## **Basics of Electron Microscopy**





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#### Agenda



1	Why Electrons?
2	Interaction with Matter
3	Electron Emitter
4	Electron Column Design
5	Operating Parameters
6	Insulating Samples

#### **Application Fields**





#### **Materials Research**

- **Task:** Understand and tailor physical properties of materials. Develop new materials.
- **Examples:** Steels, Alloys, Polymers, Ceramics, Composites
- Image: Manganese sulfide inclusions in steel. Courtesy of Georgsmarienhütte GmbH, Germany



#### Life Sciences

- **Task:** understand structure and function of biological material
- **Examples:** Cell biology, Neurobiology, Histology, Zoology, Botany
- Image: drosophila larval brain. Courtesy of C. Shan Xu, HHMI, USA



#### **Advanced Materials**

- Task: Design materials and functional nanostructures with improved or new physical properties
- Examples: Metamaterials, MEMS, Biomaterials
- **Image:** Focused-Ion-Beam Nanofabrication of Near-Infrared Magnetic Metamaterials, Enkrich et al. Adv. Mater. 17 (2005)



#### **Raw Materials**

- **Task:** find new profitable deposits of natural resources
- **Examples:** Oil&Gas (sedimentary rocks, shale), Mining
- **Image:** Pyrites and voids in shale rock. Courtesy of NanoFUN, Poland



#### **Electronics / Semiconductors**

- **Task:** Design better and more efficient electronic devices. Failure Analysis.
- **Examples:** Semiconductors, Polymer Electronics, Photonics
- Image: Cross-Section of IC with Intel 22nm-Tri-Gatetechnology. Courtesy of UBM TechInsights, Canada



#### Manufacturing

- **Task:** quality control of material systems used in different industries (e.g. automotive)
- Examples: Coatings, Oxidation, Corrosion, Irradiation
- Image: Crack in steel sheet. Courtesy of AUDI AG, Germany

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### **Carl Zeiss Microscopy** Why electrons?

- similar optical properties:
  diffraction, abberation, astigmatism etc.
- spatial resolution depends on wavelength:
  - the higher the energy (of the electrons)
    - the lower the wavelength,
      - the higher the resolution
- typical acceleration voltages of electrons:
  - Scanning electron microscopes (SEM): 0.2 30 kV
  - Transmission electron microscopes (TEM): 40 1200 kV

	Light Microscopy	Electron Microscopy
Wavelength	400 – 700 nm	0.1 – 0.002 nm (1 – 100 kV)
Spatial Resolution	~ 1 µm	~ 1 nm
Max. Magnification	1,000 x	1,000,000 x
Modes of Operation	reflected light, transmitted- light	SEM, TEM
Samples	Unmodified, hydrated	vacuum-compatible















- Bulk sample
- Sub-surface (few nm or more below)
- Rastered beam
- Detect secondary and backscattered electrons





### **Electron Microscopes** Electromagnetic Lenses







We can change the strength of the lens (i.e. the focal plane) by changing the current, but not the shape of the fields.

### **Carl Zeiss Microscopy** Components of a SEM







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### **Electron-Induced Signals** Plenty of Information Induced by Primary Beam





## Surface and volume information associated with the signals

Signals and interaction volume induced by the primary beam

### **Beam-Sample Interaction** Influence of Beam Energy – Interaction Volume



#### **Monte Carlo Simulations**



Monte Carlo simulation of the beam – sample interaction for a silicon sample at 1kV and 10kV.

### **Beam-Sample Interaction** Influence of Beam Energy – Image Quality





200 V 500 V 1 kV 2 kV



5 kV



Iron oxide/ Zirconium dioxide composite

### **Beam-Sample Interaction** Influence of Beam Energy – Image Quality



Iron oxide/ Zirconium dioxide composite



200 V

1 kV

Lower voltage provides <u>more surface detail</u>...BUT...with <u>loss of S/N  $\rightarrow$  loss of clarity!</u>

### How a SEM Works How a SEM works





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#### X-ray Detection (EDS/WDS)



The SEM beam can excite emission of **characteristic x-rays** from the sample. Detection of these x-rays by *energy-dispersive* or *wavelength-dispersive* x-ray spectroscopy (EDS or WDS) reveals a sample's **elemental composition**.



