Characteristics of materials structure by the X-ray diffraction

I

Nature and sources of the X-rays

Project WND-POWR.03.02.00-00-I043/16

International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

Project co-financed by the European Union within the European Social Funds

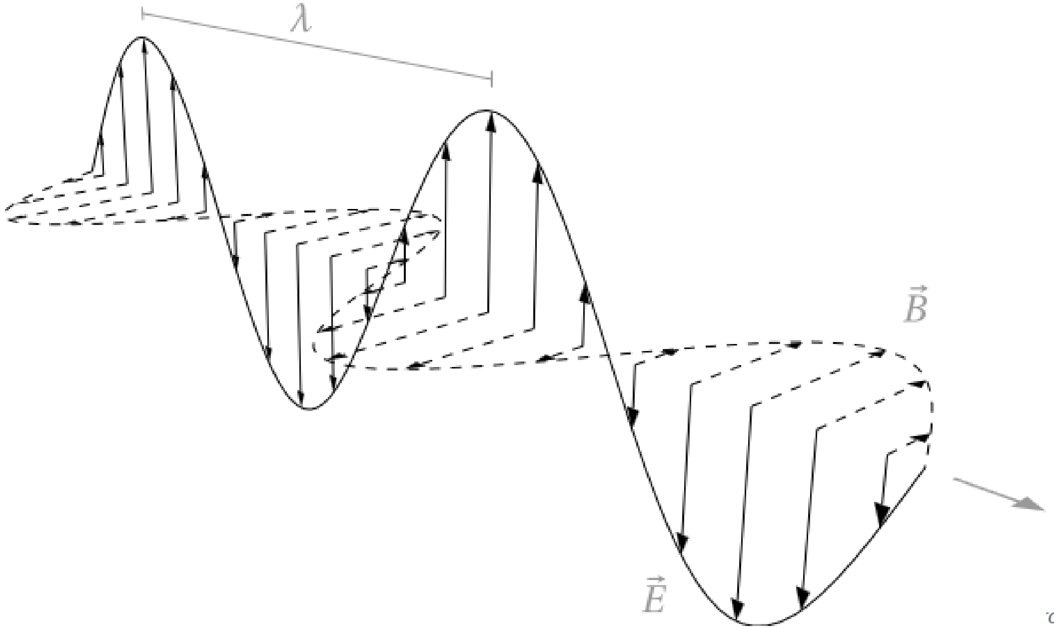
Internet survey of the lectures: http://imim.pl/studium-doktoranckie-imim-pan-uj/materiay-do-wykadow

1. Nature and sources of the X-rays
2. Diffraction phenomenon of X-ray
3. Crystallography and diffraction
4. Crystallographic texture
5. X-Ray Texture Tomography
6. Texture analysis of polycrystalline materials
7. X-ray diffraction techniques in materials engineering – measurements of **the first order stresses**
8. X-ray phase analysis, other useful methods and the newest achievements in the field of XRD
9. Demonstration of experimental set up and measurement procedures in the X-ray Laboratory

Electromagnetic radiation (electromagnetic wave)

Disturbance of the electromagnetic field in space. The electrical and magnetic components of the waves are induced by each other - a changing electric field produces a changing magnetic field, and a changing magnetic field produces a changing electric field. The properties of electromagnetic waves depend on the wavelength.

In the quantum description, electromagnetic radiation is treated as a massless stream of elementary particles called photons. The energy of each photon depends on the wavelength.

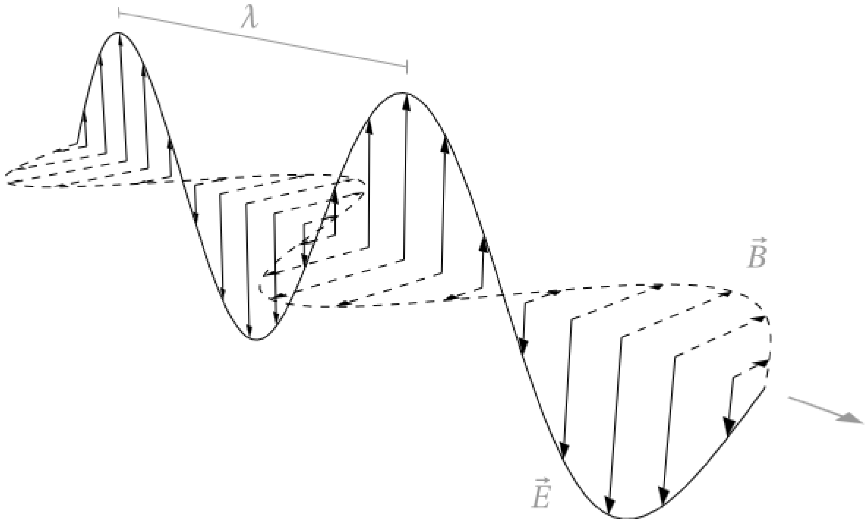


One of the most difficult issues of modern science...

Characteristics of electromagnetic waves:
Electromagnetic waves do not require the presence of a medium and may also propagate in a vacuum.
Electromagnetic waves propagate in a vacuum at a constant speed of c=299 792 458 m/s.
In material medium, this speed is always lower (equal to c/n, where n - refractive index) and depends on the type of medium and the frequency of the wave.

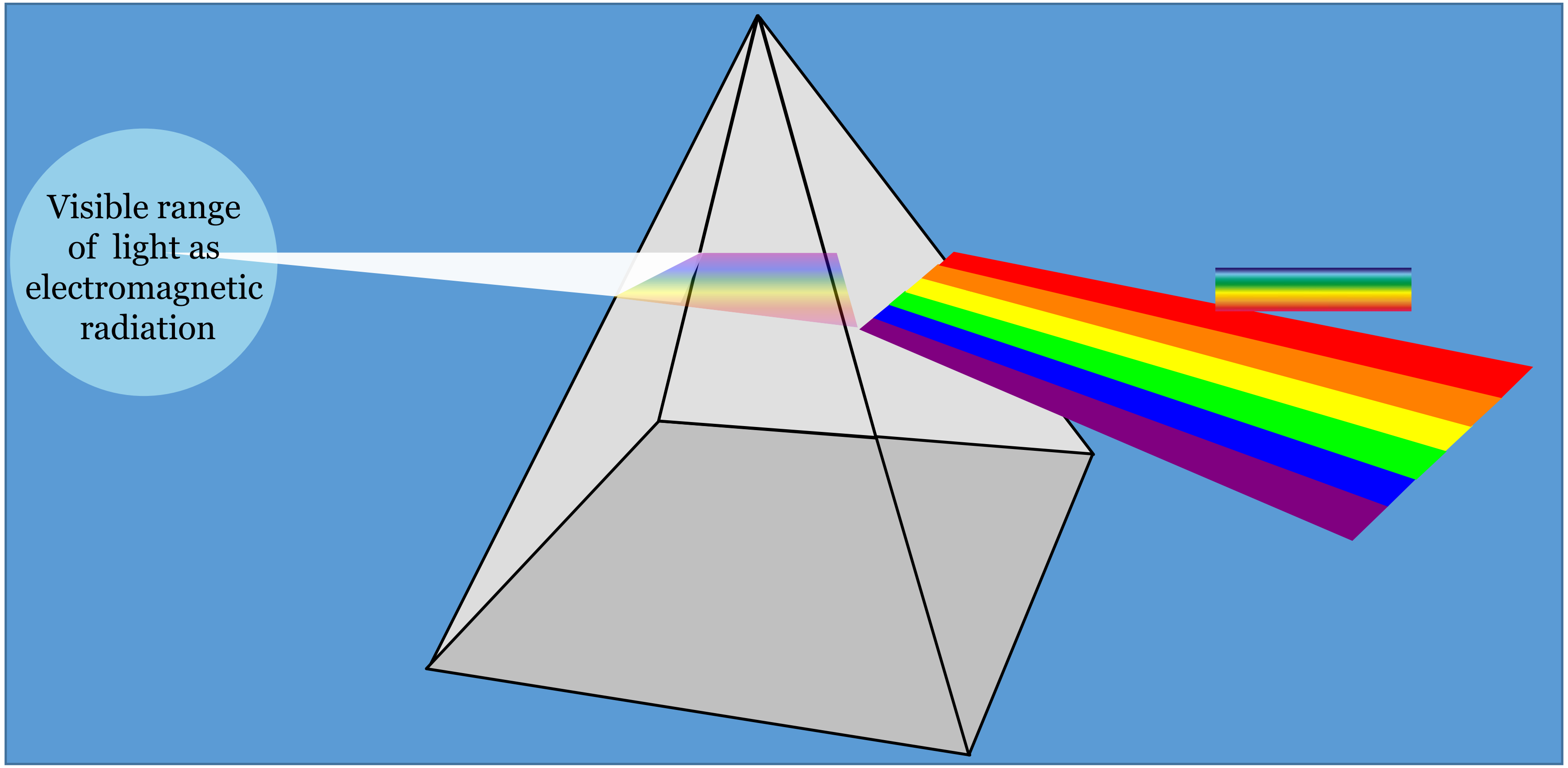
Like any mechanical wave, electromagnetic wave is characterized by:

- frequency ν, number of complete changes of magnetic and electric fields per second, expressed in Hertz (Hz),
- period of variation T, defined as the inverse of the frequency: T=1/ν, i.e. the time during which the return to the same phase of the electric and magnetic field occurs,
- wavelength λ, which is the distance between adjacent points at which the electric and magnetic fields have the same phase.



These values are interrelated: the higher the frequency, the shorter the wavelength (ν=c/λ).

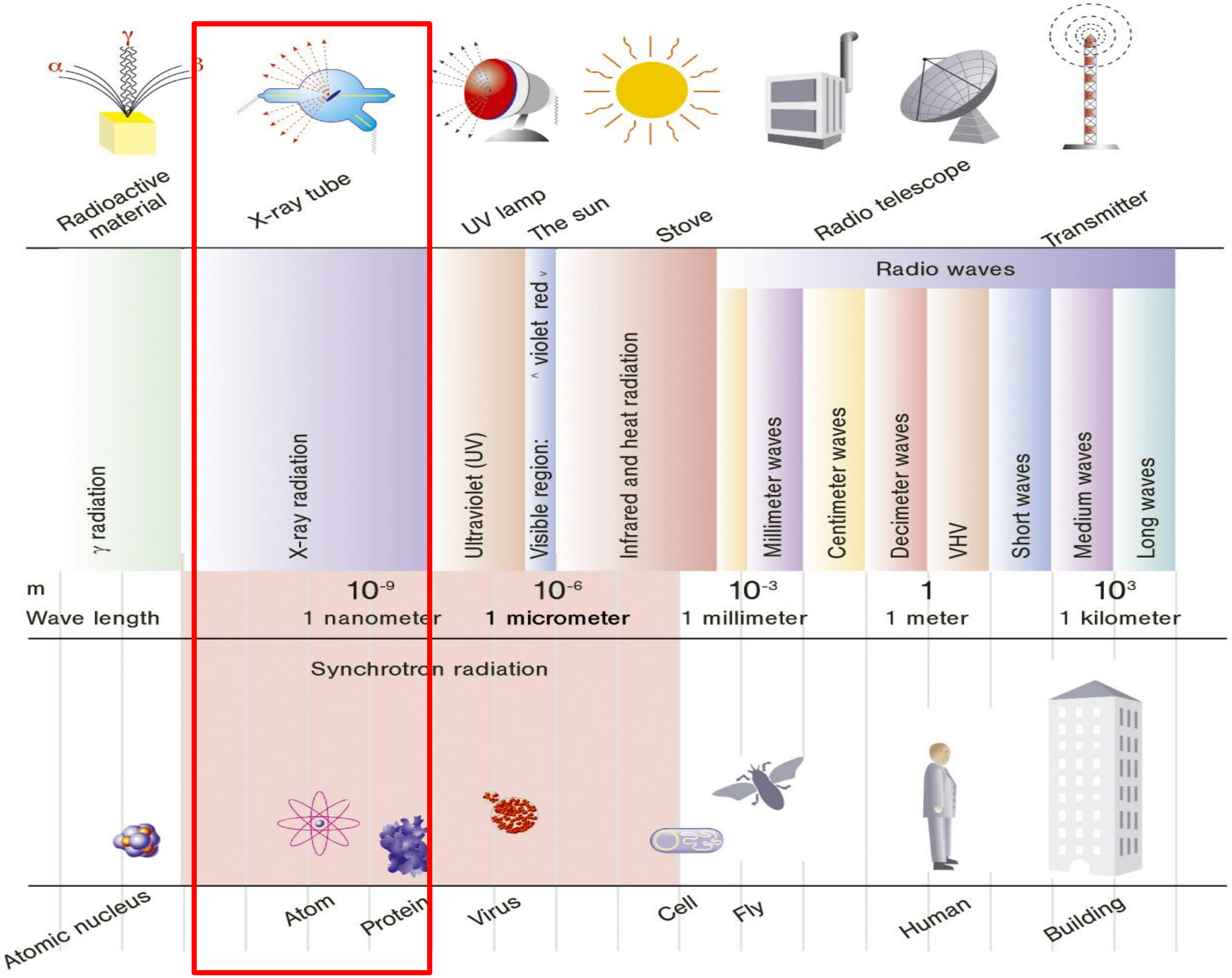
Electromagnetic waves propagate in a vacuum at a constant speed of c=299 792 458 m/s. In material medium, this speed is always lower (equal to c/n, where n - refractive index) and depends on the type of medium and the frequency of the wave.


