Electron microscopy: interaction of 80-300 keV electrons with silicon detectors

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Thermo Fisher Scientific
FEI Company

- Revenue 956M$ (2014)
- Market cap. 3,1B$ (November 2015)
- About 10,000 systems installed; 3,500 customers
- Sales and service in 50 countries
- 2700 employees
- Main sites in Eindhoven (NL), Brno (Cz) and Hillsboro (USA)
- Electron microscopes; Dual-Beam systems; software
- Acquired by Thermo Fisher Scientific in September 2016
Microscopy

Source: http://www.bates.edu/gould-research-lab/research/
Genesis of microscope

• The word microscope is derived from the Greek *mikros* (small) and *skopeo* (look at).
• One of the earliest instruments for seeing very small objects made by the Dutchman *Antony van Leeuwenhoek* (1632-1723)
• Van Leeuwenhoek was able to magnify objects up to 400x; and with it he discovered protozoa, spermatozoa, and bacteria, and was able to classify red blood cells by shape.
There’s Plenty of Room at the Bottom

- On December 29th, 1959, the physicist Richard Feynman issued an invitation to scientists to enter a new field of discovery with his lecture entitled “There’s Plenty of Room at the Bottom,” delivered at the annual meeting of the American Physical Society at the California Institute of Technology (Caltech).
- This is credited as the genesis of the modern field of nanotechnology.
- The best resolution achieved to date is 0.05 nm: we have indeed met his challenge to create a microscope powerful enough to see individual atoms.
Electron microscopes

Transmission electron microscope (TEM) – energy range: few 60 keV to 300 keV

- It detects transmitted electrons
- Resolution down to 0.5 Å
- Imaging on thin samples (< 100 nm)
- Elemental mapping by EDX or EELS

Scanning electron microscope (SEM) – energy range: few eV to 30 keV

- It detects backscattered electrons
- Resolution below 1 nm
- Imaging on thick sample (info mainly on the surface)
- Elemental mapping by EDX
- Imaging, analysis, and TEM sample preparation in semiconductor pathfinding and process development laboratories
Evolution of TEM

• TEM has evolved considerably in the past years:
  • Automated data collection
  • Digital cameras/detectors replaced photographic films
  • Easy-to-use
  • Maximized throughput

• Nowadays, the operator does not need be a TEM specialist
  • More time dedicated to sample analysis rather than tool settings

20 years ago

today
Electron microscopy customers

**Industry**
- **Electronics**
  - New Process Development
  - Failure Analysis
- **Natural Resources**
  - Minerals Liberation
  - Yield Improvement

**Science**
- **Materials Science**
  - Stronger Materials
  - Advancing Science
- **Life Sciences**
  - Understanding Biological Structure and Function

**STREAM**
Transmission electron microscope

- Electron detection 80-300 keV (for SEM energy <30 keV)
  - Imaging (TEM, STEM)
    - Life science
    - Materials science
  - Diffraction
  - Electron energy loss spectroscopy (EELS)
- X-ray detection
  - Energy dispersive spectroscopy (EDS)

Each application requires a specific type of detector!!!
Interaction of electron beam with the specimen

In transmission electron microscopy, forward scattered electrons are detected.

Two main techniques are available:

- TEM: parallel beam, uniform illumination over the sample
- STEM: focused beam, probe scanned over the sample
Standard TEM

\[ \psi = \psi_{in} \]

\[ \psi = \psi_{in} \cdot e^{i\varphi} \]

\[ \psi = \mathcal{F}\{\psi_{in} \cdot e^{i\varphi}\} \cdot e^{i\chi} \]

\[ \psi_D = \mathcal{F}\{\mathcal{F}\{\psi_{in} \cdot e^{i\varphi}\} \cdot e^{i\chi}\} \]

\[ I_{TEM} = |\mathcal{F}\{\mathcal{F}\{\psi_{in} \cdot e^{i\varphi}\} \cdot e^{i\chi}\}|^2 \]
Standard STEM

\[ \psi = 1 \cdot A e^{-ix} \]

\[ \psi_{in} = \mathcal{F}\{\psi\} \]

\[ \psi_{out} = \psi_{in} \cdot e^{i\phi} \]

\[ \phi = \sigma V_z \]

\[ \psi_D = \mathcal{F}\{\psi_{out}\} \]

\[ I_{STEM} = \iint W \cdot |\psi_D|^2 d\vec{k} \quad W \in \{W_{BF}, W_{ABF}, W_{ADF}, W_{HAADF}\} \]

E.G.T Bosch, I. Lazić, Ultramicroscopy 156 (2015) 59–72
TEM imaging

• The entire specimen is illuminated with a uniform beam
  • The image is detected with an image sensor or a sensitive film in early microscopes

• Electrons undergo different scattering events through the specimen
  • The emerging beam contains several information (structural, chemical,...)

