Advanced EELS Applications In Process Development

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Microprocessor (FIB cut)

Transistors

Cu interconnects

AMD Opteron®
Outline

• Introduction to EELS in the transmission electron microscope

• Element mapping using electron spectroscopic imaging

• Quantitative EELS of advanced gate dielectrics

• Quantitative EELS of low-κ intermetal dielectrics

• ELNES analysis of low-κ intermetal dielectrics and nickel silicides
EELS in the transmission electron microscope
Electron energy-loss spectroscopy (EELS) detects inelastic interactions of beam electrons with the atomic electrons of the probed sample volume.
Electron energy-loss spectrum

- Plasmon excitation peaks → **Dielectric material properties**
- Core ionization edges → **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
Current performance standards

Field emission gun (FEG), highly stable microscope electronics

→ sub-nanometer electron probes

Aberration correction of the probe forming electron optics

→ high SNR or sub-Angstrom electron probes

Corrected spectrometers

→ 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)
Element mapping using the three-window method
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![Graph showing element mapping using the three-window method with images of CoSi and CoSi$_2$.](image)
Element mapping using the three-window method

- 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

→ 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

Raw data

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

**Compositional analysis of arbitrarily shaped regions of interest**
Quantitative EELS of advanced gate dielectrics
MOSFET with nitrided gate oxide

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

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

→ N distribution in the 5-15 at% range can be measured by EELS at sub-nanometer resolution
EELS line scans across the gate dielectric

- 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!

3. 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:

\[
\text{Fit} = P_1 S_P + P_2 S_P \otimes S_P + P_3 S_P \otimes S_P \otimes S_P + \cdots \\
+ S_{11} S_{Si} + S_{12} S_{Si} \otimes S_P + S_{13} S_{Si} \otimes S_P \otimes S_P + \cdots \\
+ N_{11} S_N + N_{12} S_N \otimes S_P + N_{13} S_N \otimes S_P \otimes S_P + \cdots \\
+ O_{11} S_O + O_{12} S_O \otimes S_P + O_{13} S_O \otimes S_P \otimes S_P + \cdots
\]

→ Atomic ratios: \( \frac{N}{N_{Si}} \propto \frac{N_1}{Si_1} ; \frac{N_O}{N_{Si}} \propto \frac{O_1}{Si_1} \)

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

![Graph showing CCD counts and energy loss](image)
Example fits of two spectra
Example fits of two spectra
Result: spatially resolved atomic ratios of N, O and Si

Two different Si-O-N gate dielectrics

Max. 1-2 s acquisition time per point → low SNR → average N- and O- profiles from 10-20 linescans (aligning and averaging automated)

Two different Si-O-N gate dielectrics
Atomic percentages calculated from the atomic ratios

N peak shifted towards poly Si

Two different Si-O-N gate dielectrics
Comparison to AES depth profiling

Test layer stack: SiO$_2$/Si-O-N/Si

Auger Electron Spectroscopic (AES) depth profiles

EELS linescans of the same layer stack → slightly better depth resolution (about 0.5 nm)
Quantitative EELS of high-κ metal oxide dielectrics

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

- O concentration dip in the high-k oxide
  → O depletion or artifact due to strong elastic scattering in the Hf-rich layer?
- EELS quantification is problematic in the presence of strongly scattering components
  → Correction factors may have to be applied!
Quantitative EELS of low-κ intermetal dielectrics
Carbon depletion in low-κ IMDs

- Substitution of oxygen in SiO₂ by methyl groups (-CH₃) reduces the permittivity significantly (κ = 4.0 → 2.6-3.3)
  - 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)
  - 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-κ intermetal dielectrics and nickel silicides
Energy loss near-edge structure (ELNES) analysis of low-κ IMD

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

Carbon depletion zone shows modified bonding → Investigate process induced low-κ dielectric modification and damage mechanisms
Metal silicide phase identification

• 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

→ **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

Si-L$_{2,3}$ ELNES of NiSi, NiSi$_2$, and Ni$_2$Si (energy resolution 1 eV)

→ Each phase shows a distinct fine structure that can be used for phase identification (‘ELNES fingerprinting’).
Summary

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