

# Materials design & selection for Circular Economy

Module 3: Molecules and materials chemico-physical characterisation

Academic year 2026/2027

Lecturer: **Paolo Dolcet**

# Module's content

---



- This module aims at supplying the physico-chemical basics to the students towards analytical approaches to characterize molecules and materials in terms of:
  - Composition
  - Structural/morphology
  - Functional properties
- Discussed techniques:
  - UV/Vis and vibrational spectroscopies
  - Thermal gravimetry analysis and differential scanning calorimetry
  - Transmission and scanning electron microscopies

# Contacts

---



**Paolo Dolcet**

Dipartimento di Scienze Chimiche

Via Marzolo 1

35131 Padova

Email: [paolo.dolcet@unipd.it](mailto:paolo.dolcet@unipd.it)

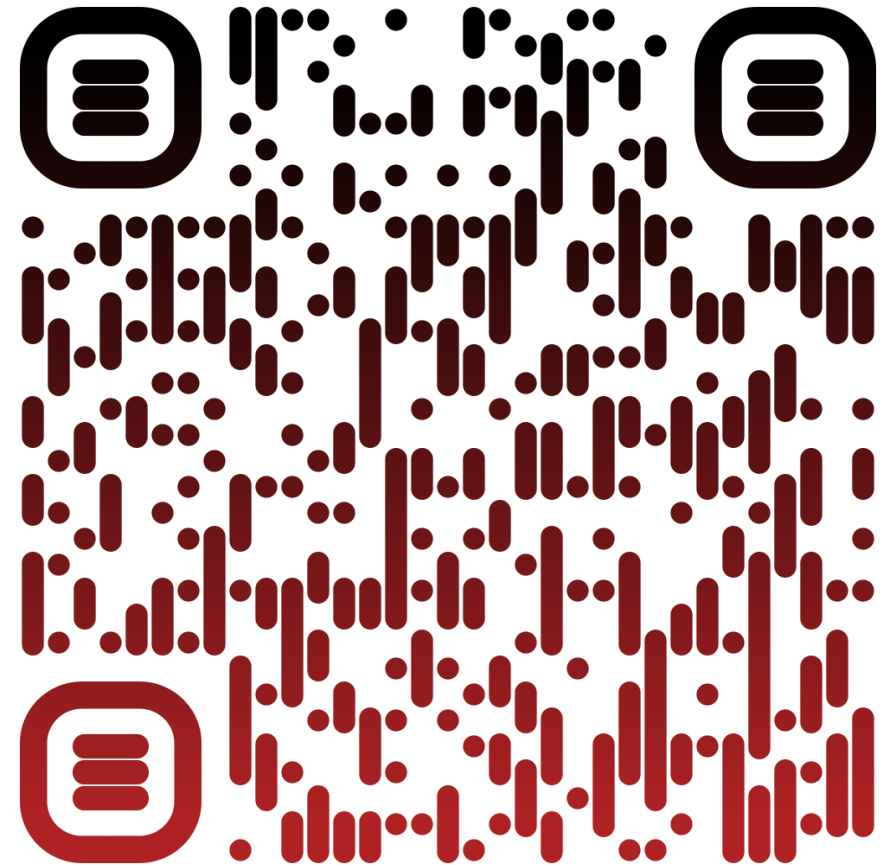
Appointments by email (please use your institutional email address  
name.familyname@studenti.unipd.it)

# Teaching material

---



- Lectures' slides will be available on Moodle:  
<https://stem.elearning.unipd.it/course/view.php?id=17043>
- Suggested textbooks
- Yang, *Materials Characterization: Introduction to Microscopic and Spectroscopic Methods*, Wiley 2013([link](#))
- Sultan, *Practical Guide to Materials Characterization: Techniques and Applications*, Wiley 2022 ([link](#))



# Why need to characterize

---



**Identification of unknown/mixed materials:** determine waste type, additives, contaminants



**Track degradation:** reuse and recycle can change structure and properties



**Quality control and compliance** along the supply chain (purity, contaminants)



Support **process decisions:** guide material choice and determine recycling route

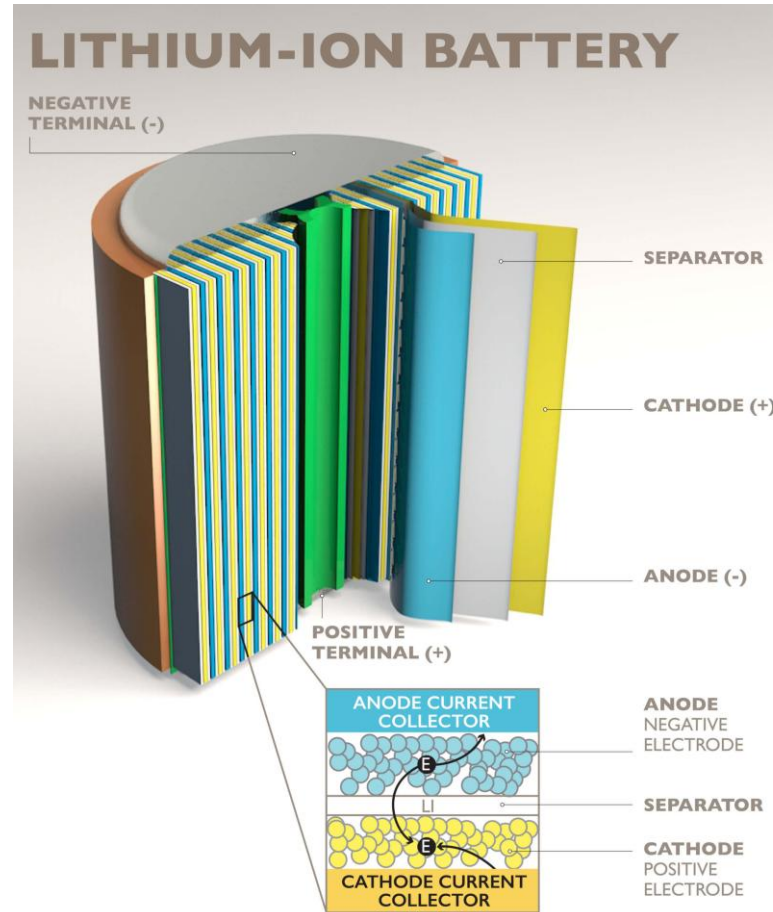
# An example: spent Li battery



“What structural phase transition have occurred?”

“What is the oxidation state of the metals?”

“Are there structural failures in the electrodes?”



“What is the degree of transition metal dissolution?”

“What impurities are present in the black mass?”

# How to choose a technique

---



## **What is the question?**

Identify,  
composition,  
structure or  
performance?

