Chapter 1

Interaction of radiation with matter

1.1 Types of radiation and basic groups of methods

Interaction of radiation with the matter is necessary in order to characterize the microstructure. The term radiation is ment in a general sense, either particles or waves. This is obvious namely for the electromagnetic radiation because photons are discrete quanta of electromagnetic radiation. The photon is identified by the wavelength, λ, energy, E, and frequency, ν , all of which are related by the equation

$$h\nu = E = hc/\lambda \tag{1.1}$$

where h is the Planck constant and c the velocity if light.

Types of radiation used for analyses are:

- photons
- electrons
- neutrons
- protons
- ions/atoms

In many cases the material is probed with one type of radiation but a second type is detected. This occurs in X-ray photoelectron spectroscopy (XPS) where the incident probe is a beam of X-ray photons but emitted electrons are detected, whereas this is reversed for the technique of energy dispersive X-ray (EDX) analysis.

We have to understand the interaction of primary radation with the material in more general sense because produced secondary radiation (photons, electrons or other particles) also interacts with the material when travelling towards the surface. Only taking this into account, we can use the emitted signals to gain an understanding of the material being examined.

There are in principle three groups of physical characterization methods:

- microscopy (optical, electron, atomic force)

- diffraction
- spectroscopy

1.2 Penetration depth

The penetration depth is a measure of how deep the radiation/particles can penetrate in the material. The penetration depth or mean free path of the incident beam determines the depth and volume of material that will be sampled. This quantity is also useful to understand from what depth the information can be obtained. In the methods that uses different primary and secondary radiations, the particle or radiation which has the shortest mean free path in the material will determine the volume analyzed (information depth).

The intensity of radiation which is transmitted through sample thickness d is

$$I = I_0 \exp(-\alpha d), \tag{1.2}$$

where I_0 is the intensity of incident radiation and α is the **absorbance**.

1.2.1 Photons

The penetration depth of photons shows considerable and dramatic variations between different types of material and photon energy or wavelength. It is defined as the depth at which the intensity of the radiation inside the material falls to $1/e$ of its original value at (or more properly, just beneath) the surface.

When electromagnetic radiation is incident on the surface of a material, it may be (partly) reflected from that surface and there will be a field containing energy transmitted into the material. This electromagnetic field interacts with the atoms and electrons inside the material. Depending on the nature of the material, the electromagnetic field might travel very far into the material, or may die out very quickly. For a given material, the penetration depth will generally be a function of wavelength.

The huge electromagnetic spectrum covers wavelengths between 10^6 m and 10^{-14} m. If we use the photons for microstructure analysis it is necessary to select the length of radiation comparable with the structure to be analyzed $\Rightarrow$ between 10^{-4}–10^{-10} m.

- IR radiation: Long wavelength infrared radiation is used to characterise materials by determining how specific wavelengths are absorbed (spectroscopy) - penetration depth strongly depends on chemical bonds;
- VIS radiation: Visible light is used in a variety of instruments mainly to obtain a visual image of the surface (microscopy). Some material are opaque while others are transparent. Even for high reflectivity opaque materials the penetration depth is in order of 50–300 nm. It can be also used for determination of film thicknesses (spectroscopic optical methods like ellipsometry, spectrophotometry).
- UV radiation: Reveals information about the electron band structure. Most substances absorb in this region.

- X-ray radiation: X-ray penetration is easier to predict than visible radiation. The penetration depth is typically μm. Absorbance increases with the atomic number. X-rays are mainly used in X-ray diffraction and XPS (probing electron states).

- Gamma (γ) radiation: has energy between 50 keV–50 MeV $\approx 10^{-2}$ nm. Absorbance is governed by the same relation for X-rays, but here α is inversely proportional with the atomic number. Gamma radiation penetrate almost all the laboratory samples. The method that uses γ-rays to characterize materials is called *Mössbauer spectroscopy* (probing states in nucleus).

1.2.2 Electrons

Penetration depth changes significantly with the electron energy and the atomic number of the material:

- for stainless steel: around one tenth of a μm for energy 10 keV and 2 μm for 30 keV,

- for energies higher than 10 keV: elements with atomic number under 20 have a penetration depth up to 10 μm while the elements with Z higher than 40 below 2 μm

