COVALENT METROLOGY

Covalent Academy

Episode 1: Spectroscopic Ellipsometry: The Swiss Army Knife of Optical Metrology

Presenter: Max Junda
max.junda@covalentmetrology.com

covalentmetrology.com

Covalent Metrology



- **Founded 2016**
- **Testing, measurement & characterization Platform**
- **30 team members (13 PhDs)**
- **9,500 ft² lab in Sunnyvale**
- **1-Stop-Shop Source for Answers**
  - 11 partner labs
  - 6 instrument partnerships
  - 6 corporate “tool shares”
- **>275 customers** (80% repeat)

Covalent Technical Groups and Organization

Electron & Scanning Probe Microscopy

(SEM/TEM, AFM, Nanoindent, acoustic microscopy)

Surface Analysis

(XPS, Auger, ToF-SIMS, dSIMS)

Optical Microscopy & Spectroscopy

(laser confocal, white light, chromatic aberration, spectral ellipsometry, UV-Vis-NIR spectroscopy)

Materials/ PC Board FA

(cross sections, inspections, dye & pry)

X-Ray Characterization

(XRD/XRR, XRF, µCT)

Chemical Analysis

(ICPMS, GCMS, DSC, TGA, DMA)

Nanoparticle Analysis/ Rheology

(DLS, PSA, zeta potential, rheometry)

Dr. Maxwell Junda



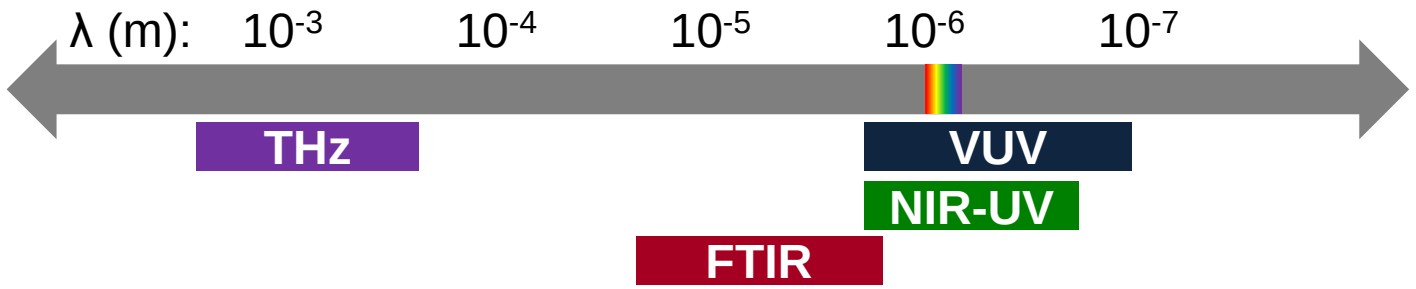
Max received his PhD in Physics from the University of Toledo in 2017 in the research group of Prof. Nik Podraza. His research work there was at the intersection of optics and condensed matter physics with a focus on solar cells. His thesis, *Spectroscopic Ellipsometry as a Versatile, Non-Contact Probe of Optical, Electrical, and Structural Properties in Thin Films: Applications in Photovoltaics*, highlights some of the broad range in which ellipsometry can be leveraged in materials science. This work and ensuing post-doc involved extensive use of both *in* and *ex situ* ellipsometry covering a spectral range from the THz to the vacuum ultraviolet.

Max joined Covalent in September 2019 and spearheads the company's ellipsometry services. As a member of the optical metrology group, he is also actively involved with Covalent's other optics-based offerings including as various microscopy and surface profilometry measurement capabilities.

Personal Ellipsometry Background

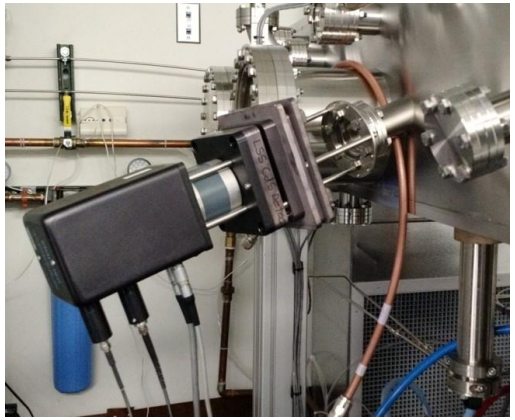
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Wide Spectral Range Instrumentation



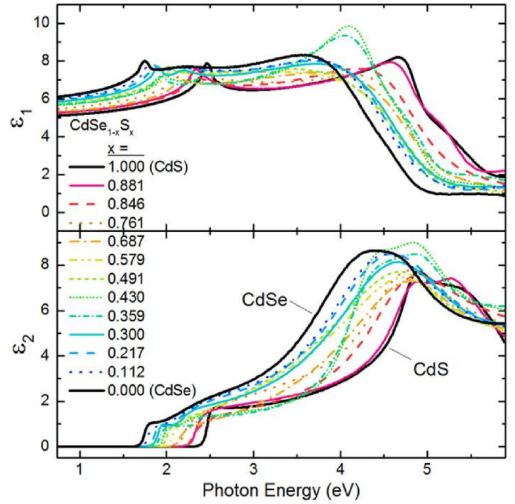
Real Time Ellipsometry

- Deposition monitoring
- Etch monitoring
- Function of temperature
- Function of ambient condition



Model & Experimental Development

- Custom optical property models
- Accessories for custom experimental configurations
- Previously unmeasured materials

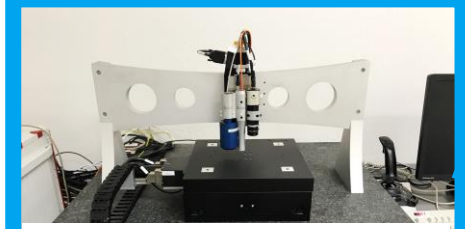


From: M. M. Junda et al., *Electron. Mater. Lett.* **15**, 500 (2019)

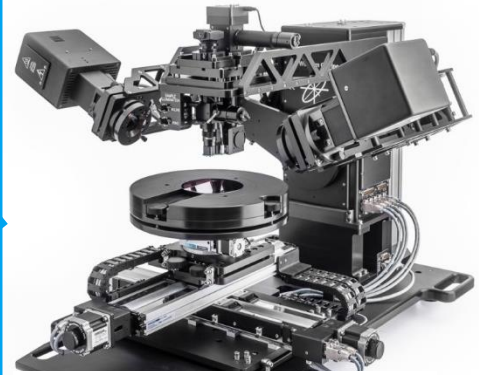
Role at Covalent Metrology

- Senior Member of Technical Staff; Optical Metrology Group





Chromatic Dispersion Profilometry



Spectral Ellipsometry



Patterned White Light



White Light Interferometry



Laser Confocal Microscopy

Outline

1 Introduction to Spectroscopic Ellipsometry

- Covalent instrumentation and capabilities
- Measurement principle
- Raw data: what we actually measure
- Modeling to derive sample properties

2 Applications and Examples

- Customer applications
- Beyond n, k, & thickness
- Large-area spatial mapping

Covalent Instrumentation and Capability



M-2000 DI

- 193 – 1690 nm
- Measurement in seconds
- 300 mm max. sample size
- Automated spatial mapping

J.A. Woollam Co.
Ellipsometry Solutions



IR-VASE

- 1.7 – 30 µm
- Measurement in tens of minutes
- 200mm max. sample size

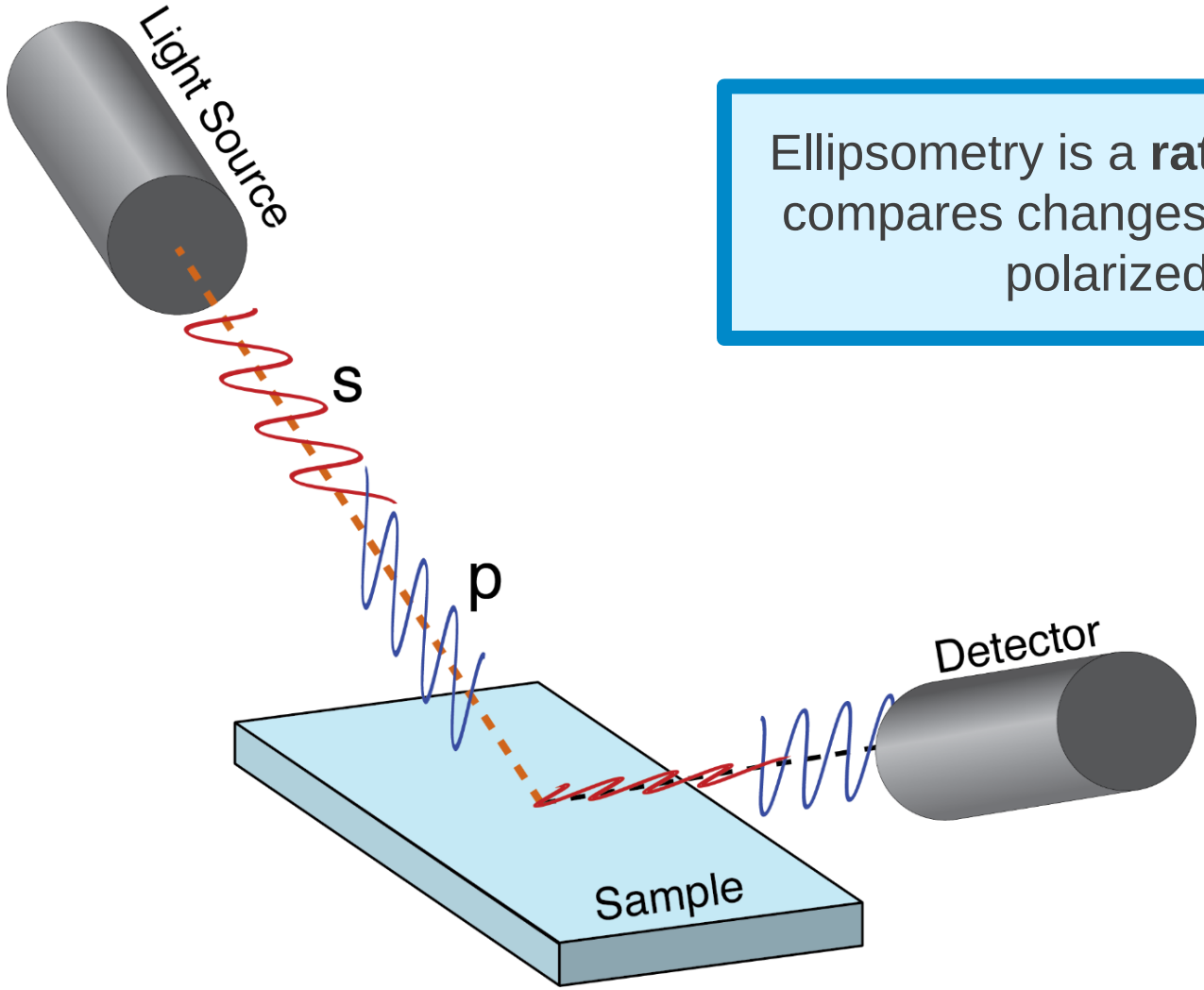
Coming to Covalent Metrology May 2020!



