The use of the

SCANNING ELECTRON MICROSCOPE

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CHAPTER 1

Introduction to Scanning Electron Microscopy

J. W. S. HEARLE

1.1. THE BACKGROUND

1.1.1. History of microscopy

The first microscopes were made in the seventeenth century. The most effective were probably those made by Leeuwenhoek—tiny glass beads mounted in a metal plate, held close to the eye, and capable of showing detail down to 1 μm on a carefully sited and illuminated object. Other types of microscopes of the same period gave a poor image, and it was not until the nineteenth century that the compound microscope achieved technical superiority over the simple-lens microscope: it was much easier to use, and by advances in design technology, the resolution was improved and aberrations were reduced. In 1876 Abbé showed by his theory of image formation that there was a limit of about 0·2 μm, set by the wavelength of light. The instrument was near its peak, and since 1900 the major advances have been mainly in techniques of use, in methods of illumination and in ways of promoting contrast.

By 1900 another possibility was available, electrons could be guided in curved paths and so could be used to form magnified images. Time was needed for the development of the technology, but in 1932 the first electron microscope was made. Contrary to the usual myth, this was based on "particle optics" and owed little to de Broglie's presentation in 1924 of a successful theory of the wave nature of the electron. Only later was this used in conjunction with Abbé's theory to calculate the limit of resolution of the electron microscope, far beyond that of the light microscope because of the much smaller wavelength of electrons. Until the 1950s development of electron microscopy was concentrated on technical improvements in the instrument; but since then advances have come more from developments in methods of use based on an understanding of the interaction between electrons and the specimen, and the methods of image formation.

It is interesting to see in Table 1.1 the similar sequence of stages in the development of each type of microscope, for the same stages are apparent in scanning electron microscopy, though some have been reduced to years rather than decades.

By 1960 the combination of light and electron microscopes covered the whole range of magnification needed to study the super-atomic world; but there were still important

limitations in their use, notably the limited depth of focus of the optical microscope and the fact that the ordinary electron microscope, which was not much use in reflection, was almost entirely restricted to the examination of very thin specimens, requiring special preparation. Scanning electron microscopy has filled these gaps, and also contributed some other new possibilities.

TABLE 1.1. STAGES IN THE DEVELOPING TECHNOLOGY OF A MICROSCOPE

Stage	Significant dates (approximate)		
	Optical microscope	Direct electron microscope	Scanning electron microscope
1. The ideal "geometrical idea" of the microscope, with no aberrations and no limit to magnification; realization limited by available technology	1600	1900	1935
2. Early design improvements	1800–1870	1932–1950	1945–1955
3. Basic understanding	1875	1935	1955
4. Commercial availability	1800	1940	1965
5. Further design improvements	1875–1900 1950–1960	continuing	continuing
6. Better techniques of use	1930–1960	continuing	continuing

1.1.2. Direct and indirect images

In an ordinary optical microscope the light passes directly into the eye and no real magnified image is formed: the image on the retina is such that the eye "sees" an enlarged virtual image. Using electrons this is not, of course, possible; and the electrons have to be focused on a screen where they stimulate light emission and so give a real, direct, enlarged image of the specimen. The same principle is used in the projection light microscope. Figure 1.1 is a schematic diagram of either type of microscope.

The best term for an electron microscope of this sort is probably a *direct electron microscope* (referring to the direct image formation) but more commonly used adjectives have been *conventional* or *traditional* (an historical distinction which will soon lose significance) or *transmission* (unsatisfactory because scanning electron microscopes are now being used in transmission).

Instead of direct image formation, it is possible, with light (as in the flying-spot microscope) or electrons, to form a magnified image indirectly by a scanning system. One way—the way in which an ordinary television system works—is to illuminate the object generally, collect the radiation, form an image and scan across it so as to pick up separately the response from each part. A corresponding signal is then transmitted on a spot scanning across a screen in register with the original scan. This method is illustrated in Fig. 1.2 (a). The magnification, or reduction, in image size is controlled by the relative areas of scan.

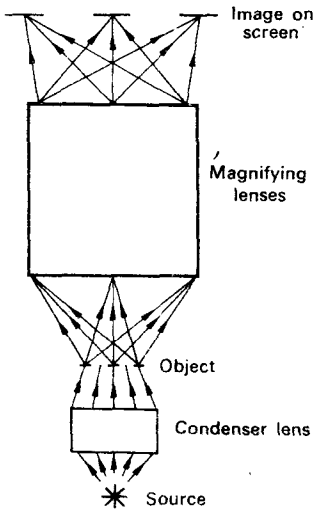


FIG. 1.1. Schematic diagram of light microscope or direct electron microscope.

For microscopy, an alternative scanning system is preferable. There is no need for general illumination. The light or electrons can be concentrated on one spot, which is then traversed across the specimen as indicated in Fig. 1.2 (b), and the complete response at each instant used to modulate the signal governing the image on the screen.