## **What is the scale?**

Molecular, bulk,  
surface or  
microstructure?

## **Can I use a destructive technique?**

Can the sample be  
consumed or must it  
be preserved?

## **Am I limited by sample preparation?**

What form, quantity  
and preparation are  
feasible?

# Spectroscopy

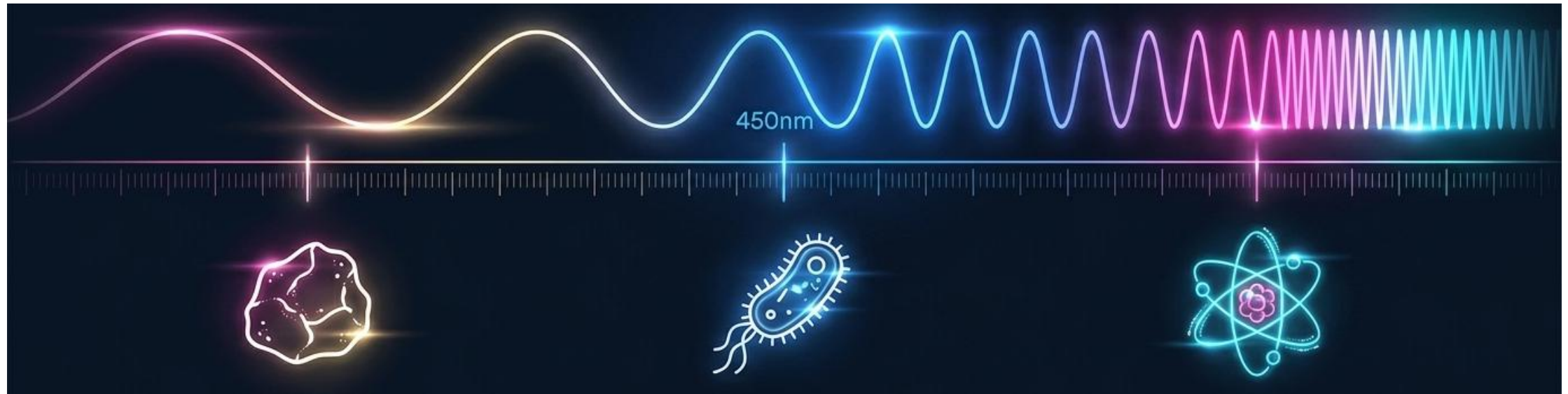
---



- **Spectroscopy** is a general term for the science investigating the interactions of radiation with matter
- **Spectrometry** (and spectrometric methods) refer to the measurement of the intensity of radiation with a photoelectric transducer or other type of electronic device
- **Spectrophotometer** is a particular kind of spectrometer that allows quantitative analysis of light

# Radiation as key to the microscopic world

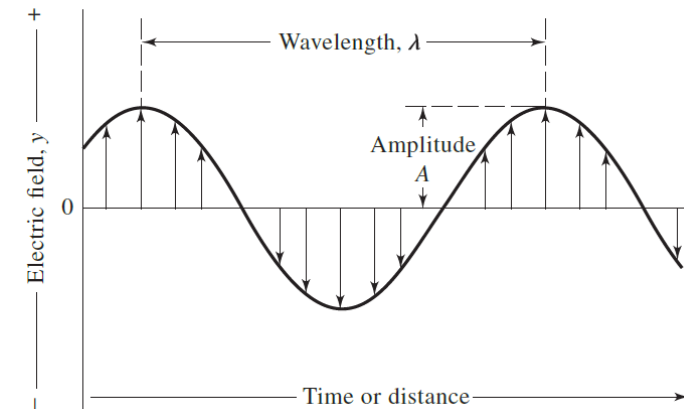
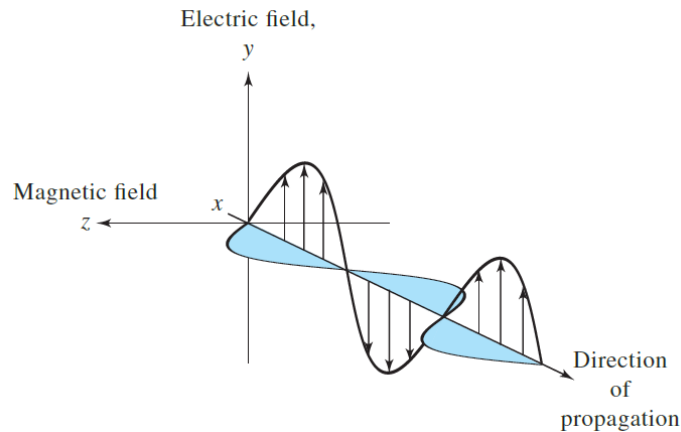
---



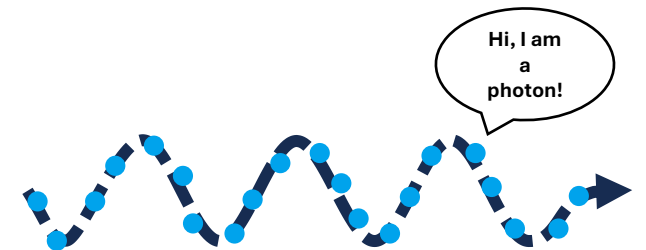
# Radiation – matter interaction



- **Electromagnetic radiation:** type of energy with several forms → most common ones: light and radiant heat



- Two components: Electric field + Magnetic field
- In first approximation: EM radiation = sinusoidal wave
- More correctly, EM is both a wave and a system of particles (photons)



# Radiation – wave characteristics



**Wavelength ( $\lambda$ ):**

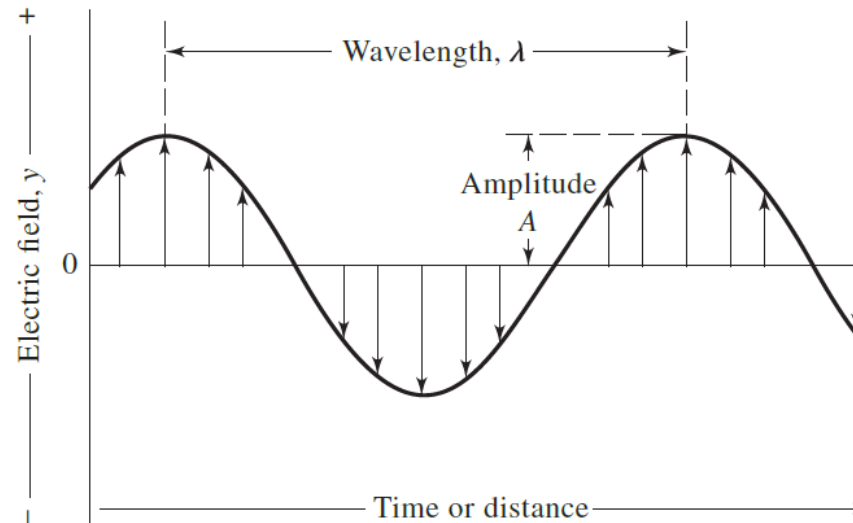
Linear distance between two successive equivalent points