RC2 DI

- 193 – 1690 nm
- Measurement in seconds
- 300 mm max. sample size
- Automated spatial mapping
- Anisotropic Samples

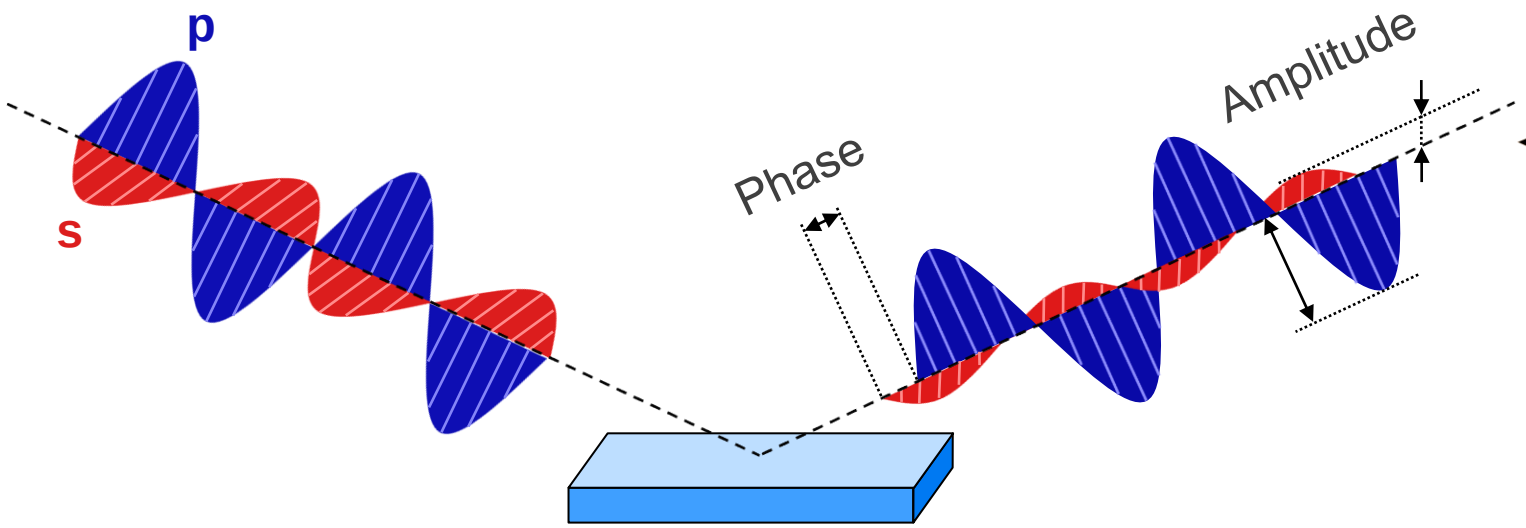
Ellipsometry Measurement Principle

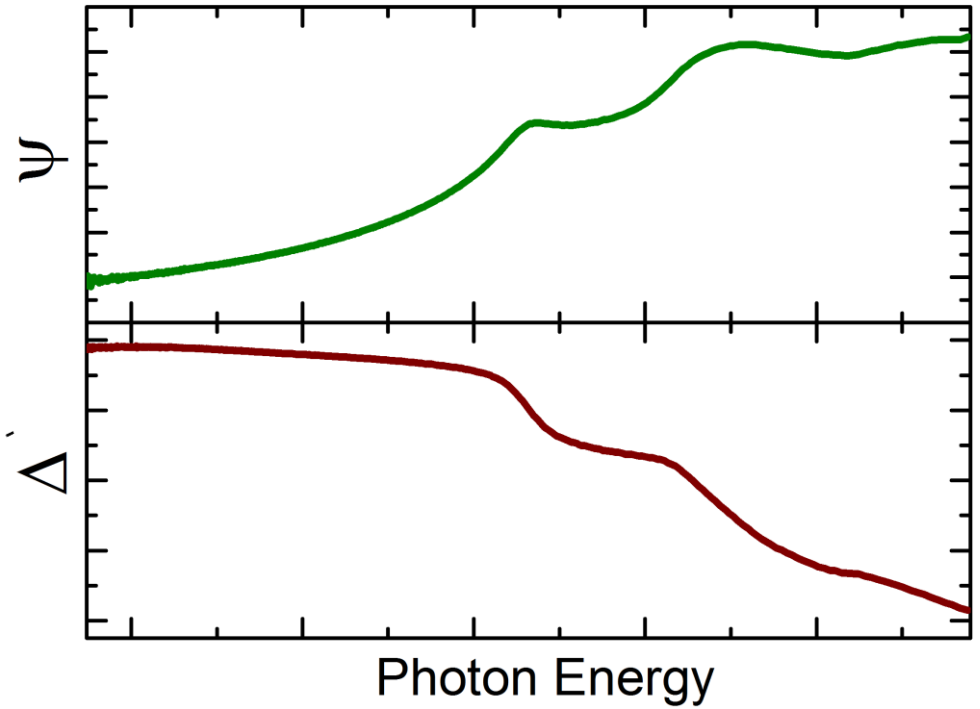


Ellipsometry is a **ratio-based** measurement that compares changes to **phase** and **amplitude** of polarized light components

Raw Data: What is Actually Measured?

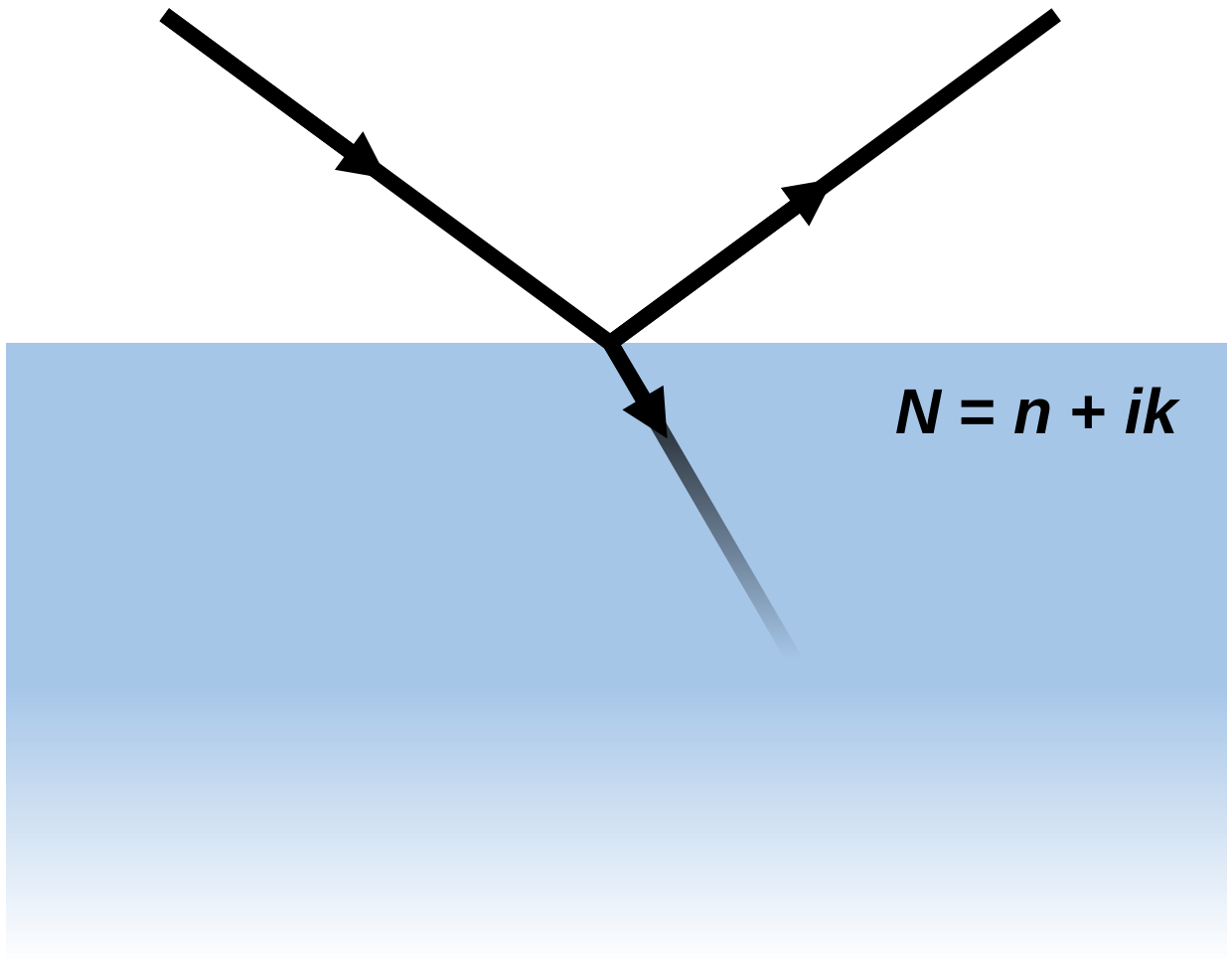
- Two values are measured at each wavelength:
  - ψ: amplitude ratio between p & s polarization
  - Δ: phase shift between p & s polarization





Optical Properties of Materials

- Optical properties described by ***N = n + ik***
  - ***n***: relative speed of light in the material compared to in vacuum
  - ***k***: strength of light absorption as light travels through the material



Calculating Effects at an Interface

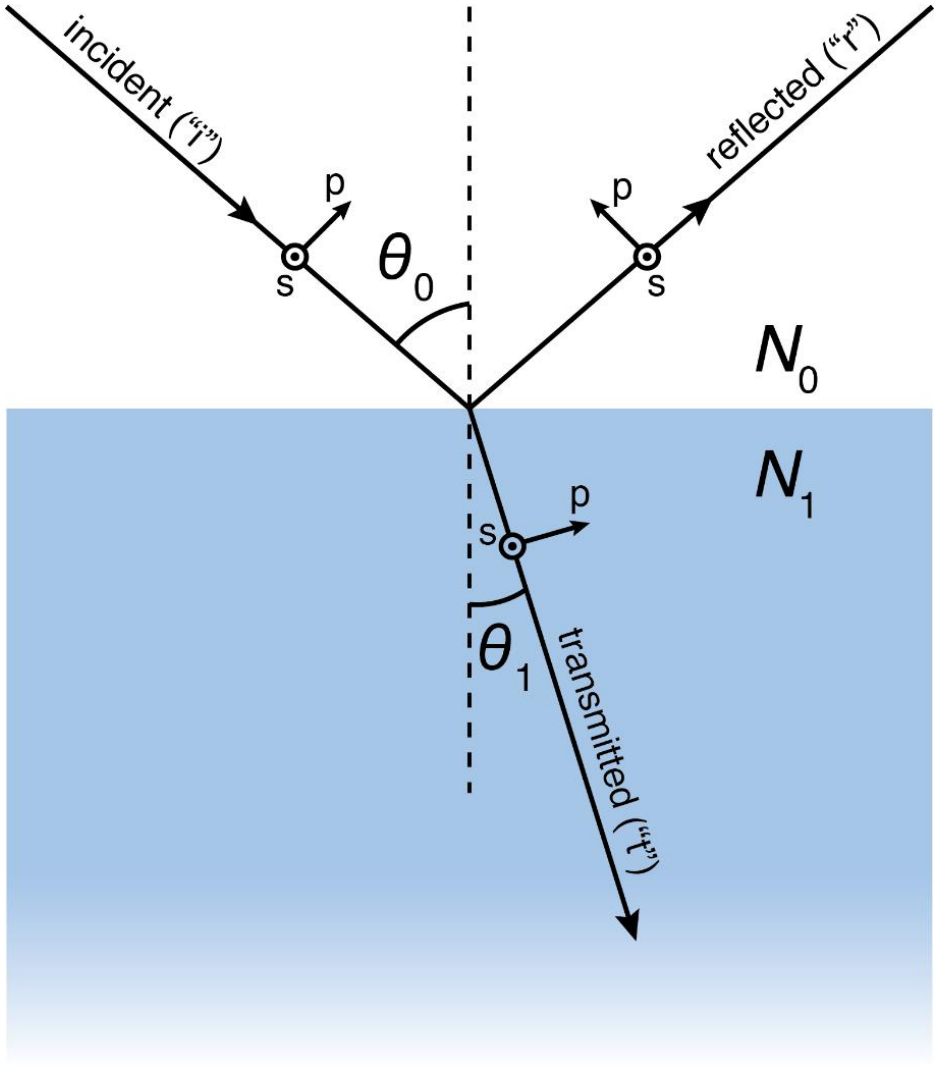
Fresnel Coefficients:

$$r_p = \frac{E_{rp}}{E_{ip}} = \frac{N_1 \cos\theta_0 - N_0 \cos\theta_1}{N_0 \cos\theta_1 + N_1 \cos\theta_0}$$

