■ IHFG Seminar 2012: Nanooptics and Nanophotonics

Structural analysis of nanostructures by electron microscopy

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This short manuscript outlines a talk I gave in 2012 at the IHFG at the University of Stuttgart as a contribution to the *IHFG Seminar 2012: Nanooptics and Nanophotonics*. It provides the reader with the conceptual foundations of electron microscopy and its primary modes of operation. The scope of this work encompasses a brief description of electron guns and electron optics followed by a survey of the most important imaging techniques regarding scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Although the original talk was motivated by the analytical requirements of nanooptics and nanophotonics, the scope of the manuscript at hand remains widely unbiased and may be useful as a general introduction to the field of electron microscopy (EM).

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Structure. This manuscript is organized as follows. In the first section (I) we motivate the use of electrons to analyze nanoscopic structures and provide some facts about the SEM and the TEM. In the second section (II) we describe common technical details of the SEM and TEM, namely electron guns and electron optics. In the third section (III) technical peculiarities and imaging techniques of the SEM are covered. The last section (IV) deals with the TEM and its modes of operation.

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I. INTRODUCTION

A. Structural Analysis of Nanostructures

Nanooptics and nano photonics is a prospering field of modern physics aiming at the manipulation of electromagnetic waves capitalizing the possibilities of quantum mechanics for the interaction of light and matter on nanoscopic length scales. Elaborate epitaxy and etching techniques facilitate the construction of structures such as photonic crystals and quantum dots which may play a crucial role in future applications. Construction-conditioned the details of such structures are characterized by a nanometer length scale (even if the whole structure has a typical length scale of micrometers). In order to control the production processes and adjust the chosen geometries and configurations, imaging techniques of nanoscale resolution and preferably more extensive analytical capabilities are required.

Scientists always were interested in structures on small length scales. Therefore the field of optical microscopy started developed in the 17th century and, until now, provided the applied sciences with a variety of highly specialized imaging techniques. For instance:

- Bright/Dark field microscopy
- Phase contrast microscopy
- Fluorescence microscopy
- Confocal microscopy
- Ultraviolet/Infrared microscopy
- Laser microscopy

However, bound by the wavelength of visible (or nearly visible) light, the resolution of these methods usually cannot exceed $\sim 100\,\text{nm}$. Furthermore, the contrast mechanisms rendering optical microscopy a viable solution are usually not met by semiconductor-based structures. Hence imaging techniques which avoid light as carrier of structural information prove more useful.

Here we mention the

- atomic force microscope (AFM)
- and the scanning tunneling microscope (STM).

Both methods reach atomic scale resolutions ($\sim 0.01\,\text{nm}$) and therefore are well established as analytical instruments for the characterization of artificial nano structures. For more details I refer the reader to a manuscript by Fabian Ripka which summarizes his talk about the AFM and the STM.

However, the most commonly used imaging techniques in the field of nanooptics and nanophotonics certainly are the

- scanning electron microscope (SEM)
- and the transmission electron microscope (TEM).

These microscopes reach atomic resolutions up to $\sim 0.05\,\text{nm}$. Not only their high resolution but also their flexibility regarding the measured signal (and therefore the employed contrast mechanism) make them the tools of choice for the analysis of artificial metal and/or superconductor based nanostructures. In the following sections the setup, technical aspects and the modes of operation of these two commonly used microscopes will be covered on an introductory level.

B. Electron Microscopy: SEM & TEM

Both, the SEM and the TEM use accelerated electrons (instead of photons) to obtain information about the structure of the specimen. The question that may arise at this point is:

1. Why Electrons?

We first note that the resolution of diffraction limited optical imaging (e.g. optical microscopes) is bound by

$$d = \frac{0.61 \cdot \lambda}{n \cdot \sin\alpha} \approx \frac{\lambda}{2 \cdot \text{NA}} \geq \frac{\lambda}{2} \tag{1}$$

where n denotes the refraction index between sample and objective lens, α is the (half) opening angle of the objective lens as seen from the sample and NA is called

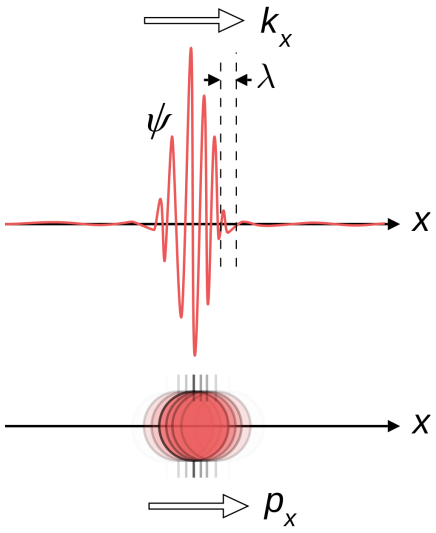

Figure 1 Particle-wave duality. In quantum mechanics an electron is described as a wave packet. Due to the uncertainty principle, position and momentum cannot be determined with arbitrary precision. Source: http://en.wikipedia.org/wiki/Particle-wave_duality

"numerical aperture". Obviously the resolution d cannot be less than half the wavelength λ of the used light. Well, this argument holds for any wave-based imaging technique and by employing the paradigm of particle-wave duality (see Fig. 1), that is the de Broglie wavelength

$$\lambda_e = \frac{h}{p_e} = \frac{h}{v \cdot m_e} \tag{2}$$

of an electron with mass m_e and velocity v (h denotes the Planck constant), we arrive at theoretical resolutions $\lambda_e \sim 0.005\,\text{nm}$ of an electron-wave based imaging technique (e.g. TEM). Here we assumed an accelerating voltage $U_a \sim 50\,\text{keV}$ which is technically feasible and also typical for modern electron microscopes (regarding the order of magnitude). If we compare this result with the typical wavelength of visible light $\lambda \sim 500\,\text{nm}$, it becomes clear why physicists and engineers strived towards the electron-analogue of optical microscopes, namely the TEM. Unfortunately the ratio of theoretical (see above) and practical improvement of resolution with respect to optical microscopes reads $100,000/1,000$ and we conclude that current electron microscopes are **not** diffraction limited. As I will point out later, the responsible drawbacks are due to imperfections of the electron optics and/or the way the electrons interact with the sample.

Let us now proceed with a short summary of facts and figures for the SEM and the TEM. The purpose of the next two paragraphs is to provide the reader with a rough feeling for the relevant scales and magnitudes

as well as the capabilities of the imaging technique in question.

2. Scanning Electron Microscopy (SEM)

A schematic sketch of an SEM is depicted in Fig. 2. The electron beam is generated at the top by means of an electron gun. Subsequently the beam is condensed and deflected by electron lenses. The objective lens focusses the beam on the sample after the latter passes trough the final aperture. The signals (e.g. secondary electrons) may now be recorded by detectors mounted above the specimen. Since the electron beam is scanning the probe, this yields a spatially resolved map of the detected signal and therefore the corresponding sample properties.

Thus SEMs are widely used to analyze the surface morphology via interaction of the sample with the beam of high-speed electrons. Since the electrons do not have to be transmitted trough the specimen, the latter may be thick; however, it has to be be conductive. Otherwise the applied charges may lead to undesired contrast mechanisms. The best resolution of SEMs depends on the imaging mode, is approximately $3 - 6\,\mathrm{nm}$ and is basically bound by the beam thickness. SEMs are characterized by a large magnification range of about $20 - 150,000\times$ and an outstanding depth of field[1] ($0.003 - 1\,\mathrm{mm}$) which is responsible for the plasticity of typical SEM images (since there is no visible focal plane). What structures appear on an SEM image depends on the detected signal. Typical SEMs facilitate the detection of several signals, e.g. secondary electrons, backscattered electrons, X-rays and photons. Each of these "information channels" is sensitive to different contrast mechanisms and therefore allows the imaging of different structures and/or material properties of the sample. A more detailed explanation follows in Section III.

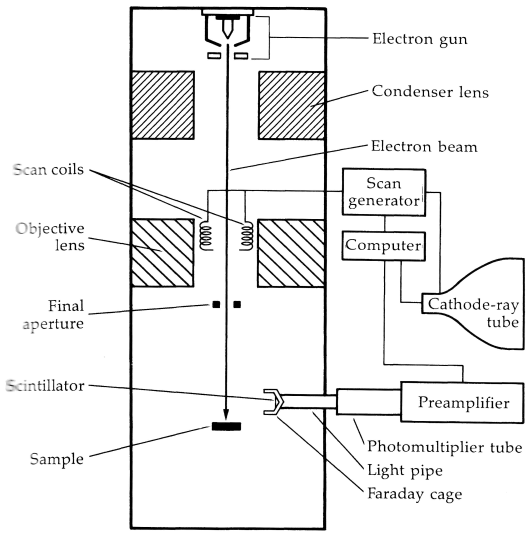

Figure 2 Schematic sketch of a scanning electron microscope (SEM). Source: Flegler *et al.* (1993)

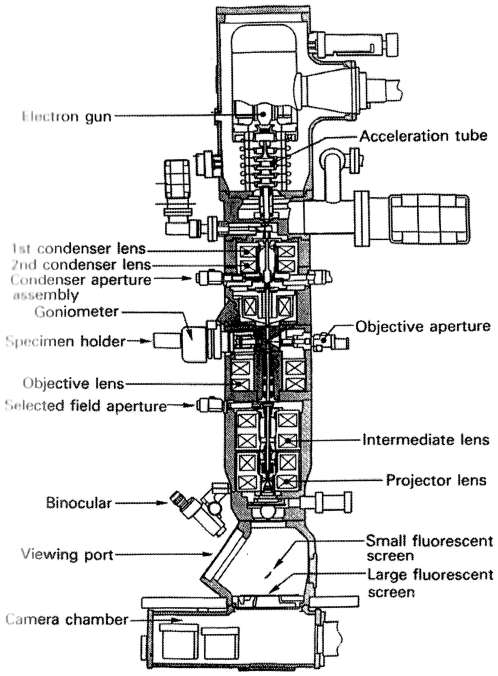

Figure 3 Technical sketch of a transmission electron microscope (TEM). Source: Fultz and Howe (2002)

3. Transmission Electron Microscopy (TEM)

A technical sketch of a TEM is depicted in Fig. 3. As in the case of an SEM, an electron gun generates a beam of electrons which is condensed by a set of magnetic lenses. The objective lens focusses the beam on the thin sample. The transmitted and scattered electrons are refocussed by a set of magnetic lenses below the specimen and form a real image on a fluorescent screen or a digital camera. Therefore the TEM is - to a certain degree - an optical microscope where the photons have been replaced by electrons.

