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# Overview of Optical Metrology of Advanced Semiconductor Materials

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- Linear Optical Response of Materials
- Spectroscopic Ellipsometry
- Advanced Materials in the Semiconductor Industry
- Photoreflectance
- Photoluminescence
- Conclusions
- Acknowledgements



## Linear Optical Response of Materials





Spectroscopic Ellipsometry: Light polarized in plane of reflection reflects differently than light polarized perpendicular to plane of reflection.



P designation used for light polarized in plane of reflection (parallel)

S designation for light perpendicular to plane of reflection (senkrecht)





$$\widetilde{\varepsilon} = \varepsilon_1 + i\varepsilon_2 = (\widetilde{N})^2 = (n + ik)^2 = (n^2 - k^2) + 2ink$$

$$\alpha = \frac{4\pi k}{\lambda}$$



Ψ - amplitude ratio, Δ – phase difference, R– Fresnel reflection coefficient, n - real part of the refractive index, k – extinction coefficient,  $ε_1$  – real part of the dielectric function,  $ε_2$  – imaginary part of the dielectric function, α – absorption coefficient, λ – wavelength of light. **cnse.albany.edu**  Advanced Materials in the Semiconductor Industry



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## Linear Optical Response of Materials





## Optical Metrology of thin metal films (Ni, NiSi, TiN and W)



V.K. Kamineni, M. Raymond, E.J. Bersch, B.B. Doris, and A.C. Diebold, Journal of Applied Physics, 107 (2010) 093525.



### SE Methods & Limitations

VUV VASE 
$$\implies \frac{r_p}{r_s} = (\tan \psi)e^{i\Delta} \implies \psi \& \Delta \implies n, k\&$$

✓ Transparent

- Interference enhancements measurements (angle)
- Transmission intensity
- In situ measurements
- Optical constant parameterization

 $\tilde{n} = n - ik$   $\longrightarrow$  Kramers-Kronig Consistency

$$\varepsilon_1 + i\varepsilon_2 = (n - ik)^2$$









## Thickness Dependent Optical Properties

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### Optical model for metals

$$\varepsilon_{1} = \varepsilon_{\infty} + \omega_{pb}^{2} \frac{\tau_{b}^{2}(\omega_{o}^{2} - \omega^{2})}{\tau_{b}^{2}(\omega_{o}^{2} - \omega^{2})^{2} + \omega^{2}} - \omega_{pf}^{2} \frac{\tau_{f}^{2}}{\tau_{f}^{2}\omega^{2} + 1}$$

$$\varepsilon_{2} = \varepsilon_{\infty} + \omega_{pb}^{2} \frac{\tau_{b}\omega}{\tau_{b}^{2}(\omega_{o}^{2} - \omega^{2})^{2} + \omega^{2}} + \omega_{pf}^{2} \frac{\tau_{f}}{\omega(\tau_{f}^{2}\omega^{2} + 1)}$$

From Ellipsometry you can extract  $au_f$ 

Mayadas and Shatzkes -

S. Marsillac, N. Barreau, H. Khatri, J. Li, D. Sainju, A. Parikh, N.J. Podraza, and R.W. Collins, physica status solidi (c) 5, 1244-1248 (2008).



#### Mayadas and Shatzkes

$$\frac{1}{\tau_f} = \frac{1}{\tau_{bulk}} + \frac{\nu_F}{\lambda} \qquad \lambda = \left[\frac{2(1-\Re)}{3\Re}\right] R_g$$





### Conclusions

- 1. An Optical model was developed to determine the thickness of thin metal films.
- 2. Model was built to show that the thickness-dependent optical properties of thin metal films are correlated to the change in Drude free electron relaxation time.
- 3. The change in free electron relaxation time was traced to the change in grain boundary reflection coefficient and grain size.