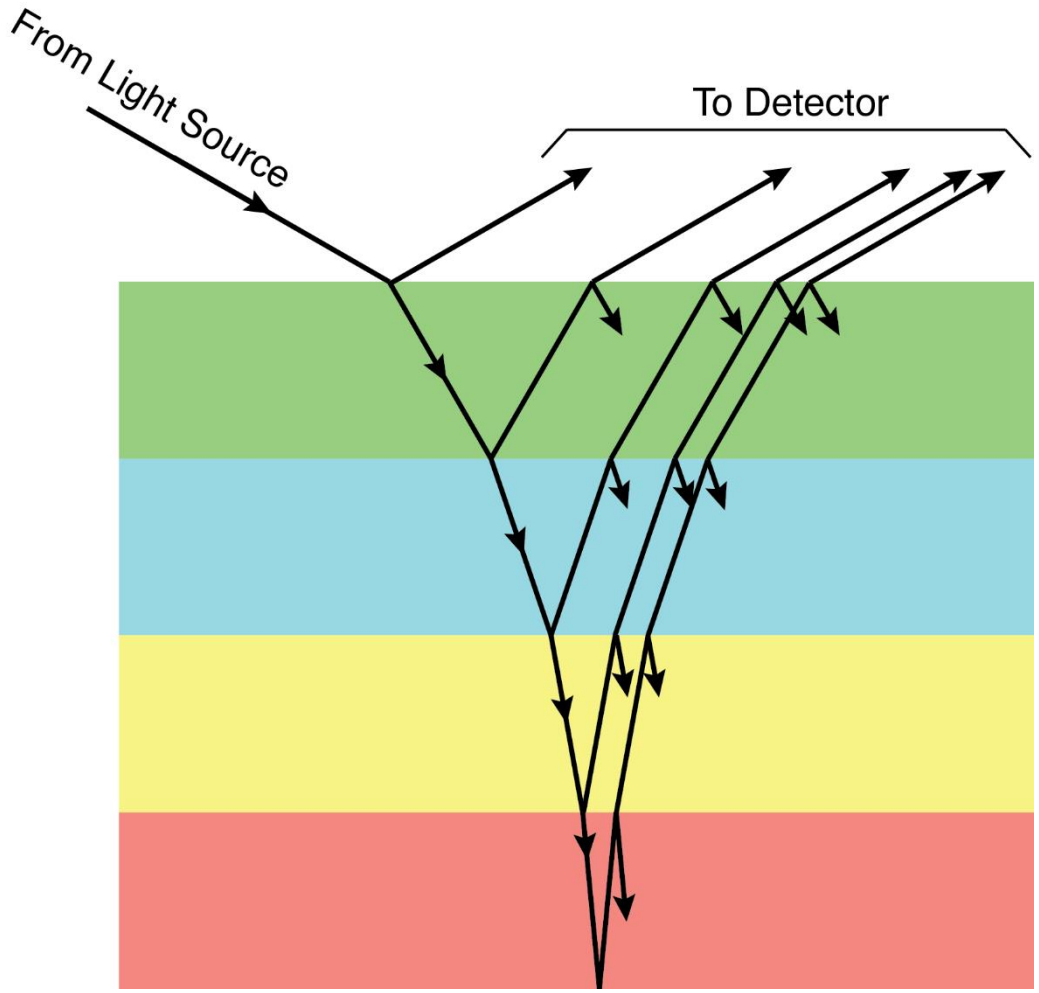
$$r_s = \frac{E_{rs}}{E_{is}} = \frac{N_0 \cos\theta_0 - N_1 \cos\theta_1}{N_0 \cos\theta_0 + N_1 \cos\theta_1}$$

$$t_p = \frac{E_{tp}}{E_{ip}} = \frac{2N_0 \cos\theta_0}{N_1 \cos\theta_0 + N_0 \cos\theta_1}$$

$$t_s = \frac{E_{ts}}{E_{is}} = \frac{2N_0 \cos\theta_0}{N_0 \cos\theta_0 + N_1 \cos\theta_1}$$



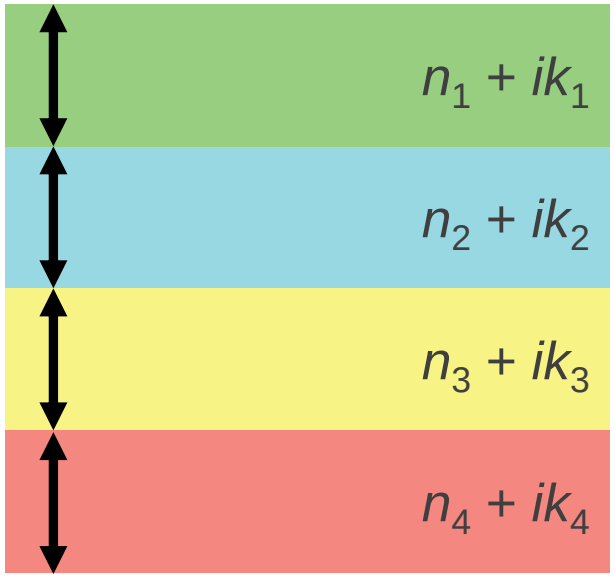
Multilayer Stacks

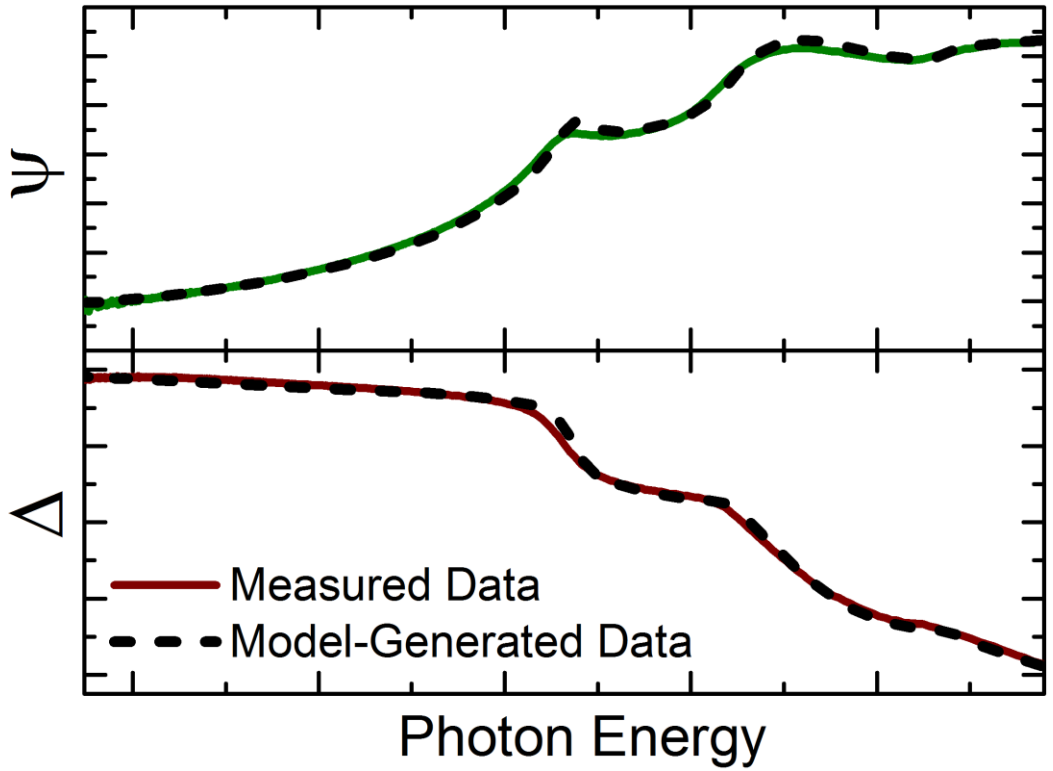


Multi-layered sample is simply a series of individual interactions between light beam and layers

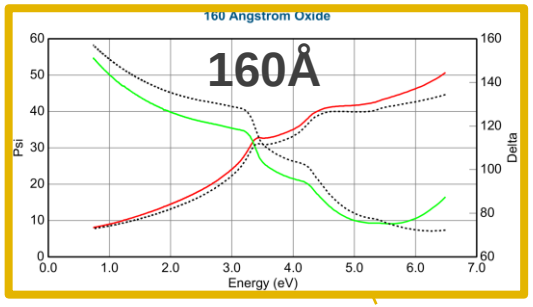
Fitting Optical Models to Measured Data

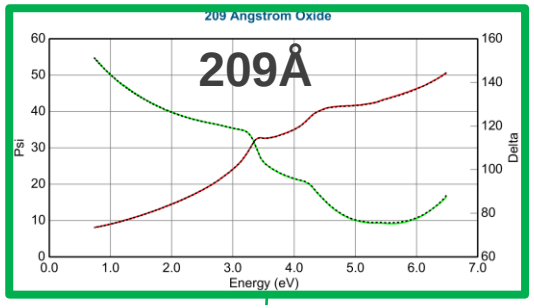
ψ and Δ resulting from optical model are calculated. Adjusting model parameters so that ψ and Δ match measured data determines unknown optical properties and thicknesses.

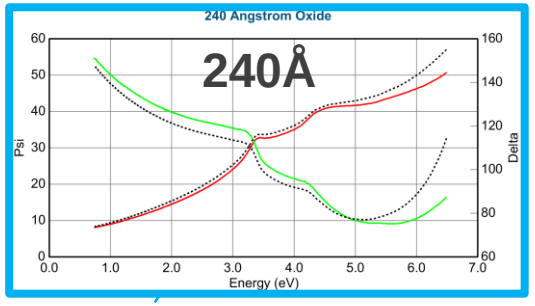


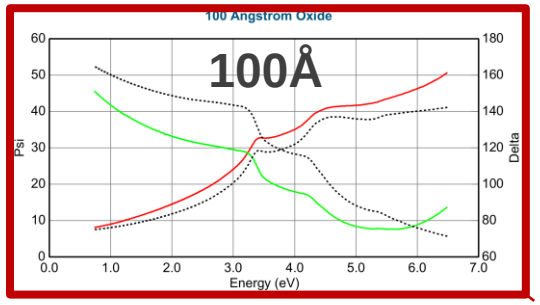


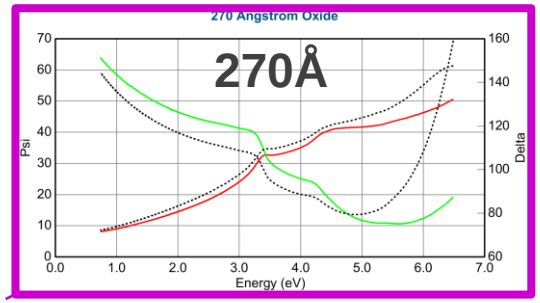
Model Fitting: Minimizing Error Function

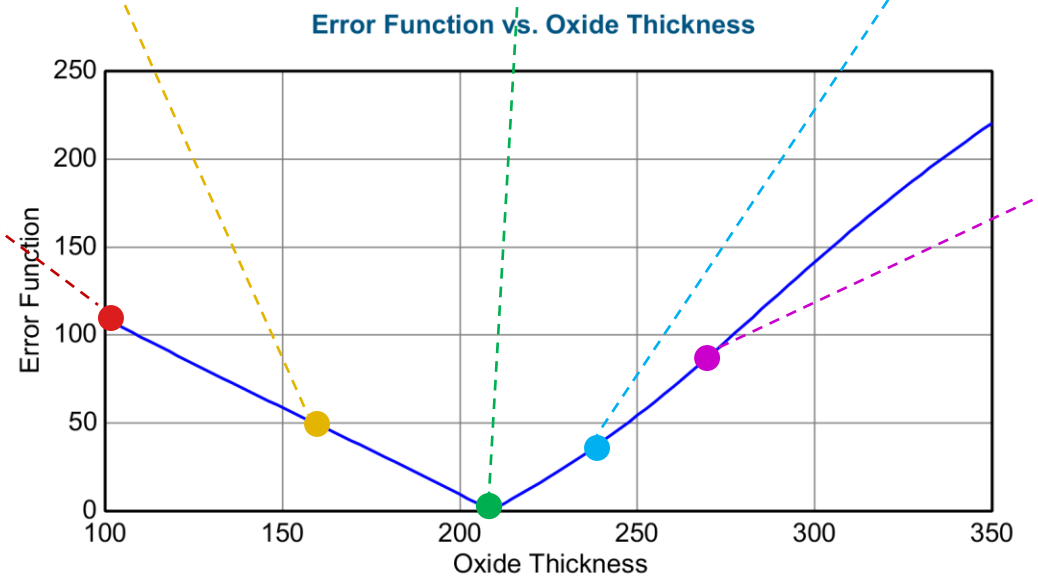












- Example shown here of a thermal oxide layer on a crystal Si wafer

Fit algorithm minimizes the error function which quantifies mismatch between model and data

Range of Applications

Basic modeling procedure can be modified for use with **many sample types** and to detect **many physical properties.**

Sometimes requires strategic sample preparation or experimental design.