Generally TEMs are used to analyze thin sections of organic or artificial structures via the transmission of high-speed electrons. Since the electrons have to be transmitted, thin samples are required ($40 - 150\,\mathrm{nm}$)

[1] The "thickness" of the focal plane, that is the interval along the optical axis in which structures appear in focus *at the same time* due to the finite resolution power.

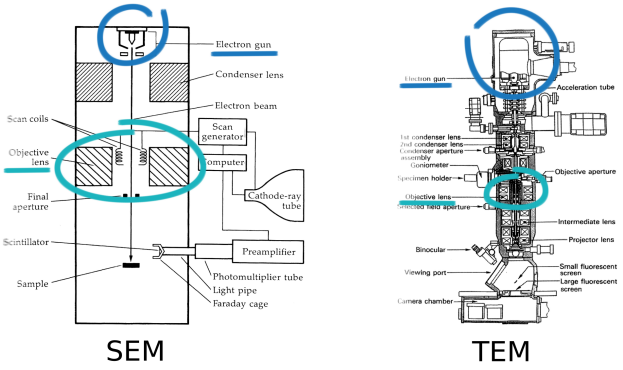

Figure 4 Technical similarities of the SEM and the TEM. In both cases an electron source and electron optics is required.

which usually leads to intricate sample preparation procedures. The resolution of TEMs surpasses the resolution of SEMs due to the reduced interaction volume of electrons and matter. Using high resolution imaging, details with 0.05 nm are resolvable. The limits on resolution power are imposed by the magnetic lenses and their inevitable aberrations. TEMs reach magnification ranges of about $500 - 500,000\times$ and a "large" depth of field: $0.004 - 0.006$ mm ("large" since the samples are much thinner). As for the SEM, a TEM provides various imaging modes, each mapping different properties of the specimen. The most important being dark field, bright field and HRTEM imaging modes. These (and a few more) will be described in Section IV.

4. SEM & TEM : Technical similarities

If one considers the schematics in Fig. 2 and Fig. 3, it is easy to see that, despite the quite different modes of operation, there are several common components to both, the SEM and the TEM. For their basic principles - electrons as information carrier - coincide, both require at least an electron source (called an "electron gun") and some controlling elements for these electrons, namely electron optics (see Fig. 4). Therefore the next section will cover these two aspects in general - providing the technical foundations for both microscopes.

II. CONTROLLING ELECTRONS

A. Electron guns

In the following the two most commonly used electron sources, namely the tungsten-hairpin design (a thermionic electron gun (TEG)) and the field emission gun (FEG) will be treated briefly.

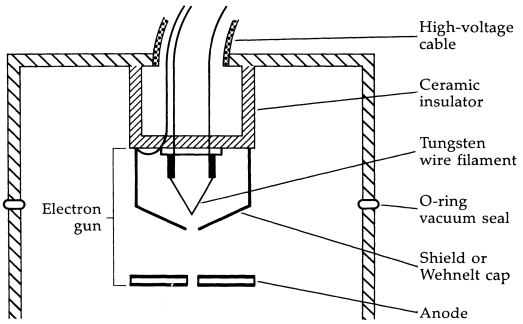

Figure 5 Schematic sketch of a thermionic electron gun with tungsten-hairpin design. Source: Flegler *et al.* (1993)

1. Thermionic Electron Gun (TEG) : Tungsten-hairpin design

Thermionic electron guns, as schematically depicted in Fig. 5, use thermionic emission of electrons from a heated filament. To this end, the tungsten-hairpin design employs a heated loop of tungsten as electron emitter. A negatively charged cap (so called "Wehnelt cap") serves as a primitive electrostatic lens and focusses the beam between the hole of the cap and the anode. Furthermore its potential controls the current of the beam. Subsequently the positively charged anode accelerates the electrons and thus controls their kinetic energy (namely their de Broglie wavelength λ_e).

The advantages of the tungsten-hairpin design are the following:

- Relatively stable source of electrons
- Inexpensive
- No ultrahigh vacuum required

This setup was the most commonly used design; however, nowadays it is successively replaced by FEGs (see next paragraph).

2. Field Emission Gun (FEG)

Field emission guns make use of the emission of electrons from the surface of conductors into the surroundings at locations of strong electric field; this effect is known as "field emission". To this end, a (cold) tungsten tip, as depicted in Fig. 6 (B), is opposed to the first anode (see Fig. 6 (A)) which creates a strong electric field at the very end of the tip. Subsequently the emitted electrons are accelerated by the second anode which is used to adjust the kinetic energy of the electrons. An adjustment of the potential of the first anode is used to control the current of the electron beam.

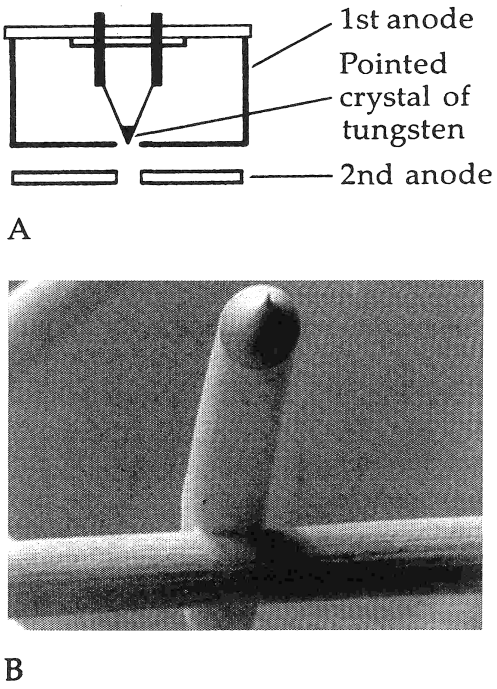

Figure 6 A: Schematic sketch of a field emission gun. B: SEM image of the cold tungsten tip. Source: Flegler *et al.* (1993)

Due to the following advantages (in comparison to the tungsten-hairpin design) FEGs are replacing TEGs gradually:

- Beam current up to 1000 ×
- Smaller area of emission
- Energy spread is about 1/10
- Duration of life 100 ×

The main advantage being the long duration of life since TEG based setups require regular replacement of the filament which in turn necessitates breaking the vacuum in the microscope. However, there are also disadvantages - mainly of technical nature:

- Dedicated design of microscope necessary (not compatible with the formerly used TEGs)
- Unstable intensity

Admittedly, most disadvantages were straightened until now due to technological progress.

B. Electron optics

Electron optics is used to control a beam of electrons in the same way as classical optical devices control the behavior of electromagnetic waves in the visible or nearly visibly frequency range. Aiming at the deflection and focussing of electrons, two approaches seem viable: One may employ electrostatic or magnetic fields. However, for technical reasons electrostatic lenses were abandoned and modern EMs rely solely on magnetic lenses.

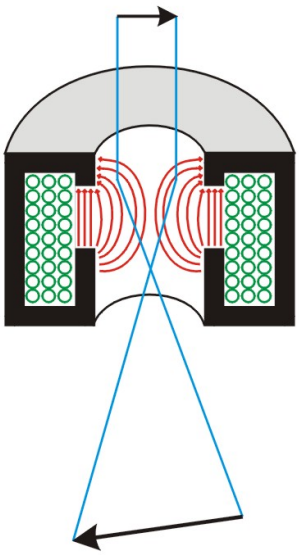

Figure 7 Schematic longitudinal section of a simple magnetic lens which, in fact, is simply a coil with adjustable current covered by soft iron to concentrate the magnetic field along the depicted gap. Source: `http://www.microscopy.ethz.ch/lens.htm`

1. Magnetic lenses

Principle. Modern magnetic lenses are so called "pole-piece lenses" which are built up of a coil around a hollow core covered with soft iron ("shroud"), see Fig. 7. A circular, narrow gap without covering concentrates the magnetic field of the coil within a small volume around the gap. The strong, inhomogeneous magnetic field causes a deflection and focussing of fast electrons due to the Lorentz force

$$\mathbf{F} = -e \cdot \mathbf{v} \times \mathbf{B}\,. \tag{3}$$

It is quite intuitive that the focal length depends on the

- **coil current**,
- the radial distance (→ spherical aberration)
- and the velocity (→ chromatic aberration).