**Period ( $p$ ):**

Distance in time between two successive equivalent points

**Power ( $P$ ):**

Energy for a given area per second



**Amplitude ( $A$ ):**

Length of vector corresponding to the maxima

**Frequency ( $\nu$ ):**

Number of oscillations per second,  $1/p$

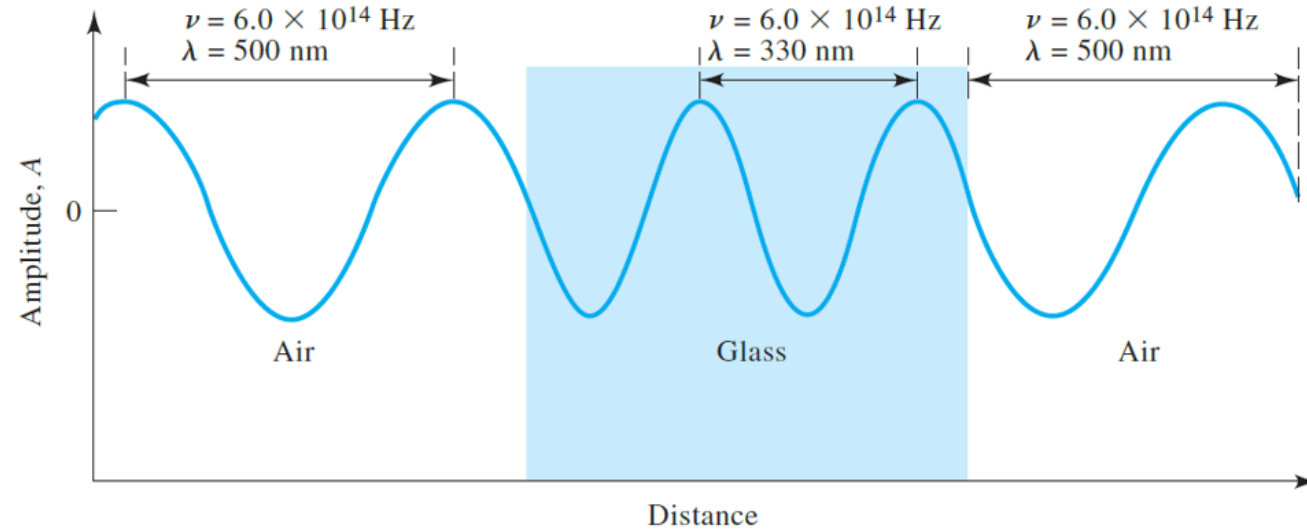
**Intensity ( $I$ ):**

Energy per unit solid angle

- **Wave behavior:**

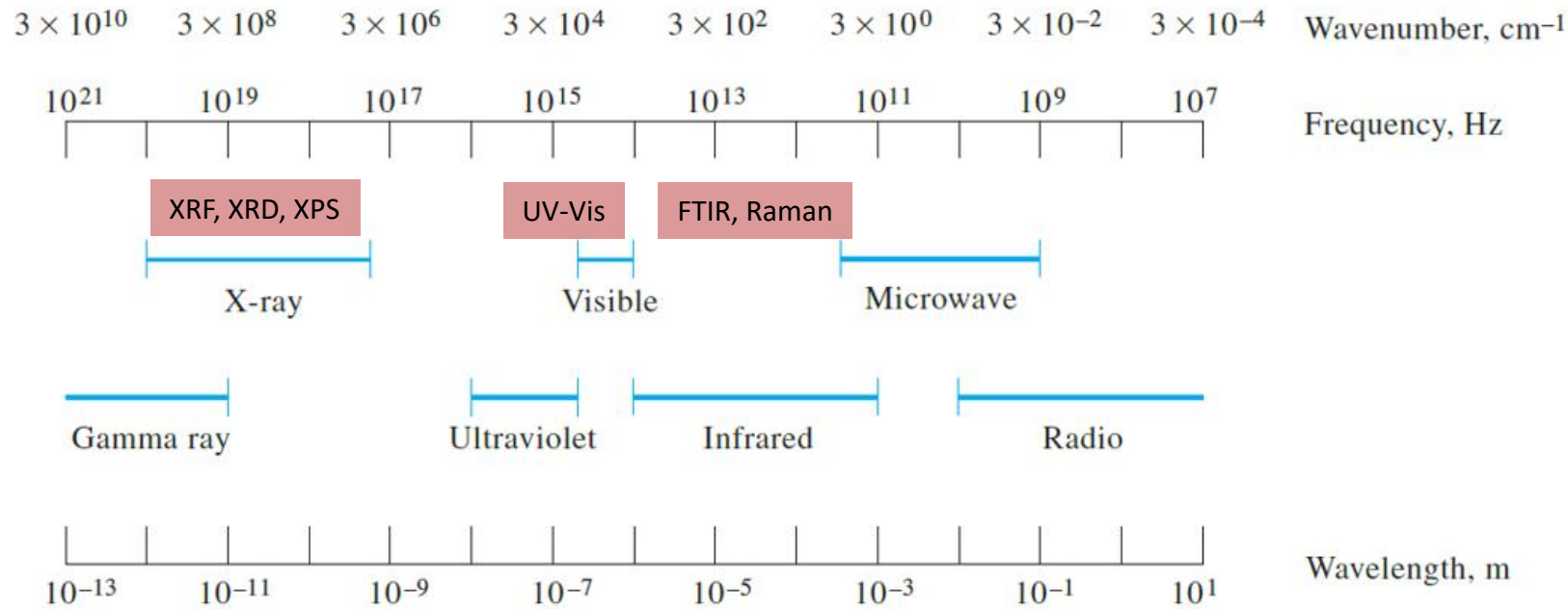
$$y = A \sin(\omega t + \varphi) = A \sin(2\pi \nu t + \varphi)$$

# Radiation – wave characteristics



- **Frequency** is determined by source only, invariant to the medium where EM travels through
- Propagation velocity  $v_i$  depends on the supporting medium:  $v_i = \nu \lambda_i$
- **Wavenumber**  $\tilde{\nu}$ : number of oscillations per unit distance ( $\text{cm}^{-1}$ ),  $1/\lambda$

