Introduction to electron microscopes: electron optics, interactions and signals

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Introduction

Present paper serves as a general introduction to the topics of the 15 tutorials following it. By sketching the general layout of electron microscopes, first we shall discuss what are the similarities and differences between the individual types of electron microscopes, namely the scanning electron microscope (SEM), the transmission electron microscope (TEM) and the analytical electron microscope (AEM). We shall see what tells apart a microprobe from a scanning electron microscope. Next, we schematically follow the scatterings of a bombarding electron within the sample and classify the elastic and different inelastic scattering processes. Both the primary scattering event and the ensuing secondary physical processes produce a multitude of signal used both in different modes of imaging and in spectroscopic and diffraction analysis. The main structure of that broad topic and the relation between the parts are delineated here. The details will be given in the ensuing lectures.

What makes an "electron microscope"?

This workshop is about the examination of different materials with electrons as we do it in an electron microscope or a microprobe. Examination includes both imaging aspects and analytical aspects. Since electrons interact strongly with any form of material and we want to restrict the interaction to the sample (in contrast to its surrounding), the samples are examined in vacuum. The schematic diagrams of the general principle is sketched in Fig. 1.

The electron source emits electrons that are accelerated. That monoenergetic primary electron beam is formed and shaped by the condenser lenses and by the (upper part of the) objective lens. This lens-system facilitates changing the current in the electron beam that bombards our sample and controls the beam convergence (from parallel broad beam illumination in a TEM to finely focused convergent beam in an SEM, EPMA or AEM). The interaction of the electrons with the matter in our sample produces different signals (electrons, photons from the infrared through visible till the X-ray range, etc.) These signals can be used for imaging different sample characteristics, like surface morphology or distribution of electrically active crystal defects or local composition, etc. Beside the two-dimensional (2D) distributions (images), 1D distributions (line-scans) and 0D characteristics (point measurements) can also be obtained. The measured characteristics can be presented qualitatively and interpreted intuitively (e.g. looking at an image and recognizing a broken metallization in an integrated circuit (IC)) or can be presented quantitatively (e.g. by determining the local chemical composition). If the sample is semi-infinite (as it is in an SEM), the detectors can only be located on the same halfspace as the electron source. Images are generated electronically using the scanning principle in these machines and imaging in the (electron)optical sense is not present. In case of thin, transparent samples, signals can be detected from both sides. In

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this latter case, further lenses may be present at the other side of the sample (as in a TEM). These lenses can be used to form a true electronoptical image or may not be present (as in a special form of the AEM: the dedicated scanning transmission electron microscope, the DSTEM). First, we are going to give a brief overview of the general components of such an instrument. Then we classify the main mechanisms of electron-matter interaction in order to understand what kinds of signals are generated and how can we detect them. Finally, we summarize how these hardware-components and signals appear in the different instruments and what are they used for.

