





EDS with Microcalorimeters

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Usage of Microcalorimeters on Scanning Electron Microscopes (SEM) for determination of elemental constitution of samples



What's EDS ?

- Measurement of Energy of characteristic X-ray radiation excited by an electron beam in a Scanning Electron Microscope (SEM) gives information on elemental constitution of sample in SEM
- In the SEM an Image and an EDS spectrum can be measured at the same time
- \Rightarrow Elemental analysis of small structures

X-Ray Excitation Volumes with 2 keV and 5 keV Electron Energies





David Joy, Monte Carlo Modeling for Electron Microscopy and Microanalysis, Oxford University Press, 1995

Motivation







geometrical dimensions and X-ray emitting volume Si(Li)-detector: spectrum of TiN @ V_{acc} = 3keV \Rightarrow Overlap of X-ray lines at low energies

Separation of X-ray lines at low energies requires different analysis method or different detector technology ⇒Low temperature detectors





Vibration performance



Detector Setup











Detector response after absorption of X-Ray



Resolution

Parameter:energy resolution @ Al-Kα (1,5 keV)Target Specification:15 eVResult:typical: around 10eV, best result: 6.4 eV



Energy resolution @ Al-K α (1.5 keV)

Energy resolution vs Energy

Problem of Countrate



□ Installation of Polycapillary Lens

Increase of solid angle due to larger Area (A=12 mm²) and lower distance (d=9 mm)



A LO BOARD

Countrate



- small detector aperture size: 200 µm
- large detector

 (factor 3 @ 5kV)
 aperture size: 600 µm
- small detector with X-ray lens (factor 120 @ 5kV), aperture size: 20 µm, converted to 120 µm by probe current ratio

Count rate of detector vs. acceleration voltage for different concepts of count rate increase, sample: bulk silicon



analytical results



Spectrum of TiN:@ V_{acc} = 4 kV, aperture: 120 μm, time: 5 min, without X-ray lens

Spectrum of TaSi₂:@ V_{acc} = 5 kV, aperture: 60 μm, time: 15 min, without X-ray lens

analytical results

Analysis of light elements

Sample: BPSG oxide (boron phosphorous silicon glass); conc.(B) = 3%



Spectrum of BPSG: @ $V_{acc} = 5 \text{ kV}$, aperture: 120 μ m, time: 8 min, with X-ray lens



analytical results

□ example: Phosphor-doped Si, N_D=4 x 10¹⁹ cm⁻³ →<1‰ with N_{Si} = 5 x 10²² cm⁻³



Spectrum von n-Si @ $V_{acc} = 5 \text{ kV}$, aperture: 30 μ m, Measurement time: 6h, with X-ray Lens



2d Elemental analysis: linescan

□ Testing structures after W-CMP: W- SiO₂







Linescan V_{acc} = 4 kV, aperture:120µm, time: 7 min., with X- ray lens

Conclusions





- Microcalorimeter shows superior performance compared to Si(Li) detectors
- Microcalorimeter based spectrometer commercially available

•Related Posters:

- F06: Development of Ir/Au Transition Edge Sensors for high Resolution X-Ray Spectroscopy in Material Science by Doreen Wernicke
- F07: STJ Detector for Material Analysis: Implementation and First Results by Michael Huber



Thin layer systems $SrBi_2Ta_2O_9(SBT) / Pt / TiO_2 / SiO_2 / Si - Substrat$



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Final assessment / analytical results

3. Analysis of samples with small volume

Sample: TiN surface w/o oxygen film



AES depth profile through TiN (80 nm) / Ti (34 nm)



Problem: low countrate

□ Detector area ~ 200µm·200µm (Si(Li) ~ 3mm · 3mm)

 \Rightarrow low countrate: 0,2cps (V_{acc} = 5kV, Ipr = 100pA)

 \Rightarrow long measurement time

 \Rightarrow high electron beam induced damage

 \Rightarrow different Types of analysis impossible:

- Linescans
- Elemental Mappings
- Measurement of low concentrations

• Possible Solutions:

– Large area detectors

- \Rightarrow 600µm x 600µm Design tested
 - Transition wide
 - \Rightarrow Resolution quite bad

- Use of polycapillary X-ray optics





Clean room





Energy calibration / heat pulses





Energy calibration / heat pulses

Possible Solutions:

•Take calibration spectrum before measurements

•Use artificial pulses to determine detector response

→current pulses heat up the detector







Energy calibration / heat pulses

Calibration of whole energy area is possible with 5 different kinds of heat pulses simulating O-K α , Na-K α , Si-K α , Cl-K α , Ti-K α



Red points: heatpulses Blue points: real pulses



Heat pulses show:
 •same pulsheight as real pulses
 •same temperature dependence as real pulses

BUT: Reproducibility of calibration at only 10eV ⇒Heatpulses have to be faster and more intensive



Analysis: C-Contamination

□ EDS analysis





X-ray spectra of spot and reference point Vacc = 4 kV, aperture: 120 μ m, time: 5 min, with lens



Measurement on black dot

reference