Therefore the coil current can be used to adjust the focus of each magnetic lens in the microscope continuously. However, the latter two dependencies are undesired and lead to aberrations which in turn limit the

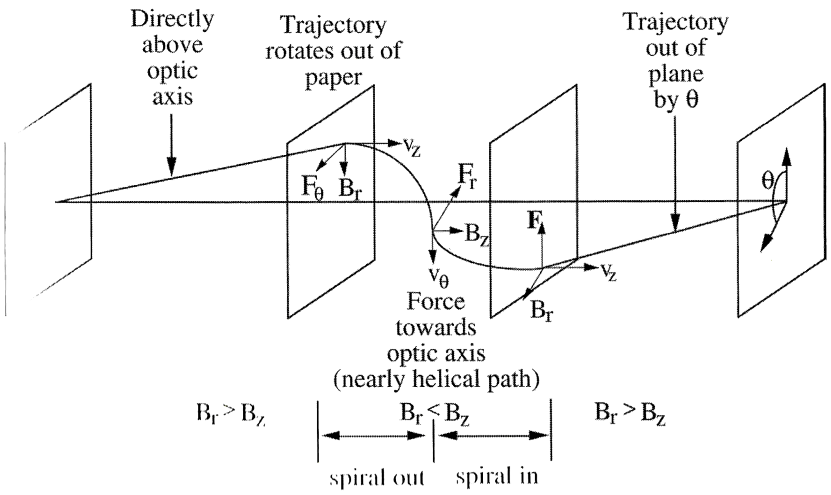

Figure 8 Trajectory of an electron flying through a simple magnetic lens. Between the two central planes the magnetic field is approximated by the field configuration of a short coil. Due to the radial component of the field on the left and right side of the coil, the electron starts spinning around the optical axis. The angular velocity component and the magnetic field collinear to the optical axis then accelerate the electron towards the optical axis. Source: Fultz and Howe (2002)

resolving power of the imaging system. Please note that chromatic aberration, caused by the energy dependence of the focal length, can be reduced by electron sources with small energy spread, namely FEGs.

Electron trajectory. To gain a more thorough understanding of a magnetic lens, consider a simple short coil (without shroud) and its magnetic field configuration as a toy model. An electron hits the magnetic field at a non-zero angle above the optical axis, as depicted in Fig. 8. Here the magnetic field of the coil is confined to the volume between the two centered planes and is radially symmetric with respect to the optical axis.

At the beginning, the radial component of the magnetic field dominates ($B_r > B_z$) and the electron starts spinning around the optical axis. Somewhere near the center of the coil the magnetic field component B_z (collinear to the optical axis) becomes the dominant one, $B_z > B_r$. Since the electron now has an angular velocity component, the Lorentz force accelerates it towards the optical axis. Henceforth the electron follows a narrowing helical path. The spinning ceases at the right end of the coil due to the strong radial component $B_r > B_z$. Only the velocity component towards the optical axis survives; thus the electron was refocussed.

Adjusting the focus. The attentive reader may have noticed that the refocussed electron in Fig. 8 is rotated by an angle θ. This angle obviously depends on the strength of the magnetic field and therefore on the focus (which is controlled by a variation of the magnetic field). We conclude that magnetic lenses impose a focus dependent rotation on the image since the aforementioned helical path "shrinks" or "expands" for different coil currents. This effect is shown in Fig. 9.

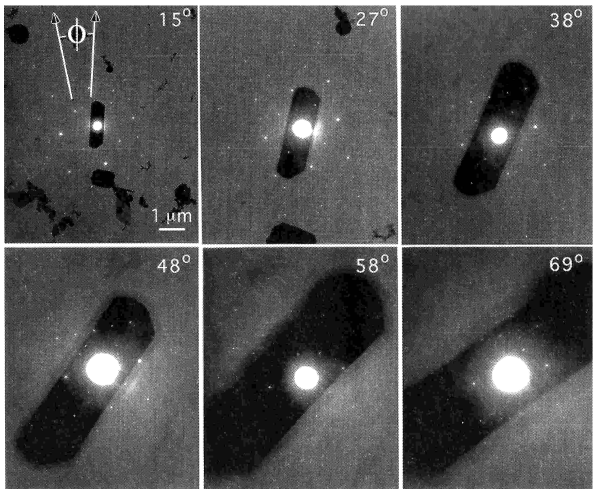

Figure 9 TEM image with a (fixed) diffraction pattern overlay (white dots). Adjusting the focus (that is, the coil current) is accompanied by a rotation of the image. Source: Fultz and Howe (2002)

This is a technical but important observation since e.g. TEM images may be compared to diffraction patterns of the same sample. To ensure comparability of images obtained under different focal setups or magnifications (that is, coil current configurations), it is crucial for the operator to know how the lens adjustments affect the image orientation.

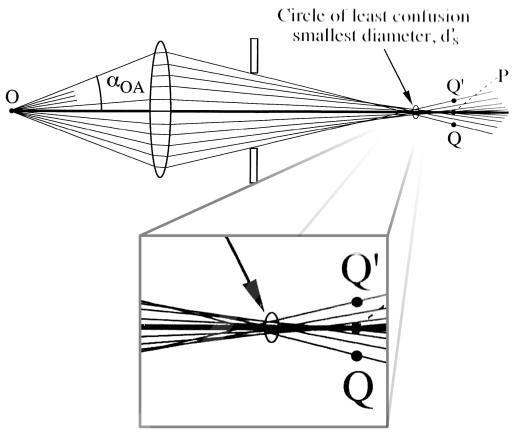

Figure 10 Spherical aberration in the case of an optical lens. Owing the radial distance dependent refraction, there is no distinct focal point. Therefore a point source will be mapped onto a small disc which certainly affects the resolving power. Source: Fultz and Howe (2002)

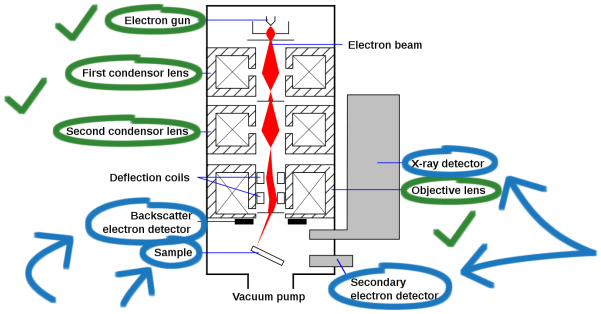

Figure 11 Schematic sketch of an SEM equipped with detectors for backscattered electrons, secondary electrons and X-rays. Source: http://en.wikipedia.org/wiki/Scanning_electron_microscopy

2. Aberrations

As for classical optical systems lens aberrations impose a limiting factor on the resolving power of EMs, see Fig. 10 for an example. As mentioned in the introductory paragraph, the order of magnitude of the theoretical improvement of resolving power[2] is about 100,000 (assumed that the imaging process is diffraction limited). However, in reality the improvement is of order 1,000. The actual limiting factors are the following aberrations which are well-known in optics:

- Spherical aberration
- Chromatic aberration
- Astigmatism

I already mentioned that chromatic aberration may be counteracted by appropriate electron guns with small energy spread. Astigmatism is a well-correctable aberration. The corresponding process is called "astigmation" and performed regularly by means of multipole lenses which are fixtures in modern EMs.

III. SCANNING ELECTRON MICROSCOPY (SEM)

The following section covers some technical details of the scanning electron microscope; especially the detection of different signals produced by the high-speed electrons interaction with the sample. The latter give rise to various imaging modes, each revealing specific structural properties of the sample. These imaging modes are subject to the second part of this section.

A. Technical Issues

1. Layout

The layout of a typical SEM is depicted schematically in Fig. 11. The green marked devices (namely the electron gun and electron optics) were already covered in the previous section. In the following the blue marked modules will be treated briefly. That is, I will outline different detection methods for photons and electrons and give an introduction to sample preparation techniques.

2. Available Signals

When the accelerated electrons, focussed by the objective lens and controlled by the deflection coils, hit the sample, several interactions between the beam and the specimen take place. The deposited energy leads to the emission of high-energy photons (X-rays) or photons in the (nearly) visible frequency range (called "Cathodoluminescence"). On the other hand, secondary electrons with comparatively low energies may be emitted (SE) and some of the incident primary electrons may be backscattered (BSE) at high angles and with high kinetic energies[3]. In crystalline samples phonon excitations will be triggered by the scattered electrons and

[2] Compared to optical microscopes.

[3] Furthermore "Auger electrons" (AE) can be observed; however, we will omit them in the following.

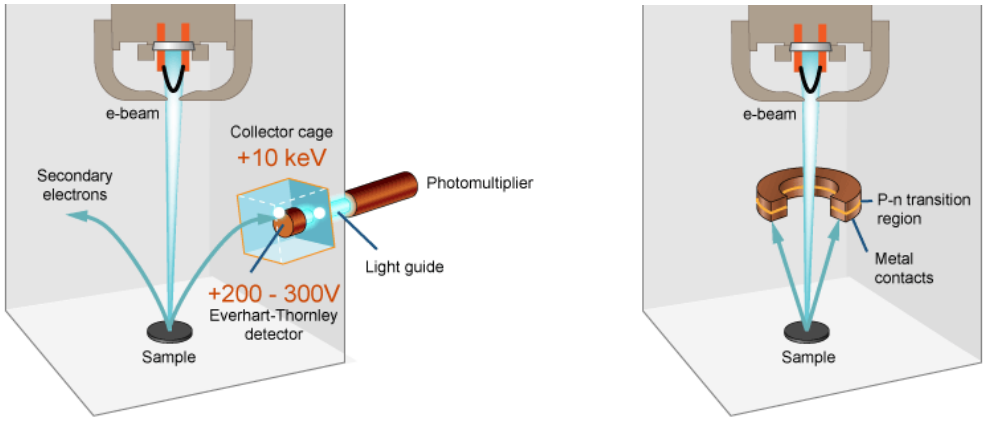

Figure 12 **Left:** Sketch of a common secondary electron detector (Everhart-Thornley detector). **Right:** Sketch of a common backscattered electron detector (solid state diode detector). Source: http://www.ammrf.org.au/myscope/sem/

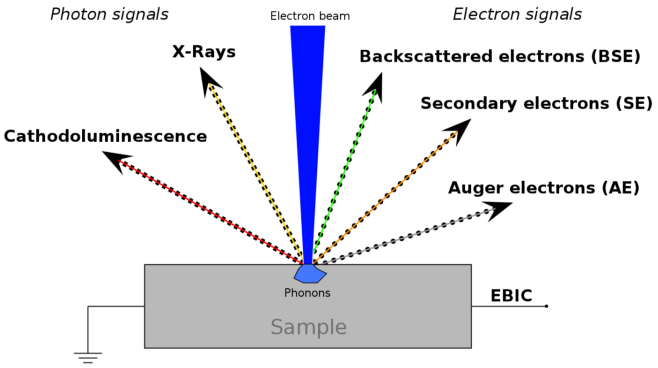

Figure 13 The incident high-energy electrons generate different signals which may be used to gain spatially resolved information about the properties of the specimen.

in semiconductors electron-hole pairs can be created by the incident electrons leading to so called "Electron Beam Induced Currents" (EBIC). All these signals or "information channels" can be employed to gain spatially resolved information about the properties of the specimen. They are depicted schematically in Fig. 13.

