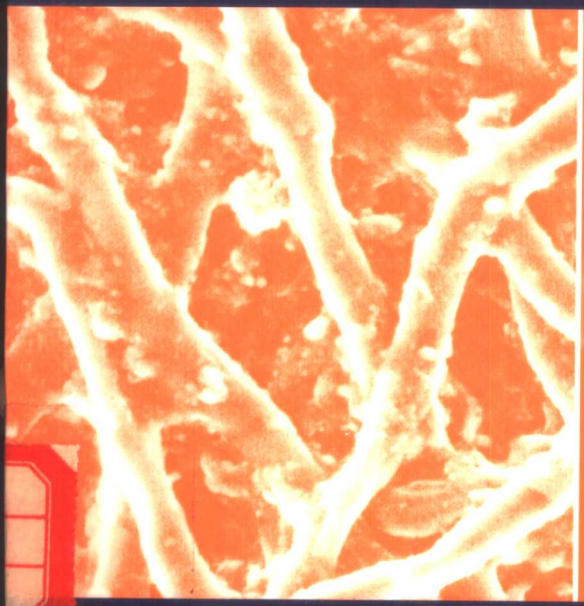
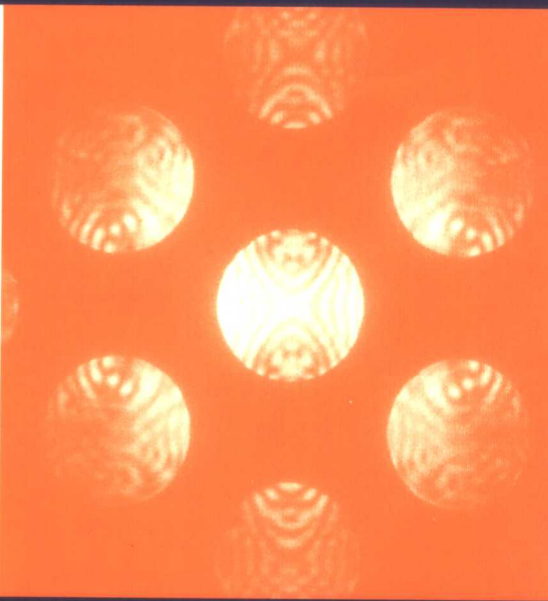
ELECTRON MICROSCOPY AND ANALYSIS

THIRD EDITION

Peter J. Goodhew
John Humphreys
Richard Beanland





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Acronyms

AEM	Analytical electron microscopy
AES	Auger electron spectroscopy
AFM	Atomic force microscopy
ALCHEMI	Atom location by channelling enhanced microanalysis
APFIM	Atom probe field ion microscopy
CBED	Convergent beam electron diffraction
CBIM	Convergent beam imaging
CTEM	Conventional TEM
EBSD	Electron back scattering diffraction
ECP	Electron channelling pattern
EDX (or EDS)	Energy dispersive X-ray analysis
EPMA	Electron probe microanalysis
ESCA	Electron spectroscopy for chemical analysis (= XPS)
FEG	Field emission gun
FIB	Focused ion beam
FIM	Field ion microscopy
HOLZ	High order Laue zone
HREM	High resolution electron microscopy
HVEM	High voltage electron microscopy
LACBED	Large angle convergent beam diffraction
LEED	Low energy electron diffraction
LEEM	Low energy electron microscopy
LIMA	Laser ionization mass analysis
MFM	Magnetic force microscopy
MS	Mass spectrometry
PEELS	Parallel electron energy loss spectrometry
POSAP	Position sensitive atom probe
RBS	Rutherford backscattering
REM	Reflection electron microscopy
SACP	Selected area channelling pattern
SAD	Selected area diffraction
SAM	Scanning Auger microscopy/Scanning acoustic microscopy

