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# SPRING COLLEGE IN MATERIALS SCIENCE ON "CERAMICS AND COMPOSITE MATERIALS" (17 April - 26 May 1989)

ELECTRON MICROSCOPY (Lectures I, II and III)

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These are preliminary lecture notes, intended only for distribution to participants.

#### General Capabilities of Electron Optical (and related) Instruments

Synopsis: Imaging methods; projection, lenses or mirrors, scanning. Imaging signals; reflected, transmitted, stimulated emissions, absorbed currents. Electron energy loss analysis. Magnification, Resolution & Contrast. Specimen requirements and preparation techniques.

#### Imaging Methods.

We can distinguish three important classes of imaging method:

Projection - straight line paths from object to image; two examples (a) X-ray topography & (b) field ion microscopy.

Lenses (mirrors) - conventional optics of microscopes and telescopes; in the present context this is the format of the <u>Transmission Electron Microscope</u>.

Scanning - simultaneous scanning of the object by a probe and of the output device, where the amplitude is modulated by a signal resulting from interaction of the probe with that object. This the format of the Scanning Electron Microscope.

These three classes are illustrated in fig. 1.1.

#### Imaging Signals.

There are many signals which may be used to form the image, but they fall into four major types:

**Reflected** – the primary beam interacts with the specimen and the reflected portion is used to form the image.

Stimulated - some secondary emission is monitored.

Transmitted - the primary beam is used after it has passed through the specimen.

**Primary "current"** - the size of the primary beam itself is monitored (e.g. scanning tunnelling microscopy).

These four types are indicated in figure 1.2. labelled R. S. T & P. respectively.

The table below gives some examples of microscope techniques:

Imaging technique	Imaging signal	Microscope acronym	Imaging resolution	Analytical range
Projection	P or R	PIN	0.3 nm	-
P	P or S	Atom probe	0.3 nm	all Z
Scanning	\$	SEM - sec	3 nm	-
\$	R	- b/s	10 nm	3 - 103
\$	5	- Auger	50 nm	3 - 103
8	S	- X-ray	1 µm	5 - 103
8	S	- CL		
\$	R	SAM (acoustic)	5 µm.	
S	P	STH	0.1 nm	-
8	S	SIMS		
S	T	STEM	0.2 nm	-
Lens	T	TEN	0.2 nm	-
L	T	- EELS		2 - 103
L	S	- X-ray		5 - 103

The signals available for detection in an electron beam instrument are illustrated schematically in figure 1.3. In many cases the instrument takes its name from the detected signal, rather than from incident beam.

The analytical capabilities of the techniques are provided by the detection of signals which depend on the atomic number of the specimen material. For secondary emissions the signal detected is of a characteristic wavelength (X-ray or CL) or energy (Auger), or a band of energies (back scattered). In the case of *Electron Energy Loss Spectroscopy* (EELS) it is the <u>loss</u> of energy which is characteristic of the element concerned.

### Magnification, Resolution & Contrast.

These three terms are related, and sometimes confused. Each will be dealt with in more detail in subsequent lectures, but at present a few points will suffice:

Magnification — must be sufficiently large that the detail of interest may be seen. The instrumental magnification must be compatible with (i.e. large enough to overcome the limitations imposed by) the resolution of the recording medium (e.g. photographic) or observer's eye. Maximum subsequent enlargement (e.g. photographic) is also limited by the resolution of the recording medium.

Resolution – in simple terms the closest spacing of two points in the object which may be discerned as separate in the image (not quite the same thing as the smallest object which can be seen). In any system with lenses (and also, in principle, in projection systems, too) a simple resolution limit is set by the Rayleigh criterion of "maximum of one diffraction peak at the first zero of the other" at  $0.61\lambda/\sin(\alpha)$ , where  $\lambda$  is the wavelength and  $\alpha$  the angular radius of the smallest aperture in the system – see figure 1.4.

Contrast – to be 'seen' the images of items of interest must have different final intensities. The eye detects these differences, and a formal measure is the contrast, defined as  $(I-I_b)/I_b$ , where I is from the point of interest and I<sub>b</sub> is from an adjacent (background) region. In the case of the Rayleigh criterion above, the signal drops to about 0.8 between the two peaks, a contrast of (-)20%.

### Specimen Requirements.

In order to detect the presence of the items of interest in the object, the specimen actually observed in the microscope must be in an acceptable form. For 'standard' SEM work this may merely be a size constraint — it must fit inside the specimen chamber! But for some detected signals (e.g. back scattered electrons, bse) the surface must be reasonably smooth (optical microscopy finish), while for others (e.g. Auger) the surface condition is critical (absolutely clean surfaces only). In the case of the TEM the high energy electrons (100 – 400 keV) will only penetrate through a few tens of nm of material without unacceptable energy loss/spread; thus very thin specimens are essential here.

#### Specimen Preparation for the SEM.

Techniques range from "as is" (subject to mounting on the specimen stage) and ensuring electrical contact, through coating with a conducting film (e.g. evaporated carbon, gold, aluminium, etc.) if an insulator, to careful grinding and polishing to "optical" standard for any quantitative analysis (e.g. bse or X-ray detection and measurement). The extreme case of Auger analysis requires that the specimen actually be prepared (or at least its surface cleaned) in the high vacuum system of the instrument, because adsorbed gaseous impurities will otherwise mask the desired result.

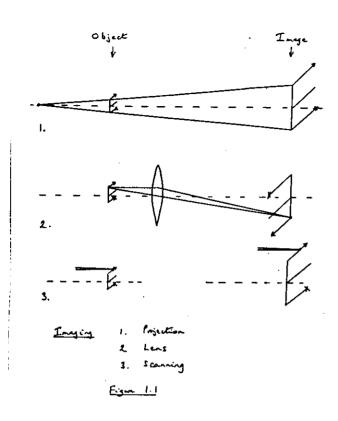
### Specimen Preparation for the TEM.