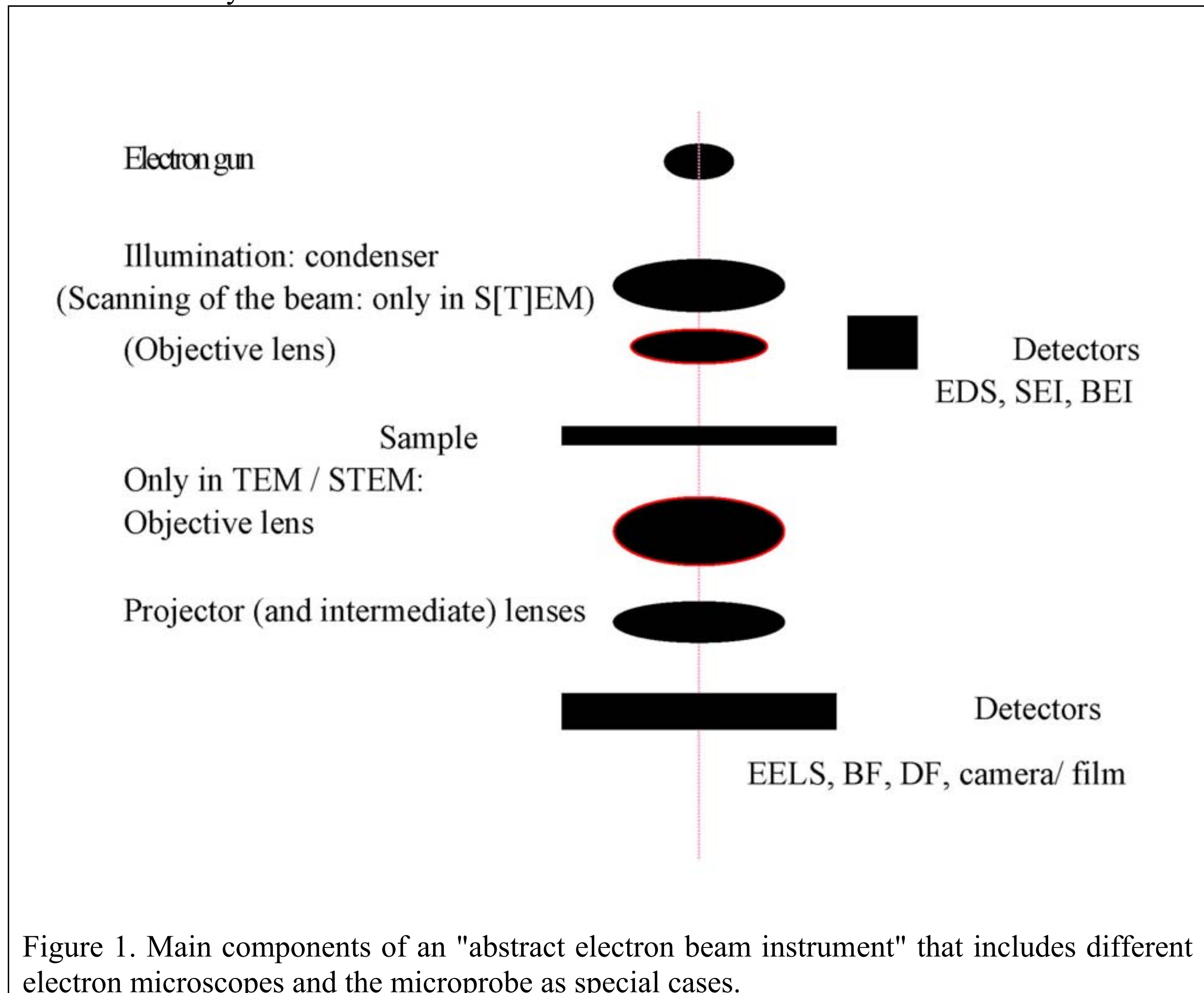
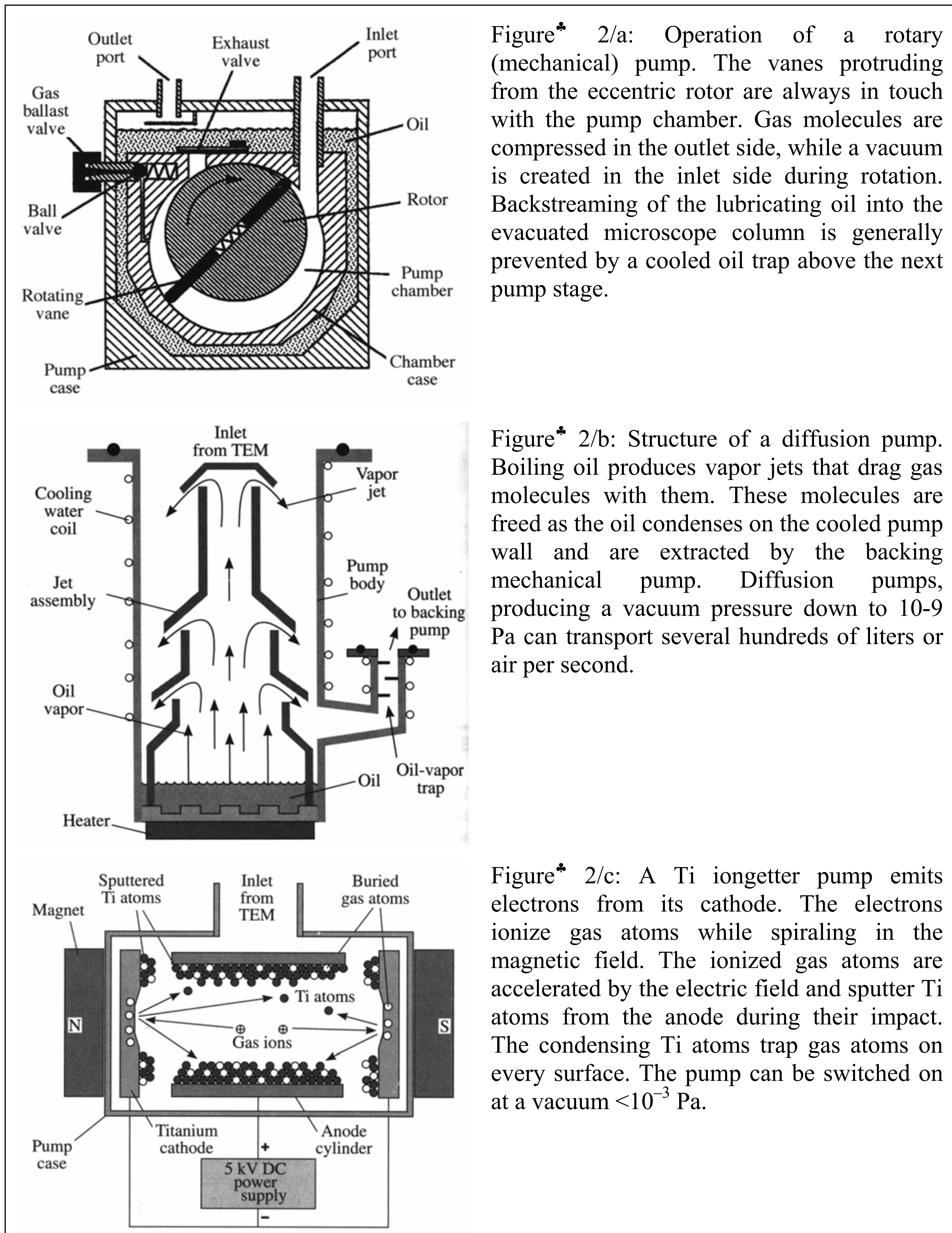


Figure 1. Main components of an "abstract electron beam instrument" that includes different electron microscopes and the microprobe as special cases.

Vacuum system

The electron column (comprising the electron gun and the lenses) and the specimen chamber (also including the specimen holder / specimen stage) form the body of the microscope. The evacuated space between the gun and the specimen is ensured by different vacuum pumps. Starting from atmospheric pressure, rough vacuum (~10^{-1} Pa) is reached with fore-vacuum pumps, namely *rotary* pumps or *membrane* pumps. These same pump-types serve for the backing of the high-vacuum (HV) pumps that further reduce the pressure into the 10^{-4}-10^{-7} Pa in the specimen chamber and into ultra high vacuum (UHV: 10^{-7}-10^{-9} Pa) in the gun chamber (in case of a field emission gun). *Diffusion* pumps or *turbomolecular* pumps can reach the normal HV (or UHV) region that can also be maintained with the help of an *iongetter* pump.



