Introduction

Welcome to the lecture training module on scanning electron microscopy. You should read these notes in conjunction with the slides of the presentation. The text has been written in a conversational style to accompany what you are seeing as a picture / graph / table. The only thing missing is the ability to get questions answered while you view the presentation. To get points clarified, send me an e-mail at qjayaram@materials.iisc.ernet.in or call (x3243) and come by my office.

I suggest that you go through the presentation once, to get a flavour of what topics are covered. Then go back and work your way through them more slowly and make sure you understand the points thoroughly. Refer to the books mentioned for more details. And finally, do the problems mentioned at the end.

After you are ready, you need to get an authorization from your research supervisor before taking the written test at the microscope centre after which you can register for the hands-on training. For logistical reasons, these will be held at fixed times and not continuously. Remember that you must remember the relevant theory when you are operating the controls to maximize the quality and speed of your work.

Good luck.

The numbers here correspond to those of the transparencies.

Image formation (Slides 1-104)

(4) Imaging (Conventional)
We are accustomed to optical imaging using a lens, both in our eye and in a camera (which form real images on a sensor, whether it is the retina or a CCD array or photographic film) as well as in a microscope which typically forms a virtual image. The principle is the same: points on the object scatter light which is "focussed" by a lens into another point. If we think of the object as consisting of a periodic array of points, then traditionally ***resolution is determined by diffraction.*** Information from ***small spacings*** **scatters** at a ***large angle ~ sin^{-1}(λ/d)***, where λ ***is the*** wavelength and ***d*** is the spacing. To accurately reproduce the object you therefore need a ***large*** lens at ***a small distance***. This is why the objective lens at high magnification comes in so close to the object in an optical microscope. Obviously, the minimum resolvable distance, i.e., the smallest spacing from which information can be transferred by the lens cannot be smaller than the wavelength. In actual fact it is ~ 0.3 λ**.** This is the reason, e.g., why blue light from a mercury vapour lamp is used for the best resolution. Similar considerations apply to forming a small focused probe from a parallel beam at the focal plane of the lens. Diffraction limits the probe size to ~ λ**.** This is why people break their heads to go to UV and Xrays in lithographic methods to define patterns for semi-conductor wafer processing. If you can use GaN blue lasers to read in an optical storage device, then you gain (in areal density) a factor of 4-5 in recording density with respect to using GaAs red emission. NOTE: A modern near field scanning optical microscope by-passes the diffraction limited resolution by sensing the scattered signal ***before*** it starts to spread due to diffraction. This requires extremely flat surfaces and the ability to collect the signal at distances ~ 10-100 nm. Then, you defeat the Fraunhoefer limit.

What is our eye's resolution (when you have 20-20 vision)? Typically, one can resolve 100 microns (0.1 mm) at 15 cm. Anything smaller will require magnification. Comfortable viewing requires that lines be separated by about 0.5 mm. These numbers are important to remember when creating images to look at.

We are not always obsessed by resolution in normal viewing. Other aspects of the signal are important. We see colour: a practiced artist / clothes designer may be able to distinguish hundreds or thousands of shades in the narrow spectral range of 400-800 nm that is visible to us. We see amplitude (i.e., bright and dark). And we see depth. Three-d vision comes from the fact the each eye sees slightly displaced images of an object due to the different position of each eye. The ***brain*** fuses these images and ***infers*** depth. Such an effect is ***learned***. For example, experiments done on adult volunteers with spectacles that turned images upside down revealed that, for a couple of days, the subjects were greatly confused and kept tripping over their own feet. After those 2 days, they began seeing everything the right side up. The brain had learned by combining touch and sight, which was **up** and which was **down.** (Of course, when they took the glasses off and returned to civilian life, they went through the same trauma!). Similarly, it was apparently shown that

very small babies do in fact see things inverted (remember that the image on the retina is a real, inverted image) and “learn” by experience, which is up and which is down.

Two eyes are not essential to see depth. We also infer what is in front and what is behind by seeing what is blocked from view by the object in front. That is how Mansur Ali Khan (Saif’s father) played cricket with one glass eye. Stereo views can be created by many methods. Newspapers sometimes carry, in their fun pages, pictures of dots that look random and meaningless. However, when you defocus, i.e., focus behind the page, a 3-d picture emerges. This is created by separating two groups of dots: one of which is seen by the left eye and the other by the right eye. The brain does the rest. In focus, of course, the 2 images seen by the eyes are identical.

A related quantity is depth of field. If you focus on a distant elephant, is your friend in front at a safe distance also in focus? The depth of focus (how much in front and back of the plane on which we are focused, are additional objects ***also*** in focus? is $\sim \lambda$ / ***sin*** θ. You can see that at the higher resolutions, θ will be high and the depth of field low. That means, at the highest magnifications, you need a surface that is extremely flat and polished (these are not the same thing!). Incidentally, to get a high depth of field in a camera, you deliberately stop it down (small aperture) and increase the exposure time to compensate. If, on the other hand, you want to highlight your friend’s face and put the background in an ill-defined blur, you use a large aperture. You are playing with θ when you do all this.

Detection of an image can also be limited by contrast. (Like colour vision is limited by brightness. You don’t see colours at night.). Camouflage relies on the fact that the contrast of the object and background are ***similar***. In the same way, when the sun goes down, the signal becomes weak and ***noise*** can make objects indistinguishable from the background. Leaves merge into one mass, though you can still make out a tree.

From the standpoint of microscopy, what are the limitations of a conventional optical system?

(1) 200 nm is the best you can resolve. That’s just not good enough for many modern materials.
(2) The sample needs to be extremely flat and smooth (depth of field is low).
(3) You don’t really get chemical information (unless your sample is fluorescent and you attach a spectrometer). Except indirectly as, e.g., when you etch the surface and say that if it looks dark, then it is cementite because you know that the etchant only attacks cementite.
(4) The specimen must usually be etched in order to reveal chemistry through topography / colour.

(5) Scanning probe microscopy
The SEM is probably the earliest commercial scanning microscope (for a history, google SEM and McMullan). It operates on a completely different principle to that of a conventional optical microscope. A probe **scans** a sample at a set of discrete points (in a scanning tunnel microscope, the probe will be a tungsten tip, in an AFM – a Si tip on a cantilever, in a near field scanning optical microscope – a laser beam, etc.). The probe ***interacts*** with the sample and produces a mess of electrons, x-rays, light, what not. (In other probe microscopes, the signal may be a tunneling current, a force, an optical signal, etc.). You need a ***detector*** to pick up the signal you want. The detector signal (it can be the magnitude or some fancy derivatives thereof) ***modulates*** a TV screen intensity that is being rastered at the same rate as the sample. What you see as bright and dark is then in one to one correspondence with where in the object the signal is high / low (as shown for points 1-15 in the slide)

How is magnification achieved? As shown in the slide, $M = d_2/d_1$ and the convenient way to increase M is to go on ***reducing*** the size of the scanned region. ***Note: magnification here is completely uncoupled from resolution.*** Even at this stage, you can sense that if you want to resolve small features you must use a small probe. In an optical microscope, the higher magnification objectives are also automatically better ground, aberration corrected and positioned closer to the object. (Remember that there is no point in going to a higher magnification if you cannot see smaller features; you may as well stay at the lower mag. and see a wider field of view!) In an SEM, ***you*** have to consciously improve resolution when you go to higher magnification. This involves a multiplicity of possible changes and not making these changes is the commonest reason why starting students take poor resolution, high magnification pictures.

---

(6) Ease of operation and viewing
This slides shows why the SEM is a popular machine. Zooming up from the top left to the bottom right involves 3 clicks. The sample (a broken iron surface) is completely unprepared and extremely rough but completely in focus everywhere. The central feature (which may be responsible for local rupture) stands out clearly and can be chemically analysed in 30 seconds for its constituent elements. The SEM can be operated at a rudimentary level with minimal effort to produce high quality (but not the ***highest*** quality!!) images.

---

(7) The lay out shows a typical possible array of detectors, the sample and scanning coils. The microscopes you will be using have the standard secondary electron (E-T), backscattered and EDS (x-ray) detectors. In addition, we have an electron backscattering diffraction CCD array and some special detectors inside the column for high resolution work (in the Sirion). Cathodoluminescence detectors will detect light from appropriate materials such as phosphors, semiconductors, etc., current detectors can measure the electrons that flow out of the same to ground, a transmitted electron detector is simlar to what exists in a TEM-STEM system.