Here the thin films have been made by a variety of techniques:

- Replication of the surface of a bulk specimen by means of a thin, electrontransparent layer which may be stripped of and used as the 'specimen'.
- 2. Cleavage of the material until thin enough.
- 3. Microtomy (direct cutting of the thin sections) widely used in biology.
- Collection of small particles, which may be either naturally occurring or produced by grinding.
- Evaporation of the material of interest to deposit a thin film on a suitable substrate, from which it is subsequently removed.
- Similar deposition from chemical (or physical) reaction in liquid or vapour phase.
- Thinning from bulk with minimal (? no) disturbance to pre-existing structures in the material, by chemical, electrochemical, or ion-sputtering techniques.

It is by this last technique that most specimens are prepared to exhibit the microstructure and defect content of the material of interest. This is where much of the <u>art</u> of electron microscopy is found!

I am indebted to Prof. P.J. Goodhew (refs. + unpublished) for much of the material in this lecture.



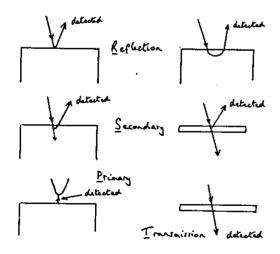
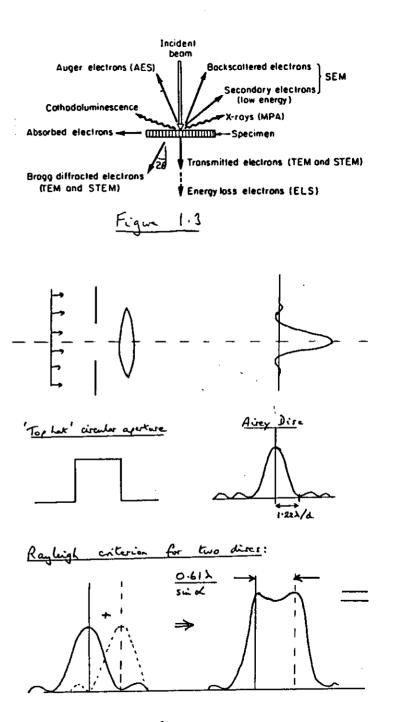


Fig 1.2

Imaging Signals



Simple Resolution Criterion

### Theory of Images

Synopsis: Resolution of images - picture points/pixels, emulsions; serial & parallel collection, rasters; noise; contrast; visibility; digital images, storage requirements, potential for image processing.

### Resolution of Images.

Since the aim in any microscope is to produce an image, or picture, we should spend a little time considering what it is that makes up that picture. Consider firstly an ordinary picture being produced by the standard photographic process. A 'normal' picture on a piece of film may be split up into a number of 'picture points' or pixels, ideally smaller than the eye can resolve at the final viewing magnification, as indicated in figure 2.1.

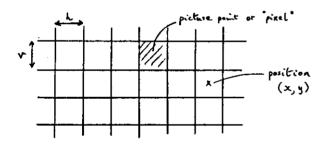
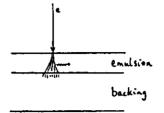


Fig. 2.1

Each picture point is lighter or darker than its neighbour over a grey-level scale, ideally of infinitesimal spacing over an infinite range. In practice we have a limited number of grey levels by emulsion grain size (grains are either developed or not) and number of grains in the picture point, so we must compromise on size of picture point versus grey level range. A reasonably fine-grained photographic emulsion can have picture points of dimensions have 25 µm (possibly as low as 10 µm), i.e. 4-6 million on a standard film or plate 50 mm wide. When printed at a reasonable maximum enlargement of 10x the (originally) 25 µm picture points begin to display the 'graininess' of the emulsion. So, without going too carefully into how many grains make up a pixel, we see

that there is a potential/actual limitation on resolution imposed by the photographic process.



A second possible limitation is imposed by a combination of 'beam spread' and X-ray or light emission from the emulsion itself or from the backing material, etc., as indicated schematically in figure 2.2.

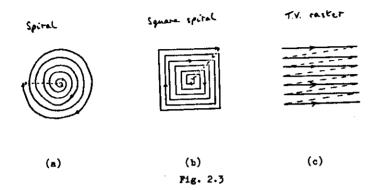
Fig. 2.2.

So take 25 µm as our 'resolution' - because what we need to be able to do is to discriminate between picture points at as small a separation as possible. The resolution in terms of the specimen itself follows trivially by the magnification! At least that is the situation for a standard, 'picture', image. But there are alternative ways of collecting images:

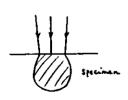
### Serial or Parallel Collection.

The normal photograph, and the image acquired in the transmission electron microscope (TEM) are both examples of what may be termed parallel collection - all picture points are recorded in parallel at the same time (or over the same period of time) during the exposure. Alternatively we may acquire the image by a scanning technique, where pixels are 'exposed' for a short time in sequence, the whole picture being built up serially over a much longer period (of necessity at least as great as the sum of the pixel exposures). Such a collection technique is used in the scanning electron microscope (SEM), where the electron probe visits each object point on the specimen in turn, while the output viewing device visits the corresponding picture point in synchronism.

In principle any scanning sequence may be adopted, provided all object points are exposed equally, e.g. in figure 2.3 (a) a spiral, or (b) a square, both working outwards from the centre, or (c) the x-y line raster of the television system.



The actual pattern traced is a compromise between a number of factors, one of which is illustrated in the figure; the beam must be 'blanked off' during the 'stray' parts of the scan. In this sense the single spiral is the most efficient, the TV raster the least. But because television technology is readily available, and also because x-à y-coordinates are easily adapted to digital technology (see below) the TV raster (whether interlaced or not) is commonly adopted.



Resolution in this case obviously depends on the final viewing device to some extent, but mostly on the probe on the actual specimen - the pixel is at minimum the area (volume) of specimen giving rise to the radiation being detected, as schematically illustrated in figure 2.4.