• Spatial distribution is detected as contrast variation in imaging mode
• Angular distribution is detected as a diffraction pattern
CMOS APS based detectors are currently used in transmission electron microscopy.
Detectors for high resolution electron microscopy

- High detective quantum efficiency (DQE) is key for high resolution reconstruction in low-dose applications

\[ DQE = \frac{(SNR_{out})^2}{(SNR_{in})^2} \]

- Key technology drivers to achieve high DQE:
  - Direct detection
  - Backthinned detector
  - Electron counting

- In 2011, FEI introduced Falcon camera for direct electron detection which has been a game changer
DQE

\[ NEQ = (SNR_{OUT})^2 = \frac{d^2MTF^2}{NPS} \]

\[ (SNR_{IN})^2 = N \]

\[ DQE = \frac{(SNR_{OUT})^2}{(SNR_{IN})^2} = \frac{NEQ}{N} = \frac{d^2MTF^2}{N\cdot NPS} = \frac{g^2\cdot N\cdot MTF^2}{NPS} = \frac{MTF^2}{NNPS} \]
Direct vs. indirect detection

**Indirect Detection**
- Electrons converted into photons
- Low sensitivity
- MTF-loss and additional noise due to scintillator
- Highly radiation-hard (depending on scintillator)

DQE: \(\sim 0.1\) at \(\frac{1}{2}\) Nq (it gets worse at low dose)

**Direct Detection**
- Direct detection of electrons
- Very high sensitivity
- MTF and noise determined by Si detector only
- Sensitive to radiation damage \(\rightarrow\) (limited lifetime)

DQE: \(\gg 0.1\) at \(\frac{1}{2}\) Nq (even at low dose)
Interaction of electrons with silicon

- An electron entering the Si sensor does not travel along a straight line
  - Lateral scattering
  - Backscattering
- The charge clouds can be few-hundreds microns wide
  - An electron can generate signal in pixels far from its impinging point
- This causes additional noise, cross-talk, MTF-loss and ultimately low DQE

*McMullan et al., Ultramicroscopy 109 (2009)


<table>
<thead>
<tr>
<th>Energy (keV)</th>
<th>Image</th>
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<td>80</td>
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</tr>
<tr>
<td>120</td>
<td><img src="image2" alt="Image" /></td>
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<td>160</td>
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</tr>
<tr>
<td>200</td>
<td><img src="image4" alt="Image" /></td>
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</tbody>
</table>
Charge sharing

If pixel size is too small, one single electrons will generate a large signal on several pixels!!

**How to deal with it?**

**Match pixel size with interaction volume**
- Small number of pixels
- Limited field of view
- All energy deposited

**Small pixel size, thin sensor**
- Large number of pixels
- Large field of view
- Only portion of energy deposited

- Sensors used for imaging require either a large field of view (single particle analysis in Life Science) or very high spatial resolution (Materials Science). Currently on the market for TEM imaging:
  - small pixels (5-15 µm)
  - large number of pixels (e.g. 4k x 4k)
  - Thin epilayer + substrate backthinning
Interaction of electrons with silicon
MTF

Back-scattering contribution

Lateral scattering and diffusion
Backthinned detectors

Falcon 1

Falcon 2

500 µm

50 µm

Passivation layer, interconnects
Active pixel
Active layer
Substrate

Thinned Substrate

Back scatter reduction plate


Electron counting can further increase DQE


1 e/px/s in single event counting mode

10 e/px/s in normal mode

Direct detection

Indirect detection

Backthinning

Falcon I
Falcon II

0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05

0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1

Spatial freq (Nq)

DQE (f)

0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
0.5

0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1

Spatial freq (Nq)

DQE (f)
Materials Science

*Courtesy of Emrah Yucelen (TMO), Au nanoparticles

- Sub-Angstrom resolution
- Typically high-contrast image
  - High dose allowed e.g. $>10^3 \text{ el/Å}^2/\text{sec}$
  - however, it depends on the case: sample may interact with beam at high current


Si [110], 0.07 nm (STEM)
Challenges for Life Science imaging (cryo-specimens)

1. **Low contrast:**
   - Protein density (C, N, O, H) comparable to vitrified water (O, H)
   - Need detectors with HIGH DQE!

2. **Radiation sensitivity**
   - Low electron dose (50 el/pix/s, 20 el/Å²/sec.
     Total: 20-100 el/Å²)
   - Trend to 1 el/pix/s electron counting

Need detectors with HIGH DQE!

