# *Nanomaterials*

2nd year Medical Physics

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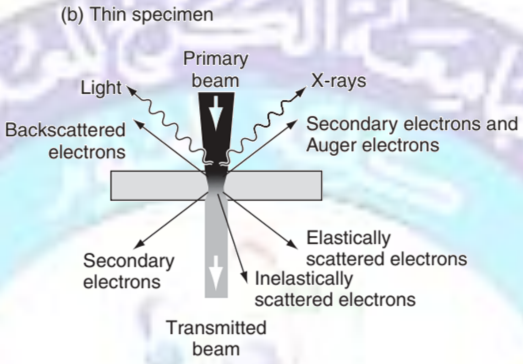
## Lecture 6: Characterisation



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## 6.1 Introduction

Characterisation methods focus mainly on both imaging (microscopy) and analysis (spectroscopy).

The type of these methods depends on the probe and what this probes excites from the sample.

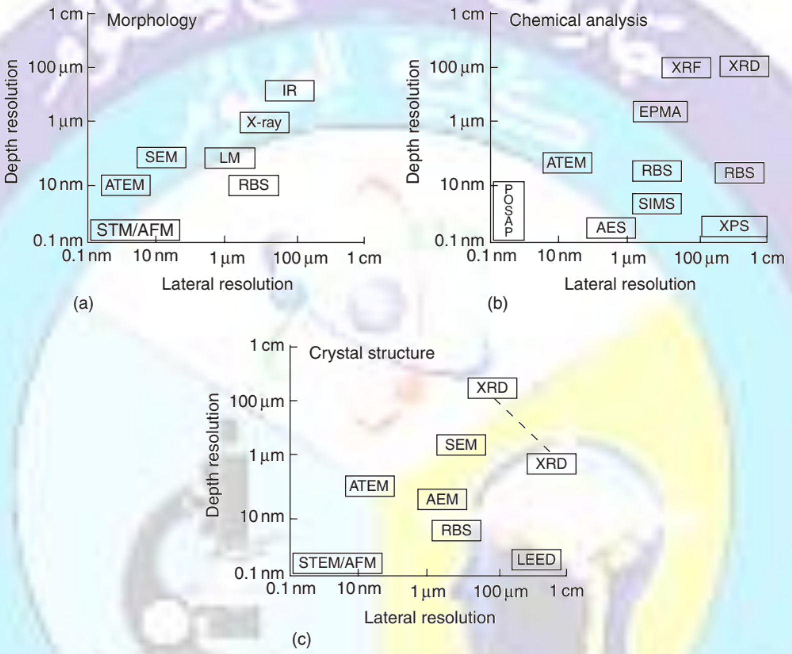
Generally, there exist around ten possible primary probes, e.g.: electrons, X-rays, ions, atoms, light (visible, ultraviolet, infrared), neutrons, sound, which may be used to excite secondary effects (electrons, X-rays, ions, light, neutrons, sound, heat, etc.) from the region of interest in the sample.

This means a maximum of around 700 characterization techniques are possible, however, only around 100 different techniques have been used to date. Most of which use ions, electrons, neutrons or photons as the primary probes.

## 6.2 Types of information

Before using a particular characterisation technique, it is essential to consider what information is required about a sample and at what resolution. We may think of this information as being divided into:

a) Morphology (the microstructural or nanostructural architecture);
b) Chemistry (the elements and possibly molecular groupings present);
c) Crystal structure (the detailed atomic arrangement in the chemical phases contained within the microstructure);
d) Electronic structure (the nature of the bonding between atoms).



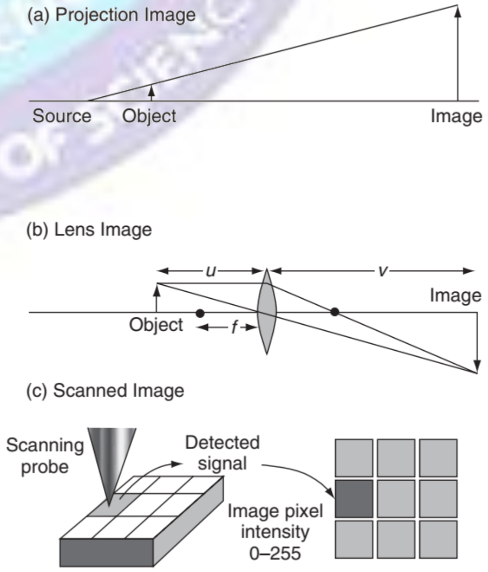
## 6.3 Image forming

The naked eye image resolution is about 0.1–0.2 mm at the near point of vision.