Fig. 2.4

Imaging theory and display modes in the SEM will be dealt with more fully in later lectures

#### Noise.

The signal in our pixel is generated by the arrival of individual electrons. If N such electrons are involved, and for simplicity each causes the same number of photographic grains to be developed (for even greater simplicity one grain each), then the variance in the image is equal to the variance in the electrons arriving. By classical statistics this variance is also N, i.e. the standard deviation or noise in the pixel is  $\sqrt{N}$ . This simple result will obviously be complicated by any variation between electrons in the efficiency of the photographic process, but it is reasonable for us to continue along these lines.

The ratio of noise to signal (the inverse of the signal-to-noise ratio) is thus:

$$\frac{\text{noise}}{\text{signal}} = \frac{\sqrt{N}}{N} = \sqrt[4]{\sqrt{N}}$$
 (2.1)

If we require this ratio to be 1% then the number of electrons per pixel must be  $10^4$ . For a typical exposure time of 5 s and pixels of size 25  $\mu m$  on the film this results in a current density, J, at the film of

$$J = \frac{10^4 \text{x} \cdot 1.6 \text{x} \cdot 10^{-19}}{5} \times \frac{1}{(25 \text{x} \cdot 10^{-6})^2} = 5 \text{x} \cdot 10^{-7} \text{ Am}^{-2}$$

with a corresponding current JxM<sup>2</sup> at the specimen. The value of J actually required at the film may be higher than this, depending on the photographic process. The (much larger) current density at the specimen is a relevant parameter in the study of beam-sensitive materials.

#### Contrast.

When we look at an image we notice <u>absolute</u> levels of darkening, <u>differences</u> between regions and also <u>rates of change</u> between areas. The absolute level depends on the signal (and development or other processing parameters), the differences are related to contrast and the rates of change to contrast and resolution. We can define the contrast, C, between a reference pixel receiving N electrons and a pixel located at some defect, which receives N+dN electrons as

$$C = \frac{dN}{N} \tag{2.2}$$

These two pixels (assumed to be magnified so as to be spatially discriminable) will be distinguishable from each other provided a visibility criterion is met:

## Visibility.

In order to be seen as meaningfully produced by the defect, the contrast C defined by eq.(2.2) must be greater than the noise intrinsic to the signal as defined by eq.(2.1) by a suitable factor, k, i.e.

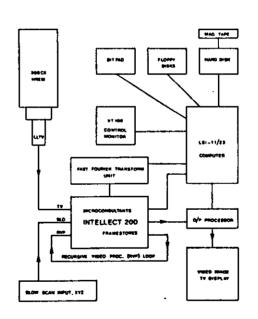
$$C = \frac{dN}{N} > \frac{k \overline{lN}}{N} = \frac{k}{\overline{lN}}$$
 (2.3)

where the factor k is usually taken to be about 5. Equation (2.3), the Rose equation, relating detectable contrast and noise, implies a minimum electron exposure necessary to detect a given contrast. Notice particularly the <u>square root</u> dependence in N acting as an impediment to reducing the detectable contrast level to lower values. Thus there is a fundamental minimum exposure, which, in beam sensitive materials, may limit the resolution attainable. Additionally, of course, if the exposure becomes too long there may also be unattainable goals set for specimen stage stability, etc.

#### Digital Images.

The terminology so far has, in fact, been deliberately cast in such a way as to permit ready transfer to digital imaging. For this transfer to occur in practice the detection system must in some way convert the incoming signal at each pixel into a number, e.g. by counting the electrons as they arrive. This could in principle be done directly just as stated, but in electron microscopes it is usually achieved via an intermediate analogue stage. In the SEM this may be via scintillator and amplifier, to analogue signal and thence back to digital via A to D converter. In the TEM a television camera may be used to view the light produced on a suitable screen and then a fast A to D converter used to produce the digitised image. Provided that each object point can then be associated reproducibly with a storage location in a suitable computer, a digital image may be built up, where a storage

location  $(n_x, n_y)$  contains the digitised intensity of the 'ordinary' image pixel at position (x,y) - figure 2.1 again. The two-dimensional coordinates are obviously well suited to handling by 2-D arrays in a computer, either in fast memory, or via access to/from a framestore.



A system for capturing and processing EM images digitally using such a framestore 18 shown schematically in figure 2.5. Images from either an SEM, or TEM via a TV camera, are digitised and added to the framestore. the output of which feeds a standard viewing monitor. Control of simple processing of both the input signal and the output signal is by means of the computer. In real time (i.e. as the picture is being captured) both of these are but rudimentary. being restricted to scaling and recursive processing (to reduce noise) at input. and acaling at output via look-up tables. These and other image processing details will be discussed later.

Fig. 2.5

Provided that the digital pixels are sufficiently numerous, e.g. >300 per TV line, then the visual impression is of a 'normal' image. In fact most of our domestic television pictures nowadays go through at least one digital framestore system between camera and our living room

(made by the same company as the system in fig. 2.5!) with a resolution of only about 750 pixels per line. So from both computing and final observation points of view a framestore of, say, 512x512 pixels = 256 k points (k = 1024 =  $2^{10}$  in computer parlance) is sensible. But is it right from the point of view of fundamental resolution?

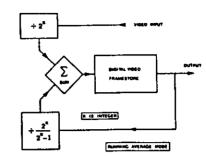
## Storage requirements for TEM digital images.

Earlier we noted 4-6 M points at least on a normal photographic plate - 16 times larger. But if we restrict ourselves to the central 20 mm of the screen (rather than the full 50 mm width) and work at a pixel size of 40  $\mu$ m<sup>8</sup> (rather than 25  $\mu$ m) we have  $(20x10^{-3}/40x10^{-6})^2 = 250~000$ . So the 'standard' 256 k framestore allows operation at a reasonable compromise between spatial resolution and storage requirement.

The other factor involved in the storage requirement (and also possibly in the quality of the A to D conversion stage) is the number of luminance levels (grey levels) associated with each pixel. For viewing the image on a TV monitor 5 or 6 bits is ample, even where colour is used (B & W discrimination is 4 bits maximum). If we were to count every electron individually into an n-bit store then N =  $2^n$  electrons could be counted, and equation (2.1) becomes

$$R = \frac{\text{noise}}{\text{signal}} = \frac{2^{(n/2)}}{2^n} = \frac{2^{(n/2)}}{2^n}$$
 (2.4)

i.e. half our bits are 'wasted' in noise, with only the more significant half of our store being meaningful. For R=10%, n=7 (i.e. 8-bit or 1-byte store is ample), while for R=1%, n=14 (i.e. 2-byte store ample). So direct counting would be feasible for R=10% or 1% by use of 'standard' stores.



