Optical Metrology of Low-κ Dielectrics

Phillip Walsh

n&k Technology, Inc.

Santa Clara, CA

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Outline

- Optical Thin Film Metrology
  - Hardware
  - Analysis Algorithms
- Correlation of Optical Properties With Thin Film Processing Conditions
- Correlation of Refractive Index With Dielectric Constant
- Examples of Low-κ Applications
  - Porous MSQ
  - SiLK
  - Inhomogenous Low-κ Films on Cu and W
  - Characterization of Plasma-Induced Damage of Treated Low-κ Films
- Conclusions
Optical System

- **Method:** Broadband Reflectometry
- **Spectral Range:** 190 nm – 1000 nm
- **Optical Design:** All-Reflective Optics

![n&k Analyzer 3300](image)

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Optical System
Analysis Model

Forouhi–Bloomer Dispersion Relations:

\[ k(E) = \sum_{i=1}^{q} \frac{A_i (E - E_g)^2}{E^2 - B_i E + C_i} \]

\[ n(E) = n(\infty) + \sum_{i}^{q} \frac{B_0_i E + C_{0i}}{E^2 - B_i E + C_i} \]

The “n&k Method” is an accurate, reproducible thin film characterization technique, that incorporates the Forouhi-Bloomer dispersion relations for \( n(\lambda) \) and \( k(\lambda) \), along with a parameterized models for inhomogeneity and interface roughness, into Fresnel equations to generate theoretical Reflectance spectrum \( R_{\text{theor}} \) in terms of \( d, n_f(\lambda), k_f(\lambda), E_g, n_s(\lambda), k_s(\lambda), \sigma_1(\text{top}) \text{ and } \sigma_2(\text{bottom}) \), whereby the wavelength and material dependence of \( n(\lambda) \) and \( k(\lambda) \) are obtained from the Forouhi-Bloomer formulation.
Porous-MSQ

4032 Å Porous-MSQ / Si-Sub

Measured

Calculated

Wavelength (nm)

Reflectance

0.0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0

200 300 400 500 600 700 800 900 1000

2D Thickness Uniformity Map

3D Thickness Uniformity Map

Porous-MSQ, \( K = 2.2 \)

Wavelength (nm)

n and k

n

k

200 300 400 500 600 700 800 900 1000

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Correlation: The higher the values of $k$ in the DUV, the higher the Si-to-N ratio of the Si$_N$$_x$ films

$n$ and $k$ Spectra of Si$_3$N$_4$ and various Si$_N$$_x$ films
Materials Characterization
Polycrystalline $\text{Si}_x\text{Ge}_{1-x}$ (10% Ge)

Reflectance

2D Thickness
Uniformity Map

3D Thickness
Uniformity Map
Materials Characterization
Polycrystalline Si$_x$Ge$_{1-x}$ (15% Ge)

Reflectance

Wavelength (nm)

Poly-SiGe (15% Ge) / 1000 Å SiO2 / Si-Sub

Measured
Calculated

2D Thickness
Uniformity Map

3D Thickness
Uniformity Map

n and k

Poly - SiGe (15% Ge)

Wavelength (nm)
Materials Characterization
Polycrystalline Si$_x$Ge$_{1-x}$ (20% Ge)

**Reflectance**

- 1082 Å Poly-SiGe (20% Ge) / 1000 Å SiO2 / Si-Sub
- Measured vs Calculated

**n and k**

- Poly - SiGe (20% Ge)
- n vs k

2D Thickness Uniformity Map

3D Thickness Uniformity Map
Materials Characterization
Polycrystalline Si$_x$Ge$_{1-x}$

![Graph 1: Poly SiGe Concentration vs. Wavelength](image1)

![Graph 2: Poly SiGe Concentration vs. Wavelength](image2)
SELETE 65nm Node Program
Source: http://www.selete.co.jp

Process Modules to be developed by Asuka

- Transistor Module (FEP)
- 65 nm Lithography (157nm, EPL) Mask
- Multi-layer Interconnect (BEP)

SiGe metal gate
High $\kappa$ gate dielectric
Low resistance source/drain
Trench isolation with a low stress

Cu interconnect
Low $\kappa$ Inter-metallic dielectric
CMP
Low resistance contact with a high aspect ratio

Semiconductor Leading Edge Technologies, Inc.

