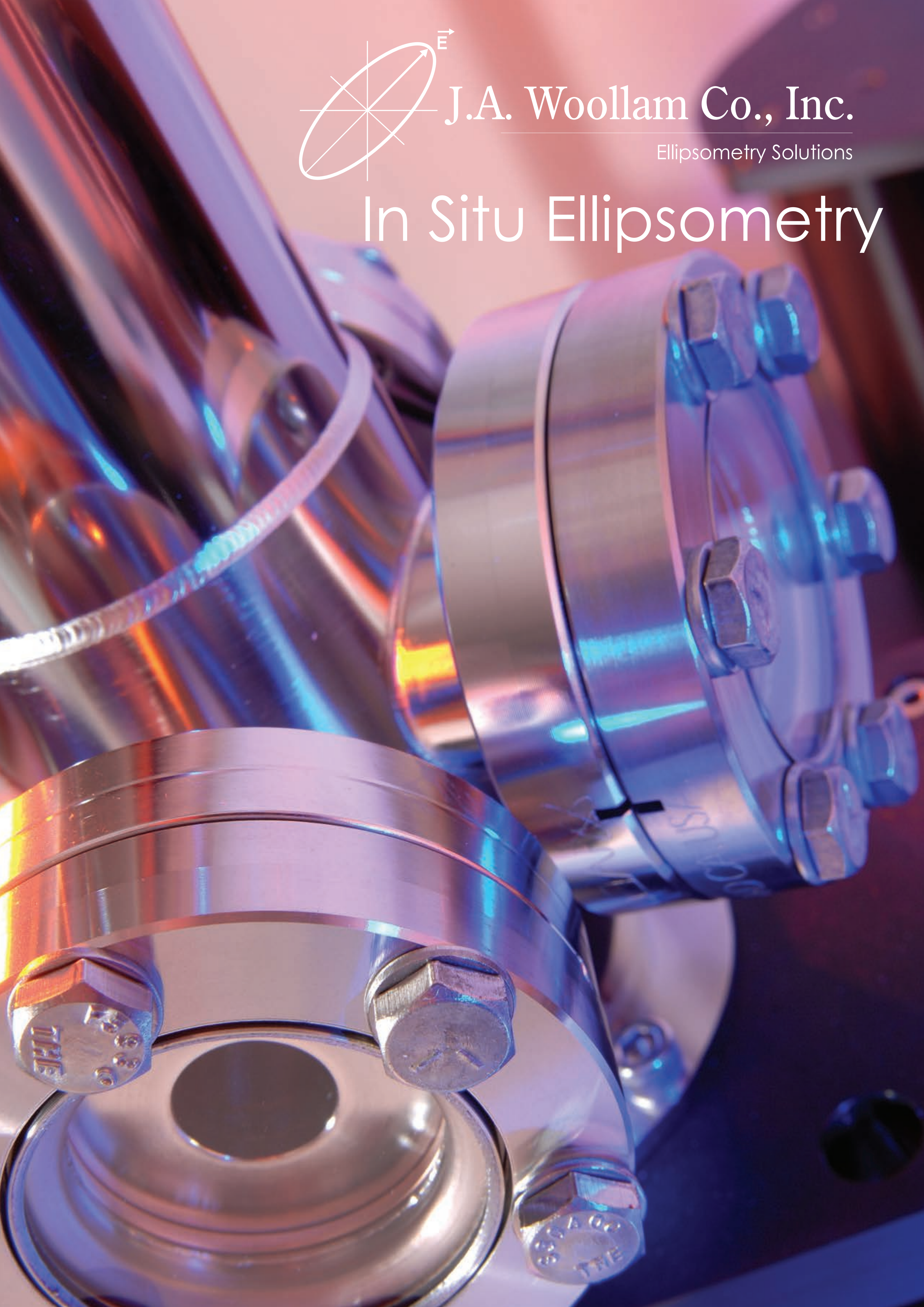
J.A. Woollam Co., Inc.

Ellipsometry Solutions

In Situ Ellipsometry



Real-Time

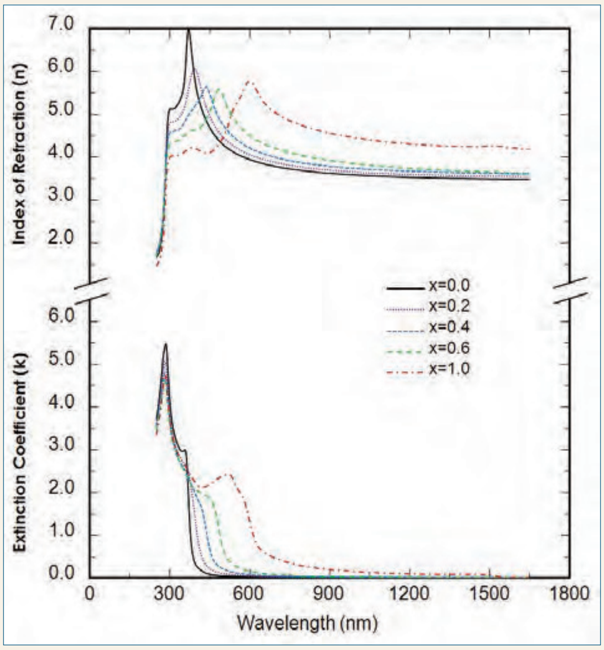
In Situ Ellipsometry

In Situ Spectroscopic Ellipsometry (SE) measures a sample “in position” as conditions are varied. This includes measurements during film growth or removal; during variation in temperature, humidity, or other environmental conditions; and during other external stimulation of the sample (magnetic, electric,…).

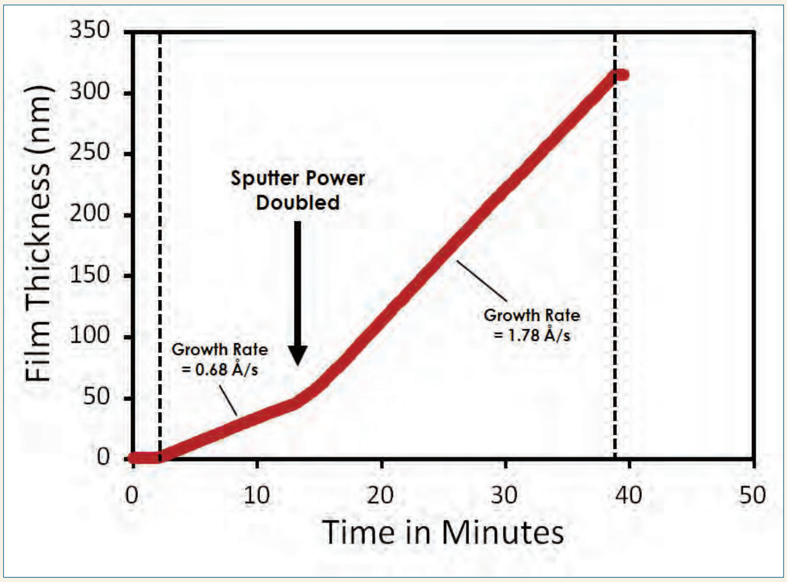
In Situ SE is routinely used to monitor growth or etch of a thin film and can be implemented for real-time feedback control. It is also common to use in situ measurements to characterize optical constants during different process conditions.

Common In Situ SE Measurements

- Thin Film Thickness from Single- or Multiple-Layers
- Growth or Etch Rate
- Optical Constants (n,k)
- Surface Quality before and after Processing
- Process Conditions that affect Optical Constants (Deposition Rate, Vacuum Pressure, Power, …)
- Real-time End-point Detection
- Material Properties that have an effect on Optical Constants
  - Composition, Crystallinity, Conductivity, Anisotropy,…



Optical constants measured for a series of different $Si_{1-x}Ge_x$ films. Process conditions are used to control the desired composition.



Film thickness determined from in situ SE measurements during sputter deposition. Note the change in growth rate that occurs when sputtering power was doubled.