---

(8) This slide is self explanatory. But it is useful to do the mental arithmetic while you sit on the machine and remember that your field of view is simply the size of your TV screen divided by the magnification!

---

(9) Let's get back to depth of field. This is the single most important reason why people use the SEM. No sample preparation and fairly easy and intuitive interpretation. Notice that the optical image on top tells you little. Very small bits, here and there, difficult to connect. The SEM image below is clear as a whistle.

(10-11) Let's see how depth of field works. When the spreading of the beam becomes large enough to compete with the size of the feature that the image is resolving, then evidently adjacent points will no longer be in focus. Spreading is governed by the beam divergence angle (the beam converges on to the same, but it is always refered to as divergence). ***High depth of field*** requires a ***low convergence angle***. From the figure, you can see (you'll see it later a few slides down the road) that it means that the aperture from which the beam emerges must be ***small*** and the distance to the sample surface must be ***large*** as illustrated in slide 11.

---

(12) This table illustrates how realistic beam divergences can give you depths of field tat are a couple of orders of magnitude larger than what you get optically. For example, a typical high mag. optical picture at 1000x has a depth of focus of 0.2 micron. In the SEM, one can easily get to 40 microns. Notice that at 100,000x, the DoF is down to 0.4 micron. This is not such a tragedy. At such a magnification, the field of view for an image spread across a 12" monitor is only 3 microns. You can still see some pretty high asperities from one end to another. And for asperities a few cm apart to **NOT** be in focus, you would have to have 0.4 micron asperities within 0.2 micron!

---

(13) There is a minimum aggregate of pixels that must be activated before an image is seen as distinct from the noise. If the smallest detail in the image covers several such aggregate picture elements, the image appears blurred, i.e., you see a large array of pixels without any features within. You then need to reduce the magnification so that it the features resolvable collapse a bit more in size on the screen **OR** you need to impove the image resolution so that additional features appear within. In both cases, the image will then appear sharp. Notice in the magnification sequence that all the features that are present are visible in the second picture. Further magnification is a waste of time (empty magnification) and only reduces the field of view. This what you get if you take someone's photo and then go on enlarging it.

---

(14) In an SEM you often have to tilt. When you do, the scanned length is increased along all directions except the tilt axis and is a maximum along a direction ***normal to the tilt axis***. Remember that magnification is inversely proportion to the scan length. That means the image is foreshortened. The 3 pictures illustrate the effect of tilt on the size of a sphere on a grid. The caption is self explanatory. Bottom line: remember that you are looking at a projection when you interpret your image.

---

(15) Similarly, the lengths you measure are all projections along the beam direction. Remember: your sample, unless polished, will have topography and you are seeing a 2-d projection of a 3-d structure.

---

(16) Let's now come to the gun and the optics. We'll only look at the bare essentials. The most common electron source is a thermionically heated W-filament. From your college physics you will remember the Richardson equation. Basically, when kT is large enough (> 2000 K), electrons will have a significant probability of escaping from near the Fermi level (where they are at 0 K) into the vacuum after surmounting the work function. This electron cloud builds up a space charge, rather like in a semiconductor junction and the field between these electrons and the positive filament deters further emission. However, if you apply a large negative potential V, to the filament with respect to an anode which is at ground, then electrons will be accelerated towards the anode. (Note: the anode and all the lenses are grounded through the cooling water. In any case it is better for the operator to be at ground and the cathode at –V, than the other way around!)

The filament fails eventually through creep thanks to the stresses at the high temperatures. Lifetimes can vary between 40 and 200 hours. Typically about 80 hours. Thus, they have to be replaced frequently but are relatively cheap.

LaB_6 is also used as a filament and you will see a summary of performance later. It is much more expensive, has to be heated carefully and is brighter and lasts longer. Field emission sources are expensive but are quite common and produce the best brightness and resolution.

---

(17) The filament is heated by a separate circuit to ~ 2700 K. Electrons emitted from the filament tip that is maintained at several thousand volts (-ve) are initially focused by what's called a Wehnelt cap. The cap has a negative bias of 100-200 V which has the effect of pushing electrons away from the cap and bringing them to a ***crossover*** of size d_0. This crossover size and the number of electrons it contains per unit solid angle and time defines the ***brightness*** that you can achieve. It is the most important parameter in determining the eventual number density of electrons that is available in the probe that is scanned across your sample and is critical for the signal achievable at high resolutions in imaging (and, to a lesser extent in spectroscopy and electron backscattering diffraction). The majority of the electrons hit the anode and flow back to complete the circuit. A small proportion goes through the hole in the anode and provides the beam for analysis.

---

(18) The reason a Wehnelt cap with a bias is used is shown here. Without a bias, the area of the tungsten tip that emits is large and you get a large current but poor focusing and therefore, poor brightness. Too large a bias and the negative equipotential lines reach the tip and emission stops.

---

(19) The first graph is self explanatory (see previous slide text). The second graph shows what happens when the filament is heated at a fixed accelerating voltage.

Because the tip is not perfect, one of the sides may start emitting before the others and a sudden jump is seen. Further heating distributes the heating and emission more uniformly and the entire tip starts emitting uniformly. ***Saturation*** at high current is essential because imaging and spectroscopy require a time invariant emission. In other words, any fluctuations in emission must damp itself out. This is accomplished (see previous slide) by using a bias resistor: any increase (decrease) in emission current leads to an increase (decrease) in i_e and in i_eR. The latter decreases (increases) the accelerating voltage ever so slightly and restores the original emission.

---

(20) Most lenses are electromagnetic and operate on the solenoid principle.

---

(21) The lens is nothing more than Cu windings in a soft iron core. In the gap between the upper and lower pole pieces an external magnetic field is produced. The key to understanding focussing is to recognize that an electron traveling along the optic axis, z, is initially only acted upon by B_r the radial component of the magnetic field(since $F=v\mathbf{x}B$ and B_z cannot act on v_z). However, the force produced is along θ (the tangential direction), Now v_θ can be acted upon by B_z to produce F_r. The net result is a spiraling of the electron under the combined actions of F_θ and F_r to cross the optic axis at some point which is a distance 'f' from where the field began to act. This is the ***focal plane*** of the lens and 'f' is the focal length. From now on you can use all the laws of paraxial ray optics for magnification. The only weird bits are that the focal length can lie within the lens and that you cannot move the lenses around but you can only change their focal lengths. The lenses are cooled by water to maintain stability of the current. Note that such a simple cylindrical lens's focal length is proportional to the ***square*** of the lens current. Therefore it is positive always and methods of correcting aberrations by combining converging and diverging lenses that can be implemented optically, are not available here.

---

(22) There are 2 configurations of the sample. The upper one is the common mode. The shape of the lower pole piece ensures that the magnetic field outside does not interfere with the electrons emitted by the sample on their way to being detected. The lower configuration enables very high fields and resolution and requires small samples that are immersed in the objective lens. This is similar to a TEM. We do not have such a facility but as we see later, sometimes the sample is so close the lower polepiece that the electrons emitted by the sample have to be detected by capturing them ***as they go back up through the lens***. How they are kept apart from the original ones coming down is a marvel of instrumentation.

---

(23) Treat these diagrams like ray diagrams in light optics and they are simple to understand. The first crossover is d_0 (in the gun) and the succeeding lenses (the first one is called the condenser and the second the objective) have the job of (a) demagnifying, i.e., reducing the size of the probe on the sample and (b) focusing the probe on the sample. For reasons that will be clear later, the entire angular range α_1, that comes through the condenser cannot be collected because of spherical aberration. An aperture is introduced to restrict the divergence to α_2 and the final probe on the sample is d_2. The quantity q_2 is the working distance. These quantities, beam

divergence and working distance, are extremely important and within the operator's control.

Increasing the working distance means reducing the strength of the objective so that it focuses at a larger distance. As you can see, α_1 comes down (depth of focus will improve) and probe size increases (resolution worsens).

---

(24) .If you want to reduce (increase) the probe size, you increase (decrease) the condenser lens strength. The position of the crossover d_1 goes up (down) and the final probe size d_2 decreases (increases).