3. Detecting Electrons

To detect electrons emitted from the surface, two different detectors are placed above the sample. The secondary electron detector is sensitive to low-energy secondary electrons whereas the backscattered electron detector responds only to high-energy electrons. Therefore a separate detection of these two types of electrons is possible which proves very useful since they carry information about different properties of the sample.

A commonly used secondary electron detector is the so called "Everhart-Thornley detector", see Fig. 12 (left). The slow electrons are attracted by a positively charged metal film which is attached to a plastic scintillator. They penetrate the anode and lead to an emission of photons in the scintillator. These photons are guided through an optical fiber to a photomultiplier tube outside the vacuum chamber of the microscope. There the photons are converted to an electrical current which may be further amplified. Since only comparatively slow electrons will be attracted by the positively charged film, the Everhart-Thornley setup is sensitive to electrons with typical energies of $\sim 2 - 50\,\mathrm{eV}$.

To detect high-energy backscattered electrons with typical energies of $\sim 20\,\mathrm{keV}$ annular solid state diode detectors are employed. Such a setup is schematically depicted in Fig. 12 (right). The basic principle is quite simple: A semiconductor based diode operates in reverse direction. An incident electron creates electron-hole pairs which in turn induce a measurable current through the pnp/npn-junction.

4. Detecting Photons

The detection of photons in the visible or nearly visible frequency range has to deal with the small numbers of emitted photons. Thus a high detection efficiency is required. Therefore the sample is placed at one focus of an elliptical mirror as depicted in Fig. 14. A light pipe is placed at the other focus and carries the photons to a photomultiplier tube (PMT). If a dispersive element (e.g. a prism- or FT-spectrometer) is placed in front of the PMT, even a spectral analysis of the emitted light is possible. As a rule, modern SEMs are capable of such a spectral analysis, provided they are equipped with a module for cathodoluminescence imaging.

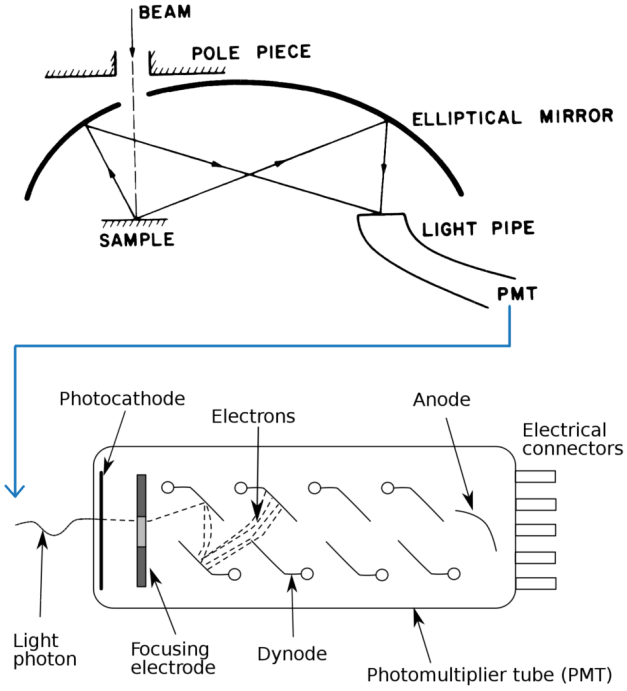

Figure 14 Detection of photons in the visible or nearly visible frequency range. An elliptical mirror collects the photons and passes them through an optical fiber to a photomultiplier tube. Source: Newbury (1986) Source: http://en.wikipedia.org/wiki/Photomultiplier

5. Detecting X-Rays

The detection of X-rays is usually combined with a spectral analysis of the photons; the most commonly used being energy-dispersive spectroscopy (EDS) which is often called "EDX". To this end a Si(Li)-crystal absorbs X-rays which leads to the creation of electron-hole pairs (their number being proportional to the energy of the X-ray photon). Subsequently these newly created charges are extracted by an applied voltage and induce a current proportional to the energy of the original X-ray photon. A schematic setup for an EDX detector is depicted in Fig. 15. A feature of EDX detectors (compared to other methods) is a wide scan range and short measuring times for a complete scan. Hence it is feasible to scan a specimen in a reasonable period of time to obtain a spatial distribution of different elements.

6. Sample preparation

The preparation procedure for an SEM imaging process depends on the sample being studied and the imaging mode being used. One of the features of scanning electron microscopy is that intact, solid samples can be imaged without sectioning (this is not possible if one prepares the sample for a TEM). However, the samples must be ...

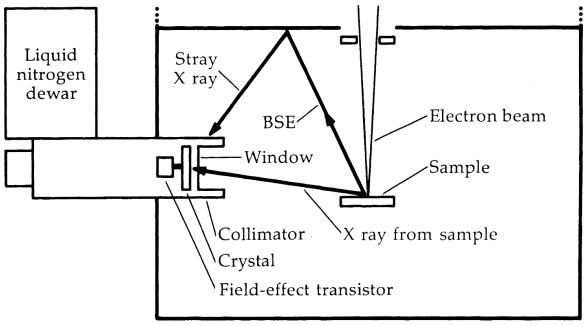

Figure 15 Schematic setup for energy-dispersive spectroscopy, that is, x-ray detection. Note that X-rays from the interaction of backscattered electrons with the adjacent equipment may cause distortions in the spectrum and should be blocked efficiently. Source: Flegler *et al.* (1993)

- devoid of volatile materials (e.g. water),
- firmly mounted (on stubs, see Fig. 16)
- and electrically conductive.

The first requirement follows from the fact that the specimen has to be stable in the hard vacuum of the microscope. Volatile materials would vaporize and destroy the sample and/or contaminate the magnetic lenses and apertures. The third requirement is due to the fact that the electron beam deposits charges on the sample which in turn distort the SEM image (especially secondary electron (SE) images).

Due to these requirements, biological samples usually require an involved preparation procedure (fixa-

Figure 16 Stubs used for mounting specimens in order to prepare them for an SEM imaging process. Source: http://en.wikipedia.org/wiki/Scanning_electron_microscope

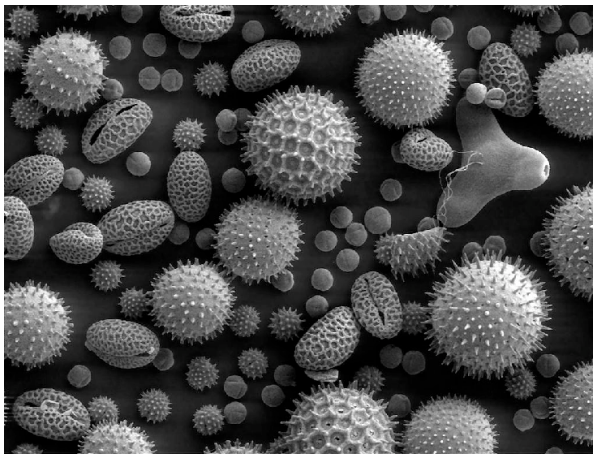

Figure 17 A typical secondary electron (SE) image which shows a collection of pollen. The plasticity of the image is due to the contrast mechanisms described in the text. Source: http://en.wikipedia.org/wiki/Scanning_electron_microscopy

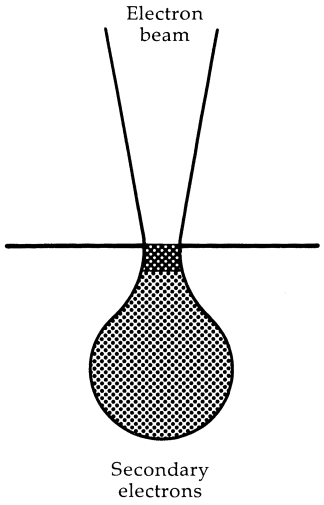

Figure 18 Interaction volume of the incident electrons (light shaded). The secondary electrons escape only from the dark shaded volume, though. Therefore the SE imaging mode provides the highest resolution possible with an SEM. Source: Flegler *et al.* (1993)

tion, coating and mounting). Plastic and ceramic based samples do not contain volatile materials and therefore require a less extensive preparation (just coating and mounting). Obviously the preparation of metals and semiconductors is fairly simple since their natural conductance is usually high enough. Therefore scanning electron microscopy is one of the most frequently employed imaging techniques within the realms of nanooptics and nanophotonics.