Visible range of light as electromagnetic radiation

Electromagnetic wave is not the only possible description of electromagnetic radiation. This radiation can also be treated as a stream of small portions of energy (quantums), called photons. To describe the movement of particles we use sizes such as momentum and particle energy, not wavelength or frequency. There must therefore be a close relationship between the quantities describing the particle and the wave. According to **de Broglie's accounts**, a single photon carries

energy $E=h\nu=hc/\lambda$ and momentum $p=h\nu/c=h/\lambda$.

The photon described in this way is a particle without mass! A constant h is a constant of nature, called the Planck constant, h=6.626-10-34 J-s. It is not clear why electromagnetic wave in some phenomena shows a wave nature (reflection, refraction, interference, diffraction, polarization), and in others a corpuscular nature (photoelectric effect, Compton phenomenon). The duality of the nature of electromagnetic radiation is called corpuscular-wave dualism and it should be attributed rather to the lack of our conceptual apparatus, which is forced to transfer its images created in contact with the macro-world to microscopic phenomena. The wave image describes sufficiently well the propagation of radio waves (large wavelengths), but light phenomena already require the use of two descriptions. The higher the frequency of electromagnetic radiation (lower wavelength), the stronger the corpuscular effects and the necessary corpuscular description. The nature of electromagnetic radiation is currently explained by quantum electrodynamics.


α
γ
β
Radioactive material
X-ray tube
UV lamp
The sun
Stove
Radio telescope
Transmitter
Radio waves
γ radiation
X-ray radiation
Ultraviolet (UV)
Visible region: ˄ violet red ˅
Infrared and heat radiation
Millimeter waves
Centimeter waves
Decimeter waves
VHV
Short waves
Medium waves
Long waves
m
Wave length
10⁻⁹
1 nanometer
10⁻⁶
1 micrometer
10⁻³
1 millimeter
1
1 meter
10³
1 kilometer
Synchrotron radiation
Atomic nucleus
Atom
Protein
Virus
Cell
Fly
Human
Building

Nature and sources of X radiation

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Wilhelm Conrad **RÖNTGEN (**1845 – 1923) Germany

Received the first Nobel Prize for Physics, in **1901**. Earlier in **1895** when experimenting with a cathode-ray tube, he noticed that some nearby barium platinocyanide fluoresced. So he proposed that an unknown type of radiation (**X-ray**) was produced.

Usage of X-rays

Diffractometry
recognizing the internal structure of matter

Medical diagnostic/therapy
human/animal treating, life elongation/comfort

Industrial radiography
operational safety, quality control

Investigation of fast phenomena
flash inspection in synchrotron

Food production
detection of forein bodies, preservation

Sterilisation
medical tools/materials

Public security
ilumination of passangers/baggage, terrorism prevention

Archeology
non-destructive inspection of historic objects

X-ray Astronomy
observation of universum

Neutralization of electrostatic charges
in manufacturing the paper and plastics

X-ray litography
patterns on Si-plates

Metrology
thickness of coatings, keeping the horizontal level

Scientific research
modification of genetic structure by irradiation

Spectrometry
identification of chemical composition

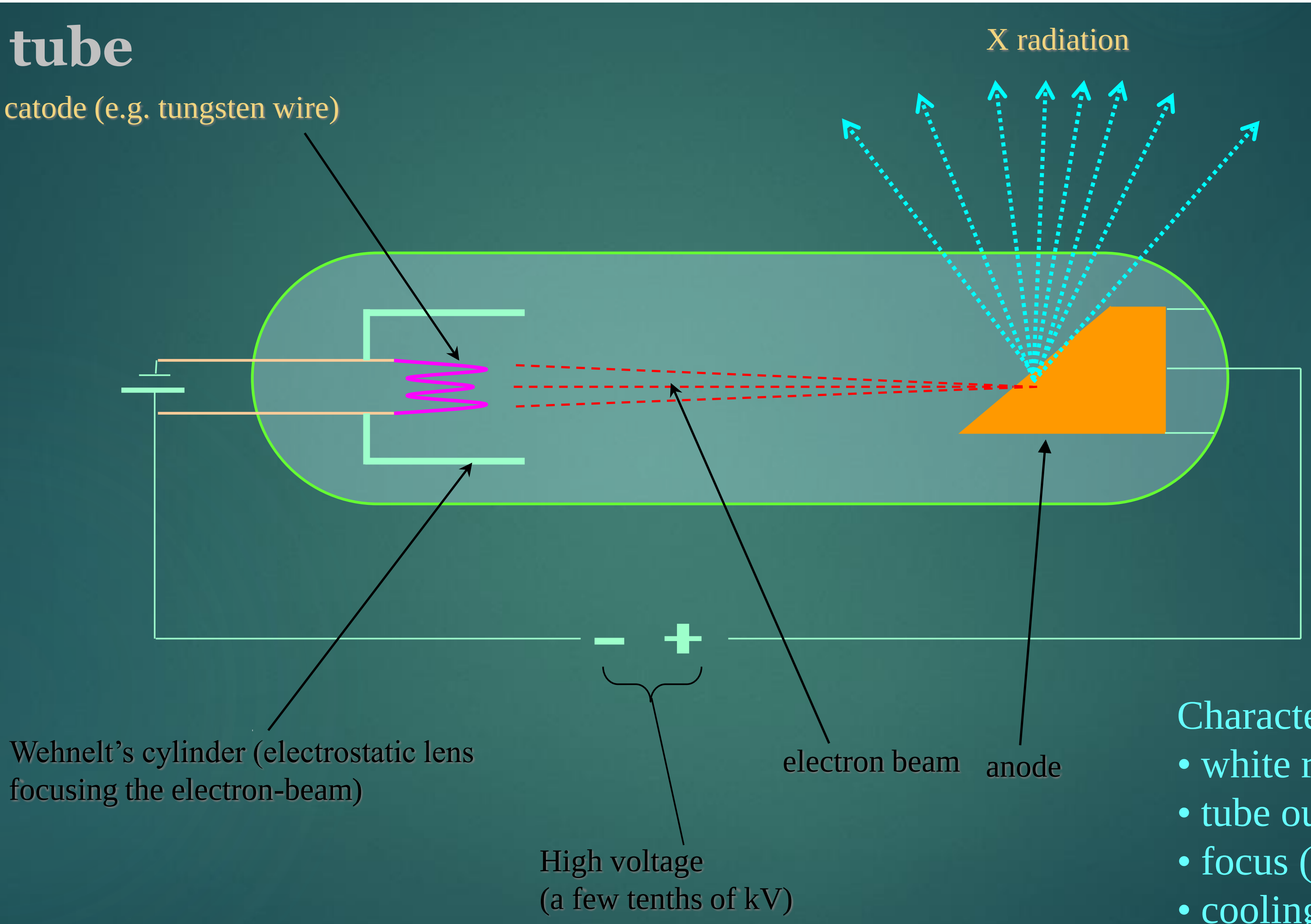
Sources of X-radiation

X-ray tube

traditional way of inducing the X-radiation

(X-rays can be generated by an X-ray tube, a vacum tube that uses a high voltage to accelerate the electrons released by a hot cathode to a high velocity. The high velocity electrons collide with a metal target, the anode, creating the X-rays. **In medical X-ray tubes the target is usually tungsten or a more crack-resistant alloy of rhenium (5%) and tungsten (95%), but sometimes molybdenum for more specialized applications, such as when softer X-rays are needed as in mammography. In crystallography, a copper target is most common, with cobalt often being used when fluorescence from iron content in the sample might otherwise present a problem.)**

X-ray tube



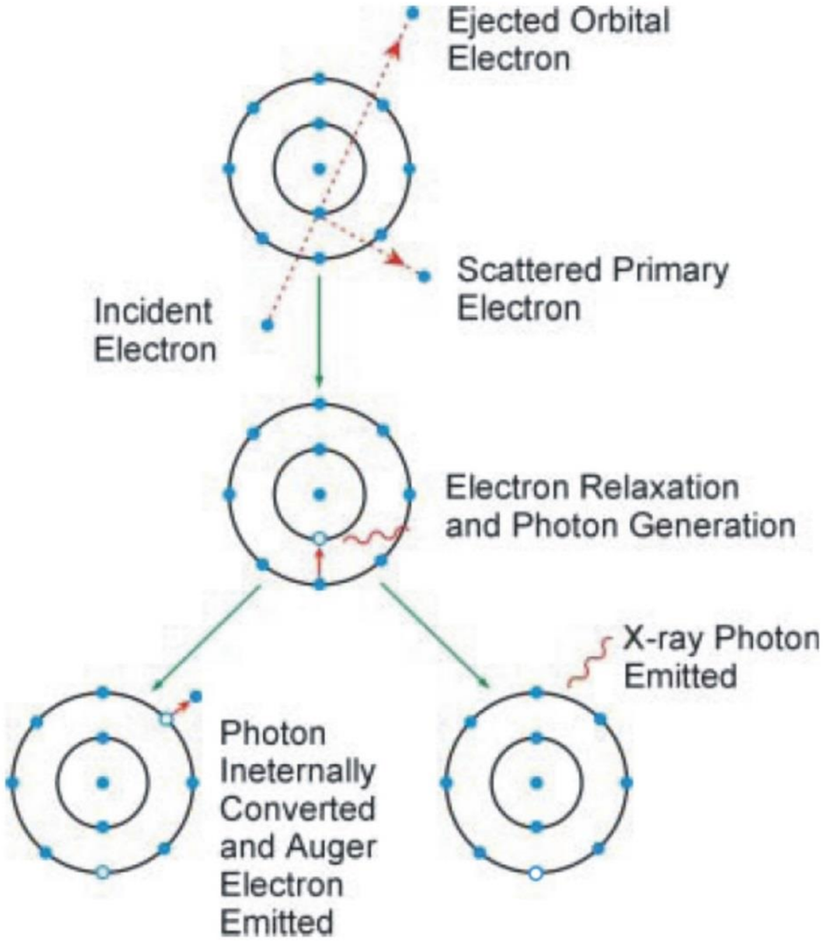
Characteristics:
- white radiation
- tube output
- focus (point/line)
- cooling system

The maximum energy of the produced X-ray is limited by the energy of the incident electron, which is equal to the voltage on the tube times the electron chargé.

