data can be fit to an air-SiO₂-interface-Si model, where the optical functions of the SiO₂ layer and the interface region are approximated using a one-term Sellmeier approximation, and a 50% voids, 50% SiO₂ Bruggeman effective medium approximation, respectively. The refractive index of the SiO₂ layer is dependent on film thickness, increasing with decreasing film thickness, but always being greater than fused quartz. The thickness of the interfacial region increases with increasing film thickness, being less than 0.2 nm for film thicknesses <20 nm.

Introduction

Ellipsometry is a technique whereby elliptically polarized light is reflected from a sample surface, analyzed with another polarizer, and detected. Historically, ellipsometric data has been used to determine the bulk optical functions of a material or the thickness of an overlaying film. Recently, ellipsometric data from more complicated sample surfaces has been interpreted in terms of surface roughness or film composition. Ellipsometry configurations can take many different forms; in all cases, the ellipsometric angles ψ and Δ are determined, where

$$\rho = r_p/r_s = \tan \psi e^{i\Delta} . \quad (1)$$

The quantities r_p (r_s) are the complex Fresnel reflection coefficients for light polarized parallel (perpendicular) to the plane of incidence. Alternately the ellipsometric data can be expressed in terms of the associated ellipsometry parameters:

$$N = \cos 2\psi , \quad (2a)$$

$$S = \sin 2\psi \sin \Delta , \quad (2b)$$

$$C = \sin 2\psi \cos \Delta . \quad (2c)$$

Note that N , S , and C are not strictly independent, since $N^2 + S^2 + C^2 = 1$.

The simplest and most accurate ellipsometers are the nulling ellipsometers (NE), which operate at a single wavelength, and the ellipsometry angles ψ and Δ are determined by extinguishing the light after the analyzer. These instruments have been commercially available for many years and are well understood.¹

Rotating analyzer ellipsometers (RAE's) are the most popular spectroscopic ellipsometers available today, and are based on a design first described by Aspnes and Studna.² In the RAE design, the incident light is plane-polarized, and the reflected light passes through the analyzer polarizer which rotates at frequencies from 20-100 Hz. The RAE measures $(\cos 2\theta_p - N)$ and $(C \sin 2\theta_p)$, where θ_p is the azimuthal angle of the polarizer. The calibration and operation of the RAE are well-understood, and have become a de facto industry standard; commercial RAE's are now available.

However, the RAE suffers from some inherent defects: (1) The time resolution of the ellipsometer is limited to about 0.1 sec (due to the analyzer rotation frequency of 20-100 Hz). (2) Without a phase-retarding optical element, the RAE is limited to measuring quantities proportional to N and C ; therefore the RAE is very insensitive to Δ when Δ is near 0° and 180° . (3) The RAE cannot measure the sign of Δ . Problems (2) and (3) can be partially solved by the incorporation of a compensator in the light path or by changing the azimuthal angle of the polarizer, but this solution is not general.

Before the design of the RAE, Jasperson and Schnatterly³ designed and built an ellipsometer which alleviates many of the shortcomings of the RAE. This ellipsometer uses a photoelastic modulator as the compensating element, and is called a polarization modulation ellipsometer (PME), since the light incident upon the sample is dynamically elliptically polarized. This instrument is capable of measuring N , S , or C , but a determination of all three requires at least 3 scans. Drevillon et al.⁴ modified the detection circuit, replacing the lock-in amplifiers with fast digitizers; by determining the amplitudes of both the fundamental and the second harmonic, S and either N or C can be determined in a single scan.