Figure[♣] 2/a: Operation of a rotary (mechanical) pump. The vanes protruding from the eccentric rotor are always in touch with the pump chamber. Gas molecules are compressed in the outlet side, while a vacuum is created in the inlet side during rotation. Backstreaming of the lubricating oil into the evacuated microscope column is generally prevented by a cooled oil trap above the next pump stage.

Figure[♣] 2/b: Structure of a diffusion pump. Boiling oil produces vapor jets that drag gas molecules with them. These molecules are freed as the oil condenses on the cooled pump wall and are extracted by the backing mechanical pump. Diffusion pumps, producing a vacuum pressure down to 10-9 Pa can transport several hundreds of liters or air per second.

Figure[♣] 2/c: A Ti iongetter pump emits electrons from its cathode. The electrons ionize gas atoms while spiraling in the magnetic field. The ionized gas atoms are accelerated by the electric field and sputter Ti atoms from the anode during their impact. The condensing Ti atoms trap gas atoms on every surface. The pump can be switched on at a vacuum $<10^{-3}$ Pa.

Pirani and *Penning* vacuum gauges (manometers) can monitor the pressure down to 1 Pa and 10^{-3} Pa, respectively, while hot cathode *ionization* vacuum meters can be used in the 1 - 10^{-7} Pa range. Pirani gauges measure the heat conduction in a thin metallic wire (Pt or W).

[♣] Reproduced from [5]

Monotonic change of heat conduction with pressure is calibrated with more expensive manometers. Spontaneous ionization of gas molecules is used in a Penning gauge. An external magnetic field is applied to spiral the charged particles between the cold cathode and the anode, enhancing further ionizations. The large number of electrons emitted from a hot cathode produces enough ionizations without a magnetic field in the ionization gauges. Current is a calibrated function of the gas pressure in both types. The current in an iongetter pump is also a monotonic function of the (low) gas pressure, so the pump is its own vacuum gauge (10^{-3} - 10^{-7} Pa).

A pressure of 10^{-1} Pa would be enough to prevent our primary electrons from colliding on the gas atoms. The better vacuum is needed to ensure as clean a sample surface, as it is possible.

Electron guns

Electron guns produce the monoenergetic (monochromatic) primary electrons used to illuminate (bombard) the sample. Their function includes both emission and acceleration of the electrons.

The electrons sources (acting as cathodes in the gun) can be classified on the basis of the physical principle behind their electron emission as either thermionic or field emission (FE) sources. The type of electron source is fixed at manufacture time and thermionic guns can not be interchanged with FE guns later.

The current density obtained from a *thermionic* source is described by the Richardson formula

$$J = A \cdot T^2 \cdot e^{-\frac{\Phi}{k \cdot T}} \qquad (1)$$

where both A and the "work function", Φ are material-constants, T is the temperature (in Kelvin) and k is Boltzmann's constant. J can be increased either by increasing T or by reducing Φ. Only a few refractory (high melting temperature) materials (like W) can withstand the required high temperature to obtain reasonable current density and increase of the temperature beyond saturation reduces cathode lifetime with minor gain in current. Materials with lower work function (like LaB_6 or CeB_6) can be operated at lower temperatures and still produce higher brightness (current density/solid angle) and live longer (See Table 1). Reactivity of these materials requires a vacuum, better by 2 orders of magnitude than a W cathode. Brightness limits microscope (and microprobe) performance when the largest signal is needed in the smallest possible spot. Brightness of thermionic sources is linearly increasing with primary beam energy (accelerating voltage). The higher voltage is the better from that point of view.

A *field emission* gun (FEG) emits electrons through the tunneling effect. The high electric field (that increases with the curvature) around a sharp tip of a W needle cathode sucks the electrons out of the cathode by tunneling them below the potential barrier (in contrast to elevating them above it). The cathode is at room temperature and must be extremely clean. That is why FEGs operate at 10^{-8}-10^{-9} Pa. Even though, regular cleaning ("flushing") of the cathode is needed. The gun contains two anodes. The first one provides the "extraction field" and the second accelerates the electrons to the required energy. Due to this construction the brightness does not depends significantly on the accelerating voltage. Although the brightness is the highest for FEGs, the maximum emitted current (needed for low magnification operation) is the lowest for them and thermionic cathodes surpass FEGs in that respect. Long term stability is also poorer with FEGs (<5%/hour with feedback regulating electronics).

Thermally assisted FEGs or *Schottky* emitters eliminate contamination of the cathode (and the need for flushing) by the elevated temperature at a price of a slightly increased energy spread. Except of a few applications, they provide a good compromise.

	Units	W	LaB_6	FE
Φ	eV	4.5	2.4	4.5
Operating temperature	K	2700	1700	300
Brightness	$A/m^2/sr$	10^9	$5*10^{10}$	10^{13}
Energy spread	eV	3	1.5	0.3
Vacuum	Pa	10^{-2}	10^{-4}	10^{-8}
Lifetime	h	100	500	>1000

Table 1. Comparison of electron sources at 100 kV.

Electromagnetic lenses

Electrons (being charged particles) can be deflected (focused) with the help of an electromagnetic field. The effect of an (electro)magnetic lens on the moving electron is twofold. On the one hand, deflection towards the optical axis is identical to the focusing effect of a convex (magnifying) lens in light optics. On the other hand, an additional rotation around the optical axis occurs in the case of the electrons. The two effects can be treated separately. Ray diagrams, based on optics can be easily drawn to understand most of the effects in the electron microscope. The additional rotation of the electron beam can be taken into account separately.