EDS map image of a paint sample from original artwork.

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#### Analysis Beyond EDS SEM + Raman spectroscopic imaging









# Nonmetallic compositions and phases:

- Polymorphs
- Molecule phases
- Polymer blends
- Crystal orientations
- Fibrillary polymer
- Degree of Crystallinity
- Kinds of Defect
- Stress and Strain
- Surface coating
- Contamination



- Nanoparticles & 2D Mat.
- Polymer
- Carbon materials
- Semiconductor
- Geology/ Mineralogy
- Forensic
- Pharma
- Biomaterials
- Coatings
- Corrosion





#### **Electron Backscatter Diffraction (EBSD)**



Bragg **diffraction** occurs when the SEM beam strikes **crystalline matter**. The resulting electron backscatter diffraction (EBSD) patterns (see image) reveal the underlying **crystal structure**.

EBSD is useful for applications with metals, inorganics and ceramics.



EBSD pattern of silicon.

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#### **Transmitted Electrons (STEM)**



When imaging very **thin samples** (<100 nm thick), electrons can pass through the sample. Detection of **transmitted electrons** yields scanning transmission electron microscopy (STEM) images of **particularly high resolution**. STEM has both materials and life sciences applications.





STEM image of mouse brain.

### **Correlative Microscopy (CLEM)**



Many scientific problems require the *combination* of SEM with other complementary microscopy techniques, such as light microscopy. This approach is called CLEM ("correlative light and electron microscopy").

In particle analysis applications, **light microscopy** provides the number and morphology of the particles, while **SEM** indicates elemental composition. Here, the targeted particle is metallic and rich in tin (Sn) and nickel (Ni).



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### **Electron Emitters** Comparison



	Tungsten	LaB <sub>6</sub>	Schottky FEG	Cold FEG
Source Size (nm)	> 50,000	5,000	50	5
Energy Spread (eV)	3	1	0.7	0.25
Brightness (A/cm <sup>2</sup> /sr)	10 <sup>6</sup>	107	> 5x10 <sup>8</sup>	> 5x10 <sup>8</sup>
Service Life (hours)	100-200	2000	> 2000	> 2000
Vacuum (Torr)	<b>10</b> <sup>-5</sup>	10-7	10-11	10 <sup>-11</sup>

Price

#### What does this mean?

Tungsten LaB<sub>6</sub> Schottky and Cold FEG "Light Bulb": High *total* emission current, but low *probe* current and large spot size. "Directed" thermal emission, somewhere between Tungsten and FEG "Laser": Lower *total* emission current, but high *probe* current in a small spot.

Performance

### **Electron Emitters** Comparison







Alloy, 1kV, 35kx, SE image

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## **SEM Column Design**

Single and Double Condensers





Effects of Beam Current (Aperture Size)





Selects the beam current (6 different diameters)

**Objective lens** 

Sample



20µm aperture Optimized optics 0.8nm @ 100pA/10kV

Effects of Beam Current (Aperture Size)





20µm aperture **Optimized** optics 0.8nm @ 100pA/10kV

7µm aperture Limited by diffraction 1.7nm @ 14pA/10kV

angle

Effects of Beam Current (Aperture Size)



**Condenser** Used to optimize the aperture angle

**Multi-hole aperture** Selects the beam current (6 different diameters)

**Objective lens** 

Sample



20µm aperture Optimized optics 0.8nm @ 100pA/10kV 7µm aperture Limited by diffraction 1.7nm @ 14pA/10kV 120μm aperture Limited by sph. aberr. 40nm @ 3.5nA/10kV