---

(25) There are 3 classes of electrons that emerge from the sample. The very low energy ones are called secondaries (SE) and form the major signal for topographic imaging! Because they have energies of a few eV, they are easily produced by the incident electron as it goes down, but they only escape from the sample closs to the surface. Deeper ones are absorbed on the way out. Thus, they carry surface sensitive information. The second class are the high energy electrons with energy of the order of that of the primary electron. These are called backscattered (BS) electrons and can escape from much deeper in the sample. The in-between range is the one with characteristic Auger emission and is not of interest here. Already you can see that the signals represent different regions of the sample: the secondary electrons from the surface and the backscattered from deep[er within.

---

(26) Secondary yield increases as voltage drops to the point where the total SE emission (primary secondary plus the backscattered generated secondaries) can reach (first crossover) and even exceed unity. (After the maximum, the yield again drops to unity, the second crossover, at a very low voltage.). Clearly, operating at this second crossover voltage eliminates charging. But aberrations and beam brightness are much poorer at such low voltages. Basically, electron penetration reduces and more secondaries are produced near the surface where they can get out.

---

(27) ***This slide is a warning***! The sample is not the only source of electrons! The signal that leaves the sample (the high energy BS electrons) can hit anything-chamber wall, pole piece, detectors,… and excite fresh secondaries and backscattered electrons. All of these can, in principle, reach the detector and be detected. In one sense it is good: the signal is amplified. In another sense it is bad: a significant part of the signal has little to do with sample structure and can degrade contrast and hence affect the visibility of features.

---

(28) This slide illustrates an aspect of the SEM that is ***absolutely critical***. The interaction region in which the signal is produced is a strong function of accelerating voltage (and, as we see later, sample atomic number and density). The shortest etching time reveals a region in which the electron beam as done the most damage. This region is about as wide as the original beam but already much deeper than the beam size. The longest etching times reveal an interaction region that is ~ microns.

This is orders of magnitude greater than the beam size. Thus, beam size is NOT the same thing as the probe interaction volume.

---

(29) This slide shows the secondary electron detector. It is based on detecting the light (through photocathodes) produced by the SE using a scintillator. The electrons are first accelerated to 12 kV and the final signal from the photomultiplier is a pulse that is amplified before going to the display. The small +ve bias on the detector ensures that secondaries (remember they have low energy) can be deviated into the detector. Thus: the SE image is NOT just line of sight. The BS electrons have high energy and will also contribute to the image. But the SE signal is larger (see previous slide) and dominates. In the old days, BSE images were got by applying a –ve bias on the detector. It didn't affect the high energy BS electrons but it repelled the SE. Today, a different solid state detector is used for the BSE.

---

(30) This slide is about basic Rutherford scattering which dominates the way in which the electron travels through the sample. There are multiple axes and an exampleof how to read the citves is as follows: The voltage axis is set at 30 kV and the Z axis at 74 (atomic no. of Au). Now you can read off the probability of an electron being scattered by 70 degrees as 10^{-2} (pn the probability axis). You can also read off the scattering cross section as ~ 2 times 10^{-19}. If you want to do likewise for Al and C, you can do so in a similar manner and find that the probabilities are ~ 5 times 10^{-4} and 10^{-4}. If you want to change voltages, just shift rigidly all curves so that the curve for Au goes through the voltage you want. The probabilities of scattering are then read off as before. There are several points to get out of this figure:

(1) A backscattering event that can be detected must come from scattering by > 90 degrees. Otherwise, the electron just keeps on going into the sample!
(2) Heavy elements are MUCH better than light ones are backscattering.
(3) Backscattering increases dramatically with increasing voltage.

---

(31) This figure is self explanatory. Just remember that the right hand set of Monte Carlo simulations tell you the maximum depth up to which a backscattered electron can be produced and ***still be detected, i.e., get out.***

---

(32) Notice in the top part of this simulation (the bottom is for x-ray production and will be discussed later) that in gold, compared to aluminium:

(a) there are more lines emerging from the surface (more backscattered signal)
(b) The lines emerge from closer to the point of impact (better lateral resolution)
(c) The lines do not emerge from as deep in the bulk (better depth resolution).

This is why microscope manufacturers quote machine performance on gold! You will never get such resolution with Al. Don't even try.

---

(33) The range of an electron in the solid reproduced in this slide must be memorized! The dependence on atomic weight and atomic no. roughly cancel. Penetration reduces with increasing density and penetration goes as the 5/3 power of voltage.
BUT…brightness gets better as voltage goes up. Thus, there is a compromise. Work at

low voltages for the best resolution if you can get a bright gun (and there are better sources than a thermionic tungsten filament) and if probe size is not limiting (because it turns out that high voltages enable smaller probe sizes). In other words, there is no use working with a bright 20 nm probe on a sample where the signal emerges from 50 nm. Better to try and reduce the 50 to 30, brightness permitting, by going to a lower accelerating voltage. And finally, just to reiterate, lighter elements lead to larger interaction volumes and poorer resolutions.

---

(34) Let's start with the backscattered signal. It monotonically increases with Z. This is a basis for contrast: ***bright regions are heavy and loghter regions are dark!!*** In addition, the derivative of the top curve drops with increasing Z. That means discriminating between nearby elements is good up to about Mo and then diminishes progressively. The signal increases with tilt. The angle here is the angle the beam makes with respect to the surface normal. This is shown in the next slide as well

---

(35-36) The reason for the behaviour with tilt (and this is true for all elements and particularly so for the lighter ones) is just geometry. An electron that goes into a ***tilted surface*** has to suffer a ***smaller angle deviation*** to get out than one that goes into a ***flat surface***. Draw it and see. This also makes the BS electron emission anisotropic. It is easier to emerge on the side that is more or less a continuation of the original trajectory than one that emerges from the other side.

---

(37) The angular distribution is less important since (except in the case of electron backscattering diffraction), we do not look at spatial / angular variations of BSE emission. So this is not shown. The energy distribution is shown (absolute and cumulative) and illustrates that heavy elements produce BSE that are mostly near the primary energy. Lighter ones have a broad distribution. This has implications for detection. BSE below 1-2 kV are hard to detect by the normal solid state detectors in use since the efficiency of BS detectors drops with decreasing electron energy.

(38-40) The next 3 slides show the spatial distribution of the BS signal, both radially from the point of impact and in the depth direction. Notice, that the heavy elements get most of their signal from a smaller fraction of the Kanaya-Okayama range than do light elements. If you also recall that this range itself decreases as Z increases (see formula from earlier slide), then you will recognize a substantial improvement in resolution with increasing Z.

---

(41) The SE signal is dominated by the surface owing to the low energy of these electrons. But they can be excited by BSE on the way out and can therefore appear far from the point of impact. These aspects reduce contrast and resolution. For example, two adhacent points in te central figure may have different topography. But a large part of the signal will come from a remote location that is common to both points. Notice that the proportion of SE produced by BSE (the so-called secondary secondaries) as shown in table 3.8, increases with Z and can even be greater than the primary secondaries.

---

(42) A reminder about the SE yield as a function of accelerating voltage

---

(43) The SE signal is not too sensitive to Z except indirectly through the BSE signal as described previously. However, it is very sensitive to surface contamination, charge, etc. Funny effects (high emission) can be seen with very low Z insulators which are not yet fully understood.

---

(44) The dependence of emission on tilt is pure geometry. SE are produced at a constant rate throughout the escape depth since the primary beam loses little energy in this thickness. When the surface is tilted, bearing in mind that the escape depth, λ, is measured along the direction normal to the surface, it is clear that for a tilt θ, SE emitted throughout a depth $\lambda/\cos\theta$ will escape from the sample, i.e., the signal increases as sec θ. This is the origin of the tilt dependence of SE images and the reason why it is often important to tilt to see good contrast. Notice also that the rate of change of emission with tilt ($d\delta/d\theta$) also increases with θ, i.e., small scale topographic features are better visible when the surface is tilted by 45 degrees (leading of course to a foreshortened image) than at zero tilt.

---

(49) The examples in this and succeeding slides illustrate some of the principles outlined earlier. The first picture shows how surface features visible at 5 kV disappear at 20 kV because the signal is dominated by the bulk at the higher voltage.