When the electrons hit the target, X-rays are created by **two different atomic processes:**

- Characteristic X-ray emission: If the electron has enough energy it can knock an orbital electron out of the inner electron shell of a metal atom, and as a result electrons from higher energy levels then fill up the vacancy and X-ray photons are emitted. This process produces an emission spectrum of X-rays at a few discrete frequencies, sometimes referred to as the spectral lines. The spectral lines generated depend on the target (anode) element used and thus are called characteristic lines. Usually these are transitions from upper shells into K shell (called K lines), into L shell (called L lines) and so on.
- Bremsstrahlung: This is radiation given off by the electrons as they are scattered by the strong electric field near the high-Z (proton number) nuclei. These X-rays have a continuous spectrum. The intensity of the X-rays increases linearly with decreasing frequency, from zero at the energy of the incident electrons, the voltage on the X-ray tube.

Characteristic Spectrum



1. Incident electron knocks off an inner-shell electron (Ionization)

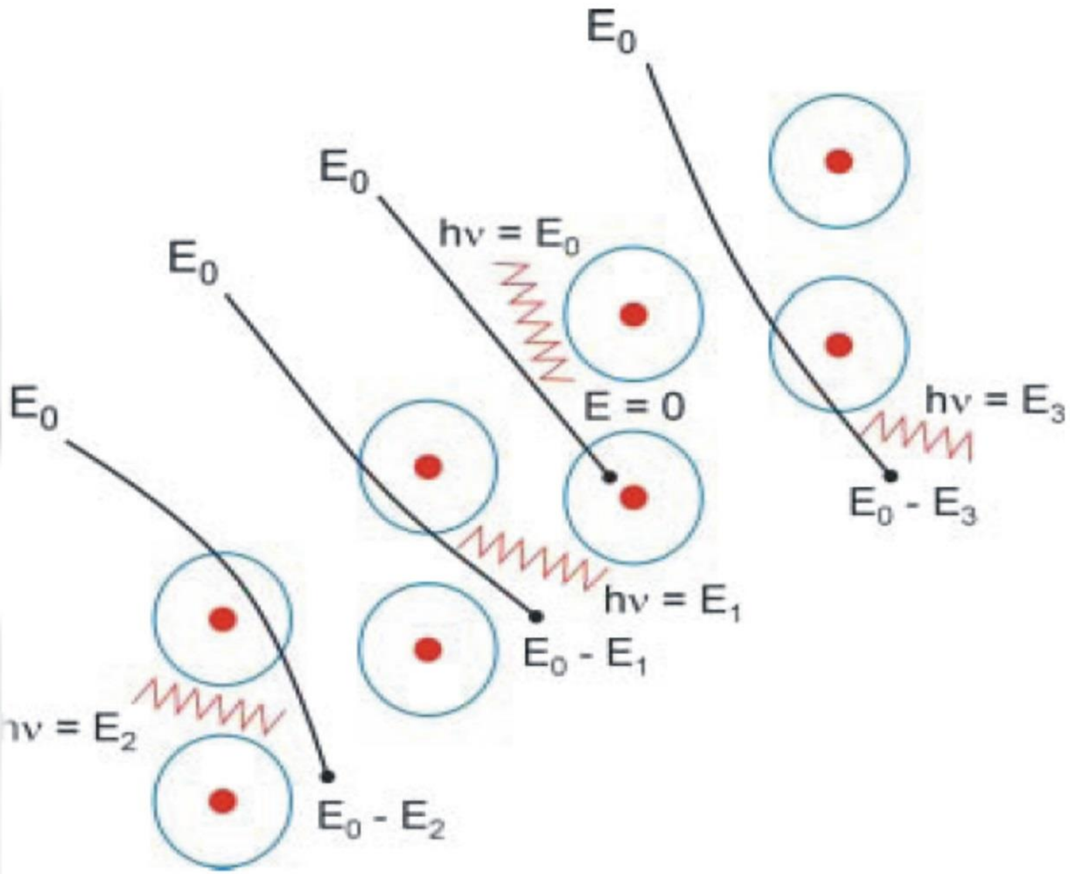
2. Outer-shell electron drops to the empty spot.

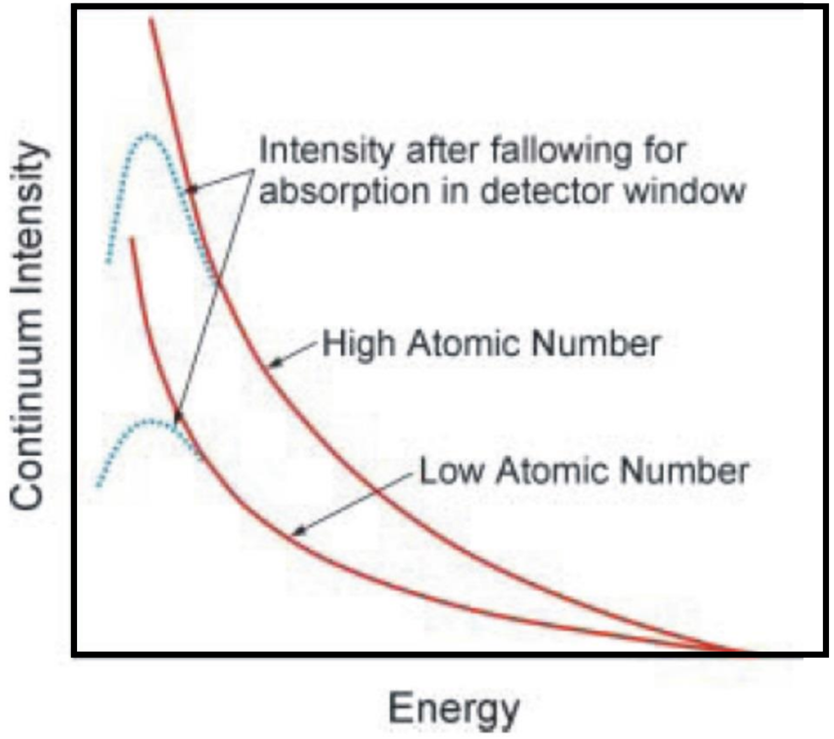
3. X-ray is emitted with the amount of an energy equal to the difference between two shells.

$$E_{X\text{-}ray} = E_{L2} - E_{M4}$$
$$= 457.8\ eV$$

➔X-rays with a specific value are Emitted.➔ Characteristic peak.

Continuum Specturm





Quantum numbers

The principal quantum number (symbolized **n**) is assigned to each electron in an atom to describe that electron's state. As a discrete variable, the principal quantum number is always an integer. As n increases, the number of electronic shells increases and the electron spends more time farther from the nucleus. As n increases, the electron is also at a higher potential energy and is therefore less tightly bound to the nucleus.

The azimuthal quantum number is a quantum number for an atomic orbital that determines its orbital angular momentum and describes the shape of the orbital. The azimuthal quantum number is the second of a set of quantum numbers which describe the unique quantum state of an electron. It is also known as the **orbital angular momentum** quantum number, **orbital quantum number** or **second quantum number**, and is symbolized as **ℓ**.

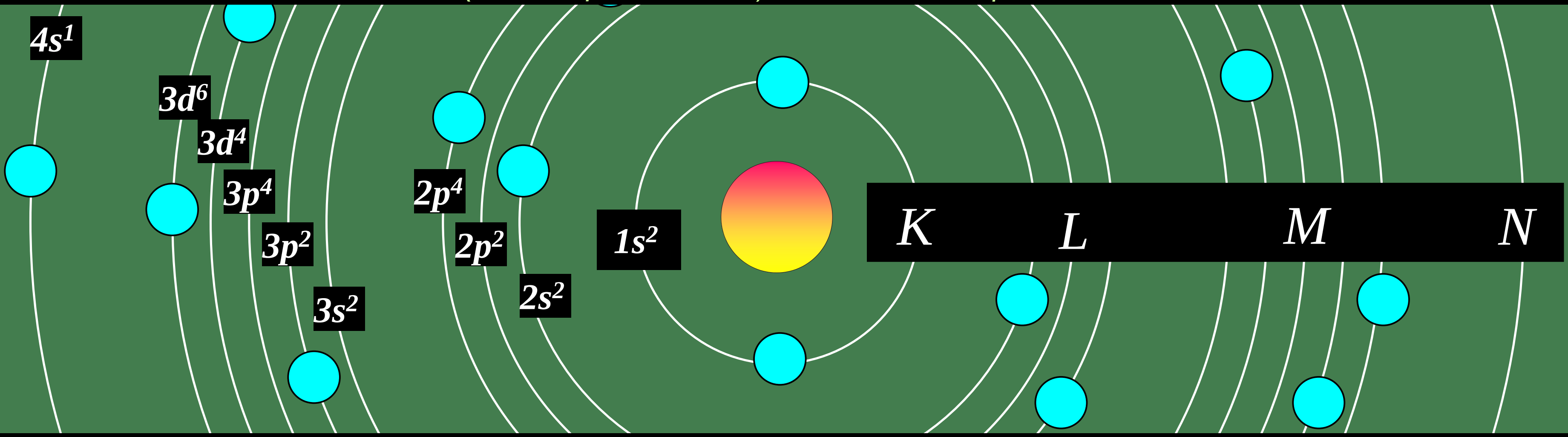
The magnetic quantum number is the third of a set of quantum numbers which describe the unique quantum state of an electron and is designated by the letter m. The magnetic quantum number denotes the energy levels available within a subshell. This quantum number indicates the possible orientation of orbital in space. The value of 'm' for a particular value of 'l' varies from +l to -l including zero.

T**he spin quantum number** is a quantum number that parameterizes the intrinsic angular momentum (or spin angular momentum, or simply spin) of a given particle. The **spin quantum number** is the fourth of a set of quantum numbers which describe the unique quantum state of an electron and is designated by the letter s. It describes the energy, shape and orientation of orbitals.

Electron configuration of ***Cu****-atom***:**

$1s^22s^2p^63s^2p^6d^{10}4s^1$

Quantum numbers describing the electron configuration are the result of description its movement by a wave equation. Non-zero solution (non-zero amplitude of electron) in the wave equation for the electron needs a complete number of its wave length for the defined orbit. For that reason the sequential numbers (quantum numbers) accurating the orbitals have been introduced: e.g. ……….. 8 states (2-"s" and 6-"p") for the electron quantum number n = 2,
……… 18 states (2-"s", 6-"p" and 10-"d") for the electron quantum number n = 3, and so on.