## Linear Optical Response of Materials





## Investigation of Optical Properties of BCB Wafer Bonding Layer used for 3D Interconnects via Infrared Spectroscopic Ellipsometry



V.K. Kamineni, P. Singh, L.-W. Kong, J. Hudnall, J. Qureshi, C. Taylor, A. Rudack, S. Arkalgud, and A.C. Diebold, Thin Solid Films, 519 (2011) 2924.

P. Singh, J. Hudnall, J. Qureshi, V.K. Kamineni, C. Taylor, A. Rudack, A. Diebold and S. Arkalgud, Mater. Res. Soc. Symp. Proc., 1249 (2010) 309.



Replacing long horizontal lines with short vertical interconnects

•Addresses RC delay, crosstalk and power consumption

- Enables integration of heterogeneous devices & technologies (memory, logic, RF, analog, sensors)
  - Enables new functionalities
  - Cost reduction compared to SoC





#### Top wafer peeling due to air pockets in the BCB film



#### SEM cross-section image

#### 37% cross-linked BCB bond





SAM image



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### **IRSE** Absorption Spectra of BCB



The thermal curing of BCB polymers is a two step process:

- 1. Thermally activated opening of the BCB ring to form an o-quinodimethane intermediate.
- 2. The o-quinodimethane reacts with residual alkene groups in the via a Diels-Alder reaction, forming a tri-substituted tetrahydronaphthalene (THN).



### **IRSE** Absorption Spectra of BCB



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## Ideal Bonded Wafer: 60% Cross-linked BCB Bond

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Hard	bake	- Croce Linking Lovel	Experiment Regulte
Temp ℃	Time (min)	- Cross Linking Level	Experiment Results
190	10	37%	Dendritic structure and voids were present
190	25	42%	Dendritic structure and voids were present
190	40	45%	Dendritic structure and voids were present
200	15	45%	Dendritic structure and voids were present
200	25	50%	Dendritic structure and voids were present
250	36 (sec)	50%	Dendritic structure and voids were present
200	40	60%	Good no dendrites or voids; passed razor blade test
250	64 (sec)	60%	Good no dendrites or voids; passed razor blade test
250	2	70%	voids were present
250	3.5	80%	No voids were present but failed razor blade test

SAM image



**Future Directions:** 

- Bonding using adhesive materials is relatively new in the semiconductor industry.
  - IRSE can aid in screening newly investigated materials.
  - IRSE will aid in process tuning and development.



## Spectroscopic Ellipsometry of Porous Low-к Dielectric Thin Films



V.K. Kamineni, C.M. Settens, A. Grill, G.A. Antonelli, R.J. Matyi, and A.C. Diebold, Frontiers of Characterization and Metrology for Nanoelectronics, AIP Conference Proceedings, 1173 (2009) 168.

C.M. Settens, V.K. Kamineni, G.A. Antonelli, A. Grill, A.C. Diebold, and R.J. Matyi, Frontiers of Characterization and Metrology for Nanoelectronics, AIP Conference Proceedings, 1173 (2009) 163.



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## Introduction to Low-κ dielectrics

- Pores reduce the effective dielectric constant of the material
- Decrease signal propagation delays (faster switching speed)
- Reduction in crosstalk and power consumption
- Lower heat dissipation
- Pore volume fraction and pore size will effect the thermo-mechanical properties





Bruggeman expression for effective medium approximation on porous SiCOH samples

$$f_{a} \frac{\varepsilon_{a} - \varepsilon}{\varepsilon_{a} + 2\varepsilon} + f_{b} \frac{\varepsilon_{b} - \varepsilon}{\varepsilon_{b} + 2\varepsilon} = 0$$
  
Silicon

	Pore Volume Fraction	Thickness (nm)		
Control	0%	1055.0 0.2		
333 nm	24.8 0.3 %	352.6 1.0		
363 nm	23.8 0.1 %	379.5 0.4		





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## Molecular Bond Vibrations (Novellus)