Both analytical applications and imaging based on the scanning principle need a finely focused beam with beam size and beam current as the two most important parameters. An ideal (aberration free) illumination system (condenser lenses + the (upper) objective lens) produces a demagnified real image of the electron source in the focused beam at the surface of the sample where every point in the electron source is imaged into an ideal point on the sample. As the magnification of the illumination system (do NOT confuse with the magnification of the imaging system!) is decreased, the (Gaussian) probe size is decreased proportionally. Probe current is reduced simultaneously, since the virtual solid angle subtended by the source (looked from the sample) is reduced. That is why brightness is the relevant parameter determining the achievable current density at the sample.

The action of the lens is realized through the magnetic field generated by the current flowing through a coil. That field is concentrated in a gap by a polepiece. Perfectness of the field is determined by the quality of the polepiece. The effect of a real lens deviates from the ideal one due to the same types of aberrations that we know from light optics. In contrast to light optics, the quality of the best electromagnetic lenses is poor. Deviation from rotational symmetry is called *astigmatism* and can be corrected for by exciting an additional quadrupol lens asymmetrically. *Chromatic* aberration means that the focal length depends on the energy of the electrons (wavelength, i.e. color in optics). Since the primary electrons are monochromatic (<2 eV for LaB_6 and <0.6 eV for FEG), chromatic aberration is not a limiting factor in reducing the probe size of the primary electrons (which is of importance in our analytical applications), at least at higher energies. The probe-size limiting aberration is spherical aberration, i.e. the imperfection of the lens that nominal focal length (Gaussian focus) is only valid for the rays parallel to the optical axis. The larger an electron (or light) ray

deviates from being parallel to the optical axis, the shorter the focal length is. The effect is a blurred disk at the focal plane, as shown in Fig. 3.

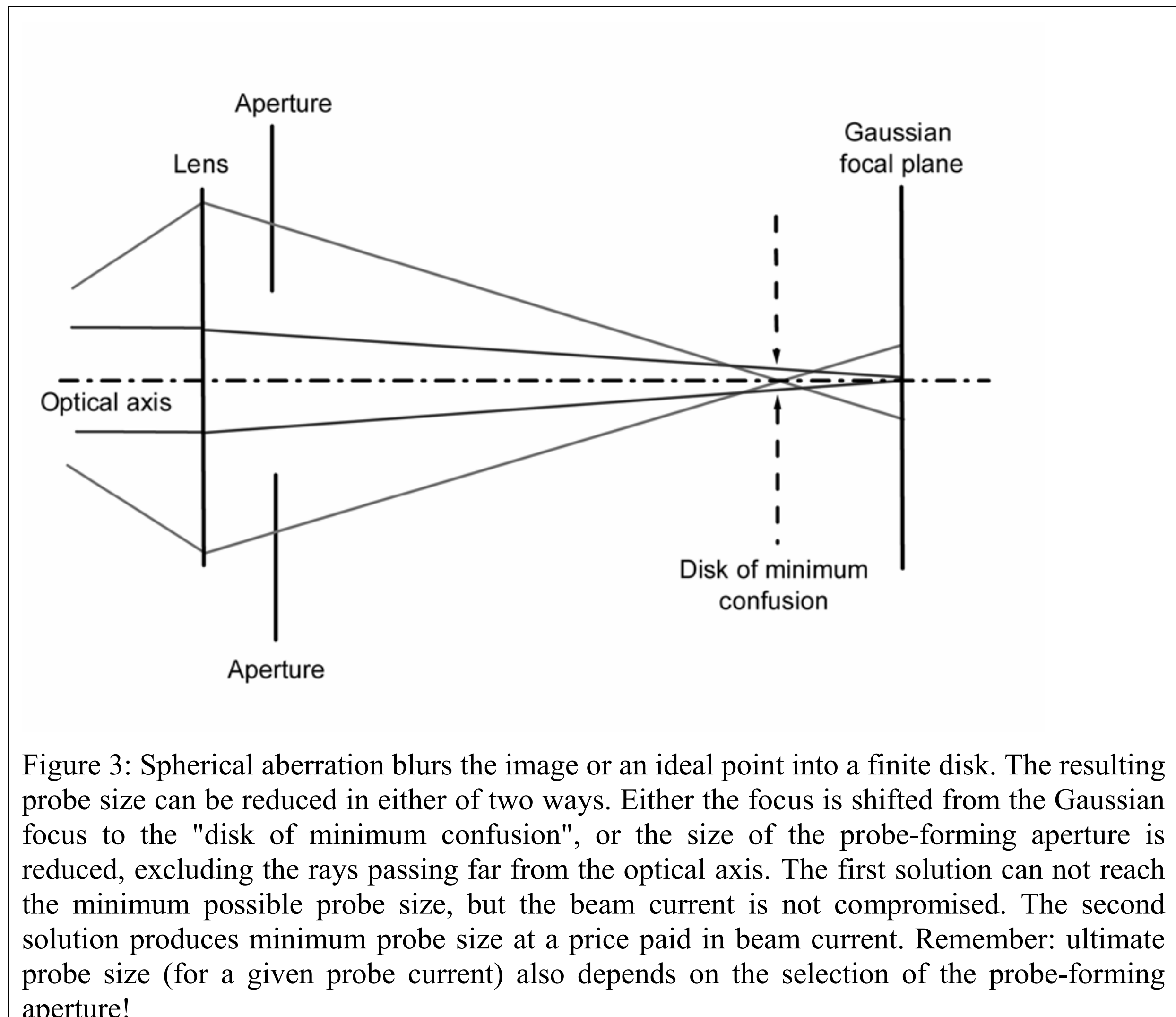


Figure 3: Spherical aberration blurs the image or an ideal point into a finite disk. The resulting probe size can be reduced in either of two ways. Either the focus is shifted from the Gaussian focus to the "disk of minimum confusion", or the size of the probe-forming aperture is reduced, excluding the rays passing far from the optical axis. The first solution can not reach the minimum possible probe size, but the beam current is not compromised. The second solution produces minimum probe size at a price paid in beam current. Remember: ultimate probe size (for a given probe current) also depends on the selection of the probe-forming aperture!