# Electromagnetic spectrum



**Nuclear transitions**  
0.005 - 1.4 Å

**Inner electrons**  
0.1 - 100 Å

**Bonding electrons**  
10 - 780 nm

**Vibration/rotation  
of molecules**  
0.8 - 300 μm

**Rotation  
of molecules**  
0.8 - 375 mm

Energy depends on  $\lambda$  !

$$E = h\nu = hc/\lambda$$

Speed of light  $c = 2.99 \times 10^8 \text{ m/s}$

# Electromagnetic spectrum



Energy depends on  $\lambda$  !

$$E = h\nu = hc/\lambda$$

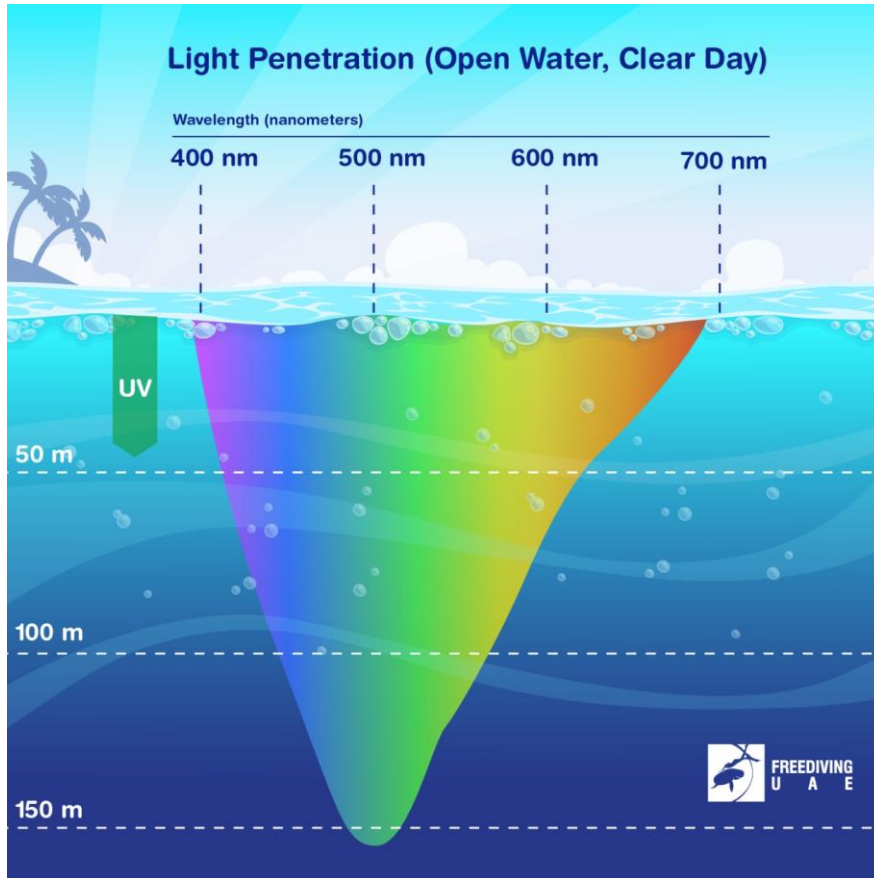
Planck constant  $h = 6.626 \times 10^{-34} \text{ J} \cdot \text{s} = 4.135 \times 10^{-15} \text{ eV/Hz}$

Speed of light  $c = 2.99 \times 10^8 \text{ m/s}$

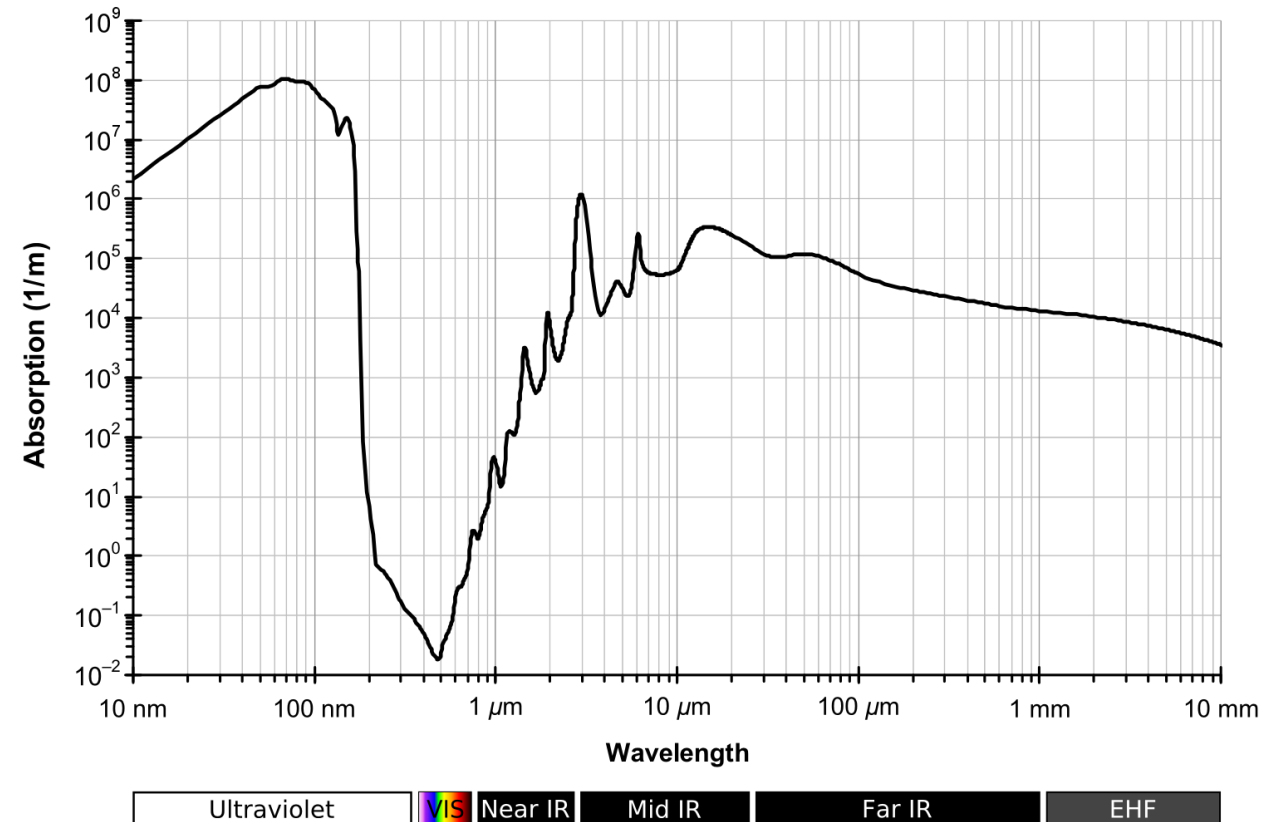
**Electronvolt (eV):** amount of kinetic energy gained by one  $e^-$  accelerating through an electric potential difference of one volt.