Refractive Index (n) Tefractive Index (n) 1.5 1.48 1.46 1.44 1.42 к = 2.75 700 800 900 100 0 κ = 2.65 0 κ = 2.55 v√avelength (nm) 500 600 700 800 900 1000 Wavelength (nm)





Relative Carbon Concentration (%) =  $\frac{A_C}{A_C}$ 

Area = Peak ×  $\frac{F.W.H.M}{\sqrt{2ln(2)}}$  ×  $\sqrt{2}$ 

к	n	Carbon (%)
2.55	1.48	4
2.65	1.461	2.4
2.75	1.453	1.5

 $A_{c}$  - area under the Si-CH<sub>3</sub> peak  $A_{o}$  - area under the Si-O peak



## **Ellipsometry Porosimetry (ULK)**







### SE can measure critical mechanical parameters (Young's modulus, CTE)

- Young's modulus is a measure of the stiffness of the porous low-κ dielectric material
- A low CTE will prevent a connection failure between via/M1 and/or via/M2



#### **SEMATECH (Alain Diebold)**





Sample	Porosity (%)	Pore radius (nm)	EP Young's Modulus (GPa)
к = 2.55	13.19	0.82	2.74
к = 2.65	11.76	0.79	3.96
к = 2.75	7.92	0.84	7.08

 $V_L$  – molecular volume

E – Young's modulus

## High Temperature Thickness Measurements (ULK)



Sample	Refractive Index (n) 632.8 nm	TER (10 <sup>-6</sup> /°C)	CTE (10 <sup>-6/o</sup> C)
к = 2.55	1.48	70.2	44.72
к = 2.65	1.461	54.9	35.54
к = 2.75	1.453	29.5	20.30

Note: Fitting for the substrate expansion using the silicon temperature library (J.A.Woollam)

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- The following parameters were determined using SE.
  - SICOH (IBM)
    - Porosity (BEMA)
    - o Grading in porosity
  - ULK (Novellus)
    - o Carbon content
    - Porosity (EP)
    - $\circ$  Pore size
    - Young's modulus
    - Coefficient of thermal expansion
- Complementary techniques such as specular XRR, off-specular XRR and SAXS were also performed on these films. Both, the optical and x-ray metrology agreed reasonably well.

Sample	Thickness (nm)	<i>n</i> (632.8 nm)	Carbon %	Porosity (%)	Pore radius (nm)	EP Young's Modulus (GPa)	TER (10 <sup>-6/0</sup> C)	CTE (10 <sup>-6</sup> /ºC)
к = 2.55	155.6	1.48	4	13.19	0.82	2.74	70.2	44.72
к = 2.65	154.7	1.461	2.4	11.76	0.79	3.96	54.9	35.54
к = 2.75	147.1	1.453	1.5	7.92	0.84	7.08	29.5	20.30



## Linear Optical Response of Materials







## Extension of Far UV spectroscopic ellipsometry studies of High-к dielectric films to 130 nm



V. K. Kamineni, J. Hilfiker, J. Freeouf, S. Consiglio, R. Clark, G.J. Leusink, and A. C. Diebold, Thin Solid Films, 519 (2011) 2894.

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## Absorption Cross-section





## Thickness metrology

SE





## Linear Optical Response of Materials



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## Metrology of Strain Engineered GaN/AIN/Si(111) Thin Films Grown by MOCVD



M. Tungare, V. K. Kamineni, F. Shahedipour-Sandvik and A. C. Diebold, Thin Solid Films, 519 (2011) 2929.

http://images.iop.org/objects/compsemi/features/thumb/7/11/5/csnit1\_11-01.jpg



## Substrate Engineering Technique



Substrate engineering consists of 4 major steps:

- 1) Growth of an epitaxial AIN buffer layer on Si
- 2) Implantation of AIN/Si with N<sup>+</sup> ions to create a defective layer in Si below AIN buffer
- 3) Annealing of AlN/Si to recover any loss of crystallinity due to implantation
- 4) Growth of thick (2  $\mu$ m) GaN on AlN/Si