In this paper, a new ellipsometer based on the photoelastic modulator is reviewed; details can be found in Refs. 5-7. This ellipsometer, called a 2-channel

polarization modulation ellipsometer (2-C PME), utilizes a Wollaston prism as the analyzing prism, whereby the reflected beam is split into two orthogonally polarized beams. This instrument has the advantage that N , S , and C can be measured simultaneously, allowing measurements to be made on materials previously inaccessible to spectroscopic ellipsometry investigations, such as thin interfaces on insulators,⁸ intermediate values of the absorption coefficient^{5,8} and study thin SiO_2 films on Si .⁹

As an example of the utility of this instrument, the results of an ellipsometric study of thin SiO_2 films on Si ⁹ will be reviewed. In general, very thin films of SiO_2 on Si have not been examined using ellipsometry techniques, since, to first order, the measured parameters are functions of the product of film thickness and refractive index. If extremely accurate ellipsometric measurements can be made, then it is possible to see second order effects, which can decouple the effects of film thickness and refractive index. The 2-C PME is ideal for spectroscopic ellipsometry studies of thin SiO_2 films on Si , since it alone can make very accurate, spectroscopic measurements of the ellipsometric angle Δ .

Description of the 2-Channel Polarization Modulation Ellipsometer

The 2-C PME ellipsometer that has been set up in our laboratory (shown schematically in Fig. 1) has been built on an optical bench for optimum flexibility and stability. The broad-band light source is a 75 W Xenon lamp in an elliptical mirror housing, which is focused onto the entrance slit of a 1/4 m single monochromator, with a 1200 lines/mm replica grating. The output light from the monochromator is collected and quasi-collimated by an off-axis parabolic mirror, and two flat mirrors are used to deflect the light beam; the beam divergence at the sample is $<0.1^\circ$.

The light passes through an aperture, a depolarizer, a filter (to limit scattered light or to remove second-order light), the polarizer/modulator combination, and is

then incident upon the sample. The reflected light passes through a Wollaston prism, which splits the beam into two mutually orthogonal beams, each of which is detected using a photomultiplier tube (see Fig. 2).

The polarizer/modulator combination consists of a high-quality calcite Glan-Taylor polarizing prism attached directly to the modulator with a manual rotator (0.01° accuracy). Both the polarizer/modulator and the Wollaston prism/detector combinations are attached to computer-controlled automatic rotators, resulting in 0.02° accuracy in the setting of the respective azimuthal angles.

A schematic diagram of the photoelastic modulator is shown in Fig. 3. An oscillating voltage is applied to a piece of crystalline quartz (c-SiO₂), precisely cut so that an oscillating stress is set up in the *i*-direction. A piece of fused quartz (a-SiO₂) is also precisely cut to resonate at the same frequency, and mechanical coupling to the crystalline quartz sets up a periodic stress in the *i*-direction of the fused quartz. The frequency of the oscillation is determined by the geometry of the quartz, and is ~ 50.2 kHz in our case. Internal stresses in the fused quartz result in an additional static retardation induced in the modulator.

The bias voltage of the phototubes are dynamically controlled such that the dc current is constant.⁷ The photocurrent from each channel, first converted to a voltage, is measured using a DVM and two lock-in amplifiers, one tuned to the fundamental frequency and the other tuned to the 2nd harmonic. The entire experiment is computer controlled, and many of the calibration routines are performed automatically. The wavelength range of the instrument is 240–840 nm, determined in the UV by the cutoff of calcite, and in the IR by the phototube response.

Theoretical Considerations

The intensity of the light reaching the photodetector will have dc, fundamental, and 2nd harmonic components (as well and higher harmonics), where the fundamental frequency is the resonant frequency of the photoelastic modulator. Generally, the light intensity of the j -th harmonic can be expressed as⁵

$$I(j\omega) = a_{j0} + a_{jN} N + a_{jS} S + a_{jC} C , \quad (3)$$

where the a -parameters are complicated functions of the sines and cosines of the azimuthal angles θ_b (the polarizer with respect to the modulator), θ_m and θ_a (the modulator and analyzer with respect to the plane of incidence at the sample), and of the time-dependent phase retardation introduced by the modulator δ .