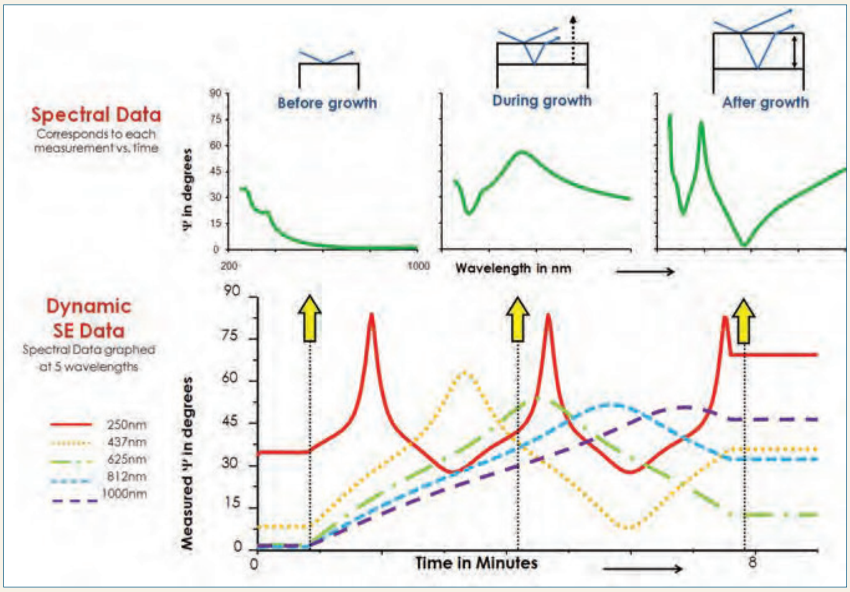
In Situ Advantages

The power of spectroscopic ellipsometry is only amplified when applied in situ. In situ measurements allow access to sample states simply unavailable during standard ex situ measurements.

Monitor Thin Films during Processing in Real-time

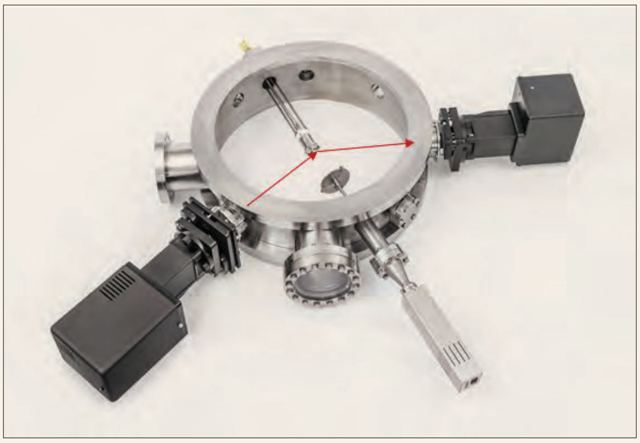
With in situ capability, the sample can be characterized:

- Prior to Film Deposition for Accurate Substrate Characterization
- In Real-time for Thickness and Optical Constants Monitoring
- Before Exposure to Air/Oxidization



Dynamic Spectroscopic Ellipsometry data is graphed at 5 representative wavelengths. Each measurement versus time actually consists of over 500 wavelengths across the spectrum from ultraviolet to near infrared. A few time-points are graphed above versus wavelength, showing the measurement before, during, and after film growth.

Non-Destructive and Non-Invasive

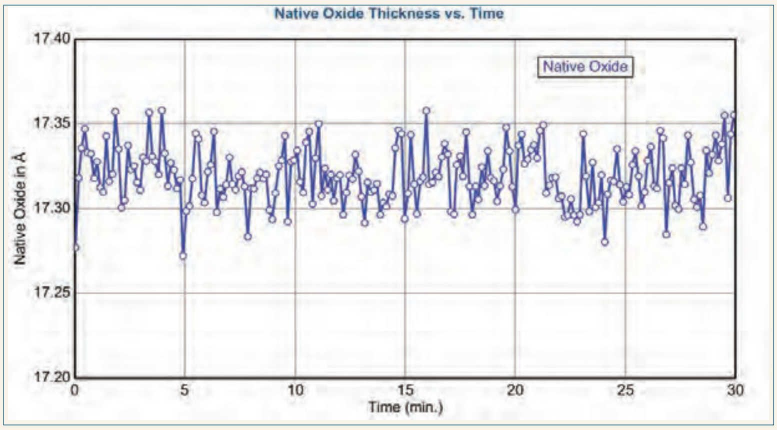


In Situ SE uses light to probe the thin film in a non-invasive manner. The ellipsometer is outside of the process chamber using windows to allow light to enter and interact with the sample. The measurement can be directly from the surface of interest without any damage or special sample preparation.

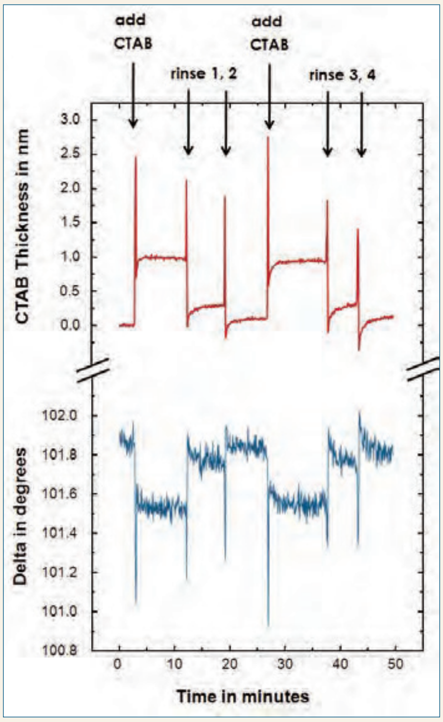
Accuracy, Precision & Sub-Monolayer Sensitivity

SE measurements determine the 'polarization' of light. For In Situ, this has significant advantages over intensity measurements (from spectrophotometer).

- Data not affected by coated windows.
- Collect accurate data even without collecting the entire beam.
  - -advantageous for moving samples
- Polarization contains 'phase' which is highly sensitive to very thin



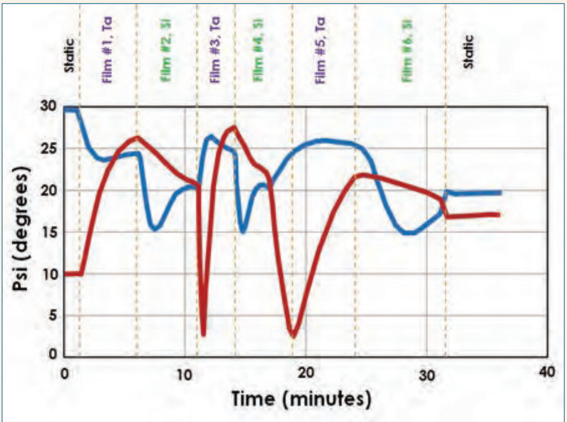
Dynamic measurements of a native silicon surface show the excellent thickness precision and stability of In Situ SE.



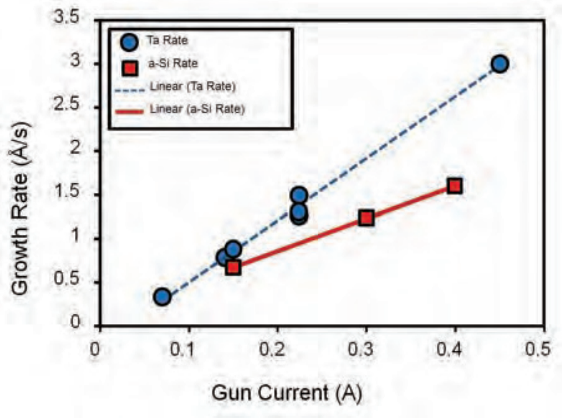
Phase (Delta) is highly sensitive to thin films, which allows precise characterization of this organic monolayer during attachment and rinsing steps.

Efficient Process Characterization

A common application of In Situ SE involves deposition of an entire series of thin films during a single process. Each film is produced with varying conditions which allows a quick understanding of the deposition process. Optical constants and growth rate are determined from each layer to characterize the process.



In Situ SE data is shown (2 of 500 measured wavelengths). The process varied between different film types and different process conditions. In this manner, a multi-layer structure was deposited, with information obtained from each individual film.



Process characterization from each layer was used to determine the optical constants and the growth rate for both Ta and a-Si films versus sputter-gun currents.

Instruments

With modular design and fast measurement capability, our M-2000® and RC2® ellipsometer models are ideal for in situ monitoring and process control. The M-2000 and RC2 are used with many different process chambers including MBE, sputter, ALD, E-beam evaporator, MOCVD, PECVD, plasma etch, PLD, ECR, and more.