(50-52) The next 3 pictures illustrate the way in which the BS signal is produced and leads to contrast in 2 different detectors, one ~ directly above the sample and one that is at a low take off angle. The one at a high take off angle requires that an electron suffers many scattering events before it can be turned around by 100-180 degrees. Such an electron will have traveled deep inside. The detector at a low angle is able to sense electrons that have been deviated by only ~ 40-60 degrees. Such electrons are concentrated near the surface. This explains why in both the microstrucures, the high angle detector senses the bulk. The low angle detector reveals the surface (whether the Cu-rich surface precipitates or the Al film on the eutectic alloy). So too with the visibility of the 200 nm film in slide 52.

---

(53) The effect of accelerating voltage is shown here emphasizing the benefits of high voltage in seeing sub-surface features. The first picture shows a buried interconnect in a semiconductor device that is only visible at 20 kV. Similarly for voids in a conductor that as failed by electromigration in the middle pictures. The bottom picture illustrates the inherent problems in quantitative assessment of volume fractions. The SEM does give you information in the depth direction. Unless that value can be quantified there is no way to assess the amount of the bright phase!

---

(54) A reminder: the SE signal is NOT line of sight. You can see inside regions that are shadowed with respect to the detector because the SE, thanks to the positive bias, travel a curved trajectory to get to the detector. Not so for te higher energy BSE. They travel in straight lines. That’s why you can see better on the bottom right compared to te bottom left. We’ll come back to the top pictures later when we discuss signal processing.

---

(55) This and the following slides illustrate effective illumination scenarios in the SEM. The text is self explanatory, I hope, in the caption, but here are some additional points. The first slide shows the equivalence between the SEM when used in the SE mode and optical illumination of the same region. Basically, the source and detector are reversed. The fact that SE can travel a curved path is equivalent to the optical illumination having a range of incident angles (which helps visibility when the surface is rough).

---

(56) For BS images, the situation nearly the same but since BSE trajectories are straight, the equivalent illumination is parallel (collimated). This makes for higher contrast, greater shadowing and a generally poorer image

---

Some points about BSE imaging (this is not covered in any slide).

(a) BS detectors are solid state devices whose gain is roughly proportional to the BSE energy. Poor sensitivity to low energy electrons means that spatial resolution is not so bad (you don't detect emission too far from the point of impact) even if it is far worse than the SE resolution. But it does mean that low kV imaging is not possible.

(b) Also, the high capacitance of BS detectors means that the relevant time constants are large and one has to scan slowly to acquire a decent image.

The next few slides show the same field of view under different detector configurations. Before looking at them, let's introduce a further detail in the BSE detector configuration. It is generally mounted concentrically around the hole in the lens through which the beam emerges and is directly above the sample. We can imagine splitting the detector into electrically independent parts: in the first few pictures we'll call them A and B i.e., 2 semi-circular portions which ***independently*** measure the BSE signal and in the later pictures we'll call them T(Top), B(Bottom), L(Left) and R(Right) i.e., a four quadrant detector.

---

(57) The 2 pictures on the left in this slide show images of a polycrystalline sample with grain facets that are formed by modulating the CRT with the sum of the signals A+B (top left) while the middle left shows the image by subtracting the 2 signals, A-B. As you can imagine, A and B will respond similarly if the atomic number does not vary across the sample. However, A and B will vary in an opposite sense (if A goes up, B comes down and vice versa) if topography changes i.e., local inclination of the surface normal in one region lies towards A and in an adjacent region towards B. Therefore the image A-B will suppress Z contrast and highlight topography while A+B will suppress topography and highlight atomic number differences. This facility is available in your machine and the effect may be seen in the images. The dark dots have disappeared in the middle left picture but the topography as seen from the shadows and contrast of the grains is superior.

---

(57) As mentioned earlier, the current that flows through the sample can also be measured and used to modulate the TV image. This specimen current image is shown in the middle right. You might imagine that this current is simply the probe current minus all the electrons that have been emitted from the top (secondary plus

backscattered). That is, broadly speaking true and that's why the dots appear bright. But there is an additional detail. The specimen current detector doesn't care about any directions. No trajectory effects are involved. Therefore the image loses most of its topographical contrast. We'll see the implications of this aspect in the Pb-Sn image later. The lower right picture reverses the specimen current contrast electronically so that it looks like the BSE sum image.

(58-59) Now we start using the 4 quadrant detector. The top picture in this slide is a conventional SE image using an Everhart-Thornley detector showing the topography of a Ni surface with pits and defects. The bottom is an image formed using the sum of all four quadrants (T+B+L+R). Notice that additional Z contrast arises in the flat portions revealing compositional segregation. The next few pictures are difference images and self explanatory (T-B, B-T, etc.). Notice that the brightness of the inclined part of the pit depends on which part of the detector is being used. When the T-B is used to modulate the image, because the lower part of the pit is inclined towards the detector and the top part is inclined away (shadowed), the top and bottom of the pit are in high relative contrast while the left and right are not too different. And so on. Thus, when you use ***ANY*** detector (single or multiple), you must be aware of its location relative to the sample. It may be necessary, for example, for you to rotate your sample and tilt the feature towards (or away) from the detector.

---

(60) This figure shows how contrast can be misleading. Lead-tin alloys are difficult to polish flat because they are so soft. The contrast in the top picture has nothing to do with atomic number. It is a polishing artifact. The bottom image is a specimen current image with no trajectory effects of electrons and shows true atomic number contrast between regions rich in Pb and in Sn.

---

(61) We now switch to signal massaging. Up to now, we have dealt with what would be called linear amplification. A large signal = bright image and the relationship is linear. The signal-distance function along a line that makes a linearly amplified image look good is typically like the one shown on the bottom. The whole range of brightness is covered without saturating (i.e., no regions brighter than fully bright or darker than black dark!).

---

(62) When contrast is low (figure a), we can improve it by first stripping the average background (b) and then amplifying it so as to increase the contrast so that the full dynamic range is covered (c). If you over-do it (d) then again signal saturation can occur. The image of such a ***differentially amplified*** signal is shown below of an Al-Si alloy. Backscattered contrast differences are not large because of the similarity in atomic number. The greater visibility of the Si needle at bottom right in the differentially amplified image is apparent.

---

(63) Non-linear amplification ***preferentially amplifies*** either the low brightness ($\gamma>1$) or the high brightness end ($\gamma<1$).

---

(64) These images show how features that appear too dark in the interior when the outside looks OK, can be made visible without making the rest of the image too bright.

(65-66) In this and the next slide, notice that combining functions (differentiating, taking the modulus, etc.) enable different types of contrast to emerge from the same region.

---

(67) The resolution of an image is governed by many aspects starting with the probe size. The minimum probe size achievable in an instrument is determined by aberrations and voltage. Generally, higher voltages give you smaller (and brighter probes). The fundamental problem with this criterion is that in MOST samples, image resolution is ultimately governed by the probe interaction volume, i.e., the size of the region from which the signal is produced, leaves the sample and is detected. This means reducing your voltage which also means reducing beam brightness and (as we shall see) worsening aberrations. At some point, contrast can limit resolution, namely one electron hits your sample every few days and noise overwhelms the signal. Thus, the theoretical resolution for ***your*** sample depends on atomic number, the type of signal, the available contrast (e.g., topography). Given these pre-conditions, you have to play with accelerating voltage, probe size, working distance and beam divergence. Let's start with aberrations.

---

(68) You may recall that we use apertures to limit the beam (see ray diagram from earlier) as we focus and change the beam size. This slide illustrates how aberrations require an optimal beam divergence (remember that beam divergence, α, is proportional to aperture size and inversely proportional to working distance). The beam spreads due to diffraction: reducing spreading requires the largest possible value of α. However, spherical aberration defocuses the image of a spot to a disc whose size varies as the cube of α. Chromatic aberration is not so bad and only varies as α. Spherical aberration is the same as in light optics but cannot be easily corrected. It is fixed for your machine. Chromatic aberration arises from 3 main causes: the first is voltage instabilities in the transformer, the second is the thermal spread due to kT in a heated source and the third is the so-called Boersch effect wherein electrons meeting at a cross-over with the same energy end up exchanging energy (electrostatic repulsion) and developing a distribution of energy (hence wavelength from de Broglie and consequent chromatic aberration). Only cold field emission sources (see later) which are extremely small and bright and where there are no cross overs along the column can avoid the latter two effects. The last aberration, astigmatism, is a correctible one (and one which many students have great difficulty in correcting), since it comes from the loss of cylindrical symmetry in the lens and is corrected by adjusting the strength of additional coils so as to make the lens symmetric. But it builds up with contamination, a lot of which can be specimen borne, and so it is contained by careful sample handling and maintaining a clean vacuum..