The time-dependent retardation introduced by the photoelastic modulator is given by

$$\delta(V_m, \lambda, t) = A(\lambda, V_m) \sin(\omega t) + \delta_0 , \quad (4)$$

where V_m is the drive voltage of the modulator, λ is the wavelength of light, $2\pi\omega$ is the frequency of the modulator oscillation, and δ_0 is the static retardation of the modulator. Since the phase retardation δ enters the a -parameters in Eq. 3 as an argument of sines and cosines⁵, the intensities of the fundamental and second harmonic will be proportional to the integer Bessel functions $J_1(A)$ and $J_2(A)$ respectively, with the $J_0(A)$ term entering as a perturbation. Generally, V_m is set such that the Bessel angle A is 2.4048 radians ($\sim 137.79^\circ$), resulting in $J_1(A)$ and $J_2(A)$ being within 15% of their maxima and $J_0(A)=0$, thus simplifying the data analysis.

Generally, the most useful angles for measurement are $\theta_b = \pm_b 45^\circ$ and $\theta_a = \pm_a 45^\circ$; we will assume these angles throughout the rest of this paper unless otherwise stated. With these restrictions, there are 3 classes of azimuthal angle orientations which are of interest in PME measurements:

$$(1) \theta_m = \pm_m 45^\circ \quad I(\text{dc}) \propto 1, \quad (5a)$$

$$I(\omega) \propto \pm_a 2 J_1(A) S, \quad (5b)$$

$$I(2\omega) \propto \mp_m 2 J_2(A) N. \quad (5c)$$

$$(2) \theta_m = 0^\circ, 90^\circ \quad I(\text{dc}) \propto 1, \quad (6a)$$

$$I(\omega) \propto \pm_a 2 J_1(A) S, \quad (6b)$$

$$I(2\omega) \propto \mp_m \pm_a 2 J_2(A) C. \quad (6c)$$

$$(3) \theta_m = \pm_m 22.5^\circ; \pm_m 67.5^\circ \quad I(\text{dc}) \propto 1, \quad (7a)$$

$$I(\omega) \propto \pm_a 2 J_1(A) S, \quad (7b)$$

$$I(2\omega) \propto 2 J_2(A) (\zeta C \pm_a \chi N) / \sqrt{2}. \quad (7c)$$

In Eqs. 5–7 and above, the notation \pm_m and \pm_a denote sign changes due to changes in the modulator and analyzer azimuthal angles, respectively. The parameters ζ and χ in Eq. 7c are ± 1 , depending upon θ_m . These equations have been developed assuming no errors in any of the azimuthal angles or window effects; the general expressions are given in Ref. 5.

Cases 1 and 2 are the conventional configurations,^{3,4} where the parameters S and/or N or C can be measured. Case (3) is new, and is discussed in more detail in Ref. 5. If the dc, fundamental, and 2nd harmonic intensities of BOTH channels are measured, then it is possible to determine N, S, and C simultaneously.

The 2-C PME has several advantages over the conventional RAE: (1) Since N, S, and C are all measured, there is no ambiguity of the sign of ψ or Δ . (2) There is no value of ψ or Δ to which the 2-C PME is insensitive; RAE's measure $\cos \Delta$, making them very insensitive to Δ when $\Delta \sim 0^\circ$ or 180° . (3) The time response of PME is ~ 500

times faster than a RAE. (4) Since N, S, and C are measured, the sum of the squares can be calculated; this allows for on-line checks of the experiment and a renormalization of N, S, and C (see Ref. 5).

Example: SiO₂ films on Si

Introduction

Thin films of SiO₂ grown on Si have been the subject of many ellipsometry studies over the last few years; in fact, single-wavelength NE is routinely used to determine the thickness of an insulating film (such as SiO₂) on silicon.¹ Generally, the analysis of ellipsometry data from SiO₂ films on Si assumes that the optical functions of SiO₂ correspond to bulk fused silica and that no interfacial layer exists between the Si substrate and the SiO₂ thin film.

Several studies¹⁰⁻¹¹ of relatively thick SiO₂ layers on Si have shown that many of these simplifying assumptions used in the interpretation of NE data are not valid. In particular, the interpretation of the ellipsometric data requires the existence of an interface layer between the SiO₂ and the Si substrate, and a refractive index of the SiO₂ layer greater than that of fused silica. Very thin films of SiO₂ on Si have generally not been examined using ellipsometry techniques, since, to first order, the measured parameters are functions of the product of film thickness and film refractive index. If extremely accurate spectroscopic ellipsometry measurements can be made, then second order effects may be used to decouple the effects of film thickness and refractive index.