B. Operation modes and contrast mechanisms

We already learned that there is a variety of potentially useful signals escaping from the specimen (recall Fig. 13). In the following these signals will be linked to properties of the sample, that is to say, we are going to establish the contrast mechanisms used by the different imaging modes of modern SEMs.

1. Secondary Electrons (SE)

The most commonly used imaging mode uses the low-energy secondary electrons (SE) collected by an Everhart-Thornley detector. Basically the SE imaging mode can be viewed as the standard mode of an SEM and as a consequence SE images are the most prevalent ones in literature, e.g. see Fig. 17.

There are several contrast mechanisms responsible for structure in SE images:

- *Surface tilt contrast*: Surfaces tilted towards the Everhart-Thornley detector appear brighter than averted ones since more secondary electrons reach the detector if their initial trajectory points towards the anode. Therefore SE images appear to be "illuminated" from the very position of the detector.

- *Shadowing contrast*: Large objects prevent secondary electrons from one side to reach the detector on the other side. That is, areas behind large objects appear darker, for instance see Fig. 17. This is coherent with the idea of an "illumination" by a light source located at the detector.

- *Edge contrast*: Regarding Fig. 18 it is easy to see that a beam which hits a (nearly) vertical structure features a larger interaction volume near the surface and consequently generates more detectable secondary electrons. In a nutshell: Sharp edges and scratches appear bright in SE images.

- *Voltage contrast*: Generally one has to think thoroughly about the significance of structures *shown* by electron micrographs and to which extent they correspond to actual structures of the sample. To illustrate such difficulties we mention the voltage contrast which is used for the spatially resolved measurement of electric potentials in semiconductor industry, see Fig. 19. The brightness of SE images obviously depends on the local electric potential since more of the low-energy secondary electrons escape from negatively than from positively charged areas. This effect also causes (undesired) contrasts between differently charged ar-

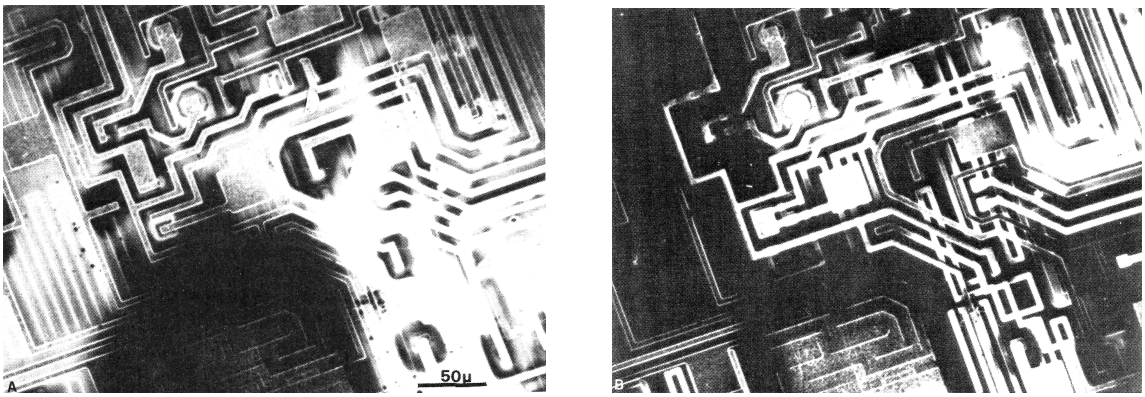

Figure 19 Illustration of the voltage contrast. **Left**: Printed circuits as part of an integrated circuit (IC) without potentials. **Right**: The same image with applied potentials. Obviously the circuits brightness depends on their potential. Actually one can use this to measure the potentials quantitatively. Source: Newbury (1986)

eas which occur on samples of low conductivity due to the inevitable charge deposition.

Due to these mechanisms (but for the last one) SE images show the topography of the sample with the highest SEM resolution ($\gtrsim 0.5\,\mathrm{nm}$) possible. The high resolution can be explained by the small fraction of the whole interaction volume from which SE electrons can escape, see Fig. 18 (remember that their kinetic energy is low).

2. Backscattered Electrons (BSE)

A second widely used imaging technique uses the high-energy backscattered electrons collected by the solid state diode detector. Here the relevant contrast mechanisms are:

- *Material contrast*: The scattering of the incident electrons is highly dependent on the (average) atomic number Z of the specimen at the current position of the beam. Therefore regions with heavy elements appear brighter in BSE images. This effect is called "material" or Z-contrast.
- *(Surface tilt contrast)*
- *(Shadowing contrast)*
- *(Voltage contrast)*

Usually the desired mechanism for BSE imaging is the Z-contrast alone. However, as in the SE imaging mode, the three additionally mentioned and already known mechanisms may distort the image and have to be taken into account to prevent misinterpretations.

In the end BSE images provide information about the spatial distribution of the average atomic number Z, that is a spacial distribution of elements. For an example see Fig. 20.

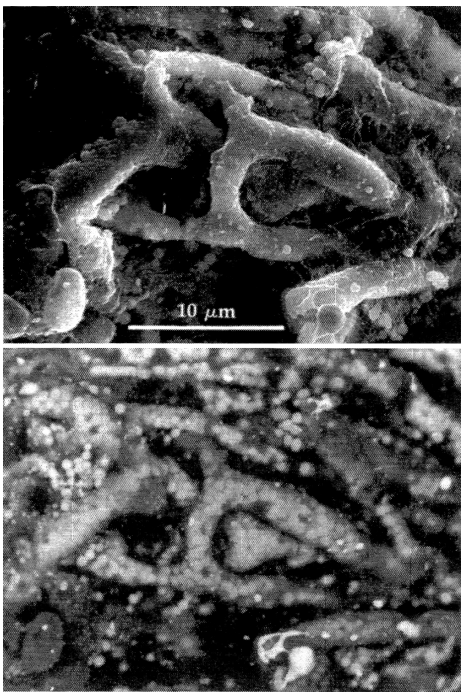

Figure 20 **Top**: SE image of an organic sample which was stained with a heavy metal in vivo. **Bottom**: BSE image of the same sample. Now the location of organelles (bright dots), into which the organism has incorporated the heavy metal, becomes visible. BSE imaging therefore provides information from "beneath the surface". Source: Flegler *et al.* (1993)

The resolution of this imaging mode is $\sim 1\,\mu\mathrm{m}$ and depends on the accelerating voltage U_a. The low resolution (compared to SE imaging) can be understood by the larger interaction volume from which backscattered electrons escape, see Fig. 21.

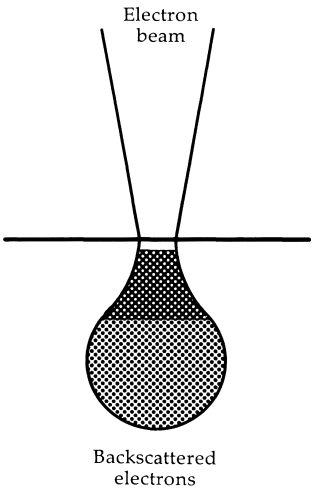

Figure 21 Interaction volume of the incident electrons (light shaded). The backscattered electrons escape only from the dark shaded volume, though. Therefore the BSE imaging mode has a lower resolution than SE images. Source: Flegler *et al.* (1993)

3. Electron Beam Induced Current (EBIC)

As already mentioned previously, the energy of the incident electrons creates electron-hole pairs in semiconductors. Measuring the electric current through the sample (by means of a picoammeter) yields information about the locally created electron-hole pairs.

This can be used to analyze semiconductor based structures, e.g. integrated circuits (see Fig. 22). There are two contrast mechanisms:

- *Depletion*: In depletion regions (p-n junctions) the created electron-hole pairs get separated by the internal electric field and induce a current which can be measured without an applied voltage (cf. photovoltaic cell). Usually a high current is encoded by bright picture elements (pixels). Consequently depletion regions appear as white lines in EBIC images.
- *Minority carrier diffusion length L*

We omit a detailed description of the second one since it is not relevant for the example shown in Fig. 22. Basically EBIC images show the location of depletion regions (that is, p-n junctions) with a resolution $\lesssim 1\,\mu$m which depends on the minority carrier diffusion length L.

4. X-ray Fluorescence (EDX)

X-ray fluorescence imaging uses the X-ray photons detected by an energy-dispersive spectrometer (EDS/EDX) to obtain spatially resolved information about the constituents concentration. It has a wide range of applications in materials science and various other fields where the local composition of samples has to be determined. The measurable quantities are

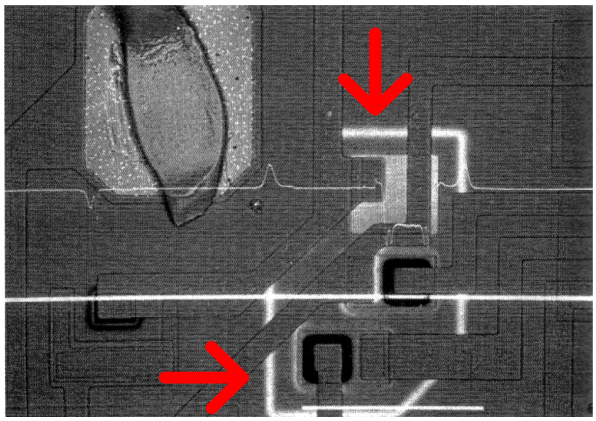

Figure 22 Typical EBIC image of an integrated circuit featuring p-n junctions (bright lines). The bold horizontal line illustrates a single scan line and the thin horizontal curve above describes the corresponding current (compare the peaks with the bright areas along the bold line!). Source: Flegler *et al.* (1993)

- the *emission rate*
- and the *photon energy*.

The emission rate conveys information about the concentration of particular elements whereas the photon energy can be used to identify elements by means of their characteristic X-ray spectrum.