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Relationship Between Dielectric Constant, $\kappa$, and Refractive Index, $n$

- Complex Index of Refraction: $N(E) = n(E) - ik(E)$
- Complex Dielectric Function: $\varepsilon(\nu) = \varepsilon_1(\nu) - i\varepsilon_2(\nu)$

$\varepsilon = N^2$

Therefore:

$\varepsilon_1 = n^2 - k^2$

$\varepsilon_2 = 2nk$

- Dielectric Constants: $\kappa = \lim_{\nu \to 0} \varepsilon_1(\nu)$

Thus: $\kappa \sim n^2$

$v$ (frequency), $E$ (Energy), and $\lambda$ (wavelength) are used interchangeably in this presentation.
# Classification of Low-κ Dielectric

(Slide adapted from a presentation by Dr. Osamu Aoki, JSR Microelectronics, Inc., USA.)

<table>
<thead>
<tr>
<th>Material</th>
<th>κ = 4.1</th>
<th>κ = 3.5</th>
<th>κ = 2.6 – 3.2</th>
<th>κ = 2.0 – 2.5</th>
<th>Process</th>
<th>Vendor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inorganic</td>
<td>SiO2</td>
<td>SiO2:F</td>
<td>FOx</td>
<td>XLX</td>
<td>CVD</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>CVD</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Spin-on</td>
<td>Dow Corning</td>
</tr>
<tr>
<td>Hybrid</td>
<td>Dense-MSQ</td>
<td>Porous-MSQ</td>
<td>Sp-in-on</td>
<td>Sp-in-on</td>
<td>JSR</td>
<td>Applied Novellus</td>
</tr>
<tr>
<td></td>
<td>BlackDiamond CORAL</td>
<td>BlackDiamond POLA</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Organic</td>
<td>SiLK</td>
<td>Nautilus</td>
<td>Spin-on</td>
<td>Dow Chemical</td>
<td>Honeywell</td>
<td></td>
</tr>
<tr>
<td></td>
<td>FLARE</td>
<td>FLARE</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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Comparison of $n$ Spectra for Various Low-$\kappa$ Materials

Refractive Index, $n$

SiO$_2$

SiO$_2$:F

Dense-MSQ

Low-$k$

Porous-MSQ

Porous-MSQ < Low-$k$ < Dense-MSQ

2.2 < Dielectric Constant < 3
**Dense-MSQ**

4043 Å Dense-MSQ / Si-Sub

- Measured
- Calculated

2D Thickness Uniformity Map

Dense-MSQ, $\kappa \approx 3.0$

2D Thickness Uniformity Map

**n and k**

Wavelength (nm)

Reflectance

Wavelength (nm)

n

k

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Porous-MSQ

4032 Å Porous-MSQ / Si-Sub

Measured
Calculated

Porous-MSQ, $K = 2.2$

2D Thickness Uniformity Map

3D Thickness Uniformity Map
Correlation Between $n$ and Porosity

$n$ at 633nm versus Porosity

$y = -0.4022x + 1.3968$

$R^2 = 0.9922$
Correlation Between $n^2$ and $\kappa$

\[ y = 2.6317x - 1.9049 \]
\[ R^2 = 0.9737 \]
The following set of SiLK samples were provided to n&k Technology for characterization.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cure Temp / Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>320 °C / 90 s</td>
</tr>
<tr>
<td>2</td>
<td>420 °C / 120 s</td>
</tr>
<tr>
<td>3</td>
<td>440 °C / 120 s</td>
</tr>
<tr>
<td>4</td>
<td>440 °C / 120 s</td>
</tr>
<tr>
<td>5</td>
<td>440 °C / 120 s</td>
</tr>
<tr>
<td>6</td>
<td>470 °C / 120 s</td>
</tr>
</tbody>
</table>

