# Modern Electron Optics and the Search for More Light: The Legacy of the Muslim Golden Age

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| Section | Title | Page |
|---------|-------|------|
| 6.1     | Introduction | 120  |
| 6.2     | Electron Optics | 120  |
| 6.3     | Parallels with Optical Microscopy | 121  |
| 6.4     | JJ Thomson and His Discovery, the Electron | 123  |
| 6.5     | The Principle of Electron-Solid Interaction | 124  |
| 6.6     | The Basic Components of Electron Microscopes | 128  |
| 6.6.1   | The Electron Source | 128  |
| 6.6.2   | The Probe-Forming Column (Electron Lenses) | 131  |
| 6.6.3   | The Detectors | 137  |
| 6.7     | Fourth-Dimension Electron Microscopy or Time-Resolved Electron Microscopy | 138  |
| 6.8     | Lensless Electron Microscopy | 139  |
| 6.9     | Application of Electron Microscopy Towards Light-Producing Devices | 139  |
| 6.10    | Conclusions | 143  |

References | 143
6.1 Introduction

This chapter is a brief survey of some of the fundamental premises of electron optics with an emphasis on electron microscopy and its relevance to modern life. The reader is introduced to the basic concept of electron microscopy and the parallels to the more familiar optical microscopes that depend upon the use of light optics. Some recent developments in the technique will be surveyed. The UN General Assembly proclaimed 2015 as the international year of light and light-based technologies, where electron microscopy has played and continues to play a pivotal role in the development of efficient, environmentally friendlier alternative light sources to the incandescent light bulb. These developments follow a scientific method of enquiry that had its roots laid down in the eleventh century by the Arab scholar, Al-Hassan Ibn al-Haytham, known in the west by his Latinised name as ‘Alhazen’. Ibn al-Haytham also discovered and correctly explained and described a puzzling effect observed in the field of optics; known as spherical aberration. The correction of this lens-related deficiency in electron microscopy is shown to have been fundamental in developing a new class of efficient Light Emitting Diodes (LED) light bulbs. The occurrence of ‘spherical aberration’ is also equally important in other imaging devices such as telescopes, and an example showing its pivotal role in the Hubble Space Telescope (HST) is subsequently demonstrated.

6.2 Electron Optics

The field of electron optics is concerned with the formation of a fine beam of electrons to use in a variety of applications as listed below. In one such relevant example; electron microscopy, the electron beam is directed to bombard a solid specimen for the purpose of learning more about the properties of such a sample, but with the particular emphasis on obtaining such information at the smallest possible dimensions. This electron-solid interaction results in an array of signals, or by-products, ranging from electrons to photons. The collection of one of these signals has often led to a specialised technique associated with such a signal. This will be explained in more details later on. There is estimated to be about 100,000 instruments around the world that use electrons to map the features of a given specimen.

Another important technical area concerns the use of a focussed electron beam, which is known as electron beam lithography (EBL). In such applications, the smallest device components like diodes and transistors; being small is essential for high-speed electronics, are drawn using small-diameter electron beams for producing the required masks used in transferring a given pattern composed of these components to a semiconductor wafer. Electron beams have also been used in direct writing on devices, in locally functionalising a surface, as well as in electron-beam-induced deposition of materials, to name but a few relevant examples.

In addition to, the use of a fine beam of electrons to map the surfaces of samples, small-diameter energetic electron beams have also been used in welding metals. The instruments employed for such applications share with the imaging instruments a great deal of commonalities, but the emphasis for welding of metallic materials is on producing an energetic spot of electrons with sufficient current to fuse together the parts of the samples being welded.

Another equally important area that also requires the production of a small-diameter electron beam, albeit not as fine as in electron microscopy, is in conventional television sets and in cathode ray tubes (CRT) used in oscilloscopes for scientific applications. Neither of these methods requires the production of a small
beam diameter, because the human eye can only resolve features of the order of
~200 micro metres (μm) (1 μm is one of a millionth of a metre whilst 1 nm is one
billionth of a metre).

As can be appreciated from the foregoing discussion, the subject of electron
beam optics is vast in nature and almost impossible to cover all its aspects in a
non-specialised book chapter such as this. I will therefore confine the material
presented to two main parts: the first is an introductory review of some of the more
widely used instruments that are employed to spatially map the constituents
(elements) of the solids under investigation. The depth of information gathered
in this exercise ranges from the top atomic layer of such a sample down to depths
of few microns below its surface. Second, I will briefly turn my attention to some of
the latest developments in electron microscopy, and specifically the techniques
that have been introduced in the last few years to advance this method. This
second part will include some important highlights of the use of electron micros-
copy in relation to light and light-related techniques as a mark of this year’s
celebration.

6.3 Parallels with Optical Microscopy

In order to better appreciate electron microscopy, it is helpful to draw a parallel
with a much older technique that is also used to image a variety of samples;
including solids and liquids; the optical microscope. This instrument is perhaps
better known and more widely used, given that many of us can claim to have used
one either during science-based education or at work. This fascinating instrument
has also over many decades seen extensive research and development to optimise it
to the state we are familiar with today. Such advances have in many ways paved the
way for the development of the more recent technique, the electron microscope,
which has somehow been easier and faster to develop, particularly in its early days.
There are, indeed, many distinctive similarities between the propagation of
photons (light) and electrons, where examples of ray (photon) optics can be
used to illustrate and understand electron optics. Parameters such as focal dis-

dtance, linear magnification and angular magnification, which define the ray optical
systems of optical microscopy, can therefore be understood.

In optical microscopy, we observe an important phenomenon: the way
photons behave as they cross a boundary between two media of differing refractive
indices causes the incident ray to change direction through a different angle from
that of incidence. This phenomenon was formulated, and has come to be known to
us, as Snell’s Law (Willebrord Snellius 1580–1626—with René Descartes having
also independently reached the same formulation in his 1637 essay, ‘Dioptrics.’).
However, recent research findings suggest that this relationship was in fact discov-
ered more than six centuries earlier than the reports of Snellius by an Arab
scientist, Abu Sa’d al-Alaa Ibn Sahl (940–1000 CE), commonly known as Ibn
Sahl, who resided in Baghdad, capital of today’s Iraq. The relatively recent
discovery of a manuscript by the French Arab historian of science, Rashed [1]
has unveiled the work of Ibn Sahl, who developed this relationship while studying
the properties of burning mirrors and lenses. Ibn Sahl showed for the first time the
refraction of light by glass lenses, as depicted in Fig. 6.1. He went on to describe
an elaborate instrument design which could be used to manufacture glass lenses of
regular and varying shapes [1]. Astonishingly, this discovery went unnoticed and
uncredited for almost 1000 years!