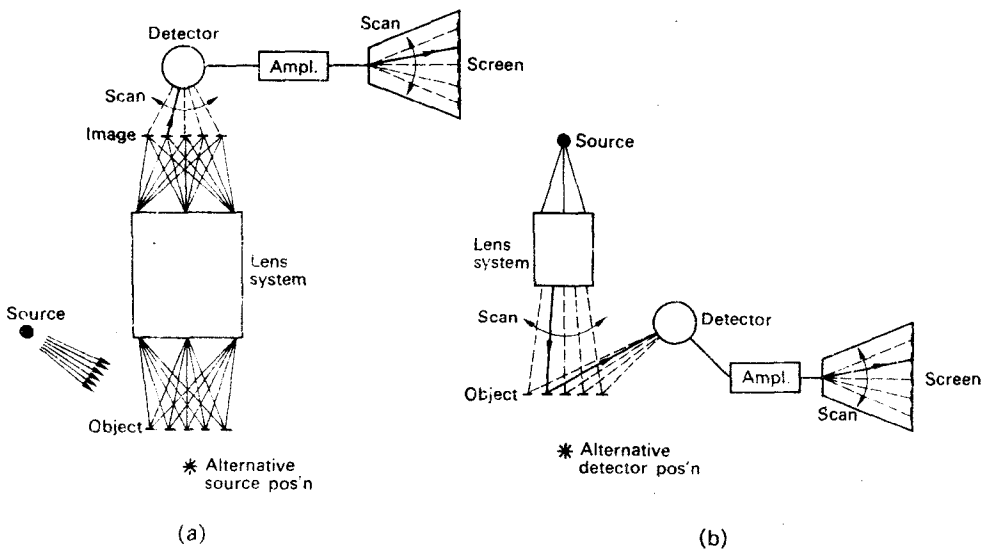


FIG. 1.2. (a) Scanning system applied to radiation which has come from specimen. (b) Scanning system applied to radiation incident on specimen. Asterisks (*) show position of source or detector for viewing in transmission.

1.2. THE SCANNING ELECTRON MICROSCOPE

1.2.1. The instrument

The idea of making an electron microscope on this principle was suggested by M. Knoll in 1935, and as a result M. von Ardenne constructed a scanning electron microscope in 1938. This, and other early instruments, seemed of little practical value: the resolution was little better than an optical microscope and recording times were long. Great improvements in design were made by C. W. Oatley and his group at Cambridge, and resolution of the order of 250 Å, similar to that of the first commercial

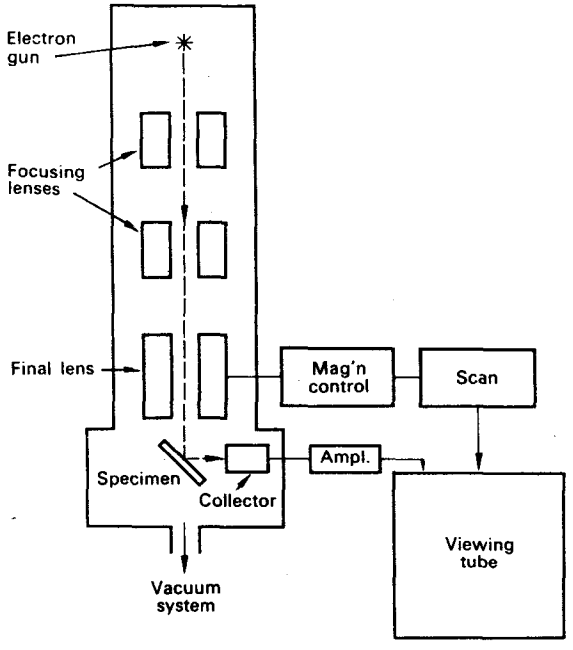


FIG. 1.3. Block diagram of scanning electron microscope.

instruments, had been achieved by 1955. This was still much worse in resolution than could be achieved with direct electron microscopes. Consequently with all the thrust in electron microscopy directed towards magnification and high resolution, the fact that most electron microscope work does not make use of the available resolution,* and that the humdrum virtues of depth of focus and convenience in use are very important, was not appreciated. Widespread exploitation of the scanning electron microscope was delayed; but, once started, it grew at an enormous rate. In 1955 there were only two or three instruments; in 1965, when the first commercial model was introduced, perhaps ten; in 1970 there were over 500 in use.

* A recent survey indicated that 60% of conventional electron micrographs were taken at less than 40,000 times magnification.

The essential features of a scanning electron microscope, indicated in Fig. 1.3, are:

1. an electron source,
2. a means of focusing a tiny spot of electrons from the source on the specimen,
3. a means of scanning the spot across the specimen,
4. a means of detecting the response from the specimen,
5. a display system, capable of being scanned in register with the incident scan,
6. a means of transmitting the response from the specimen to the display system.

