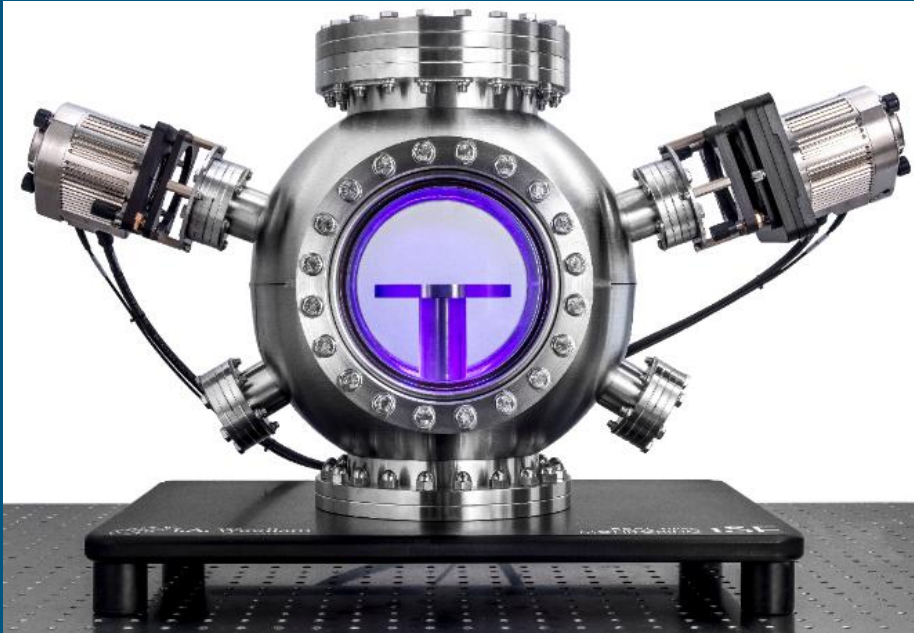




J.A. Woollam

Ellipsometry Solutions



Spectroscopic Ellipsometry for *In Situ* Applications

Greg Pribil
Applications Engineer

Harvard University, NNCI ALD/MOCVD/MBE Symposium – October 2019



Outline

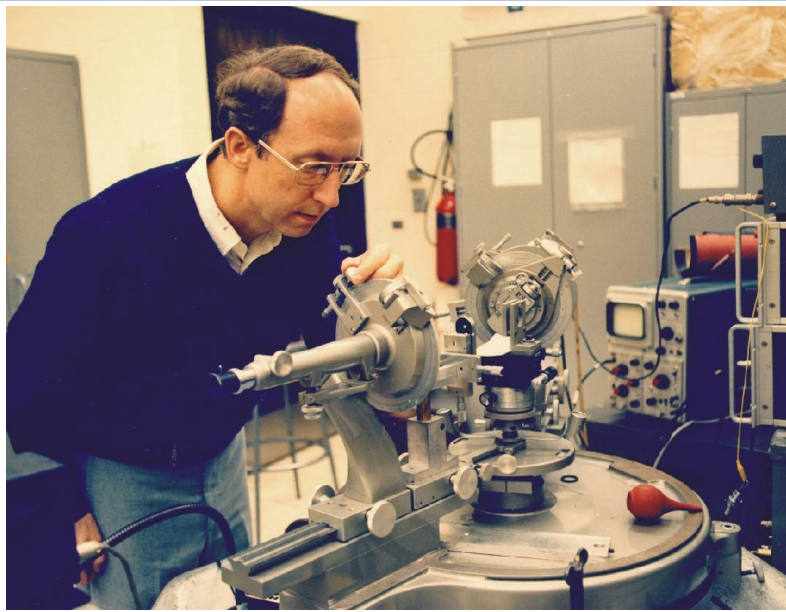
- Spectroscopic Ellipsometry (SE) Introduction
- In Situ SE Integration and Common Considerations
- In Situ SE Examples





J.A. Woollam Co. History

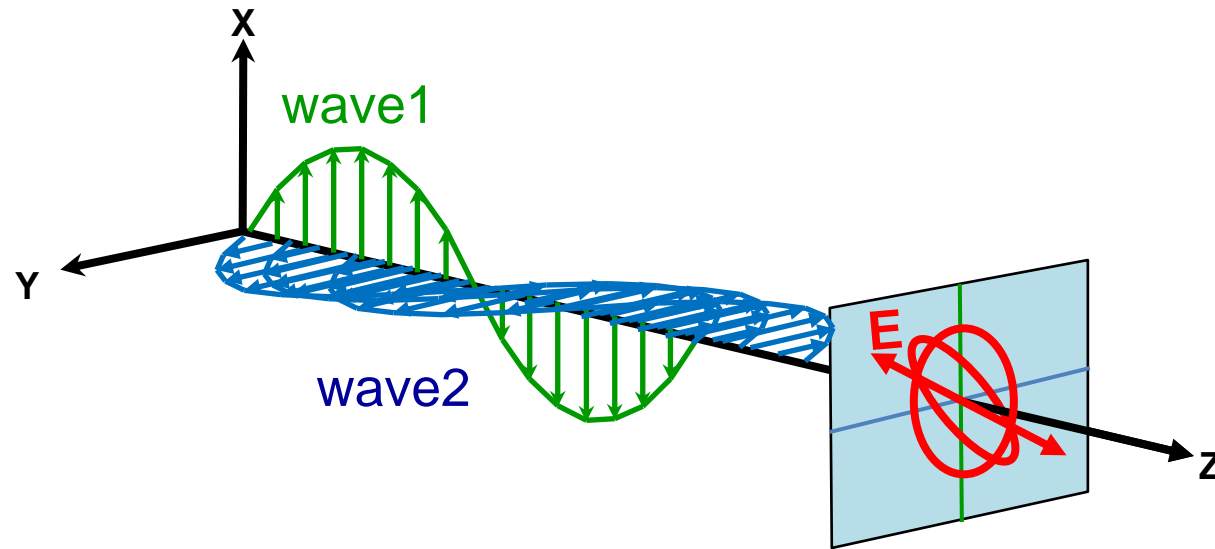
- Founded in 1987 by Prof. John A. Woollam based on spin-off of research at University of Nebraska-Lincoln.
- > 60 employees
- > 190 patents



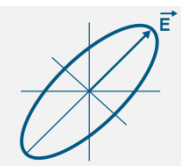


What is Polarization?

- Describes shape of Electric Field relative to direction of travel.



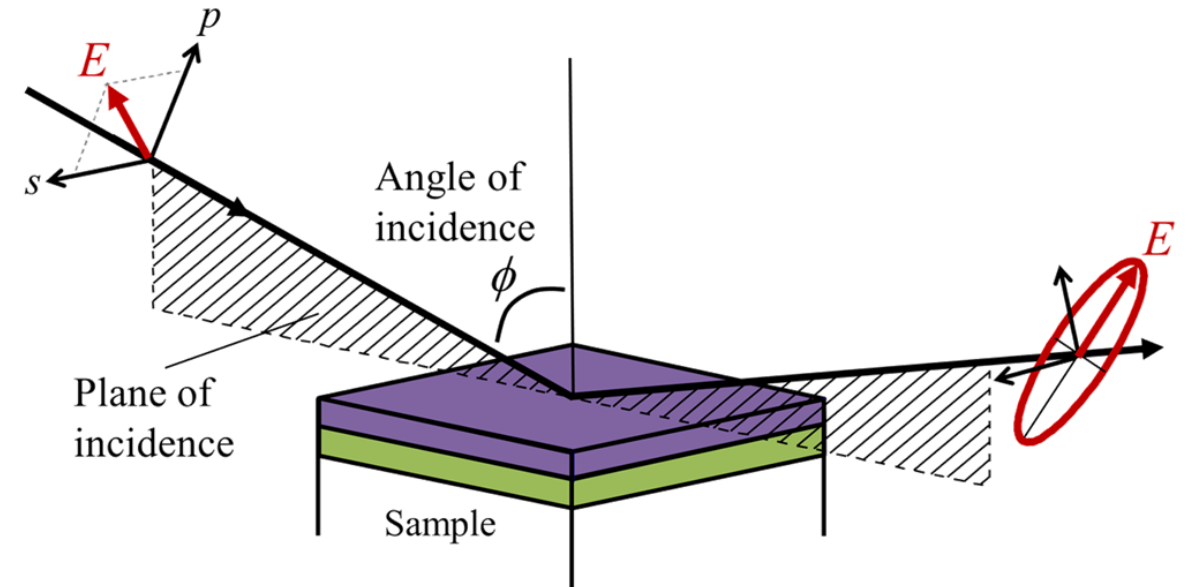
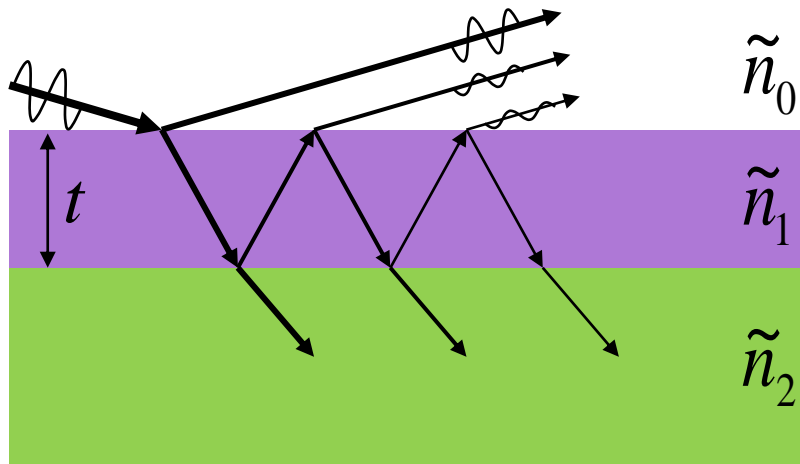
- Linear: arbitrary amplitudes, in-phase
- Circular: equal amplitudes, 90° phase difference
- Elliptical: arbitrary amplitudes, arbitrary phases



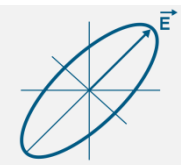
Spectroscopic Ellipsometry (SE)

- SE measures the change of polarization as light interacts with a thin film coated surface

$$\tilde{\rho} = \frac{\tilde{r}_p}{\tilde{r}_s} = \frac{|\tilde{r}_p|}{|\tilde{r}_s|} e^{i(\delta_p - \delta_s)} = \tan \Psi e^{i\Delta}$$



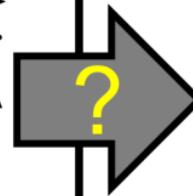
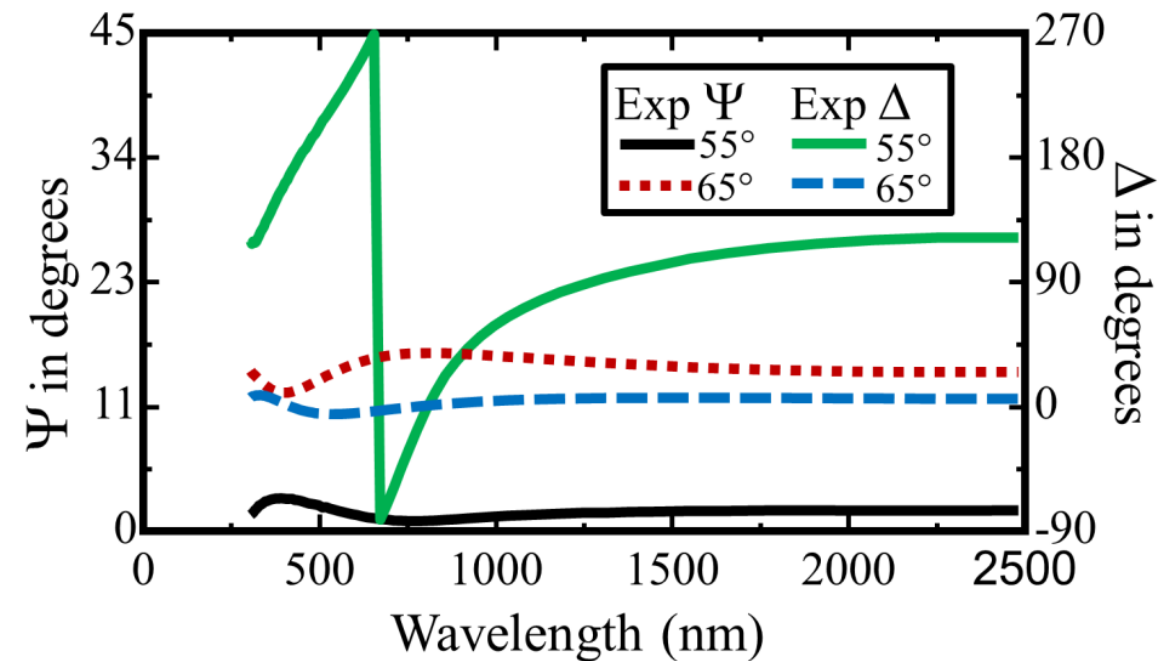
- Coherent interference occurs when light recombines after traveling different paths through thin film



What Can SE Determine?