Real beam size is a superposition of the Gaussian probe size and the broadening caused by the spherical aberration. Since a given signal is needed for detection with a given precision, the best resolution of our image (or analysis) will be a function of gun brightness, lens aberration and *selection* of the operating parameters (beam forming aperture and correct alignment).

Sample stages (eucentricity)

The function of specimen stages / specimen holders is that a preselected location of the larger sample should be positioned suitably for imaging and analysis. Selection of the area requires lateral movement in two independent directions (called "x" and "y" directions). Tilting (in at least one or preferably two directions) is needed for orienting the preselected sample feature into the direction optimal for either imaging or analysis. For optimal examination, the preselected sample point should be on the optical axis at the object distance, determined by the objective lens. (The object distance is strictly determined for a TEM and can be varied for an SEM with variable working distance.) Lateral movement in the plane of the holder is trivial, so let's examine tilting.

It is desirable that tilting do not induce shift in the image (the examined object should not move out of the field of view when we want to view it from a different orientation). Separation of tilt from lateral shift is achieved by eucentric stages (holders). In a eucentric holder, any preselected point of the sample can be made coincide with the tilting axis (which crosses the optical axis in a well-aligned microscope). Points on the tilting axis remain stationary (but viewed from different angles) while tilting. Points at a distance "r" from the tilting axis move along an arc with radius "r" while tilting. Fine shift possibility perpendicular to the directions of lateral shifts (called "z"-direction) ensures that the preselected point can be placed on the tilting axis. In double-tilting holders, it is only the main tilt that is eucentric (in order to keep both manufacturing and operation simple). The secondary tilt usually introduces lateral shift.

Samples and sampled volume

Most of our microscopes and microprobes operate in a vacuum and bombard the sample with energetic electrons. Consequently, solid samples can be examined which are not altered by either the vacuum or by the electron bombardment. Electrical conductivity is also needed in order to prevent artifacts and instabilities caused by charging. (Exception is the low pressure (environmental) SEM, which can examine insulating, wet and even living objects.) Many of the charging artifacts can be suppressed by evaporating a thin conductive layer on the sample. Au (or Pd/Au) is preferred from imaging in the SEM and carbon is preferred for TEM or analysis. SEM imaging does not need further sample preparation. Quantitative microprobe analysis relies on correction calculations, which assume defined sample geometry. Consequently, a polished, smooth and flat sample surface, positioned perpendicular to the electron beam is assumed and that requires sample preparation. Maximum sample size is determined by the sample stage (holder). For a TEM, thickness is additionally restricted to ensure electron transparency. High-resolution imaging and analysis in a TEM further restricts thickness down to a few nanometers. Sample preparation (thinning) is more complicated correspondingly. Last but not least, the sample should be representative of the property or process we want to examine. The rules of correct sampling can not be overemphasized.

The volume of signal generation and the penetrating power of the selected signal together determine the volume we sample. For signals that cross the material without significant loss (like hard X-rays), the sampled volume coincide with the excited volume (varying from the μm^3 range for bulk samples EPMA down to nm^3 range for thin samples in an AEM). For strongly absorbed signals (like seconder electrons or Auger electrons) only a depth of a few atomic layers is sampled in an area determined by the probe size, irrespective of the excited depth.

The physical basis of both imaging and analysis: electron scattering

Whenever the energy balance of the collision of two particles (bodies) can be described with kinetic energies only, we speak of elastic scattering. When either other forms of energy or the appearance of new particles must be taken into account for energy conservation, the scattering is inelastic. Conservation of momentum applies to both forms of scattering.

Elastic scattering

It is known from classical physics that the partitioning of the kinetic energy between two colliding bodies (with masses m1 and m2) only depends on the ratio of their masses: m1/m2. Since the mass of the electron is negligible as compared to the mass of any finite sample (or

even with a single atomic nucleus), the kinetic energy taken by the sample can be neglected. Energy conservation during an elastic scattering means conservation of the kinetic energy for the primary electron (i.e. the conservation of its velocity). Elastic scattering means a change in the direction of the electron's speed and it is mainly caused by the collision with atomic nuclei. General theory of microanalysis is based on the assumption that the sample is homogeneous and isotropic, so crystalline effects are not taken into account. For such materials (like an amorphous sample), calculation of elastic scattering can be restricted to the calculation of scattering angle. Elastic scattering is characterized with relatively large average scattering angles (in contrast to inelastic scattering), resulting that a considerable fraction of the primary electrons may "turn back" and leave the bulk sample, producing the useful backscattered electron signal. The isotropic approximation is used in Monte Carlo (MC) calculations which calculate the measured signal intensity by following the individual scattering events of the primary electron (and of the secondaries) and summing up the probabilities of signal generation during all of these scattering events. Effects due to crystallinity are out of the scope of such calculations and of all the correction procedures used to convert measured X-ray signals into chemical compositions. Consequently, the presence of crystalline material is a source of systematic error for X-ray microanalysis. This error is rare and small for EPMA of bulk materials, but it can be significant during EDS analysis of thin samples.