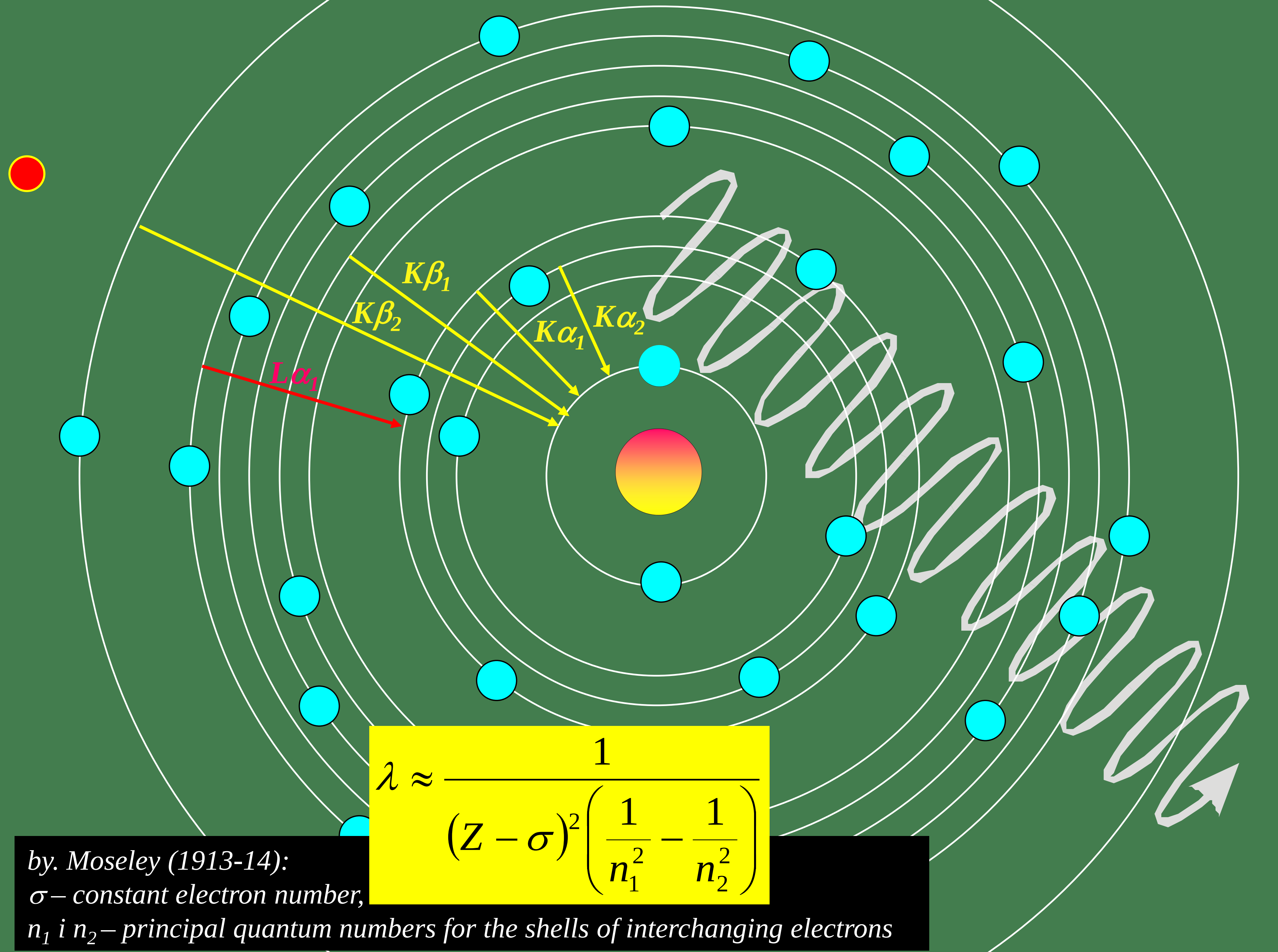
n (1, 2, 3…) **The principal quantum number**(reflects the complete wave length of the electron on its orbit with r-radius) using to estimate the electron energy.

l (s, p, d…) **The azimuthal quantum numer** (introduced for non-circular orbits) reflects the orbital moment of momentum: *s* - relates to *l* = 0, *p* – to *l* = 1, *d* – to *l* = 2,…). *l* = 0, 1, 2, 3… n-1

Additional two quantum numbers ***m*** and ***s*** are required for a complete description of the electron state in the atom:

m **The magnetic quantum number** regards a various orientation of the orbital which corresponds to specific movement state: *m* = -l, (-l+1), …, 0, +1, +2, …, +*l*.

s - **The spin quantum number** of electrons: *s* = ±½



by. Moseley (1913-14):

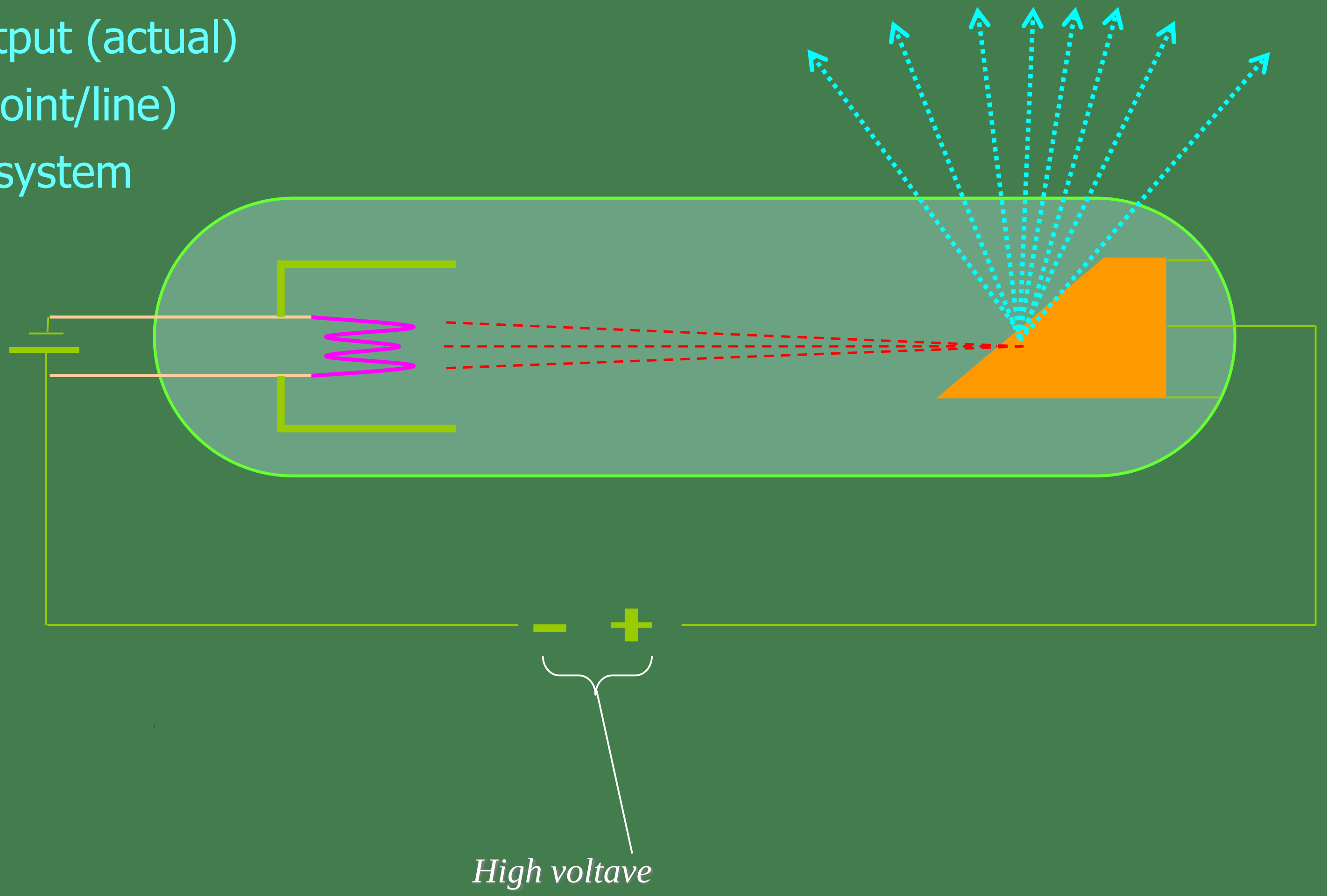
$$\lambda \approx \frac{1}{(Z-\sigma)^2\left(\frac{1}{n_1^2}-\frac{1}{n_2^2}\right)}$$

σ – constant electron number,

n_1 i n_2 – principal quantum numbers for the shells of interchanging electrons

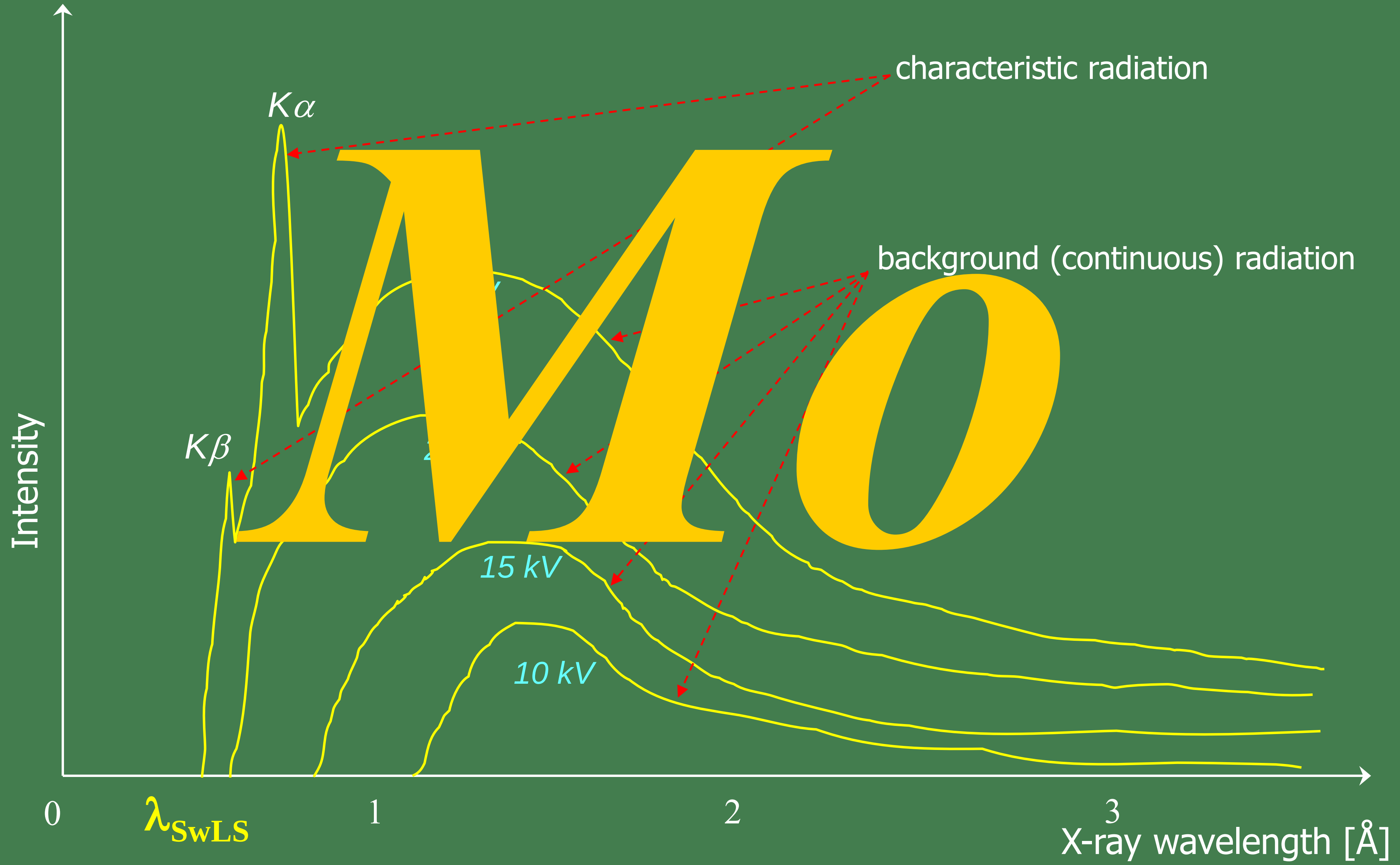
Characteristics:

- white radiation (Bremsstrahlung)
- tube output (actual)
- focus (point/line)
- cooling system



$$kinetic\ \ enery\ \ of\ \ \ elektron = eV = \frac{1}{2}mv^2$$

Short-wave limit of spectrum



X-rays can also be produced by fast protons or other positive ions. The proton-induced X-ray emission or particle-induced X-ray emission is widely used as an analytical procedure. **For high energies, the production cross section is proportional to $Z_1^2 Z_2^{-4}$, where Z_1 refers to the atomic numer of the ion, Z_2 to that of the target atom.**

Particle-induced X-ray emission or **proton-induced X-ray emission** (**PIXE**) is a technique used in the determining of the element make-up of a material or sample. When a material is exposed to an ion beam, atomic interactions occur that give off EM radiation of wavelenghts in the x-ray part of the electromagnetic spectrum specific to an element. **PIXE is a powerful yet non-destructive elemental analysis technique now used routinely by geologists, archaeologists, art conservators and others to help answer questions of provenance, dating and authenticity.**

MicroPIXE allows protein analysis. The advantage of microPIXE is that given a protein of known sequence, the X-ray emission from sulfur can be used as an internal standard to calculate the number of metal atoms per protein monomer. The relative concentrations of DNA to protein (and metals) can also be measured using the phosphate groups of the bases as an internal calibration.