#### Resolution depends on the beam current! Optimized imaging conditions for discrete probe currents

Benefit of Double Condenser



**Condenser 1** Selects the beam current

**Fixed** aperture

**Condenser 2** Selects the aperture angle

**Objective lens** 

Sample



35µm aperture 0.8nm @ 100pA/10kV

Benefit of Double Condenser





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01/28/2019

Benefit of Double Condenser





Continuous regulation of probe current and optimized imaging at any voltage

## **Achieving Greater Resolution**

Moving Boundaries with Column Design





## **Objective Lens Design**

Magnetic + Electrostatic Hybrid



Electrostatic lens eliminates magnetic fields below the final lens to mitigate image distortion



## **Stage Biasing**





- Improve resolution (sub-nanometer from 1 kV to 30 kV)
- Enhance contrast by tuning bias voltage
- Improve detector efficiency for inlens detection as well as BSD detection
- Suppress charging artifacts.
#### **GEMINI Column** Beam Boost + Deceleration





- The energy of the primary electrons in the column is kept at above 8keV at all times
- Maintains beam at high tension throughout the column preserving brightness until final deceleration at the objective lens

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### **Optimizing Imaging Parameters** Accelerating Voltage



The <u>accelerating voltage</u> can typically be varied by the operator from < 1 kV to 30 kV on SEMs.

Increasing accelerating voltage will:

- decrease lens aberrations → smaller probe diameter → better resolution.
- increase the probe current at the specimen. A minimum probe current necessary for good contrast and a high signal to noise ratio
- potentially *increase* charge-up and damage in specimens that are non-conductive or beam sensitive

evaporated gold particles



30 nm carbon film over a Cu TEM grid







#### **Optimizing Imaging Parameters** Beam Probe Diameter



The <u>probe diameter or spot size</u> can be varied altering current to a condenser lens.

*Decreasing* the probe diameter will:

- decrease probe current
- enable greater resolution. Resolving small specimen features requires probe diameters of similar dimensions.
- *decrease* lens aberration due to a stronger lens setting





Smaller spot size: image is *sharper* but also *grainier* in appearance due to the lower signal to noise ratios associated with a lower beam current



Larger probe size: results in a *less sharp* but *smoother* image in appearance.

#### **Optimizing Imaging Parameters** Objective Aperture Size



The <u>aperture size</u> can be selected by the user

*Decreasing* the diameter of the aperture will:

- decrease lens aberrations → increase resolution
- decrease the probe current
- *decrease* the convergence angle of the beam → *increase* depth of focus



Light bulb filament





### **Optimizing Imaging Parameters** Working Distance



Working Distance is the distance from the final lens to the top of the sample

*Increasing* the working distance will.

- increase depth of focus • increase probe size BUT decrease resolution
- increase the effects of ٠ stray magnetic fields BUT decrease resolution
- increase aberrations due ٠ to the need for a weaker lens to focus



Specimen

#### Light bulb filament





#### **Optimizing Imaging Parameters** Focus and Stigmation







#### **Objective lens astigmatism**

- Revealed as streaking when going in and out of focus
- Corrected by applying current differently to a ring of stigmator coils around the objective lens
- Correct to obtain circular beam





Under-focused



Focused, Astigmated



Over-focused



Focused, Stigmated

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### **Electron Microscopes** Imaging difficult samples



Insulating or non-conductive samples: charging leads to artifacts and distorted images.



Example: paper fibers imaged at 5keV

### **Electron Microscopes** Imaging difficult samples



Insulating or non-conductive samples: charging leads to artifacts and distorted images.



#### **Local Charge Compensation**





- Surface discharging with nitrogen ions
- No additional high pressure detectors needed (full InLens detection)
- Fully automated, discharging within a few seconds



#### **Easily Eliminate Charging Problems** By switching on local CC





video link

### Variable Pressure (VP) Design





#### **VP** design

- Up to 133 Pa chamber pressure
- Gas molecules dissipate charge
- Loss of S/N, resolution

## **Difficult Samples**

Insulating or non-conductive

Imaging of insulating samples with Variable Pressure mode (VP)







#### **Effective Charge Compensation in NanoVP**



Fibrous polymer microstructures imaged at 3 kV in normal VP modes.