Crystalline effects are also needed to explain all diffraction patterns, including electron backscattering (EBSD) patterns in an SEM and to simulate the contrast in a (HR)TEM. Kinematic (single scattering) theory corresponds to the Bragg-equation of diffraction. The Kikuchi lines in a selected area electron diffraction (SAED) pattern in a TEM or the (pseudo)Kikuchi-lines in an EBSD pattern manifest the traces of the atomic planes in Bragg-condition (for the given orientation of the given structure). Interpretation of diffraction patterns is based on the calculation of electron states in a periodic crystal that are described by Bloch-waves, which are the solutions of a Schrödinger-like equation. Dynamic (multiple elastic scattering) effects are also included in the Bloch description. Deviation from the kinematic theory is mainly restricted to the crossing-points of such lines. Elastic scattering permutates the electron between Bloch-states corresponding to the same total energy. Interference between such coherent Bloch-waves produces the atomic resolution phase contrast image in the conventional HRTEM. Correct interpretation of such images can only be done on the basis of extended image simulations.

Inelastic and quasielastic scattering

"Loss" of kinetic energy (of the primary electron) is mainly caused by the interaction with the electrons of the sample. The simplest inelastic scattering process is single electron excitation or ionization. In that case a bound electron of the sample is kicked off its bound state and receives kinetic energy. It is called a secondary electron (in contrast to the primary electron). The most probable value of the received kinetic energy is a few eV and it is below 50 eV for most of the cases. Strictly speaking of quantum mechanics, the primary and secondary electrons can not be distinguished. However, we shall call primary electron the one with the large part of the kinetic energy and secondary electron the one with the small kinetic energy. If the secondary is generated close to the sample surface, it can leave the sample with high probability and can be collected as a signal electron. If it is generated deep in the volume, it is absorbed with high probability and can not escape. The kinetic energy lost by the primary is a sum of the kinetic energy and the original binding energy of the secondary. The binding energies are quantized and characteristic of the atoms (for inner shells), so the minimum energy-loss for each such event-type represents a characteristic loss (onset of an edge-type

loss) and serves as a basis for electron energy loss spectroscopy (EELS) in the TEM. Since the binding energies of inner shells are large, these infrequent inelastic collisions cause large individual losses (up to several tens of keV). One of the two competing processes that brings the atom back to its ground state after inner shell ionization is X-ray emission used in EPMA and AEM. The frequent event of single electron excitation in an outer shell is characterized by a binding energy in the 1 eV - multiples of 10 eV range and by the production of secondary electrons. The production of electron-hole pairs in a semiconductor also belongs to single electron excitations.

The second type of inelastic scattering is the interaction with electrons of the conduction (valence) band. These electrons respond with a collective oscillation (correlated motion) to the excitation by the primary electron. The quantized pseudo-particle describing this collective oscillation is the so-called plasmon. Its energy (lost by the primary) is in the 10-30 eV range. It is a frequent scattering event resulting in a strong peak in the EELS spectrum. The same process is responsible for the multiple scattering that smears out the characteristic EELS edges when the sample thickness increases.

The third type of inelastic scattering is frequently called quasielastic, because (although strictly speaking it is inelastic) the loss in the kinetic energy of the primary is so low that we can not separate it from elastic scattering with the usual electron energy loss spectrometers. These losses of the order of 10^{-2} eV are caused by excitation of lattice vibrations, the so-called phonons (dissipating as heat). Since the nuclei are well localized, the scattering in reciprocal space extends to large angles, resulting in diffuse scattering between the Bragg peaks. The high angle annular dark field (HAADF) electron detector unique of the best AEM, the so-called dedicated scanning transmission electron microscope (DSTEM) uses this thermal diffuse scattered signal to produce atomic resolution incoherent images that can be interpreted intuitively. Thermal diffuse scattering is the most probable event at room temperature.

The resulting effect of the multitude of inelastic scattering events is that the primary electron is slowed down in a bulk sample. Most of the inelastic scattering events cause a small angle scattering (as compared to elastic scattering). That is why MC calculations frequently approximate the net effect of many inelastic scatterings with the so-called continuous slowing down approximation which is nothing else than calculating the most probable energy loss between two consecutive elastic scatterings from an analytical formula (in contrast to calculating many inelastic events individually).

Sample thickness, single, multiple and plural scattering; Monte Carlo simulations

The larger the sample thickness, the larger the probability that the primary electron is scattered more than once within the sample. It is always the case for bulk samples in the SEM. If a few scattering happens we call it multiple scattering. Reduction of the elastic peak and appearance of peaks at multiples of plasmon energy in the EELS spectrum are characteristic of multiple scattering for thin samples. EELS edges are rounded, elemental analysis is difficult in this range. The name of a large number of consecutive scatterings of a primary electron is plural scattering. EELS analysis is impossible in this thickness range.

Sophisticated MC calculations follow all the elastic scattering events and the inner shell ionizations (with large individual energy losses) individually. They take the effect of plural inelastic scattering events into account by the continuous slowing down approximation. The Monte Carlo name originates from probability. (Reasoning that generating random numbers is the same as gambling in a Casino in Monte Carlo.) The most probable values of scattering angle, traveled distance and lost energy are determined by random numbers. The probabilities

are scaled to give back physically meaningful values for the sum of a large number of scattering events. The main advantage is that arbitrary geometrical boundary conditions can be taken into account during the calculations.

Generated signals and signal detectors

As we mentioned in connection with inelastic scattering, a large number of *secondary electrons* (SE) with low energy are generated in the sample and the SEs generated close to the surface escape and can be detected. Due to their low energy, SEs can be efficiently collected by an attractive electric field (of 10 keV) into the scintillator of an Everhardt-Thornley detector. The attractive field collects the SEs from practically every direction, ensuring that the collection solid angle approximates to a hemisphere. The scintillator converts the electron signal into light, which is detected by a photomultiplier. The analog output of the multiplier can modulate the intensity of a TV display (old terminology is cathode ray tube, CRT) when the image is formed on the basis of the scanning principle. SE images are sensitive to surface morphology. Edges and sharp tips show up bright, since an inner point is close to the surface in multiple directions in these regions, resulting in enhanced SE emission. Emission of these low energy SEs is sensitive to even small changes of local surface potential. The resulting *potential contrast* (voltage contrast) can be used to map the local electric potential (e.g. over the surface of an operating integrated circuit).