Sample Types:

- Liquid samples
- Bulk materials
- Powders
- Anisotropic samples
- Etch / deposition processes
- Multilayer thin film stacks

Sample Properties:

- Composition
- Interfacial diffusion
- Resistivity of conductors
- Surface roughness
- Crystal strain
- Defect densities in disordered materials
- Temperature effects
- Chemical bonding states
- Spatial uniformity (i.e. mapping)

Range of Applications

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Typical SE Advantages:

- Fast
- Non-destructive
- Can be spatially mapped

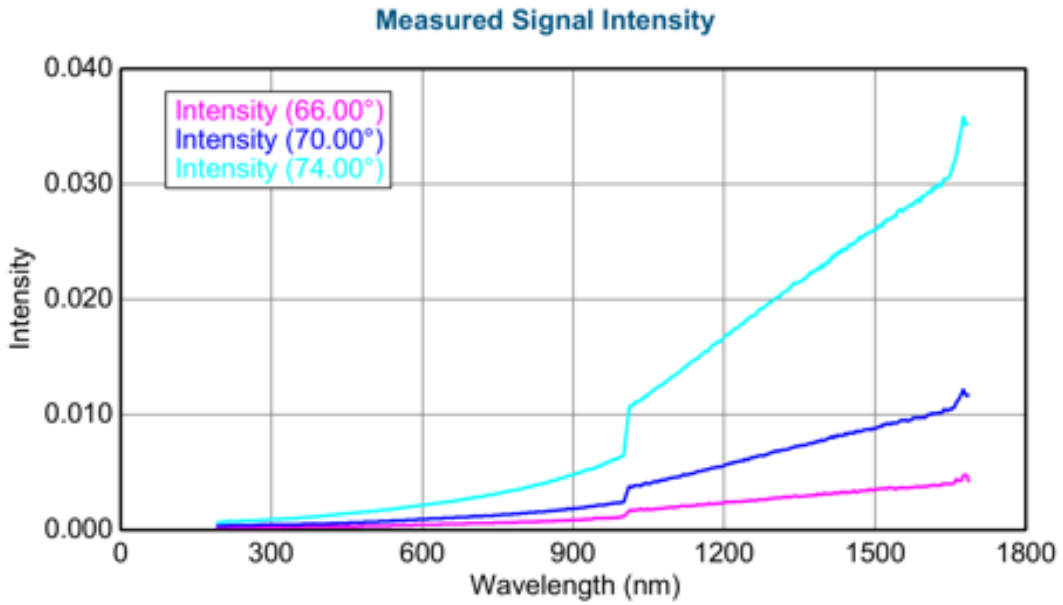
Property	Advantage	Disadvantage (Strengths of Alternatives)
Composition	[Typical]	Requires known correlations / optical databases
Interfacial Diffusion	[Typical]	Requires material transparency above interface
Resistivity of Conductors	Can access buried layers non-destructively	Spectral-range dependent accuracy (better at longer wavelengths) Slower than contact methods
Surface Roughness	[Typical]	Usually models roughness using approximations Can't determine roughness details (Ra, Rq, Sku, etc.)
Crystal Strain	[Typical]	Requires known correlations
Defect Densities in Disordered Materials	[Typical]	Only applicable to materials with many defects Insensitive to type / source of defects
Temperature Effects	Fast; Non-destructive	N/A
Chemical Bonding States	[Typical]	Requires bond vibration modes to be IR active Identification of states only through other measurements
Spatial Uniformity	[Typical]	Spatial resolution limited to beam size (~5mm unfocused; ~300µm focused)

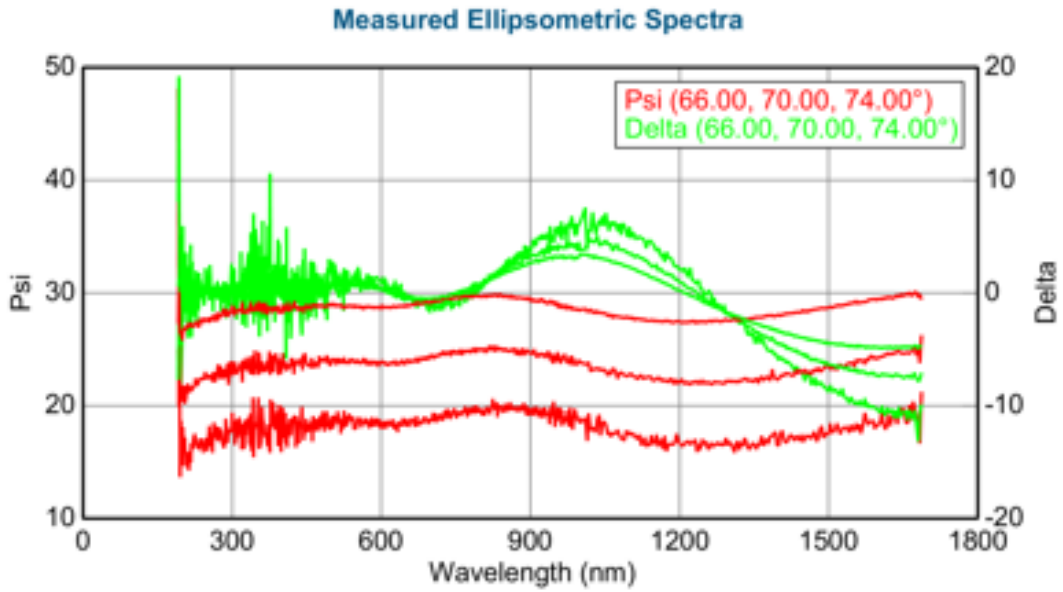
Example 1: Classic Ellipsometry Example with a Twist

Samples:	Printed circuit boards with water-resistant dip coating
Problem:	Would like an understanding of coating thickness, uniformity, and coverage
Experiment Design:	• Measure an uncoated board to model bare PCB surface • Measure coated boards for coating coverage and thickness • Confirm surface roughness with alternate measurement

Example 1: Rough Samples

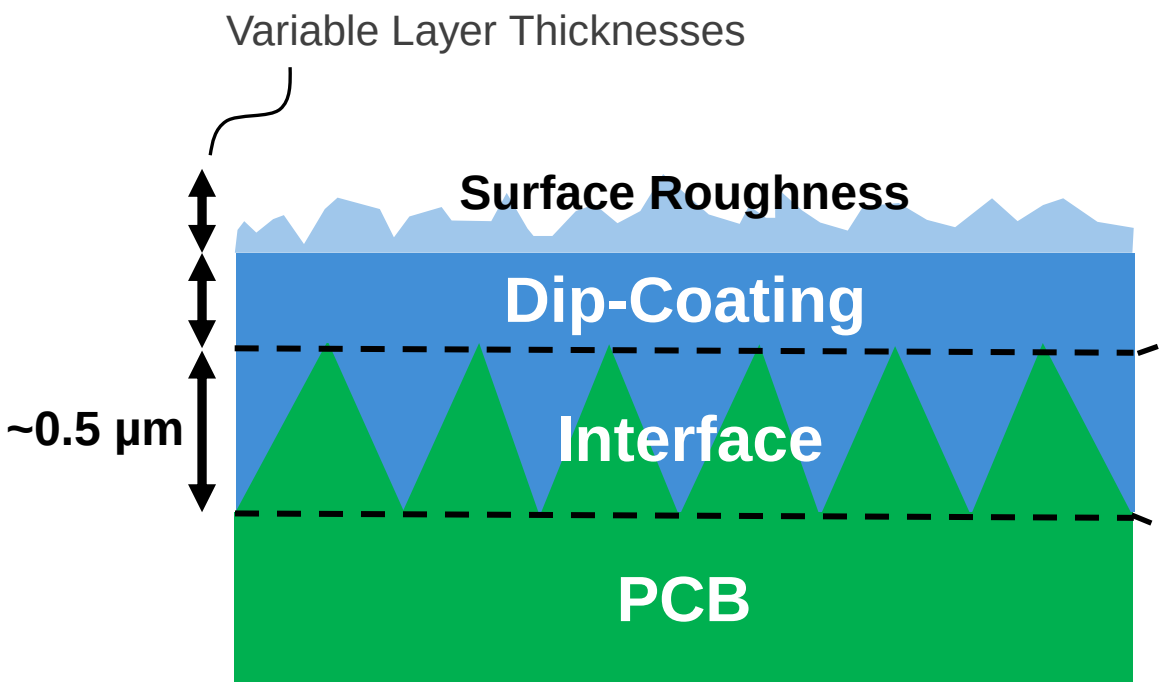
- Challenge: signal intensity very low due to high surface roughness

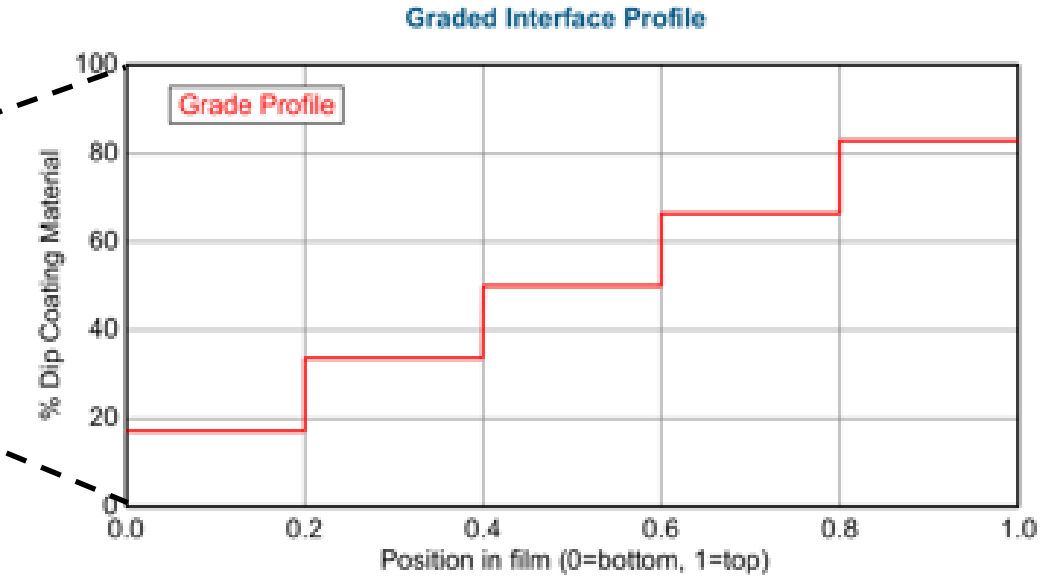




Example 1: Rough Samples

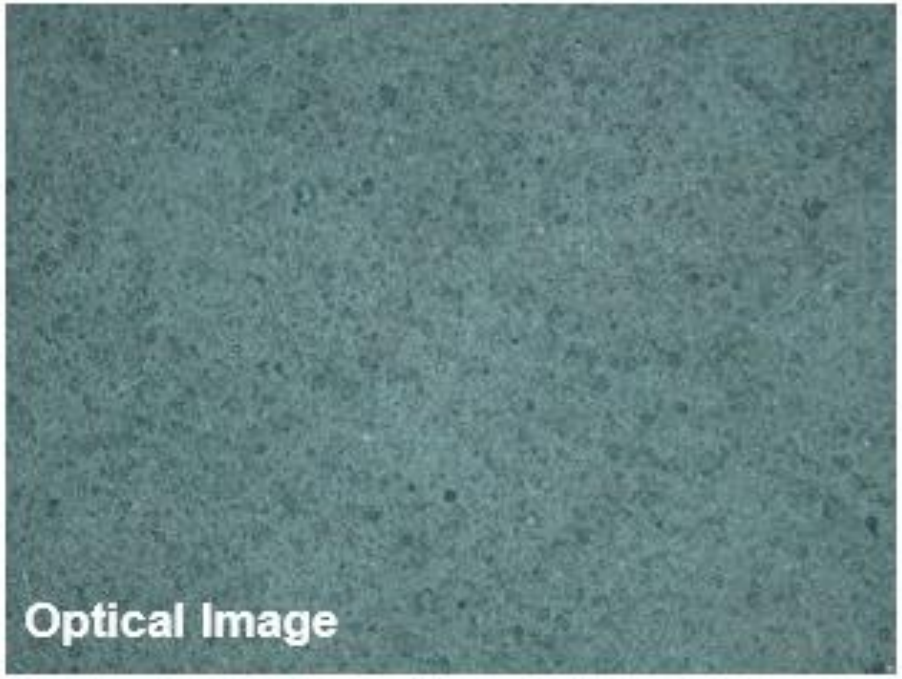
- Model incorporated an interface layer that was ~0.5 µm thick which approximated the roughness of the PCB that was filled with dip-coating material