SiLK on samples 3, 4, and 5 was processed identically.
SiLK

Dependence of k on Curing Conditions

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cure Temp / Time</th>
<th>Thickness (Å)</th>
<th>Band Gap Eg (eV)</th>
<th>Extinction Coefficient k (at 330nm)</th>
<th>Index of Refraction n (at 314 nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>320 °C / 90s</td>
<td>5133</td>
<td>3.86</td>
<td>0.009</td>
<td>1.969</td>
</tr>
<tr>
<td>2</td>
<td>420 °C / 90s</td>
<td>5188</td>
<td>3.77</td>
<td>0.025</td>
<td>1.925</td>
</tr>
<tr>
<td>3</td>
<td>440 °C / 90s</td>
<td>5192</td>
<td>3.70</td>
<td>0.034</td>
<td>1.910</td>
</tr>
<tr>
<td>4</td>
<td>440 °C / 90s</td>
<td>5191</td>
<td>3.69</td>
<td>0.034</td>
<td>1.909</td>
</tr>
<tr>
<td>5</td>
<td>440 °C / 90s</td>
<td>5200</td>
<td>3.70</td>
<td>0.033</td>
<td>1.912</td>
</tr>
<tr>
<td>6</td>
<td>470 °C / 90s</td>
<td>520.7</td>
<td>3.62</td>
<td>0.046</td>
<td>1.900</td>
</tr>
</tbody>
</table>

- Curing condition of SiLK films are conventionally monitored through determination of n at 314 nm
- Correlation is significantly improved when considering k-spectra from 320 - 380 nm range

Plots of the extinction coefficient k of six spin-on polymer films near the UV absorption edge demonstrate the uniform increase of k with cure cycle in this spectral region. The data taken at the wafer centers are illustrated by the solid curves. The range of within-wafer variations are shown by the crosses.
For the demonstration of this capability two samples with the following nominal film structures were measured:

**Case Study 1:** Black Diamond™ (3000 Å) / SiC (500 Å) / Cu (3000 Å) / Si Substrate  
**Case Study 2:** Black Diamond™ (3300 Å) / SiC (500 Å) / W (3000 Å) / Si Substrate

In order to analyze the samples described above we first had to characterize underlying SiC, Cu and W films. For this purpose, two background samples of the following nominal film structures were provided:

**Background Sample 1:** SiC (500 Å) / Cu (3000 Å) / Si Substrate  
**Background Sample 2:** SiC (500 Å) / W (3000 Å) / Si Substrate

As the results of the analysis of these samples we were able to obtain $n$ and $k$ spectra for SiC film, Cu and W films.
Low-κ Black Diamond™ Film
Detection of Inhomogeneity

For the characterization of the optical properties of the Black Diamond™ film, a single-layer Black Diamond™ on Silicon Substrate background sample was provided. Immediately upon inspection of the Reflectance spectrum it became evident that the film is inhomogeneous, i.e. optical properties of the film change with depth.

**Calculation 1**
Homogeneous Black Diamond™ Model
GOF = 94.76%

**Calculation 2**
Inhomogeneous Black Diamond™ Model
GOF = 98.16%

**Figure 2A.** Plots of measured (red) and calculated (green) Reflectance spectra versus Wavelength (nm).

**Figure 2B.** Plots of measured (red) and calculated (green) Reflectance spectra versus Wavelength (nm).
The most physically plausible explanation of the bad fit is a possible presence of an extra layer in the film stack. A new computational model for analysis was developed, where on top of the inhomogeneous Black Diamond™ layer an ultra-thin “terminating layer” was placed.

Inhomogeneous Black Diamond™ Model with the Ultra-Thin Terminating Layer.

Goodness of Fit: 99.35%
Low-\(\kappa\) Films Deposited on Cu and W

Case Study 1: (Terminating Layer / Inhomogeneous Black Diamond\(^\text{TM}\) / SiC / Cu / Si Substrate)

**Figure 6A.** Thickness uniformity map of the SiC layer.

**Figure 6B.** Thickness uniformity map of the inhomogeneous Black Diamond\(^\text{TM}\) layer.

**Figure 6C.** Thickness uniformity map of the ultra-thin terminating layer.
Low-$κ$ Films Deposited on Cu and W

Case Study 2: (Terminating Layer / Inhomogeneous Black Diamond™ / SiC / W / Si Substrate)

Figure 7A. Thickness uniformity map of the SiC layer.

Figure 7B. Thickness uniformity map of the inhomogeneous Black Diamond™ layer.

Figure 7C. Thickness uniformity map of the ultra-thin terminating layer.
The next challenge was to measure a similar film structure on a patterned wafer. For this purpose, a patterned sample of the following nominal film structure was provided:

**Patterned Sample: Black Diamond™ (7700Å) / SiC (500Å) / Cu (3000Å) / Si Substrate**

**Figure 8A.** Thickness uniformity map of the SiC layer.

**Figure 8B.** Thickness uniformity map of the inhomogeneous Black Diamond™ layer.

**Figure 8C.** Thickness uniformity map of the ultra-thin terminating layer.
Two SEM cross section scans were performed on the same wafer: one at a location near the center of the wafer, and another one at the location near the edge.