Figure 1.3 indicates the commonest mode of use in which it is the electrons scattered by the specimen which are collected as the response of the specimen.

1.2.2. The practical advantages

Table 1.2 compares the three important instruments—optical, direct electron, and scanning electron microscopes. They should not be regarded as competitive, but as complementary; and, indeed, some of the most useful research results come from a combination of the use of more than one type of microscope, or from a combination with other instruments.

TABLE 1.2. COMPARISON OF MICROSCOPES

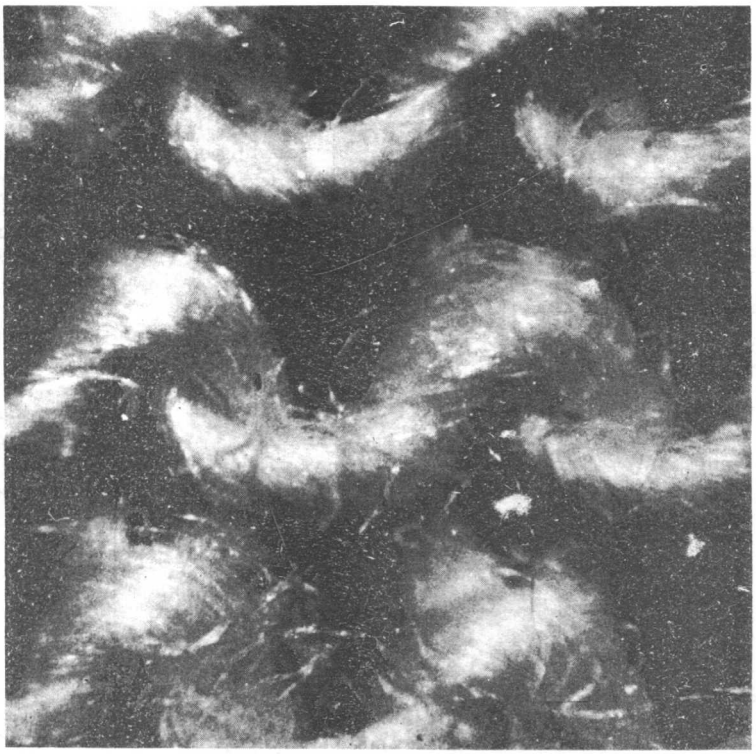
	Optical	Scanning electron	Direct electron
Resolution—easy	*5 μm*	0·2 μm	**100 Å (10 nm)**
—skilled	*0·2 μm*	100 Å (10 nm)	**10 Å (1 nm)**
—special	*0·1 μm*	5 Å (0·5 nm)	**2 Å (0·2 nm)**
Depth of focus	*poor*	**high**	moderate
Mode—transmission	yes	yes	yes
—reflection	yes	yes	*not satisfactory*
—diffraction	yes	yes	yes
—other	some	**many**	*no*
Specimen—preparation	usually easy	**easy**	*skilled, liable to artefacts*
—range and type	versatile real or replica	versatile real or replica	*only thin, or replica*
—maximum thickness for transmission	**thick**	medium	*very thin*
—environment	**versatile**	usually vacuum but can be modified	*vacuum*
—available space	small	**large**	small
Field of view	large enough	large enough	*limited*
Signal	only as image	**available for processing**	only as image
Cost	**low**	high	high

Advantages over others are indicated in bold type; disadvantages in italics.

The obvious advantages of the scanning electron microscope are:

(a) great depth of focus, as illustrated in Fig. 1.4 in comparison with the use of an optical microscope at comparatively low magnification—this is a much greater asset than might at first be imagined;
(b) the possibility of direct observation of the external form of real objects, such as complex fracture surfaces, at high magnification—thus avoiding the necessity to make thin replicas for use in direct transmission electron microscopy;
(c) the ability to switch over a wide range of magnification, so as to zoom down to fine detail on some part identified in position on the whole object;
(d) the ease of operation, and the large space available for dynamic experiments on the specimen.