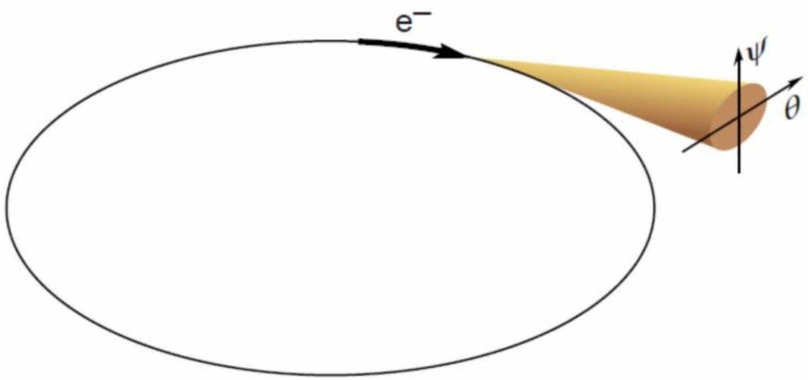
Synchrotron radiation

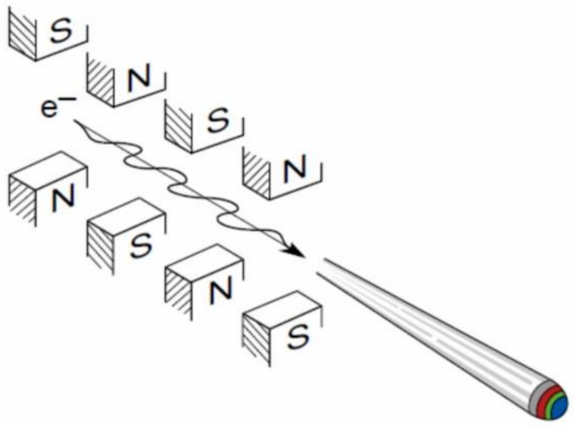
Generation of synchrotron radiation

The synchrotron was originally built for nuclear physics. In such a device, electrons or other particles are accelerated on a circular track, the circumference of which is often hundreds of metres. The curvature of the particle path is carried out by a vertically oriented magnetic field of the electromagnets. **The deviation of the track from the straight line is accompanied by the emission of electromagnetic radiation.** In past, this radiation was considered to be a necessary evil and appropriate shielding needed to be built against it.

The first devices working for solid state physics were properly adapted synchrotrons. However, the solutions applied so far proved to be inefficient, and the need to constantly repeat the work cycle of the synchrotron was also unfavourable. Currently, so-called storage rings are being built, which consume less power and do not lose electron beams as a result of bombardment of the shield, and thus can serve much more effectively for the purposes of solid state physics.

Bright and Powerful X-Rays from Relativistic Electrons





Synchrotron radiation

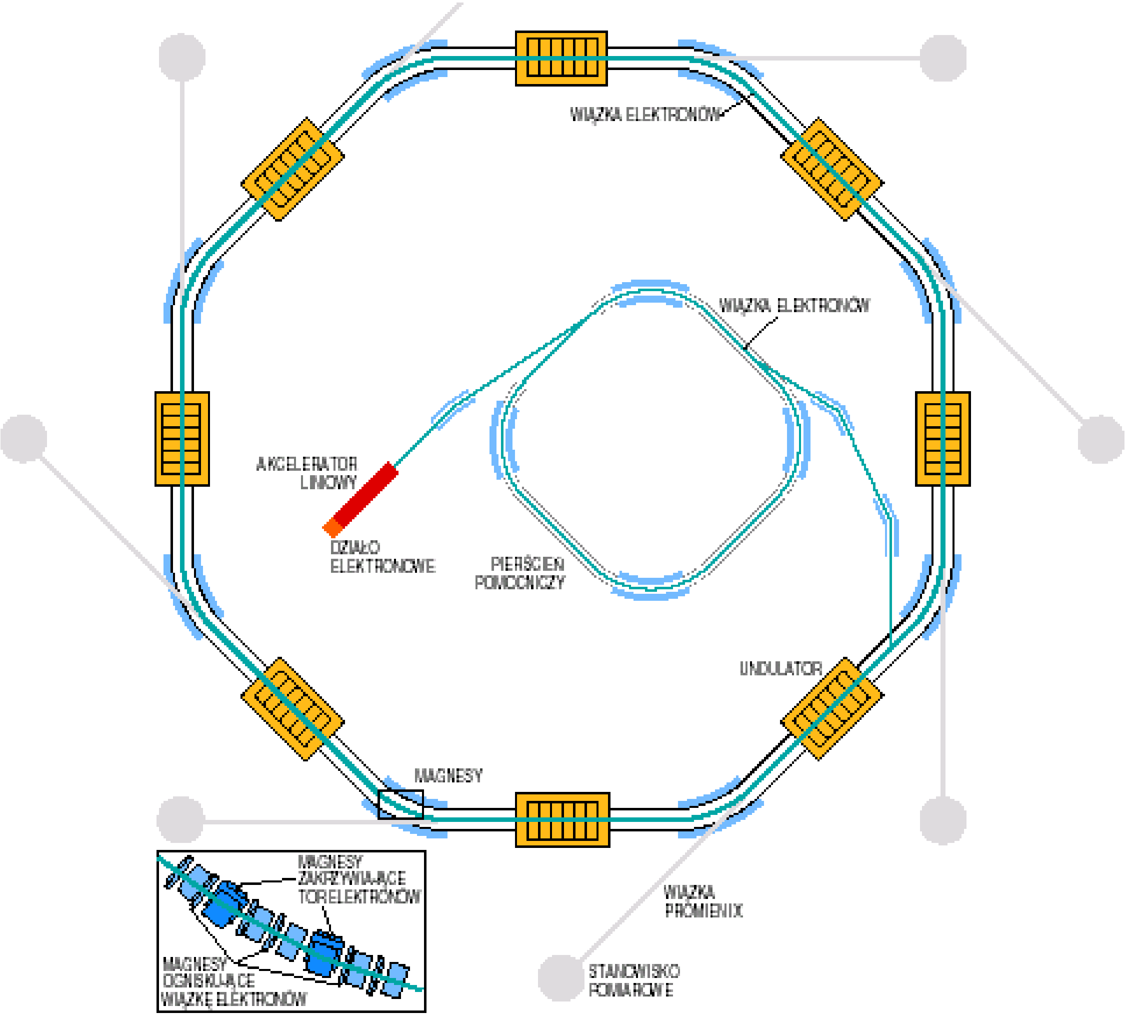
- 10^{10} brighter than the most powerful (compact) laboratory source
- An x-ray "light bulb" in that it radiates all "colors" (wavelengths, photons energies)

Undulator radiation

- Lasers exist for the IR, visible, UV, VUV, and EUV
- Undulator radiation is quasi-monochromatic and highly directional, approximating many of the desired properties of an x-ray laser

Storage ring

The acceleration of electrons takes place in a high frequency chamber. Initially, the speed of electrons increases, followed mainly by mass (relativistic effects) increases. In order to constantly increase the energy of electrons, the accelerating field must be synchronized with the time of the circuit by electrons. When the required energy is reached, the electrons fall on the disc where the test nuclear reaction takes place. This cycle shall normally be resumed at a frequency of approximately 50 Hz.