Experiment and data reduction

As an example of the utility of the 2-C PME, the results of an ellipsometry study of SiO₂ on Si will be reviewed (see Ref. 9 for details). For this study, 32 different samples of thin film SiO₂ on crystalline Si [5 Ω -cm, float-zoned, (100)] were

prepared in a dry oxygen environment using growth temperatures from 800°C to 1050°C; the resulting sample thicknesses were from ~3 nm to 325 nm.

In order to interpret the measurements, the data were first converted to complex ρ (see Eq. 1); sample spectra in the ρ representation are shown in Fig. 4. The complex ρ data were then fit to various models, using an unbiased estimator as a figure of merit.¹² The fitting procedure minimizes the χ^2 given by

$$\chi^2 = \frac{n_\lambda}{[1/(n_\lambda - m_z - 1)]} \sum_{i=1}^{n_\lambda} [\rho_{\text{calc}}(\lambda_i, \mathbf{z}) - \rho_{\text{exp}}(\lambda_i)]^2 / \delta \rho_i(\lambda_i)^2, \quad (8)$$

where the sum goes over the n_λ wavelength points in the spectrum. The calculated spectrum ρ_{calc} is determined from the parameter vector \mathbf{z} , which has m_z elements. The elements of the parameter vector \mathbf{z} , which may be film thicknesses, constituent fractions, or Sellmeier coefficients, are varied to minimize χ^2 . The χ^2 is minimized using a Levenberg-Marquardt minimization algorithm, and the variance-covariance matrix and a "goodness of fit" parameter are calculated^{11,12}.

As is discussed at length in Ref. 12, the χ^2 has significant meaning in determining whether the fit actually is consistent with the data only if the errors in the data ($\delta \rho_i$) are included in Eq. 8, and the errors are normally distributed. Under these circumstances, $\chi^2 \sim 1$ indicates that the fit is consistent with the data, while $\chi^2 \gg 1$ indicates that the model does not fit the experimental data.

The models used to fit the data each assume that the ambient was vacuum ($n=1$) and consist of 3 media: (1) a SiO₂ film, modelled using a single-term Sellmeier approximation, (2) a thin interfacial layer, modelled the Bruggeman effective medium approximation¹³, assuming a 50% mixture of fused SiO₂ and crystalline Si, and (3) a crystalline silicon substrate.

The fits to the data were performed by varying some of the following parameters: (1) the thickness of layer 1, d_1 ; (2) the Sellmeier A coefficient of layer 1; (3) the Sellmeier λ_0 coefficient of layer 1; and (4) the thickness of layer 2, d_2 . The optical functions of the SiO₂ layer were determined from a single-term Sellmeier approximation:

$$n^2 - 1 = A \lambda^2 / (\lambda^2 - \lambda_0^2), \quad (9)$$

where it was assumed that the film was non-absorptive (i.e. $k=0$). A single-term fit to the data of Malitson¹⁴ between 240 and 780 nm yields $A_{\text{SiO}_2} = 1.09905$ and $\lambda_{\text{SiO}_2} = 92.27$ nm, with an average absolute residual in the refractive index of ~ 0.0002 .

The optical properties (refractive index n and extinction coefficient k) of the silicon substrate were determined from separate 2-C SPME measurements of a (100) sample of crystalline silicon which had been stripped of most of its oxide overlayer. The data were then mathematically corrected for the ~ 7 Å excess oxide remaining after the cleaning, which was modelled using the Sellmeier approximation, with $A = 2.0$ and $\lambda_0 = 92.27$ nm (see below). The thickness of the overlayer was chosen such that the extinction coefficient $k_{\text{Si}} = 0.018$ at $\lambda = 630$ nm.¹⁵