In practice we do not usually count individual electrons, but in the SEM at least integrate over a considerable number before A to D conversion, but our amplifiers and conversion may introduce their own noise before that conversion. In the TEM TV camera mode our signal certainly is noisy at standard frame rate (50 fields per sec = 25 frames).

Fig. 2.6.

In either case direct integration (addition) of frames, or a 'leaky bucket' running average mode may be used to reduce the noise. The latter technique is illustrated schematically in figure 2.6.

#### Potential for image processing.

With the image stored in digital form (however fleetingly) the whole range of the available image processing techniques is opened up. These may for convenience be split into three operator time-scales, (i) image analysis at leisure, (ii) real-time (i.e. available on the microscope within a few seconds at most) for the information of the operator and (iii) automatic, feedback processing for control.

### (i) Image Analysis:

One or two examples will suffice - segmentation and area counting, displaying segmented images in false colour, particle sizes and shapes, etc., of images previously saved to disc.

#### (ii) Real-time Operations:

As already noted the output may be scaled or stretched to fill the full range of grey levels of the viewing monitor. Also, as may be seen from figure 2.5 a fast Fourier transform unit enables the transform of an image to be displayed - of interest to HREM applications to show the state of astigmatism, defocus, etc..

#### (iii) Automatic, feedback Processing:

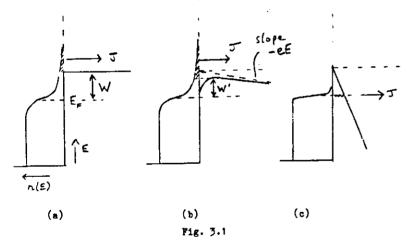
If these last operations, or similar variance calculations, are available sufficiently rapidly, then they may be used for automatic focusing and astigmatism correction. These facilities are now beginning to move out of the development phase into manufacturers' catalogues.

### Electron Optics

Synopsis: Electron emission, electron guns, optical principles, electrostatic and magnetic lenses, aberrations; condenser lens systems, objective lens, projector lenses; the complete TEM. Electron emission analysers. Scanney systems: the SEM.

# Electron Emission.

The electrons required for the microscope may be emitted from a filament either as a result of being thermally excited (thermionic emission) or by being 'sucked out' by an electric field (field emission), or possibly by a combination of both. Schematic energy diagrams are shown in the figure for (a) thermionic emission, (b) field enhanced thermionic emission and (c) cold field emission.



The actual source of the electrons is a filament 'tip' in all cases. In the thermionic case (a) a heated tungeten hairpin (or for greater emission a crystal of LaB<sub>6</sub>) provides this tip. The current density emitted from a hot wire at temperature T is given by the Richardson-Dushman equation

$$J = A_0 T^2 \exp(-W/kT),$$
 (3.1)

where  $A_0$  is a constant for the material, of order 1.2x10<sup>6</sup> Am<sup>-2</sup>. The

compromise between work function, W, and temperature of operation, T, usually means that tungsten is used, but greater current densities may be achieved using a single crystal of lanthanum hexaboride, LaB<sub>6</sub>, at the expense of a more stringent vacuum requirement and greater care in operation.

Some increase in emission may also be obtained by allowing the filament to 'see' the attractive electric field from the anode, case (b). In this field-enhanced case the work function, W (eV), is reduced to W', where

$$W' = W - e\sqrt{(eE/4\pi \xi_{\bullet})}$$
, (3.2)

where E is the field at the tip (of order 10<sup>7</sup> Vm<sup>-1</sup> for a reduction of 0.1 eV). Thus an increased current is available at the same temperature, or alternatively the same current at lower filament temperature, thus increasing lifetime.

At even higher fields, case (c), quantum mechanical tunnelling of the electrons takes place to produce even higher current densities given by the Fowler-Nordheim equation:

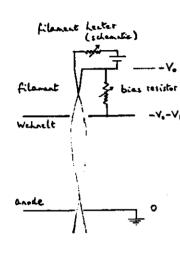
$$J = A_f E^2 \exp(-B/E)$$
, (3.3)

where A<sub>f</sub> and B are constants for the material and the field, E, is of order 10<sup>9</sup> Vm<sup>-1</sup>. Such a high field implies even more stringent vacuum (10<sup>-10</sup> torr) and a very sharp, small tip. This is usually achieved by a suitably oriented tungsten single crystal. Although the current density is high, the emission is from a very small region and thus the total current obtainable is small. Field emission sources are of use in conditions where a very small, intense source is required by the condenser lens system, such as in STEM analytical microscopes.

### Electron Guns

The principles of operation of electron guns are similar to those of a thermionic valve, namely an electron source (filament or cathode), a biased control electrode (grid) and an anode. This is shown schematically in the figure where, apart from the filament itself,

components have axial symmetry. For convenience in the rest of the microscope the anode is at earth potential, so that the filament is at the large negative potential. In fact the largest negative potential is



at the control electrode (known as the Wehnelt cylinder), the difference between that and the cathode controlling the current - a larger negative bias restricting the current passing on towards the anode. Unlike the thermionic valve, the current is not collected by the anode, but passes on through a hole at its centre. The focusing effects of the electrostatic fields (see later section on lenses) produce real images of the initial source. crossovers, at various positions along the axis. It is the final crossover which is used as the effective source for the condenser lenses (see below).

Fig 3.2 .

The detail of operation of the field emission gun is somewhat different, but will not be dealt with here for reasons of space.