M-2000

Measurement Speed	**Fastest**		**Typical**
	~20 complete spectra per second		complete spectrum in 1-2 seconds
Technology	Patented Rotating Compensator with CCD detection		
Wavelength Options	**Model**	**Wavlength Range**	**# Wavelengths**
	V	370 - 1000nm	390
	X	245 - 1000nm	470
	X-210	210 - 1000nm	485
Measurement Type	D	193 - 1000nm	500
	+I (NIR Extension)	Add 1000 - 1690nm	Additional 190
	Fast, accurate Ellipsometry measurements with good sensitivity over full range of values		

M-2000 shown attached to UHV process chamber.

RC2

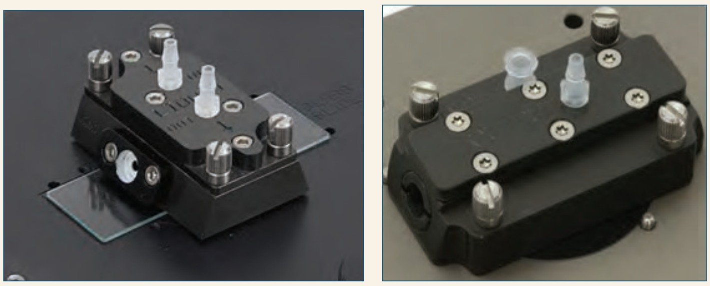
Measurement Speed	**Fastest**		**Typical**
	complete spectra in 0.3 seconds		complete spectrum in 1-2 seconds
Technology	Patented DUAL Rotating Compensator with CCD detection and Achromatic Compensators		
Wavelength Options	**Model**	**Wavlength Range**	**# Wavelengths**
	U	245 - 1000nm	470
	D	193 - 1000nm	500
	+I (NIR Extension)	Add 1000 - 1690nm	Additional 190
Measurement Type	Advanced Measurements within 0.3 seconds Fast, accurate Ellipsometry masurements with great sensitivity over full range of values Advanced measurements including Generalized SE and Full Mueller-Matrix SE		

Modular Source and detector heads for an RC2 shown along with electronics control box

Accessories

In situ experiments are not limited to large process chambers. Many experiments can be made with accessories directly integrated onto the ellipsometer base. The following accessories are available for use with the M-2000 or RC2:

Liquid Cells



Variable Temperature Liquid Cell

Cryostat

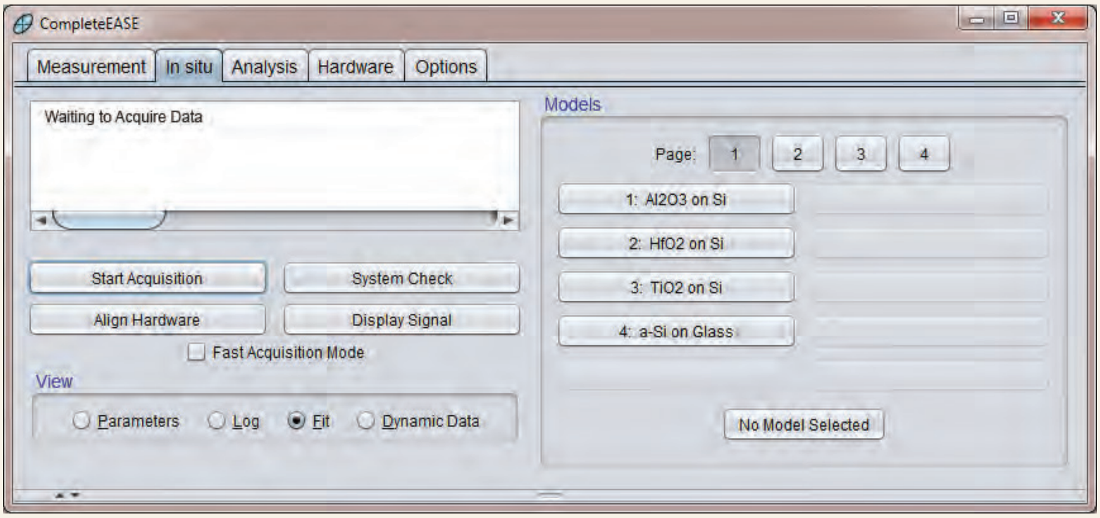
QCM-D

Heat Cells

CompleteEASE Software

CompleteEASE® software is the ultimate In Situ software package: from simple dynamic ellipsometry measurements to complex process control through integration with external program.

In Situ SE measurements can be synchronized with substrate rotation period to avoid substrate wobble effects. Uses patented method to correct for window effects from process chamber view ports. Multiple ellipsometric analysis models can be loaded in sequence. Results from an early stage of the process can be forwarded to analysis of a later stage and merged together.



Waiting to Acquire Data

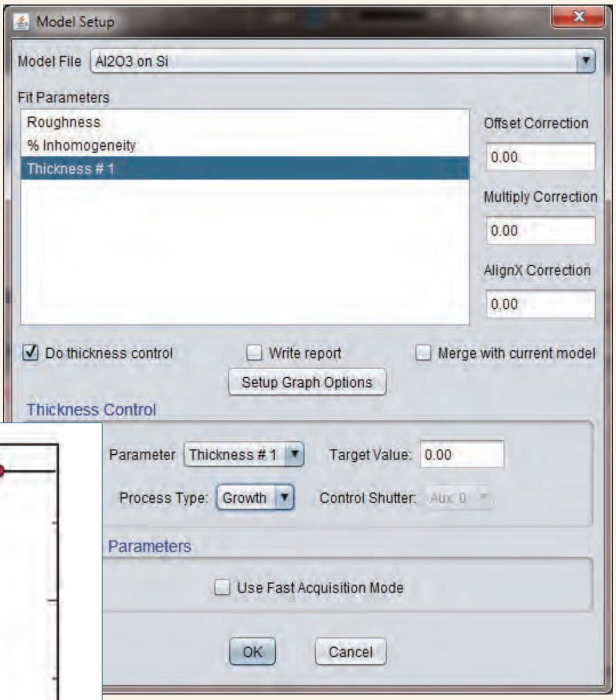
☐ Fast Acquisition Mode

○ Parameters ○ Log ● Fit ○ Dynamic Data

1: Al2O3 on Si
2: HfO2 on Si
3: TiO2 on Si
4: a-Si on Glass

CompleteEASE can be used in conjunction with external programs for process modification allowing feedback control of:

- Thickness Endpoint/Shutter
- Temperature or Composition through heater or sputter gun current
- Synchronization of a moving process



Model File: Al2O3 on Si

Offset Correction: 0.00

Multiply Correction: 0.00

AlignX Correction: 0.00

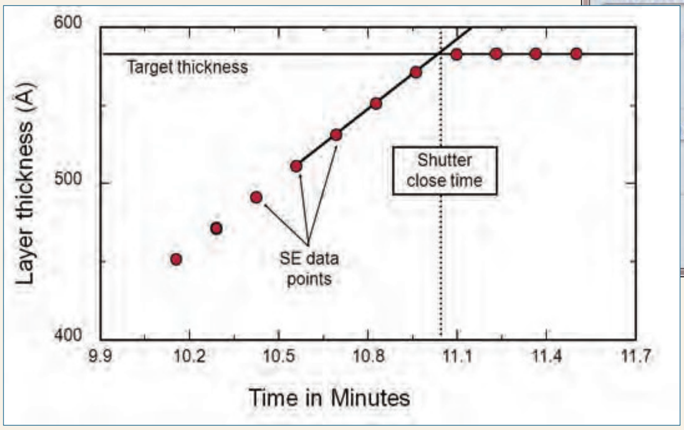
☑ Do thickness control ☐ Write report ☐ Merge with current model

Parameter: Thickness # 1 Target Value: 0.00

Process Type: Growth Control Shutter: Aux 0

☐ Use Fast Acquisition Mode

Parameters determined via CompleteEASE analysis of dynamic data can be used to trigger changes in process. ASCII-based command strings are sent back and forth between CompleteEASE and the external program for total control.