Snell’s law describes the relationship between the incidence angle α and the
refractive angle β and speeds v₁ and v₂ of a photon as it crosses the boundary of
two media of differing refractive indices, n₁ and n₂, respectively. This is shown
diagrammatically in Fig. 6.2 and can algebraically be stated as:
In a simplified way, the behaviour of electrons when moving through a boundary of two areas of differing potentials could also be described by a similar relationship to (6.1) above. Consider the speed \( v_{1,2} \) of an electron of mass \( m \).
and charge \((e)\) in relation to the potential acting upon it as it moves between two areas of potentials \(V_1\) and \(V_2\):

\[
\frac{1}{2} m v_1^2 + eV_1 = \frac{1}{2} m v_2^2 + eV_2
\]  

(6.2)

The electric field only applies a perpendicular force on the moving electron. This then causes only the electron perpendicular momentum to be affected; so, as it crosses the (potential) boundary, we can write:

\[
m v_1 \sin \alpha_1 = m v_2 \sin \alpha_2
\]

(6.3)

The above equation can then be rewritten to get:

\[
\sin \alpha_1 / \sin \alpha_2 = \sqrt{\frac{V_2}{V_1}}
\]

(6.4)

Equations (6.1) and (6.4) have a close resemblance with \(\sqrt{(V_1,2)}\) acting as the refractive index. Such close relationships allow one to reflect on the same optical theory of light optics, and to expect to gain similar results up to a certain extent.

This chapter, however, is mainly concerned with the electron microscope family of instruments. Therefore, whilst there has been a great deal of developments and advancements in optical microscopy in the last few decades, they will not be covered here.

### 6.4 JJ Thomson and His Discovery, the Electron

It would be unfair, if not incomplete, to discuss electron microscopy without mentioning its pivotal component; the electron. Many questions arise: so what is this electron, and how was it discovered and by whom? How do we generate electrons for use in microscopy? Are there different electron sources in use today? What are these and is this important?

The discovery of the electron in 1897 by Joseph John Thomson, widely referred to as JJ Thomson, came amidst intensive research activities in a related instrument to the present electron microscope; namely, the CRT. In Cambridge, where JJ Thomson was just appointed as a Professor of Experimental Physics at the Cavendish Laboratory (the Physics Department at the University of Cambridge, UK), he pursued his research into electrical discharges in CRT. The CRT is a vacuum tube which was made of glass in its early days, with a heated filament acting as the cathode opposite a fluorescent screen acting as the anode. The work carried out in one form of the CRT, called the Crookes tube, has shown these rays to cast a shadow on the glowing walls of the glass tubes and on the fluorescent screens opposite and, as such, move in straight lines. Being charged particles, it meant that they could also be deflected by either electrical or magnetic fields (for more details on the history of the cathode rays, see the Flash of the cathode rays—Dahl [2]).

At that time, there were many opinions on the nature of cathode rays as being waves, atoms or molecules. It was JJ Thomson, however, who carefully designed an experiment to measure the electrical charge to mass of the emitted particles, which established them as unique elementary sub-atomic particles. He called these particles ‘corpuscles’, which later on were given the name ‘electrons’ by Fitzgerald as a result of combining the words ‘electric’ and ‘ion’ [3]. In addition, such particles, which can be accelerated, have a wavelength which is shorter than that of light photons by up to 100,000 times (depending on the electron’s speed). These properties led researchers later on to accelerate a focussed beam of electrons to illuminate the surface of a specimen—as light is used in optical microscopy—and
hence develop electron microscopy as an imaging tool. As a result of this, a resolution imaging limit of about 50 pico-metres (pm) was estimated for electron microscopy, which is yet to be realised, in comparison to an upper limit of some 200 nm for traditional light microscopy. These exciting prospects have encouraged research into developing imaging tools using energetic electron beams. It was in 1931 that Ernst Ruska and Max Knoll [4] finally succeeded in demonstrating the first working electron microscope.

There are two major types of electron microscope; the first of which uses a high energy beam of electrons to penetrate thin samples, and the transmitted electrons are used to form an image of that sample in what has become known as the transmission electron microscope (TEM). The electron beam energy in this case ranges from 50,000 V up to 1,000,000 V, but most widely used electron energies in TEMs today are in the range 100,000–300,000 V. In the second type, known as the ‘scanning’ type, the incident electron beam is normally in the range 100–30,000 V. These electrons are arranged to impinge the solid surfaces, and the reflected electron signal is used primarily to map the surface topography of the said sample. This type is known as the scanning electron microscope (SEM) and is the most widely used type in industry and academia alike. The major difference between the two instruments is in the collection of the signal from each, in the TEM the signal is of the transmitted electrons through the thin sample, whilst in the SEM the signal is normally of the reflected electrons (i.e. scattered back off the sample’s surface). Both types use electrostatic and electromagnetic electron lenses to control the electron beam energy, but more importantly for the SEM is to focus it in the smallest possible spot which, when scanned, could be used to form an image of the area the electron impinges.

These electron optical lenses are analogous in function to the glass lenses of the optical light microscope. The remainder of this chapter will review the principle of the electron microscope, its major components, and provide a short overview of its applications, particularly in relation to light-related technology, before finally concluding with future trends in electron microscopy.

### 6.5 The Principle of Electron-Solid Interaction

Imagine an electron beam of an infinitesimally small diameter is incident on a solid surface, as depicted in Fig. 6.3 below. As these electrons penetrate the solid, they will interact with the sample’s constituent atoms. This interaction is referred to as ‘electron scattering’ and is further divided into two categories: (a) an inelastic scattering, where the incident electron gives up part of its energy as a result of its collision with the solid’s atoms; and (b) an elastic scattering, which causes the electron to change its direction of travel with almost no energy loss. It is the first scattering type, however, that gives rise to the signals enumerated in Fig. 6.3, whilst the second type is normally what determines the shape of the interaction volume of the incident electrons within the solid under study, as depicted in Fig. 6.4. The shape and size of the interaction volume depends on the incident electron energy, its angle of incidence with respect to the surface and on the average atomic number of the sample under study. If one concentrates on the emitted electron signal alone and plots their number against their energy, one would in principle collect a distribution similar to that shown in Fig. 6.5.