#### 20 Pa in normal VP mode, VPSE detector

Sample in courtesy of Dr. Hans-Georg Braun Leibniz-Institute of Polymer Research Dresden Max-Bergmann-Center of Biomaterials

#### Effective charge compensation in NanoVP



Fibrous polymer microstructures imaged at 3 kV in NanoVP modes.



#### 150 Pa in NanoVP mode, VPSE detector

Sample in courtesy of Dr. Hans-Georg Braun Leibniz-Institute of Polymer Research Dresden Max-Bergmann-Center of Biomaterials

## **Difficult Samples**

Insulating or non-conductive





Environmental SEM – freeze dried coffee beans and water vapor



to the full saturation of the coffee grain

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#### **Electron Microscopes** In summary



#### **Electron Microscopes...**

- ... use electrons instead of photons
- ... can achieve much higher resolution than light microscopes. The best ones can do < 1 nm (SEM) or 0.05nm (TEM)!
- ... use electromagnetic lenses instead of glass lenses
- ... adjust voltage, probe current, aperture size and working distance to optimize image quality and information
- ... have challenges imaging insulating samples, but modes to address available
- ... but can do more than "just" imaging

#### **ZEISS Microscopy Portfolio** Multi-Scale Characterization for Multi-Scale Research



#### A complete microscopy portfolio...



...to address multi-scale research challenges.



## We make it visible.



Resources:

- ZEISS <u>– Electron / Ion Microscopy</u>
- ZEISS <u>EM for Life Sciences</u>
- ZEISS <u>EM for Materials Science</u>
- ZEISS Correlative Microscopy
- ZEISS X-ray microscopy

Bob Hafner; Scanning Electron Microscopy Primer; Univ of Minnesota; 2007; link

#### **Electron Microscopy in Various Sectors** Serving Diverse Applications



**Materials Science** Industry Life Science **Basic Materials**, **Semiconductor Bio**, Pharma, **Functional Materials**, Cosmetics, Data Storage, **Medical Diagnostics**, Nanomaterials, **Telecom**, **Flat Panel Brain Research** 2D Materials **Electronics Battery**, Solar Metals, Alloys Nano Bio Tech Ceramics, Minerals, Silicon, Resist, Metals, **Drugs, Cells, Tissues Polymers, Composites Particles**, Catalyst







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- spatial resolution depends on wavelength:
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#### • typical acceleration voltages of electrons:

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	Light Microscopy	Electron Microscopy
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Spatial Resolution	~ 1 µm	~ 1 nm
Max. Magnification	1,000 x	1,000,000 x
Modes of Operation	reflected light, transmitted- light	SEM, TEM
Samples	Unmodified, hydrated	vacuum-compatible

De Broglie wavelength





## **Electron Emitters**

Tungsten







- Tungsten has the highest melting point and lowest vapor pressure of all metals, thereby allowing it to be heated for electron emission.
- V-shaped hairpin type
- Source size about 50µm
- Energy spread about 3eV
- Service life 100-200 hours
- Very cheap

Brightness  $\beta = 10^6 \text{ A/cm}^2/\text{sr}$ 

### **Electron Emitters** Lanthanum Hexaboride LaB<sub>6</sub>







- LaB6 filaments are single Lanthanum Hexaboride crystals ground to a fine point
- Source size about 5µm
- Energy spread about 1eV
- Service life up to 2000 hours
- More expensive than tungsten (10-20x)

Brightness  $\beta = 10^7 \text{ A/cm}^2/\text{sr}$ 

### **Electron Emitters** Schottky Field Emitters (FEG)







- Tungsten rod with a very sharp tip
- Tip is heated to about 1750K. Electrons are pulled out of the tip by applying an extraction voltage (tunnel effect)
- Source size about 0.05µm
- Energy spread about 0.7eV
- Service life > 2000 hours