- for energies between 0–2 keV: the penetration depth is reduces to 0.4–300 nm

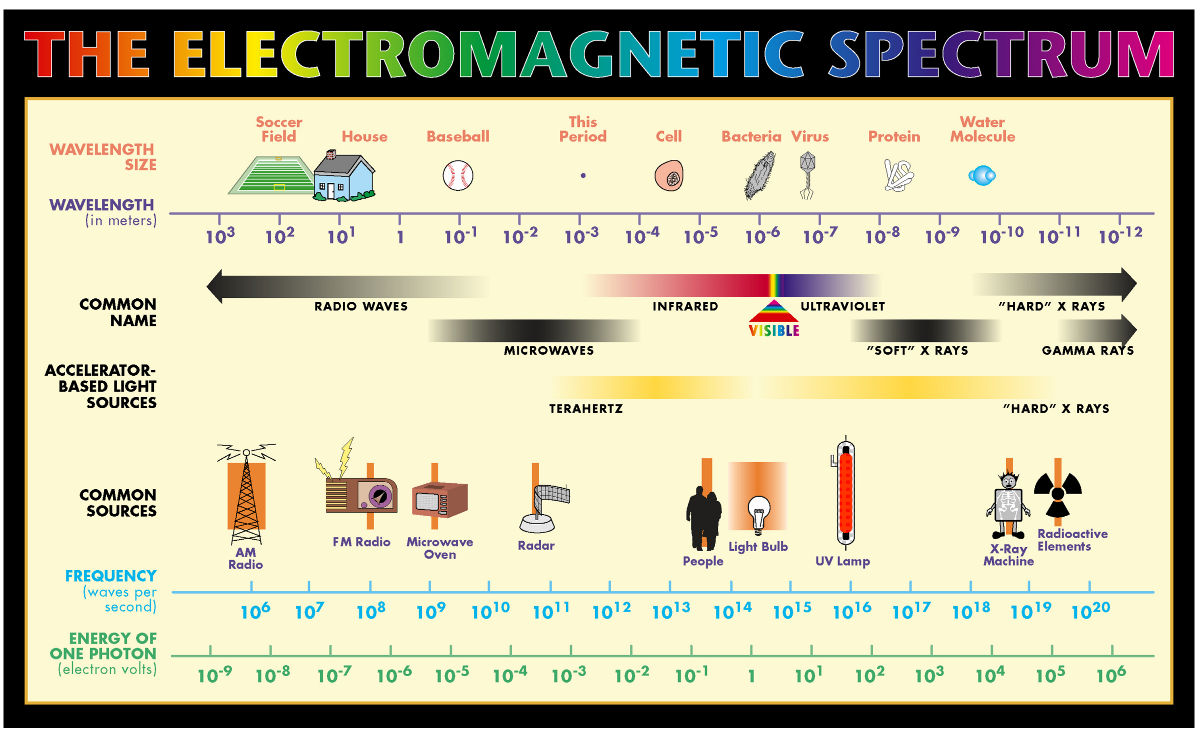

Figure 1.1: The electromagnetic spectrum illustrating the relationship between wavelength, frequency and energy of photons.

1.2.3 Neutrons

Although neutrons are thousand times the mass of electron and as a consequence is more particle like, it still possesses sufficient wave character to be diffracted by materials. Since it does not possess an electrical charge it is not affected by the electron cloud surrounding the nucleus and on passing through a material effectively interacts only with atom nucleus. ⇒ the penetration depths are larger than those of electrons or X-rays and are in order of few millimetres. Neutrons can be used to study microstructure within the bulk of a material.

1.2.4 Protons

The interaction of a proton beam with a material has many similarities to the electron but there are some important differences. The proton being charged is influenced by elstat forces within the material but because the mass is 1836 times that of electron, a proton of a few MeV energy has a much greater momentum than electrons of say 50 keV. The proton loses a small fraction of momentum in each atom collision and will not be deviated significantly from the incident beam direction. ⇒ Therefore, protons will travel much further into a material (with a little scattering) than electrons of the equivalent energy penetration depth is greater than for electrons. For 2.5 MeV proton, the penetration depth is around 55 μm for carbon and 28 μm for silver. It decreases with the the proton energy and Z of the material.

Protons are frequently used to excite X-rays in a technique known as a particle induced X-ray emission (PIXE). Other method using protons is Rutherford backscattering spectroscopy (RBS).

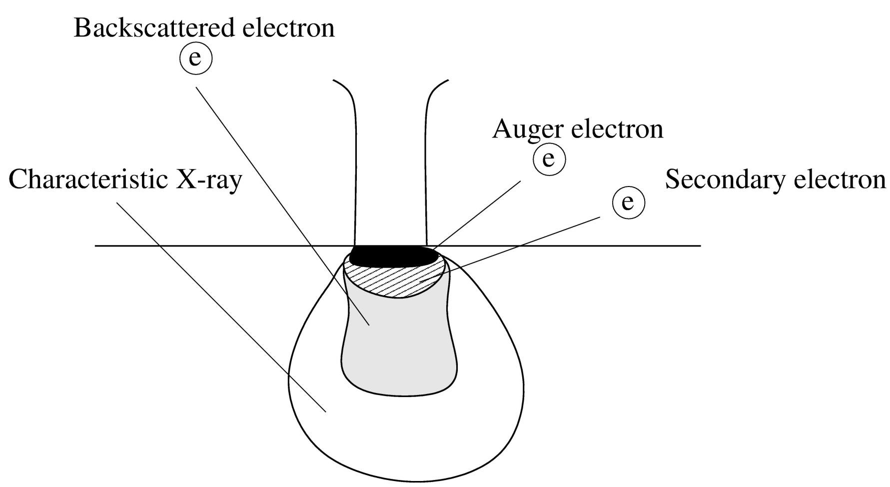

Figure 1.2: Example of different information depths for interaction of primary electron beam with material.