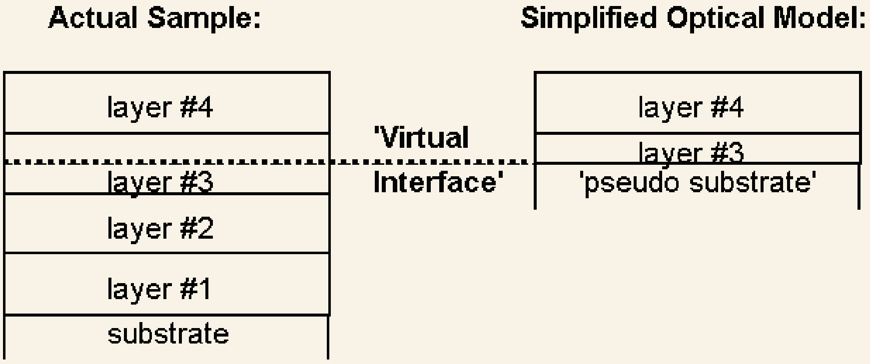


Example of real-time thickness control via control of shutter.

In Situ analysis requires powerful software with capabilities not generally needed for ex situ data. CompleteEASE includes several features essential for advanced in situ analysis.

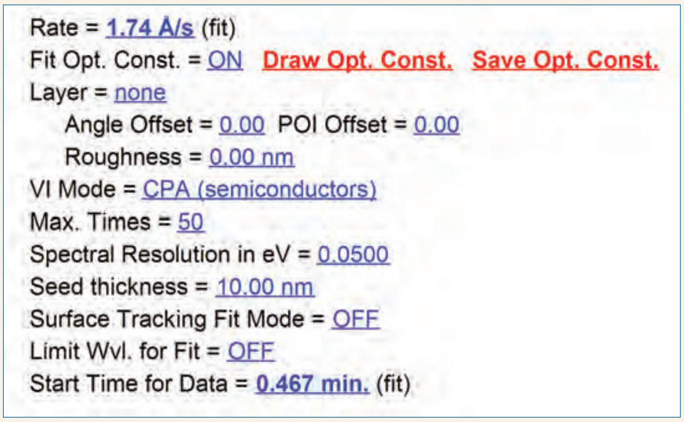
Virtual Interface

Sample structure below the layer of interest may not be known or difficult to model. Virtual Interface models the underlying structure as a single ‘pseudo substrate’, simplifying the optical model of a multi-layer sample.



GROC Model

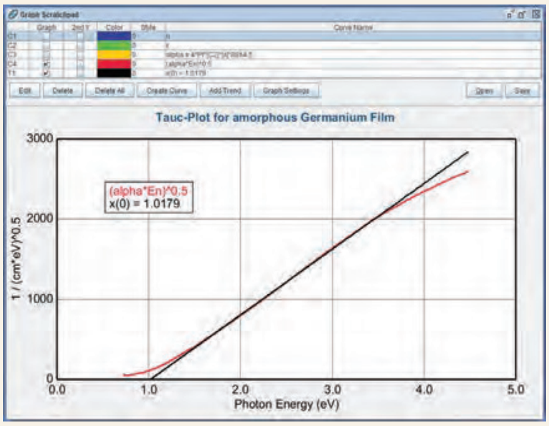
The Growth-Rate and Optical Constant (GROC) Model automatically determines growth rate & optical constants of a film by analyzing real-time data over a selected wavelength range, using a virtual interface to calculate the pseudo substrate from the first time-slice of data. The GROC model works well for high-index materials such as semiconductors, metals, and high-index dielectrics.

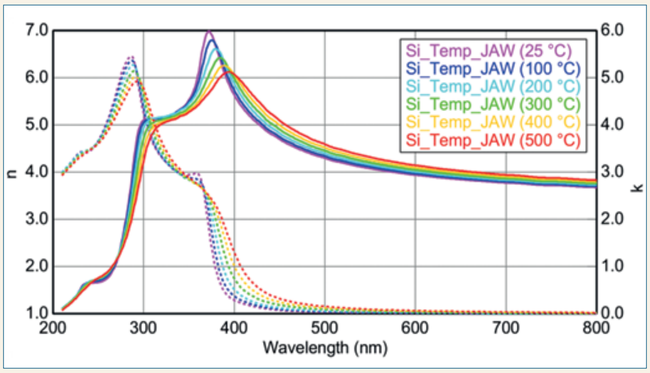


Rate = 1.74 A/s (fit)
Fit Opt. Const. = ON   Draw Opt. Const.   Save Opt. Const.
Layer = none
Angle Offset = 0.00   POI Offset = 0.00
Roughness = 0.00 nm
VI Mode = CPA (semiconductors)
Max. Times = 50
Spectral Resolution in eV = 0.0500
Seed thickness = 10.00 nm
Surface Tracking Fit Mode = OFF
Limit Wvl. for Fit = OFF
Start Time for Data = 0.467 min. (fit)

Graph Scratchpad

The Graph Scratchpad is useful for comparing optical constants, uniqueness tests, and dynamic fit results. Equation-based curves and trend lines can be added which allow users to extrapolate properties such as band gap or transition temperatures. The Graph Scratchpad can also be used simply for manipulating fonts, colors, and labels of plots to create presentation-worthy graphs of your results.



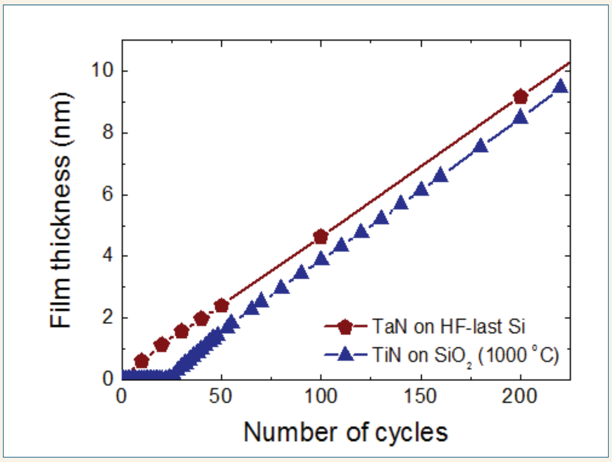


Build Temp/Comp Libraries

Users can create their own temperature or composition libraries for various materials. After fitting data for multiple temperatures or compositions, the results can be compiled into one material file, allowing optical constants for intermediate temperature or composition values to be easily extrapolated.

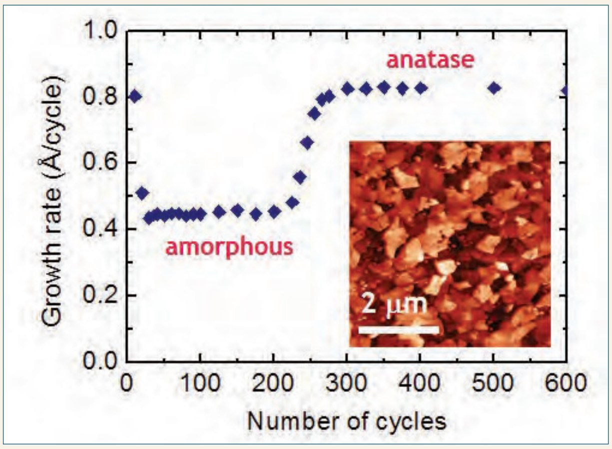
Atomic Layer Deposition

Atomic Layer Deposition (ALD) has demonstrated monolayer control for thin film growth. In Situ SE is the perfect tool to match monolayer sensitivity to this unique process capability. Significant advantages have been demonstrated through the combination of In Situ SE and ALD. Researchers at Eindhoven University have written an excellent review of their work.[1]

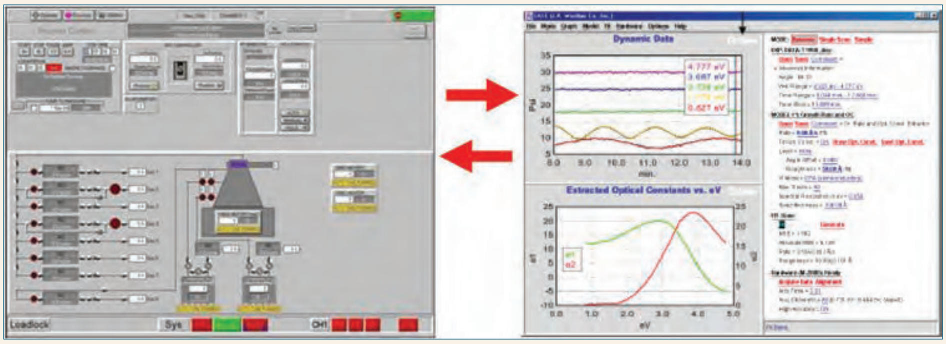


Film thickness from in situ SE for TaN and TiN show a nucleation delay for the TiN film.[1]

In Situ M-2000 spectroscopic ellipsometer mounted to an Oxford Instruments ALD chamber.