### Current densities & Gun brightness.

As seen in figure 3.2 the negatively biased Wehnelt cylinder focuses the beam to a crossover of diameter  $d_c$ . Also from this crossover the beam exits from the gun as a cone of semi-angle  $a_c$ , the beam divergence. The brightness of the gun, B, (crucial in determining the resolution and visibility of the final image) is defined as the current density per unit solid angle and is given by

$$B = \frac{J_0 e V_0}{\pi kT} = \frac{4 I_b}{\pi^2 d_c^2 a_c^2} \qquad \text{A m}^{-2} (\text{steradian})^{-1} . \quad (3.4)$$

Here  $J_0$  is the current density at the filament,  $V_0$  the accelerating voltage, k Boltzmann's constant and T the filament temperature;  $I_b$  is

the beam current.

Typical operating values at 100 kV are:

filament	J <sub>o</sub> /Am <sup>-2</sup>	d <sub>c</sub> /µm	I <sub>b</sub> /pA	a <sub>c</sub> /rad	$B/Am^{-2}(sterad)^{-1}$
tungsten	2-3x10 <sup>4</sup>	30	100	0.01	4x10 <sup>8</sup>
LaB <sub>6</sub>	2.5x10 <sup>5</sup>	5	200	0.01	5x10 <sup>10</sup>
field emitter	1x10 <sup>7</sup>	0.5	1x10-82	1x10 <sup>-4</sup>	2.5x10 <sup>12</sup> .

Operating lifetimes may be approximately 100 hrs, 500 hrs and 1000 hrs respectively, under vacuum conditions  $10^{-5}$  torr,  $10^{-6}$  torr and  $10^{-10}$  torr respectively, for tungsten, LaB<sub>6</sub> and field emitter.

Note that the brightness defined as above cannot be increased in the system - as condenser lenses focus the 'probe' to smaller diameters the divergence goes up, and vice versa.

# Resumé of simple lens principles:

Radiation (be it light or electron beam) has a wave nature. For light, change in refractive index causes 'bending' of the beam, e.g.

$$\frac{\sin(i)}{\sin(r)} = \frac{n_{f} 2}{r} \text{ etc.}$$
(3.5)

The simplest way of making a lens for light is to have glass with spherical interfaces, necessarily axially symmetric:

Converging lens
of focal length 'f'

cardinal planes

Fig. 3.3

The focal length may be positive (as here) or negative (diverging lens) by different combinations of refractive indices 'n' and curvatures (concave/convex).

Often we simplify to a 'thin lens', by assuming single cardinal plane:

Thin lens

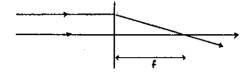


Fig. 3.4

The change in properties need not be abrupt, however, any (ideally) axially symmetric variation can have a lens effect:

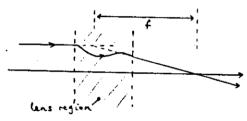


Fig 3.5

This may also be schematically shown as a 'thin lens' by extrapolation of the entry and exit beams.

Anything which has these sort of properties is potentially a lens for that sort of 'radiation'. For electron beams either electrostatic or static magnetic fields may be suitably shaped, as will be discussed below, to produce converging lenses only. Thus we only need to consider converging lenses from now on.

Magnification 'M', focal length 'f', object distance 'u' and image distance 'v' are related by standard formulae:

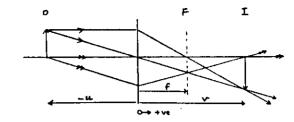


Fig 3.6

$$-\frac{1}{u} + \frac{1}{v} - \frac{1}{f} \tag{3.6}$$

$$M = \frac{v}{-u} - \frac{-f}{u+f} = \frac{v-f}{f}$$
 (3.7)

The minimum distance between object and its real image comes out as

$$(-u+v)_{m+n} = 4f ag{3.8}$$

Lenses may also be combined along the optic axis, either 'in contact' or, more usefully for electron optics, separated from each other, e.g. two thin lenses separated by 'D'.

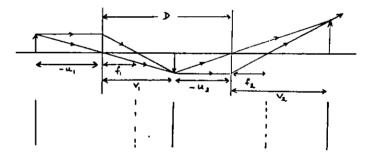


Fig 3.7

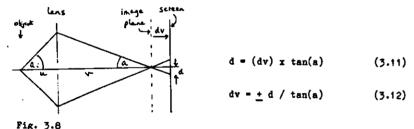
For the situation of interest we require that all 'objects' for lenses should be real. Thus

$$D > v_1 + f_2$$
 (3.9)

The final magnification, M, is given by
$$M = M_1 \times M_2 = \frac{-v_1}{u_1} \times \frac{-v_2}{u_2} \quad \text{etc.} \quad (3.10)$$

Also shown in the lower part of figure 3.7 are the conjugate planes of these two lenses (extension to more lenses 'downstream' is obvious). The solid vertical bars indicate positions on the axis at which information could be displayed (e.g. by insertion of a screen) as pure 'image', while the dashed bars indicate positions where the same information is present in 'diffraction' form. The importance of conjugate planes for the electron microscope is that anything done, e.g. insertion of an aperture, in one plane may be considered as having been done in all the planes to which that plane is conjugate (after having applied the necessary magnification and inversion factors between the planes concerned).

Depth of focus, 'dv', is concerned with the extent to which the imaging screen can be misplaced along the axis and the image remain acceptably sharp, while depth of field, 'du', is concerned with the extent to which the object may be displaced, the imaging screen remaining fixed. If fuzziness 'd' is acceptable (see lecture 2 by MJG on images) then



Relating this back to fig. 3.6 and eqs(3.6) = (3.7).

$$du = dv / H^2 = d / (H^2 tan(a))$$
 for small du. (3.13)  
=  $d/M tan(a_i)$ 

### Electron lenses.

As mentioned earlier anything which can 'bend' the electron beam is a candidate for making a lens. Electrostatic and static magnetic

fields are both suitable, and in use in practice. In both cases only positive (converging) lenses can be constructed.