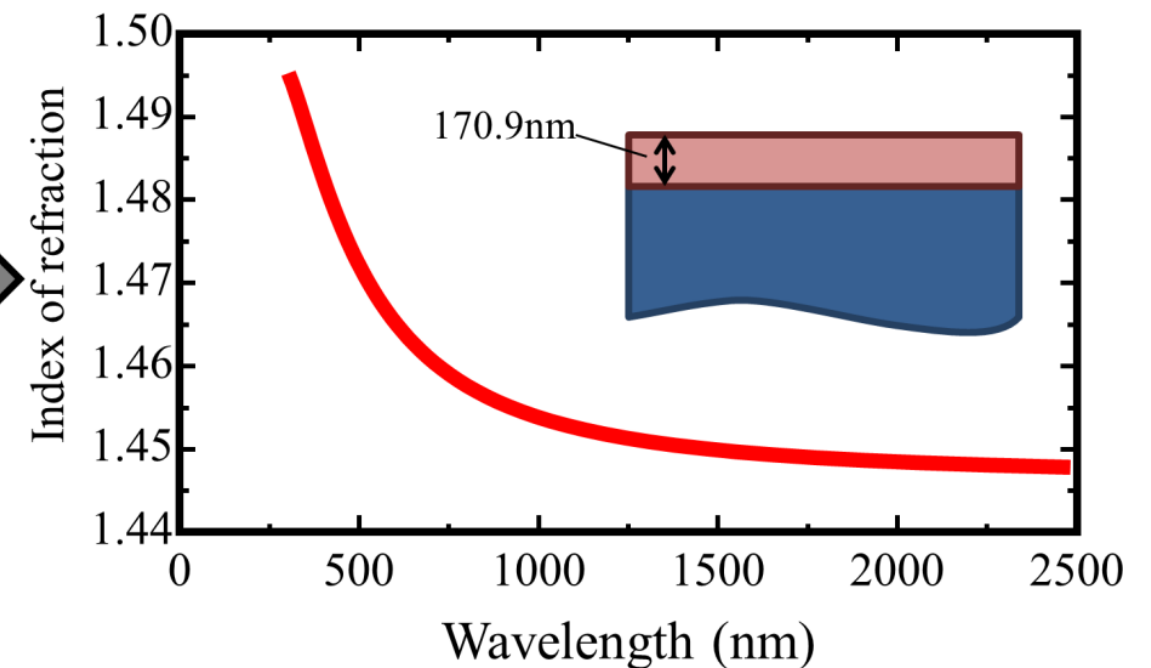
Experimental Data

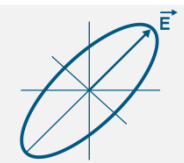
Psi, Delta



Sample properties

Film thickness, Optical constants
Surface roughness, Composition, Crystallinity...

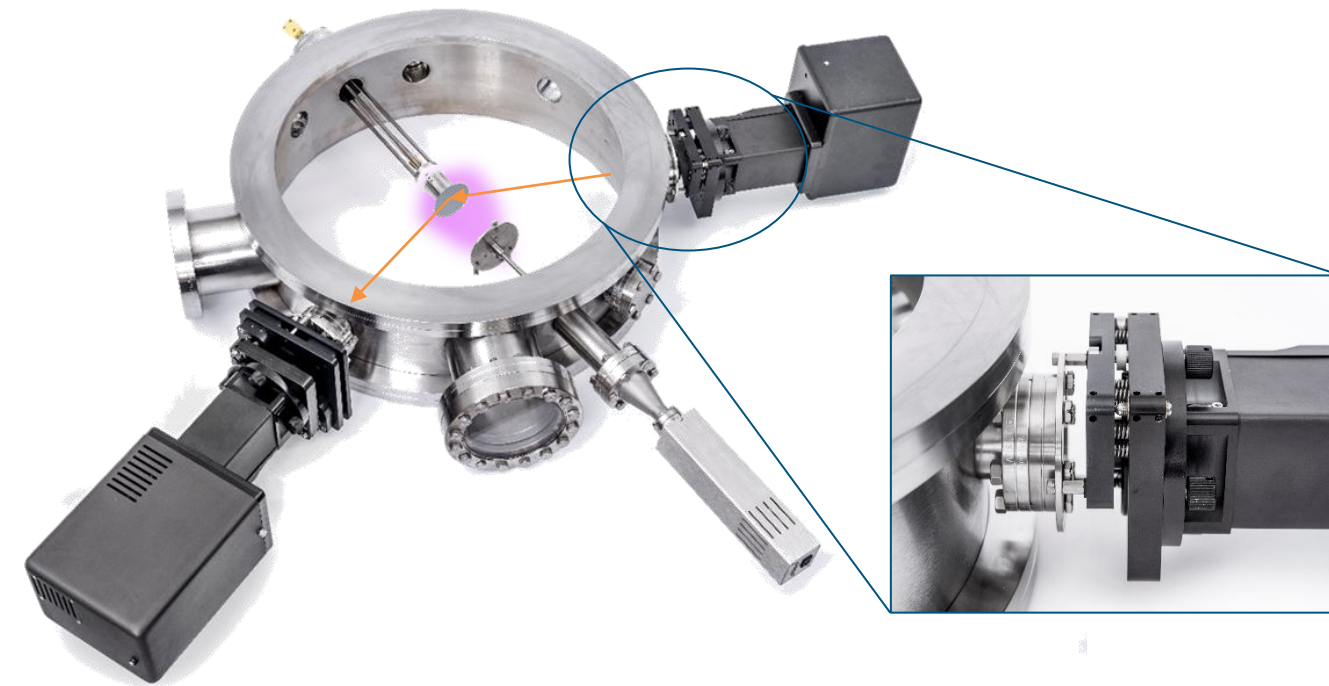
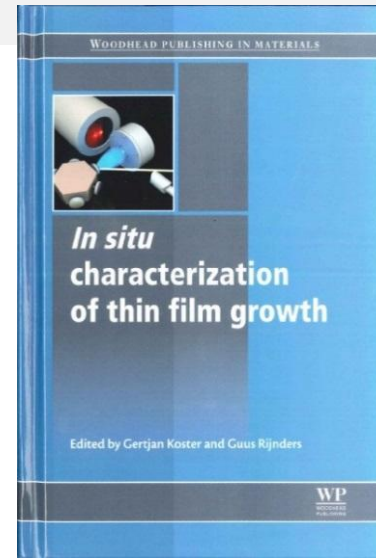




In Situ Ellipsometry

- Real-time monitoring and control
- Complete sample history
 - Before, during and after process
- Direct measurement of surfaces & interfaces

J.N. Hilfiker "In situ spectroscopic ellipsometry (SE) for characterization of thin film growth", chapter in *In situ characterization of thin film growth*, G. Koster and G. Rijnders, eds., Woodhead Publishing: Cambridge, UK (2011)

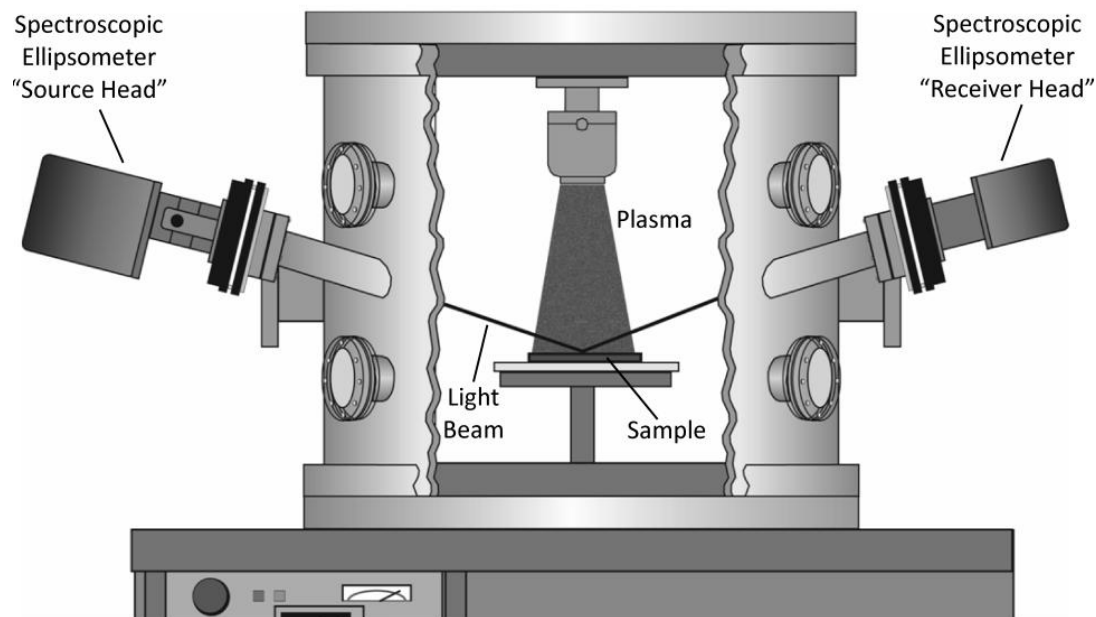


**Atomic Layer
Deposition**



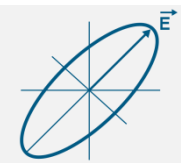
In Situ Spectroscopic Ellipsometry

- Source and Receiver attach to standard window flanges on process chamber for non-invasive, non-contact measurements.



- CCD detection: Possible to measure >1000 wavelengths from UV to NIR – collected multiple times each second.
- High Sensitivity to Thin Film properties: Thicknesses from <1nm to 10+ microns.
- Measurement is ratio, so accurate with fluctuating intensity due to window coating, moving sample, etc.