If one counts only, say, photons of a characteristic transition of silicon, the intensity of these photons correlates with the concentration of Si. At the bottom line, one obtains a so called "dot map", see Fig. 23, which encodes the distribution of a particular element. The resolution of such dot maps is quite low ($\sim 5-30\,\mu$m) and depends on the material. Fig. 24 tells us why: X-rays can escape from each point of the interaction volume as they are barely screened by the surrounding material.

5. Cathodoluminescence (CL)

Cathodoluminescence imaging uses the information carried by the light photons emitted by the sample. Collected by an elliptical mirror, analyzed by a dispersive element and detected by a PMT, these photons can be used to study the composition and/or growth of semiconductors, minerals and ceramics. Brightness and color of the image is determined by

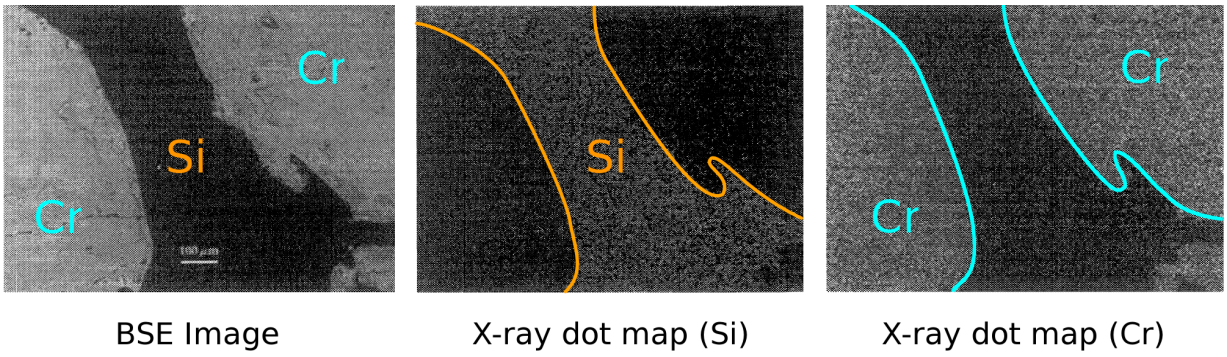

Figure 23 **Left**: BSE image of a metallurgic sample. *Z*-contrast tells us that there are two species characterized by different atomic numbers *Z*. **Middle**: X-ray dot map of the same sample for Si. **Right**: X-ray dot map of the same sample for Cr. Thus we identified the centered species as silicon bordered by chromium. Source: Flegler *et al.* (1993)

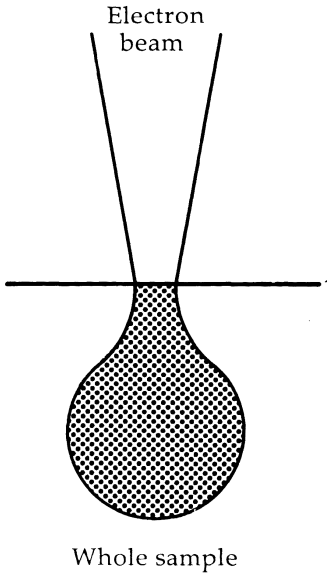

Figure 24 Interaction volume of the incident electrons. X-rays escape from the whole (dark shaded) volume. Therefore EDX imaging offers comparatively low resolutions. Source: Flegler *et al.* (1993)

- the *recombination rate*
- and the *photon energy* (wavelength).

These are material-dependent parameters. As a result, CL images can be used to localize heterogeneities and defects or perform laterally resolved band gap measurements in semiconductor based structures. An example showing a spatially varying band gap (that is, photon energy) is given in Fig. 25. CL images offer high spatial resolutions up to $\gtrsim 1\,\mathrm{nm}$.

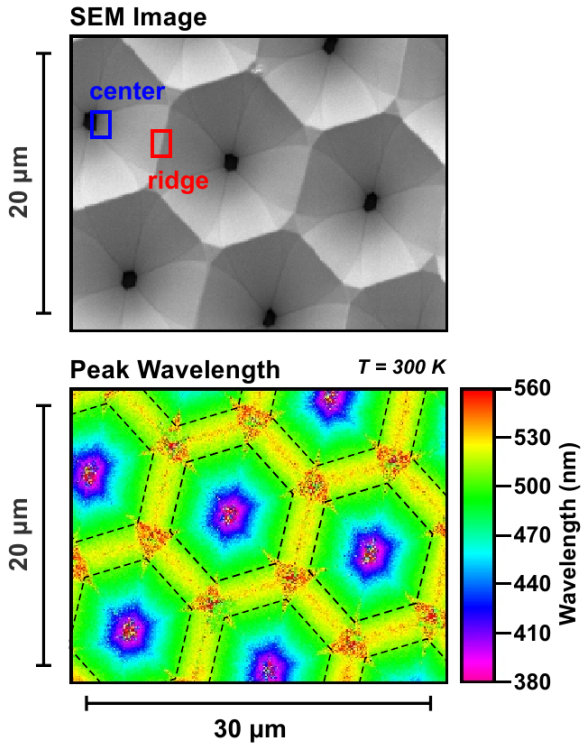

Figure 25 **Top**: SE image of an artificially created, semiconductor based nanostructure. **Bottom**: Spectrally resolved CL image of the same region. Obviously the band gap changes spatially due to local variations of the constituents concentration (which may be a consequence of the manufacturing process). Source: Metzner (200X)

IV. TRANSMISSION ELECTRON MICROSCOPY (TEM)

The following section covers some technical details of the transmission electron microscope; especially sample preparation techniques and the electron-specimen interaction. The latter give rise to various imaging modes, each revealing specific structural properties of the sam-

ple. These imaging modes are subject to the second part of this section.

A. Technical issues

1. Layout

A typical TEM is depicted in Fig. 26. In contrast to SEMs, TEMs require additional electron optics below the sample to form a real image on the fluorescent screen or the CCD camera; consequently TEMs are larger than SEMs and require more vertical space.

As SEMs, modern TEMs are able to detect several different signals to extract as much information as possible. Each signal corresponds to a distinct imaging mode and reveals characteristic properties and structures of the specimen. For instance, the setup shown in Fig. 26 (right) features (in addition to the viewing screen and the CCD camera) detectors for X-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS). Additionally, many modern TEMs are capable of scanning transmission electron microscopy (STEM) and feature even the capabilities of SEMs (e.g. there is a BSE detector in Fig. 26).

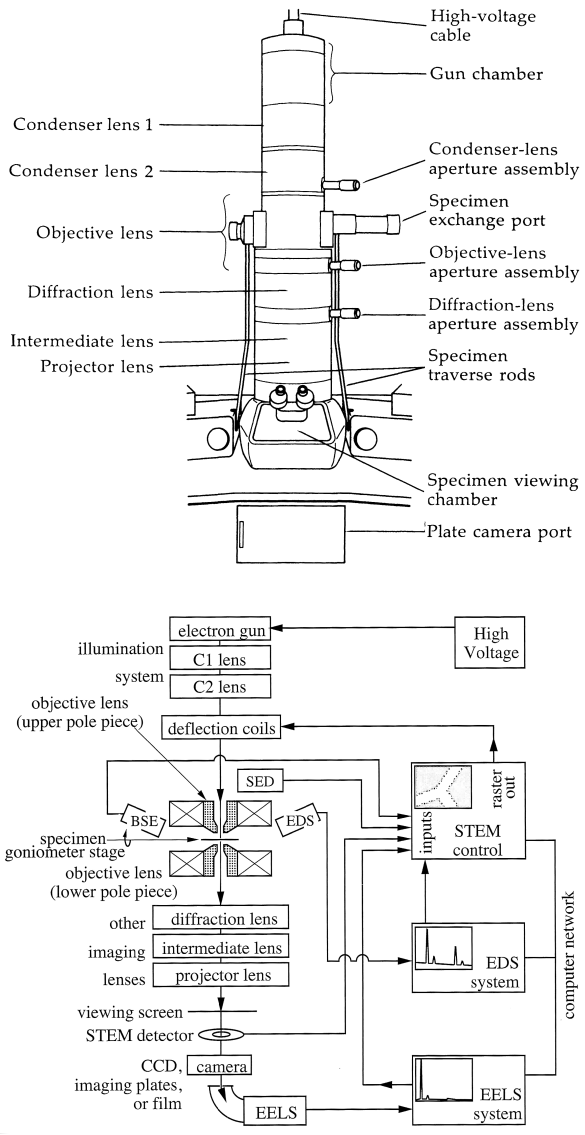

Figure 26 **Left**: Exterior view of a typical transmission electron microscope. Source: Flegler *et al.* (1993) **Right**: Schematic sketch of a typical transmission electron microscope equipped with several detectors. Source: Fultz and Howe (2002)

2. Electron-specimen interaction

As the name suggests, transmission electron microscopy uses the transmitted electrons to create real image of the thin sample or analyze these electrons otherwise. The transmitted electrons "feel" the bound shell electrons and the nuclei via the coulomb interaction. This leads to the following relevant mechanisms:

- Absorption
- Elastic scattering
- Inelastic scattering
- Diffraction

Compared to optical microscopy, absorption does not contribute significantly to the image contrast since the samples are too thin so that the variations in electron absorption are marginal. Elastic scattering occurs due to interactions with nuclei and generally causes large deflections and little energy loss since the mass ratio m_N/m_e is very large. On the contrary, inelastic scattering is caused by interactions with electrons of the specimen and is characterized by slight deflections with significant energy loss, see Fig. 27. Diffraction is a coherent process and requires us to treat electrons as waves. In the case of periodic structures, diffraction becomes manifest as the observable diffraction patterns cannot be explained by incoherent scattering alone (that is, the classical scattering of particles).