Specimen in vitreous ice

Carbon foil ~20nm

3.2 Å of Thermoplasma acidophilum 20S proteasome

Falcon II camera for life science

- Electron energy: 80-300 keV
- 4k x 4k pixels
- Sensor size: ~60 mm x 60 mm
- Thickness: 50 um
- Dose rate: towards 0.025 el/pix/frame
- Radiation hardness: < 1 Gpe/pix (~35 Mrad)
A focused beam is scanned through the sample. Each scanning position corresponds to a pixel in the final image. Forward scattered electrons can be acquired at different angles:
- BF: < 10 mrad
- ADF: 10-50 mrad
- HAADF: >50 mrad off axis

Transmission Electron Microscopy, A Textbook for Materials Science
David B. Williams, C. Barry Carter
STEM detectors

Segmented detectors

Solid state detectors with one segment or multi-segments
- One intensity level per each scanning position

Pixelated detectors

Complete diffraction pattern recorded per each scanning position
- BF, ADF, HAADF can be emulated offline

**Diffraction patterns**

Typical diffraction pattern

Intensity variation from undiffracted beam and outmost diffraction spot can be 6 orders of magnitude

- Typical requirements to serve this application
  - High dynamic range ($10^6$) (→ EMPAD made at Cornell University)
  - Single electron sensitivity
  - High speed (> 1 kHz)
  - Extremely high radiation hardness (all electron current mainly in the undiffracted beam)
  - 128x128 to 1024x1024 pixels
  - Typically, large pixel size (> 100 µm) for 100-300 keV

• One diffraction pattern generated for each scanning position
• Depending on the element present at that position, electrons can undergo different scattering angle
• The diffraction pattern at a specific scanning position is used to generate a grey level corresponding to that position in the final image
Radiation damage

• Electrons energy range in TEM 80-300 keV (moving towards 60 keV)

• Radiation damage
  – Ionizing damage
  – Displacement damage

• The energy threshold for electrons to create displacement damage is 260 keV*

• Radiation damage in detectors for TEM is mainly ionizing damage
  – Silicon oxide and silicon nitride are the areas where ionizing damage is localized
    • Minimize oxide and nitride
    • Use radiation tolerant design (surface passivation, enclosed layout for transistors)

CMOS APS vs. Hybrid detectors

Epilayer (e.g. 5-25 μm)

Substrate (e.g. 10-500 μm)

Oxide (3-5 μm)

Depletion region

80-300 keV electron

transistor

Readout

(not directly exposed to radiation)

Sensor layer (e.g. 500-700 μm)

Pixelated ASIC (e.g. 500 μm)

Thin Oxide

Fully depleted

80-300 keV electron
CMOS APS vs. Hybrid detectors

**CMOS APS**

**Ionizing damage**
- Thick oxide (pre-metal and inter-metal dielectric)
- Possible formation of fixed positive charges and interface traps
  - Dark current increase
  - Threshold voltages shift
- Limited radiation hardness
- The thick oxide creates a large dead layer, limiting max electron energy in front illumination

**Displacement damage**
- Partially depleted epilayer
- Displacement damage occurs in the diffusion region: traps can be active (effect on dark current, gain, MTF, ...)
- Limited radiation hardness

**Hybrid**

**Ionizing damage**
- Thin oxide and/or thin metallization
  - Suited for low energy too
  - Very limited effect of interface traps
- Passivation by highly-doped P+ or N+ layer along the entire surface (very efficient)
- Sensor layer thicker than electron penetration depth in Si
  - It acts as “shield” for the ASIC

**Displacement damage**
- Fully depleted sensor layer
  - Displacement damage occurs in the high-field region of the sensor layer: traps not active
  - No displacement damage in the ASIC
- Very-thin dead layer
- Very high radiation hardness of the sensor layer
- ASIC can degrade over time due to X-rays
Single electrons interaction in CMOS APS

Non backthinned  backthinned
Single electrons interaction in CMOS APS

Non backthinned

backthinned
Single electrons interaction in hybrid detectors (Pixel size 150 μm)

80 keV, non fully depleted

80 keV, fully depleted
Single electrons interaction in hybrid detectors (Pixel size 150 μm)

300 keV, non fully depleted

300 keV, fully depleted
Conclusions

• TEM is a growing field and detectors are one of the key elements

• Different markets are served with different applications
  – Specific detectors are developed for each market/application

• Electrons in the energy range 80-300 keV have a different interaction with the matter than high energy particles
  – This has an influence on the detection and radiation damage mechanisms
Thanks
Q&A