---

(69) Notice how with astigmatism there is streaking in the image when you go out of focus. The direction of streaking changes by 90 degrees when the sign of focus is

reversed (i.e., when going from focusing above to below the sample). Minimising the streaking is one way of eliminating astigmatism.

---

(70) A few words of clarification on this routine optimization of a function in which probe size increases with α due to aberrations and decreases as α increases due to diffraction. The current in the beam is an ***independent*** parameter which affects the probe size. If you need a large current, then ***automatically*** your beam size goes up because you have to use a large α. (There is a modern generation of JEOL microscopes that apparently gets around this problem, but in the machines we have, this is the constraint). What do we mean by ***"if we need a large current"***? Current dictates signal. If your current is too low, your resolution becomes limited by noise and all the fineness in probe size or interaction volume will not help you. Because the probe / beam divergence requirement ultimately stems from the brightness of the original source, the brightness, β, comes into the first equation. Make your source brighter and the first part of the first term drops. This is part of the reason why field emission sources (and LaB_6 if you don't have so much money) are so effective compared to thermionic tungsten sources.

---

(71) The next few slides illustrate the effects of aberrations. The first one combines everything: diffraction, spherical and chromatic along with a ***specific beam current of 10^{-10} A.*** The three straight lines for diffraction, spherical and chromatic show the dependence on beam size when each operates ***independently.*** The sum of all of tem is shown by the curve for $\sqrt{(d_g^2+d_s^2+d_c^2)}$ which will depend on the amount of chromatic aberration you have (1V, 2V, etc.). Remember that chromatic effects go as $\sqrt{\Delta V/V}$, where V is the voltage. Therefore, low accelerating voltages make this particular aberration worse. Thus, at low beam divergence you are limited by diffraction; at high by spherical aberration; the transition by chromatic aberration.

---

(72) This slide shows the final smallest achievable beam size taking only one aberration at a time, and subject to different overall beam currents for a W filament operating at 20 kV. At high current, the spherical aberration limit is higher and at low currents the chromatic effect is higher. The higher curve always applies (you always lose!) Remember that the choice of beam current is dictated by sample contrast.

---

(73) Here we see the effect of aberrations on the optimum beam divergence. The lowest curve applies, i.e., at high beam current you are limited by spherical aberration and at low currents by the appropriate chromatic aberration line.

---

(74) Notice how much brighter and more stable in energy are the field emission (FE) sources. This is why they provide such excellent images at high magnification. However, their total current can be low. This is not mentioned in the table but W-hairpin filaments can produce a total current of microamps, while the corresponding amount for a FE source may be in the nanoamps. That means if you want to do imaging of a low contrast feature at low magnification or spectroscopy of a trace element without high spatial resolution, you ***may*** be better off with a tungsten source. So too with electron backscattering diffraction (EBSD).

---

(75) Reminder of the difference between merely going up in magnification and making the changes in operating conditions that allow you to get the resolution that justifies doing so.

---

(76) Now we come to signal-noise. This is like any other data acquisition ystems that suffers from noise and background as you may have encountered in x-ray diffraction or photoelectron spectroscopy. The longer the acquisition time (i.e., the slower the scan speed and the longer the time the beam sits on one point), the better the signal relative to noise. Thus, a challenging feature shown in this picture is picking out a small gold particle sitting on a big one. The only difference in signal is the part of the electron emission that comes from the small particle itself and this will be small compared to the emission from the substrate. This difference is detectible at the larger beam current scan which is also for a longer time shown in the bottom. The features in the upper figure are starting to blend with the random noise.

---

(77) The bottom line is that when you want to detect a feature with low contrast (C) you need more beam current (i_B) or a longer acquisition time (τ_f). If you do not wish to compromise on resolution, increasing τ_f is the only option. Providing the beam is stable (and does not wander off), providing the specimen is stable (and does not wander off) and provided hydrocarbon based contamination is minimal (i.e., you plasma cleaned the surface and did not handle it), you can do experiments with scan acquisition times of tens of minutes and extending to one hour, ***if necessary***.

---

(78) The bottom of this slide illustrates the serious loss in resolution when contrast degrades. And remember that contrast is higher when Z is higher and more topography is present. That is why fracture surfaces are gold coated (when chemical analysis by x-ray spectroscopy is not critical).

---

(79) This slide returns to the old point that if you want to see something that only differs slightly in scattering amplitude with respect to its background, you must eliminate as much of the background signal a spossible. Remembering that SE emission from the walls of the chamber and pole pieces can contribute to background (this is the tertiary SE signal, the secondary being the SE generated by departing BSE), the bottom picture shows how the same Au particles on Au become visible when the chamber is coated by a low SE emission material.

(80) Small particles often appear very bright but featureless. This can be understood from the schematic in the lower picture. Because of the curved surface, it is easy for BSE to emerge through the side from regions far from the point of impact. Basically, the entire volume of the particle is explored by the beam, regardless of the location of the beam. Thus, surface topography takes a back seat in the image.

---

(81) We now change subjects to come to insulators and charging. It is normally not possible to view insulators because if the incident charge cannot flow to ground through the sample, then the sample develops a potential and this repels the incoming

beam. In a dramatically bad case as shown here, the beam turns around and hits the pole piece, never actually reaching the sample.

(82-84) The secondary emission coefficient approaches and can even exceed unity (1) for materials at low voltages. That means nearly as many electrons are emitted as hit the sample. This greatly reduces or eliminates charging but the catch is that you have to work at voltages below ~ 2 kV where beam brightness is usually too small for conventional electron guns; only field emission sources are viable. Notice the artifacts when charging occurs.

---

(85) Biological tissues are frequently stained by heavy metal salts (Os) to provide conductivity.

---

(86) The standard way to eliminate charging is to coat with Au-Pd. This sequence shows why sputtering is best. Gold atoms get scattered by the Ar ions and arrive on the surface from all directions. Evaporation in high vacuum, in contrast, is a line of sight method and rough surfaces / fibres, etc. can suffer from shadowing and some parts never get coated. The artifacts in the image when charging is present range from lines in the scan to sudden very bright or dark regions to time variable contrast.

---

(87) Modest conductivity is sufficient to deter charging as in this mica insulator at 50 C.

---

(88) High energy backscattered electrons are less affected by charging. Similarly, fast scanning can help. Both of these degrade resolution but that is better than not seeing anything.

---

(89) Charging artifacts are insidious as shown by the wavy interface that disappears when charging is properly eliminated.

(90-92) Stereo imaging relies on taking two pictures at slightly different tilts and viewing each with one eye. This can be done in a stereo viewer or, by holding them in front of your nose and focusing behind them. Each image splits into 2 when out of focus. When the inner images of both eyes are superimposed on each other, you will see stereo. (The outer images are a distraction and must be ignored.) This requires practice.

(93-101) The following pictures illustrate application of SEM to different types of samples. The fracture surfaces in this picture come from different alloys that display different modes of failure.

---

(96) Remember that specimen preparation is important. This sample looks so dramatic because of the deep etch and the tilt.

---

(97) Many subtle features of failure in the glass fibre reinforced plastic can be seen, including the length of pull out, the size of the flaw in the fibre that initiated failure, the onset of dynamic instability during glass fracture, etc.

---

(98) The samples here are from semiconductor processing and is self-explanatory.

(99-101) In-situ experiments can be done in modern machines with the necessary attachment, including straining, heating (this picture), chemical reaction, etc.

---

(103) The field emission (FE) tips are of 3 types. The cold emitter uses only voltage to extract electrons. Because emission is surface sensitive, it is important to periodically clean off contaminants by a process called “flashing”. A thermal FE heats the filament as in a thermionic gun, so that the tip stays clean, but at the cost of a thermal energy spread in the electron energy (chromatic aberration). The Schottky emitter uses ZrO_2 to reduce the workfunction and produces a large current from a large area but with a small virtual source (project the diverging beam back to get to the virtual source). This means that small probes require crossovers as in a thermionic gun (Boersch effect; see slide on resolution) with consequent loss of resolution.