Interesting results from in situ SE during growth of TiO2 show the transition from amorphous to anatase crystal phase.[1]

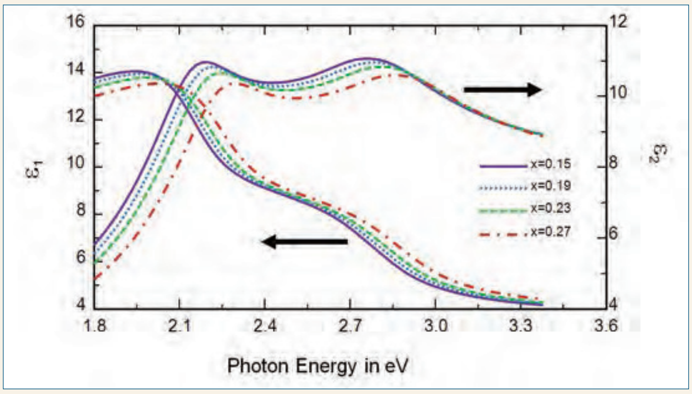


Communication between the Woollam CompleteEASE ellipsometry software and the ALD software allow real-time information to be shared about the process.

1. E. Langereis, et al. "In situ spectroscopic ellipsometry as a versatile tool for studying atomic layer deposition", J. Phys. D: Appl. Phys. 42 (2009) 073001 (19pp).

Compound Semiconductors

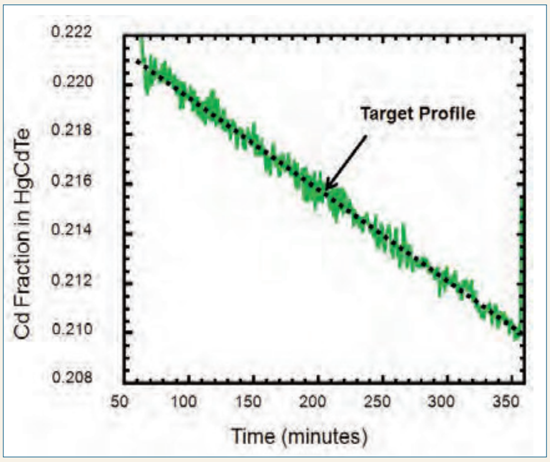
The semiconductor industry is able to use In Situ SE to characterize many material properties due to the natural link between these properties affect the material optical constants. In Situ SE is used to monitor temperature, composition, crystallinity, and more.



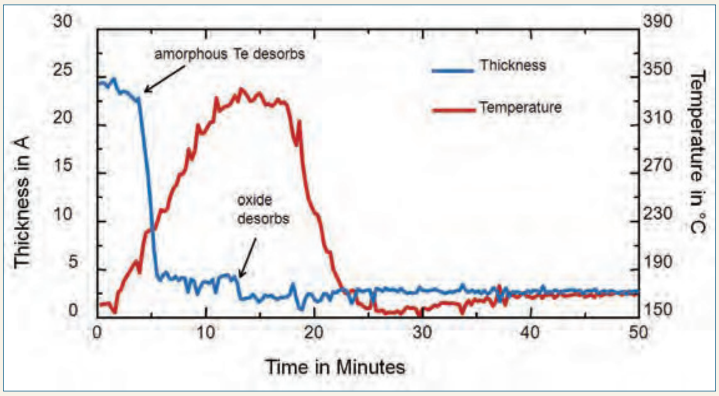
Shifting optical constants due to change in $Hg_{1-x}Cd_xTe$ composition

To get the most benefit from In Situ SE, libraries of each material are created which contain information about the material properties associated with different optical constants.

Library of information can be used to carefully control (in real-time) the composition for an intentionally graded device structure.



Composition profile of HgCDTe controlled real-time using In Situ SE.

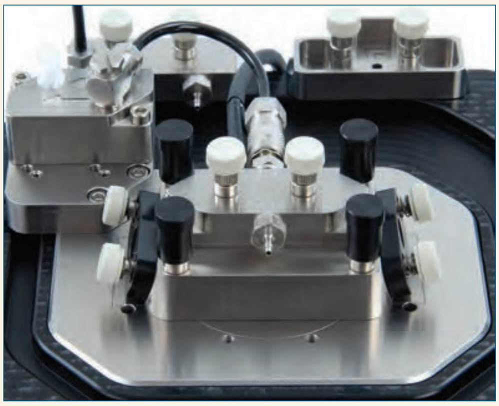


Surface temperature are monitored real-time with In Situ SE.

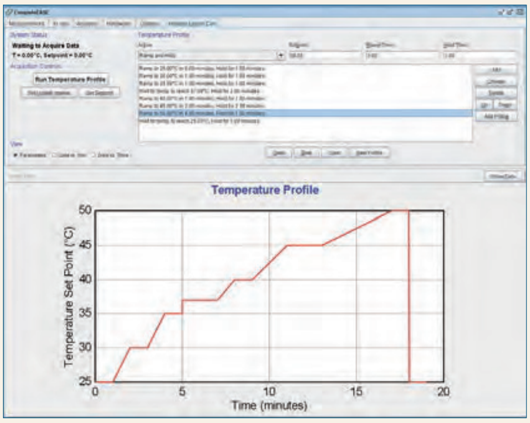
Optical constant changes also occur with temperature which allows the In Situ SE to measure the substrate temperature while surface layers are being removed.

BioChem Films

Liquid ambients, rough metal substrates, and ultra-thin self-assembled monolayers are common in biological experiments. In situ SE is used to characterize substrate surface, monolayer adhesion, reagent conditions, rinsing, etc.



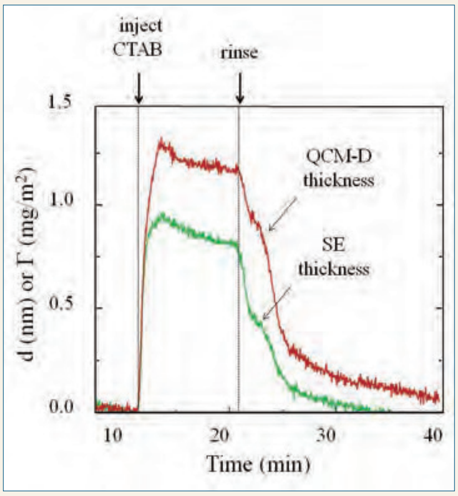
5mL Heated Liquid Cell



Waiting to Acquire Data
T = 0.00°C, Setpoint = 0.00°C

QCM-D

An interesting recent biochemical research area combines In Situ SE measurements with quartz crystal monitors (QCM). Each measures the thin film within a liquid as biomolecules attach to the surface. In Situ SE measurements are sensitive to the total density of attached molecules and do not detect the trapped water, as it shares the same index of refraction with the surrounding ambient. The QCM thickness measurement is related to total mass coupled to the surface. This can include trapped water with the polymer and thus may report a larger thickness than SE measurements. The combination of both measurements on the same surface have led to interesting conclusions about biomolecule dynamics.[2]



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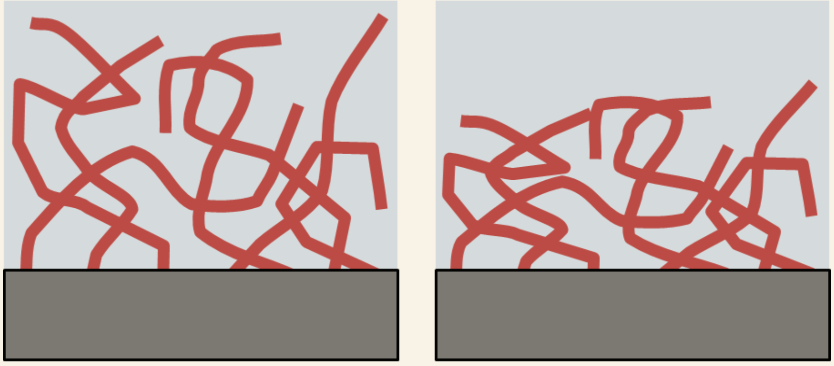
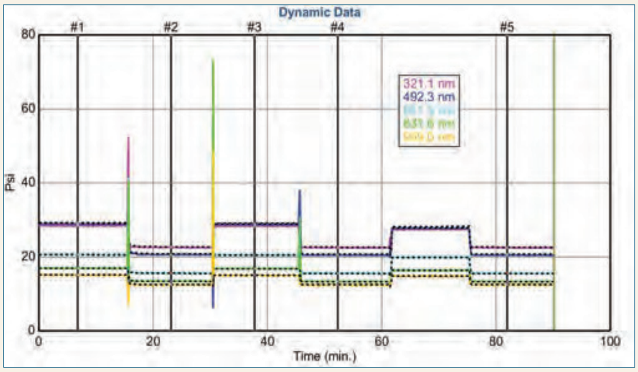
[2] Rodenhausen, K.B. and Schubert, M. “Virtual separation approach to study porous ultra-thin films by combined spectroscopic ellipsometry and quartz crystal microbalance methods”, Thin Solid Films, 519 9 (2011) 2772-2776.