1.2.5 Ions/atoms

Penetration depth depends on the number of protons in the ion, its energy and the target.

- For low energies (several eVs) it is reflected from the target and part of its energy is transferred to the surface atom, causing ejection of atoms, ions or clusters. Kinetic energy is transferred to the surface atom M_2 but the impinging ion does not penetrate into the surface.

- At higher energies, the atom burrows into the material, causign atoms, atom clusters, ions and ion clusters to be ejected from the surface while, at the same time, atoms are knocked further into the material. These in turn collide with other atoms establishing a collision cascade process in which atoms collide with one another and move in both forward and backward directions. The original ion will either come in rest in the material or may be ejected on the other side.

The range R - the total distance that the ion (projectile) travels in coming to rest, is longer than the penetration depth x. The **projected range** R_p is defined as the total path length of the projectile with energies $0.002 \leq \epsilon \leq 0.1$ keV measured along the direction of incidence.

$$R_\mathrm{p} = C_1(\mu) M_2 \left[\left(\frac{Z_1^{2/3} + Z_2^{2/3}}{Z_1 Z_2} \right) E \right]^{2/3}, \tag{1.3}$$

where M_2 is the atomic mass of the target , Z is the atomic number while $C_1(\mu)$ is experimentally determined. For projectiles with energies $0.5 \leq \epsilon \leq 10$ keV

$$R_\mathrm{p} = C_1(\mu) M_2 \left[\left(\frac{(Z_1^{2/3} + Z_2^{2/3})^{1/2}}{Z_1 Z_2} \right) E \right]^{2/3}. \tag{1.4}$$

Ion channelling is possible along crystallographic axes with small critical angles. In this case the penetration depth is substantially increased.

1.3 Material damage

The material can be damaged by the interaction with radiation:

- **photons**
  Regarded in general as the least harmful, the photons will damage the target nevertheless. Damage can occur by heating the target, which depends on the radiant energy, photon flux and the depth of penetration. Moreover X-rays can induce surface oxidation and lasers can burn holes into material. In general the most photon radiation causes very little damage but some maetrials can be sensitive even to visible light (photographic layer, photoresists) $\Rightarrow$ it is used at the beginning of target characterization.

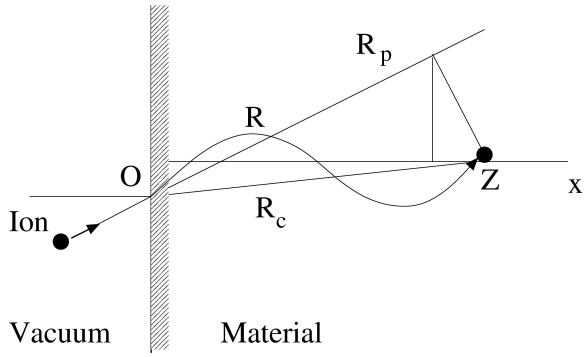

Figure 1.3: Calculation of total distance of ion.

- **electrons**
  Although electrons have a dual nature, their weight leads to a relatively large transfer momentum if they are accelerated to several hundreds keVs. The resulted damage depends on the heat transferred and the thermal conductivity of the material. For metals and alloys the damage is minimum but in polymers and oxides the damage is worse. It is possible to cover the surface with a conductive material (usually gold) but the disadvantage is that the composition cannot be later investigated.

- **ions and atoms**
  When atoms and ions penetrate the target, they can react with it or not cause any damage at all (e.g. Ion Scattering Spectroscopy, where ions interact elastically with the target) or they cause significant damage:

  - shift of atoms from their normal lattice position (amorphization)
  - breaking the bonds (requires 10x more energy) and transfer of such high energy that atoms of the material can travel through the material and leave it (sputtering)

  For low ion fluxes the damage areas are isolated (the amorphous material is surrounded by the intact one) but for high fluxes, the ions create an amorphous layer and cause significant material sputtering.

It is necessary to choose the right sequence of methods according to their damage characteristics.

1.4 Resolution

Mean free path determines the information depth and also the spatial resolution (defined as the perpendicular to the direction of the incident beam). Generally, the spatial resolution is influenced by the beam size and the wavelength.

The image can be obtain by:

- illumination of the sample and use of lens for focusing the reflected or emitted radiation. The spatial resolution depends on the lens system used and the wavelength of emitted or reflected radiation. Optical, X-ray and some ion microscopes are using this method.

- a narrow beam is directed on the sample and absorbed or reflected radiation is detected. The sample surface is scanned by a narrow beam. In this case the spatial resolution depends in the wavelength, beam size and the scattered radiation in the sample. Most of the equipment are using electrons and ions. Nowadays, also optical microscopes use the scanning procedure in so called confocal microscopy (or confocal laser scanning microscopy).