Spectroscopic Ellipsometry from the Vacuum Ultraviolet to the Far Infrared

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Presentation Outline

- Ellipsometry basics. Thin-films.
- Modern Instruments: VUV to far-IR.
- *Ex situ* examples.
- *In situ* examples.
- What’s new?
  - Generalized ellipsometry: anisotropy
  - Depolarization: backside/roughness; Patterned wafers
  - Integrated metrology
Ellipsometry measures the change in polarization state of light reflected from a sample surface.

\[ \rho = \tan(\Psi) e^{i\Delta} = \tan\left(\frac{R_p}{R_s}\right) e^{i(\delta_p - \delta_s)} = \frac{E_p^r}{E_s^r} / \frac{E_p^i}{E_s^i} = \frac{R_p}{R_s} \]
Interaction of Light with Thin Film

- Optical constants $N = n + ik$, or $\varepsilon = \varepsilon_1 + i\varepsilon_2 = N^2$ determine reflected/transmitted intensities, phase change, and angle change.

![Graphs showing reflectance and transmittance for 200nm and 1micron oxide on silicon](image-url)
SE Data Analysis FlowChart

Measurement → Model → Fit → Results

Exp. Data

n,k

Gen. Data

n
n-1

1

0

Compare

Fit Parameters

n,k

Thickness
Roughness
Uniformity
Ellipsometry Advantages

- Measures ratio of two values
  - Highly accurate & reproducible (even at low light levels)
  - No reference necessary
  - Not as susceptible to scatter, lamp or purge variation

- Measures a 'phase', ‘Δ’
  - Very sensitive, especially to ultrathin films (<10 nm)
  - Provides TWO values at each wavelength

- Spectroscopic Ellipsometry (SE)
  - More Information – More Film Properties
  - Data at wavelength of interest (157nm, 193nm, 248nm…)
VASE®

- Spectral Range: 193nm to 1700nm
- Automated Angle
- Variable Angle Spectroscopic Ellipsometry, Transmission and Reflection.

Generated and Experimental Data

<table>
<thead>
<tr>
<th>Energy in eV</th>
<th>Delta in degrees</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0</td>
</tr>
<tr>
<td>1.0</td>
<td>100</td>
</tr>
<tr>
<td>2.0</td>
<td>200</td>
</tr>
<tr>
<td>3.0</td>
<td>300</td>
</tr>
<tr>
<td>4.0</td>
<td>400</td>
</tr>
<tr>
<td>5.0</td>
<td>500</td>
</tr>
<tr>
<td>6.0</td>
<td>600</td>
</tr>
<tr>
<td>7.0</td>
<td>700</td>
</tr>
</tbody>
</table>

- Model Fit
- Exp E 40°
- Exp E 75°

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IR-VASE® Hardware

Spectral Range:
- 2 μm to 33 μm
  (300 to 5000 cm⁻¹)

Drude tail
Si-O Bond absorption
VUV-VASE™ Instrumentation

- Auto Angle: 10°-90°
- Wavelength: 146nm-1100nm
- Nitrogen Purge
- AutoRetarder
- Sample Load Lock
- Automated Sample Alignment
Spectral Choices:
- 193 nm to 1000 nm
- 245 nm to 1000 nm
- 370 nm to 1000 nm

Thickness in Å
Mean = 4283.555
Min = 4209.600
Max = 4331.700
Std Dev = 36.489
Uniformity = 0.85 %

Hundreds of wavelengths
Ex Situ Applications

Ellipsometry is Sensitive to:

- Layer thickness
- Optical Constants
- Surface and Interfacial Roughness
- Composition / Crystallinity
- Optical Anisotropy
- Uniformity (over film area and depth)
- Any physical effect that induces changes in material optical properties
Semiconductor Industry

- Dielectrics (oxides, nitrides, carbides)
- Polymers (Low-Dielectric constant)
- Polysilicon
- Multilayers (ONOPO, SOI, ...)
- Lithography Applications
  - Photoresists
  - Antireflective coatings
  - Photomasks
- Compound Semiconductors
Optical constants depend strongly on crystallinity, which can vary with process conditions.
**Si₃N₄ Film Thickness and Index**

**Initial Modeling Attempts**

**Optical Model #1**

<table>
<thead>
<tr>
<th>Cauchy Layer</th>
<th>2896.2 Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass substrate</td>
<td></td>
</tr>
</tbody>
</table>

MSE = 176.6

**Optical Model #2: Add Roughness**

<table>
<thead>
<tr>
<th>Surface Roughness</th>
<th>108.34 Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cauchy Layer</td>
<td>2878.1 Å</td>
</tr>
<tr>
<td>Glass substrate</td>
<td></td>
</tr>
</tbody>
</table>

MSE = 165.7

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After analysis, we found out film was deposited in two passes!