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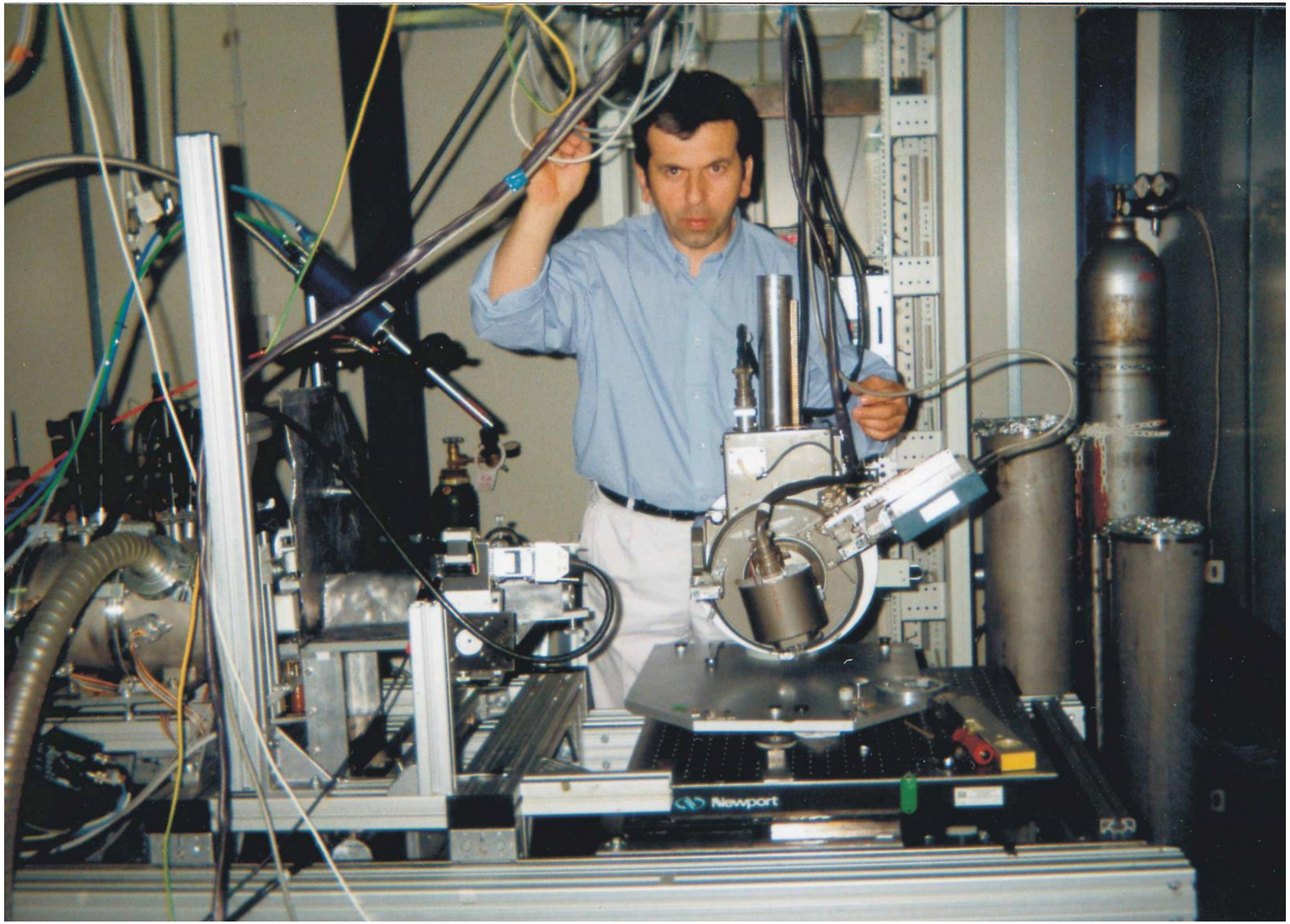
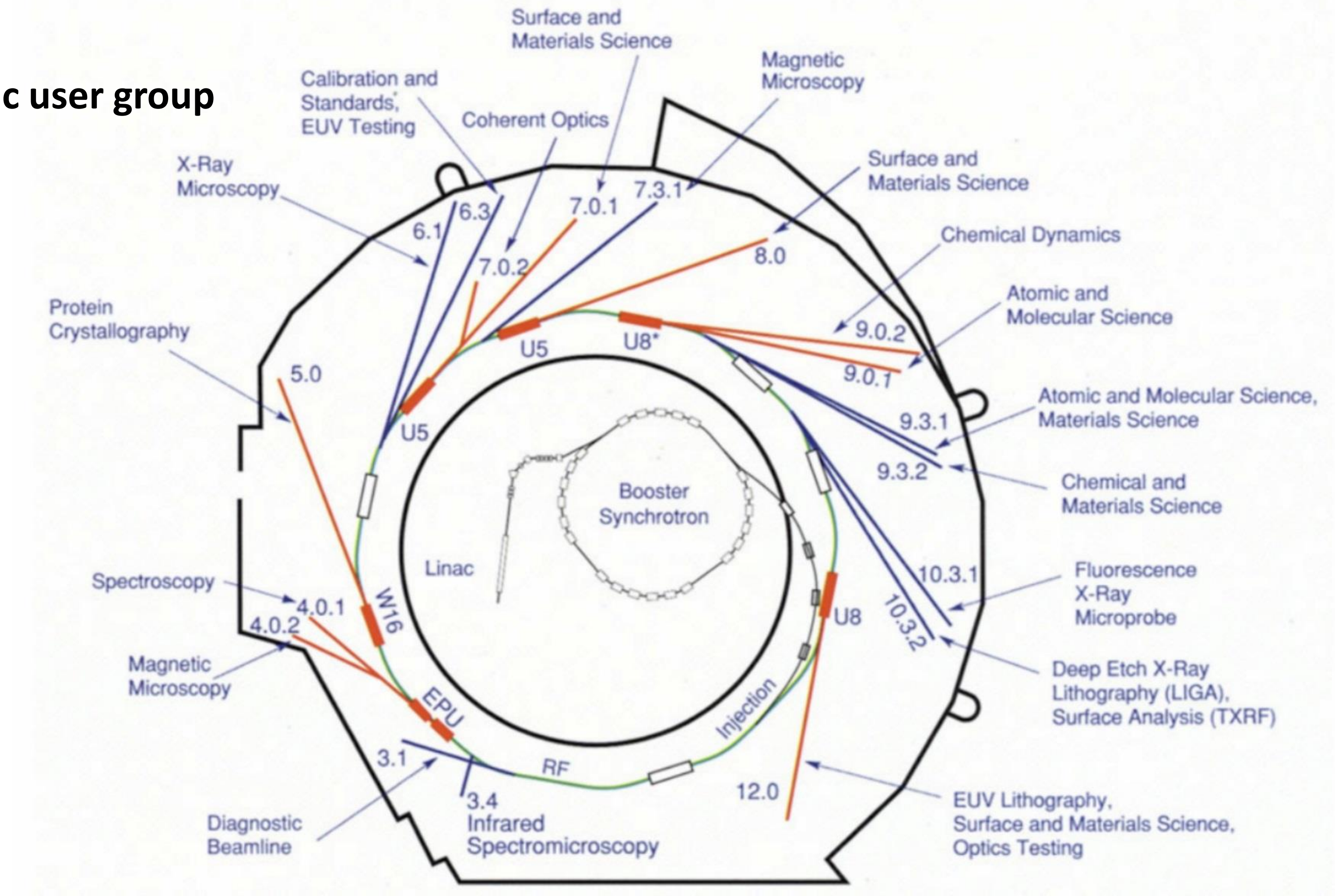
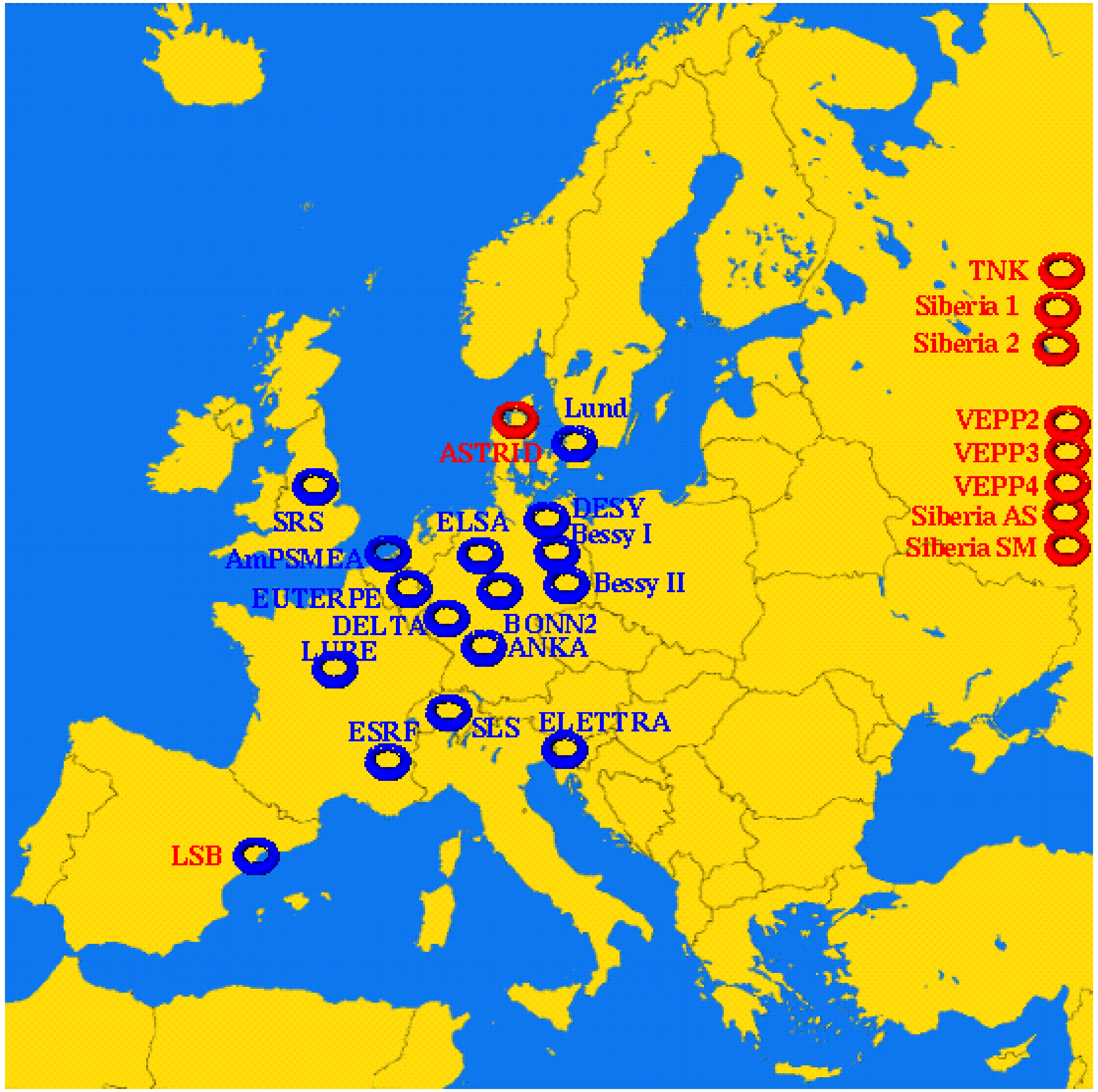
ELETTRA Synchrotron Centre (Trieste),
SAXS BeamLine 5.2. Year 2000



A single storage ring serves many scientific user group




TNK
Siberia 1
Siberia 2
Lund
ASTRID
VEPP2
VEPP3
VEPP4
Siberia AS
Siberia SM
SRS
ELSA
DESY
Bessy I
AmPSMEA
EUTERPE
Bessy II
DELTA
BONN2
LURE
ANKA
ESRF
SLS
ELETTRA
LSB

X-ray detectors

X-ray detectors are devices used to measure the flux, spatial distribution, spectrum and/or other properties of X-rays.

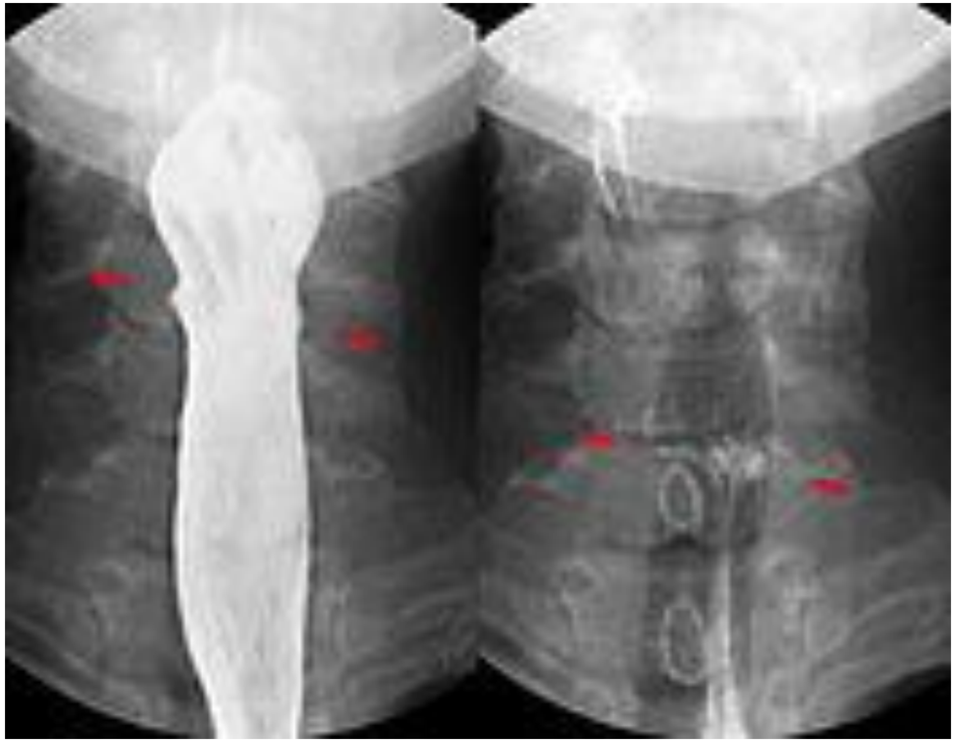
X-ray detectors vary in shape and function depending on their purpose. They can be divided into two major categories: imaging detectors and dose measurement.

- Imaging detectors such as those used for radiography were originally based on photographic plates and and later photographic X-ray films, but are now mostly replaced by various digital detector types such as image plates and flat panel detectors.