$$E = \frac{hc}{\lambda} = \frac{4.135 \times 10^{-15} \text{ eV/Hz} * 2.99 \times 10^8 \text{ m/s}}{\lambda} \approx \frac{1240 \text{ eV} \cdot \text{nm}}{\lambda}$$

# Electromagnetic spectrum



<https://freedivinguae.com/2020/07/what-happens-to-light-at-depth/>



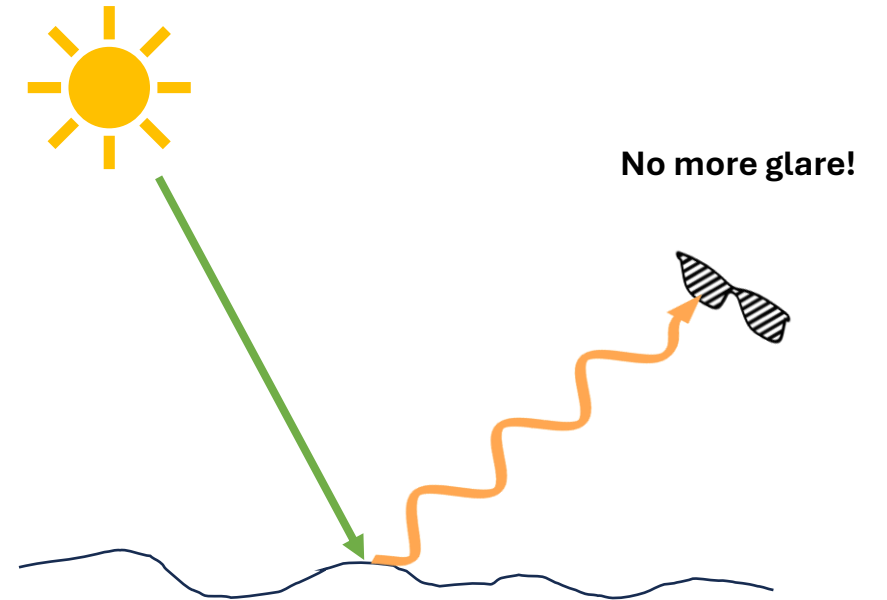
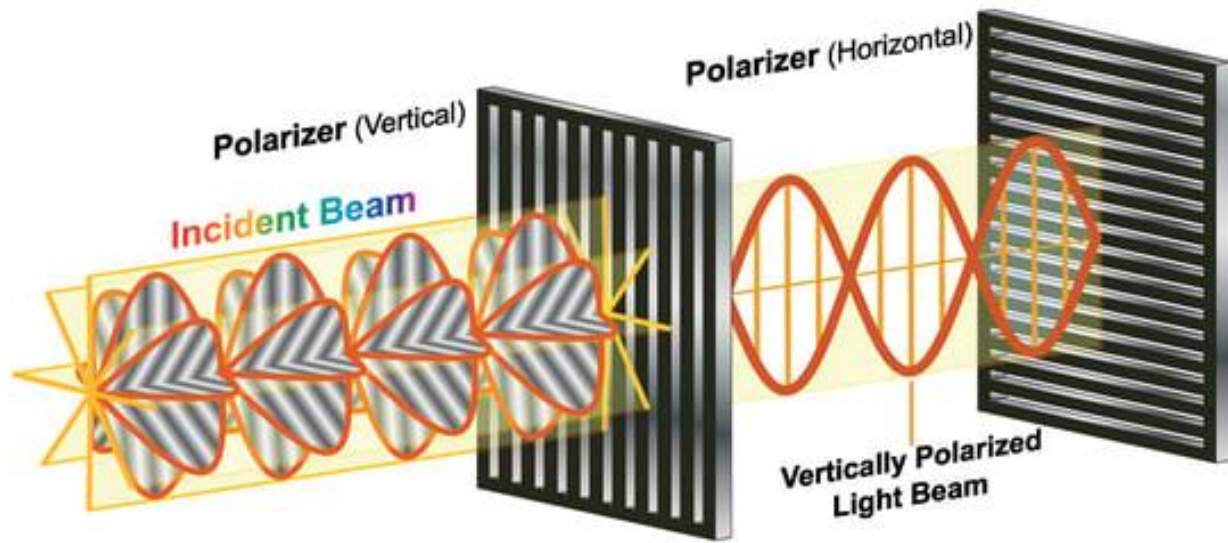
[https://en.wikipedia.org/wiki/Electromagnetic\\_absorption\\_by\\_water#/media/File:Absorption\\_spectrum\\_of\\_liquid\\_water.png](https://en.wikipedia.org/wiki/Electromagnetic_absorption_by_water#/media/File:Absorption_spectrum_of_liquid_water.png)

