

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

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 SiO₂ 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

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- κ 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

NiSi₂

?

?

?

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

Energy loss [eV] Si-L_{2,3} ELNES of NiSi, NiSi₂, and Ni₂Si (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