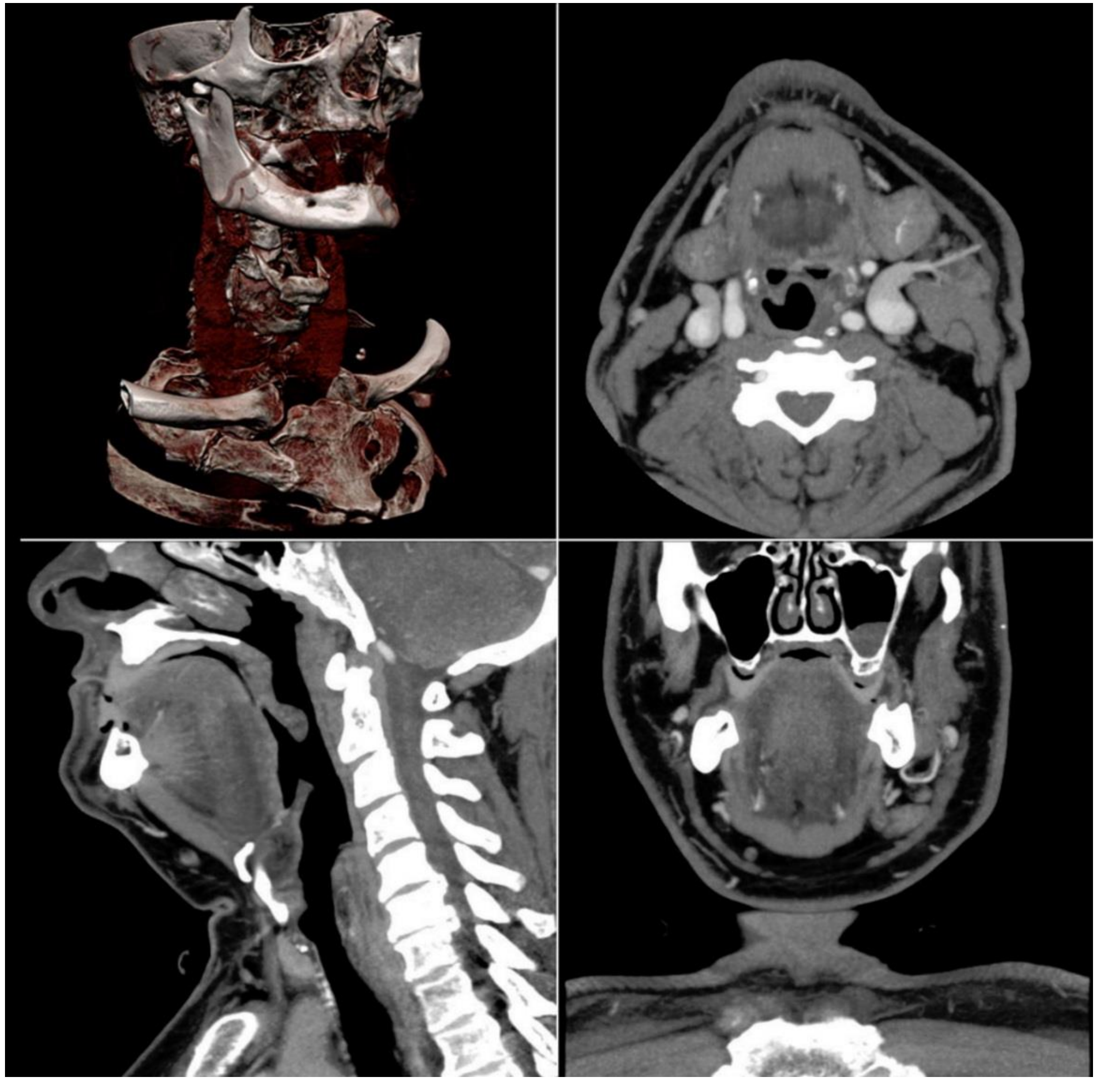
- For radiation protecttion direct exposure hazard is often evaluated using ionization chambers, while dosimeters are used to measure the radiation dose a person has been exposed to. X-ray spectra can be measured either by energy dispersive or wavelength dispersive spectrometers.

Imaging detectors for radiography: originally photographic plates and X-ray films; now mostly replaced by various digitizing devices.

Angiogram showing a transverse projection of the vertebrobasilar and posteriori celebral.



Fish bone pierced in the upper esophagus. Right image without contrast medium, left image during swallowing with contrast medium.



X-ray computed tomography (**X-ray CT**) or **computerized axial tomography scan** (**CAT scan**),

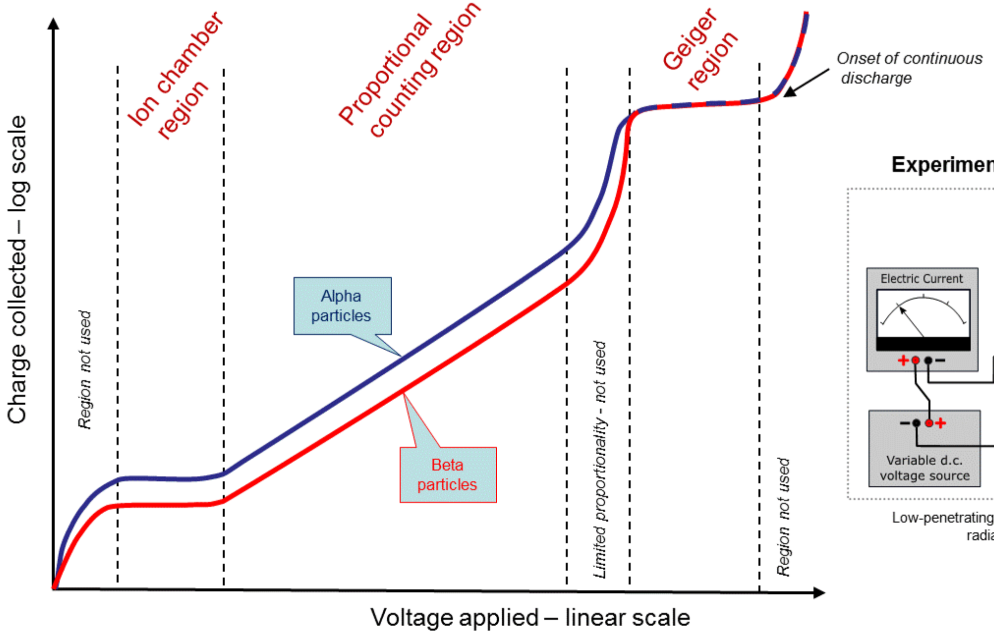
(makes use of computer-processed combinations of many **X-ray** images taken from different angles to produce cross-sectional images (virtual 'slices') of specific areas of a scanned object, allowing the user to see inside the object without cutting).

Typical screen layout for diagnostic software, showing one 3D and three MPR views.

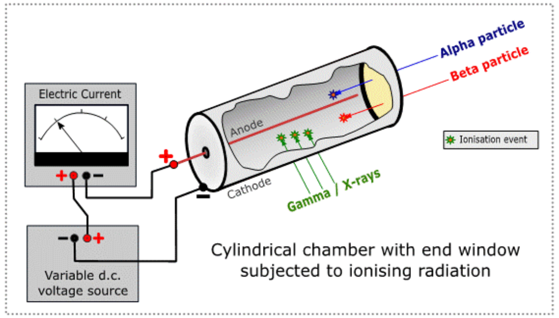
Practical Gaseous Ionisation Detection Regions

This diagram shows the relationship of the gaseous detection regions, using an experimental concept of applying a varying voltage to a cylindrical chamber which is subjected to ionising radiation. Alpha and beta particles are plotted to demonstrate the effect of different ionising energies, but the same principle extends to all forms of ionising radiation.
The ion chamber and proportional regions can operate at atmospheric pressure, and their output varies with radiation energy. However, in practice the Geiger region is operated at a reduced pressure (about 1/10th of an atmosphere) to allow operation at much lower voltages; otherwise impractically high voltages would be required. The Geiger region output does not differentiate between radiation energies.

Variation of ion pair charge with applied voltage



Experimental set-up of a cylindrical chamber



Low-penetrating radiation enters via an end window, but high-penetrating radiation can also enter via the cylinder side wall.

DS

Properties of X-rays

(electromagnetic wave)

- absorption (attenuation in material medium)
- scattering (coherent and fluorescent)
- refraction (air - solid body; $1\text{-}n = 10^{-6}$)
- total reflection (q = 10’ ÷ 30’)
- magneto-”optical” Kerr effect

Absorption (attenuation in material medium)

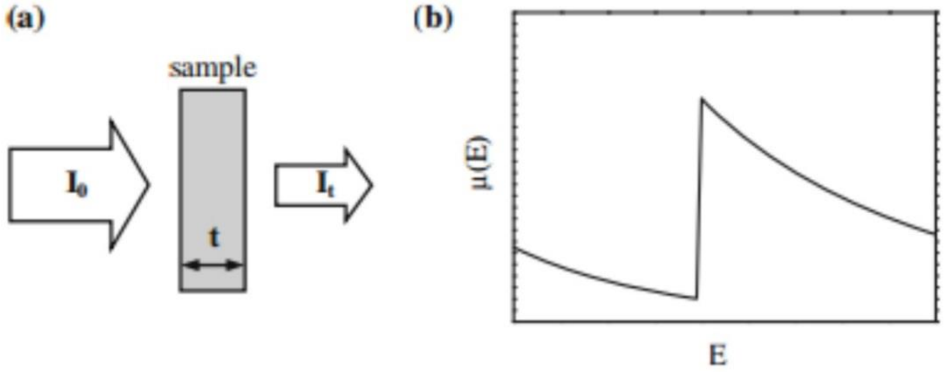
Attenuation (in some contexts also called **extinction**) is the gradual loss in intensity of any kind of flux through a medium

If X-rays of intensity I_0 are incident on a sample, the extent of absorption depends on the photon energy E and sample thickness t. According to Beer's Law, the transmitted intensity I_t is:

$$I_t\,(t) = I_0 e^{-\mu(E)t}$$

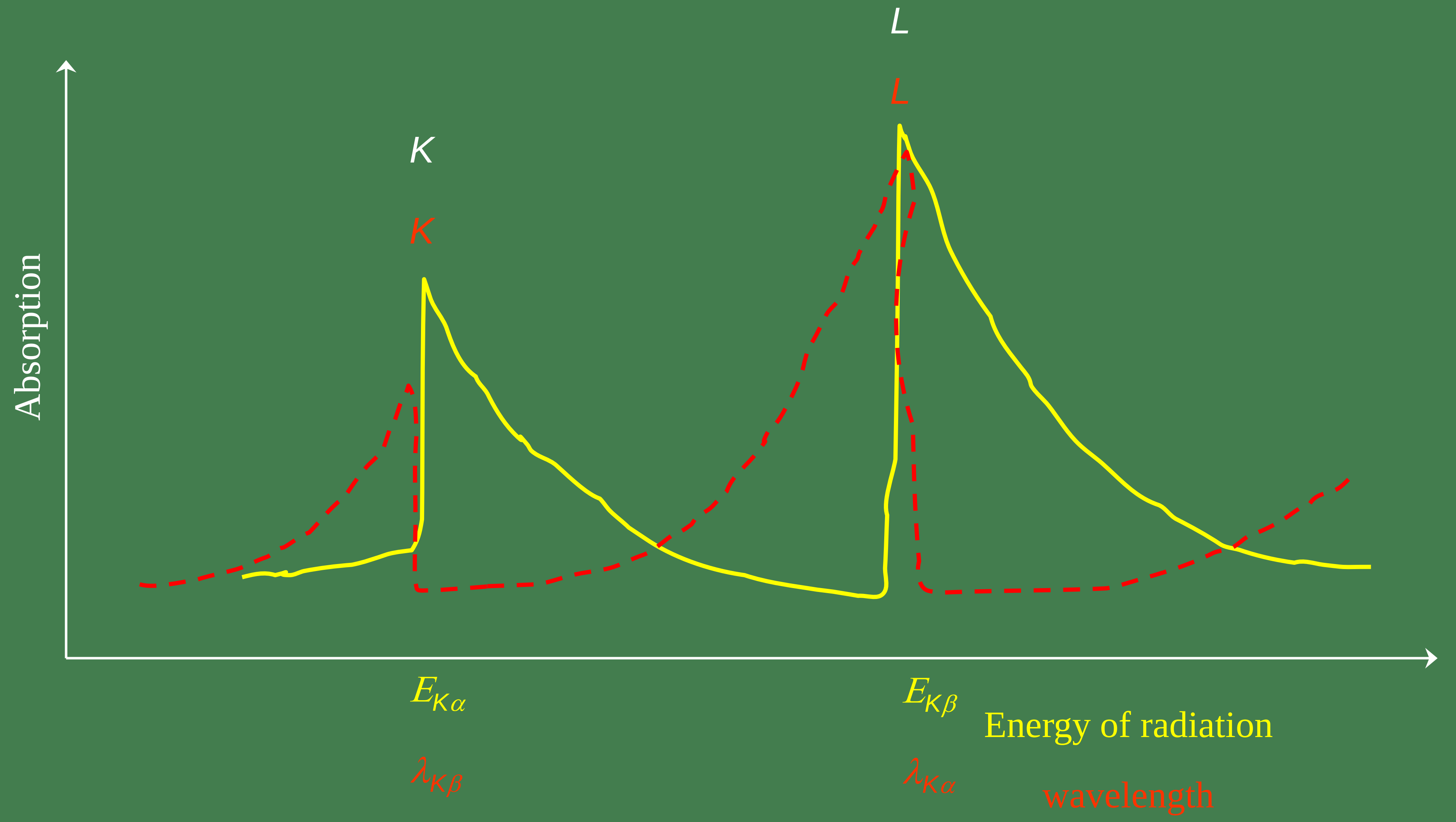
where μ(E) is the energy-dependent X-ray absorption coefficient.