3. Sample preparation

In contrast to scanning electron microscopy, TEM requires thin ($\lesssim 100\,\mathrm{nm}$) samples since electrons have to be transmitted through the specimen. For most samples are too thick, sectioning is usually required and therefore preparation is often an intricate procedure. The actual preparation procedure depends on ...

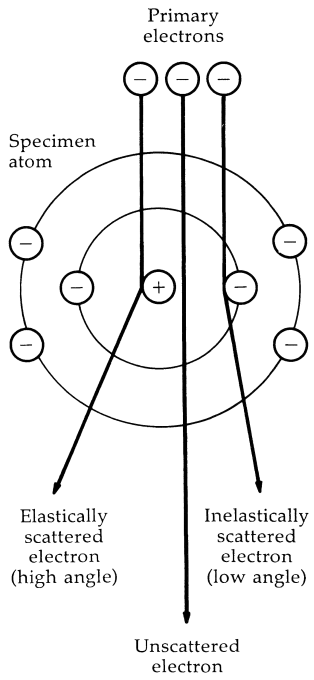

Figure 27 Interaction mechanisms between incident primary electrons and specimen atoms. Electrons are inelastically scattered at bound shell electrons; elastic scattering occurs if an incident electron scatters at a nucleus (due to the large mass ratio m_N/m_e). Source: Flegler *et al.* (1993)

- the material (organic samples, semiconductors, ...)
- and the imaging technique/contrast mechanism.

As a consequence, the field of TEM sample preparation has developed into a full-grown field of science which goes beyond the scope of this introduction. Let us just note some of the most common techniques:

- Fixation/Dehydration
- Staining (heavy metals)
- Ultrathin sectioning (by means of a microtome)
- **Mechanical milling** ("Polishing")
- **Chemical etching**
- **Ion etching**

Whereas the first three methods are primarily used for the preparation of organic samples, the last three prove most useful for crystalline and amorphous structures. That is, they are prevalent within the field of nanooptics and nanophotonics.

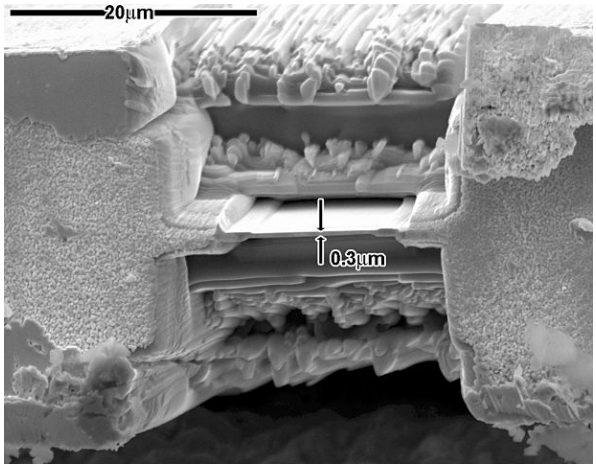

Figure 28 SE image showing a metallurgic TEM sample milled by a focused ion beam. Since 0.3 μm is not thin enough for TEM imaging, an additional method such as chemical etching has to be employed subsequently. Source: http://en.wikipedia.org/wiki/Transmission_electron_microscopy

B. Operation modes and contrast mechanisms

1. Bright-field (BF) & Dark-field (DF) imaging

The two most conventional imaging modes of TEM are called bright- and dark-field imaging. Both use the transmitted electrons and the electron optics below the sample to create a real image on the screen. To this end the intermediate lens is focused on the image plane of the objective lens, as depicted in Fig. 29. Without an objective aperture the images would be of poor contrast since only mass-thickness contrast (the absorption of electrons depends on the mass density and sample thickness) conveys any structure. Therefore an (objective) aperture is placed in the back focal plane of the objective lens. Hence only electrons scattered by a specific angle contribute to the image and the structural information encoded in the scattering angles can be used to create this image. As an aperture in the focal (or *Fourier*) plane represents a k-space truncation, the resolving power is diminished, that is, no atomic resolution is possible. In a nutshell: We traded resolution for contrast.

The **bright-field** (BF) imaging mode uses a *centered* aperture, as depicted in Fig. 29 (left). Therefore only unscattered electrons contribute to the image. One finds the following contrast mechanisms:

- *Mass-thickness contrast*: Areas with heavy atoms/thick regions appear dark since the incident electrons are absorbed.
- *Diffraction contrast*: Crystalline areas appear dark

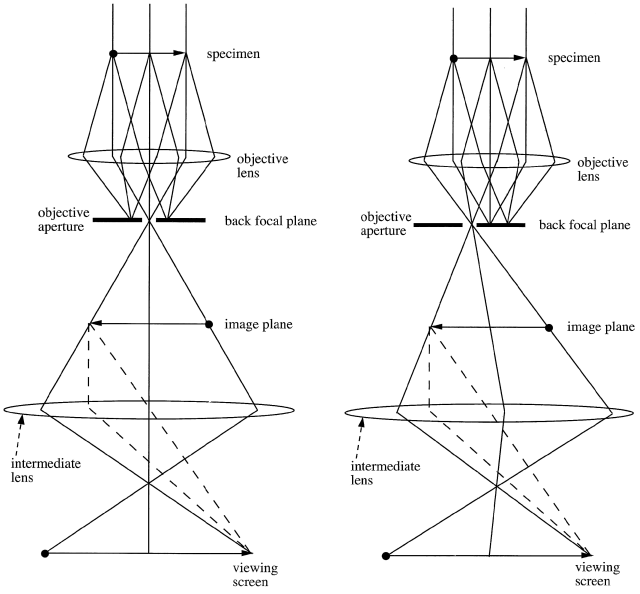

Figure 29 **Left**: Setup with centered objective aperture in the back focal plane of the objective lens used for bright field imaging (BF). **Right**: Setup with shifted objective aperture as used for dark field imaging (DF). Only scattered electrons contribute to the image. Source: Fultz and Howe (2002)

since the diffracted electrons are blocked by the aperture.

Please note that areas without any material (e.g. a hole) appear bright; therefrom the name "bright-field" imaging.

the **dark-field** (DF) imaging mode uses a *shifted* aperture, as depicted in Fig. 29 (right). Therefore only scattered/diffracted electrons contribute to the image. The key contrast mechanism is diffraction contrast which is highly sensitive for lattice defects. Recall that only electrons scattered/diffracted under a certain angle contribute. Small variations of this angle (e.g. due to lattice defects) result in dark areas on the image. Especially areas without any material (viz. holes) appear dark; therefrom the name "dark-field" imaging.

2. Selected Area Diffraction (SAD)

In combination with BF and DF imaging, selected area diffraction (SAD) belongs to the conventional imaging modes of transmission electron microscopy.

To obtain a diffraction pattern, the intermediate lens is focussed on the back focal plane of the objective lens, namely the Fourier plane, see Fig. 31. Without an intermediate aperture in the image plane of the objective lens, the obtained diffraction pattern contains information on the whole sample. Inserting an intermediate

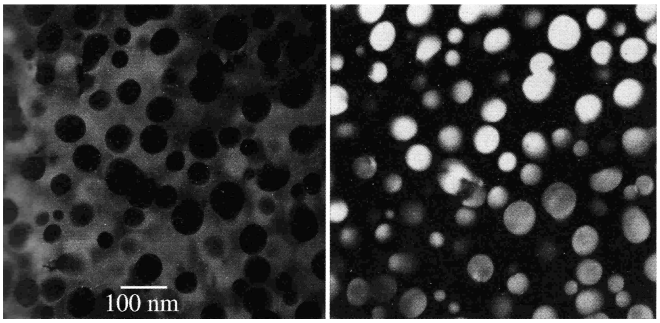

Figure 30 **Left**: BF image of an Al-Li alloy. **Right**: DF image showing the same region of the sample. Obviously the embedded grains scatter incident electrons intensively, thus they appear dark in the BF and bright in the DF image. Source: Fultz and Howe (2002)

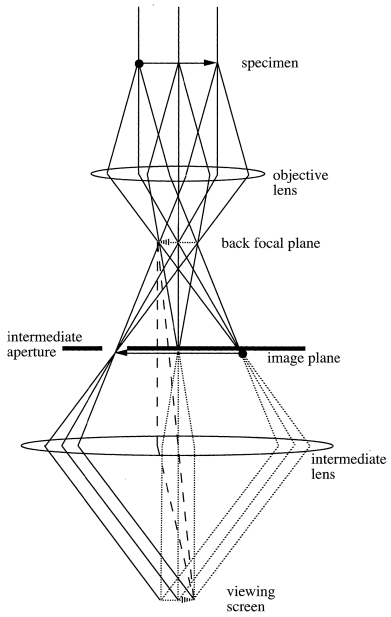

Figure 31 Setup used for selected area diffraction (SAD) imaging. An (intermediate) aperture is placed in the image plane of the objective lens and allows a localization of diffraction patterns in real space. Source: Fultz and Howe (2002)

aperture in the image plane allows a localization of the diffraction pattern in real space. That is, only electrons from the selected region contribute to the diffraction pattern.

In combination with BF and DF imaging, SAD is a powerful tool for the analysis of polycrystalline specimens and lattice defects. An example how to image the diffraction pattern of a specific grain using a combination of BF, DF and SAD imaging is shown in Fig. 32.