SEM	Scanning electron microscopy
SIMS	Secondary ion mass spectrometry
SPM	Scanning probe microscopy
STEM	Scanning transmission electron microscopy
STM	Scanning tunnelling microscopy
TEM	Transmission electron microscopy
TOFSIMS	Time of flight SIMS
UPS	Ultraviolet photoelectron spectroscopy
WEDX	Windowless EDX
WDX	Wavelength dispersive X-ray spectrometry
XPS	X-ray photoelectron spectrometry (= ESCA)
ZAF	Atomic number, absorption and fluorescence correction

Preface

It has been gratifying to discover in how many countries of the world the first two editions of this slim textbook have been read. It is now twelve years since the second edition was written in 1987 and this has been a period of rapid development in both electron microscopy itself and in related techniques. Many of the most visible developments are associated with the introduction of computer control into microscopy – it is now commonplace to find an SEM driven from a keyboard or mouse rather than from dedicated knobs on the instrument fascia. Other developments have only become feasible since the advent of cheap high power computing; confocal light microscopy and scanning probe microscopy are both good examples.

In writing this third edition we have been conscious that the basic principles of SEM and TEM have not changed, whereas the outward appearance of many microscopes has. During the 1980s scanned probe microscopies have also grown up. They are no longer specialized laboratory instruments but some of them (e.g. AFM) have become off-the-shelf everyday imaging tools. We have tried to reflect these changes without removing the material which leads to fundamental understanding of microscopy itself.

The book now covers a wide range of topics and it is a pleasure to acknowledge the help of many colleagues who offered help in particular areas, especially Dr Helen Davock who provided several of the illustrations and Anne Beckerlegge who imposed order on our chaos.

Peter J. Goodhew, John Humphreys and Richard Beanland
Liverpool, Manchester and Towcester

Contents

Chapter 1

Microscopy with light and electrons

1.1 Introduction

A microscope is an optical system which transforms an 'object' into an 'image'. We are usually interested in making the image much larger than the object, that is magnifying it, and there are many ways in which this can be done. This book deals with several sophisticated techniques for magnifying images of very small objects by large amounts, but many of the principles involved are just the same as those which have been developed for light microscopes over the past 400 years. The concepts of resolution, magnification, depth of field and lens aberration are very important in electron microscopy and so we deal with them in this first chapter in the more familiar context of the light microscope. When we consider electron microscopes in later chapters it will be found that instead of becoming more complicated, many areas of the subject become simpler because we are dealing with electrons rather than light. Thus although apparently more complex, and certainly much more expensive, electron microscopes are almost as easy to understand (in principle) as their humble stable-mate, the magnifying glass.

The techniques which are given the most detailed coverage in this book are scanning electron microscopy (SEM), transmission electron microscopy (TEM) and the analytical techniques which are made available by using them. At the simplest level an SEM can be thought of as providing images of external morphology, rather similar in appearance to those formed by the eye, while a TEM probes the internal structure of solids and gives us access to microstructural or ultrastructural detail not familiar to the human eye. In both cases several different types of image can be formed. Consequently it is necessary to understand not only how such microscopes work, but also how to interpret the images which they produce. This is particularly true for the TEM, and accounts for the length of Chapter 4.

It will become apparent that there is much more to both SEM and TEM than is implied by the preceding paragraph, since there is almost infinite scope for control of the imaging processes to reveal specific types of detail in a specimen. In addition, the potential for analysis of local regions of a

specimen while it is being examined in an electron microscope has been exploited very widely since the first edition of this book was published. Chapter 6 deals in some detail with several of the analytical methods such as X-ray and electron spectrometry, which are now standard facilities on a modern microscope. It is also easy to obtain structural as well as chemical information by using electron diffraction in either an SEM or a TEM and this topic is treated in Chapter 3. Altogether the modern family of electron microscopes are extremely versatile tools for revealing the nature and behaviour of matter.

Several of the concepts which are essential to the understanding of electron microscopy are common to any imaging system, and many of these ideas will have first been met in the context of the light microscope. In the remainder of this chapter we therefore deal with some general features of imaging systems and then introduce the ideas of magnification, resolution and lens aberrations as they apply to simple and familiar light-optical systems.