The collection of one of the signals resulting from the interaction depicted in Fig. 6.3 has over the years resulted in a specific class of instruments reflecting the type of information gathered from such interaction. For example, if one collects the resulting X-ray photons of a given element making up the solid, the collected image would be a map of the distribution of such an element in the solid, normally
to a depth ranging from ~0.05 μm to few μm depending on the incident electron energy and the sample’s atomic number. This technique is referred to as the electron microprobe analysis (EPMA) and is heavily used in material science research and applications. If, however, one uses the Auger electrons of a given element, then these would normally produce a map of the distribution of this element on the very top atomic layers of such a sample—a technique known as
Scanning Auger Electron Microscopy (SAM). Auger electrons are an alternative to X-ray emission from atoms and also, like the X-ray photons used in EPMA, are material specific, i.e. almost the equivalent of fingerprints of the elements [5]. Whilst X-rays originating a few microns below the surface could still be collected, the collected Auger electrons come from only the top few atomic layers of the solid under study (i.e. the depth of information of the X-rays could be >100 times or more greater than that of the Auger electrons). The collection of other signals would give rise to techniques associated with such other signals.

However, the main and most popular type of electron microscope is the SEM, where use of the reflected low-energy ‘secondary’ electrons, which mainly have an energy of <50 eV (refer to Fig. 6.5), is made to map the topography of the solid surface. One important application of the SEM is in the semiconductor industry, both during the development of the integrated circuits and the various electronic devices constituting it but, equally as important, during their production (i.e. in what has become known as fabrication lines). In the latter case, there are normally two types of microscope in use. The first is referred to as a Critical Dimension Scanning Electron Microscope (CD-SEM). This type of instrument is normally used on semiconductor fabrication lines for quality control, with the main function of measuring the dimensions of some components on the circuits/devices being produced. It is also interesting to note that such inspection is usually made automatically and lasts no more than few seconds per test, making the inspection of some 10–20 areas on a full 300 mm diameter wafer last no more than 1 min.

The second type is normally also used for quality control, with the main purpose being searching for ‘defects’ or foreign particles appearing on the wafer which could cause the ultimate failure of the circuit built on or next to it, for example, where a particle is found lying between two active parts of the circuit and it unintentionally connects them. These defects can be due to anything ranging from small particles that find their way into the production line as a result of poor practice to failure in procedure. Sometimes these also appear as a result of the use of poor quality or incompatible materials in the fabrication process including, for example, the ‘pure or de-ionised’ water used for washing the wafers. This class of instruments is referred to as ‘defect review SEMs’, which are normally equipped with a number of elemental/chemical detectors used to analyse such defects. The SEM is also equally vital in biological and other physical sciences, such as physics.

**Fig. 6.5** Electron spectrum of a typical distribution in energy and number of electrons that exit the surface of a solid which is bombarded by a beam of energetic electrons. AE Auger electrons, SE secondary electrons, BSE backscattered electrons.
and engineering. More discussion will be devoted to the SEM later on. In recent years the development of compact and sometimes novel X-ray and various detectors has enabled these methods to be used as add-on techniques to the electron microscope, thus enhancing the instrument’s analytical capability and widening its use.

If, on the other hand, the sample under study could be made in the shape of a thin enough section to allow a beam of energetic electrons to penetrate it and be collected from the other side, then a whole new array of signals would result in allowing one to gain more information from such a sample on atomic dimensions. This class of instruments is referred to as TEM. The working of the TEM, however, is quite subtle and different than that of the SEM. To understand the basic principle of TEM, let us investigate the fate of some energetically incident electrons on a thin sample. The transmitted electrons will pass through the thin specimen but, in so doing, will be subjected to one of three possible mechanisms:

1. To pass through with no scattering,
2. To pass through with some angular deflection (elastic scattering) and
3. To lose some energy as it passes through (i.e. inelastic scattering).

The above three possibilities are normally a function of the energy of the incident electrons and the arrangement of the sample’s atoms. How we see the varying contrast in the obtained TEM images is quite interesting though.

To better appreciate the underlying principle behind the operation of the TEM, it is useful to consider the following simple example. Imagine a hypothetical specimen made out of four regions of carbon and lead as depicted in Fig. 6.6 below (where lead is a much higher atomic number material than carbon). If 100 energetic electrons are incident perpendicularly on the surface of this sample and the transmitted electrons are classified according to them being scattered (i.e. change direction of travel) through a small angle of 0.5° or more in comparison with those which pass through without suffering any scattering, or less than 0.5°, then the number of transmitted electrons varies in terms of their scattering.

![Fig. 6.6](image-url) A hypothetical sample used to illustrate the principle of transmission electron microscopy by following the fate of 100 energetically incident electrons, and counting those that scatter through an angle of more than 0.5°. (a) carbon film 10 nm thick of randomly distributed atoms, (b) same carbon film but of 20 nm thickness, (c) 20 nm film of randomly distributed lead atoms, whilst (d) is 20 nm thick lead but of regularly distributed atoms (i.e. crystalline), (e) the objective aperture which sits below the sample in the TEM to stop electrons scattering through more than 0.5° (adopted from [7]) from passing through and hence contributing to the used signal.
angle in a very interesting way. In part (a) of the sample, nine electrons will scatter through 0.5° or more, in part (b) about 17 will scatter, while part (c) shows a much larger number of about 95 electrons scattering by more than 0.5°. However, in part (d) the number scattered depends on the angle that the incident beam of electrons makes with the sample atoms in what is known as ‘Bragg diffraction’ [6]. If a small aperture (i.e. a hole cut in a metal plate) is placed below the specimen such that any electrons scattered by 0.5° or more are stopped by this plate whilst those scattered by less than this angle go through, then a number of different ‘in-value’ electron signals could be collected below the small aperture. The position of this aperture is therefore the key part in the working of the TEM. The detectors used in the TEM also vary from principally detecting the crystallinity of the sample, via the distribution of its atoms; or they determine its atomic number, via the measurement of very small energy losses that the incident electrons suffer in passing through the thin sample.