<table>
<thead>
<tr>
<th>Layer</th>
<th>Thickness (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 Surface Roughness</td>
<td>90.742 Å</td>
</tr>
<tr>
<td>3 Cauchy (same as #1)</td>
<td>1304.8 Å</td>
</tr>
<tr>
<td>2 ‘Interface’ (50% void)</td>
<td>274.84 Å</td>
</tr>
<tr>
<td>1 Cauchy Layer</td>
<td>1324.6 Å</td>
</tr>
</tbody>
</table>

Best Fit Optical Model

Final MSE = 10.31
Organic Film on Si

- **Isotropic Model**

<table>
<thead>
<tr>
<th>Cauchy Layer</th>
<th>7919.3 Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si substrate</td>
<td></td>
</tr>
</tbody>
</table>

MSE = 316.2
Spin-Cast Organic Film on Si

- Uniaxial anisotropy model, optical axis normal to surface

MSE = 16.9

<table>
<thead>
<tr>
<th>aniso-Cauchy</th>
<th>9951.8 Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si substrate</td>
<td></td>
</tr>
</tbody>
</table>

Anisotropic Film Optical Constants

Index of refraction 'n'

Ordinary (in-plane) Index
Extraordinary (out-of-plane) Index
Photoresists

- Measure refractive index at each lithography line
  - 248 nm, 193 nm, 157 nm...

- Monochromator before sample allows accurate optical characterization of resists in UV without exposing.
Multilayer: Resist on ARC

- Both layer thicknesses can be determined because of the DUV data where there is high optical contrast between layers.

<table>
<thead>
<tr>
<th>0</th>
<th>Silicon substrate</th>
<th>1 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>i-line AR Coating</td>
<td>3107.7 Å</td>
</tr>
<tr>
<td>2</td>
<td>Photoresist</td>
<td>6117.9 Å</td>
</tr>
</tbody>
</table>
Resist and ARC Optical Constants

- Similar Dispersion except at shorter wavelengths where both films become absorbing.
Phase Shifting Photomasks

- Complex material structures (index grading)

![Graph showing index of refraction (n) and extinction coefficient (k) vs. distance from substrate in Å]
IR-VASE® Advantages

- **High Sensitivity to Ultra-thin films**
  The "phase" information of Spectroscopic ellipsometry (not available from FTIR reflectance or absorptance) is highly sensitive to ultrathin films.

- **Directly measure accurate Optical Constants**
  No Kramers-Kronig analysis with extrapolation required, as in FTIR.

- **Non-destructive Characterization**
  Measurements do not require vacuum; can study liquid-solid interfaces, e.g. wet-etch, biological, or medical applications.

- **No Baseline or Reference sample required**
  Samples smaller than the beam diameter can be measured because the entire beam does not need to be collected. Ellipsometric measurements are accurate and quantitative. FTIR reflectivity and absorbance measurements are relative.
Silicon Epitaxial Layers on Silicon

- Drude equation used to model free carrier optical absorption

**Measured:**
- Epitaxial Si Layer Thickness ($t_{epi}$) = 273nm
- Native Oxide Thickness ($t_{ox}$) = 1.2nm
- Epitaxial Layer Resistivity ($\rho_{epi}$) = 5.13 $\Omega$-cm
- Substrate Resistivity ($\rho_{sub}$) = 0.0175 $\Omega$-cm

**Model**

<table>
<thead>
<tr>
<th>Layer Type</th>
<th>Thickness/Model</th>
</tr>
</thead>
<tbody>
<tr>
<td>Native Oxide</td>
<td>$t_{ox}$</td>
</tr>
<tr>
<td>Silicon with free carriers</td>
<td>$t_{epi}$</td>
</tr>
<tr>
<td>Silicon with free carriers</td>
<td>0.6 mm</td>
</tr>
</tbody>
</table>

\[\varepsilon_{Drude} \left(E_{ph} = \varepsilon_0 \omega \right) = \frac{-\varepsilon_0 \rho}{\varepsilon_0 \rho \cdot 10^{-2} \left( \frac{\tau \cdot 10^{-15}}{E_{ph}} + i \frac{1}{E_{ph}} \right)} \quad \text{and} \quad \rho = \frac{m^*}{Nq^2 \tau} = \frac{1}{q \mu N}\]
Doping Concentration (resistivity)

- Dopants introduce free-carrier absorption in the Infrared.