The most appropriate fit for the thickest films ($d_1 > 100$ nm) included the variables d_1 , A , λ_0 , and d_2 , holding $f_2=0.5$; thinner films ($25 \text{ nm} < d_1 \leq 100 \text{ nm}$) were most appropriately fit using the variables d_1 , A , and d_2 , holding $\lambda_0 = 92.27$ nm and $f_2 = 0.5$; and the thinnest films ($d_1 \leq 25$ nm) were most appropriately fit using the variables d_1 and A , keeping $\lambda_0 = 92.27$ and eliminating the interface layer.⁹

Results from the fitting procedure

Figure 5 shows the values of the Sellmeier A coefficient and selected error limits plotted versus the log of the SiO₂ film thickness. The value of A is greater than the value of A for fused silica (shown by the solid line in Fig. 5) for all samples, and the fitted values of A and their errors increase with decreasing d_1 . A linear plot shows that the extrapolated value for $A \sim 2.0$ for very thin SiO₂ films on Si. (Hence the rationale for using $A=2.0$ in calculating n and k for Si mentioned above.) This increase of A corresponds directly to an increase in the refractive index with decreasing film thickness. There is, however, no clear dependence of the Sellmeier A coefficient with growth temperature.

Figure 6 shows a plot of the calculated interfacial layer thickness (d_2) from samples with oxide thicknesses >25 nm. Clearly, the interfacial layer thickness increases with increasing film thickness, although there is sizable sample-to-sample variation; there is no discernable dependence of d_2 on growth temperature.

Table I presents a listing of some of the polymorphs of SiO₂, along with some of their characteristics. The table lists two types of amorphous SiO₂: the normal fused silica, and a high pressure form found by Hongsen et al.¹⁷ Several crystalline forms are listed, including tridymite and cristobalite, which are metastable and have a relatively low refractive index, and coesite and stishovite, which are stable, but only at high pressures and temperatures. Coesite, which consists of SiO₄ units in 4-membered rings, represents the highest packing density (hence the highest refractive index) that can be obtained with SiO₄ units; stishovite consists of SiO₆ units. The Sellmeier A coefficients (at $\lambda = 589$ nm, assuming $\lambda_0 = 92.27$ nm) are shown by the arrows to the right of Fig. 5 for some of the polymorphs.

The Sellmeier A data (or equivalently, the refractive index) presented in Fig. 5 and the data on crystalline materials shown in Table I suggest that the structure of thin SiO₂ films consist of amorphous analogs of coesite, quartz, keatite, cristobalite, and tridymite. Since the lattice mismatch between Si and any form of SiO₂ will

result in a very large, compressive stress, it is reasonable that there will be a tendency for denser forms to be created. The thinnest films (2–5 nm) would consist primarily of SiO_4 tetrahedra arranged in 4-membered rings, analogous to coesite. Thicker films (5–10 nm) would increasingly add 5- or 6-membered rings, with relatively close packing (similar to quartz), while even thicker films (>10 nm) would consist of an increasing number of looser packed SiO_4 units, arranged in 6-membered rings, reminiscent of cristobalite or tridymite. Even the thickest films do not have as low an α coefficient as fused silica, and so are closer packed than fused silica.

ACKNOWLEDGEMENTS

This research is sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

Table I

Various properties of polymorphs of crystalline SiO₂ and fused silica. The quantity n is the refractive index at 589 nm and A is the Sellmeier coefficient (see Eq. 5b), assuming $\lambda_0=92.27$ nm. The density is given in gm/cm³ and CN is the Si coordination number for each form. Stable temperatures (°C) and pressures (GPa) are listed in the description column

Material	Density	n	A	CN	Ref	Description
fused silica	2.20	1.459	1.099	4	a	normal glass form
α -tridymite	2.26	1.475	1.147	4	a	metastable below 117 °C
α -cristobalite	2.33	1.485	1.176	4	a	Metastable below 267 °C
keatite	2.50	1.519	1.275	4	a	Synthetic: 380-585 °C; 0.33-1.2 GPa
HP-glass	2.56	1.532	1.314	4	b	300 °C; 9.8 GPa
α -quartz	2.65	1.547	1.359	4	a	Stable below 573 °C
coesite	2.92	1.596	1.509	4	a	Stable for >500 °C; >2.0 GPa
stishovite	4.28	1.815	2.238	6	a	Stable for >7.6 GPa

a Ref. 16 and references therein.