The TEM is a more complex instrument to manufacture and operate than the SEM. Moreover, its price is normally several times more than that of the SEM. The number of TEM instruments worldwide is perhaps less than 10 % of the installed user base of SEMs. It should, however, be understood that the information gathered from either instrument normally complements the other rather than being an alternative to it.

### 6.6 The Basic Components of Electron Microscopes

Figure 6.7 depicts a schematic of the two most widely used types of electron microscope: the SEM and the TEM. The major components of either instrument could be divided into the following parts: the electron source, the probe-forming column, the specimen chamber and finally the detectors. A brief coverage of these components will be given below; however, for more detailed discussion of the modelling of these components the reader is referred to [8]. It should also be noted that most, if not all, modern instruments are computer controlled, but this will not be covered here.

#### 6.6.1 The Electron Source

The first type of electron source used in electron microscopes consisted of a heated filament made out of a thin tungsten wire, a similar material to that used in conventional light bulbs. Over the years more and more electron source types have been developed to address the fundamental problem of using the highest possible number of electrons per unit area per unit angle of emission; a concept referred to as the ‘source brightness’. The higher the brightness value, the smaller the focused point that can be formed with the same number of incident electrons. Table 6.1 below summarises the currently available and widely used types of electron sources and their relative properties. The development of field electron emitters as high-brightness electron sources in the last 40 years or so has in particular moved both types of electron microscope discussed here and other probe-forming systems closer towards realising their ultimate resolving power.

The basic principle of electron emission from the various sources is depicted in Fig. 6.8. The important property that underpins all of these is the material’s work function, a property that dictates how much energy is required to liberate an electron from its bound state in the material’s atom. For thermionic sources, electrons are liberated by heating the source to ‘boiling point’, the consequences
Fig. 6.7 A schematic of the major components of the electron microscopes (a) SEM and (b) TEM. Note that most microprobes and EBL instruments share the same configuration as the SEM.

Table 6.1 A comparison of the characteristics relevant to electron optics for some of the most widely used electron sources (York Probe Sources Ltd, http://www.yps-ltd.com/)

| Emitter type          | Thermionic | Thermionic | Schottky FE | Cold FE |
|-----------------------|------------|------------|-------------|---------|
| Cathode material      | W          | LaB$_6$    | ZrO/W (100) | W (310) |
| Operating temperature (K) | 2800      | 1900       | 1800        | 300     |
| Effective source radius (nm) | 15,000   | 5000       | 15 (*)      | 2.5 (*) |
| Normalised brightness (A/cm$^2$ sr kV) | $1 \times 10^4$ | $1 \times 10^5$ | $1 \times 10^7$ | $2 \times 10^7$ |
| Energy spread @ cathode (eV) | >0.59     | >0.50      | 0.5–0.8     | >0.23   |
| Beam noise (%)        | 1          | 1          | 1           | 5–10    |
| Operating vacuum (mbar) | $<1 \times 10^{-5}$ | $<1 \times 10^{-6}$ | $<1 \times 10^{-9}$ | $<1 \times 10^{-10}$ |
| Typical cathode life (h) | ~100      | ~1000      | >10,000     | >10,000 |
of which make the lifetime of this type of source quite limited to mostly no more than few 100 h of use.

The work function of a material is measured in units of electron volts (eV). An eV is the amount of energy gained or lost by the charge of a single electron that is moved across an electric potential of 1 V. For tungsten (W), the work function is about 4.6 eV. The work function acts as a barrier between the electrons in the metal and the vacuum outside the metal. There are a number of materials and combination of materials that have a lower work function than W. One of these is a compound known as lanthanum hexaboride (LaB₆), which has a barrier height of only 2.8 eV; and, when heated to a moderate temperature of about 1800 K, the electrons have enough energy to jump over the barrier thus enabling them to be used as an electron source. The lifetime of this type of source is of a factor of $\frac{3}{5}$ longer than the heated W filament; and their brightness is also more than $\frac{10}{5}$ higher. The vacuum environment for the operation of this source is, however, more stringent than that of the W counterpart, normally in the region of $10^{-7}$ mbar.

Field electron emission [9], on the other hand, relies on a totally different mechanism, which exploits the unique wave-particle duality of the electron (see Chap. 1 for more explanation). This wave nature of the electrons, when utilised, has allowed scientists to develop electron sources with a brightness value of more than 100,000 greater than that obtained from the early tungsten filament counterpart (see Table 6.1 above). In this method, the electron source is made of a very sharp needle, often less than 1 μm in diameter. When this is subjected to a high electric field, $>10^9$ V/m, the barrier between the electron position in the metal and that of the vacuum level becomes so thin (refer to Fig. 6.8 [10]) that electrons can tunnel through it and are liberated into the vacuum. Depending on the electron emitter’s diameter and its position relative to an electrode positioned in front of it, called the extractor, the high electric field needed for the electrons to tunnel through can be achieved by applying a moderate voltage value between the emitter and the extractor of less than 5000 V.

Figure 6.9 depicts the various materials and configurations of electron sources currently in use in electron optical instruments which give brightness values spanning a wide range, but which for brevity we will not cover here. Suffice it to say, however, that over the last few decades, field electron emitters, and

![Fig. 6.8 A simplified schematic of the electron potential energy at the solid–vacuum interface showing the modification (bending) of the energy barrier as a result of the application of the electric field. For a more detailed description of this phenomenon as applied to various sources, see [10]](image)
particularly those which are known as thermally assisted or ‘Schottky’ field electron emitters, have become so popular and relatively easy to use that at least half of the modern SEMs and TEMs are manufactured using this class of electron sources. But the heated tungsten filament, in spite of its clear disadvantages, is still expected to be with us for many years to come because of its relatively low cost and ability to function in poor vacuum conditions.

Recent advances in electron source technology are currently directed at developing small-diameter sources of novel materials in pursuit of even higher brightness. Such materials include carbon nano-tubes (CNT) and nano-rods of conducting materials in general. The CNT is a sheet of carbon atoms rolled to form a cylinder of one or several layers. The diameter of this cylinder ranges from few nm to several tens of nm (see Fig. 6.10 for illustration). However, in spite of the potential advantages of such sources in terms of their superior brightness and small source size ([14], it is expected to be sometime before we will see such a source in use due to the great technical challenges in reliably producing and using a stable electron emitter of this type.

