

### Advanced EELS Applications In Process Development

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### Microprocessor (FIB cut)



- •Introduction to EELS in the transmission electron microscope
- •Element mapping using electron spectroscopic imaging
- •Quantitative EELS of advanced gate dielectrics
- •Quantitative EELS of low-к intermetal dielectrics
- •ELNES analysis of low-κ intermetal dielectrics and nickel silicides



### EELS in the transmission electron microscope

# Electron beam – specimen interaction





Electron energy-loss spectroscopy (EELS) detects inelastic interactions of beam electrons with the atomic electrons of the probed sample volume

### Electron energy-loss spectrum





- Core ionization edges  $\rightarrow$  **Compositional analysis**
- Core ionization near-edge structure (ELNES) → local atomic environment, chemical bonding

### Electron energy-loss spectroscopy in the TEM



#### Imaging energy filters allow to record spectra and energy selective images



•spatial resolution limited by the size of the focused electron probe

•energy resolution limited by the energy width of the electron source

spatial resolution limited by filter optics
energy resolution limited by the width of the energy selecting slit

Field emission gun (FEG), highly stable microscope electronics

#### $\rightarrow$ sub-nanometer electron probes

Aberration correction of the probe forming electron optics

#### $\rightarrow$ high SNR or sub-Angstrom electron probes

Corrected spectrometers

### $\rightarrow$ energy resolution limited by the energy width of the electron source

(Standard Schottky FEG: 0.5–1 eV,

Monochromated FEG: 0.1-0.3 eV)



### Element mapping using Electron Spectroscopic Imaging (ESI)











•Three-window method is routinely used for physical failure analysis at specific sites (e.g., identifying etch residuals or contaminating particles)

- Results depend on the quality of the edge background extrapolation - user has little control over this process
- Detection of low concentrations unreliable
- → It is often preferable to examine an actual spectrum from a region of interest
- $\rightarrow$  use Image-EELS

### Principle of Image-EELS

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Record a series of energy-filtered TEM images and extract spectra from any desired region of interest

#### Image-EELS of an SOI contact after TiN barrier deposition



•Cross-section prepared by FIB cutting

•100 images in 5 eV-steps (80-575 eV), energy slit width 5 eV, 4 s/image.

•Specimen drift during acquisition corrected off-line by cross-correlation image alignment





After alignment

## Image-EELS of an SOI contact after TiN barrier deposition





•Abnormal features (e.g., residual layers) can be investigated in detail

•Characteristic near-edge structures of the Si- $L_{2,3}$  edge can be distinguished



### Quantitative EELS of advanced gate dielectrics

### MOSFET with nitrided gate oxide



#### TEM of a MOSFET



•Si-O-N gate dielectric - less than 10 atomic layers!

•The N distribution affects the properties of the Si-O-N layer

 $\rightarrow$  N distribution in the 5-15 at% range can be measured by EELS at subnanometer resolution

- Si-O-N deposited by plasma-enhanced CVD
- Specimen thickness 20-80 nm
- Electron probe size  $\approx$  0.35 nm
- •Line scans: 40 points in 0.15 nm steps across the gate dielectric
- Max. 1-2 s per point due to specimen drift



## Conventional quantitative spectrum processing





- 1. Model the edge background ( $\propto E^{-r}$ ). NOT GOOD FOR OVERLAPPING EDGES!
- 2. Area under the edges is proportional to the concentrations per area, BUT ONLY FOR SINGLE SCATTERING!
  - Differential scattering cross-sections needed for quantification. PROBLEM: THEORETICAL CROSS-SECTIONS INACCURATE!

## Improved spectrum processing by reference spectra fitting



Decomposition of the measured spectrum into its single, double,... scattering components:

 $\begin{aligned} \text{Fit} &= \mathsf{P}_1\mathsf{S}_\mathsf{P} + \mathsf{P}_2\mathsf{S}_\mathsf{P}\otimes\mathsf{S}_\mathsf{P} + \mathsf{P}_3\mathsf{S}_\mathsf{P}\otimes\mathsf{S}_\mathsf{P}\otimes\mathsf{S}_\mathsf{P} + \cdots \\ &+ \mathsf{Si}_1\mathsf{S}_{\mathsf{Si}} + \mathsf{Si}_2\mathsf{S}_{\mathsf{Si}}\otimes\mathsf{S}_\mathsf{P} + \mathsf{Si}_3\mathsf{S}_{\mathsf{Si}}\otimes\mathsf{S}_\mathsf{P}\otimes\mathsf{S}_\mathsf{P} + \cdots \\ &+ \mathsf{N}_1\mathsf{S}_\mathsf{N} + \mathsf{N}_2\mathsf{S}_\mathsf{N}\otimes\mathsf{S}_\mathsf{P} + \mathsf{N}_3\mathsf{S}_\mathsf{N}\otimes\mathsf{S}_\mathsf{P}\otimes\mathsf{S}_\mathsf{P} + \cdots \\ &+ \mathsf{O}_1\mathsf{S}_\mathsf{O} + \mathsf{O}_2\mathsf{S}_\mathsf{O}\otimes\mathsf{S}_\mathsf{P} + \mathsf{O}_3\mathsf{S}_\mathsf{O}\otimes\mathsf{S}_\mathsf{P}\otimes\mathsf{S}_\mathsf{P} + \cdots \end{aligned}$ 

 $\rightarrow$  Atomic ratios:

$$N_N/N_{Si} \propto N_1/Si_1$$
 ;  $N_O/N_{Si} \propto O_1/Si_1$ 

Determine the proportionality factors from calibration measurements

→ Edge background modelling, removal of multiple scattering effects, separation of overlapping edges, and quantification in a single workstep!

### Set of reference spectra



### Example fits of two spectra



### Example fits of two spectra



## Result: spatially resolved atomic ratios of N, O and Si





## Atomic percentages calculated from the atomic ratios





#### Comparison to AES depth profiling



Auger Electron Spectroscopic (AES) depth profiles

EELS linescans of the same layer stack

 $\rightarrow$  slightly better depth resolution (about 0.5 nm)

## Quantitative EELS of high-k metal oxide dielectrics



TiN/poly Si-capped Hf-O-Si gate electrode stack



• O concentration dip in the high-k oxide

- $\rightarrow$  O depletion or artifact due to strong elastic scattering in the Hfrich layer ?
- EELS quantification is problematic in the presence of strongly scattering components
- $\rightarrow$  Correction factors may have to be applied!



### Quantitative EELS of low-к intermetal dielectrics

- Substitution of oxygen in SiO<sub>2</sub> by methyl groups (-CH3) reduces the permittivity significantly ( $\kappa = 4.0 \rightarrow 2.6-3.3$ )
- $\rightarrow$  Carbon doped intermetal dielectric materials (IMD) reduce interconnect delay, power dissipation, and crosstalk noise
- Plasma processing for resist stripping, trench etching and post-etch cleaning removes molecular groups that contain C and H from the near-surface layer (10-20 nm)
- $\rightarrow$  Increased water absorption and dimensional changes
- → Quantitative EELS analysis of structured IMD films with nanometer resolution for process optimization

## EELS line scans across carbon depletion zones







Cu interconnect lines embedded in SiCOH (HAADF-STEM image)

Atomic ratios calculated from EELS line scans



# ELNES analysis of low-k intermetal dielectrics and nickel silicides

## Energy loss near-edge structure (ELNES) analysis of low-к IMD

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ELNES of the C-K edge at three different FEG monochromator settings

 $\rightarrow$  three different energy resolutions





**Carbon depletion zone shows modified bonding**   $\rightarrow$  Investigate process induced low- $\kappa$  dielectric modification and damage mechanisms

- The formation properties of self-aligning metal silicides on narrow lines depend on process temperatures, dopant concentrations, and line width
- The introduction of nickel mono-silicide (NiSi) requires a thorough investigation of these effects and their relation to process parameters

 $\rightarrow$  Identify silicide phases with nanometer resolution for process optimization

## Metal silicide phase identification by electron microdiffraction





Results often ambiguous due to strong crystal orientation dependence of the diffraction patterns NiSi







NiSi<sub>2</sub>



?

?

?





## Metal silicide phase identification by ELNES of the Si- $L_{2,3}$ edge





Energy loss [eV] Si-L<sub>2,3</sub> ELNES of NiSi, NiSi<sub>2</sub>, and Ni<sub>2</sub>Si (energy resolution 1 eV)

 $\rightarrow$  Each phase shows a distinct fine structure that can be used for phase identification (`ELNES fingerprinting')

- Advanced TEM-EELS techniques provide valuable high spatial resolution information for process development:
- Accurate compositional analysis using Image-EELS
- Quantitative N, O, Si, C, ... concentration profiling by means of reference spectra fitting of EELS linescans
- •Chemical bonding analysis of low-κ dielectric materials using ELNES analysis
- Phase identification of metal silicides by ELNES fingerprinting