Polymer Swelling

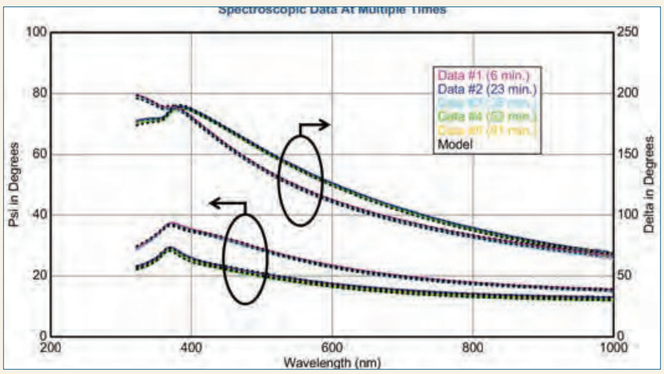
A polymer thin film was measured in aqueous ambient, where the pH was cycled through different values. This caused the polymer layer to swell and then compress. In situ SE measurement captured the details of this process.



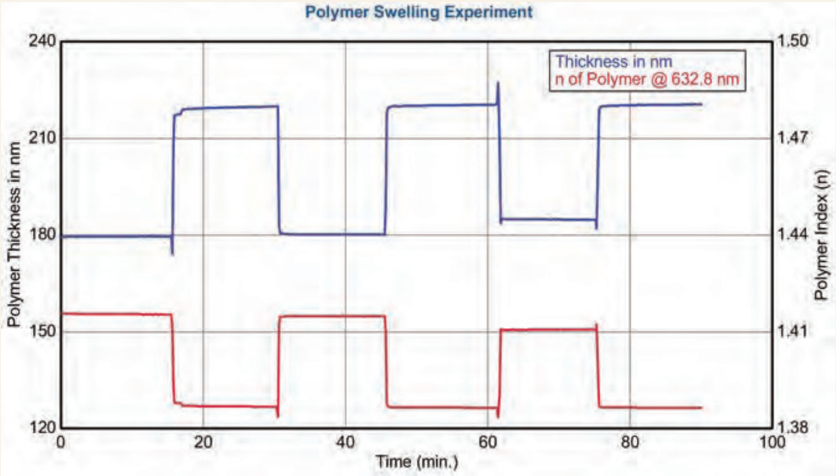
Polymer swelling (left) and compressed polymer (right)



Individual time-slices were used to build a model for the polymer film thickness and refractive index – even as it varies under different swelling conditions



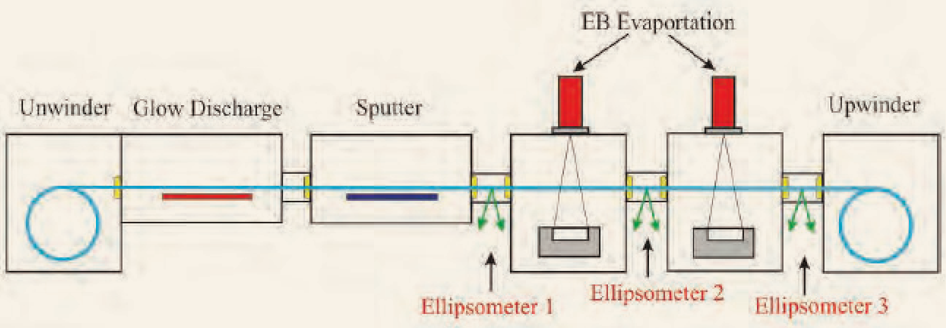
This model was used to characterize the thin film during the entire in situ process

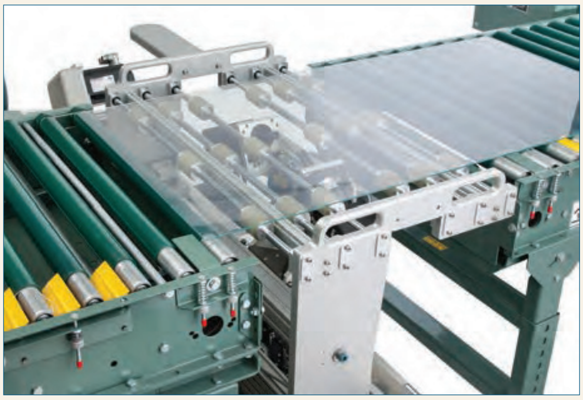


Film thickness and index are determined versus time, watching the film swell (increase thickness, decrease index) and compress (decrease thickness, increase index)

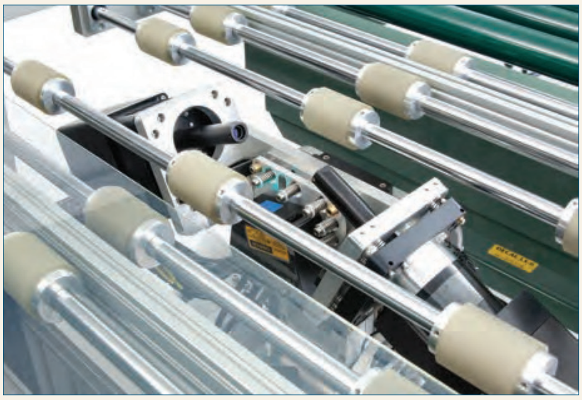
In-Line

Measurements can be made away from process chamber without breaking vacuum. Sample measurements are made in close proximity to processing – directly before or after. Multiple ellipsometers can be synchronized along the process path for multi-layer coatings. Monitor thickness of each layer along roll-to-roll processes.

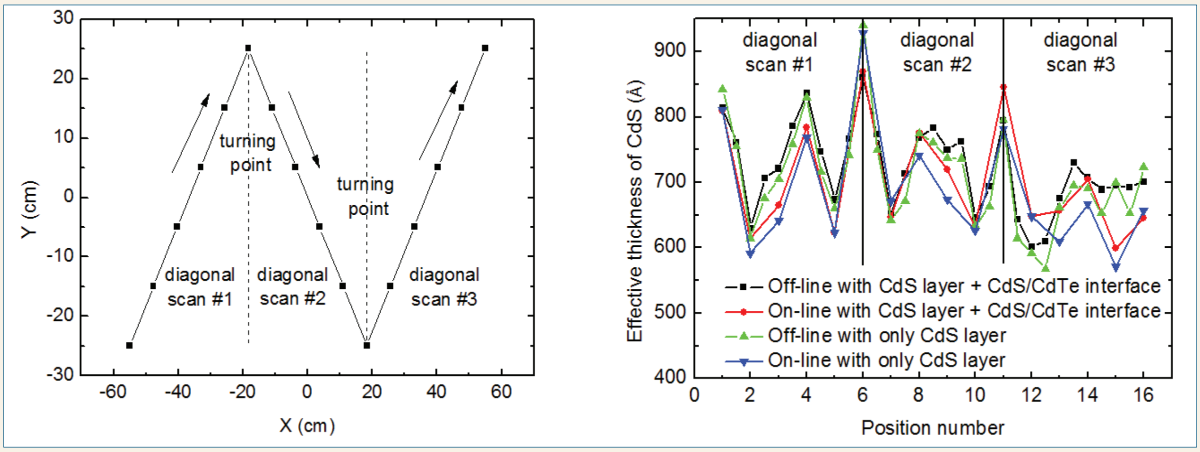




In-Line ellipsometer measuring a substrate on a glass panel.



Close-up of In-Line ellipsometer on a conveyor belt.