# Resolution and penetration depth



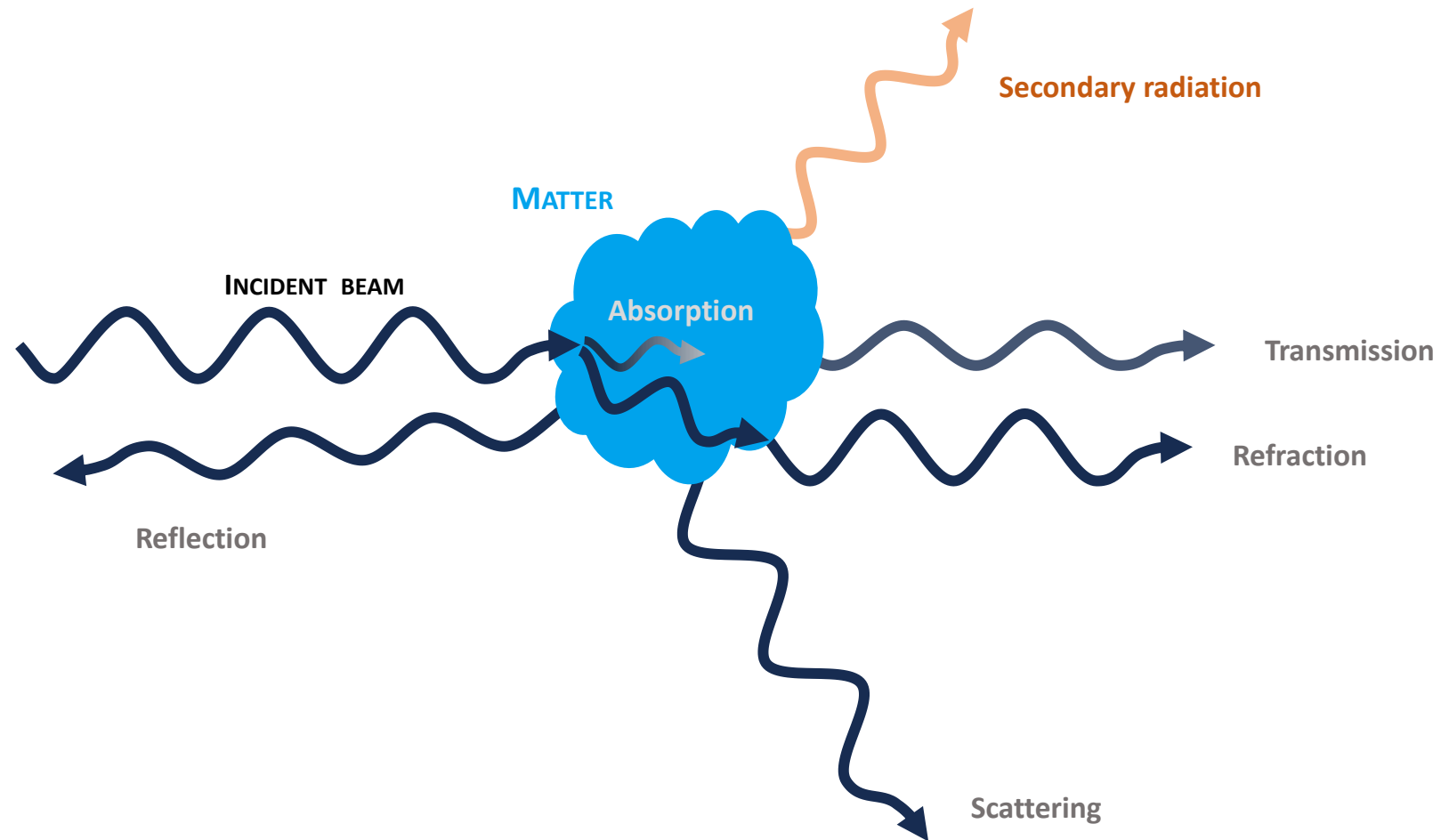
| Technique | Radiation     | Energy range | Spatial resolution | Penetration depth  |
|-----------|---------------|--------------|--------------------|--------------------|
| FTIR      | IR            | 0.01-0.5 eV  | $\mu\text{m}$ - mm | $\mu\text{m}$      |
| Raman     | Visible/NIR   | 1-3 eV       | $\mu\text{m}$      | $\mu\text{m}$      |
| UV-Vis    | UV-Visible    | 1.5-6 eV     | mm                 | $\mu\text{m}$ - mm |
| XPS       | X-ray         | 1-2 keV      | $\mu\text{m}$      | nm                 |
| SEM-EDX   | Electron beam | 0.5-30 keV   | nm - $\mu\text{m}$ | nm - $\mu\text{m}$ |
| XRF       | X-ray         | 1-50 keV     | mm - cm            | $\mu\text{m}$ - mm |
| XRD       | X-ray         | 5-20 keV     | mm                 | $\mu\text{m}$ - mm |
| TEM       | Electron beam | 100-300 keV  | $\text{\AA}$ - nm  | nm                 |