The general purpose of microscopy is to resolve finer details than that for human eyes.

For nanostructured materials we need to resolve details down to a level of 100 nm and below. In transforming an object to an image, there are essentially three methods available, as shown in the figure:

a) A projection image formed in parallel; e.g., field ion microscopy (FIM).
b) A lens image formed in parallel; e.g., TEM and light microscopy. Here, a lens of focal length *f* will form an image of an object at a magnification *v/u*, where u and v are the object and image distances, respectively. The



relationship between these parameters is given approximately by the thin lens formula

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

c) A scanned image formed in serial; e.g., scanning electron microscopy (SEM) and scanning transmission electron microscopy (STEM).

## 6.4 Light microscopy

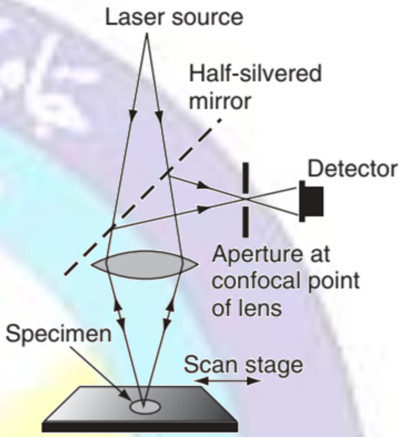
Light microscopy uses visible or UV light microscopies for imaging.

The wavelength of these rays is insufficient for the direct visualization of nanostructures.

Light microscopy images surface regions, since light does not penetrate the sample, and provides up to $10^3$ magnification.

Key aspects to note are that, owing to the nature of the radiation, the sample can be viewed under ambient conditions and can also be subjected to dynamic experiments whilst being simultaneously observed.

Light microscopy techniques require some sample preparation such as polishing or thin sectioning.



The signal is either reflected, fluorescent or transmitted light and it creates a 2D image.

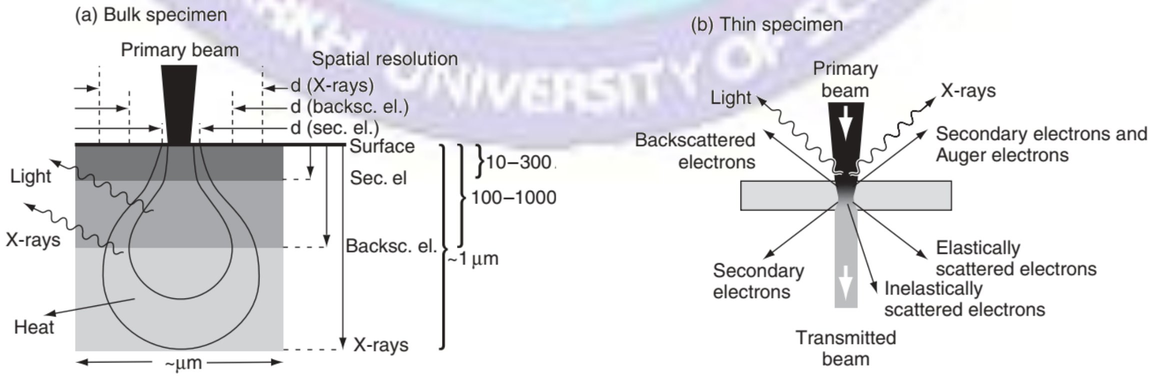
## 6.5 Electron Microscopy

### 6.5.1 Interaction of electrons with specimen

The figure below summarises the various signals produced as a result of electron–specimen interactions in bulk and thin samples.

It shows the different signals from the specimens that arise from the interaction of electrons with the specimen.

The interaction volume is defined as the volume within which 95% of the electrons are brought to rest by scattering, and has a characteristic teardrop shape (as shown in the figure).