Both cross sections closely correspond to the results obtained using n&k Analyzer (7706.4Å at the center and 7691.2Å at the edge, using n&k Analyzer; 7710Å at the center and 7690Å at the edge, using SEM).
Low-κ Damage Due To Ashing

- Plasma damage during ashing, etching, etc. densifies top layer of the low-κ film.
- This results in two layers, consisting of a thin damaged layer on top of the original low-κ film.
- Generally, densification causes an increase in $n$ for the damaged layer.
Low-κ Damage Due To Ashing

**Sample Split**

<table>
<thead>
<tr>
<th>Ashing Chemical</th>
<th>Remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No Treatment</td>
</tr>
<tr>
<td>2</td>
<td>He/H2 Ashing</td>
</tr>
<tr>
<td>3</td>
<td>NH3 Ashing</td>
</tr>
<tr>
<td>4</td>
<td>O2 Ashing</td>
</tr>
<tr>
<td></td>
<td>Ref.</td>
</tr>
<tr>
<td></td>
<td>Low Damage Ashing (Selete)</td>
</tr>
<tr>
<td></td>
<td>Low Damage Ashing</td>
</tr>
<tr>
<td></td>
<td>Conventional Ashing</td>
</tr>
</tbody>
</table>

Low-κ Damage Due To Ashing
n&k Analyzer Measurements

Single Layer-Analysis on Plasma-treated Low-k

Low-$\kappa$ Damage Due To Ashing
n&k Analyzer Measurements

2 Layer-Analysis on Plasma-treated Low-$\kappa$

<table>
<thead>
<tr>
<th>Goodness of Fit</th>
<th>$E_\gamma (\mathrm{eV})$ - 6.42 eV</th>
</tr>
</thead>
<tbody>
<tr>
<td>VL (nm)</td>
<td>193 248 365 633</td>
</tr>
<tr>
<td>$n$</td>
<td>1.375 1.355 1.341 1.335</td>
</tr>
<tr>
<td>$k$</td>
<td>0.000 0.000 0.000 0.000</td>
</tr>
<tr>
<td>Refract. %</td>
<td>60.2 60.3 44.3 19.3</td>
</tr>
<tr>
<td>Absorb. %</td>
<td>65.5 65.5 44.6 18.4</td>
</tr>
</tbody>
</table>

Film Structure
Thickness (Å)

<table>
<thead>
<tr>
<th>Damaged Layer</th>
<th>243</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low-$\kappa$</td>
<td>3597</td>
</tr>
<tr>
<td>Si</td>
<td>1.000 nm (F)</td>
</tr>
</tbody>
</table>

Unknown Material: previous result
Recipe Datas: +/- 1000 Å, 0.0 nm, Film, Energy

$N_{\text{damaged}}$ (1.335) > $n_{\text{bulk}}$ (1.213)

Low-$k$ Damage Due To Ashing

n&k Analyzer Measurements

Low-κ Damage Due To Ashing
n&k Analyzer Measurements

Low-\(\kappa\) Damage Due To Ashing

n&k Analyzer Measurements

Relative Damage: \((n_{\text{damaged}} - n_{\text{ref.}}) \times \text{Thickness}_{\text{damaged}}\)

Low-κ Films Deposited on Cu and W
Measurements on Patterned Wafers

Note that the terminating layer in the previous Black Diamond example is consistent with the He/H2 ashing condition:

**Patterned Sample: Black Diamond™ (7700A) / SiC (500A) / Cu (3000A) / Si Substrate**

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**Figure 8A.** Thickness uniformity map of the SiC layer.

**Figure 8B.** Thickness uniformity map of the inhomogeneous Black Diamond™ layer.

**Figure 8C.** Thickness uniformity map of the ultra-thin terminating layer.
Conclusions

- Can use optical metrology to:
  - Rapidly, nondestructively, and simultaneously determine film thickness, $n$, and $k$
  - Correlate $n$ and $k$ to film processing conditions
- Optical metrology of low-$\kappa$ films:
  - Correlation of $n$ to dielectric constant
  - Porous films
  - Inhomogeneous films
  - Films on metallic substrates
  - Characterization of plasma-induced damage