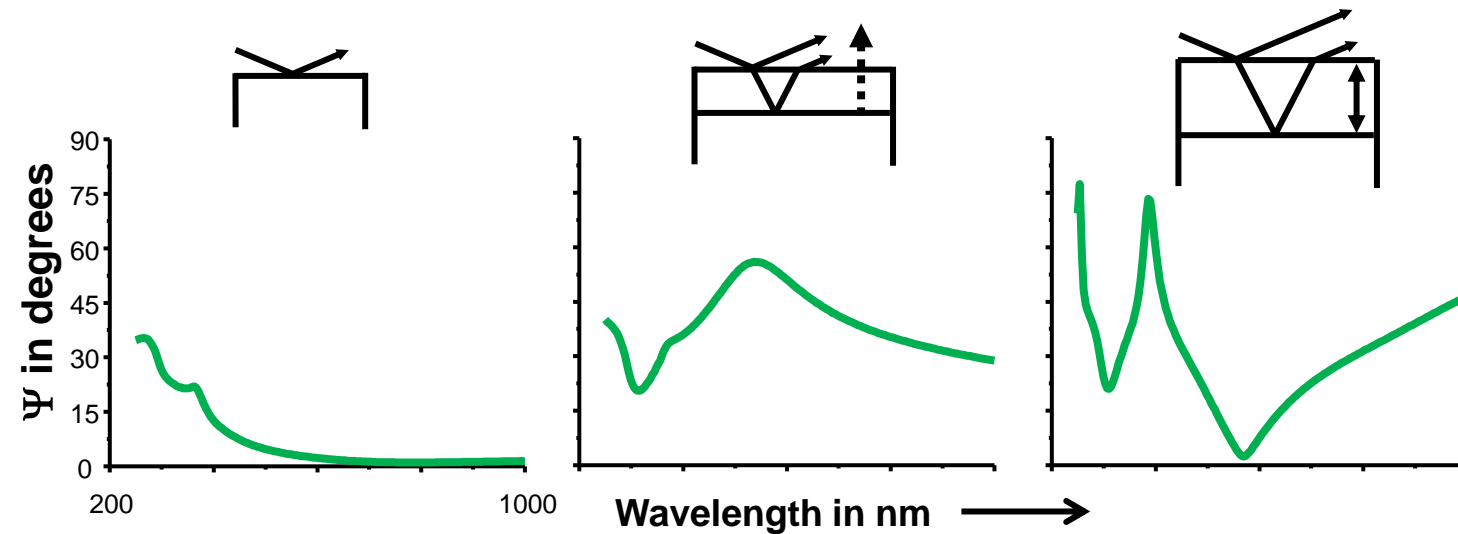
$$\frac{\tilde{E}_p}{\tilde{E}_s}$$



In Situ Spectroscopic Ellipsometry Data

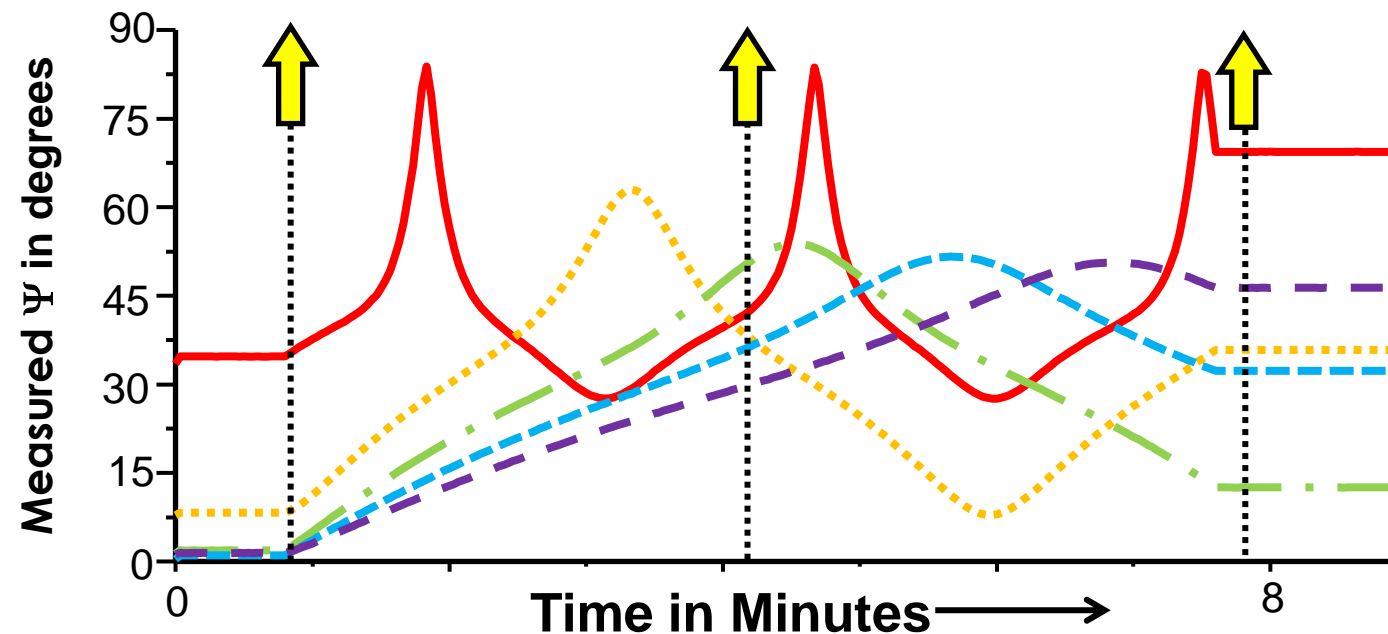
Spectral Data

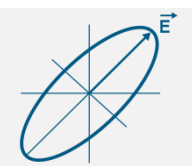
study sample
before, during, &
after film growth



Dynamic SE Data

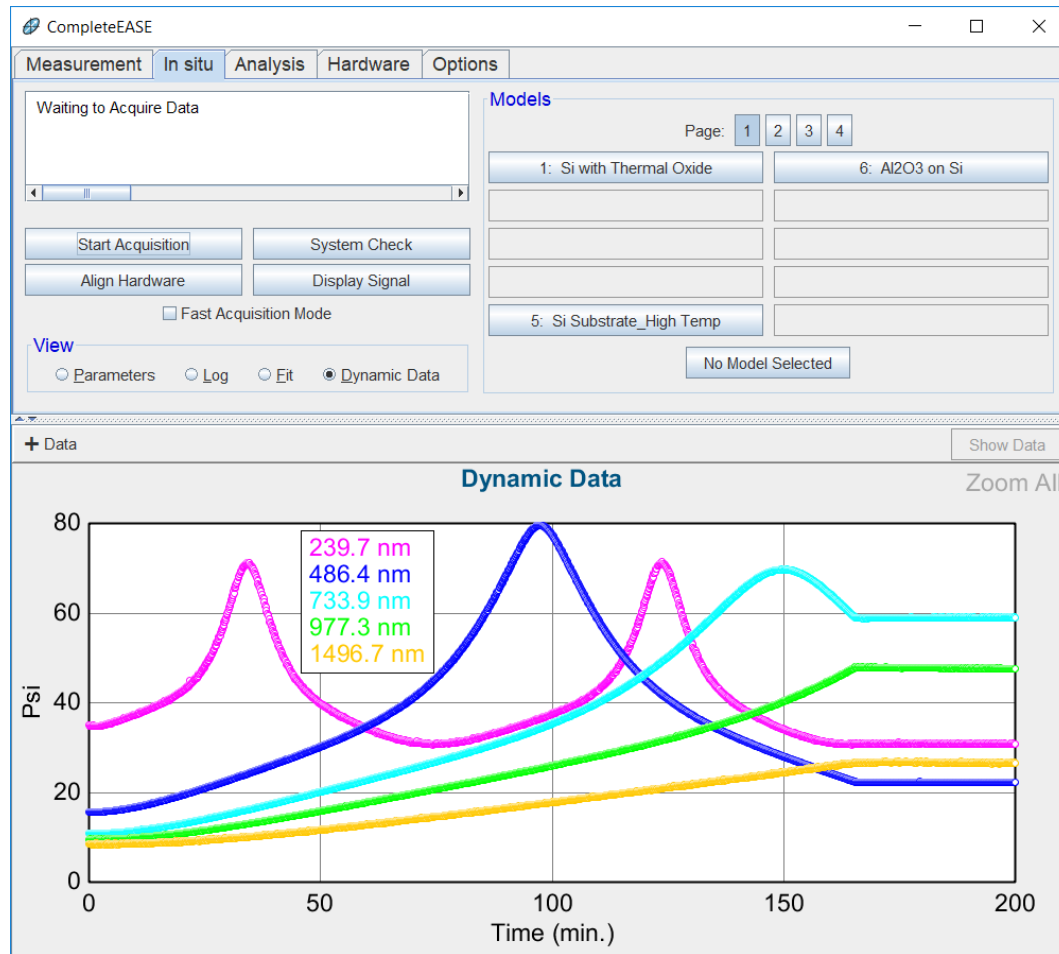
contains
continuous
Spectral Data



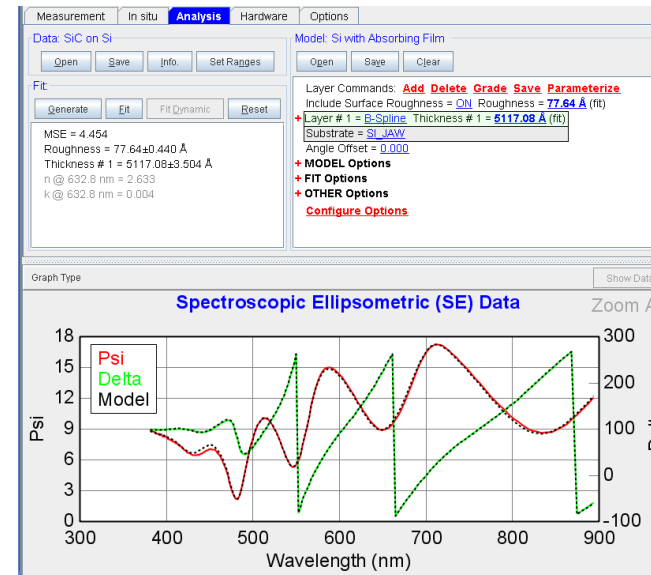


CompleteEASE User Interface

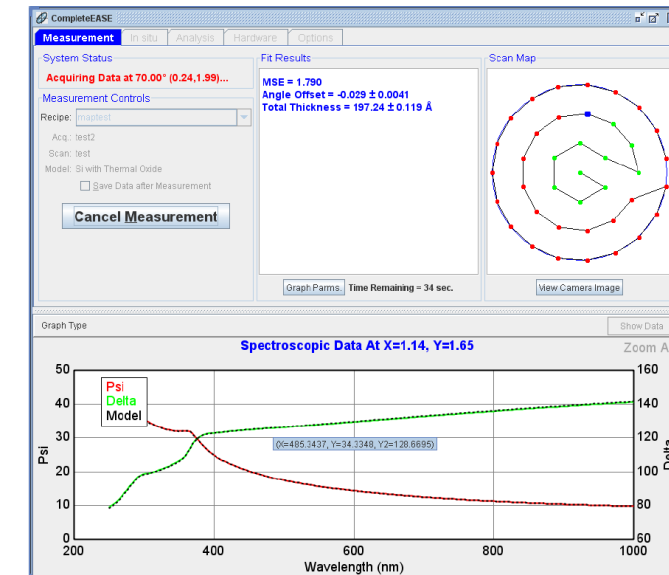
■ In situ Tab:



■ Analysis Tab:

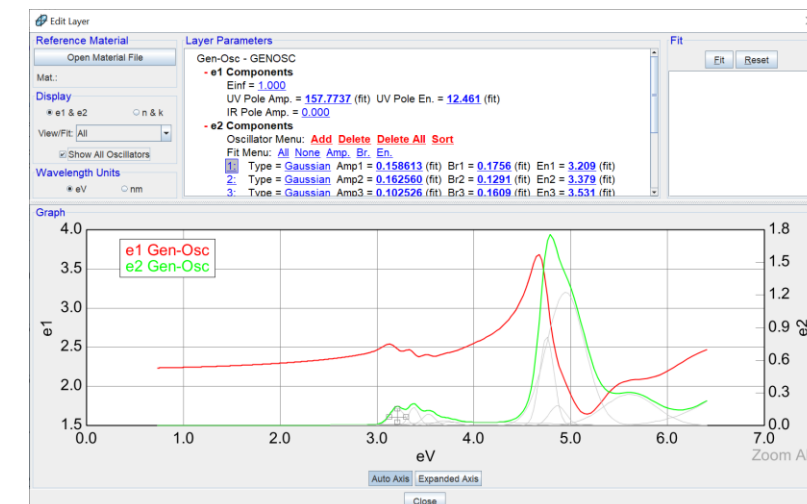


■ Measurement Tab:

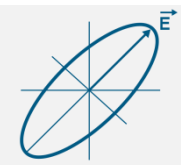


■ Optical Models:

- Gen-Osc
- B-Spline
- Cauchy
- ...