As we saw, many primary electron turn back from a bulk sample due to the large angle of elastic scattering and due to the large number of scattering events. (It does not make too much difference if some of them were inelastic. It only reducing the energy if the backscattered electron, resulting in a broader spectrum of BE energies.) These energetic *backscattered electrons* (BE) carry twofold information. On the one hand, elastic scattering (and hence the fraction of electrons that backscatter) depends on the average atomic number in a monotonic manner, serving as a basis for atomic number contrast. On the other hand, backscattering coefficient also depends on the elevation of the emitting surface, resulting in a topographic contrast component. The two signal components can be separated with a pair of symmetrically positioned BE detectors. In most of the cases a large area, reverse-biased p-n junction serves as a BE detector. The energetic BEs generate a wealth of electron-hole pairs in the detector collected at the opposite electrodes by the bias field. The current of the detector is proportional to the number of BEs. The detector is more efficient at higher accelerating voltages (more energetic BEs).

Directional dependence of the BEs from a single crystal (due to Bragg reflection) can be mapped with a 2D charged couple device (CCD) detector. The small modulation superposed over a large isotropic background is separated in *EBSD*. Computer processing identifies the Kikuchi-lines in the weak contrast and compares to patterns simulated for the given (known) crystal structure. Orientation of individual grains is determined and processing of thousands of grains yields an orientation map. Pole figures for textures and misorientation maps can also be deduced.

If the sample is connected to Earth-potential, the *absorbed current* through its connection gives a mixed contrast of both atomic number and topographic contrast. Although no special detector is needed, separation of the contrast components is impossible, limiting its usage.

Transmitted electrons (in case of a thin sample in the TEM) carry mass thickness (mainly for amorphous samples) and diffraction contrast information (for crystalline samples). The bright field (*BF*) and the dark field (*DF*) detectors in a STEM use these contrast mechanisms.

Internal electric fields in the sample can be imaged in a (semiconducting) sample if an external short-circuit (with an included current meter) is attached to it. When the primary beam excites a small volume far from the internal field region, the generated electron-hole pairs are recombined within their diffusion range and no signal is generated in the external short circuit. However, the electron-hole pairs are separated by the internal field when they are generated at the field region (within the diffusion range of the charge carriers) and a compensation current flows in the external circuit. This signal is called electron beam induced current (EBIC). Typically p-n junctions, Schottky-barriers and electrically active dislocations in semiconductors and grain boundaries in several ceramics are examined by EBIC.

Cathodoluminescence (CL) gives information complementary to EBIC: the distribution of recombination centers for the charge carriers. Recombination of the electron-hole pairs emits a photon in the infrared (IR), or in the visible range. The signal of a photon detector shows the regions of recombination centers bright in the CL image while the beam is scanned.

Electron energy analyzers are applied for two different purposes. On the one hand, the *EELS* detector in a TEM measures the energy distribution of electrons having passed the thin sample. Beside additional information, the characteristic edges of inner shell ionizations provide the chemical composition of the sample. On the other hand, the *Auger electron* spectrometer (AES) measures the kinetic energy of electrons leaving the surface of a bulk sample. The Auger-process is the alternative of X-ray emission after inner shell ionization and identifies the atoms by the characteristic energies of the emitted electron, similarly to the characteristic X-rays (see Fig. 4).

The main signal for this community is *X-rays*, generated in the secondary process (after the primary process, ionization). The inner hole is filled from an outer shell and the difference in the energies of the two shells is carried away by an X-ray photon (see Fig. 4). The energies of a given X-ray line are a monotonic function of the atomic number, as described by Moseley's law. This monotonic dependence facilitates qualitative analysis. By measuring the energies of the characteristic X-ray lines (from Moseley's law) we obtain a list of the elements present. The two usual types of X-ray detectors are treated in two different lectures. Quantitative analysis requires the measurement of the number of photons for one selected X-ray line per every element present in the sample. (In X-ray analysis we superficially call the number of photons "intensity", although strictly speaking, intensity in physics has a different dimension.) The correction procedure, needed to convert the measured X-ray intensities into chemical compositions is elaborated in a different talk.

The effect of crystal structure; ALCHEMI

The systematic error in the chemical composition (measured from X-ray intensities) caused by dynamic electron diffraction in a single crystal is turned into a source of additional information by the method atomic locations by channeling enhanced microanalysis (ALCHEMI). Crystallographic location of minor components in a thin crystalline sample can also be determined via this method (beside composition) in a TEM.

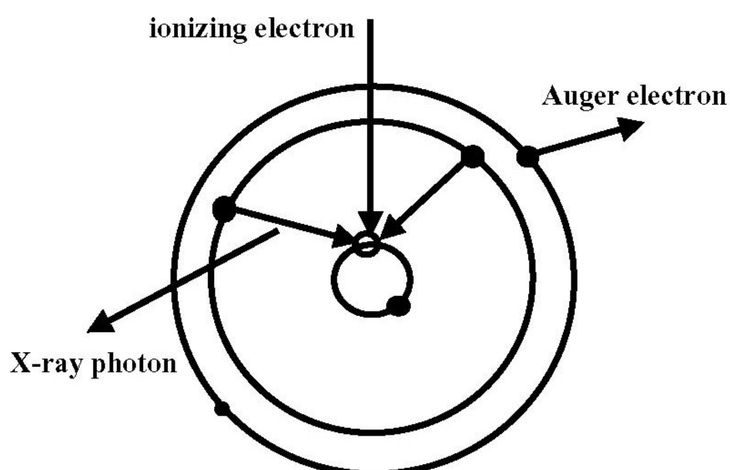


Figure 4. There are two alternative ways of deexitation from the highly excited state of an inner shell being ionized. These are the emission of either an X-ray photon or an Auger electron. *X-ray emission* is a single-electron process that shifts the inner "hole" (i.e. the electron state that is empty due to ionization) to an outer shell. The difference between the energies of the two shells is transferred to the X-ray photon.

