

Optical Metrology of Low-к Dielectrics

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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-*k* 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



Optical System





Analysis Model

Forouhi–Bloomer Dispersion Relations:

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

$$n(E) = n(\infty) + \sum_{i}^{q} \frac{B_{0_{i}}E + C_{0_{i}}}{E^{2} - B_{i}E + C_{i}}$$

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_{theor}) in terms of d, $n_f(\lambda)$, $k_f(\lambda)$, E_g , $n_s(\lambda)$, $k_s(\lambda)$, $\sigma_1(top)$ and $\sigma_2(bottom)$, whereby the wavelength and material dependence of $n(\lambda)$ and $k(\lambda)$ are obtained from the Forouhi-Bloomer formulation.



Porous-MSQ



Materials Characterization Si-to-N Ratio in SiN_x Films





Materials Characterization Polycrystalline Si_xGe_{1-x} (10% Ge)



Materials Characterization Polycrystalline Si_xGe_{1-x} (15% Ge)



Materials Characterization Polycrystalline Si_xGe_{1-x} (20% Ge)



Materials Characterization Polycrystalline Si_xGe_{1-x}





SELETE 65nm Node Program

Source: http://www.selete.co.jp





Relationship Between Dielectric Constant, *κ*, and Refractive Index, *n*

- Complex Index of Refraction:
- Complex Dielectric Function:

$$N(E) = n(E) - ik(E)$$
$$\varepsilon(v) = \varepsilon_1(v) - i\varepsilon_2(v)$$

 $\varepsilon = N^2$

Therefore:

$$\begin{aligned} \varepsilon_1 &= n^2 - k^2 \\ \varepsilon_2 &= 2nk \end{aligned}$$

• Dielectric Constants: $\kappa = \lim_{v \to 0} \varepsilon_1(v)$ Thus: $\kappa \sim n^2$

v (frequency), *E* (Energy), and λ (wavelength) are used interchangeably in this presentation.



Classification of Low-*k* **Dielectric**

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

| Material | <i>κ</i> = 4.1 | <i>κ</i> = 3.5 | $\kappa = 2.6 - 3.2$ | $\kappa = 2.0 - 2.5$ | Process | Vendor |
|-----------|----------------|----------------|----------------------|----------------------|---------|--------------|
| Inorganic | SiO2 | | | | CVD | - |
| | | SiO2:F | | | CVD | - |
| | | | FOx | XLX | Spin-on | Dow Corning |
| Hybrid | | | Dense-MSQ | Porous-MSQ | Spin-on | JSR |
| | | | BlackDiamond | BlackDiamond | CVD | Applied |
| | | | CORAL | POLA | CVD | Novellus |
| Organic | | | SiLK | Nautilus | Spin-on | Dow Chemical |
| | | | FLARE | FLARE | Spin-on | Honeywell |



Comparison of *n* Spectra for Various Low- κ Materials





Dense-MSQ



Porous-MSQ



Correlation Between *n* and **Porosity**

n at 633nm versus Porosity





Correlation Between n^2 and κ





SiLK

The following set of *SiLK* samples were provided to n&k Technology for characterization.

| Sample | Cure | | |
|--------|----------------------------|--|--|
| | Temp / Time | | |
| 1 | 320 ^O C / 90 s | | |
| 2 | 420 ^O C / 120 s | | |
| 3 | 440 ^O C / 120 s | | |
| 4 | 440 ^O C / 120 s | | |
| 5 | 440 ^O C / 120 s | | |
| 6 | 470 ^O C / 120 s | | |

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



SiLK

Dependence of k on Curing Conditions

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

• 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

0.07 0.06 Extinction Coefficient, k Temp / Time Wafer 320°C/ 90 s. 0.05 420°C/ 120 s 0.04 440°C/120 s 345 470°C/120 s 0.03 0.02 0.01 0.00 340 320 330 350 360 370 Wavelength (nm)

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.



Low- κ Films Deposited on Cu and W

For the demonstration of this capability two samples with the following nominal film structures were measured:

Case Study 1: Black DiamondTM (3000Å) / SiC (500Å) / Cu (3000Å) / Si Substrate **Case Study 2:** Black DiamondTM (3300Å) / SiC (500Å) / W (3000Å) / 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 (500Å) / Cu (3000Å) / Si Substrate **Background Sample 2:** SiC (500Å) / W (3000Å) / 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 singlelayer 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 DiamondTM Model

Calculation 2 Inhomogeneous Black DiamondTM Model

GOF = 94.76%





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).