6.6.2 The Probe-Forming Column (Electron Lenses)

The probe-forming system consists of a number of ‘lenses’ that act to form an image of the source of electrons which is used in a focussed spot of electrons. The basic underlying principle of an electron lens is a structure that controls the electron trajectory, ultimately forcing them to converge to a focussed spot. Two types of lens are used in probe-forming systems: an electrostatic one where an electric field is formed by the application of suitable voltage to a specially shaped metal structure which the electrons travel through; or a magnetic lens where an electric current passes through a coil enclosing a carefully shaped iron structure, thus producing a magnetic field that acts on the electrons in a similar fashion to the electrostatic lens. The electrons that pass through such lens structures could then change their path in a diverging or converging manner to a point somewhere away from its starting position (i.e. to form a focused image of the emitted electrons away from its starting point). Such a focused spot could then be used to scan the
surface of a solid in a raster fashion (i.e. moving the spot across the surface in a straight line and then moving quickly back to the start point and scanning across the next line down) where, at each pixel of the raster, an interaction similar to that depicted in Figs 6.3 and 6.4 takes place. Note that the focussed spot could be smaller or larger than the area where electrons are emitted from depending on the source of electrons used and the applications for which the probe is intended. Note also that the choice to use either an electrostatic or a magnetic lens, or indeed a combination of both, is largely dictated by the intended applications. However, it should be noted that magnetic lenses are more favourable for high resolution applications because of their favourably smaller aberration coefficients (see below for detailed discussion on lens aberrations). The focussed spot diameter \( d_f \) is therefore composed of a number of contributing sources where their diameters can be simply expressed as the original source of emitted electrons \( d_o \), the chromatic

**Fig. 6.10** (a) TEM image of the end-form of a CNT showing the nickel seed used to grow the CNT. (b) TEM image of the ultra-fine tip (nano-needle) of the as-grown In-doped ZnO nano-pencil with a diameter in the range of 13–15 nm. (c) SEM image showing the morphology of In-doped ZnO nano-pencils on silicon substrate prepared by thermal evaporation process. The image shows a useful nanostructure made of two parts: ultra-fine tip connected to a base of ZnO nano-rods which could be useful as an electron source. (d) Shows a CNT grown on top of a sharp tungsten emitter and the bar measure 1 μm. All of these sources are from the author’s research group at the University of York [11–13]
aberration \( (d_C) \), spherical aberration \( (d_S) \) and diffraction effects \( (d_D) \). These contributions could be added in the following manner:

\[
d_T = \left( d_o^2 + d_C^2 + d_S^2 + d_D^2 \right)^{0.5}
\]

The lenses within an electron microscope behave in a similar fashion to the glass lenses of optical microscopes and these, too, suffer from a number of fundamental properties (or deficiencies) that adversely affect the quality of the focussed image. These are referred to as lens aberrations which basically blur the focussed image, and would therefore require a correction procedure to resolve. Some of these are inherent to electron microscopy, such as the chromatic aberration, due to the nature of the emitted electrons and the type of source used having a range of different initial energies as they leave the surface of the electron source. The effect of such varying energies is that the focussed electrons would correspondingly land, for each electron energy, at a slightly different distance away from the point of emission, as depicted in Fig. 6.11a.

Table 6.1 above lists the energy spread typical of the various electron sources in use. Modern electron microscopes, particularly in the case of TEMs, have started to use an energy mono-chromator to correct for the chromatic aberration, but these are not yet commonly used due to the cost they add to the microscope. It is clear from Table 6.1 that field electron emitters have a smaller energy spread than thermionic sources and these give this class of electron sources a clear advantage for use in electron microscopy and particularly so in the case of the SEM, which only use chromatic aberration correctors in the top of the range instruments.

Another optical effect inherent in all cylindrical or spherical lenses (and mirrors) is the spherical aberration, which in the present case is caused by electrons focussing at different points, depending on how far the electrons are incident from the centre of the lens as it passes through it. The reason for this is that for each of the emitted electrons going through the lens’s hole when these are projected back would subtend a range of angles at the point of emission (the source). Again the positions of the focussed electrons would converge differently for each electron trajectory, as the electrons travel through regions of different potentials, and hence will obey the formula given above (Eq. (6.4)). As a result they will focus at a range of distances away from the point of emission. The extent of this focal distance away from the point of emission (or the ideal focal point of such a lens) is a function of the angles subtended, i.e. the size of the lens’ hole. This in turn means that the larger the aperture-hole diameter, the worse is the effect. Spherical aberration \( (C_s) \) is a feature of all round lenses which causes image distortion and limits the ultimate microscope resolving power. In practice, however, is that the various foci of the electrons past the lens will go through a minimum called the ‘disk of least confusion’ (DLC), which is normally assumed

![Fig. 6.11](a, b) Schematic diagram illustrating chromatic and spherical aberrations and the disk of least confusion (DLC)
as the focal point of the lens. The spherical aberration is schematically illustrated in Fig. 6.11b.

It is interesting to note that spherical aberration was first noted by the Arab scientist, Ibn al-Haytham (d1040) in his magisterial work on optics, ‘Kitab al-Manazer’ (see Chap. 7). Whilst this defect was known early on during the electron microscopy development, an effective resolution was only realised and proposed in 1997 (see [15] for a full account). Again, the effect of spherical aberration tends to be more obvious in the TEM case which, when corrected, allows one to resolve structures a small fraction of a nanometre. Its correction in the SEM is also equally important but only applicable in the top of the range instruments. This has in great part been due to the use of field electron emitters, which has made it easier to reduce its effect in SEMs. Figure 6.12 shows the effect of correcting spherical aberration in a modern TEM.

Spherical aberration is an effect typically found in imaging instruments, such as cameras and telescopes. Its presence compromises the quality of the resulting image with potential catastrophe, as the scientists in charge of the Hubble telescope found [17]. The HST project cost several billion US dollars and took several years to complete. The first images collected were of very poor quality, as shown in Fig. 6.13a. The fault causing such a poor image was identified to be due to spherical aberrations of one of the telescope’s mirrors and a repair mission was launched to fix it. It consisted of a number of astronauts, one of whom was an experienced experimental scientist who was able to walk in space to repair the fault. Figure 6.13b shows the effect of correcting the spherical aberration on the quality of the images obtained.