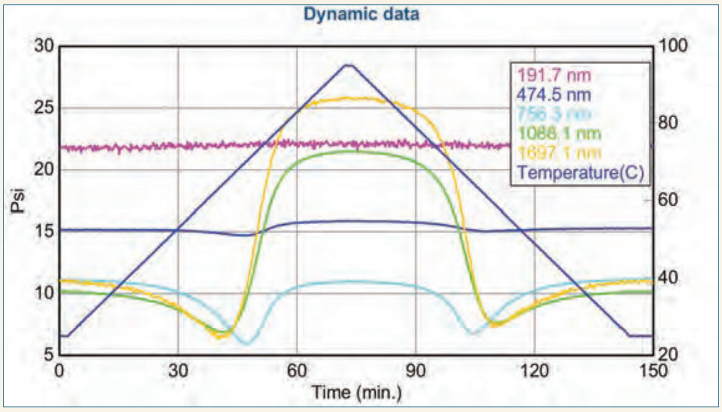


16 points on the panel are obtained at an average of ~ 3.7 s per point. A single panel, 120 cm in length moves by the In-Line SE in 1 min.[3]

3. Chenl, J., Koiralal, P., Salupol, C., Collins, R.W., Marsillac, S., Kormanyos, K.R., Johs, B.D., Hale, J.S., Pfeiffer, G.L. (2012) 'Through-the-Glass Optical Metrology for Mapping 60 cm x 120 cm CdTe Photovoltaic Panels in Off-Line and On-Line Configurations' Photovoltaic Specialists Conference (PVSC), 2012 38th IEEE, pp. 377-381.

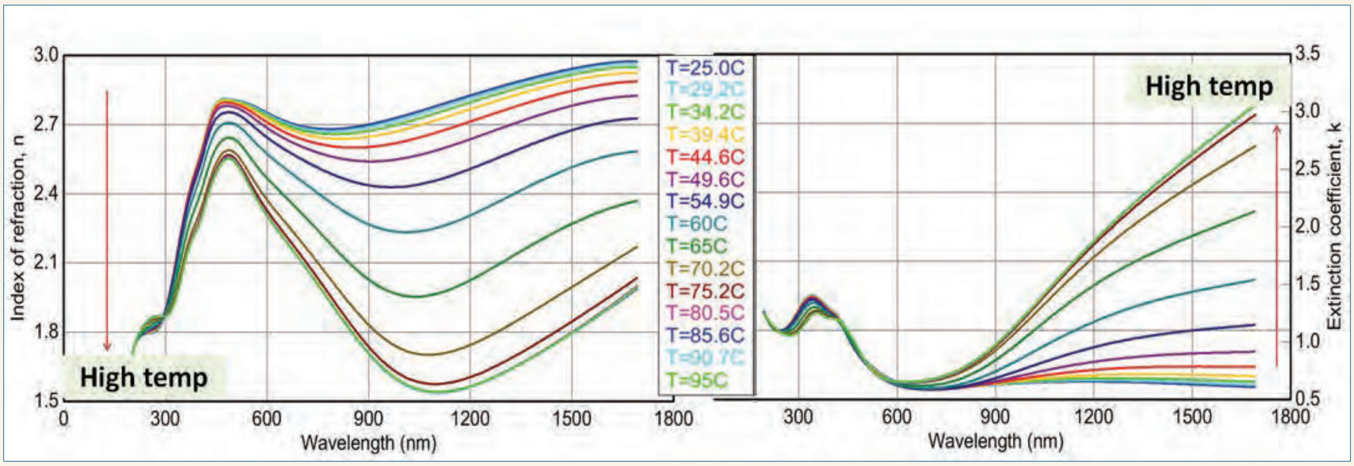
Temperature Studies

In Situ SE measurements versus sample temperature can provide interesting optical constant information. Materials such as vanadium oxide (VO_2) exhibit a remarkable transition between semiconducting and metallic phases. This transition is clearly seen in the dynamic In Situ SE measurement data.



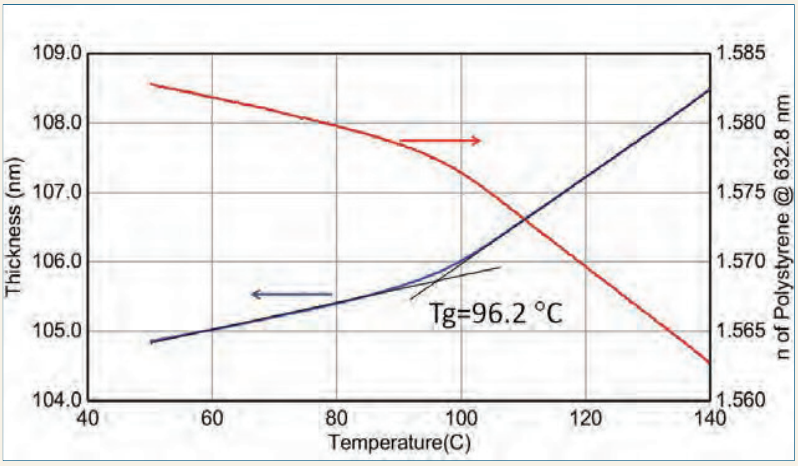
Dynamic data during temperature ramps show clear transitions as the phase of VO_2 changes.

From In Situ SE measurements at different temperatures, the large variation in optical constants is accurately determined.



Optical constant variation versus temperature during the VO_2 phase transition.

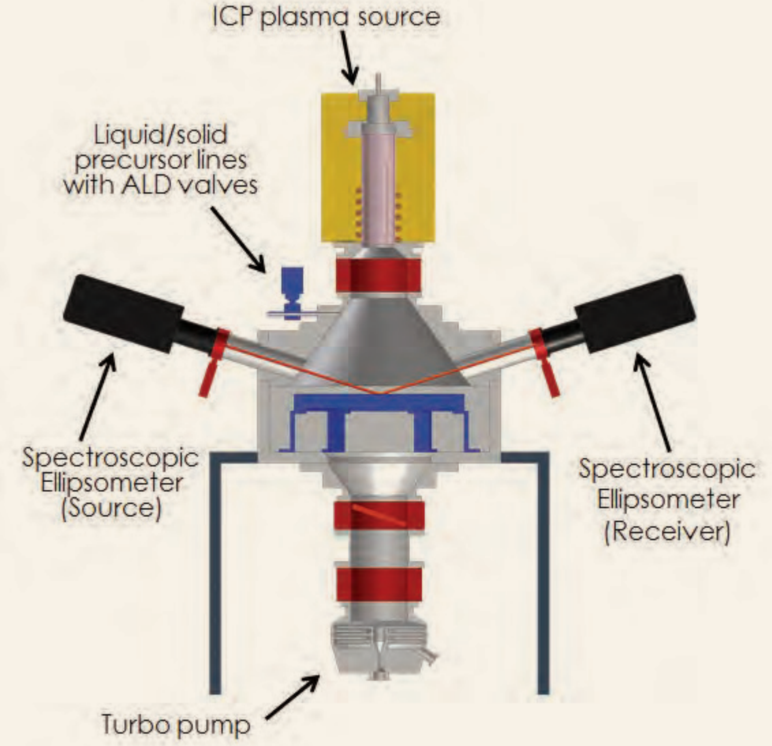
For organic films, a more subtle phase transition occurs – called the glass transition. As the temperature is heated or cooled, the organic material goes through a transition that is clearly identified when measuring the thickness and index versus temperature. This transition may vary with film properties and even with thickness, which makes In Situ a valuable tool for study.



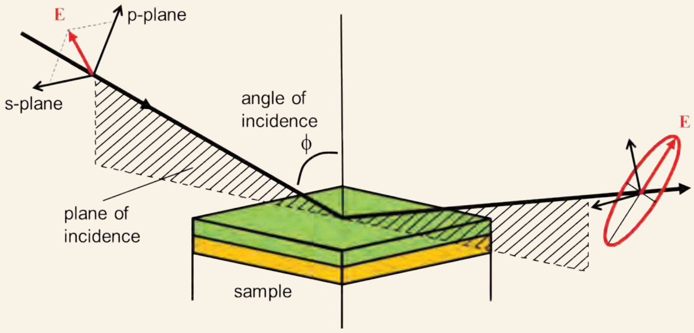
Change in rate for both index and thickness as temperature is ramped clearly identifies the glass transition temperature for an organic film

Cryostat mounted on RC2.