Since the energies of the shells are well defined in an atom (being quantized), the energy of the photon is characteristic to the emitting atom. Several outer shells are available for a given inner shell as possible routes of deexitation by photon emission. The appearance of a series of X-ray lines produced in response to the ionization of a single inner shell is the manifestation of the existence of the alternative routes. If the ionized shell is the innermost (most bound) K-shell with principal quantum number equal to 1, the series of lines is called the K-series. If the shell with principal quantum number 2 (L-shell) is ionized, the series is the L-series, etc. The shells with principal quantum number >1 (L, M, etc.) are separated into *subshells* with slightly different energies (as compared to the energy difference between the main shells). The alternative *Auger-electron emission* is a two-electron process. While the inner hole is shifted to one of the outer shells, the energy difference is given to another outer-shell electron (in contrast to a photon). This energy exceeds the binding energy of this other electron and it is emitted from the atom (leaving it in a doubly ionized state). Since the kinetic energy of the emitted electron is a difference between 3 binding energies, it is also characteristic of the atom, making Auger spectroscopy an alternative analytical technique. The probability of X-ray emission is called the *fluorescence yield* (ω), while the probability of Auger emission is $1-\omega$, the two processes being the only alternatives for deexitation. Since ω gives the total probability of photon emission (irrespective of which line of the series it belongs to), we need a further atomic constant, the *weight of line* to obtain the probability of the emission of a selected X-ray line. A special type of Auger transition is when the two outer subshells involved in the transition belong to the same shell. In that case we speak of a *Coster-Kronig transition*, characterized with Coster-Kronig transition *rates*, f_{ij} for transitions between the ith and j^{th} subshell of the same shall. The importance of Koster-Cronig transitions in X-ray analysis is that they redistribute the (primary) holes between the different inner subshells prior to the X-ray emission. Consequently they change the intensities of the individual X-ray lines from the values pertinent to the primary ionization by our bombarding electrons.

Scanning electron microscopes (SEM)

All SEMs use the scanning principle for imaging (hence the name). The focused electron beam is scanned over the surface of the bulk sample synchronized with the scanning of another electron beam in a TV tube. Any selected signal from the sample (varying as a

function of beam position) can modulate the intensity of the TV tube. Magnification is determined by the ratio of the lengths of the two respective scanned lines.

Typical accelerating voltage varies from 30 keV down to 200 eV. Resolution is the best at the higher voltages (~1-2 nm for FEG at 30 keV).

The main advantages of an SEM is good spatial resolution (intermediate between optical microscopy and TEM), large depth of focus (giving a clear image simultaneously from hills and valleys of a rough surface) and moderate (or no) sample preparation.

If an X-ray detector is attached (either EDS or WDS), the analytical capacity becomes close to a microprobe.

Working distance is variable in many SEMs; low for high-resolution imaging and large for EDS/WDS.

Conventional SEMs operate in HV. Low vacuum (low pressure / environmental) SEMs do not evacuate the vacuum chamber to that level. Even critical water pressure can be maintained to examine a water droplet or a living creature. Insulating samples can also be examined, because the gas atoms carry away the excess charge and prevent the sample from charging.

Transmission electron microscopes (TEM)

TEMs are operated at 100-400 keV to image thin samples. Elastic scattering is used in the formation of true electronoptical images by electrons transmitted through the sample. Diffraction contrast dominates low and medium magnification images. High-resolution images are based on phase contrast. Assessment of crystal defects is a main application of conventional TEM. The sample is illuminated by a broad (nearly) parallel beam and the image is formed by the imaging lenses at the other side of the sample. Magnification has nothing to do with the size of the illuminated area, it is determined by the imaging lenses. The image is formed on an electroluminescent screen (or film or detector) in a fixed position in both modes of operation. In imaging mode, the image plane of the objective lens is projected to the screen. In diffraction mode, the focal plane of the same objective lens (containing a diffraction pattern) is projected to the same screen.

Focused (convergent), fine electron probe is used for analysis in an AEM. The possibility to form an intense fine beam and the presence of an EDS or an EELS detector makes an AEM from a TEM.

The microprobe (EPMA)

A microprobe is similar to an SEM that is equipped with (an EDS and) several WDS detectors for X-ray analysis. Quantitative point analysis is the main feature with possibilities of elemental mapping and line scan. The point to analyze is typically selected from the compositional BE image of the polished sample. The three main characteristics that tell apart a microprobe from an SEM is stability, maximum beam current and the number of spectrometers. SEMs are optimized for SE resolution, while microprobes are optimized for long-term stability and higher currents.

Quantification of the results is based on the fact that the measured X-ray intensities (generally) do not depend on the chemical bonding of the given element. In that sense EPMA / EDS / WDS gives "elemental" concentrations (in contrast to chemical composition in the chemical sense that includes the chemical form the element is in). In the exceptional cases (of mainly light elements), a chemical shift and some distortion of the measured peak can be detected, as will be elaborated in a separate lecture.

Conclusions

The introduction tried to delineate the structure of the "forest". The individual "trees" will be examined in detail during the workshop. Although most of the topics were touched superficially, I hope this skimming helps the audience to place the "trees" of the coming lectures into their correct positions and to see the "forest" in its full beauty.

Further reading

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