Brightness  $\beta > 10^8 \text{ A/cm}^2/\text{sr}$ 

#### LaB6 versus Tungsten Comparison



A comparison of the main characteristics of LaB6 and tungsten electron sources:

	Lower work function allows		
Characteristic	electrons to be more easily emitted from LaB6 source (e.g. it is more efficient)	LaB6	Tungsten
Work function /eV	Lower work function allows a	2.4	4.5
Operating temperature /K	lower operating temperature → longerLaB6 lifetime	1700	2700
Brightness /relative to tungsten	LaB6 can be more than 50x as bright as a tungsten	>>50	1
Spot diameter improvement /relative to tungsten		1.5	1
Gun vacuum /Pa	Labb can produce a spot 1.5x tighter than tungsten	10-4	10 <sup>-2</sup>
Typical lifetime in normal mode /hrs	The above parameters ensure LaB6 has a lifetime of ~ 20	1000	50 - 100
Typical lifetime in long filament life mode /hrs	times that of tungsten	2000	>100
Resolution at 30 kV, 1pA /nm	The brightness and spot size contribute to a higher res.	2	3
Drift /% per hour	Less drift for LaB6	0.5	1
Approximate cathode cost /€	Although the cost of the cathode is higher, downtime costs need to considered	> 900	9

## **Limits to Resolution**

#### **Optical Aberrations**





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## **Limits to Resolution**

#### **Optical Aberrations**

What limits the resolution in a SEM?





Spherical aberration:

### **Chromatic Aberrations Effects on Probe Size**



Probe Size:

$$d_{P} = \sqrt{d_{S}^{2} + d_{C}^{2} + d_{d}^{2} + (M \cdot d_{g})^{2}}$$

 $d_c = C_c \frac{\Delta U}{\Delta U}$ 

 $-\alpha$ 

Chromatic aberration:

At low voltages the dominating effect that increases probe size is chromatic aberration.

There are 2 strategies to reduce the effect of chromatic aberration:



## **Chromatic Aberrations**

#### **Effects on Probe Size**



Probe Size:

$$d_{P} = \sqrt{d_{S}^{2} + d_{C}^{2} + d_{d}^{2} + (M \cdot d_{g})^{2}}$$

 $d_c = C_c \Delta U$ 

Chromatic aberration:

At low voltages the dominating effect that increases probe size is **chromatic aberration**.

There are 2 strategies to reduce the effect of chromatic aberration:



Energy width of a Schottky field emitter.

- 1. Reduce the energy spread *dU* (monochromator, filter)
  - usually accompanied by a huge loss of beam current!

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## **Chromatic Aberrations**

 $d_P = \sqrt{d_S^2 + d_C^2 + d_d^2 + (M \cdot d_g)^2}$ 

 $d_c = C_c \frac{\Delta U}{U} \alpha$ 

#### **Effects on Probe Size**

Probe Size:

Chromatic aberration:

At low voltages the dominating effect that increases probe size is **chromatic aberration**.

There are 2 strategies to reduce the effect of chromatic aberration:

- 2. Keep the electron energy *U* has high as possible and then decelerate the beam to the desired landing energy. One technique is stage biasing ("Beam Deceleration"), **but** 
  - Sample must be flat
  - Sample cannot be tilted
  - Sample must not be rough
  - Conductivity must be homogenous
  - Discharging can happen





# **Chromatic Aberrations**

Benefit of Beam Booster



Probe Size:

Chromatic aberration:





The GEMINI Beam booster advantage:

- The electron energy *U* is kept at above 8keV followed by *deceleration in the column*.
- No stage biasing is required
- No energy filtering needed
- Very high resolution even at (ultra) low voltages
- No loss of brightness and probe current at low voltages
- No alignment needed when switching from high to low voltages
- Does not influence the focused ion beam

### **GEMINI Column** Improvements in GEMINI Technology - GEMINI II column





### **GEMINI Column** Improvements in GEMINI Technology - GEMINI II column





Continuous variation of beam current and acceleration voltage. No realignment needed.