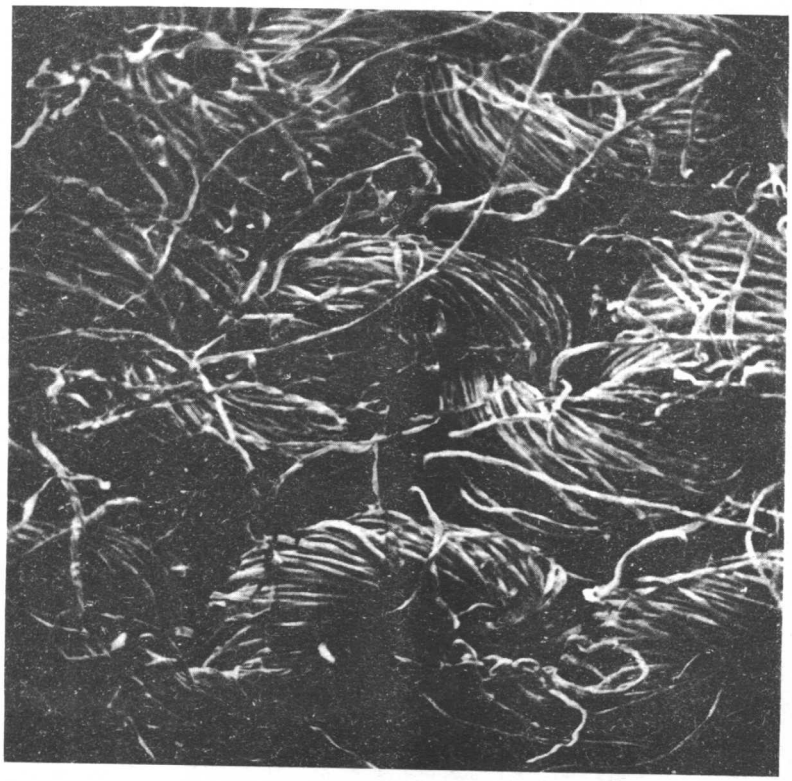
There are two other advantages which are more specialized. Firstly, an image can be formed as a result of any response of the specimen stimulated differentially by the electron spot which impinges on the specimen. The usual mode of operation is to pick up secondary electrons knocked out of atoms on the surface of the specimen, but scattered primary electrons, light emission, X-ray emission, current in the specimen, and many other responses described in the next chapter can also be used to generate



(a)

FIG. 1.4. Comparison of depth of focus in optical and scanning electron microscope. (a) Optical micrograph of knitted cotton fabric.

an image and obtain useful information about the specimen. Secondly, since the information is available in the form of an electric signal, it can be processed in various ways to present images in different forms. For example, two different responses may be added or subtracted; a contour map of image intensity may be presented; the image may be tilted or rotated; or quantitative measurements made.



(b)

FIG. 1.4 (*cont.*). (b) SEM micrograph of the same fabric.

Apart from its cost, the obvious limitations of a scanning electron microscope are:

(a) lack of the highest resolution;
(b) the vacuum environment of the specimen;
(c) the inability to show up internal detail, visible in an optical microscope;
(d) the lack of colour response, which gives a means of contrast in light microscopy, additional to intensity differences (though some analagous contrast mechanisms may be involved in scanning electron microscopy).

Some of the factors which limit the quality of a scanning electron microscope image in practice are listed in Table 1.3. Much of this book will be concerned with ways of minimizing these faults.

TABLE 1.3. FACTORS LIMITING QUALITY OF A SCANNING ELECTRON MICROSCOPE IMAGE

Spot size + penetration and spread in specimen.
Spherical aberration.
*Distortion, anisotropic distortion.
*Astigmatism, anisotropic astigmatism.
Chromatic aberration, rotational chromatic aberration.
Space-charge distortion.
Diffraction.
*Departure from symmetry.
*Distortion by external fields.
*Scan faults.
*Other design or operational faults.
Distortion by internal fields, e.g. charging on specimen.
Uncontrolled emission due to charging of specimen.
Noise.
Vibration.
Specimen damage.

* Avoided by good design and maintenance.

1.3. THE LOGIC OF SCANNING ELECTRON MICROSCOPY

1.3.1. Image formation

Image formation can be approached in various ways, and, at first sight, the method indicated in Fig. 1.2 (b) or 1.3 looks very different from what happens in other microscopes. Indeed, it was apparently a source of surprise that the image formed on the screen looked just like the object! And what is more, like the object viewed from the source of electrons.

While, in the absence of ordinary familiarity with the viewing of objects, the relations between incident radiation, object, emitted radiation and image might most logically be dealt with in other ways, it turns out that the analogy with a conventional microscope is surprisingly close, and extremely useful.

1.3.2. The analogy with the reflected light microscope

For the commonest usage of the scanning electron microscope, with an image formed by scattered electrons in order to show up topographical detail, the analogy is with a projection light microscope used to examine a specimen in reflection. Figure 1.5 is a diagram which applies to either type of microscope, with the following differences:

	Direct optical	*Scanning electron*
Radiation	light	electrons
Direction of radiation	$C \rightarrow B \rightarrow A$	$A \rightarrow B \rightarrow C$
Focusing action *or* object–image relation	$PBQ \equiv LAM$ (image formed by separation in space)	$A \equiv P$ at time t_1 $A \equiv B$ at time t_2 $A \equiv Q$ at time t_3 (image formed by separation in time)