![Graph showing Extinction Coefficient 'k' vs Wavelength (µm) for different resistivities (0.05, 0.1, 0.5 ohm-cm)]
Measure epi-layer thickness and substrate resistivity

<table>
<thead>
<tr>
<th>Layer</th>
<th>Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epitaxial Silicon</td>
<td>0.95435</td>
</tr>
<tr>
<td>Silicon Substrate</td>
<td>1</td>
</tr>
</tbody>
</table>

0   Silicon Substrate 1 mm
1 Epitaxial Silicon 0.95435 µm
Epi-silicon: Far-IR data & fit

Generated and Experimental

- Model Fit
- Exp $\Psi-E$ 72°
- Exp $\Delta-E$ 72°
Ion-implanted doping profiles

- Implant-dose: $10^{15} \text{ As-cm}^{-2} @ 80 \text{ keV}$

![Graph showing doping profiles](image)

- RTA model
- RTA exp. data
- Furnace exp. data
- Furnace model
- SRP RTA
- SRP Furnace
- IR-SE RTA - $7.1 \times 10^{14} \text{ cm}^{-2}$
- IR-SE Furnace - $5.6 \times 10^{14} \text{ cm}^{-2}$


SRP: Spreading Resistance Probe
Au Before and After Cleaning

- Sample was removed for cleaning, and remounted for measurement
- Note the high level of both precision and reproducibility in the data
Optical Measurement of Phonon Structure in III-V Semiconductors

- IR-VASE® determined phonon spectra in agreement with published values
- Data fit with simple Restrahlen model; without free-carrier effects
- RCE provides much better data than RAE
In situ Spectroscopic Ellipsometry has been used in a wide variety of applications:

- Sputter Deposition
- Etching (RIE, ECR, …)
- Electrodeposition
- e-Beam Evaporation
- and more...

- Annealing
- MBE
- PVD
- CVD
What Can We Measure *In Situ*?

- Substrate Temperature
- Substrate Surface Quality
- Oxide Thickness and Desorption
- Interfacial regions
- Growth Rates
- Substrate and film optical constants
  - (without surface oxide)
- Alloy Composition
- Multi-layer Thickness
UNL Sputter Chamber

- Sputter chamber retrofit with optical ports for \textit{in-situ} ellipsometry.

- Measure growth rate and optical constants for thin films.
Real-time Thickness Prediction based on last ‘n’ points

- Predict when to turn off process with high precision
Hg$_{1-x}$Cd$_x$Te (MCT) grown by MBE

Purpose / Goals of *in situ* Ellipsometry:

- Monitor Substrate **Temperature** before growth
  - good quality MCT will grow only in a $10^\circ$ temperature window

- Monitor Substrate **Surface Quality** before growth
  - the substrate is heated to desorb the oxide

- Monitor and *Control* the MCT **Composition**
  - IR devices *require* a composition accuracy of ±0.001!
Composition Accuracy

- 6 independent MCT growth runs (without control)
  - post-deposition *in situ* ellipsometry analysis

\[ y = 1.012x - 0.0038 \]
Long Term Run-to-Run InGaAs Composition Accuracy

- Std. Dev. in SE composition error of \(\pm 0.002\) achieved over a 6 month period (at ASU)

InGaAs Composition Comparison: \textit{in situ} SE vs. XRD

- Composition tolerance to achieve lattice match
Generalized Ellipsometry...

- Does not assume zero off-diagonal components for the Jones matrix. Uses the following relationship...

\[
\begin{bmatrix}
    p_{out} \\
    s_{out}
\end{bmatrix} =
\begin{bmatrix}
    r_{pp} & r_{sp} \\
    r_{ps} & r_{ss}
\end{bmatrix}
\begin{bmatrix}
    p_{in} \\
    s_{in}
\end{bmatrix}
\]

- Thus, a generalized sample is described by...
  - AnE
    \[
    \tan(\Psi) \cdot e^{i\Delta} = \frac{r_{pp}}{r_{ss}}
    \]
  - Aps
    \[
    \tan(\Psi_{ps}) \cdot e^{i\Delta_{ps}} = \frac{r_{ps}}{r_{pp}}
    \]
  - Asp
    \[
    \tan(\Psi_{sp}) \cdot e^{i\Delta_{sp}} = \frac{r_{sp}}{r_{ss}}
    \]
Depolarization Measurements

- Requires AutoRetarder™ or Rotating Compensator (e.g. M-2000®)

- Potential Applications
  - transparent substrates (back-side effects)
  - patterned samples
  - layer thickness non-uniformity
  - finite spectral bandwidth
Patterened Samples (depolarization)

<table>
<thead>
<tr>
<th></th>
<th>oxide_final</th>
<th>98.752 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>oxide_final</td>
<td>1.8176 nm</td>
</tr>
<tr>
<td>0</td>
<td>si_final_tabulated</td>
<td>1 mm</td>
</tr>
</tbody>
</table>

\[ \text{MSE} = 4.605 \]

\[ \text{ThkUni} = 14.03 \text{ (% of unetched area in beam)} \]

Conclusions

- 142 nm to 200 micron spectral range.
- Variable angle; focusing; mapping; monochromator or diode-based; polarization control: autoretarder.
- Ex situ applications:
  - oxides, poly-silicon, stacks, photoresists, surface contamination/cleaning, optical constants.
  - Advanced applications: graded layers, anisotropy, depolarization, patterned material.
- In situ monitor/control of:
  - Growth/etch, thickness, temperature, composition