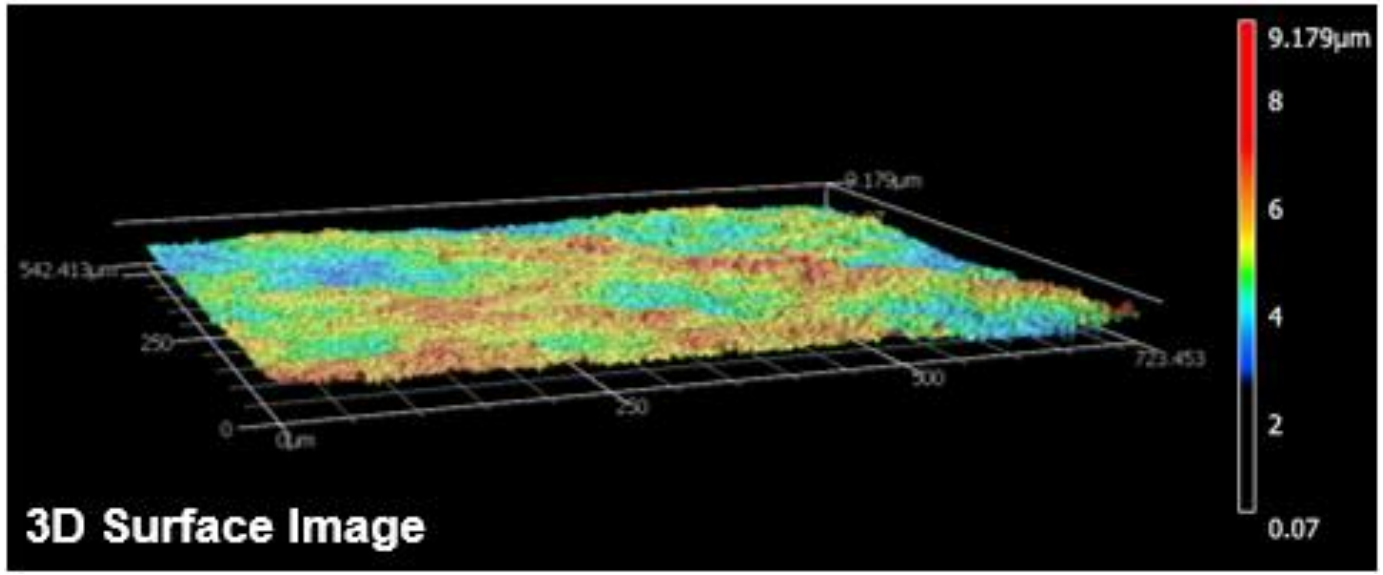




Example 1: Rough Samples

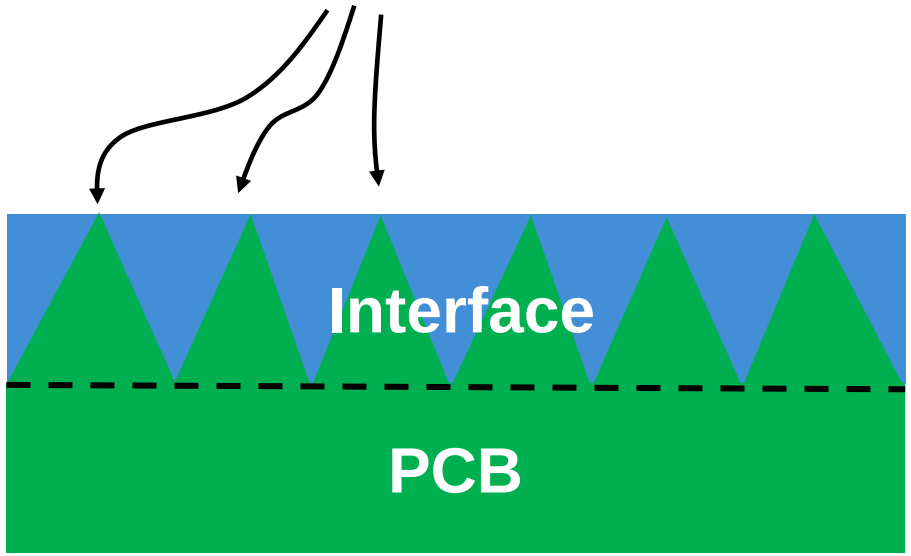
- Separate measurements using a Keyence VK X-1100 laser confocal microscope verify surface roughness of uncoated board
- Arithmetic mean roughness found to be **0.691 µm**
- This surface topography measurement justifies the use of the very thick interfacial layer where the coating has filled the voids of the bare board roughness





Example 1: Rough Samples

- Results provided thickness at multiple points on each board – measure of uniformity
- In a few cases, the coating and surface roughness thickness were found to be zero indicating a sample structure depicted below
- Thin coating at “high points” of PCB surface may be ineffective moisture barrier



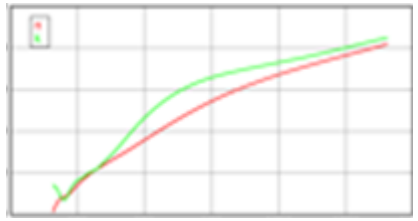
Beyond a simple measure of coating thickness, SE able to provide understanding of coating-substrate interface

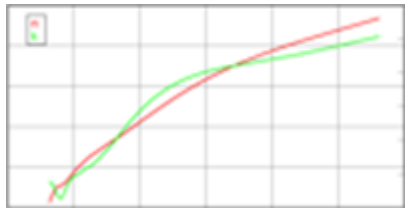
Example 2: Ellipsometry as Composition Probe

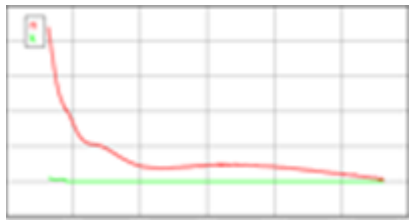
Samples:	TaN thin films on glass wafers
Problem:	Correlate process variables with resulting film (1) **composition** and (2) **uniformity**
Experiment Design:	• 5× TaN films systematically varying N_2 gas flows • Measure 11 point line scan across wafer • Correlate IR optical properties with processing

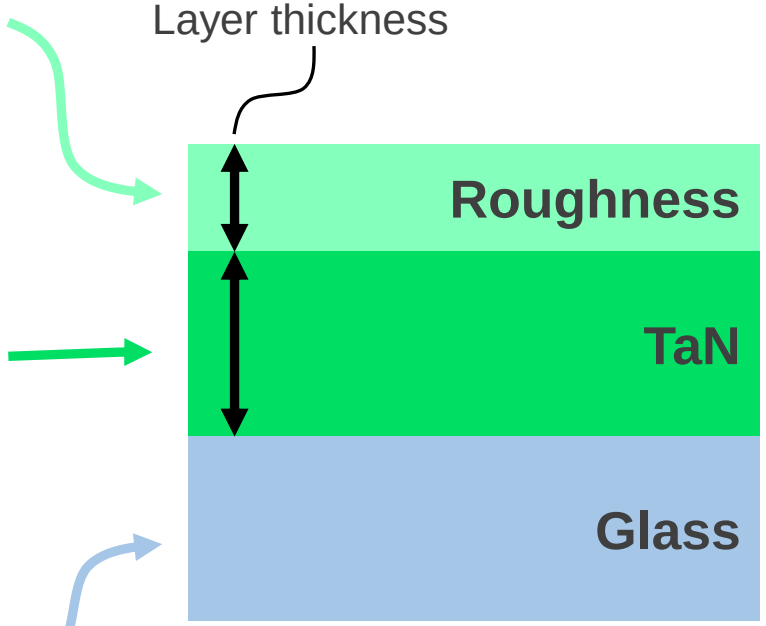
Example 2: Optical Model

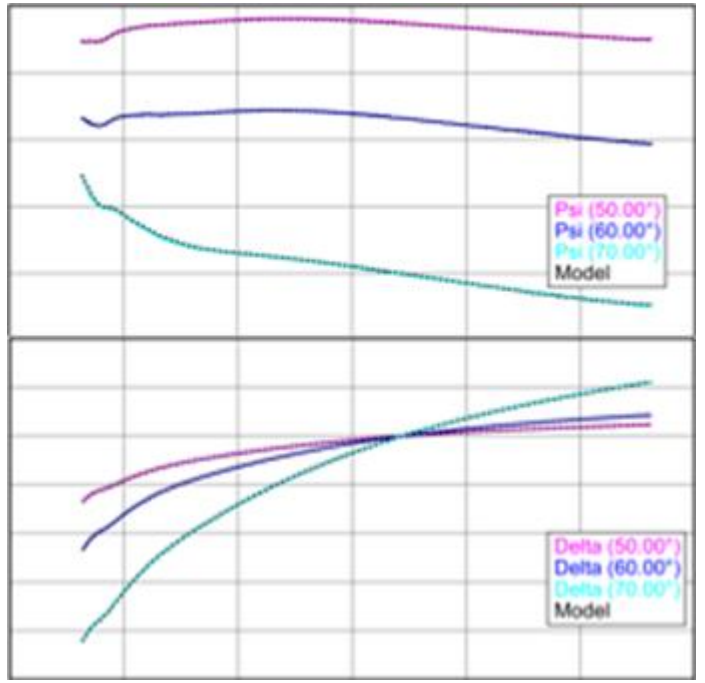
Optical properties of each layer









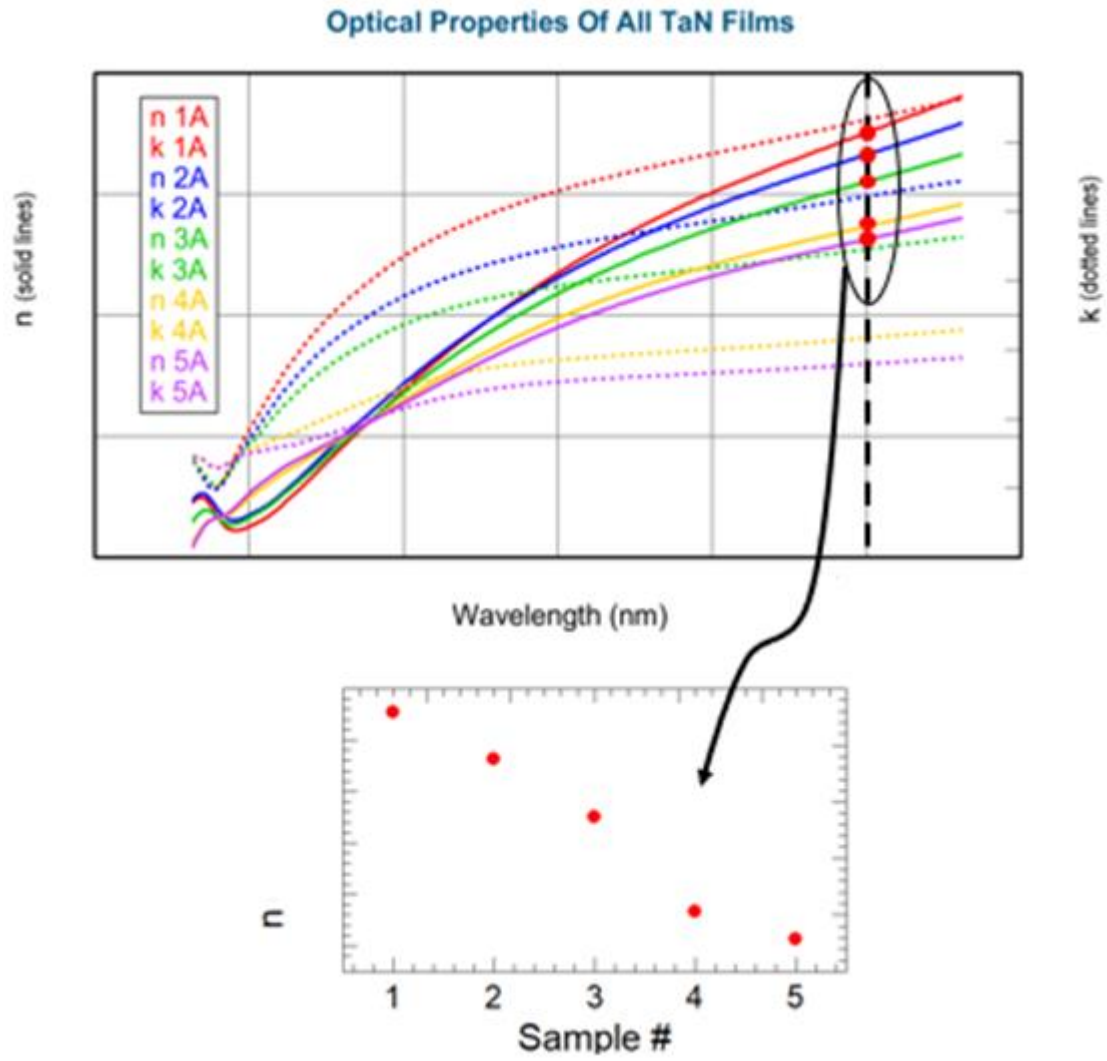


Fit procedure varies optical properties and layer thickness until model matches measured data