1.2 Methods of image formation

There are three basic ways in which an image can be formed. Perhaps the simplest to imagine is the *projection* image, of which the commonest example is the formation of shadows when an object is placed in front of a point source of illumination, as shown in Figure 1.1. The second type of image is formed by conventional lens systems, as for example in Figure 1.2, and we shall call this an *optical* image. This is not a strictly accurate term, since optical simply means 'involving light' and one of the key lessons of this book is that similar images can be formed using electrons or ions. We will try to use the terms 'electron-optical' and 'ion-optical' where they are appropriate.

Both projection and optical images are formed in parallel, that is all parts of the image are formed essentially simultaneously. However the third type of image we need to consider is the *scanning* image, in which each point of the picture is presented serially. The best-known example of this type of image is a television picture, in which several thousand picture points are displayed consecutively, but the process is repeated with such a high frequency that the image appears to the eye in its entirety.

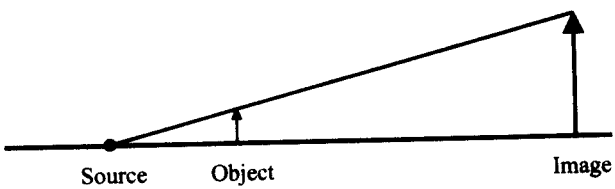

Figure 1.1 The formation of a projection (or shadow) image. Each point in the object is projected directly at the equivalent point in the image.

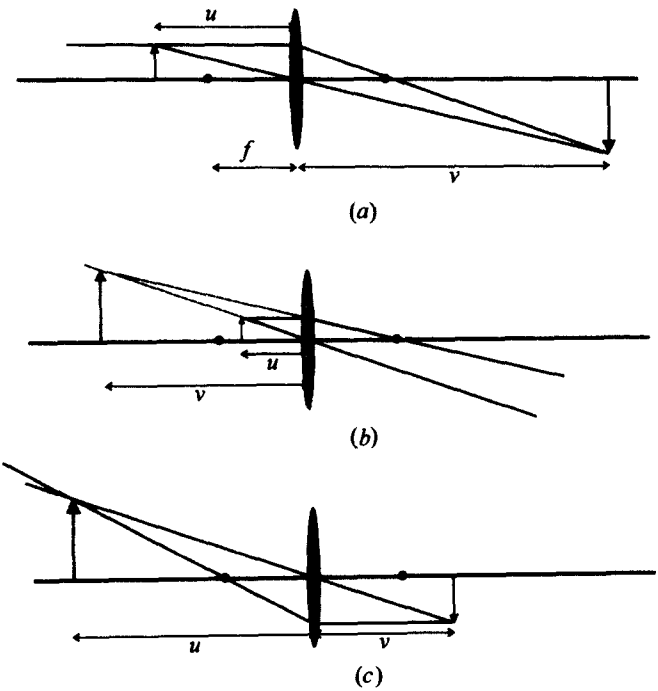

Figure 1.2 Ray diagrams illustrating the formation of an image by a single lens of focal length f. An animated version of this ray diagram can be found in MATTER: Introduction to Electron Microscopes.

In later chapters we will devote a great deal of attention to one optical technique, the TEM, and to one scanning technique, the SEM. We will also, for comparison, describe a projection technique, the Field Ion Microscope (FIM).

1.3 Pixels

One of the most important ideas concerned with images arises from the scanning image as typified by television. A European TV image contains about 700×625 discrete points and it is fairly obvious that the smallest piece of information about the image is contained in one of these *picture points*. They are generally called *pixels*, which is short for picture element. A single domestic TV picture therefore consists of more than 200 000 pixels, each of which can be of a different intensity or colour. The smallest detail which can possibly be shown in the image is a single pixel in size, that is 1/625 of the screen height for a TV image. The idea of the pixel arose from consideration of scanned images but it turns out to be universally applicable to images however