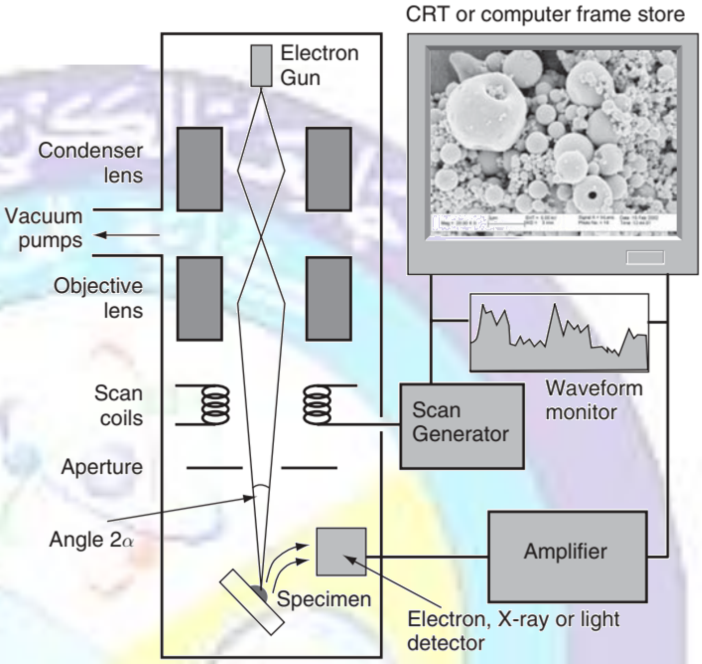


The depth and lateral width of electron penetration in the specimen are roughly proportional to $V^2$ and $V^{3/2}$, respectively, where V is the accelerating voltage.

Besides the primary electrons, there are various types of emitted electrons which leave the surface of a bulk sample and may be used for imaging or analysis.

### 6.5.2 Scanning Electron microscopy (SEM)

SEM is extremely useful for imaging surface and subsurface microstructure. The basic layout of SEM instrumentation is shown in the figure.



The electron source (gun) usually consists of a tungsten or LaB6 filament or field emission gun (FEG).

The accelerating voltage is usually between 1 and 30 kV.

In the SEM, two or more condenser lenses are used to demagnify the crossover produced by the gun, while the objective lens focuses the electron probe onto the specimen.

The final probe diameter lies between 2 and 10 nm.

For creating images, SEM uses secondary electrons or other signal, such as backscattered electrons or X-rays, as they are emitted from each point on the surface.

The intensity of each pixel on the monitor is directly related to the emission intensity of the selected at a corresponding point on the specimen surface.

The image produces can be 3-dimensional and the magnification can be up to $10^6$.

SEM uses samples of any thickness, but imaging is limited to the interaction volume of electrons-specimen (figure above). Hence, the image will reveal information for surface and sub-surface regions.

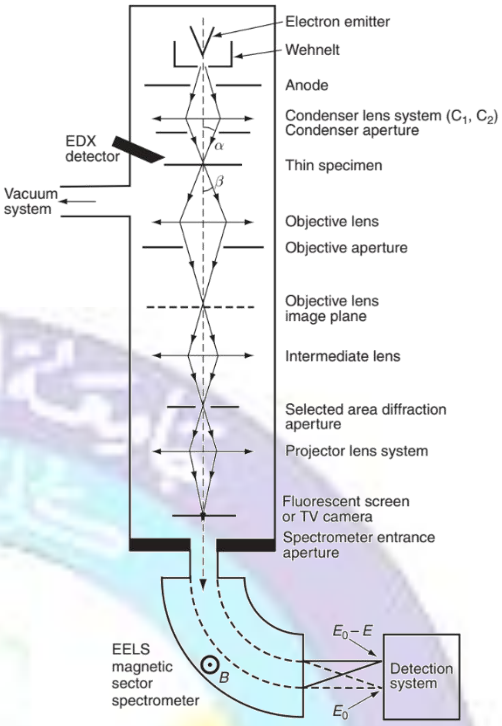
SEM requires a conductive sample.

If SEM imaging is needed for a non-conductive sample, a thin layer of conductive coating is applied to the sample.

SEM requires vacuum environment since it uses electrons, which limits dynamic and biological experiments.

### 6.5.3 Transmission Electron microscopy (TEM)

The conventional TEM is a key tool for imaging the internal microstructure of ultrathin specimens.



The electron gun is usually thermionic tungsten or LaB6, however FEGs are becoming increasingly common.

The accelerating voltage is considerably higher than in an SEM and is typically 100–400 kV.

The benefits of high voltage include increased imaging resolution, increased penetration and thus the ability to study thicker samples.

Two or more condenser lenses demagnify the probe to typically 1 mm in diameter are used.

The specimen must be no more than a few hundred nanometres (typically <150 nm) in thickness, and is usually in the form of a 3 mm diameter disc.

The combination of the objective lens and the projector lens system provides an overall magnification of around $5\times10^7$ times.

TEM provides information on the internal structure of the sample.

TEM specimens are normally in the form of an ultrathin disc, prepared by cutting, mechanical polishing or chemical dissolution, followed by electropolishing.

TEM also requires vacuum environment.

A comparison between the resolutions of different characterisation techniques is shown below