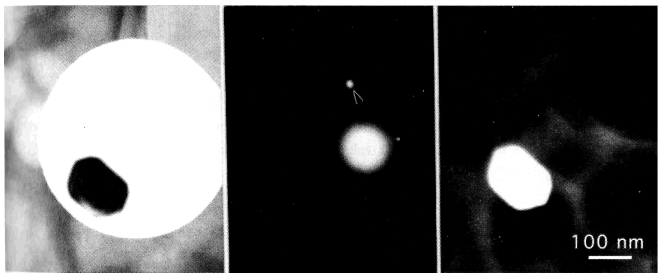

Figure 32 Interplay of BF, DF and SAD imaging modes. **Left**: BF image of an $Al_{12}Mn$-grain with SAD aperture (which is visible since it is placed in the image plane). **Middle**: Focussing the intermediate lens on the back focal plane yields a real image of the Fourier transform, that is, a diffraction pattern. Since the intermediate aperture selects the area in real space which contributes to the diffraction pattern, this is called an SAD image. **Right**: By means of an additional (objective) aperture (which is visible in the SAD image since it is placed in the Fourier plane), the diffraction spot of the $Al_{12}Mn$-grain is chosen (see arrow in the SAD image). Focussing the intermediate lens on the image plane yields a DF image of the marked diffraction spot. Source: Fultz and Howe (2002)

3. High-Resolution Imaging (HRTEM)

High-resolution imaging (HRTEM) is characterized by atomic-scale resolutions $\gtrsim 0.05\,\text{nm}$ and therefore provides the highest resolving power of all TEM imaging modes.

To this end the intermediate lens is focussed on the image plane of the objective lens. To allow the contribution of higher Fourier components, the objective aperture in the Fourier plane has to be large so that at least the first diffraction order is transmitted.

In this case *phase contrast* occurs. The interference of transmitted and diffracted electrons conveys information about the phase shift caused by the sample and the lenses. Since the phase shift depends on the electron optics, the settings and the sample, interpretation of HRTEM images is a highly non-trivial task. Usually models of the specimen and the microscope are used to simulate the imaging process and compare the results with the actual HRTEM image; this allows a verification or falsification of the assumed models.

HRTEM images are commonly used for the analysis of crystallographic structures, defects and interfaces, see Fig. 34. Another application (DALI) will be introduced in the next paragraph.

4. DALI

The acronym DALI stands for *Digital Analysis of Lattice Images* and describes a method to analyze HRTEM images of crystalline samples algorithmically.

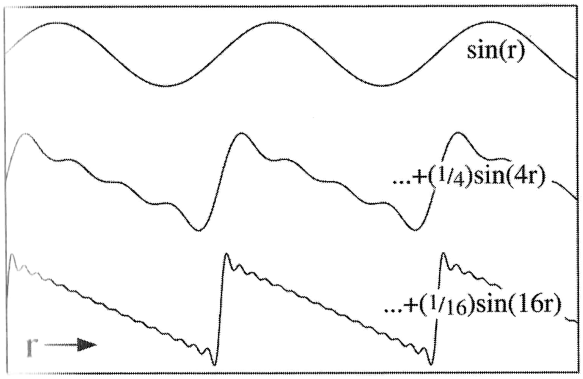

Figure 33 HRTEM imaging employs more Fourier components than conventional imaging modes. The more Fourier components are used to describe a real space structure, the higher is the resolution; here illustrated by the first three Fourier approximations of a sawtooth. Source: Fultz and Howe (2002)

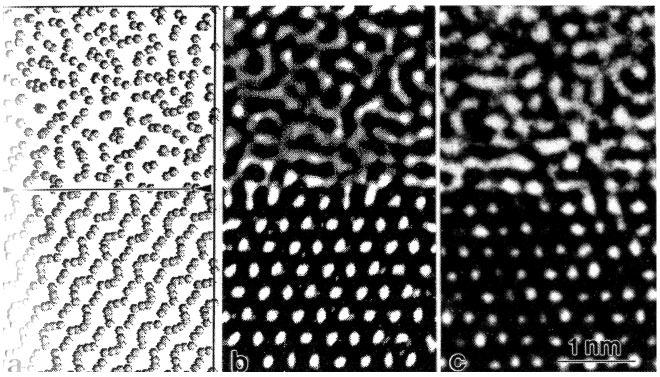

Figure 34 Example for the HRTEM imaging and interpretation procedure. Depicted is an interface of crystalline Pd_3Si and amorphous $Pd_{80}Si_{20}$. **Left**: Atomic model of the sample. **Middle**: Simulation of the imaging process based on the model of the sample and a model of the microscope. **Right**: The actual HRTEM image verifies the employed model of the specimen. Source: Fultz and Howe (2002)

To this end, consider an HRTEM image of a semiconductor based sample with locally varying concentration of another element (e.g. Si and Ge as shown in Fig. 35). The lattice constant of this structure depends on the concentration ratio of both elements continuously - this relationship is known as Vegard's law. The DALI-program detects the lattice sites visible in the HRTEM image automatically and computes the lattice sites displacement with respect to a reference lattice (e.g. given by the pure Si-substrate). The result is a vector field which describes the lattice deformation due to concentration gradients. By Vegard's law, this vector field corresponds to a concentration map, see Fig. 35.

DALI is widely used in the field of semiconductor

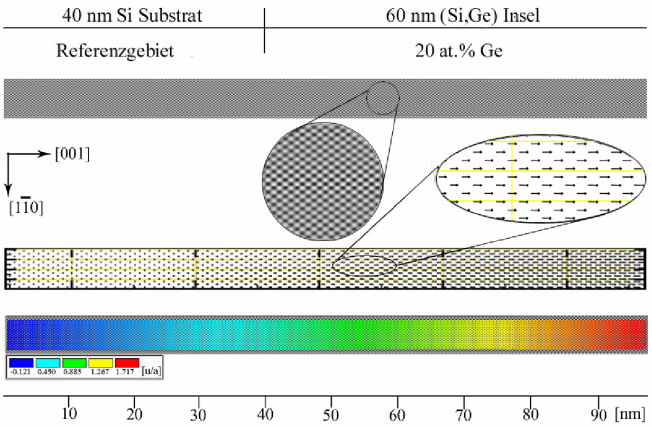

Figure 35 Based on an HRTEM image of a Si-(Si,Ge) interface (top), the DALI-program computes a vector field (middle) which shows the displacement of atoms with respect to a reference area (here the pure Si on the left). By Vegard's law, information about the local lattice constant can be converted to a concentration map of Ge (bottom). Source: `http://crysta.physik.hu-berlin.de/~ines/freiburg_2006_dgk/haeusler_vortrag_dgk_2006_freiburg.pdf`

based nanostructures, such as quantum dots and wells, to obtain information about the locally varying composition and hence on the manufacturing process.

5. High-Angle Annular Dark-Field (HAADF)

To conclude this section (and, actually, the manuscript) we introduce an imaging mode which uses *scanning* transmission electron microscopy (STEM). In this mode of operation, a focussed electron beam scans the (thin) sample. In opposition to SEM, the emitted signals (e.g. scattered electrons) are detected *below* the sample. Using this "scanning approach" resolutions up to $\sim 0.1\,\mathrm{nm}$ are possible.

The high-angle annular dark-field imaging mode (HAADF) uses electrons which are scattered through an angle of $\sim 6^\circ$ (Within the realm of electron optics, this is a high angle!). These electrons are collected by an annular detector as depicted in Fig. 36. Therefore HAADF uses incoherent elastically scattered electrons (in contrast to HRTEM which uses coherently scattered electrons). Since the scattering depends strongly on the atomic number Z, Z-contrast is responsible for structures in HAADF images which therefore constitute atomic-resolution compositional maps of the specimen. For an example see Fig. 37.

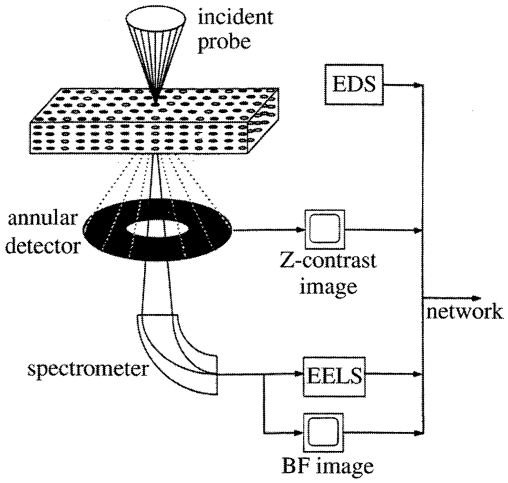

Figure 36 Schematic showing the setup used for HAADF imaging. The annular detector is placed below the sample and collects the scattered electrons ($\sim 6^\circ$). The slightly scattered (or even unscattered) electrons passing through the centre may be used for electron energy loss spectroscopy (EELS) and/or BF imaging. Source: Fultz and Howe (2002)

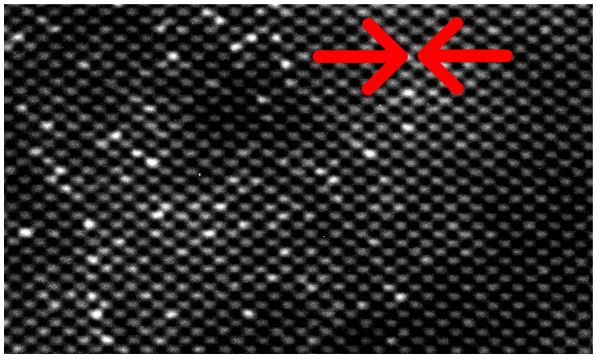

Figure 37 HAADF image of an interface between Sb-doped Si (left) and undoped Si (right). The sample slice is several atom layers thick. If a Sb-atom is located anywhere in a column of Si-atoms, its projection appears as bright dot in the HAADF image. Source: Fultz and Howe (2002)

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