they are formed. This is particularly relevant when an image is to be stored by a computer, and again it must be broken down into the smallest necessary units of information. Domestic television is only just becoming a 'digital' medium and the signals are currently sent in analogue form. However the images produced by electron microscopes are often stored in computer memory and need to be in a digital form, that is each pixel is coded so that its brightness is represented by a single number (usually between zero and 255). Such images are often composed of a number of pixels which is a power of two, and common image sizes are 256×256 $(=2^8 \times 2^8)$ pixels or 1024×1024 $(=2^{10} \times 2^{10})$ pixels. Large amounts of computer memory are then needed to store such images. If 256 $(=2^8)$ brightness levels (known as grey levels) are permitted, each pixel takes up 8 bits of memory and a complete 1024×1024 pixel image needs $1024 \times 1024 \times 8$ bits, which for many computers is 1 megabyte (often abbreviated to 1 MB). Such an image will just fit on the conventional floppy disc of a microcomputer. However, image compression techniques are making it possible to reduce the storage requirement, often by an order of magnitude.

1.4 The light-optical microscope

Both optical and scanning types of microscope usually use lenses in some form, so we will now review some of the basic ideas of lens optics and define the necessary terminology. The simplest optical microscope, which has been in use since the early seventeenth century, is a single convex lens or 'magnifying glass'. The ray diagram for this is shown in Figure 1.2 and serves to illustrate the concepts of focal length, f, and magnification, M. The image is magnified, real and inverted if the object distance u (between lens and object) is between f and $2f$, as shown in Figure 1.2(a) and in MATTER: Introduction to Electron Microscopes. The image is erect but virtual if the object is within the focal distance (i.e. the object distance is between zero and f, Figure 1.2(b)). If an image is to be recorded on a photographic plate or viewed on a screen then it must be real, and therefore we will not be concerned with optical arrangements which give rise to virtual final images.

If the object is further from the lens than $2f$ (Figure 1.2(c)) the image is demagnified, that is the magnification is less than unity. Notice that Figures 1.2(a) and 1.2(c) are essentially the same (although drawn backwards) if we interchange the words 'object' and 'image'. This illustrates one of the important features of an optical system – its effect on the light rays does not depend on the direction in which they are supposed to be travelling. This 'principle of reciprocity' was propounded by Helmholtz in 1886 and will be of use when we consider the scanning transmission electron microscope (STEM) in Chapter 7.

The conclusions drawn above about the behaviour of a convex lens of focal distance f are summarized in the thin lens equation

$$\frac{1}{f} = \frac{1}{u} + \frac{1}{v} \tag{1.1}$$

where u is the 'object distance' (the distance from the lens to the object) and v is the 'image distance'. Figure 1.2(b) shows that, by similar triangles, the magnification M produced by the single lens is given by v/u. Substitution in the lens equation gives

$$M = \frac{f}{u-f} = \frac{v-f}{f} \tag{1.2}$$

from which it can be deduced that for large magnification $u-f$ must be small and positive. This is achieved by placing the object just outside the focal point of the lens.

Magnification of an object without severe distortion is very limited using a single lens. Strictly the image in Figure 1.2(a) should be curved so that all points on it are equidistant from the lens centre. If the magnification is high this effect is considerable and the image seen in any one plane will appear distorted. For high magnifications therefore, combinations of lenses are used so that the total magnification is achieved in two or more stages. A simple two-stage photomicroscope will have the ray diagram shown in Figure 1.3.

The first lens, the *objective*, provides an inverted image at B with a magnification $(v_1 - f_1)/f_1$ and the second lens, the *projector*, gives a final upright image at a further magnification of $(v_2 - f_2)/f_2$. The image is viewed on a screen or recorded on a photographic plate at C with a total magnification of

$$M = \frac{(v_1 - f_1)(v_2 - f_2)}{f_1 f_2} \tag{1.3}$$

If higher magnifications are required it is quite straightforward to add a second projector lens to provide a third stage of magnification.

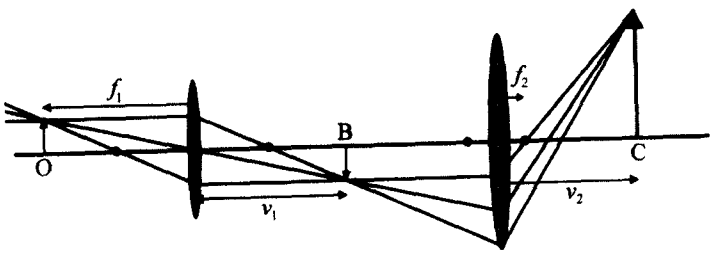

Figure 1.3 The ray diagram of a simple two-stage projection microscrope. The object is at O and the final image at C, with an intermediate image at B.