#### Electrostatic lenses.

Assuming that our electrons are non-relativistic their speed, v, depends on the potential,  $\emptyset$  through which they have been accelerated. The refractive index for electron waves is directly proportional to velocity (oppositely to light waves) and thus, for an electrostatic field, to the square root of  $\emptyset$ . Thus Snell's law for light becomes Bethe's refraction law for electrons

$$\frac{\sin \theta_1}{\sin \theta_2} = \frac{n_2}{n_1} = \sqrt{\frac{\theta_2}{\theta_1}}$$
 (3.14)

Two examples of lenses will be shown, one asymmetric and the other symmetric.

### Asymmetric cylinder lens.

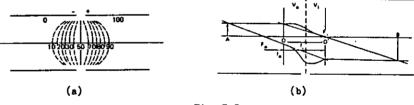


Fig. 3.9

Here two tubes at different potentials in close proximity along the axis produce a varying refractive index in the 'fringing' region at their junction - see equipotential lines sketched in figure 3.9(s). The

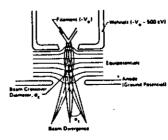
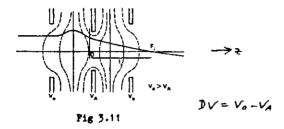


Fig. 3.10

exact focusing effect depends on the direction of travel, but is as shown schematically in figure 3.9(b). As drawn this lens is not actually used directly in the microscope (used in cathode ray tubes), but the lens action of the electron gun itself is somewhat similar as may be seen from figure (3.10) at the left. The

focusing action of the asymmetric plates (Wehnelt and anode) produces the first crossover inside the gun.

# Unipotential (Einsel) lens.



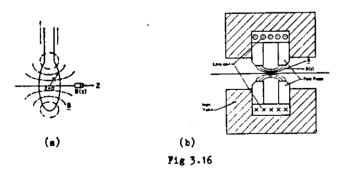
The unipotential lens is symmetric, having a unique focal length, f. of the form

$$1/f = (3/16) \int (DV)^{-2} (d\phi/dz)^2 dz$$
 (3.15)

which is independent of e/m. This fact makes the electrostatic lens of particular interest in ion microscopy, where magnetic lenses, which vary with e/m, are too weak.

#### Magnetic leases.

7.12



Field lines for two simple geometries are shown schematically in the figure. In figure 3.16(a) a single loop produces a varying field along its axis and, more importantly a diverging field with components normal to the axis increasing with distance away from that axis. The force on a moving charged particle (Lorentz) is perpendicular to both the magnetic field and the direction of movement, which results in a focusing action. The strength of this action varies with the induction, B, so soft ferromagnetic polepieces are used to concentrate the field as in (b).

A further, and this time inconvenient, effect of the mutual perpendicularity of the force, movement and field is that there is an additional result - the whole imaging system appears to rotate as the beam passes along the axis. Some impression of this may be gained from figure 3.17 below.

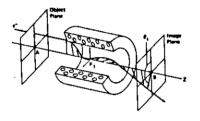


Fig. 3-17

The two lens parameters of most interest are its focal length, f, and the total angle of rotation, 0, as a function of the axial magnetic induction, B<sub>e</sub>. These are given by

$$(1/f) = (D/mE) \int_{\mathbb{R}}^{2} dz$$
 (3.16)

and

$$\Theta = (C/mE) \int_{B_{\mathbf{z}}}^{B_{\mathbf{z}}} d\mathbf{z}$$
 (3.17)

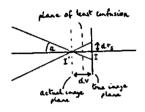
where D & C are constants and E is the energy of the electron. Notice that the focal length is directly proportional to the particle mass.

### Information transfer.

In mathematical form the relationship between 'images' and 'diffraction patterns' may be considered as a set of Fourier Transforms - the action of the lens from its object plane '0' to its focal plane 'F' is a forward FT, followed by a second forward FT in the space to the

image plane 'I'. The quality of the lens (e.g. aperture size, perfection of the lens) may thus be expressed in mathematical terms as modifications to the Fourier transform (as used in multislice calcualtions of high resolution images, for example) or in conventional optical terms as aberrations. Only three of these aberrations will be considered here as examples, and only in the most important lens, the objective lens.

# Spherical Aberration.



The axial change in focus for beams close to the axis, but inclined to the axis at an angle 'a' turns out to be

$$dv = -c_{\rm g} a^2$$
, (3.18)

where C<sub>g</sub> is termed the <u>spherical</u> aberration coefficient. Hence

Pig. 3.18

fuzziness 'drg' in the true image plane of

$$dr_a = C_a a^2 \tan(a). ag{3.19}$$

If 'a' is sufficiently small (as is the case in electron optics), then this becomes

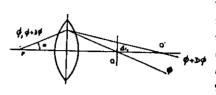
$$dr_a = C_a a^3$$
 (3.20)

In practice an operator is likely to refocus to the plane roughly mid-way between the two image planes in the diagram, to the 'plane of least confusion', which modifies the detail of the numerical factor in eq.(,), but the principle is maintained.

Again, like the focal lengths dicussed in the section on electron lenses, it is not possible with static fields (whether electrostatic or magnetic) to produce negative spherical aberration coefficients. Thus the best that can be done is to minimise the value of C<sub>S</sub> by design and manufacture and then to take its effects into account in the interpretation of our images.

7 44

### Chromatic Aberration.



When electrons leave the specimen with different energies, related to potentials,  $\emptyset$  and  $\emptyset$  +D $\emptyset$ , they are brought to focus at different points on the optic axis, as shown in figure 3.19 at the left. The result is a disc of radius drc given by

fig. 3.19 
$$dr_{c} = C_{c} = \frac{D\phi}{2} , \qquad (3.21)$$

where  $C_{\rm C}$  is defined as the chromatic aberration coefficient. As with spherical aberration it is only possible to minimise the effects of chromatic aberration by (i) minimising the energy spread in the incident beam and (ii) (if possible) losing as little energy as possible in passing through the specimen. This second condition implies that chromatic aberration may limit resolution except for the very thinnest of specimens.

### Astigmatism.

So far we have assumed accurately axially symmetric properties to the lenses. The first effect of failure in that condition, whether by inaccuracies in lens manufacture or by unexpected internal or external fields, is the production of astignatism. The lens has different effective focal lengths for paraxial rays in the two principal asymmetry planes as shown in figure 3.20.