# Polarized radiation

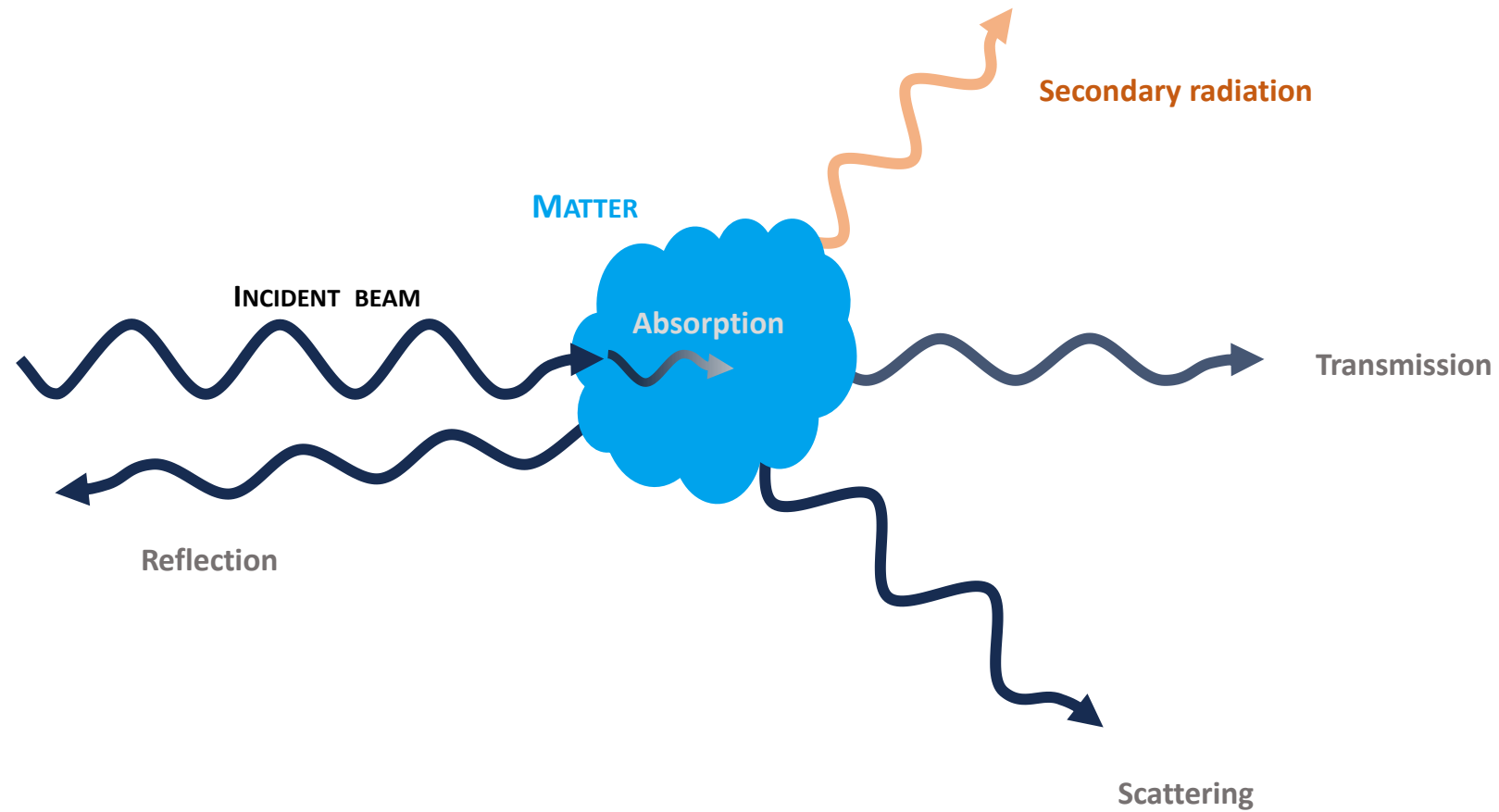


<https://sciotex.com/polarization-definition-when-to-use-in-machine-vision/>

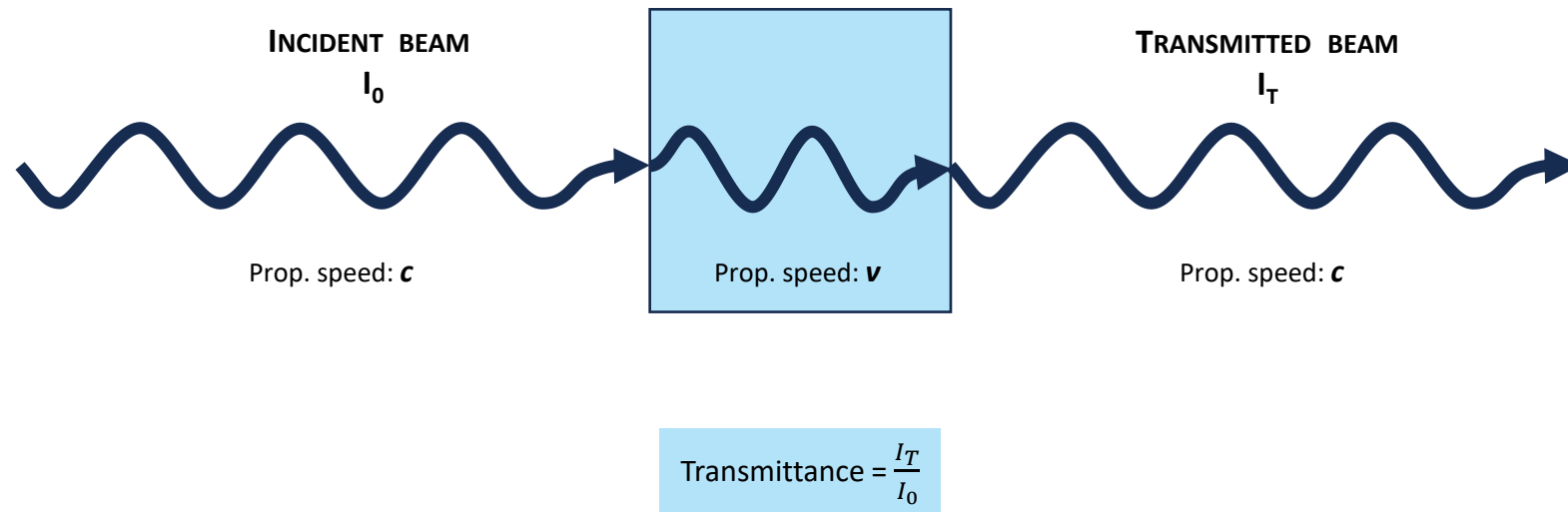
# Radiation – matter interaction



# Radiation – matter interaction



# Transmission



In a medium  $\neq$  vacuum, the interaction between the EM wave and the electron in the atoms **slow the light wave down**

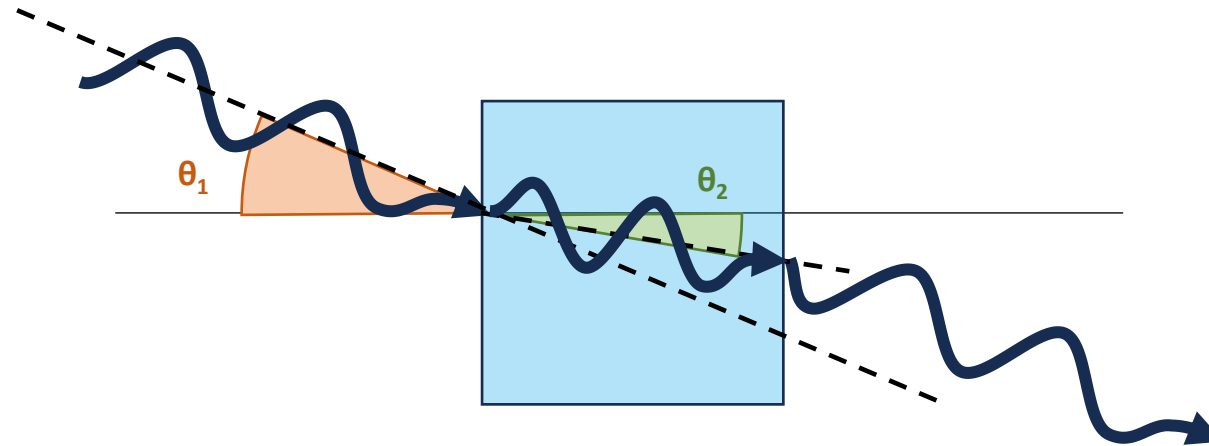
→ Index of refraction  $n = \frac{c}{v}$

P.S.: there is no frequency change, only the velocity of propagation is affected

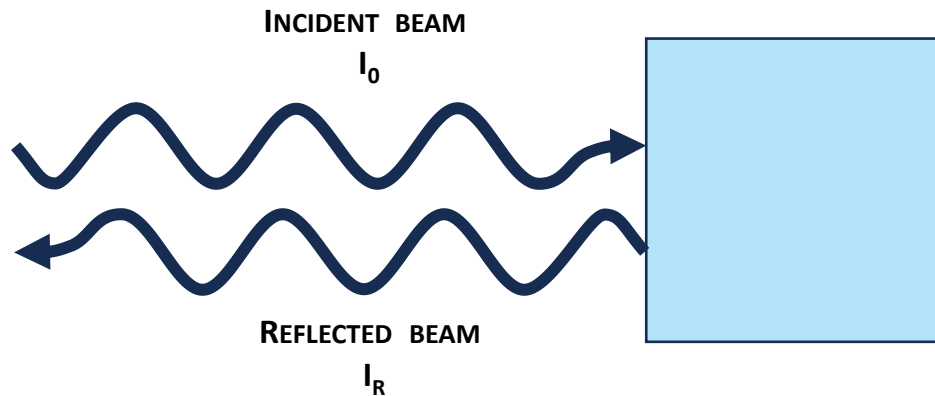
# Refraction & reflection



If the incident beam strikes at an angle  $\theta_1 \neq 0^\circ$ ,  
the angle inside the medium changes →  
**Refraction**