---

(104) Top shows work function and energy levels during field emission. Bottom shows the 2-anode structure. The first is optimized at ~ 5 kV to extract the electrons from the tip. The second anode accelerates (or decelerates) the beam to the desired energy.

X-ray microanalysis (Slides 105-151)

(105,106) We now come to x-ray microanalysis. X-rays are emitted when a high energy electron or photon ejects a core electron. (See a basic text on atomic structure if you have forgotten the terminology.) The vacancy is filled according to the selection rules governing transitions (change in spin, angular momentum etc.) A transition from L→K leads to an energy reduction that can appear as an x-ray photon that is labeled as K. Transitions ending from a higher state to an L level will lead to the emission of an L x-ray and so on. Correspondingly, transitions from L to K are called K_α and transitions from M to K are called K_β. Clearly, making a vacancy in a K state can lead to the emission of a wide variety of x-rays and their relative emission intensity is fixed. But most importantly, these energies are independent of the original impinging electron energy and are unique to the elements in the sample, so they provide a fingerprint by which to identify the elements present. The x-ray energy, unlike in XPS, is not sensitive enough to reflect changes in bonding since only core electrons are involved. **Note:** The vacant electron position can also be filled through Auger emission. This process is more efficient for light elements. X-ray efficiencies for many important elements are poor (C,N,O).

---

(107) A critical energy (and therefore accelerating voltage) is needed to eject an electron to an unoccupied state. This is approximately the ionization potential. Below this voltage, x-rays are produced only by the deceleration of the electron and this leads to a continuous spectrum of x-ray energies. This brehmstrahlung is always present as a background.

---

(108) By analogy with the Kanaya Okayama range for the electron in the solid, one can also see that there is a distance over which the incoming electron retains enough energy to ionise the sample and this distance is obviously lower than the distance over which the electron comes to a rest. Notice that the critical voltage is simply the ionization potential. Thereafter, the depth from which x-rays are produced increases rapidly with energy. Once again, if spatial resolution is important, heavy elements are better and so are low voltages (but staying above E_c). The only problem with low voltage is that the intensity of emission also drops.

---

(109) From the previous slide it should be clear that each element will contribute x-rays from a different volume of the pear-shaped region. Also different x-rays of the same element will come from different regions. Thus, ***there is no linear correlation between intensity and elemental concentration.*** In addition, x-ray production can happen from deep inside compared to SE emission. Thus, ***it may be possible to see features in an image but not be able to analyse them with respect to their background.*** Imaging resolution is measured in nm. X-ray analysis resolution is measured in microns! The task of identifying elements, quantitatively estimating them and then possibly forming elemental maps that show the spatial distribution of specific elements will take us through the next ~ 30 slides.

---

(110) Before an x-ray can be detected it has to leave the sample and suffers partial absorption along the exit. (Absorption is of various kinds: all we mean here is that an x-ray that is absorbed is no longer continuing in the same direction towards the detector.) Because an x-ray of one element can excite ionization in a neighbouring element, absorption (because it appears in the exponential) can lead to dramatic differences in detection. For example the absorption of Ni K_α in Mn and Fe is several times that of Mn and Fe K_α x-rays in Ni.

There are two types of x-ray detection systems. The most commonly used one is an energy dispersive detector (EDS) that "simultaneously" analyses all energies, generally from 0-20 kV (more energetic x-rays go right through the silicon detector). This type of detector is fast, has an energy resolution of 130 eV (cf electron spectrometers with 0.1 eV in XPS) and has a modest signal to noise. The other type disperses the spectrum using a crystal grating according to Bragg's law (wavelength dispersionm WDS) and has much better sensitivity, quantification and energy resolution. We will look primarily at EDS.

---

(111) Notice that $K\alpha_1 - \alpha_2$ splitting is not seen. Notice how the L-lines merge into one another. But α- β splitting is seen.

---

(112) The relative proportion of K_α to K_β and L to K lines is a good check against proper identification. If you see only the L and not the K, maybe your voltage is too low to excite the higher energy K. But if that is not the cause, maybe your original identification was wrong!

(113,114) When quantitative analysis is carried out, proper background subtraction is necessary. Weak peaks may become apparent only when this is done. Notice the characteristic shape of the Brehmstrahlung in 114.

---

(115) X-ray generation is an interplay between efficiency of production and the loss of electrons through backscattering. Broadly, with increasing atomic number, the incident electron loses its energy faster as it goes into the bulk or disappears through backscattering and more of the x-rays are produced close to the point of impact.

---

(116) One reason more x-rays are produced slightly inside than at the surface is that electrons at the surface can leave through backscattering! Another is that when the electron goes in a bit, its direction is no longer normal to the surface because it has suffered a collision. Effectively, this means that its path length increases (cosine effect) at a given depth from the surface from what it would have been if the trajectory were normal to the surface. The function $\varphi(\rho z)$ is the number of x-rays in a slice dz of the sample divided by the no. produced in dz of a free standing film.

---

(117) Notice that emission decreases even faster as Z increases than apparent. The density (by which the x-axis is normalized) of Cu > Ti>Al.

---

(118) As mentioned before, the price of high spatial resolution is poor signal. Working close to the critical voltage will minimize internally generated signal. May be practical when the amount of the element is large. Notice also that the strong dependence of the size of the region of x-ray production on voltage implies that larger probe sizes can be used for microanalysis than are necessary for imaging. (You gain signal and you don't sacrifice resolution. Do not blindly work with the same probe when you switch from imaging to analysis.)

---

(119) The next 3 slides are on powders. There are artifacts when analyzing samples that are not semi-infinite. Here is one. Because the powder is not infinite in extent (i.e., electrons leave the sample at the bottom while still retaining useful energy), the number of x-rays produced is a function of powder size AND element! The lower slide illustrates the effect of tilt. As backscattering emission increases (see earlier), x-ray production suffers!

(120,121) The other aspect of a powder's topography (this slide and the next one) is that depending on where the detector is, one part of the powder may give you a different spectrum from another part due to absorption (and maybe fluorescence) differences.

---

(122) Once you get your spectrum, you can do many things with it You can use the number of counts in a specific window of energy to modulate your TV image. This gives you elemental maps. Thus, the BSE image tells you that compositional variations are present. The elemental maps tell you exactly where the Ag, Cu and Sn are. Similarly for the line scans showing the variation of intensity of a particular x-ray across a line in the sample (3 slides later.)

---

(123) Setting contrast and brightness levels during x-ray mapping so as to ensure that the full range of elemental concentrations is seen without noise or saturation

---

(124) Use common sense in interpretations!

(125) Once a digital map is acquired, the data can be read across arbitrary lines to get line profiles

---

(126) How much can be detected?

---

(127) Function massaging can be done as with differential processing once the information is available in digital form. For example, the function Au **x** Pt tells you where both elements are located to a significant extent.

---

(128) All elements have to be specified for proper quantitative analysis as we will justify later. In some detectors oxygen cannot be detected and in the best of detectors it is not easy to quantify as we will see later. But if an oxide is stoichiometric, then oxygen can be specified by proportion with the detected metal elements. When that is done, the "dark holes" in (d) are identified to be really holes.

---

(129) Sensitivity again. Here, we are able to clearly see a difference of 5% in Ag in a Au-Ag alloy. Obviously, in this sample, even 1% difference would have been detectible.

(131 to 136) The next 6 slides on EDS are self explanatory. The lower part of the last one deals with fluorescence. Because absorption often involves the emission of another x-ray and because x-rays are so penetrating, the volume involved in fluorescence is very large.

---

(137) This slide summarises the ranges of interaction for all the signals so far. Notice the wide variation and also the fact that fluorescence can take place miles away! Of course, all these numbers change with voltage and atomic number, but the trend remains the same. As far as x-rays are concerned, remember that production is followed by absorption on the way out and is complicated by fluorescence which can greatly increase the region contributing to the signal.