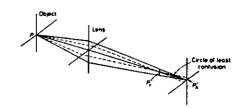


Fig. 3.20

Again the circle of least confusion is indicated mid-way between the two principal image planes - indicating where an operator would actually 'focus'. Correction of astigmatism is in principle straightforward - microscopes are provided with field coils at suitable positions along the optic axis which produce (as accurately as possible) the opposite astigmatism to that present so as to 'cancel it out'. In practice, of course, there is quite an art to that procedure, particularly at the highest resolution! Unlike spherical and chromatic aberrations, astigmatism is thus correctible.

All the other optical aberrations (distortion, etc. as in fig. 3.21 below) are present in the electron microscope, but are less important than those discussed above.

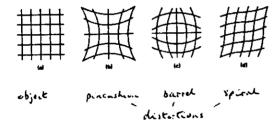
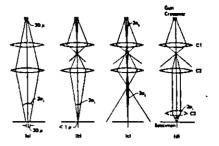


Fig. 3.21

#### Condenser Systems.

The electron gun produces a 'source' of electrons (actually a crossover point) which is used by the condenser lens system to illuminate the specimen. The condenser lenses are required to tailor this illumination to the desired properties, small area irradiated, small divergence, etc.. Possible configurations of single, double and even triple condenser lens systems are illustrated in figure 3.22, where a source crossover of 30 µm is assumed. For a single condenser (a) placed mid-way between source and specimen the minimum size of the illuminated area is also 30 µm, when the convergence angle a<sub>1</sub> is defined by an aperture placed in the condenser (C2). If this convergence is to be reduced (as it must be for high resolution imaging) the lens must be

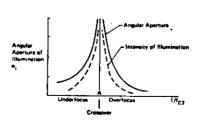


defocused, which results in a rapid decrease in the intensity as the illuminated area increases in size. Greater flexibility is introduced by having two condenser lenses. The first lens, C1, is strongly excited, producing a much reduced 'source' for C2, resulting

Fig. 3.22

in a much smaller minimum area of illumination when C2 focuses it on to the specimen as in (b). By underfocusing C2 a reasonably-sized area may be illuminated with smaller convergence than in (a), while by overfocusing C2 almost parallel illumination may be achieved as in (c). Finally we should in principle always take account of the fact that the part of the objective lens field before the specimen (see next section) inevitably acts as a further condenser lens to some extent. This effect, illustrated in (d), is used in 'condenser/objective' systems to produce the extremely small 'probes' required for microanalytical purposes.

For the double condenser case, (b)  $\hat{a}$  (c) above, the divergence,  $a_i$  depends on both C1 and C2; for small  $a_i$ , C1 must be strong (to produce a small crossover for C2) and C2 adjusted to have its crossover at or below the specimen, depending on the lowest illumination level required



to view the specimen. The illumination intensity varies as the square of the divergence a<sub>1</sub> and thus reduces very quickly as C2 is defocused in either direction. This is shown schematically, for a particular value of C1 in figure 3.23. The minimum divergence is seen to be for C2 overfocused, as mentioned earlier with reference to fig. 3.22(c).

Fig. 3.23

The total current passing through the condenser lenses is affected by apertures placed in both C1 (usually of fixed size) and C2 (variable

- current proportional to square of its diameter), as well as by the strength of C1 (reducing as its strength is increased).

For alignment purposes it is also necessary for the condenser lens systems to contain sets of alignment coils. Although it is in principle possible to manufacture the system sufficiently accurately to ensure exactly axial illumination for 'bright field' operation, it is also essential to be able to tilt the illumination to enable diffracted beams to pass through the objective lens axially for high resolution 'dark field' operation. Thus most microscopes are fitted with two independent sets of alignment controls (bright & dark field), which control both angle of incidence and exact positioning of the illumination.

### Objective lenses.

The most important lens (in governing the ultimate resolution) is the objective lens. As noted above part of the effect of this lens is that of an additional condenser lens, which should always be taken into account. However, for most purposes we may consider the condenser and imaging properties of the lens separately. Thus in this section we concentrate on 'imaging' in the most general sense.

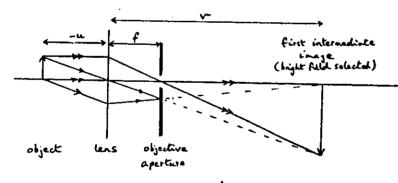


Fig. 3.22

In figure 3.22 we see that the objective lens produces both an inverted image (in its image plane) and a diffraction pattern (in its focal plane). Typical values are focal length, f=2 mm, object distance, u=2.1 mm, giving an image distance, v=42 mm and a magnification, M=20x. The angles, Ø, involved in diffraction of electrons are of order 0.01 rad (half degree) and thus lateral distances in the focal plane of

shown in the figure, must be of this diameter in order to discriminate between the incident beam direction (the optic axis here) and any diffracted beam. As noted earlier image and object planes are conjugate. Thus an aperture placed in the image plane can be thought of as equivalent to an aperture placed physically upon the specimen. This is indicated in the figure; after allowing for magnification and inversion the area selection aperture of diameter d defines a region d/M of the apecimen, e.g. d=50 µm, with M=20x, defines 2.5 µm. As far as any later lenses are concerned an opaque aperture (opaque apart from the hole, that is!) defines that area as the only part of the specimen which exists, and this applies to both image and diffraction information. Thus the aperture is used for selected area diffraction.

## Selected Area Diffraction.

As shown in figure 3.23'below the diffraction pattern 'seen' by any later lenses in the microscope can only have arisen from the area of the specimen selected by the aperture - hence the term selected area diffraction. The same applies, of course, to the image 'seen' by the later lenses. Thus, if these later lenses can be focused successively on the image, to view the area selected, and then on the diffraction pattern in the focal plane of the objective lens, to observe the pattern itself, diffraction information will be displayed from a very small area over which the operator has complete control.