Over large energy regions, μ(E) is a smooth function of the photon energy, **varying approximately as $\mu(E) \sim d\,Z^4/m\,E^3$** . Here d denotes the target density while Z and m are the atomic number and mass, respectively. Thus, μ(E) decreases with increasing photon energy. **If the latter equals or exceeds the binding energy of a core electron, however, a new absorption channel is available in which the photon is annihilated thereby creating a photoelectron and a core-hole.** This leads to a sharp increase in absorption coefficient. Above the absorption edge, the difference between the photon energy and the binding energy is converted into kinetic energy of the photoelectron and μ(E) continues to decrease with increasing photon energy. After a short time of the order of 10^{-15} s, the core-hole is filled by an electron from a higher energy state**. <u>The corresponding energy difference is released mainly via fluorescence X-ray or Auger electron emission.</u>**



(a) Schematic of incident and transmitted X-ray beam, (b) absorption coefficient μ(E) versus photon energy E around an absorption edge

Absorption edge



X-ray scattering techniques are a family of non-destructive analytical techniques which reveal information about the crystal structure, chemical composition, and physical properties of materials and thin films. These techniques are based on observing the scattered intensity of an x-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy. (**X-ray scattering is different from X-ray diffraction**).

Elastic scattering

Materials that do not have long range order may also be studied by scattering methods that rely on elastic scattering of monochromatic X-rays.

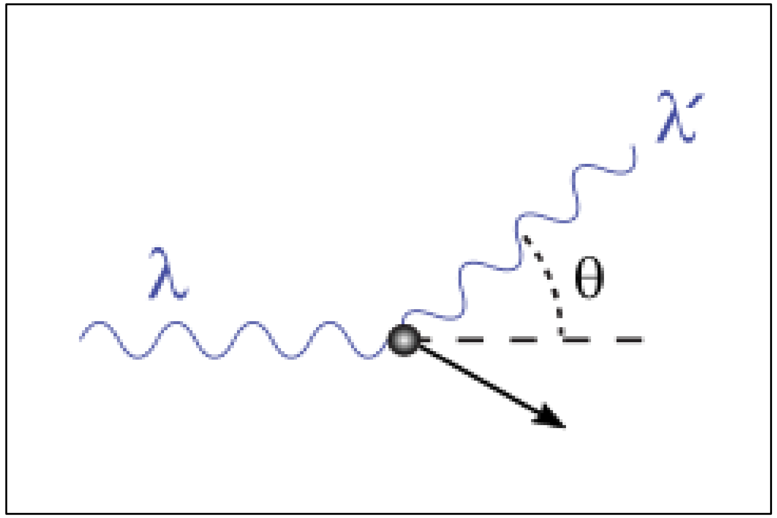
- **Small-angle X-ray scattering** (SAXS) is a technique where the elastic scattering of x-rays (wavelength 0.1 ... 0.2 nm) by a sample which has inhomogeneities in the nm-range, is recorded at very low angles (typically 0.1 - 10°). This angular range contains information about the shape and size of macromolecules, characteristic distances of partially ordered materials, pore sizes, and other data. SAXS is capable of delivering structural information of macromolecules between 5 and 25 nm, of repeat distances in partially ordered systems of up to 150 nm. USAXS (ultra-small angle X-ray scattering) can resolve even larger dimensions.
- X-ray reflectivity is an analytical technique for determining thickness, roughness, and density of single layer and multilayer thin films.
- Wide-angle X-ray scattering (WAXS), a technique concentrating on scattering angles 2θ larger than 5°.

Inelastic scattering

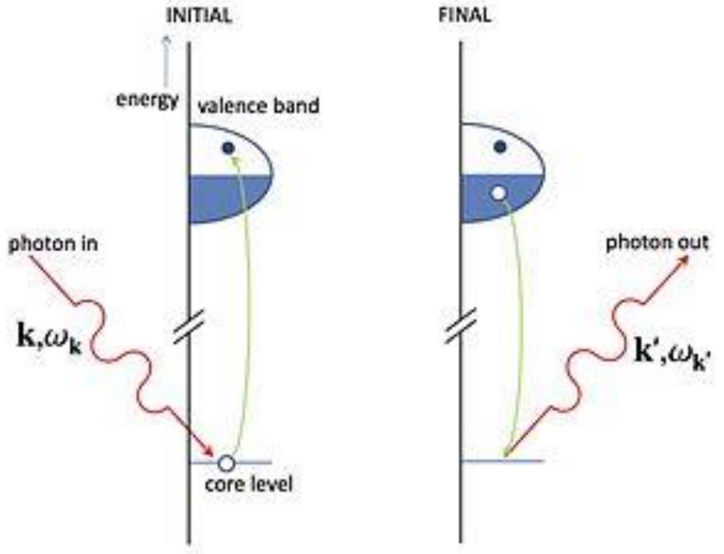
When the energy and angle of the inelastically scattered X-rays are monitored, scattering techniques can be used to probe **the electronic band structure of materials.** Inelastic scattering alters the phase of the diffracted x-rays, and as a result do not produce useful data for x-ray diffraction. Rather, inelastically scattered x-rays contribute to the background noise in a diffraction pattern.

- **Compton scattering** is the **inelastic scattering** of a photon by a charged particle, usually an electron. It results in a decrease in energy of the photon (which may be an x-ray or gamma ray photon), called the **Compton effect**. Part of the energy of the photon is transferred to the recoiling electron. **Inverse Compton scattering** exists, in which a charged particle transfers part of its energy to a photon.

A photon of wavelength λ comes in from the left, collides with a target at rest, and a new photon of wavelength λ' emerges at an angle Θ.



- **Resonant Inelastic X-ray Scattering** (RIXS) is an X-ray spectroscopy technique used to investigate the electronic structure of molecules and materials. It is a resonant technique because the energy of the incident photon is chosen such that it coincides with, and hence resonates with, one of the atomic x-ray absorption edges of the system. The resonance can greatly enhance the inelastic scattering cross section, sometimes by many orders of magnitude. The RIXS event can be thought of as a two-step process. Starting from the *initial* state, absorption of an incident photon leads to creation of an excited *intermediate* state, that has a core hole. From this state, emission of a photon leads to the *final* state. In a simplified picture the absorption process gives information of the empty electronic states, while the emission gives information about the occupied states. In the RIXS experiment these two pieces of information come together in a convolved manner, strongly perturbed by the core-hole potential in the intermediate state. RIXS studies can be performed using both soft and hard x-ray.



Direct RIXS process. The incoming x-rays excite an electron from a deep-lying core level into the empty valence. The empty core state is subsequently filled by an electron from the occupied states under the emission of an x-ray. This RIXS process creates a valence excitation with momentum k′ − k and energy ħω − ħω′ .

X-ray Raman scattering (XRS) is non-resonant inelastic scattering of x-rays from core electrons, in which a high-energy x-ray photon gives energy to a core electron, exciting it to an unoccupied state. The process is in principle analogous to x-ray absorption (XAS), but the energy transfer plays the role of the x-ray photon energy absorbed in x-ray absorption, exactly as in Raman scattering in optics vibrational low-energy excitations can be observed by studying the spectrum of light scattered from a molecule. Because the energy (and therefore wavelength) of the probing x-ray can be chosen freely and is usually in the hard x-ray regime, certain constraints of soft x-rays in the studies of electronic structure of the material are overcome. For example, soft x-ray studies may be surface sensitive and they require a vacuum environment. This makes studies of many substances, such as numerous liquids impossible using soft x-ray absorption. One of the most notable applications in which x-ray Raman scattering is superior to soft x-ray absorption is the study of soft x-ray absorption edges in high pressure. Whereas high-energy x-rays may pass through a high-pressure apparatus like a diamond anvil cel and reach the sample inside the cell, soft x-rays would be absorbed by the cell itself.

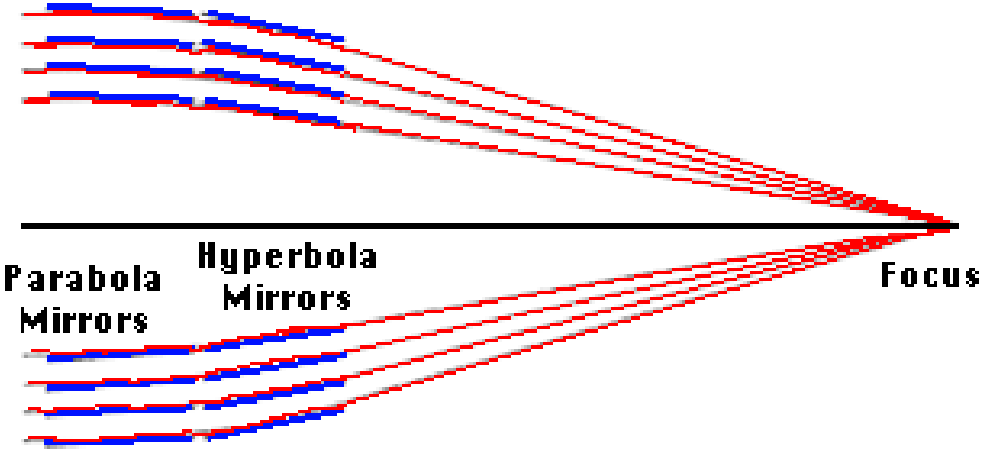
Refraction

X-ray optics is the branch of optics that manipulates X-rays instead of visible light. While lenses for visible light are made of transparent materials that can have a refractive index substantially larger than 1, for X-rays the index of refraction is slightly smaller than unity. The principal methods to manipulate X-rays are therefore by reflection, diffraction and interference. Examples of applications include X-ray microscopes and X-ray telescopes Refraction is the basis for the compound refractive lens, many small X-ray lenses in series that compensate by their number for the X-rays' minute index of refraction. The imaginary part of the refractive index, corresponding to absorption, can also be used to manipulate X-rays: one example is the pin-hole camera, which also works for visible light.

Reflection

The basic idea is to reflect a beam of X-rays from a surface and to measure the intensity of X-rays reflected in the specular direction (reflected angle equal to incident angle). It has been shown that a reflection off a parabolic mirror followed by a reflection off a hyperbolic mirror can lead to the focusing of X-rays. Since the incoming X-rays must strike the tilted surface of the mirror, the collecting area is small. It can, however, be increased by nesting arrangements of mirrors inside each other.

Several designs have been used in X-ray telescopes based on grazing incidence reflection: the Kirkpatrick-Baez design and a couple of designs by Wolter.



A **Wolter telescope** is a telescope for X-rays using only grazing incidence optics. Visible light telescopes are built with either lenses or parabolic mirrors at nearly normal incidence (that is, a nearly perpendicular angle of reflection). Neither works well for X-rays. Lenses for visible light are made of a transparent material with an index of refraction substantially different from one, but there is no equivalent material for x-rays. Conventional mirror telescopes work poorly in the X-rays as well, since the light hits the mirrors at near-normal incidence, where the X-rays are transmitted or absorbed, not reflected. **X-rays mirrors can be built, but only if the angle from the plane of reflection is very low (typically 10 arc-minutes to 2 degrees). The most commonly used reflective materials for X-ray mirrors are gold and iridium.**

Thank you

Project WND-POWR.03.02.00-00-I043/16

International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

Project co-financed by the European Union within the European Social Funds