- Resolution = 2 cm<sup>-1</sup>
- E<sub>1</sub> (TO) Bulk AIN = 666 cm<sup>-1</sup>
- Isotropic model due to very thin AIN films (IR Probe)
- Tensile Biaxial Strain
- Broadening of the E<sub>1</sub> (TO) phonon mode in implanted AIN is clear sign of presence of defects
- Not sensitive to the A<sub>1</sub> (TO) due to c-plane orientation

Sample List	E1(TO) cm <sup>-1</sup>	γ (cm <sup>-1</sup> )	∆ω (cm⁻¹)	dw <sub>o</sub> /dP	Stress (GPa)
Si_AIN	662.2 0.08	19.5 0.2	3.8	4.5	0.84
Si_AIN_Implanted	664.5 0.24	56.0 0.4	1.5	4.5	0.33
Si_AIN_Annealed	663.8 0.16	21.2 0.2	2.1	4.5	0.48



## Linear Optical Response of Materials



## Spectroscopic Ellipsometry of CVD Graphene

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F. J. Nelson, V. K. Kamineni, T. Zhang, E. S. Comfort, J. U. Lee, and A. C. Diebold, Appl. Phys. Lett., 97 (2010) 253110.

## Wafer Mapping- CVD Graphene on 300nm SiO<sub>2</sub>/Si

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## Linear Optical Response of Materials





## Temperature and thickness dependence of the dielectric function and interband critical points in ETSOI



Dielectric response of bulk Si and nanoscale silicon films in different

V. K. Kamineni and A. C. Diebold, "Evidence of the phonon confinement on the linear optical response of nanoscale silicon films", <u>arXiv:1103.4102v2</u>.

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## Introduction to Silicon-on-insulator (SOI)

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Advantages of SOI technology:

- Excellent isolation of active devices
- Lowers the parasitic capacitance
- Lower leakage current
- Faster device operation
- Lower power consumption



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## Electronic transitions



<sup>1</sup>C. Kittel, Introduction to Solid State Physics (Wiley, New York, 1996)

<sup>2</sup>M. Cardona and F. H. Pollak, Phys. Rev. B 142, 530 (1966).



## **Direct space analysis**

### Direct space analysis:

Lorentzian line shape

$$\varepsilon = C e^{i\beta} \left( E - E_g + i\Gamma \right)^{-\mu}$$

Double derivative of the Lorentzian line shape

$$\frac{d^{2}\varepsilon}{dE^{2}} = \begin{cases} \mu(\mu+1)Ce^{i\beta}\left(E-E_{g}+i\Gamma\right)^{-\mu-2}, \mu \neq 0\\ Ce^{i\beta}\left(E-E_{g}+i\Gamma\right)^{-2}, \mu = 0 \quad \mathbf{k} \end{cases}$$

C – amplitude

$$\beta$$
 – phase factor

$$E_g$$
 – threshold energy ( $E_{CP}$ )

 $\mu$  – order of singularity

 $\mu = \frac{1}{2}$  (3D one-electron approximation)  $\mu = 0$  (2D one-electron approximation)  $\mu = -\frac{1}{2}$  (1D one-electron approximation)  $\mu = 1$  (excitonic critical points)

### Dielectric function of Si (experimental):



<sup>1</sup>P. Y. Yu and M. Cardona, Fundamentals of Semiconductors (Springer, Berlin, 2001).



Imaginary part of the dielectric function and its second derivative



- Change in the dielectric function of c-Si nanoscale films at different thickness
- Blue shift in the critical point energy is observed

Imaginary part of the dielectric function of *c*-Si QWs (~ 5 nm)

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Altering the optical response by changing the top dielectric layer: changes in quantum confinement and electron-phonon interaction



## In-line optical metrology

#### Thickness dependent dielectric function



What does it mean to in-line metrology?