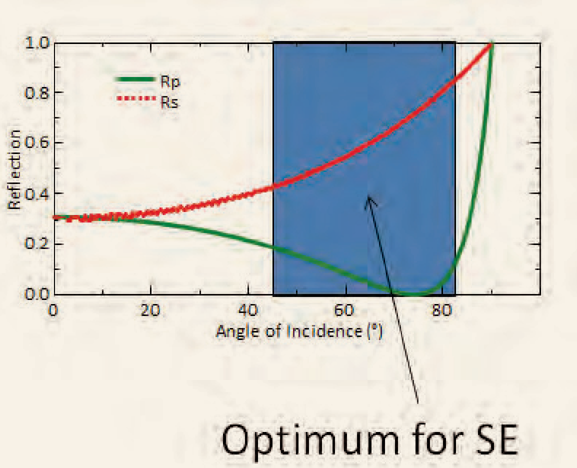
Hardware Integration



Example integration of In Situ SE on an ALD chamber[4]. Source and receiver heads are mounted to windows which allow access for the light beam to reflect from the sample surface at an oblique angle.



Plane of incidence for an SE measurement.



The ‘sweet’ spot for ellipsometry is generally at an angle of incidence between 50° and 80° - providing the best change in polarization.

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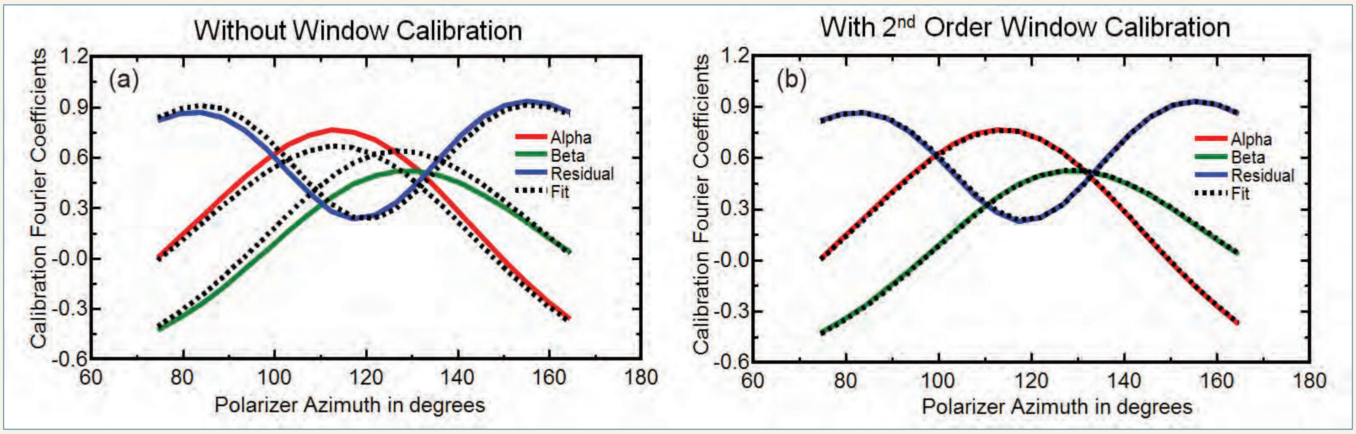
4. Langereis, E., Heil, S.B.S., Knoops, H.C.M., Keuning, W., van de Sanden, M.C.M., and Kessels, W.M.M. (2009) ‘In situ spectroscopic ellipsometry as a versatile tool for studying atomic layer deposition’, J. Phys. D: Appl. Phys., vol. 42, 073001 (19pp).

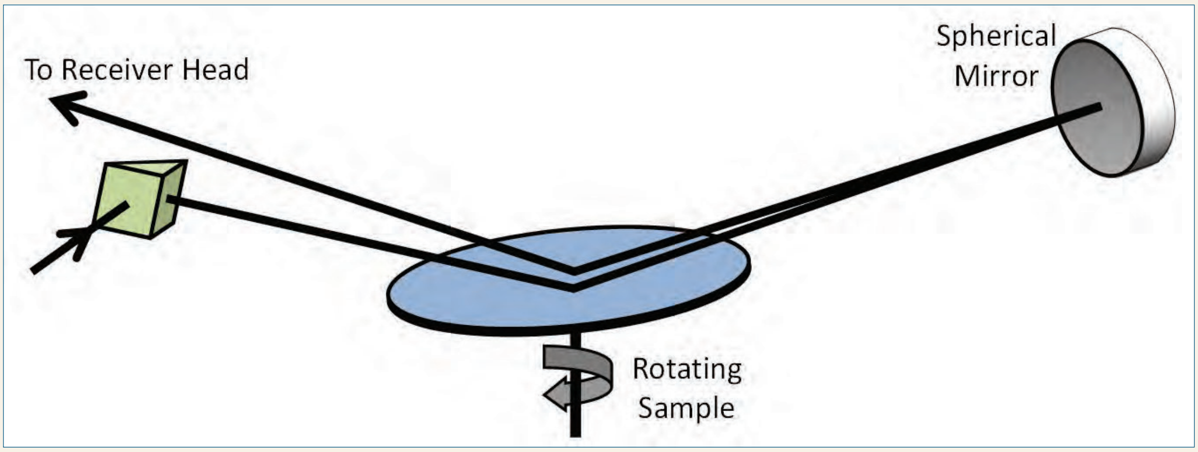
Advanced Technology

Windows provide the In Situ SE probe beam access to the sample. To improve measurement accuracy, we have developed a patented method to correct for any window birefringence.

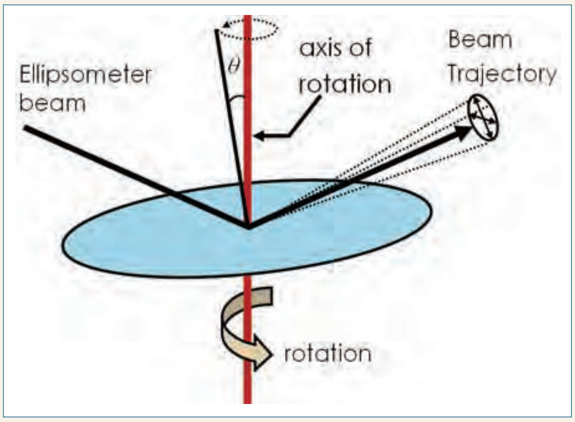


(a) Calibration through windows shows inaccuracy due to window retardation. (b) This is corrected by using our patented window calibration.

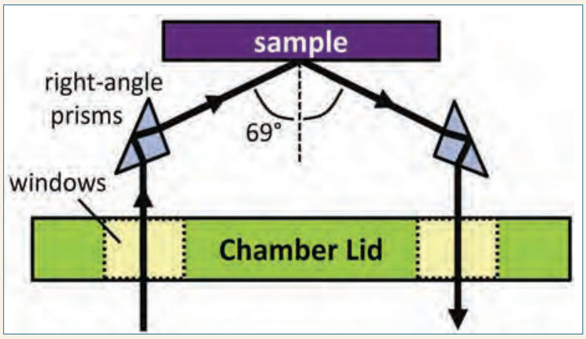
Sample rotation can lead to beam wobble which may reduce measurement accuracy. We have significant experience working with rotating samples. One method to minimize beam wobble incorporates reflection from a spherical mirror to compensate for any movement[5]. Other process chambers may require novel beam steering to incorporate the In Situ SE.



Wobble can be compensated through this unique approach.[5]



Sample rotation may lend to beam wobble.



Prisms and mirrors can be used for custom beam steering.

5. Johs, B.D., He, P., (2011) 'Substrate wobble compensation for in situ spectroscopic ellipsometry measurements', J. Vac. Sci. Technol. B, vol. 29 issue 3. (5pp)

In Situ Package

In-situ Package consists of:

- General ellipsometer, including:
  - Source unit with lamp housing, and polarization state generator (polarizer rotating compensator)
  - Receiver unit with polarization state detector (Polarizer, rotating compensator (RC2 only))
  - 19” rack-mount control boxes
    - -M-2000 (2)
    - -RC2 (1)
- Computer and Monitor
- CompleteEASE data acquisition and analysis software
- 1 set (2 pieces) flat fused silica UHV windows, 2.75” (CN 40) flange
- 1 set of in-situ mounts and stands to attach the in-situ tilt stages
- 1 set of in-situ tilt stages
- Alignment jig
- Ex-situ test base (65°, 90°-straight through)

RC2 In Situ Package (shown w/o computer)

Common In Situ References:

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