The SEM, on the other hand, has also seen a great deal of advancement to improve the quality of the obtained images as well as increasing its resolving power. This has largely been not only due to the use of Schottky and cold-field electron emitters as electron sources, both having lower energy spread and much higher brightness than its conventional thermionic counterpart, but also due to a range of efficient electron detectors that have recently been developed. As a result most modern SEMs offer high resolution imaging in the range of 1–2 nm. These field emission sources have particularly benefited the use of low voltage imaging where most modern instruments operate down to few 100 eVs and sometimes even less to obtain an image resolution of the order of few nm. This high resolution
low-electron energy mode is particularly important in imaging biological and novel and radiation-sensitive materials, as encountered in the field of nanotechnology.

By reducing the incident electron beam energy, the interaction volume between the incident electrons and the specimen also reduces and becomes much closer to the specimen surface, as seen in Fig. 6.4 above. This extends the use of the SEM to previously unchartered areas. One recently developed field of application is in imaging doped regions of semiconductors, which have traditionally been almost impossible to image using conventional high-electron energy imaging (>5 keV). This is because the current secondary electron detectors used in the SEM are incapable of resolving differences in the atomic number \(Z\) of the constituting elements of a sample of less than \(Z = 1\). However, the amount of doping material used in semiconductor devices is normally less than 0.001%; and yet by imaging with electron energy of less than, say, 2000 eV one could differentiate between regions of a sample like silicon, which are differently doped, as depicted in Fig. 6.14. Doping of semiconductors is the crucial step in making electron devices, such as the p-n junction diode; the basic building block of integrated circuits. This low-energy imaging mode is proving valuable in the fabrication lines in the semiconductor industry, where its use is in quality control. The explanation of the mechanism that gives rise to such a contrast is outside the scope of this chapter, and the reader is referred to recent articles discussing this phenomenon [18, 19].

A recent development in SEM technology to cope with the limited space available in modern laboratories is the advent of the table-top electron microscope. A number of microscope manufacturers now specialise in producing such instruments. It is estimated that many thousands of such microscopes have been produced and sold to date. It is expected that further developments will continue to be exerted in this endeavour. One such development from the author’s laboratory, depicting a small-size electron column, is shown in Fig. 6.15. This column also contains a novel electron detector inside the column [20]. One other parallel development has been in using silicon wafer technology to make the various microscope column components, which include the lenses, the deflectors and the stigmator correctors, as depicted in Fig. 6.16 [21]. The whole column in the latter case measures only a few mm in height making the whole microscope with its control electronics, vacuum system and specimen chamber comparable in size to a laser printer. This SEM is now commercially available.
Fig. 6.14  The imaging of doped semiconductors with low-voltage electrons (Image (a) collected at 6 keV) and (b) collected at only 2 eV). The contrast of the p-regions, which are the bright areas, is increased as the beam energy is decreased. Note in particular the appearance of some small surface particles (contaminants) when imaging at such low-electron energies. In images (c, d) another effect is shown which is related to the scanning speeds the images are collected (c) at high speeds (TV rates) and (d) at slow scanning speeds of only 2–5 s per frame [18–20]

Fig. 6.15  A whole electron column from the author’s laboratory, developed for imaging at ultra-low voltage use, down to 1 eV. It contains all the necessary components to form an image, a Schottky field emission source, the electron lenses and def deflectors as well as a novel in-lens electron detector [20]
The Specimen Chamber

The specimen chamber of the electron microscope is an airtight metal vessel that is pumped to a pressure of the order of $10^{-5}$ to $10^{-6}$ mbar (standard sea level pressure is defined as 100 mbar). Again, the size of the specimen chamber differs greatly between the TEM and the SEM. The former can only accommodate samples measuring a few mms in diameter, where the specimen to be studied occupies only a small fraction of this size. The SEM, on the other hand, could accommodate samples a few cm in diameter or even larger, as in the semiconductor industry where full wafers measuring up to 30 cm in diameter are normally used in critical dimension type SEMs. Often, however, in material science, biology and general engineering, samples seldom measure in excess of several mm in diameter. Therefore most specimen chambers in SEMs measure between 20–40 cm to accommodate the various samples from these disciplines.

6.6.3 The Detectors

It is also customary that the specimen chamber, particularly for the SEM, accommodates a number of detectors to capture the various signals emitted from the sample being studied, as depicted in Fig. 6.3. Associated with the choice of a given detector is a requirement to control the total pressure around the sample. For example, if one needs to map the distribution of a given element on the top few atomic layers of a solid sample (as in Auger electron spectroscopy [4]), then the pressure around the sample should be much lower than if the signal being captured is only to reflect the surface topography (i.e. using the low-energy electrons depicted in Fig. 6.5). In the former, a pressure in the region of $10^{-9}$ mbar or lower is needed, whilst for the latter the environment around the sample could be as high as $10^{-5}$ mbar. There is now a class of instruments, referred to as environmental SEM (ESEM), where the pressure is even higher than conventional SEMs ($>10^{-2}$ mbar). This is to allow for the inspection of insulated or biological specimen, including glass, wood and even meat.
The development and use of energy-dispersive X-ray detectors (referred to as either EDS or EDX) has, however, given electron microscopes, and particularly the SEM, an added dimension in analytical power. Traditionally, X-ray analysis of solid samples has been carried out using dedicated instruments that employ wave-dispersive type detectors (WDS) (i.e. where the emitted X-rays are detected according to their wavelength for the WDS rather than according to their energy, as in EDS). Detecting the emitted X-rays was the task of the first electron microprobe for use as an analytical tool, and it is referred to as the electron probe microanalyser (EPMA) [22]. The EPMA is a much more complicated instrument than the SEM due to the special X-ray detectors used, but of course it has more powerful analytical abilities, too. In addition it has a higher X-ray resolving power than the EDS type and thus enables smaller amounts of materials to be detected. However, EDS is cheaper and simpler to operate. It is estimated that up to about half of all electron microscopes in the world are equipped with an EDS detector, which is mainly used for qualitative elemental analysis with detection limits of around one atomic %.

There are many other types of detectors that are also used to gain more information from the sample under investigation, and the semiconductor industry leads in this respect. Some of these are used to check the operation of the integrated circuits being manufactured and are normally employed on fabrication lines for quality control and monitoring during fabrication. The SEM has, indeed, underpinned the progress made in the semiconductor industry over the years and is most likely to continue to play such a role in the future.