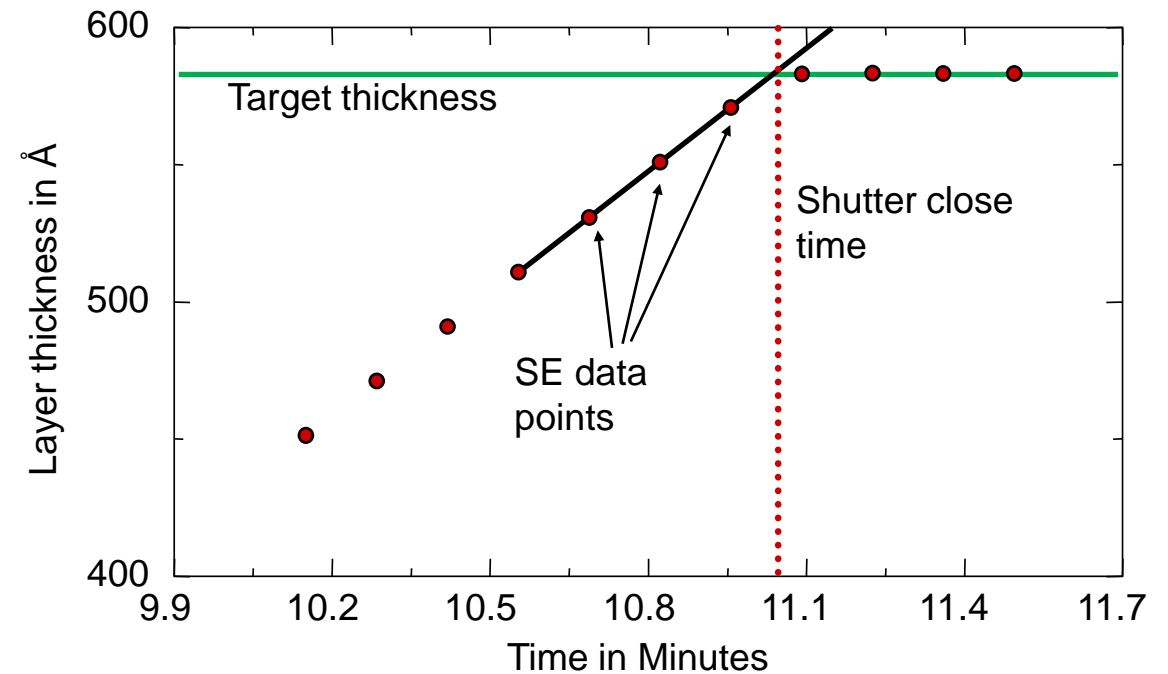
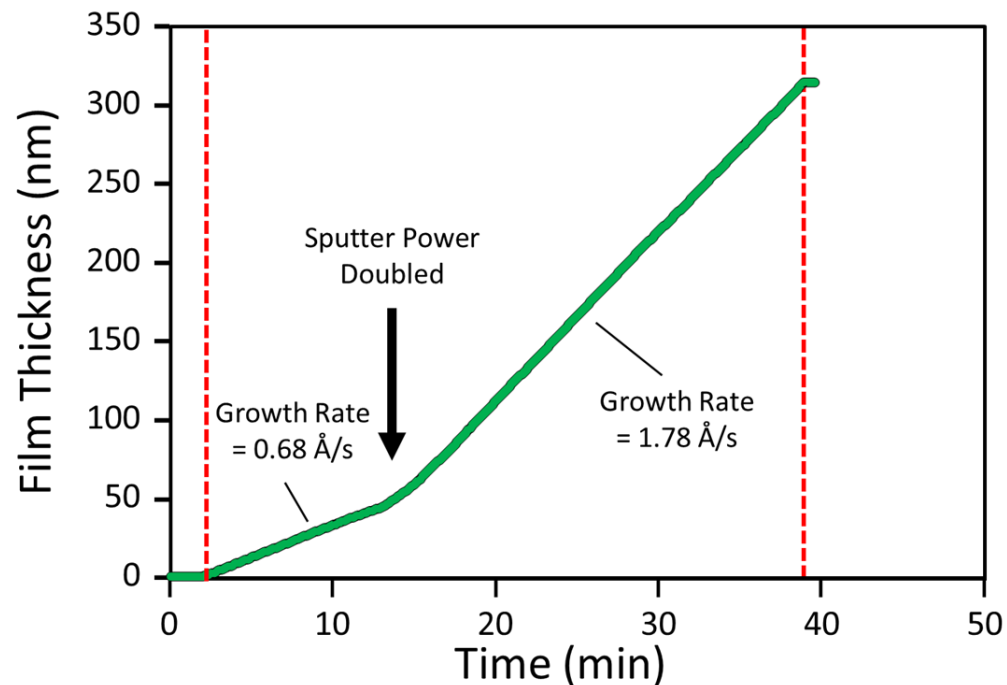


■ “Custom” tabs (e.g., Heat Cell)



Application: Thickness and Growth Rate

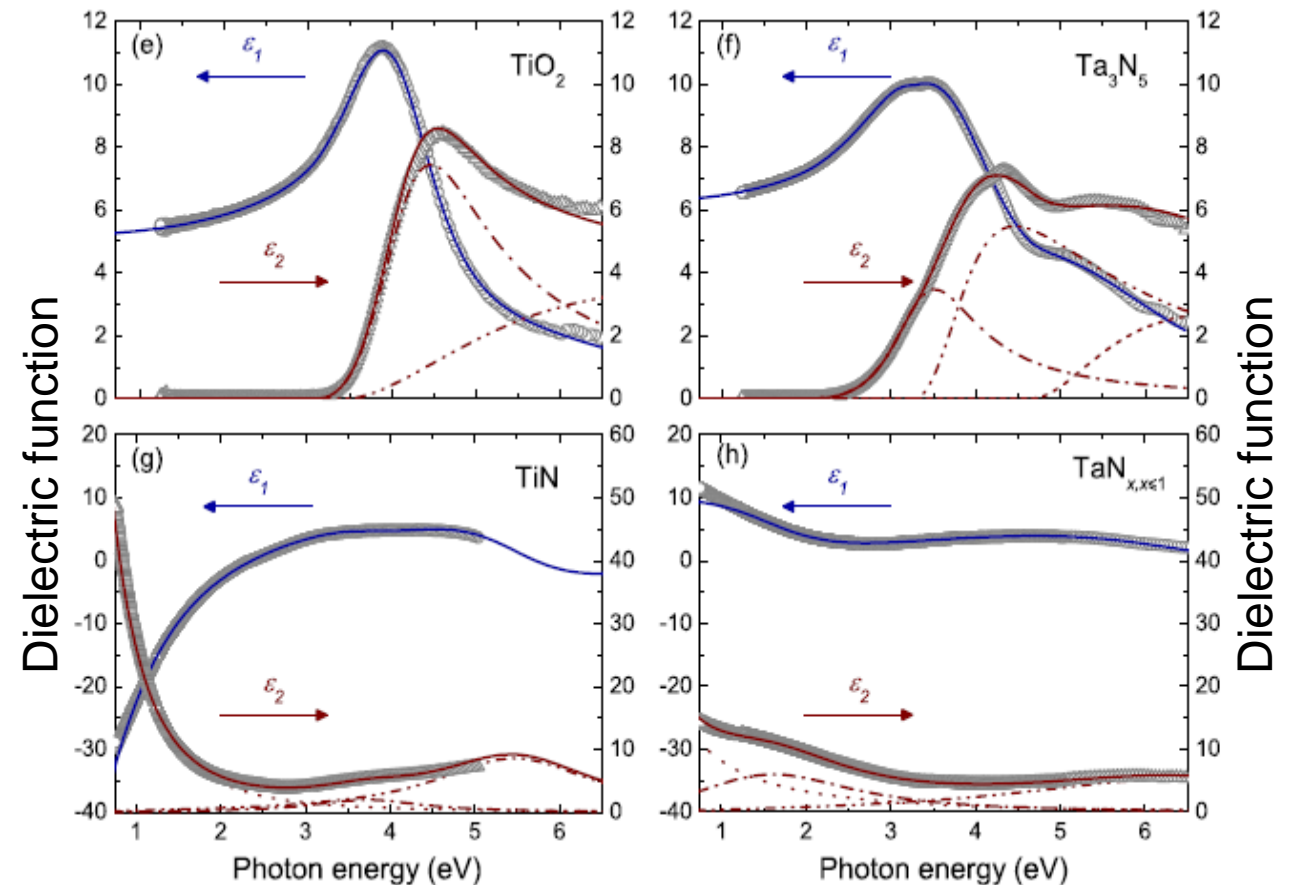
- Most common in situ SE application: Real-Time film thickness.
- Film thickness and growth rate shown for sputter deposition of SiO_2 thin film.
- Thickness control algorithm for precise process control.
- Controlled growth shown for a 583\AA GaAs thin film.



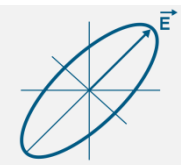


Advantages of Spectroscopic Ellipsometers

- **Wide spectral range**
 - Near IR range for conductive film
 - UV range for semiconductors and dielectrics
- **No gaps in spectrum**
 - Interference enhancement to study thin metal films
- **Software with extensive modeling options for most-general samples**
 - General Oscillator models
 - Drude for conductivity
 - Tauc-Lorentz for amorphous materials
 - Parameterized semiconductor models
 -
 - B-spline parameterizations

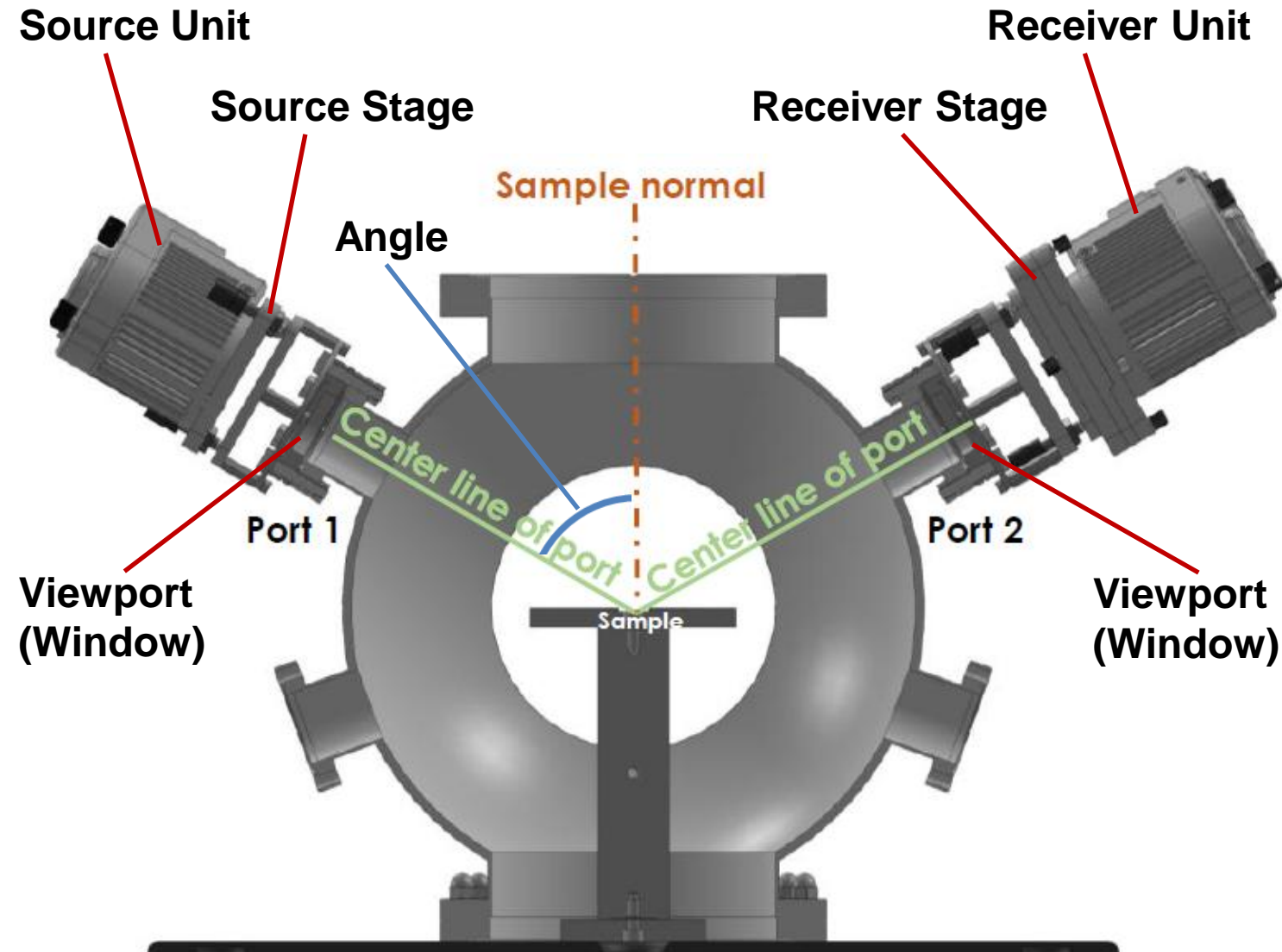
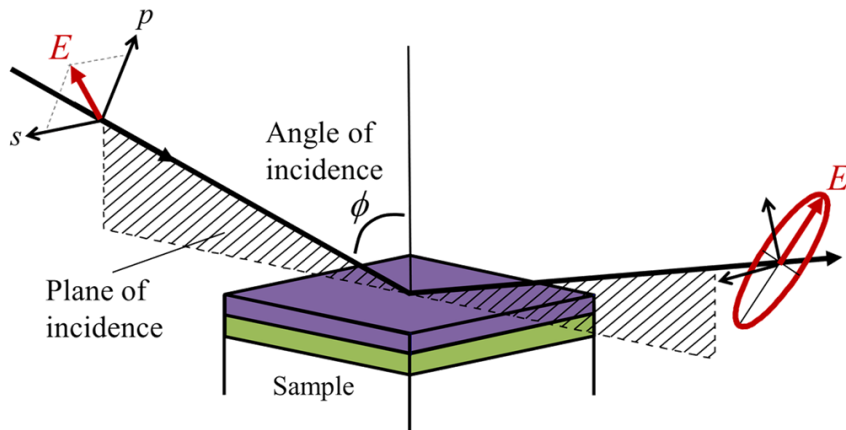


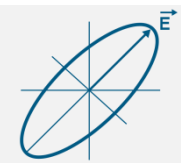
Langereis et al., *J. Phys. D: Appl. Phys.* **42**, 073001 (2009)



Chamber Integration

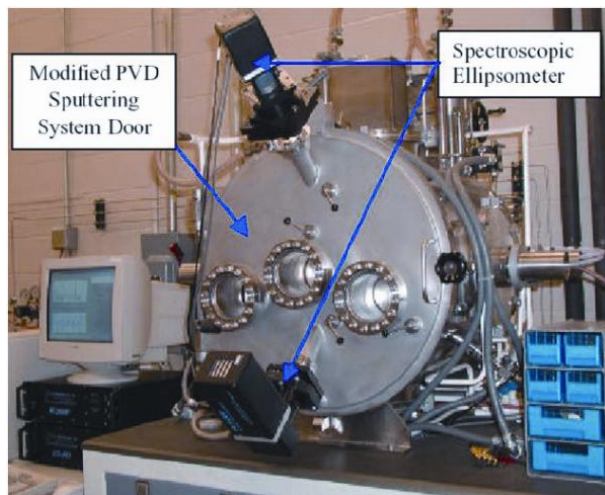
- Requires optical access to the Sample during measurement.
- SE measurements can be continuous, periodic, or triggered.
- SE measurement angle defined from sample normal to ellipsometer viewport.
 - Source Angle = Receiver Angle
 - Typical “default” port angle $\sim 70^\circ$, with most chambers designed for port angle between 60° to 75° .