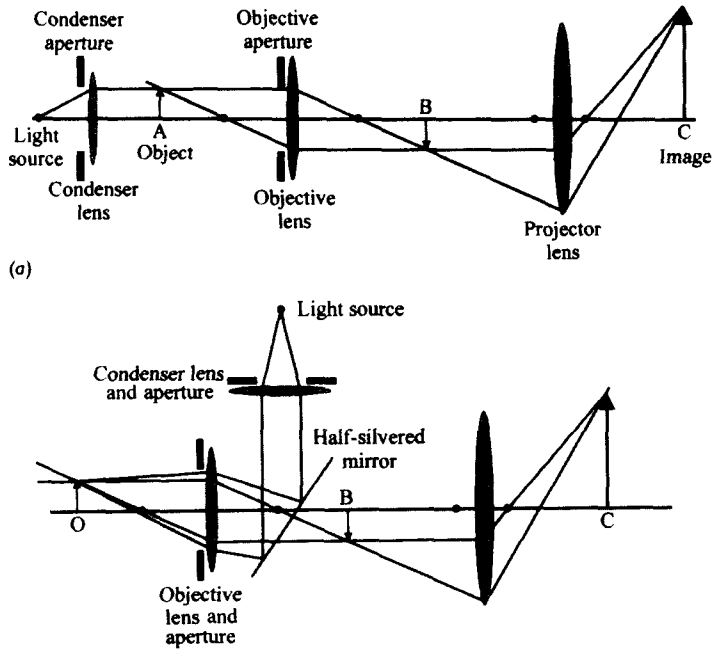

Figure 1.4 The optical systems for the two common types of projection microscope. (*a*) Transmission illumination, (*b*) Reflected illumination.

We have so far assumed that the object itself is self-luminous, and we have therefore shown the 'rays' starting at the object and ending at the viewing screen. In practice we are rarely able to look at this sort of specimen and we must illuminate it with light from a convenient source. We are now forced to consider whether the object is mainly transparent, in which case we illuminate it from behind, or whether it is opaque, in which case we must illuminate it from the front. Thus, immediately, we have a division into two classes of optical microscope: the biologist who needs to look at very thin sections of tissue uses a transmission arrangement such as that shown in Figure 1.4(*a*), while the materials scientist or geologist who needs to examine the surface structure of a solid specimen uses a reflection arrangement as shown in Figure 1.4(*b*). The same two types of electron-optical arrangement arise in electron microscopy, leading to TEM and SEM instruments. In this case both types of instrument are used in almost all fields of science.

The essential parts of any illumination system are a light source and a condenser system. The condenser is necessary to collect the light which is diverging from the source and to direct it at the small area of the specimen

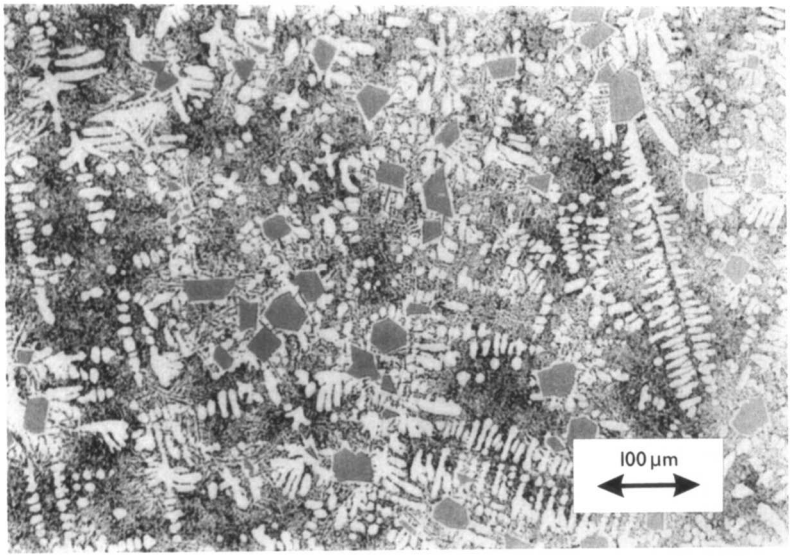

Figure 1.5 An optical micrograph of a polished specimen of Al–16% Si. (Monica Hughes, University of Liverpool)