b Ref. 17

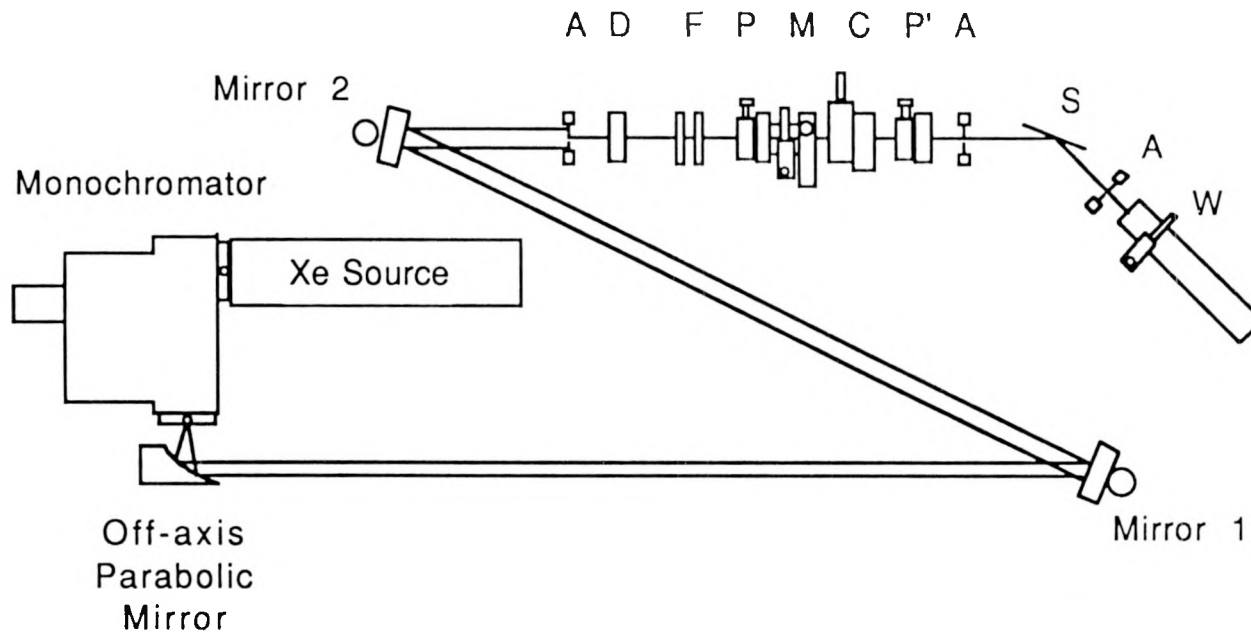
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FIGURE CAPTIONS

1. Schematic diagram of the 2-channel polarization modulation ellipsometer.
2. A schematic diagram of the photoelastic modulator. The applied ac voltage V is applied between the front and back faces of the crystalline quartz component. The black strips represent an aperture which restricts the light to the central part of the fused quartz.
3. Wollaston prism and phototube assembly.
4. The real (bottom) and imaginary (top) parts of the complex Fresnel reflection ratio ρ for 6 thin SiO_2 films on Si, plotted versus photon energy. The nominal thicknesses of the films are listed in the legend in nm.
5. Sellmeier A coefficient plotted versus the log of the thickness of the SiO_2 layer. The values of A of various high-index polymorphs of crystalline SiO_2 are shown to the right by the arrows. The straight line at $A = 1.099$ corresponds to the A for fused silica. The data sets are labeled by their growth temperatures.
6. The interfacial layer thickness in nm plotted versus the log of the SiO_2 thickness for several samples included in this study. The data sets are labeled according to their growth temperatures.