Example 2: Optical Property Correlation with Composition

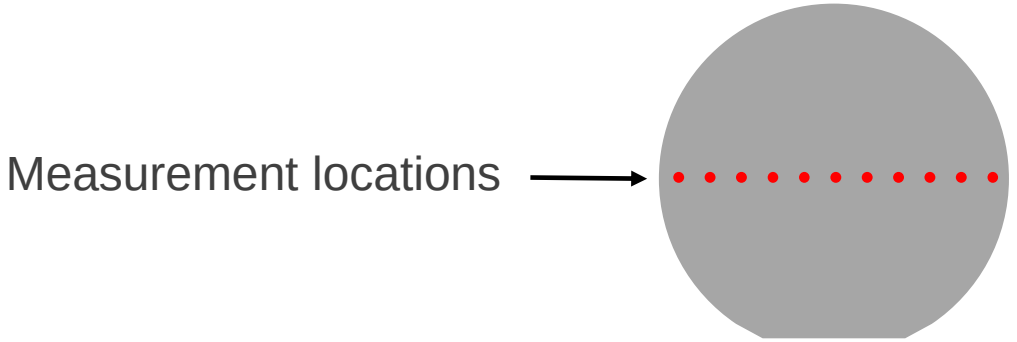
- Each film deposited with varying nitrogen gas flow, resulting in different film stoichiometry
- Determined relationship between *n* and deposition condition
- Best sensitivity in IR: largest variation in *n*
- Can be used for efficient process control

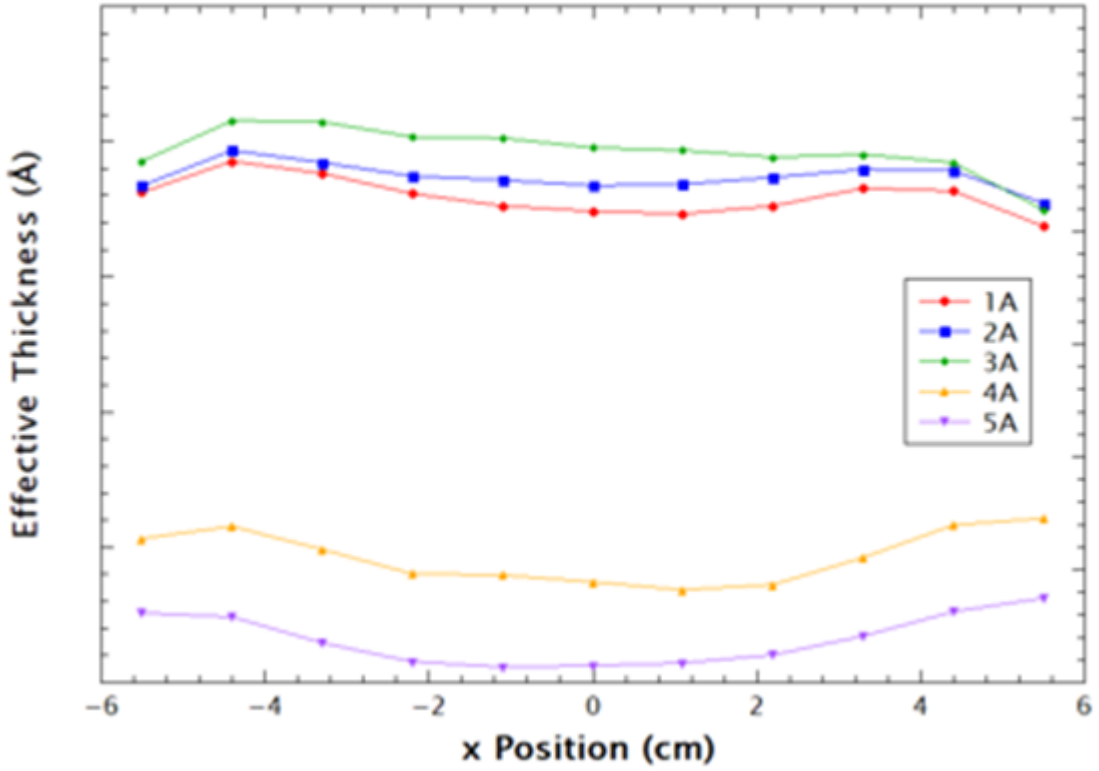
Relationship between optical properties and composition can be used for real-time monitoring or fast post-processing feedback



Example 2: Assessing Film Thickness Uniformity

- For the same project, 11-point line scans were collected across each wafer
- Line scan or full spatial mapping is fully automated instrument feature
- Usually, analysis is straightforward once an appropriate model is developed – track changes in model parameters vs. position





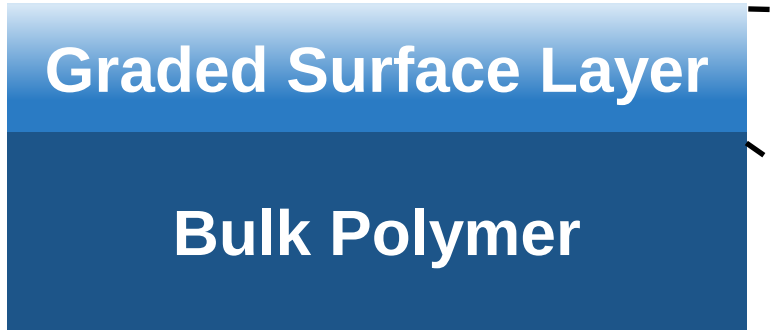
Example 3: Temperature Dependence

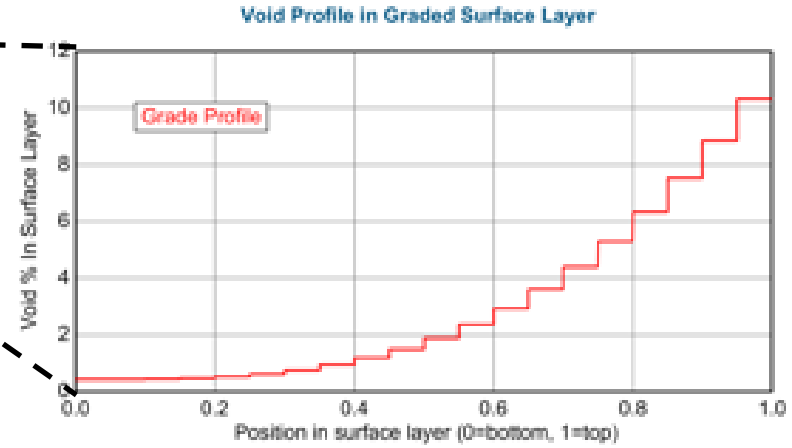
Samples:	Bulk polymer slab for use with high-intensity lasers
Problem:	Would like quantitative understanding of how laser induced-heating affects optical performance
Experiment Design:	• Embed thermocouple directly below sample surface • Use room temp. measurements to build optical model • Collect SE data in real time while preheated sample cools

Example 3: Room Temperature Modeling

- Polymer optical properties not homogeneous near surface
- Room temperature spectroscopic ellipsometery *and* spectroscopic unpolarized transmittance measurements collected to build accurate optical model

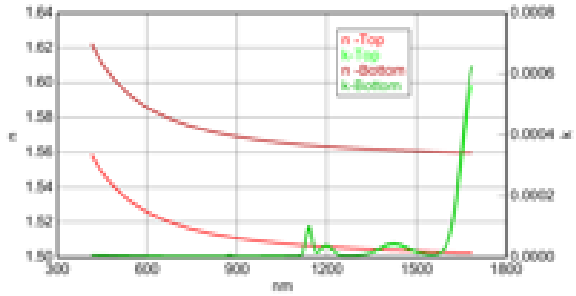
Density decreases closer to the surface

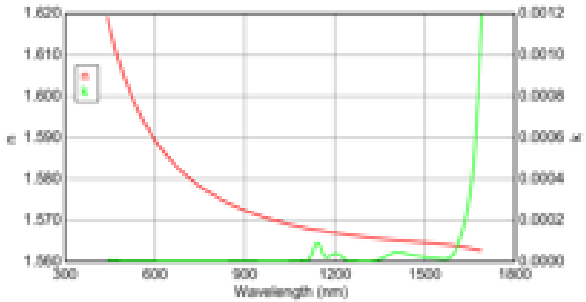




Example 3: Room Temperature Modeling

Optical properties of each layer

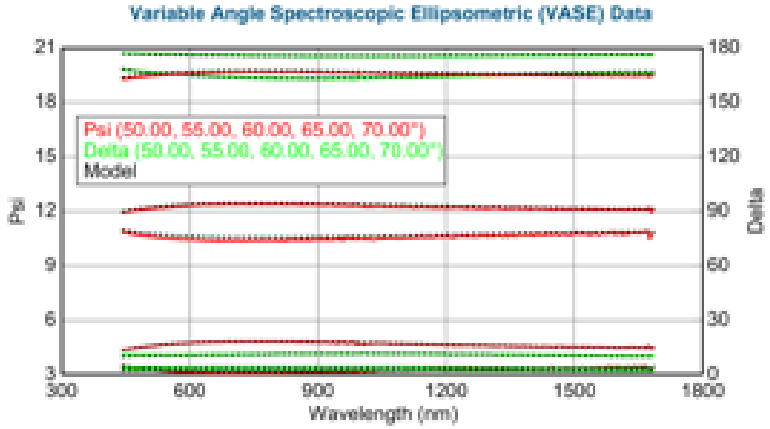


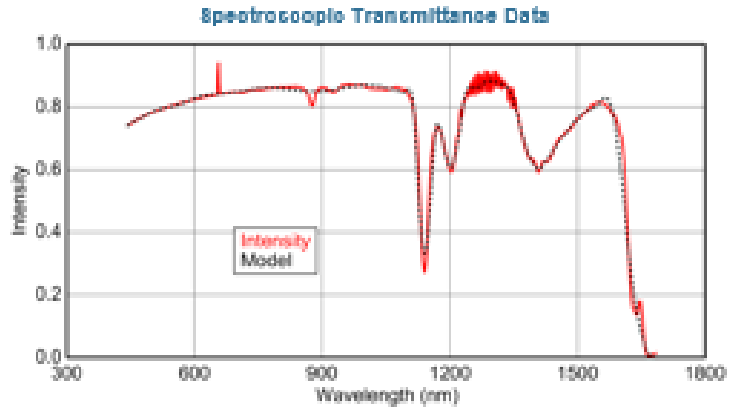


Layer thickness

Graded Surface Layer

Bulk Polymer



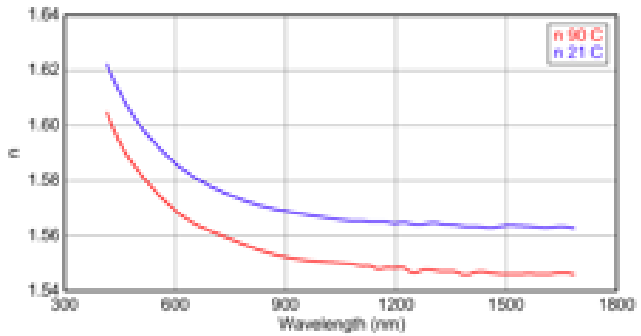


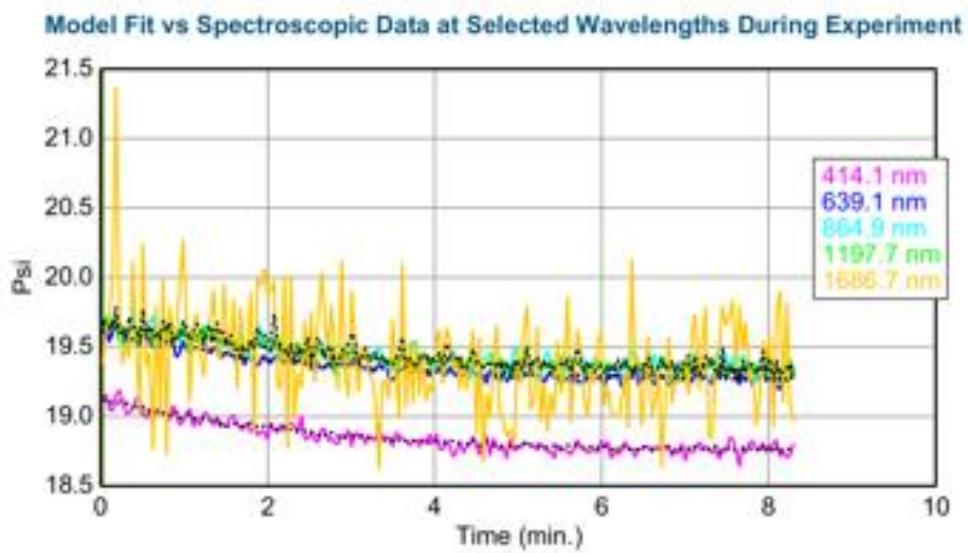
Example 3: Modeling Real Time Measurements