- A recipe which has thickness dependent optical properties
- A recipe which has top dielectric layer dependent optical properties





- Linear Optical Response of Materials
- Spectroscopic Ellipsometry
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### Modulation spectroscopy (non-linear optical spectroscopy)



Complementary technique to measure the shift in the energy and lifetime broadening of the  $E_1$  critical point



Xenon source (probe)





### Photoreflectance



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## Photoreflectance of Strained SOI

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Fig. 1. Raman spectra corresponding to the sSOI sample using visible (514 nm) (a) and UV (325 nm) excitation (b).

Fig. 4. PR spectrum of strained silicon layer at room temperature (dotted curve). The solid line is corresponds to the theoretical fit.

- Three transitions energies are (i) 3.19 eV, (ii) 3.30 eV and (iii) 3.45 eV.
- Calculations show tensile strain of 1% in the strained Si overlayer
- This value is again in good agreement with the nominal tensile strain and with the values obtained from RS and Low Temp PhotoLuninescence.



# Franz Keldysh Oscillations



g. 1. Photoluminescence and photoreflectance spectra of Ga-and N-polar thick GaN layers grown by HVPE.

Franz Keldysh Oscillations
Toue to internal electric field in polar GaN



where  $\hbar\theta$  is the electro-optic energy,  $\Gamma$  is the linewidth,  $\phi$  is the phase factor, F is the electric field, and  $\mu$  is the electron hole reduced mass ( $\mu = 0.2 m_e$ ). The field estimated from the period of FKOs is 215 kV/cm (see Fig. 3). Such a huge

#### OPTO-ELECTRONICS REVIEW 12(4), 435.439

Internal

**E Field** 

# GaAsSb

Sample	А	В	С	D	Е
%Sb	38.3	38	45.7	51	52.5
Thickness d (nm)	41	72	50	72	41
$E_{\rm GHH}~({\rm eV})$	0.730	0.732	0.705	0.694	0.692
$E_{\rm GLH}~({\rm eV})$	0.681	0.681	0.689	0.695	0.696
$\Delta E_{\text{GHH-GLH}}$ (meV)	49	51	16	1	4
F (kV/cm)	92	52	80	54	96





FIG. 1. Room temperature PR spectra for all samples.

### Chouaib, APL 93, (2008) 041913



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## Photoluminescence

Photoluminescence is used to study the changes in the indirect transitions due to quantum confinement and change in dielectric medium

Photoluminescence peak energy (2D)

 $E = E_{gap} + E_{QC} - \frac{R_y}{\left(n - \frac{1}{2}\right)^2} + \frac{\hbar^2 \left(k_x^2 + k_y^2\right)}{2M}$   $E_{QC} = \frac{\hbar^2 \pi^2}{2ML^2}$   $R_y = \frac{m^*}{2} \left(\frac{e^2}{4\pi\epsilon\hbar}\right)^2 \rightarrow \text{Rydberg} \quad \mathbf{k} = \frac{\pi}{\sigma} (1,1,1)$ 



Band structure of Si calculated from the  $\mathbf{k} \cdot \mathbf{p}$  method<sup>1</sup>

<sup>1</sup>M. Cardona and F. H. Pollak, Phys. Rev. B 142, 530 (1966).



## Photoluminescence



As the power is increased, at high densities, and at low temperatures the free excitons condense to form a liquid phase.

This liquid phase manifests itself in the formation of EHD (Electron-hole droplets) – a broad feature in luminescence.

BE – Binding Energy



## Photoluminescence of SOI Quality



Fig. 2. PL mapping on (a) SIMOX, (b) Unibond<sup>®</sup>, and (c) ELTRAN<sup>®</sup> wafers with t<sub>SOI</sub> = 170 - 200 nm at room temperature: Upper and lower figures are on top Si layers and substrates, respectively.



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- A wide range of wavelengths makes Spectroscopic Ellipsometry a very powerful method capable of measuring more than just thickness and refractive index
- Non Linear Optical methods such as Photoreflectance and Photoluminescence provide information not available from spectroscopic ellipsometry