In biological applications, the SEM is equally important. However, because the samples are of tissues from living beings and/or plants, these are normally ‘special’ treated to withstand the low pressure of the SEM (e.g. in a process called dry-freezing, where the sample is first subjected to a low temperature treatment to freeze-dry it followed by slicing and inspection). This is normally followed by coating its surface with a conducting film to avoid being charged by the incident electrons. These special treatments have by and large been avoided in the ESEM, which is now becoming an important imaging tool in biological applications. But, on the other hand, there are still a lot of instruments which are not of that type in addition to there being some applications which may require inspections in conventional SEMs. In these cases the sample would normally be covered by a conducting metal film prior to inspections. Such films are so thin that they still allow one to observe fine details.

### 6.7 Fourth-Dimension Electron Microscopy or Time-Resolved Electron Microscopy

In 2009, the Arab laureate, Ahmed Zewail, announced the success of his research group in introducing a new dimension to electron microscopy by utilising femtosecond laser pulses to radiate the electron cathode of the microscope, thus releasing single packets of electrons for bombarding the samples instead of a continuous beam. The laser used is carefully chosen to just exceed the work function of the microscope’s electron source, which is normally of the field electron emitter type, as discussed in Sect. 6.6.1 above. The bound electrons at the Fermi level of the electron source are released upon the laser pulse striking the tip of the electron emitter. The released ‘packet’ of electrons is then accelerated by the electron optical column towards a sample under investigation. Note that these electrons are also focussed by the column in exactly the same way as a continuous stream of electrons would have been; so in effect one is using a focussed packet of electrons to bombard the sample. Zewail called this new microscopy mode ultra-fast electron microscopy, which now has come to be known as 4D UEM (see Chap. 3).
In these experiments, one can follow the dynamic response of the sample at such incredibly short time scales.

The pioneering work of Zewail is in carefully choosing the wavelength of a laser (which corresponds to a given energy) to slightly exceed the work function of the electron source of the microscope. This then eliminates the need to apply a voltage pulse to the emitter, which is limited to microseconds at best. Laser-pulsing at the femtosecond is now standard in 4D electron microscopy. It should be said that 4D electron microscopy is in its infancy, and there are several research groups around the world who are actively engaged in the development of this exciting 4D microscopy technique (see Chap. 3 for a full list of references and the various areas of application of the technique). Their efforts are likely to open up new areas of applications and enhance our understanding of materials and biological processes.

### 6.8 Lensless Electron Microscopy

The 1986 Nobel Prize in Physics was awarded for the development of a new type of electron microscopy called scanning-tunnelling microscopy (STM) and for the development of the first electron microscope, given to one of the survivors Ernst Ruska. The STM was demonstrated by two Swiss scientists, Heinrich Rohrer and Gerd Binnig, who proposed a new method to map surfaces at the atomic scale. In this technique, a voltage-biased sharp-field electron emitter, as discussed above, is brought ever so close to the specimen surface (i.e. in the order of only several atomic layer distances) where electron tunnelling via field emission starts with the application of very small voltages of less than 50 V. The emitter is then scanned in a raster fashion over the surface, and the tunnelling current obtained is used to map the spatial distribution of the surface atoms. The original STM experiment was essentially carried out under ultra-high vacuum conditions (i.e. about $10^{-10}$ mbar). Today there is an array of different configurations of the same principle in what is referred to as scanning-probe microscopy (SPM). It is ironic that the majority of the SPM methods can be carried out under atmospheric pressure. Scanning-probe microscopy today is used in a variety of disciplines ranging from biological applications to physics and engineering. The reader is referred to recent publications for up to date references [23].

Finally, whilst much work has been invested in developing ever more sophisticated electron lenses to map surfaces at the highest possible resolution, a recent development has demonstrated the possibility of producing high resolution secondary electron images at the sub-10 nm region without the use of an electron lens [24]. There is a great deal of research still to be carried out to optimise the technique and to quantify it, but this has been an interesting development in electron microscopy and could yet lead to further if not different areas of applications of this indispensable instrument.

### 6.9 Application of Electron Microscopy Towards Light-Producing Devices

In this international year of light and light-based technologies, it is appropriate to cite one area where electron microscopy has been an invaluable research tool in its own advancement. The use of electron microscopy has over the years been fundamental in developing the light-producing devices we have been using, ranging from the incandescent light bulb to the latest development of known as ‘light-emitting diodes (LEDs)’ (see later on an explanation of LED). There are a number of benefits in developing an efficient ‘white’ light source like LEDs closely
resembling natural light. For example, it is known that sunlight is responsible for replenishing 90% of the vitamin D in human bodies (via exposing our skins to direct sunlight), compared to only 10% replenishment via food [25]. This vitamin is of great medical importance to humans. Natural light is also believed to have a protective effect against certain types of cancers (breast and prostate types) by preventing the over-production of cells [26]. In addition, its deficiency could lead to a weak immune system against colds, coughs, etc., and many studies link it to body fatigue (seasonal affective disorder—SAD), broken bones and fracture. The conventional light bulb as well as the fluorescent tubes is very inefficient in resembling sunlight, which makes the search for alternatives all the more important.

In addition to the medical benefits LEDs bring to human life, their development has potential impact on the amount of electricity used worldwide. It is currently estimated (in the year 2015) that lighting amounts to at least 20% of the total world electricity produced. For a country like the UK, for example, this amounts to the equivalent cost of about $3bn/annum. If white-colour-based LEDs could replace the conventional light bulbs around the house and in offices, then energy saving of more than 80% could result. The US Department of Energy estimates a projected energy saving amounting to about $30bn/annum, if LED replace incandescent lighting, with the added bonus of avoiding about 1800 million metric tons of carbon emissions [27]. Imagine this being applied worldwide; it would have huge savings on world resources but would particularly allow less developed countries to offer their citizens lighting resources at modest cost. Furthermore, LEDs are more compact in size, contain no mercury—hence safer to use and dispose of—and have more than 30 times longer life (~50,000 h of use).

But what is a light-emitting diode, and what does it consist of? And more crucially what does electron microscopy have to do with its development?