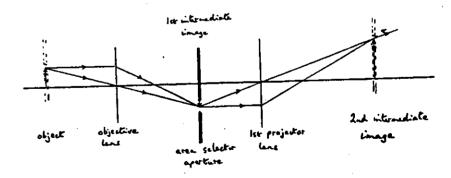
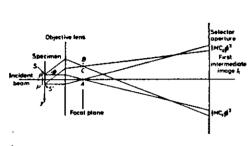


Fig. 3.23



As noted earlier, spherical aberration produces a 'smearing' of dr<sub>a</sub> in the focus of a cone of radiation of semiangle a (see eq. 3.20). For a beam at a particular angle p this is equivalent to a lateral displacement of the region to which the aperture is conjugate. This displacement, illustrated in figure 3.24, differs with diffraction spot used, being zero for the beam on the

Fig. 3.24

optic axis. For typical low order diffracted beams from metal crystals the displacement in the specimen plane turns out to be of the order of a hundred nm - see exercise - and this should be taken as the accuracy of selected area diffraction.

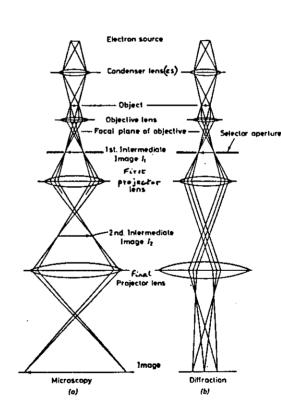
# Projector lenses.

The image produced by the objective lens and the diffraction pattern in its focal plane are still very small. To further magnify either (and to make the switch between the two feasible) a number of projector lenses is required. If each lens magnifies the image by about 20x then three further lenses would produce a total instrumental magnification of approximately (20)<sup>4</sup> = 160 000x and this value is typical of a high resolution instrument.

Thus, if the first projector is focused on the image plane of the objective lens, this high magnification image is produced, the exact magnification depending on the detail of the strengths of all the projector lenses. If, alternatively, the first projector lens is weakened so that it is focused on the focal plane of the objective, a magnified diffraction pattern is obtained, the magnification, more normally considered as an effective camera length. From an objective lens of focal length 2 mm a total magnification of 50x in the projector system yields a final camera length of the required value, approximately 1 m.

As noted earlier, in eq (5.12), the depth of focus will be large if the angular aperture, a, involved is small. In the TEM we are dealing with aperture angles as small as 10<sup>-2</sup> (objective aperture radius/focal length at most). This results in a depth of focus sufficient to include the final viewing screen (whether flat or lifted for viewing), a photoplate below, or a transmission viewing screen even further below, all 'in focus' (but with different magnifications) at the same time.

## The complete TEM.



Putting all the items in sequence vields the complete TEM. illustrated in figure 3.25. For simplicity single condenser stage is drawn, and only projector lenses are included. At the left in (a) the first projector lens is set for microscopy. i.e. the final screen is conjugate with the 1st intermediate image. hence with the specimen. On the right, in (b), the first projector has been weakened so as to make the screen conjugate with the focal plane of the objective so as to display the diffraction For simplicity the condenser and objective apertures have been omitted.

Fig. 3.25

#### Electron Energy Analysers.

The basic principle of an electron-energy analyser is to use some dispersive property to 'spread out' the electrons spatially according to their energies, and then to measure the number of electrons of a given range of energies either (a) – serial collection – by placing an aperture (slit) to allow through only those of that energy range, or (b) – parallel collection – by having a position-sensitive detector to measure the electron flux in each energy range. Either electrostatic or magnetic fields may be used to spread out the electrons, but in the case of the TEM most systems are now magnetic. An example is shown schematically in figure 3.26.

Неге magnetic induction. B. in the prism. perpendicular to the plane of the paper, causes electrons to travel along arcs of circles, radius R. because of the Lorentz force F = e wxB acting on them as they travel with velocity v. radius R depends on the kinetic energy of the electron through

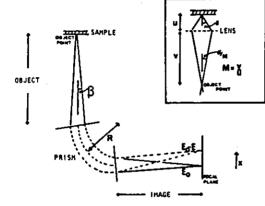


Fig. 3.24. The electron ray paths through the magnetic prism spectrometer showing the object and image planes and the direction of dispersion. The inset shows how the use of a lens to couple the analyzer to the microscope reduces the angular divergence of the beam passing into the spectrometer.

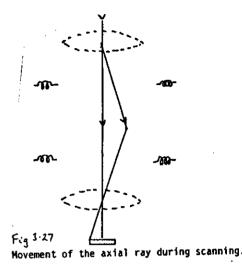
$$R = \underline{n} \cdot \underline{y} = 1 \cdot (\underline{2n} \cdot \underline{x})^{2}$$
  
eB B ( e )

and hence the position, x, of arrival of different energy electrons in the focal plane.

### Scanning systems.

For scanning in general, it is possible to produce the necessary relative movement of the probe and specimen, while maintaining identical optical properties, by moving either the specimen (as in the scanning optical microscope, SOM) or the lens (as in some versions of the scanning acoustic microscope, SAM). Such mechanical movements are of necessity slow, so, in the case of the SEM the beam is scanned by the necessary small amounts by suitably placed deflector coils. Thus a focused electron probe may be moved with almost no change in the optical properties. A single set of

coils (four coils making up a set, one each side of the optic axis (z-direction) for each of the x- and ydirections of scan) would work, but it is usual to have two sets as indicated schematically in figure 3.27. Note that the beam changes both its position and its angle of incidence as it is scanned, the angular variation being greater the greater the magnitude of the positional displacement. It is possible to maintain a fixed angle of incidence in a three-set coil system, but this complication is usually not warranted.



#### The Scanning Electron Microscope.

The complete electron optical layout of the SEM is shown schematically in figure 3.28 - to be discussed again in lecture 8.

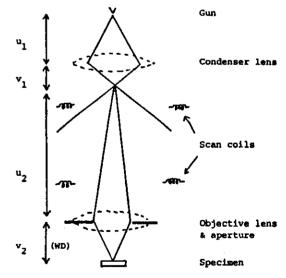


Figure \$.28 Ray diagram of SEM