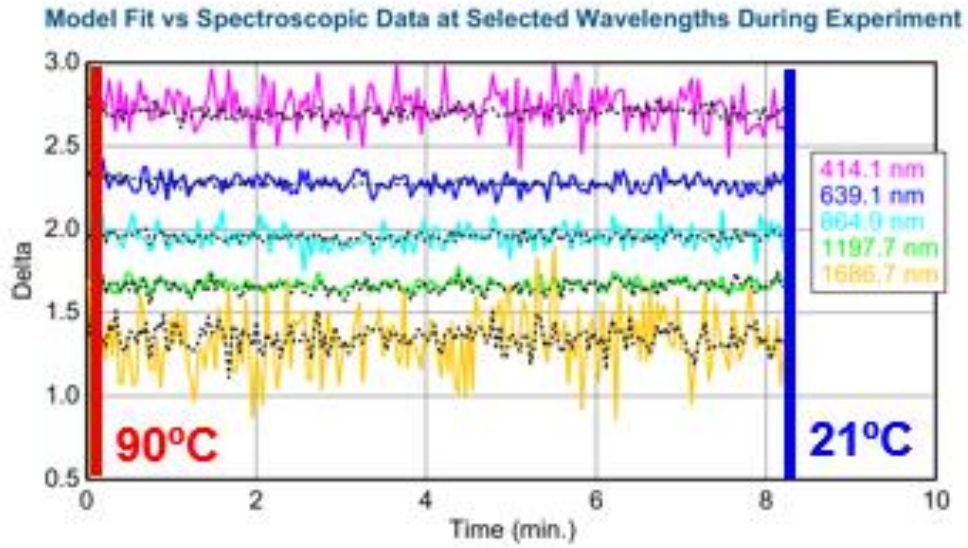
- Fit optical properties of polymer to measurements as sample cools
- Measure temperature over time as sample cools

Graded Surface Layer

Bulk Polymer

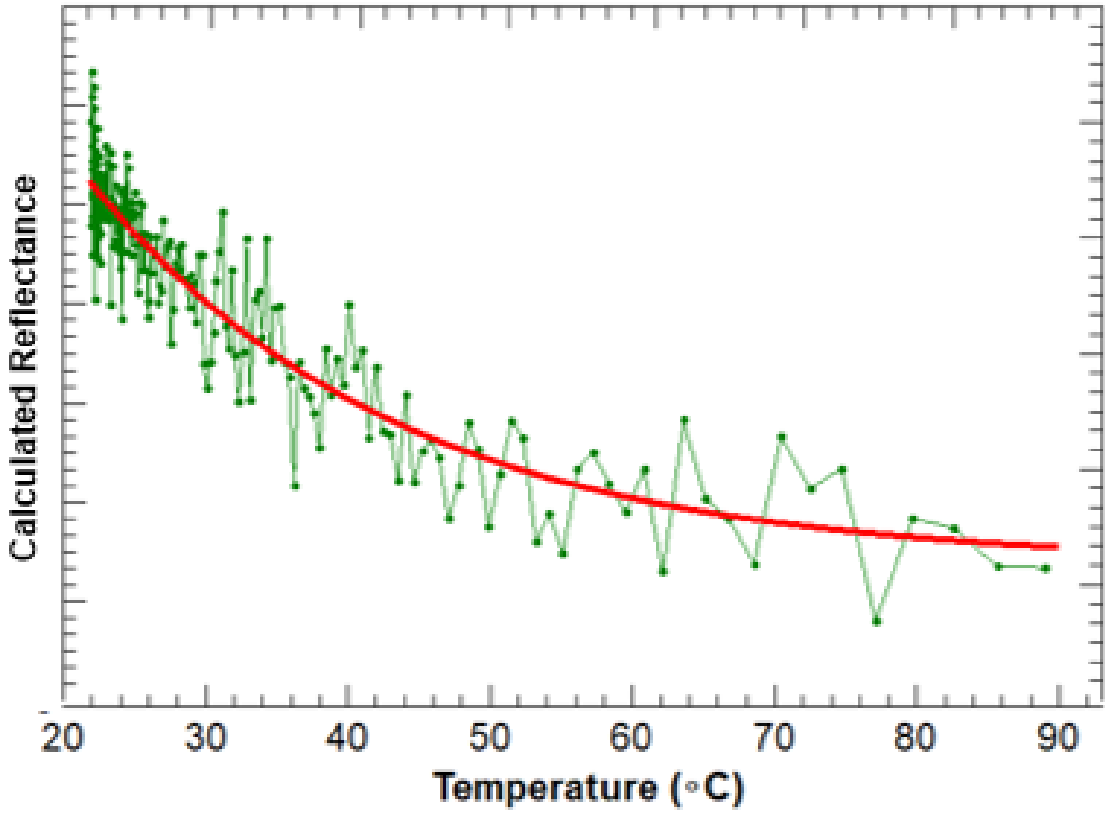






Example 3: Reflectance vs. Temperature

- Sample reflectance can be calculated from optical properties and layer structure
- Here, the reflectance at a specified wavelength of interest is calculated as a function of temperature

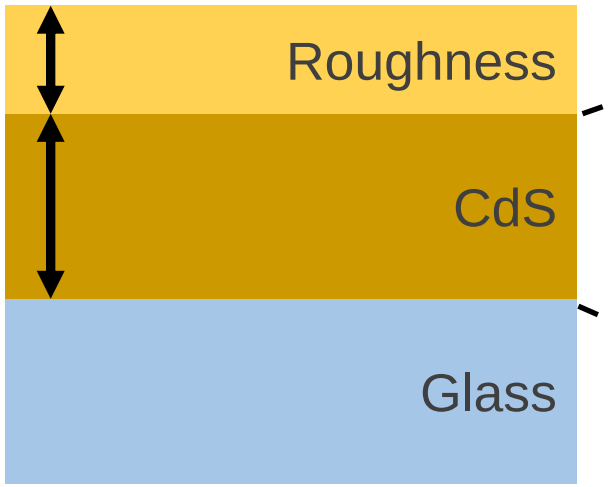


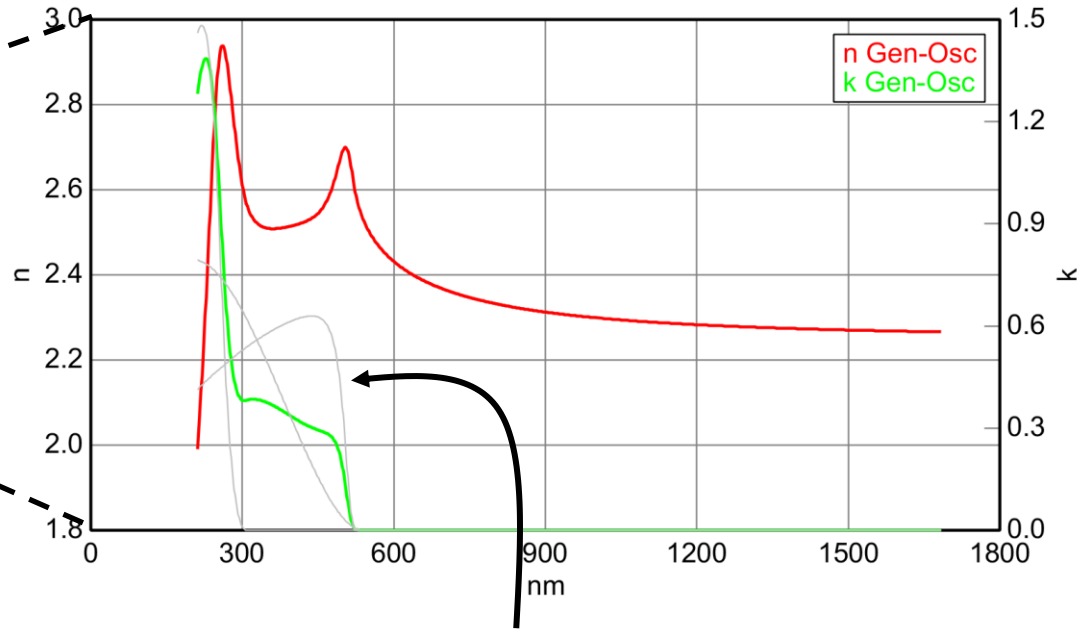
Example 4: Uniformity Mapping

Samples:	CdS thin film
Problem:	Understand how annealing affects spatial uniformity of (1) **film thickness** and (2) **optical properties**
Experiment Design:	• Collect spatial mapping SE data before and after annealing • Develop parametric optical model • Map optical parameters and thickness

Example 4: Parametric Optical Modeling

- Basic model for thin film includes bulk film layer and surface roughness
- With appropriate parametric modeling of optical properties, optical parameters can be mapped





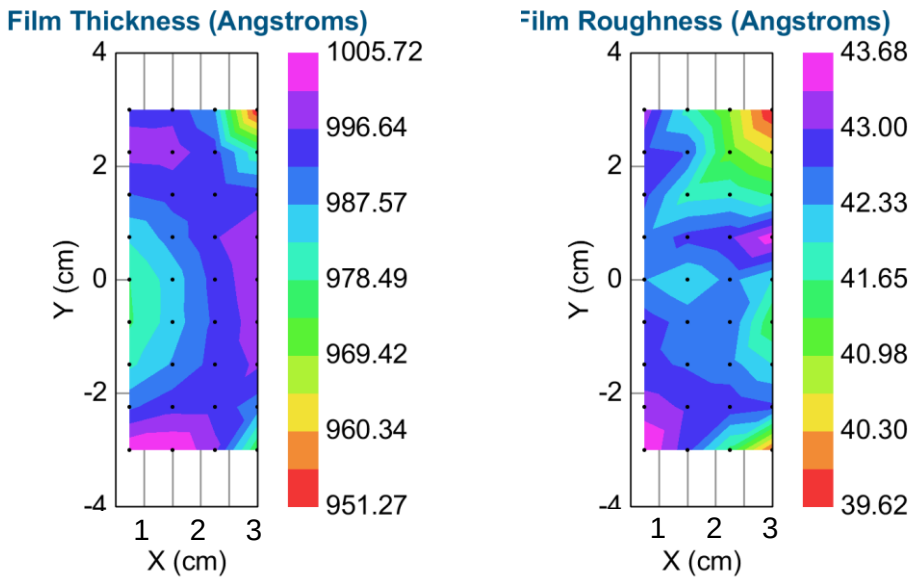
- Each grey line is an individual spectral feature defined by an equation (oscillator)
- Parameters in these equations sometimes have physical meaning and can be mapped

Acknowledgement: Thank you to C. R. Grice (U. Toledo) for the fabrication of this sample