The light-emitting diode is one of many types of the basic diode that are used as the building blocks in semiconductor devices. However, there are some crucial differences between diodes used in general electronic devices, such as the indispensable silicon p-n junction diodes used in most electronic devices, and those employed for lighting. In the former case, a piece of silicon, for example, is designated into two parts. In one part a material like boron (B) replaces the host silicon atoms to a value not exceeding 0.01% of the total volume which makes the p-region, while on the adjoining part a material like phosphorous (P) is used to a similarly low concentration and this makes the n-region of what becomes a p-n device. The n-region is the one which has more electrons per unit volume than the host silicon whilst the p-region has more ‘holes’, i.e. less electrons. It is the electron movement within the two regions which causes the flow of electrical signal throughout the electron devices.

The development of white LED technology has eluded scientists for many years. Red and green LEDs have been available for almost 50 years, but it was the blue LED that was required to make up the white light. The case for a successful and functioning blue LED is rather special, though. For brevity, one can summarise the material requirements for producing blue LEDs to be:

1. The semiconductor material has to be of direct type (i.e. when an electron loses energy by falling across the band gap, no phonons—which leads to heat—are also produced.). For more explanation of direct and indirect band gap semiconductors, see Chap. 10.
2. The energy gap should be intermediate in the range 1.77 and 3.1 eV, which corresponds to a wavelength in the visible spectrum between 400 and 700 nm.
3. The material should be amenable to the formation of p-n junction diodes.

The above requirements eliminate widely used semiconductor materials like silicon and germanium which are both of the indirect type (see [28] for more
discussion of semiconductor fundamentals). Although this is the case, this did not stop researchers from developing LEDs based on silicon compounds, such as silicon carbide (SiC) which has been successfully used to produce commercial LEDs, albeit with very low efficiency.

The search for a material or a compound of materials to satisfy all the above three requirements looks as if it has finally been found in gallium nitride (GaN). This material is a direct III–V semiconductor with an energy gap of 3.36 eV. It is now considered as one of the leading candidates in this technology and commercial devices using this material have already been on sale for the last year or two. The development of this compound material owes heavily to the work and contribution of two groups, Isamu Akasaki, Hiroshi Amano of Nagoya University, Japan and Shuji Nakamura of the University of Santa Barbara, USA, who shared the Nobel Prize for Physics for 2014 ‘for the invention of efficient blue light-emitting diodes which has enabled bright and energy-saving white light sources’. Whilst they demonstrated the blue-light laser using GaN during the late 1980s and early 1990s, it took many research groups in academia and industry alike all over the world nearly three decades to develop efficient methods for the reliable and large-scale production of GaN. Professor Sir Colin Humphreys of the University of Cambridge, UK (see Chap. 5) has been one of the world’s leading scientists in developing GaN-based LEDs, and his research group’s work has built upon the demonstration of the Japanese Nobel Prize winners and developed new methods for the large-scale production of commercial material. The use of electron microscopy has been crucial in this endeavour, and examples from work published by Humphreys’ group will be briefly reviewed here, although parallel stories indicative of the crucial role that electron microscopy has played apply in the case of all the other examples and in the various developments by scientists all over the world towards this goal.

The challenge faced by the community researching GaN for use in LED technology, following the Japanese demonstration, was in producing high-quality material free of defects. It is important to appreciate that the role of defects in semiconductors is similar to that of the ‘dopant’ materials one adds to form the p-n junctions. Further, the small amount of this foreign material or defect that alters the electrical behaviour of the semiconductor is comparable in size to that of a golf ball and a football pitch. However, in terms of the foreign material one adds to make a p- or n-type semiconductor, one knows a priori its characteristics and how much to add to achieve the required results. In the case of material defects, this is uncontrolled with devastating consequences, if their numbers exceeds a certain limit.

One of the challenges faced by Humphreys’ group was in using silicon wafers as the base on which to grow the various layers of materials needed to produce the LED device. A schematic of a typical LED device based on GaN is shown in Fig. 6.17 [26]. The substrates used in this technology and almost all other semiconductor devices, which do not have an active role in the working of the device, are normally made from silicon wafers to reduce the cost of the more expensive active materials like GaN. However, it is the substrate material that causes some of the dislocation defects seen in the grown GaN materials. Such dislocations reduce the efficiency of the LED and their reduction is therefore of utmost importance in the search for efficient devices.

In addition, depositing GaN on Si causes the former to react with the Si to form a Ga-Si alloy and melt-back etching that alters the concentration of the constituting elements. To avoid this from happening, a layer of aluminium nitride (AlN) is first deposited on the Si as a nucleation layer. The quality of this layer and its interface with the Si substrate are of great importance for the produced LEDs; and the interfacial layer between the Si and the AlN, as depicted in Fig. 6.18, was only possible to study by using a state-of-the-art TEM with a resolution of 0.1 nm.
This TEM resolution could only be achieved after the correction of the microscope’s spherical aberration. It is interesting to note that the solution offered by Humphreys’ research group has now successfully been taken up by UK industry (Plessey Semiconductors).

It is also interesting to note that whilst most researchers thought that the use of indirect bandgap semiconductors is not suitable for efficient LEDs, nanotechnology is providing a route where this may not necessarily be the case. The work reviewed by Professor Nayfeh in this book demonstrates that indeed there is life for silicon in producing efficient lighting if one uses silicon nano-particles (see Chap. 10).
6.10 Conclusions

The development of the blue LED, which earned its developers the Nobel Prize in 2014, and the subsequent tireless work of scientists and engineers in its development as a working device, could not have been realised had they not used state-of-the-art TEMs which are equipped with aberration correctors, but equally if scientists and engineers had not followed the scientific method of enquiry in their research and development. The use of the law of refraction is fundamental in electron and light optics, and the name of Ibn Sahl, the tenth century Arab mathematician, should be on a par with that of Snell. It is also clear that the correction of the spherical aberration defect in electron optical instruments and in other imaging devices is crucial for achieving the ultimate performance of such devices. Whilst Ibn al-Haytham’s early discovery of spherical aberration should be correctly acknowledged, as well as his many other pioneering work in optics, his legacy in science goes far beyond these achievements (see Chap. 2 for more details). More importantly in relation to science and engineering, Ibn al-Haytham again deserves credit and recognition for his role in laying down the foundations of the scientific method of enquiry in his work on optics some 1000 years ago entitled, Kitab al-Manazir. It is fitting therefore for UNESCO to recognise this year and celebrate the contribution of Ibn al-Haytham as a pioneering polymath.

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