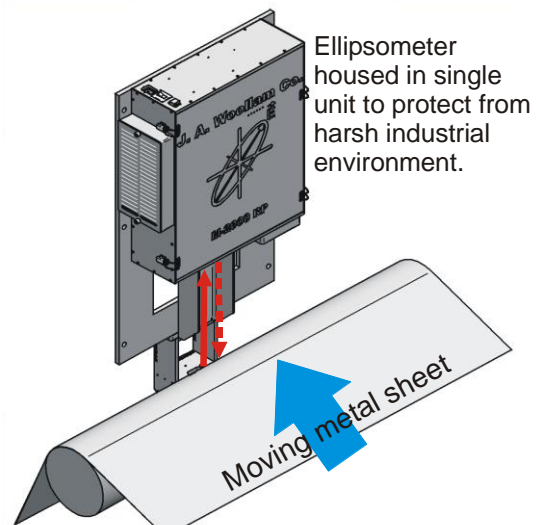


Examples: In Situ/In-Line SE Implementations

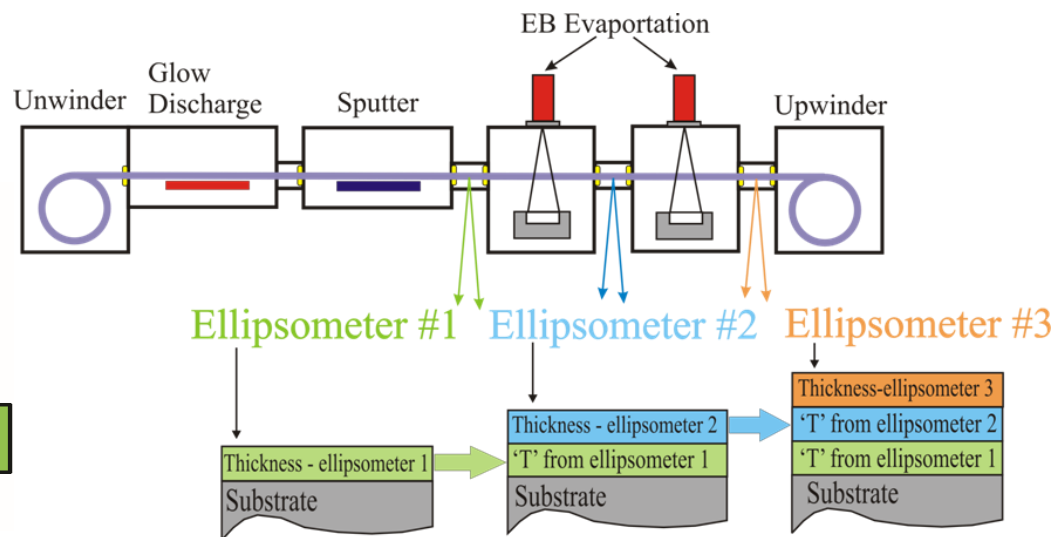
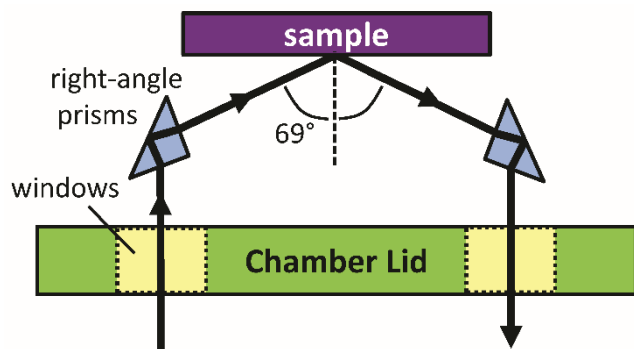
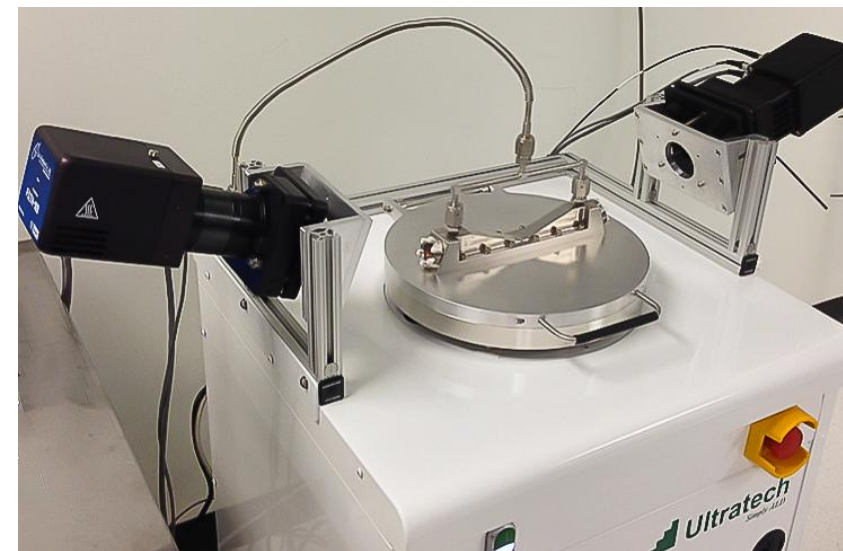
Door mounted



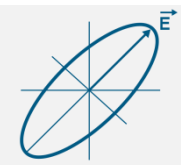
Metal Roll-to-Roll



Atomic Layer Deposition

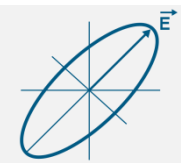


Conveyor belt




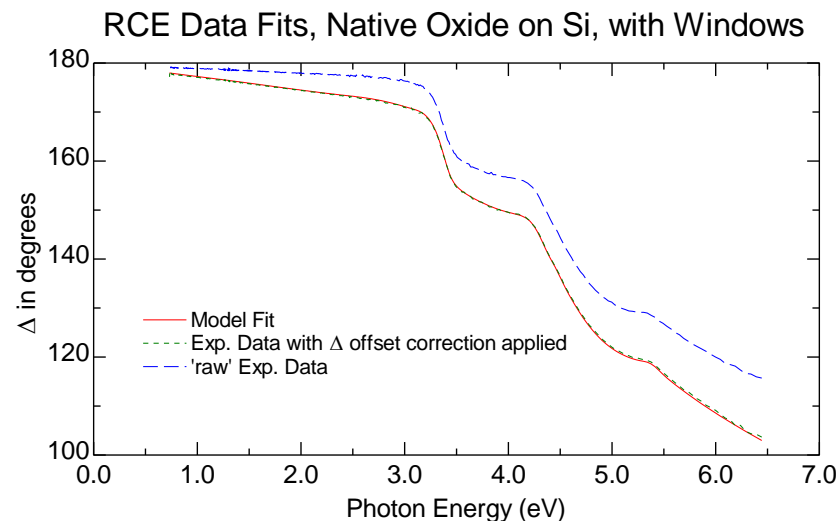
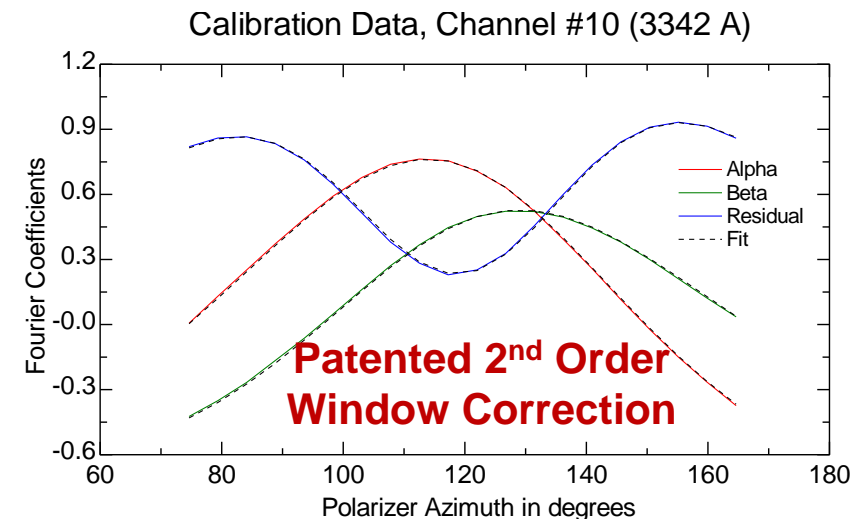
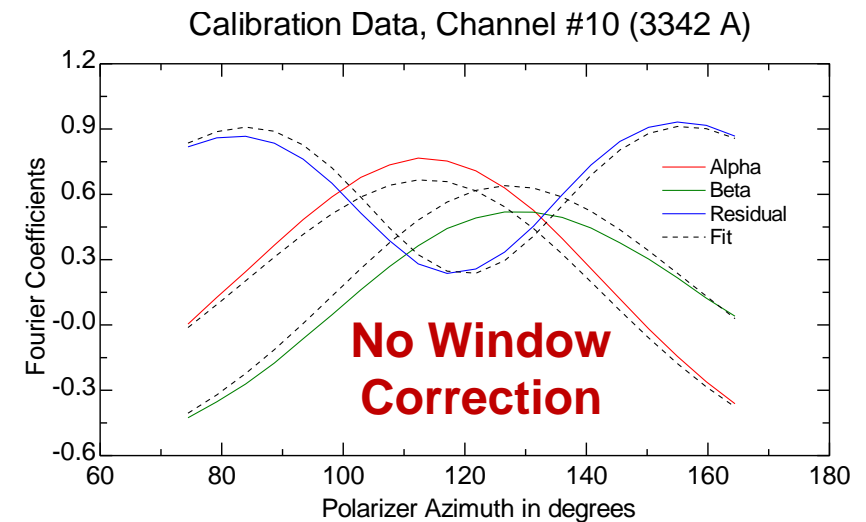
Common In Situ Considerations

- Nominal Angle of Incidence
 - Determined during ‘System Check’ calibration
- Window Effects
- Substrate Temperature
- Substrate Wobble



Viewport Window Effects (Strain)

- SE polarization measurements are not affected by transparent coatings on chamber windows.
 - Beam transmits through window at/near normal incidence.
 - Absorbing coatings can decrease signal-to-noise or eventually limit spectral range if coatings become opaque.
- Calibration is required for window birefringence, which changes the polarization.
 - Patented 2nd order window correction applied.