(138,139) Some routine considerations for collecting and viewing a spectrum. Low acquisition times tell you the principal elements. Longer times are needed for trace elements. Notice that the same peak can belong to the K family of one element or the L (or M) family of heavier elements. Overlapping peaks can complicate identification and quantification. Light elements are poor x-ray producers and show weak peaks despite being abundantly present.

---

(140) Now, a brief excursion into wavelength dispersive spectroscopy. Because the detector begins with a Bragg grating, the wavelength range that a given crystal can detect is limited and a full range of wavelength detection will require multiple crystals all of which have to be located in a parafocusing geometry (see any text on x-ray diffraction for details). The wavelength resolution depends on the width of the diffraction maximum of the crystal planes which in turn depends on perfection, absence of defects, etc. Similarly, the sensitivity will depend on the structure factor, distance from sample, etc. The figures here show the enormously improved energy resolution of the WDS spectrum compared to that of EDS.

---

(141) Another comparison between WDS and EDS. Look at the same range of x-ray energies and see the difference in peak differentiation. The bottom pictures show the WDS spectra from 2 different crystal gratings.

---

(142) This slide is a quick summary of error analysis that illustrates the importance of a low background. Noise is not discussed here, but clearly the ability to quantify the counts under a peak depends on the statistical uncertainty involved in peak smoothing. The bottom line is that a quick check of the total number of counts under the particular peak in the spectrum can allow you to estimate the ***uncertainty*** in your analysis. Routine work often gets you about 10% (relative, i.e., 50% ± 5) accuracy in elemental content. To get to 1% you may have to count for long times. Remember

that if spatial resolution is not important, accelerating voltages, probe sizes and apertures can be increased.

(143-147) The slides on quantitative analysis (ZAF) are self explanatory.

(148,149) The advantages of low voltage EDS if you have a bright field emission source and exploit the limited x-ray emission volume at low kV without sacrificing signal

---

(150,151) The next couple of slides illustrate in-situ experiments at high pressures (low vacuum) and in moisture that can be carried out in special microscopes. In environmental machines like our ESEM, materials that would degrade in high vacuum or low water pressure (like tissues) can be viewed. A further advantage of low vacuum viewing is that charge can leak through the air to ground and therefore insulators can be viewed without coating. However, at the highest magnifications, resolution will be poorer due to scattering of the beam by air.

Electron Backscattering Diffraction (EBSD) Slides 152-158

(152,153) We now come to electron backscattering diffraction. So far, we have ignored crystallography. However, if we think of backscattered electrons as emerging from a point about 100 nm inside the sample, it is clear that along certain directions the BSE will be diffracted by the lattice planes of the sample. (Something similar happens during Kikuchi line formation in the TEM.) Under these circumstances, the angular distribution of BSE intensity (which we have ignored so far; we simply put a detector somewhere and measure the total charge that hits it) will have discontinuities wherever the crystal is at a Bragg angle. This is schematically shown in the figure below, where we show the general case where the beam is incident at a slight deviation from the Bragg angle. The Bragg scattered beam is always at $2\theta_B$ with respect to the incident beam. This means that along the original direction (before diffraction) the BSE intensity will drop, while at an angle of $2\theta_B$ with respect to this direction, the BSE intensity will rise. The locus of all directions that can hit the lattice plane at a given angle is a cone and therefore the directions along which the intensity is reduced or increased also lie along a cone ***at a specific semi-angle to the lattice plane,*** i.e., the normal to the lattice plane, which is the cone axis, makes a specific angle with respect to the cone surface. What's more, the cone rotates with the crystal. The next slide, 153, will show that the intersection of the cone with a recording plane (film or CCD array) is a hyperbola which, because of the low value of the Bragg angle, is practically a straight line.

The situation is actually a little more complicated and full details can be found in any book on electron diffraction. When the electron beam is incident at an angle that is close to the Bragg angle, the amplitude in the crystal can be represented by 2 (or more) Bloch waves which represent the solution for the wave function under a periodic potential. In the simplest case, when only one lattice plane is diffracting strongly, there are 2 waves inside the crystal that peak respectively at and in-between the atomic planes. The wave, let's call it wave 1, that peaks in-between atomic planes, channels easily and is transmitted. The other one, wave 2, being high in amplitude at the atom core, interacts with the atom and is inelastically scattered and heavily absorbed. When the incident angle is ***less than the Bragg angle, wave 2 is more strongly excited. Thus, most of the electrons are dumped in a basket that is heavily absorbed.*** As you can see, (see upper figure) if the incident angle is less than θ_B, then the scattered angle will be greater than θ_B, i.e., between 3 and 4 or between 1 and 2. Thus, backscattered intensity will be low The central part, i.e., directions corresponding to an incident angle greater than θ_B excite wave 1 more and lead to easy channeling and large BSE signal. Thus, the lines consist of a bright central band without a slightly darker outside. Remember that the centre of the band corresponds to $\theta = 0$, and this is parallel to the projection of the lattice plane on the CCD camera. Thus, the BSE pattern tells you the orientation of lattice planes in the sample relative to the CCD camera and the beam. This pattern can be indexed and thus, grain orientation can be measured to the resolution of a backscattered image, namely the size of the region that constitutes the BSE source. The most intense part of the BSE emission region may be as small as 50 nm if you work at a low voltage such as 5 kV

in something like Fe. Thus, one can do texture studies, grain boundary misorientation, phase identification, etc. along with microanalysis and imaging, thus completing the full range of information that is needed (the only thing missing is bonding and valence state).

---

(154) The crystallographic information was originally got by rocking the beam (or by viewing single crystals at very low magnification) in what were called channeling patterns. Thus, this technique goes back many decades. However, it was only with the introduction of fast image processing and transform methods along with sensitive detectors that it became a practical tool for routine analysis. It is still a slow method since the variation in BSE signal due to electron channeling is small (i.e., weak contrast).

(155,156) Texture in a superconductor is shown here after the orientation at each point on a polycrystalline film has been determined. Comparison of neighbouring points enables the creation of maps wherein boundaries separate regions whenever the misorientation exceeds a specified angle. Thus, when the specified angle is small, the sample show many “grains”. When the allowed misorientation is allowed to reach 6 degrees, practically the whole field of view is a single grain. Such measurements can lead to pole figures (C-5) as routinely obtained by x-ray diffraction.

---

(157) This is an orientation map showing how many grains, not fully recrystallised, are present in the interior of a poorly heat treated Ti-containing steel. Contrast with the optimum microstructure above.

---

(158) EBSD orientation map reveal that the dendrite of Si seen in the SE image on the left are composed of twinned orientations wherein each colour in the right hand picture corresponds to a specific orientation.

High resolution, low voltage imaging, detectors (Slides 159-177

This section can be skipped for basic operation.

At high magnifications, one needs to work at such low working distances that secondary electrons cannot be effectively captured by the E-T detector outside. Instead, they are allowed to travel back up through the bore of the pole piece (because of their low energy, they travel a spiral path that does not interfere with the incoming high energy beam) and are detected inside the column. Backscattered detection at low voltage (remember that signal and probe size permitting, beam spreading decreases at low voltages and therefore contributes to better resolution) is also a challenge since detector efficiency drops rapidly below 1-2 kV. Many methods are available commercially to deal with these problems.

(159-163) In Zeiss microscopes (159-161), the SE and BSE detectors exploit the physical separation of the low and high energy electrons at a particular height. The SE travel in the annular outer region and hit the detector while the BSE go through the hole and are detected above. Similar designs are used in JEOL and in Hitachi (162,163) microscopes. In Hitachi, the ExB filter (162) uses the fact that an electric and magnetic field act in opposite directions for electrons traveling down, but in the same direction for electrons traveling up the column. This, the fields are matched so that there is no net force on the primary electron but the SE and BSE are deviated towards detectors. In some instances, low energy BSE are detected by making them hit a plate where they eject secondaries and then detecting the secondaries. Slides 162 and 163 show how by biasing the collecting electrodes, one can attract SE, repel them, enhance BSE by allowing them to hit a plate where they eject secondaries which are allowed to leave (negative bias) or prevented from leaving (+ve bias)

(164-167) These pictures show how one is able to mix the SE and the BSE signal in different proportions. Thus, in figure 164 left, the addition of the BSE signal reduces charging and the unwanted high topographic contrast at the edge as well as charging (BSE are less prone to specimen charging since they have more energy). So too in figure 166.