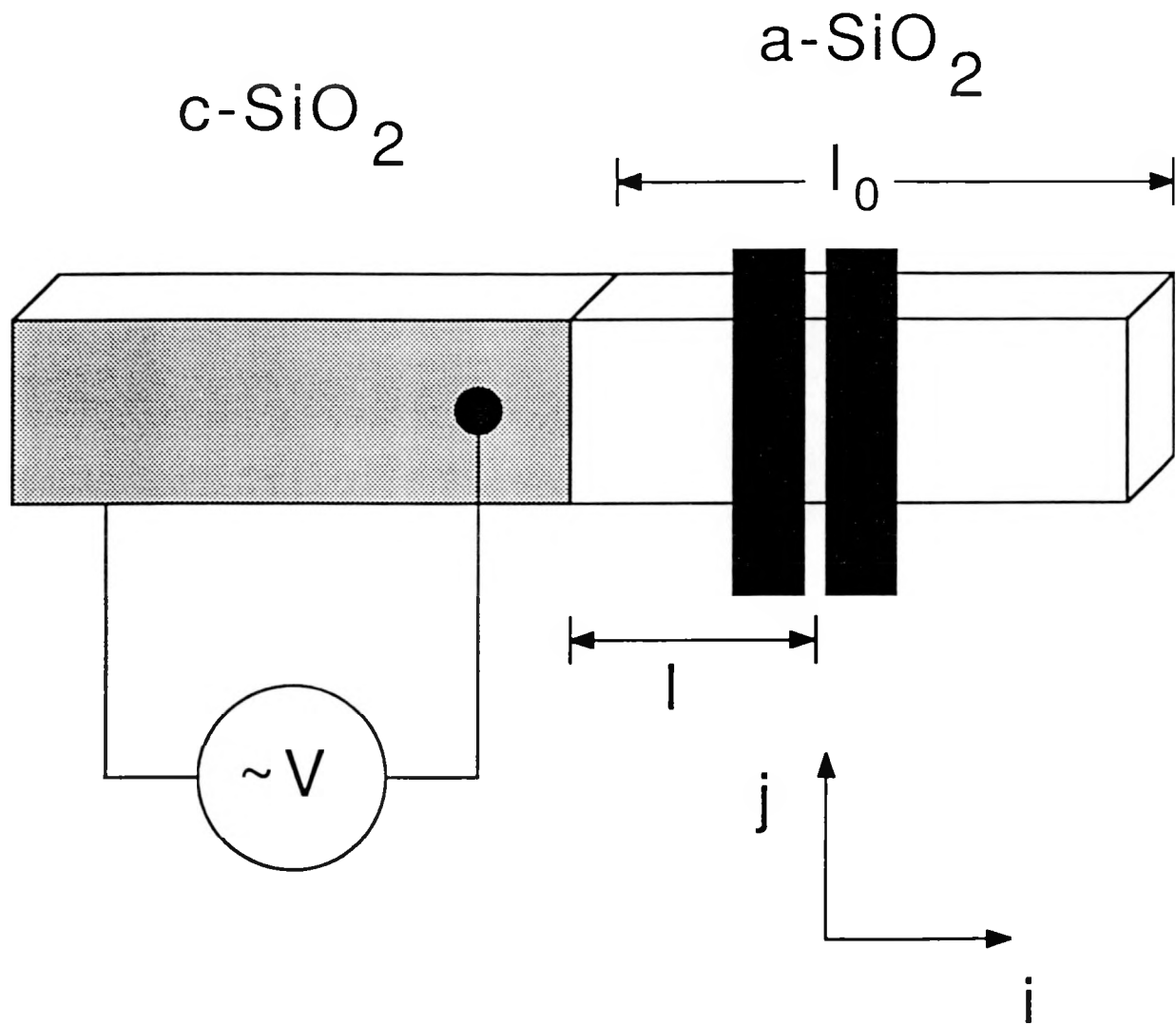
Polarization Modulation Ellipsometer



LEGEND

A	Aperture	C	Cal. Compensator
D	Depolarizer	P'	Cal. Polarizer
F	Filters	S	Sample
P	Polarizer	W	Wollaston Prism
M	Modulator		+ Detector

Photoelastic Modulator



Wollaston Prism and Detector Assembly