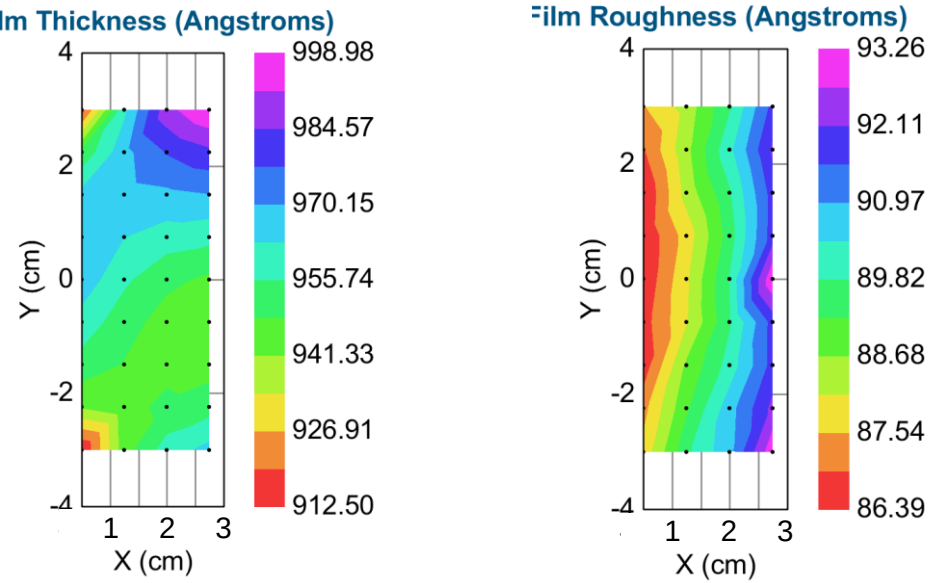
Example 4: Thickness Uniformity Mapping

- Layer thickness spatial mapping is a common application for SE

Pre-Annealing

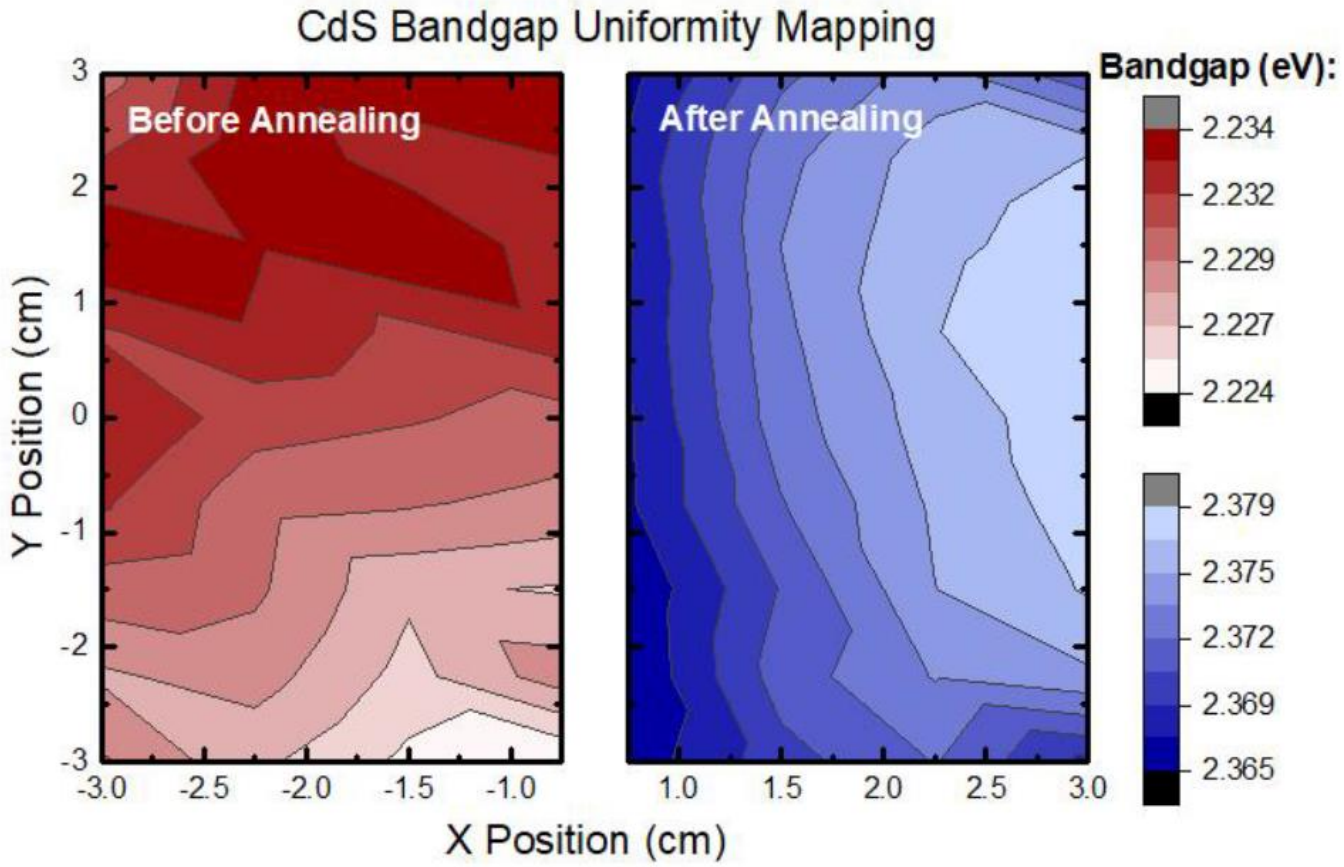


Post-Annealing



Example 4: Optical Uniformity Mapping

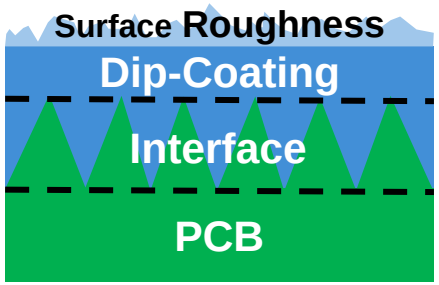
- Mapping can be accomplished with any other optical parameters
- Here, the spatial changes in bandgap upon annealing are mapped
- Reflectance and transmittance can also be calculated from sample structure and mapped



Summarizing These Applications

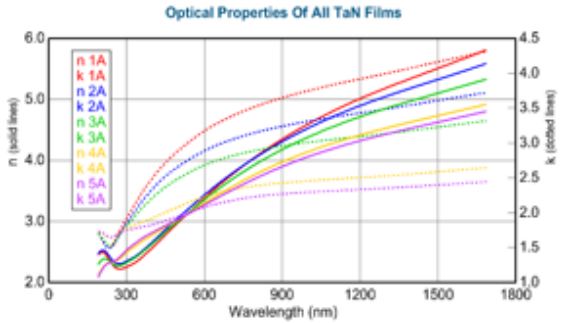
1. Coated Circuit Board

- Buried interfacial character
- Rough surfaces
- Evaluate conformal coating



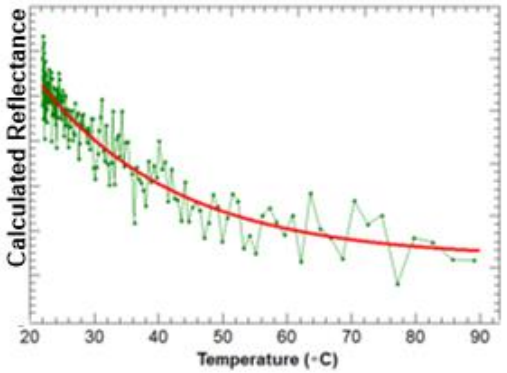
2. Varied Composition TaN

- Compiled database correlating *N* with fabrication condition
- Can be applied to future samples
- Test for process drift



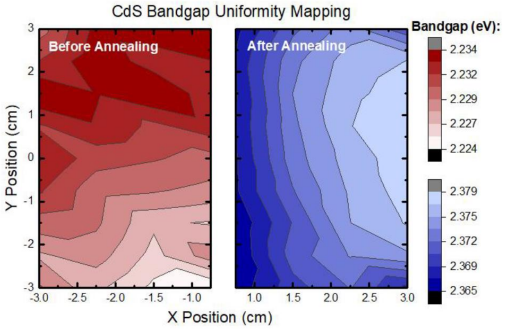
3. Optical Properties vs. Temp.

- Fit SE and Transmittance data simultaneously at room temp.
- Measured in real time as a function of changing temp.
- Determined how reflectance changes as sample is heated



4. Mapping Optical Parameters

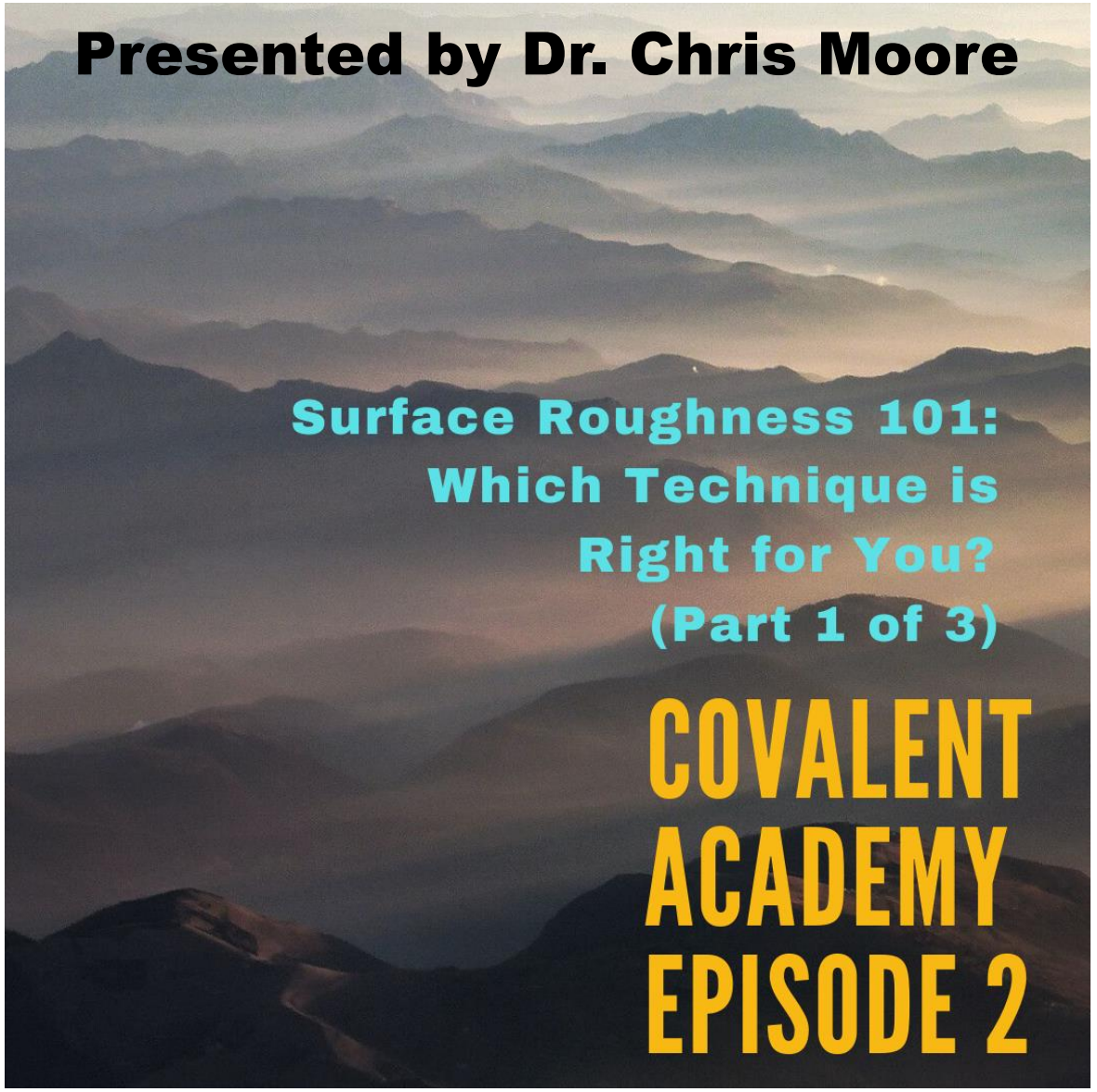
- Before and after processing
- Mapped thickness & roughness
- Mapped uniformity of optical bandgap
- Can be extended to any other optical parameters (e.g. *n*)



Future Episodes

- This episode intended to be high-level overview of basics and select applications
- Future episodes on ellipsometry intended to highlight more applications:
  - Powders
  - Liquids
  - Diffusion
  - Crystal strain
  - Etc.
- Can customize topics based on viewer preferences

Next Week: Episode 2



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