(168,169) YAG scintillators offer the best BSE discrimination by converting them to light and detecting the optical signal

(170) The top 3 pictures are self explanatory. In the bottom set of 4, notice that the BSE signal enables the true size of the feature to be seen despite the carbon contamination since the high energy electrons are less impeded by the carbon and simply do not register it at all. It is the SE signal whose resolution is poorer. Thus, BSE and SE resolutions approach one another at low kV.

(171) We have seen earlier that probe sizes are finest at the higher voltages. In particular, chromatic aberration is ruinous at low V. One way of exploiting the low beam spreading in the sample at low kV with the better probe resolution at high kV is

to decelerate the beam just before it hits the sample. Originally patented by Zeiss, variants now come from all makers.

(172) Graph showing how resolution is maintained in the gentle beam mode by deceleration just before hitting the sample and images showing how a low landing voltage heightens topography.

(173) Remember that the point here is that in gold, the resolution achievable at 500 V by decelerating a 2000 V beam by 1500 V is comparable to a 1000 V undecelerated beam. In your sample, which is not gold, the comparable probe size achievable at a net voltage of 500 V can be exploited to reveal features that the low voltage enables in light elements in which otherwise beam spreading would be limiting.

(174) Deceleration also enables sensitive materials to be viewed with good resolution at a voltage that avoids damage.

(175) A new revolutionary technology from JEOL promises the same probe current at smaller probe sizes through an aperture control lens (remember that because of spherical aberration, traditionally, we have to use smaller apertures and throw away electrons traveling at a large angle when we reduce the probe size). Little change in resolution in going from 50 pA to 1 nA (a factor of 20 in current).

(176,177) Secondary electrons have an energy distribution in part because the barriers to escape are different when the compositions and dopants change in a semiconductor. Filtering the electrons arriving at the detector enables a large contrast difference between p and n type regions (H-18 schematic and H-19 actual)

Some tips during operation

1. Start at a low magnification and get a feel for your sample. Gradually go up.
2. Check your accelerating voltage and probe size. Generally, one starts at 10-20 kV.
3. The SE mode is the standard one to start with. If contrast is poor, consider increasing the probe current, tilting, going to a higher voltage.
4. At high magnification, if the resolution is poor, consider a smaller working distance and smaller probe size. If signal is not an issue, maybe reducing your voltage will help.
5. If depth of field is poor, consider a smaller aperture or a larger working distance.
6. If contrast remains poor, consider increasing the acquisition time (slower scan).
7. If there is significant topography (shadowing, holes), remember that you can rotate the sample and tilt so that you get a different perspective on the same feature.

Specimen preparation

The great advantage of the SEM is that no special preparation is mandatory for viewing. However, the usual considerations of metallography apply. If you want to see contrast from different phases, it is a good idea to etch and convert it into topographical contrast. Specimens must be electrically grounded. This means that even if your sample is conducting, it must be well mounted so that there is a good electrical connection between the surface being examined and the holder. Usually this is done with graphite or silver paint when the sample is mounted in plastic or is some jagged shape with poor contact. Plasma cleaning (removes hydrocarbons before they crack under the beam) is essential for the highest resolution work (and for prolonged microanalysis when the beam sits on one place) to ensure that contamination is minimized. (Never handle the surface being examined, blow off dust before evacuating.) For insulating samples that can be coated, Au-Pd coating is the first route to take. Only if you need to do quantitative microanalysis do you need to avoid Au and maybe try carbon. Working at high pressure is another possibility on the ESEM. Low voltages are yet another possibility if you forgot to coat. But remember that microanalysis may not be possible at low voltages. But as long as you don’t care about locating the probe in a specific place, you can still do microanalysis at higher kV when the specimen is charging. Your image will be bad, that’s all.

SEM Quiz

Choose the best answer

1. The typical accelerating voltage range that is used in a conventional (thermionic) SEM is (in kV):
   (a) 1-10
   (b) 5- 30
   (c) 10-40
   (d) 1-40

2. A secondary electron's energy range is typically (in eV):
   (a) 0-100
   (b) 50-100
   (c) 0-50
   (d) 500-2000

3. This question is about backscattered electron (BSE) emission and its change with tilt angle (i.e., deviation from normal incidence of the electron probe):
   (a) BSE emission decreases as the sample is tilted.
   (b) BSE emission is independent of tilt
   (c) BSE emission increases with tilt and gives you a brighter image
   (d) BSE emission increases with tilt but this increase can only be exploited if the detector is placed at a low take off angle.

4. Secondary electron emission :
   (a) is strongly dependent on topography alone
   (b) is strongly dependent on atomic number alone
   (c) is dependent on both topography and atomic number
   (d) is relatively independent of topography and atomic number but depends on the material's conductivity

5. The resolution of a SE image can improve at lower voltages because:
   (a) Direct SE emission is more efficient
   (b) More secondaries are produced because BSE emission increases
   (c) The depth of SE production drops
   (d) The lateral range of BSE reduces thereby reducing the indirectly produced SE.

6. When an image is viewed at 1,000,000 times on a typical 17" TV monitor, the scanned field of view is about:
   (a) 0.3 by 0.25 microns
   (b) 0.4 by 0.3 microns
   (c) 0.8 by 0.6 mcirons
   (d) 10 by 8 microns

7. The depth from which the bulk of the secondary electron signal can emerge is (in nm) about:
   (a) 10
   (b) 100
   (c) 50
   (d) 200

8. To observe a very rough surface, one needs to work with
   (a) a small probe size
   (b) a small aperture
   (c) a small working distance
   (d) a low accelerating voltage

9. If the accelerating voltage is increased from 10 to 20 kV, the depth to which electrons penetrate increases by a factor of about:
   (a) 2 (b) 3 (c) 4 (d) 5

10. An insulating sample cannot be viewed with the following modification:
    (a) Coating it with a conducting metal
    (b) Working at about 1 kV
    (c) Working at high pressure
    (d) Working at very low scan rates

11. The contrast of an image in the case of a sample with light elements **cannot** be improved by:
    (a) Working at high accelerating voltages
    (b) Coating the surface with :a high atomic number element, such as gold
    (c) Reducing the probe size
    (d) Increasing the acquisition time

12. This question is not multiple choice. A sample of carbon nano tubes is being imaged at high magnification. List requirements (high or low) for the following operating parameters:
    (a) accelerating voltage
    (b) spot size
    (c) working distance
    (d) aperture size

13. Which of the above requirements might change if gold was the sample?
14. When increasing magnification from 500x to 10,000 x, one must remember to do the following:
    (a) reduce the spot size
    (b) reduce the working distance
    (c) increase the accelerating voltage
    (d) switch from SE to BSE mode.

15. When one wants to get topographic contrast in the backscattered mode, the 2 signals from each half of the detector are:
    (a) added
    (b) subtracted
    (c) divided
    (d) multiplied

16. When SE images are acquired, the following can help to improve the signal without degrading resolution:
    (a) tilting the sample
    (b) keeping the sample flat
    (c) reducing the working distance
    (d) increasing the working distance

17. A network of Cu lines is buried 1 micron below a surface layer of Si. The voltage at which the Cu is seen is about 15 kV. If the situation is reversed (i.e., Cu on Si), the critical voltage to see the Si would be:
    (a) slightly larger
    (b) slightly smaller
    (c) much larger
    (d) much smaller

18. K x-ray fluorescence of element Z_1 by K x-rays of element Z2 are ,most probable when:
    (a) Z_2 is slightly > Z_1
    (b) Z_2 is slightly < Z_1
    (c) Z_2 is much < Z_1
    (d) Z_2 is much > Z_1

19. Carbon particles are embedded in a thick alumina film made by CVD. The volumes of sample from which different K x-rays are generated are V_C, V_O and V_{Al}.
    (a) $V_C > V_{Al} > V_O$
    (b) $V_{Al} > V_C > V_O$
    (c) $V_C > V_O > V_{Al}$
    (d) $V_{Al} > V_O > V_C$

20. If spatial resolution is the critical requirement for x-ray microanalysis, then one should use:
    (a) a very small probe size
    (b) an accelerating voltage just larger than the critical one for x-ray emission
    (c) " " " much larger " " " "
    (d) a tilted sample