Low-κ Black Diamond[™] Film Detection of Ultra-Thin Terminating Layer

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 DiamondTM Model with the Ultra-Thin Terminating Layer.



Goodness of Fit: 99.35%





Low- κ Films Deposited on Cu and W

Case Study 1: (Terminating Layer / Inhomogeneous Black DiamondTM / SiC / Cu / Si Substrate)



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



Figure 6B. Thickness uniformity map of the inhomogeneous Black DiamondTM 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 DiamondTM / 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.



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

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 DiamondTM (7700A) / SiC (500A) / Cu (3000A) / 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.



Low-K Films Deposited on Cu and W SEM Comparison

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



Figure 9A. Thickness uniformity map of the inhomogeneous Black DiamondTM layer.



Figure 9B. SEM cross section image at a location close to the center of the wafer.



Figure 9C. SEM cross section image at a location close to the edge of the wafer.

Both cross sections closely correspond to the results obtained using n&k Analyzer (7706.4A at the center and 7691.2A at the edge, using n&k Analyzer; 7710A at the center and 7690A at the edge, using SEM).



Low-к 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.



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Low-к Damage Due To Ashing

Sample Split

| | Ashing Chemical | Remark |
|---|-----------------|-------------------------------|
| 1 | No Treatment | Ref. |
| 2 | He/H2 Ashing | Low Damage Ashing (Selete) |
| 3 | NH3 Ashing | Low Damage Ashing |
| 4 | O2 Ashing | Conventional Ashing |

Ref: Katsumi Yoneda (SELETE), presented at Semicon Japan, December, 2004.



Single Layer-Analysis on Plasma-treated Low-k

| Goodness | of Fit | = 0.9835 | Eg(V) = | 6.49eV |
|----------|--------|----------|---------|--------|
| WL (nm): | 193 | 248 | 365 | 633 |
| n : | 1.304 | 1.283 | 1.269 | 1.262 |
| k : | 0.001 | 0.000 | 0.000 | 0.000 |
| Rexp %: | 68.2 | 68.3 | 44.3 | 19.3 |
| Rcal %: | 66.4 | 66.9 | 43.7 | 17.8 |
| | | | | |

| Film Structure Thio | e ckness (Å) |
|------------------------|-----------------|
| Low-k | 3693 |
| Si Substrate | 1.000 mm (F) |

Unknown Material: previous result Recipe Details: +/- 1000 A, 0.0 nm, Film, Energy



Ref: Katsumi Yoneda (SELETE), presented at Semicon Japan, December, 2004.



2 Layer-Analysis on Plasma-treated Low-k

| Goodness | of Fit | = 0.9953 | Eg(V) = | 6.49eV | |
|----------|--------|----------|---------|--------|--|
| WL(nm): | 193 | 248 | 365 | 633 | |
| n : | 1.375 | 1.355 | 1.341 | 1.335 | |
| k : | 0.000 | 0.000 | 0.000 | 0.000 | |
| Rexp %: | 68.2 | 68.3 | 44.3 | 19.3 | |
| Rcal %: | 68.5 | 68.9 | 44.6 | 19.4 | |

Low-k 3597 Si 1.000 mm (F) Substrate

Film Structure

Damaged Layer 243

Thickness (Å)

Unknown Material: previous result Recipe Details: +/- 1000 A, 0.0 nm, Film, Energy



Ref: Katsumi Yoneda (SELETE), presented at Semicon Japan, December, 2004.





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<u>Relative Damage : (ndamaged – nref.) × Thicknessdamaged</u>



Ref: Katsumi Yoneda (SELETE), presented at Semicon Japan, December, 2004.



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 DiamondTM (7700A) / SiC (500A) / Cu (3000A) / Si Substrate



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

Figure 8B. Thickness uniformity map of the inhomogeneous Black DiamondTM 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-κ films:
 - Correlation of *n* to dielectric constant
 - Porous films
 - Inhomogeneous films
 - Films on metallic substrates
 - Characterization of plasma-induced damage