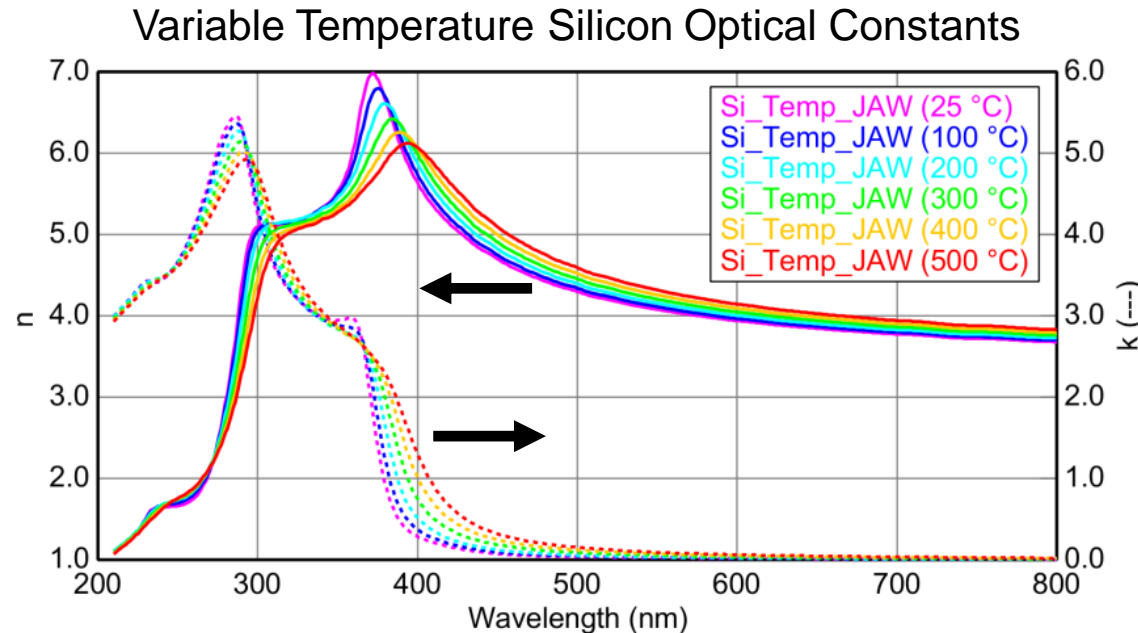


Mount windows / viewports evenly to minimize induced window mounting strain



Substrate Temperature

- Substrate commonly not at room temperature.
- Optical constants are temperature dependent.

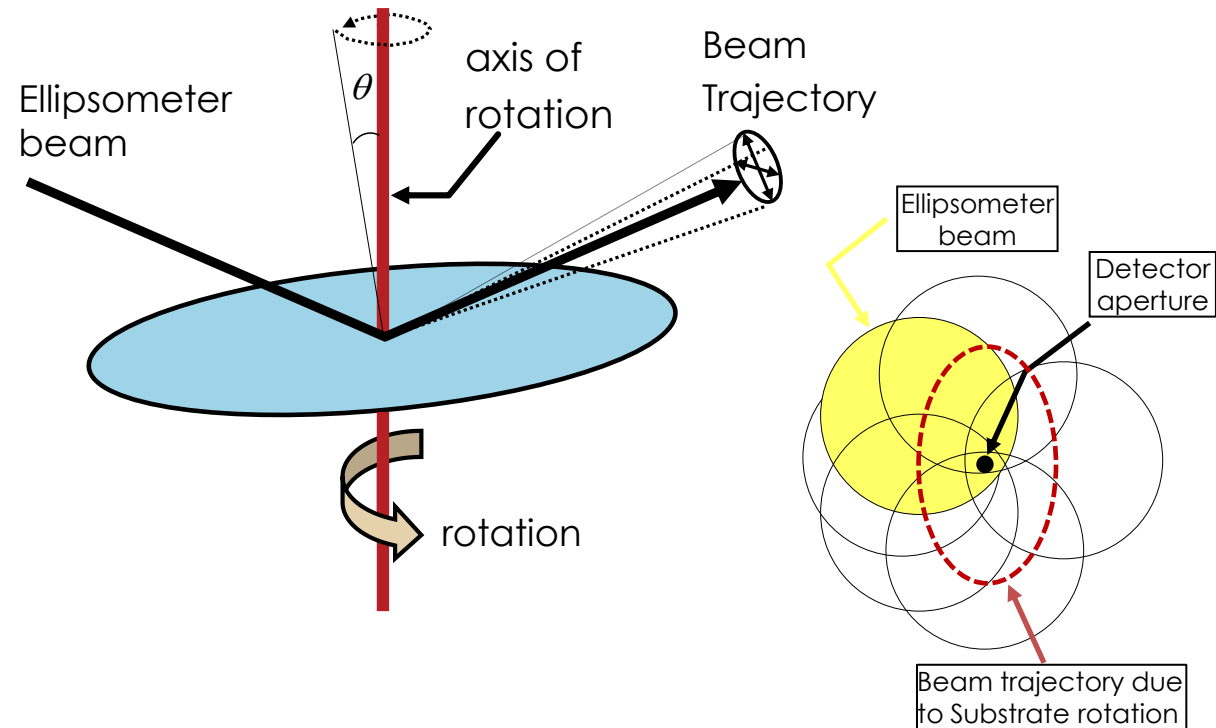


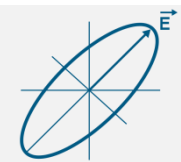
- Temperature and composition dependent material files available or can be created within the software.



Beam Wobble During Substrate Rotation

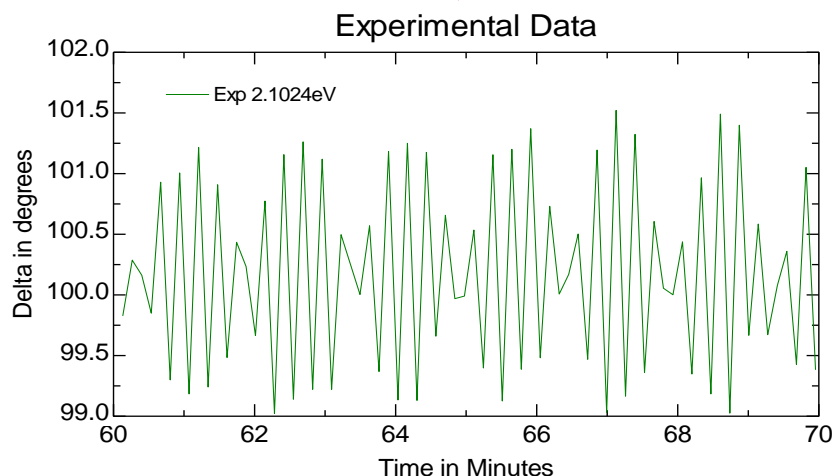
- Substrate rotation may cause beam “wobble”.
 - Minimize rotator wobble if possible.
 - Use large beam diameter to always cover receiver (detector) aperture.
 - Synchronize data acquisition period with substrate rotation period.
Or Trigger measurements for specific position.



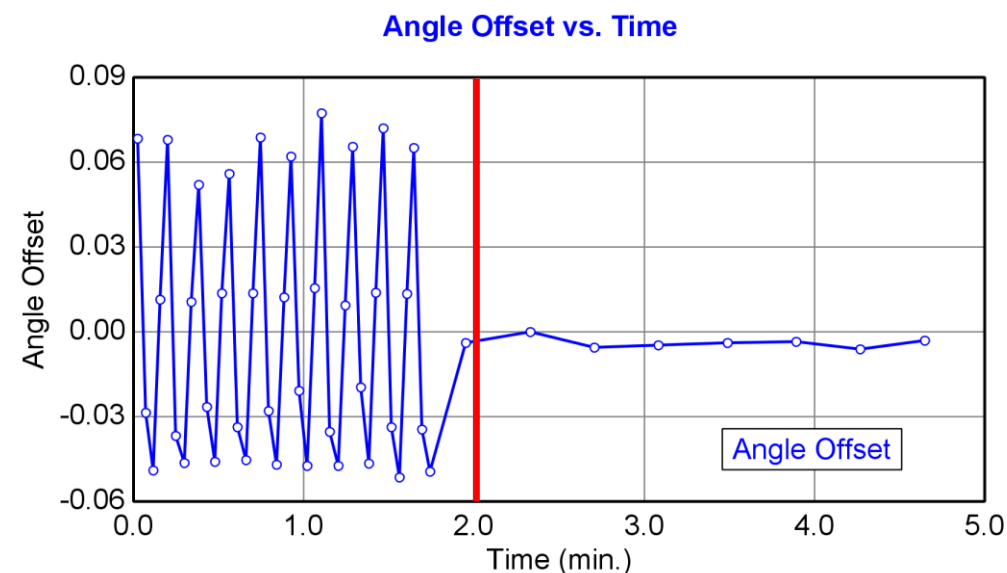
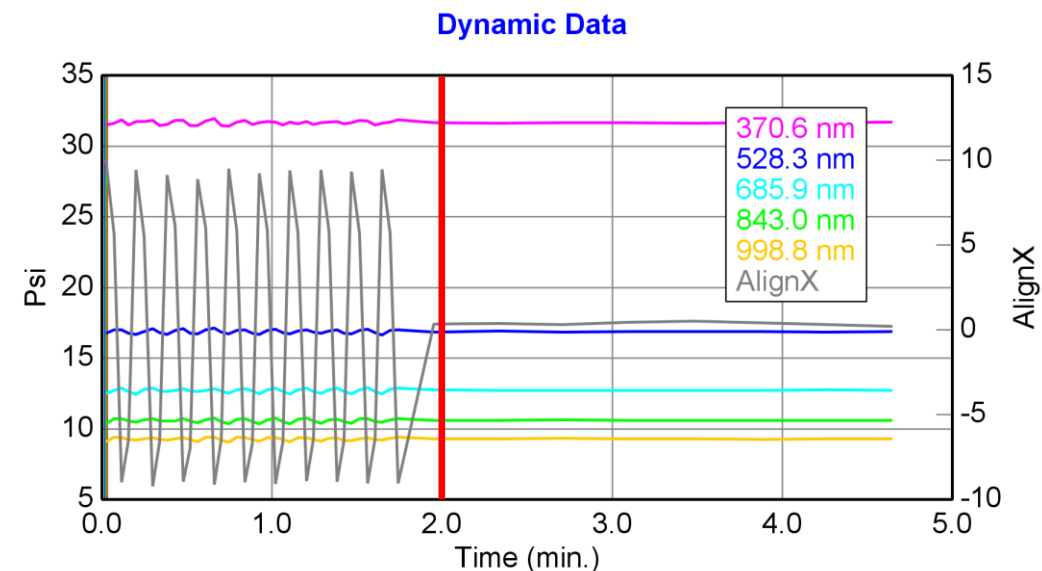


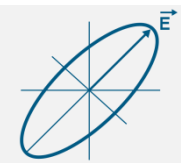
Synchronization with Rotation Period

- Beam wobble from rotating substrates can produce beat pattern in dynamic data when acquisition period does not match substrate rotation period.