which is to be examined. This serves two purposes; it makes the object appear brighter so that it can be seen more easily (also improving its *contrast*) and it also enables the microscopist to control the angle at which the illumination arrives at the specimen. The beam can be made to converge on the specimen or can illuminate it with parallel rays. It will be shown later that in electron microscopy the concepts of contrast and convergence angle are rather important and we will deal with them in greater detail in Chapters 3, 4 and 5.

In early light microscopes the sun or ordinary diffuse daylight was used as a source and a concave mirror was used to direct the light towards the specimen. For many purposes this is adequate but for more demanding work it is more usual to find a built-in light source and a condenser lens as shown in Figure 1.4. With the addition of two variable apertures near the condenser lens and the objective lens it is possible to control the area of specimen which is illuminated and the angular spread of the light collected from the specimen. With a well-made microscope, micrographs such as that shown in Figure 1.5 can be taken.

1.5 Magnification

In principle it is possible to make a light microscope which will produce any selected magnification. However, since for convenience the instrument should be compact, without too many adjustments, it is usual to alter f_1 or f_2 in equation 1.3 rather than v_1 or v_2. This means that in order to change the

magnification, one lens is usually exchanged for another with a different focal length, giving a limited set of fixed magnifications. The alternative is to alter the distances between all the components of the microscope and this is generally less convenient. We will see later that this problem does not arise in electron microscopes, where all the parameters are more easily adjusted.

Although it was stated in the last section that the total magnification of the microscope can easily be increased by adding additional lenses it turns out that for the vast majority of purposes the two-lens system shown in Figure 1.3 is quite sufficient. The reason for this is simple; the smallest details which can usefully be distinguished in a light microscope are about 200 nm in size (2×10^{-7} m: 1000 nm = 1 μm; 1000 μm = 1 mm). The reason for this limit is discussed in the next section but for the moment let us consider its implication. The unaided human eye can easily detect detail only 0·2 mm in size. Therefore there is very little point in magnifying the smallest details which can be resolved (200 nm) up to a larger size than 0·2 mm (200 μm). Thus any magnification greater than 1000× only makes the details bigger. We cannot make finer details visible by magnifying the image an extra ten times. An example of this 'empty magnification', as it is called, is shown in Figure 1.6. The first micrograph has a magnification of 70× and we see a lot of detail. Magnifying this several times more, to 300× reveals more detail. However a further stage of magnification to 1400× or higher shows us no more; the features are further apart but no clearer. If a large display is needed, for example in order to view the micrograph at a distance, it is more sensible to enlarge a 1000× micrograph photographically than to build a microscope capable of higher magnifications. Now it is relatively easy to provide magnifications of 1000× with only the two-lens system of Figure 1.3, for example using an 80× objective lens and a 15× projector lens. Consequently it is not necessary to build a light microscope with three or more stages of magnification, since this will not improve the resolution but will rather degrade it by introducing extra aberrations (see section 1.8). However the scanning electron microscope has inherently better resolution and it makes sense to use it at higher magnifications, as Figure 1.6 shows.

1.6 Resolution

In order to compare the electron microscope with the light microscope we need to know what factors control the resolution (often called resolving power) which we will define as the closest spacing of two points which can clearly be seen through the microscope to be separate entities. Notice that this is not necessarily the same as the smallest point which can be seen with the microscope, which will often be smaller than the resolution limit.

Even if all the lenses of the microscope were perfect and introduced no distortions into the image, the resolution would nevertheless be limited by a diffraction effect. Inevitably in any microscope the light must pass through a