#### **Comprehensive Signal Detection**





**Inlens Duo:** Inlens SE and BSE detector for high resolution topographical and compositional imaging of surfaces, thin films and nano particles.



ETSE Detector: High resolution topographic imaging with 50% more signal to noise and reduced charging at low kV in high vacuum mode



VPSE-G4: Fourth generation Variable Pressure SE detector provides 85% more contrast



**C2D**: Cascade Detector for the ultimate performance in VP mode even at higher pressures.



**aSTEM:** Annular STEM detector provides HAADF modes for "TEM-like" imaging of thin films or biological sections



HDBSD: High definition BSE-detector for excellent low kV compositional imaging of all samples in all vacuum modes



AsB Detector: Angular selective BSE-detector enables crystallographic & channelling contrast imaging of metals and minerals mounted in the final lens.



**YAG-BSD**: YAG crystal based scintillator BSE-detector provides fast, easy compositional imaging



**BSD4**: 4 parallel output BSE-detector provides "real-time" 3D imaging and surface metrology.



**Dual EDS**: Diametrically opposed EDS detectors at 8.5 mm working distance & 35 degree take-off angle delivers highest analytical productivity with results delivered twice as fast.
# **On-Axis In Column Detection System** Different electrons carry different information





### **Comprehensive Signal Detection** Inlens Duo



### **Inlens Duo:**

- Affordable in-column SE & BSE detection - two detectors in one
- Sequential compositional and topographic imaging of layers, coatings, thin films and nano-particles







Compositional imaging with on-axis in-lens BSE detection Imaging with Quad mode ...more information in less time Sample courtesy of D. Willer, MPA Stuttgart



#### InLens

**EsB** 

N 47 - 11/

SE2

0.76

Ag / Ni / Cu sample

ate water was

InLens + SE2

CALIFORNIA DAY IN AN INTERACTION OF A DAY OF AND A DAY OF A DAY OF

 Mag =
 1.31 K X
 3 μm
 WD =
 5.2 mm
 EHT =
 1.80 kV
 Signal A =
 SESI
 E

 Crossbearn 540
 →→→
 FIB Imaging =
 SEM
 Noise Reduction =
 Line Avg
 FIB FIB

ESB Grid = 1400 V Date :14 Jul 2014 Time :17:36:26 FIB Probe = 30kV:700pA System Vacuum = 1.79e-006 mbar

# **In-lens EsB Detection** Tunable energy selective backscatter



EsB detector on CNT with catalyst nanoparticles



# Analysis Beyond EDS ZEISS FESEM + Raman spectroscopic imaging









# Nonmetallic compositions and phases:

- Polymorphs
- Molecule phases
- Polymer blends
- Crystal orientations
- Fibrillary polymer
- Degree of Crystallinity
- Kinds of Defect
- Stress and Strain
- Surface coating
- Contamination



- Nanoparticles & 2D Mat.
- Polymer
- Carbon materials
- Semiconductor
- Geology/ Mineralogy
- Forensic
- Pharma
- Biomaterials
- Coatings
- Corrosion





# Why Correlative Microscopy? Zoom in from micro to nano





Rapid light microscopy overview – low resolution

Targeted SEM imaging– high resolution

### **Correlative Microscopy** Correlation of functional and structural information





# X-ray Tomography MicroCT and X-ray Microscopy



### Sample Center of Rotatation Source Detector Detector Detector Detector Detector

Resolution degrades substantially as

the sample moves away from the source

**Projection** based architecture

٠

**Conventional MicroCT** 





- Two-Stage Magnification for unprecedented resolution and contrast
- High resolution is maintained as the sample moves away from the source