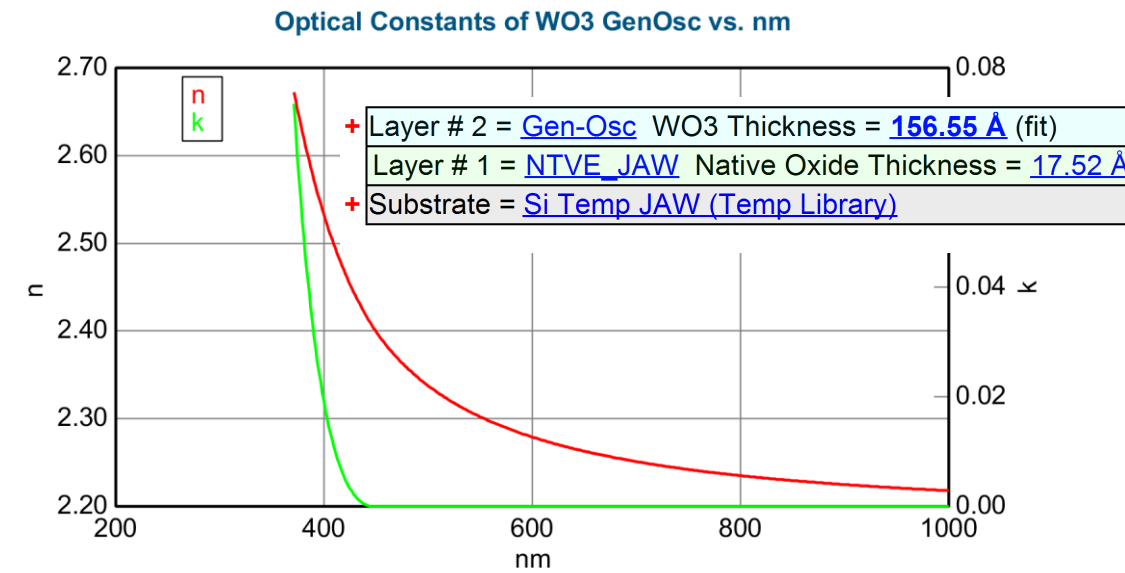
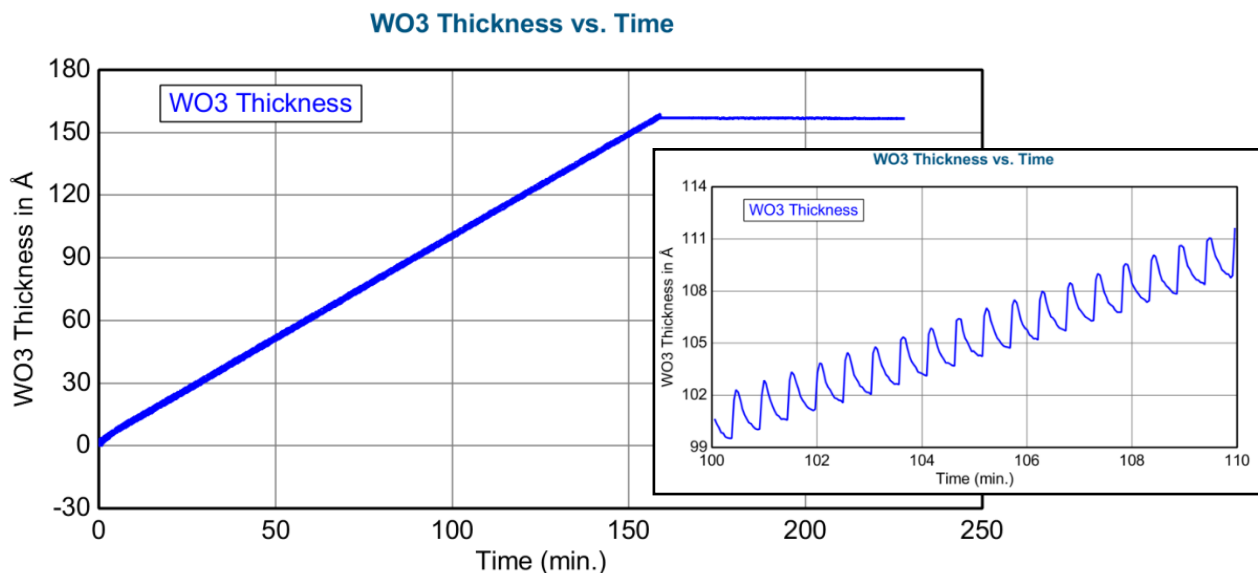
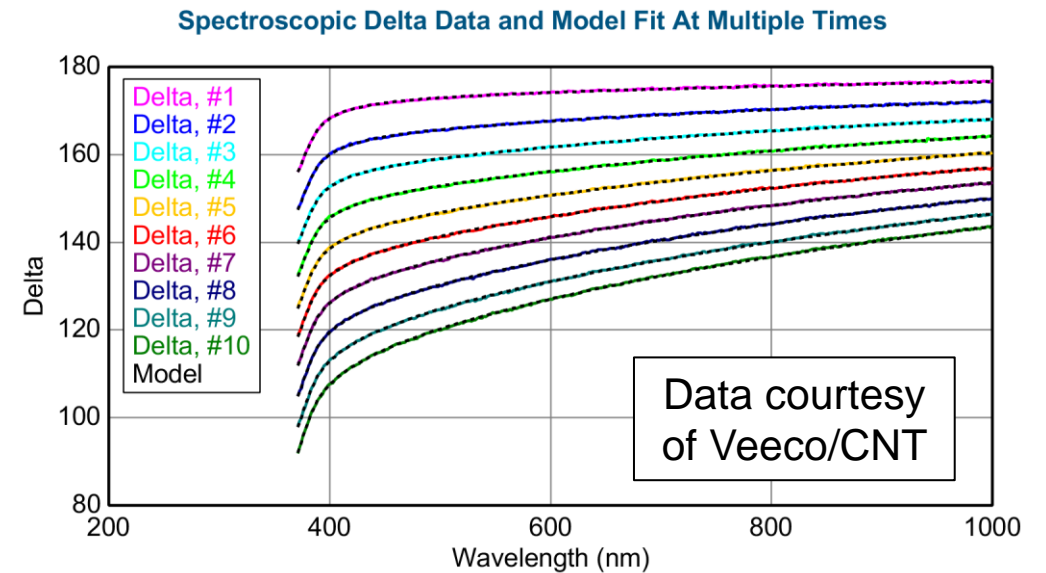
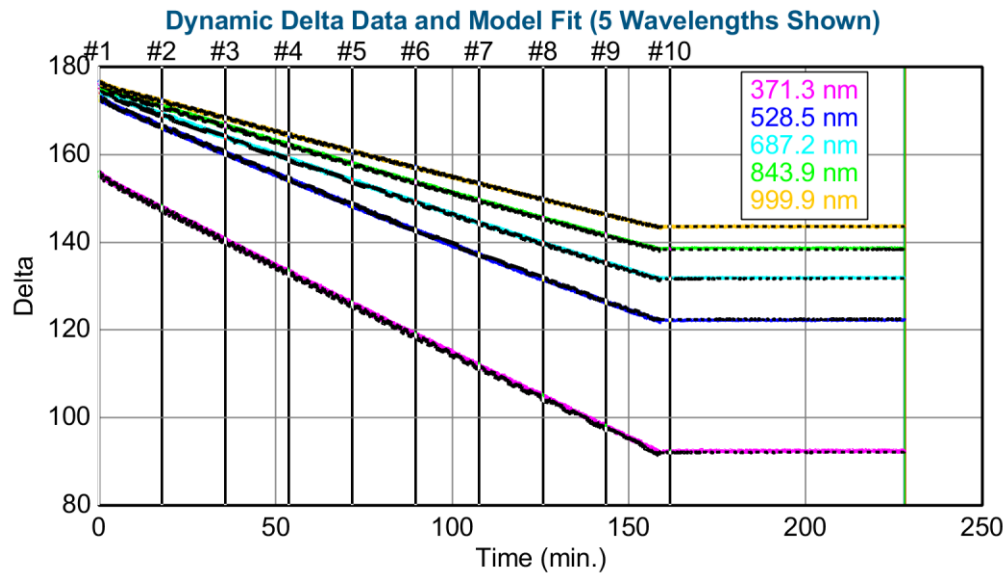


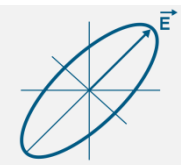
- Right figures show dynamic data initially out of sync with substrate rotation period. Data acquisition time changed after 2 minutes to match substrate rotation period.





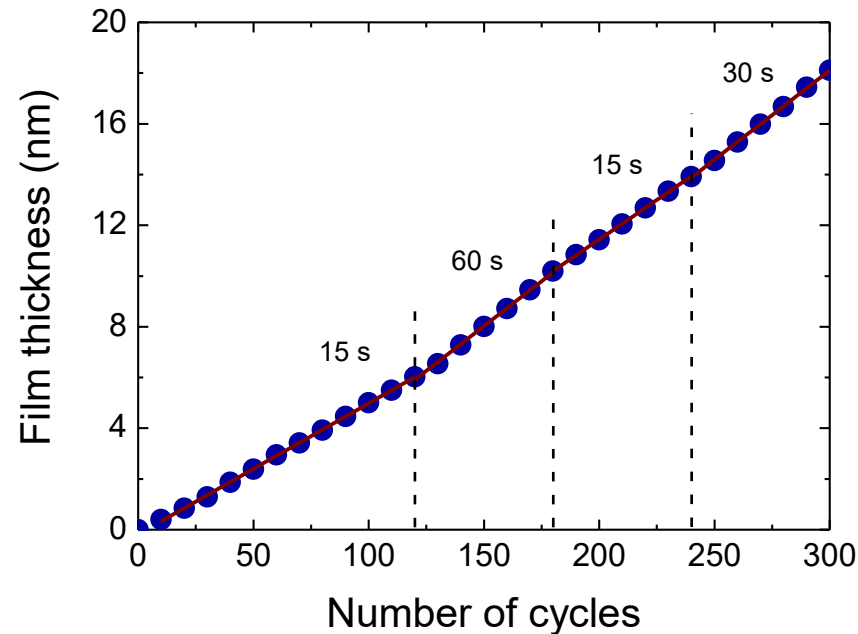
Growth Example: ALD WO₃



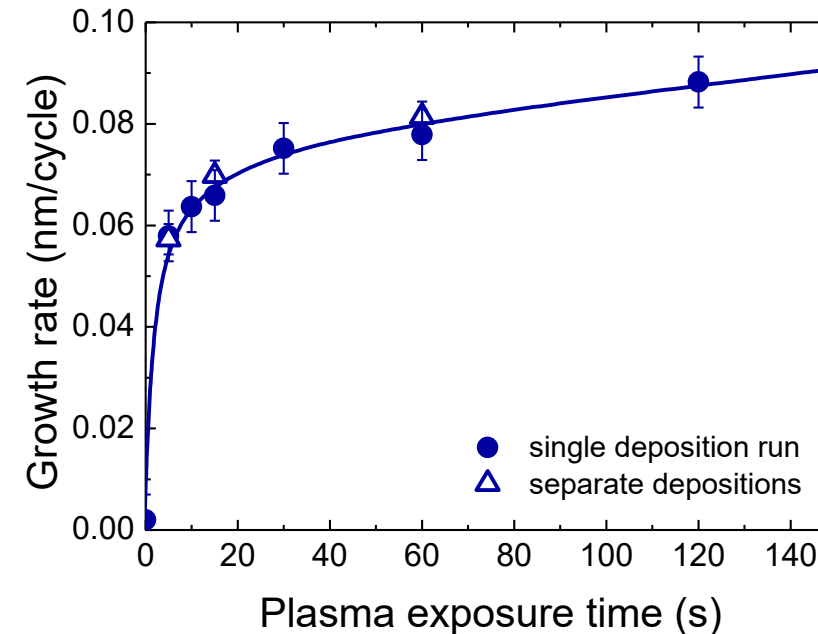


Fast Process Development & Optimization

Monitor film thickness while changing precursor/reactant dosing time



Calculate growth per cycle from slope to establish saturation curves

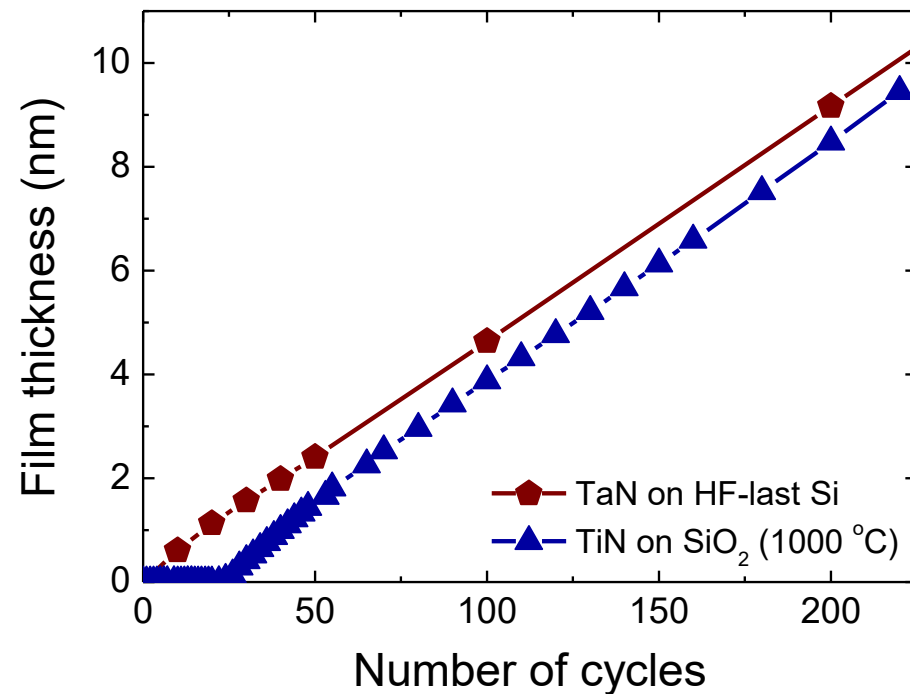


Ex situ measurements **not** strictly necessary as **single deposition run** yields saturation curves, etc.

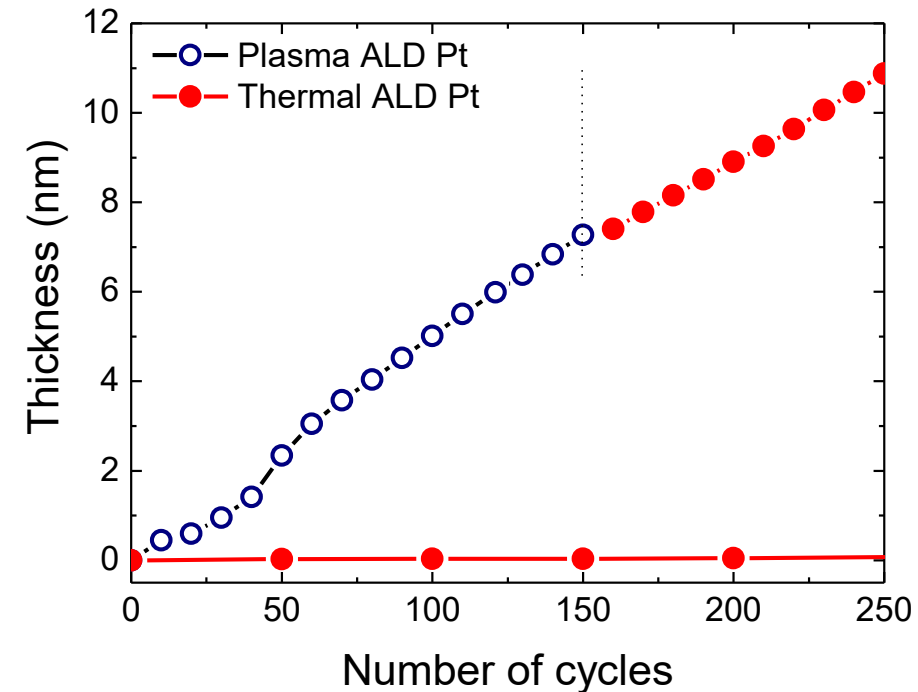


Initial Growth: Substrate Dependence, Nucleation Delay

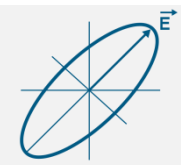
TiN from TiCl_4 nucleates poorly on surfaces with low $-\text{OH}$ densities,
TaN from $\text{Ta}[\text{N}(\text{CH}_3)_2]_5$ nucleates easily



Very long nucleation delay of Pt by thermal ALD on oxide surfaces,
plasma-assisted ALD nucleates easily

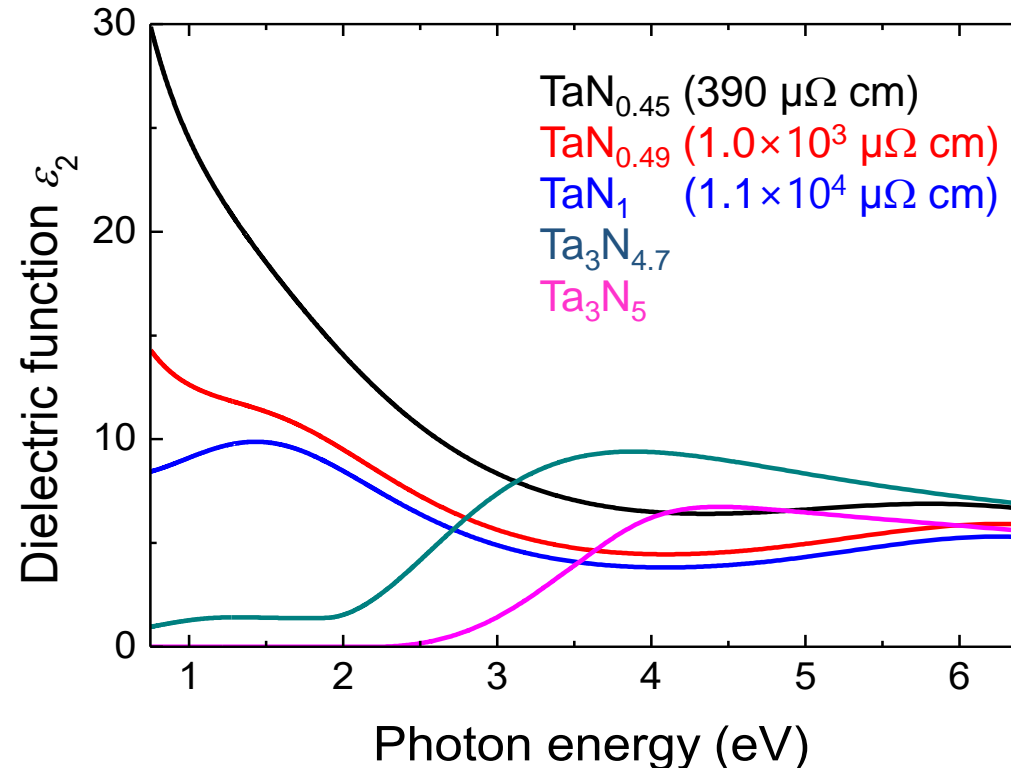


With *in situ* SE nucleation delay of ALD processes can be investigated
and film thickness can be **controlled exactly**



Distinction of Phase Compositions

Different TaN_x phases deposited by varying ALD conditions



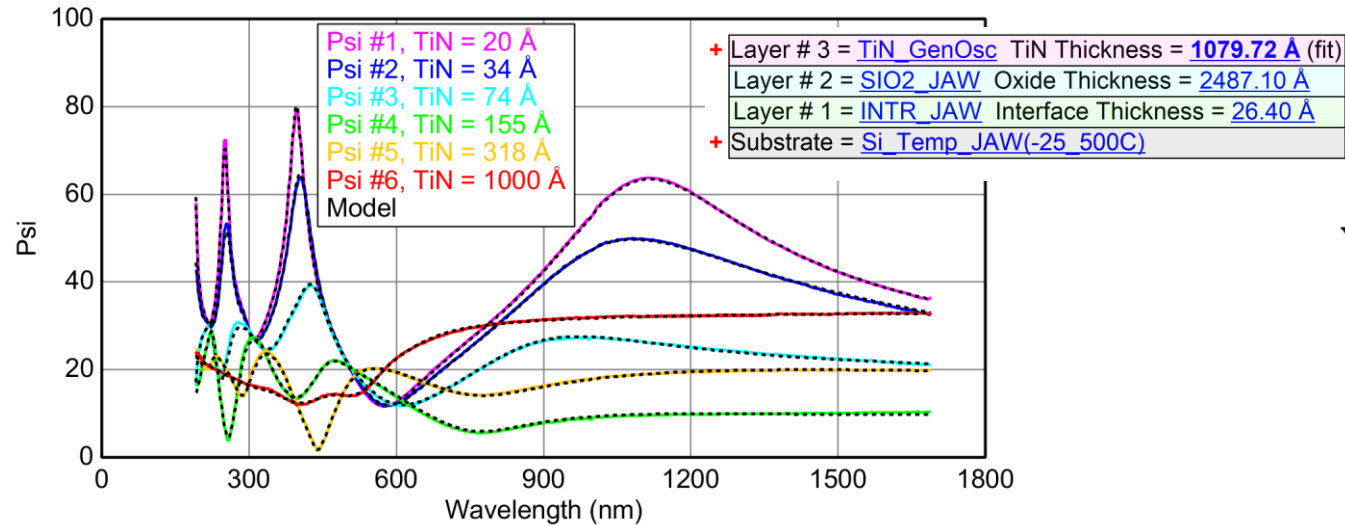
Clear **distinction TaN_x phases** from dielectric function:

- **Conductive TaN** shows Drude absorption by free electrons
- **Semiconductive Ta_3N_5** shows Tauc bandgap behavior

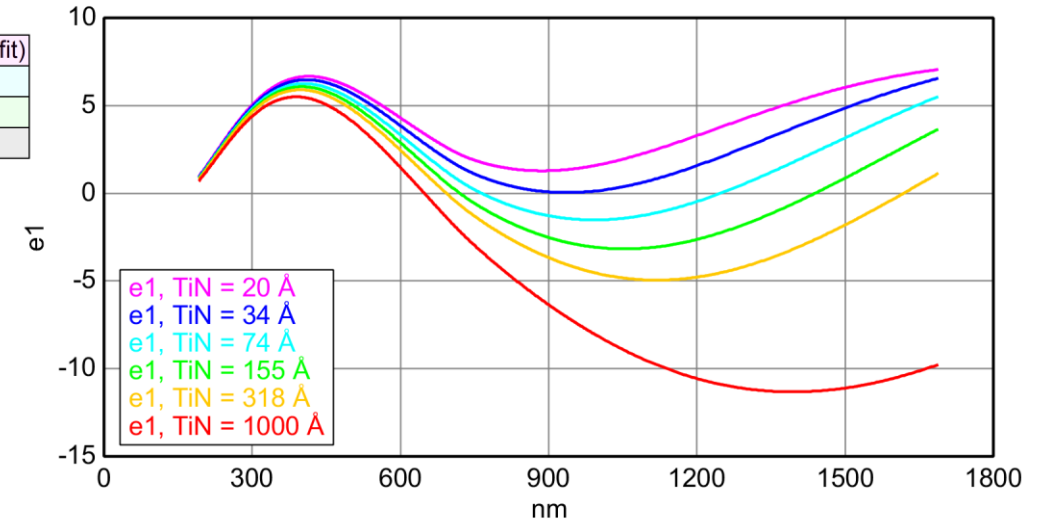


Evolving Film Properties: ALD TiN

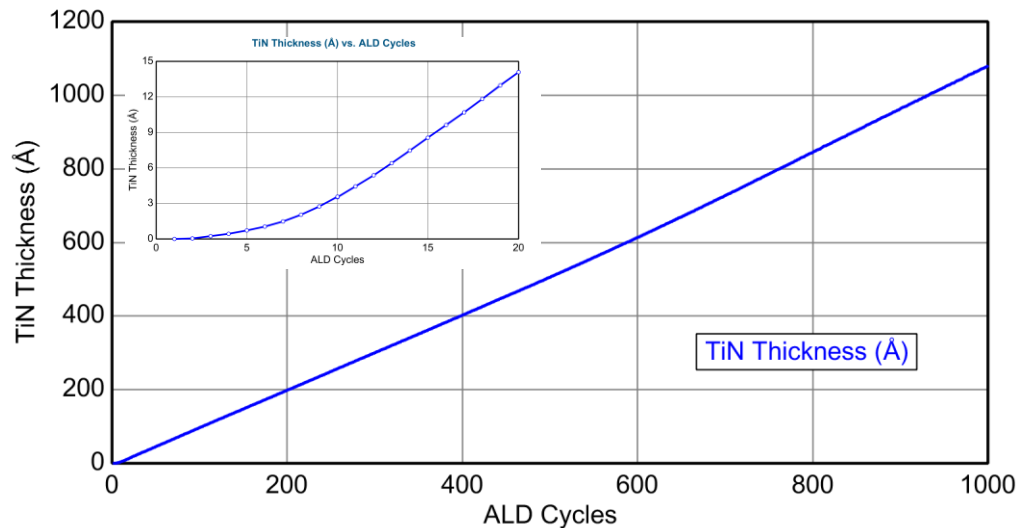
Spectroscopic Psi Data vs. TiN Thickness



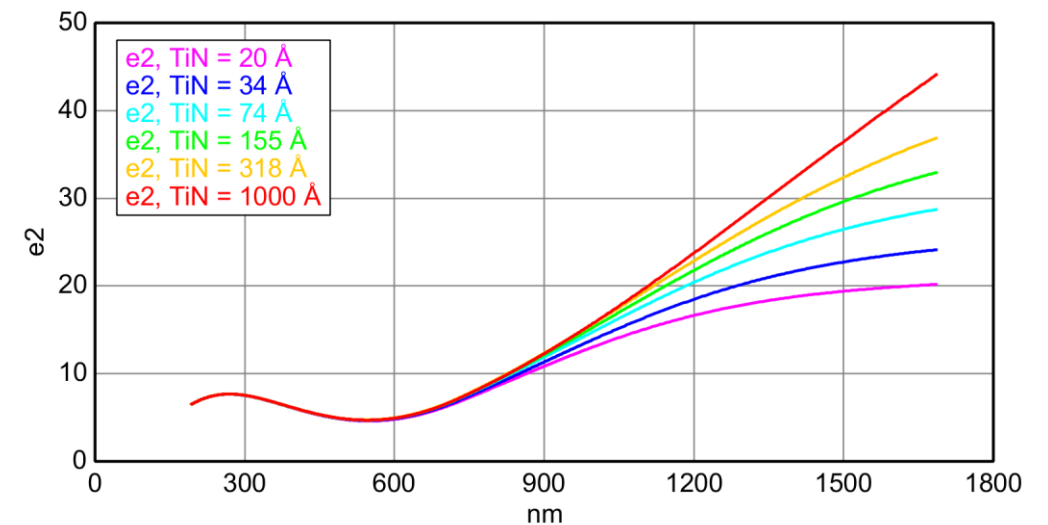
Optical Constants vs. TiN Thickness

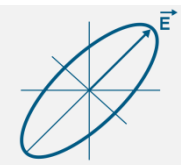


TiN Thickness (Å) vs. ALD Cycles



Optical Constants vs. TiN Thickness





Summary

- Spectroscopic Ellipsometry is a useful technique for monitoring in situ dynamic processes.
 - Determine thickness, growth/etch rate, optical constants...
- Determine other material properties such as composition, resistivity, and crystallinity from optical property changes.
- Spectroscopic ellipsometers have been integrated on a variety of in situ and in-line systems.
- THANK YOU!