**Snell's law**  
$$n_1 \sin(\theta_1) = n_2 \sin(\theta_2)$$

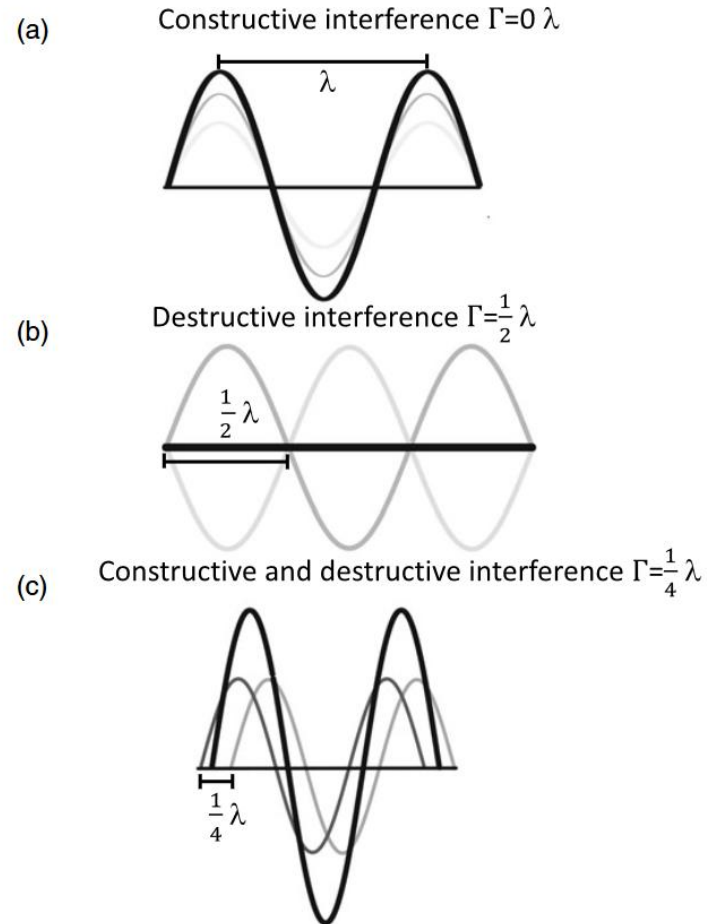


When an incident beam crosses the interface where  $n_1 \neq n_2$  →  
**Reflection**

$$\frac{I_r}{I_0} = \frac{(n_2 - n_1)^2}{(n_2 + n_1)^2}$$

The fraction of reflected radiation increases by  
increasing difference between  $n_1$  and  $n_2$

# Interferences

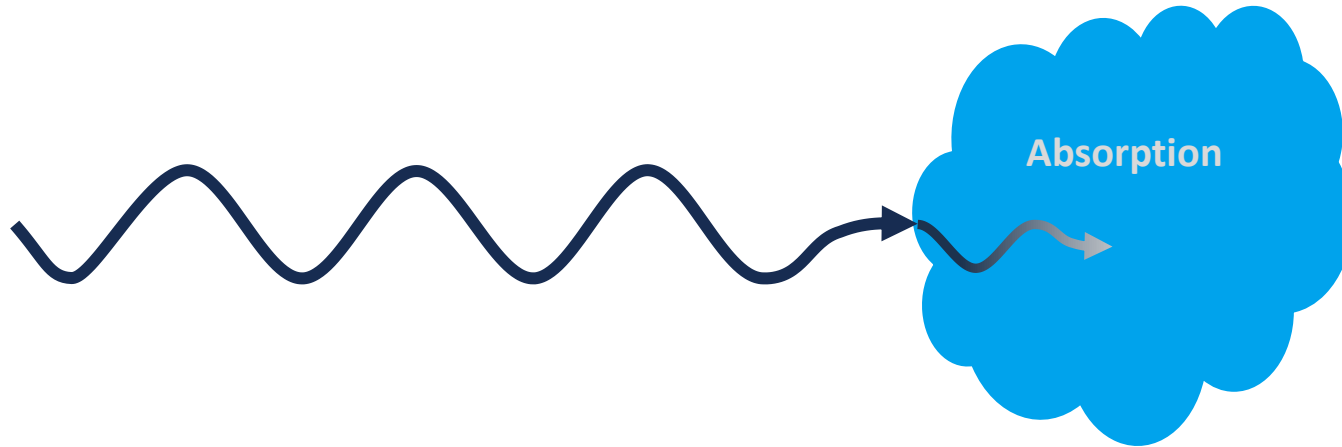


When two (or more) waves vibrate in the same plane, with same propagation direction, they will **interfere**

**Retardation  $\Gamma$ :** distance that one wave lags behind another wave

# Absorption

---

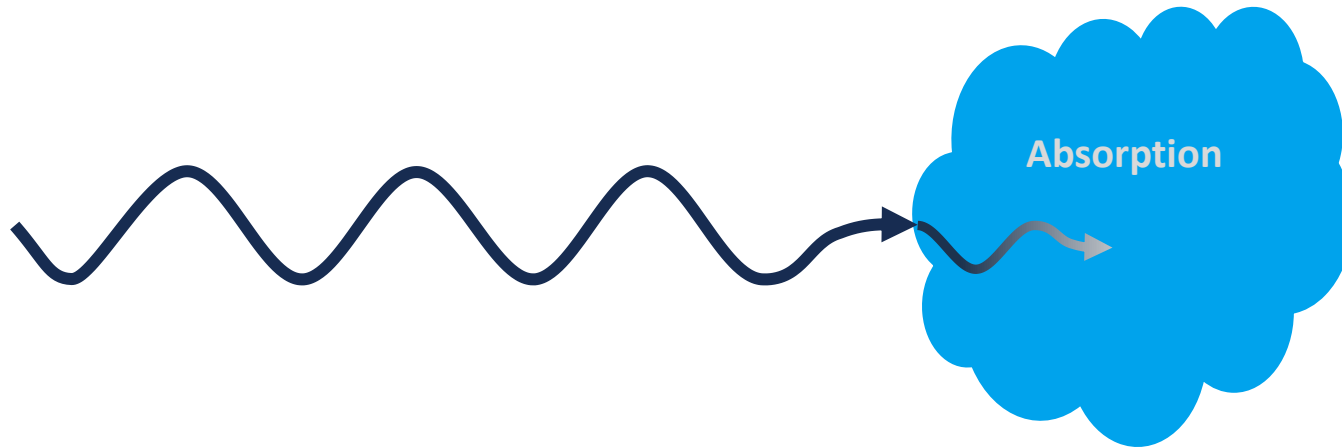


When there is energy transfer to the atoms/molecules/ions → **Absorption**

Absorption is a selective process → **specific frequencies** are removed

Absorption occurs if the energy of the radiation matches a specific atomic/molecular/ionic state

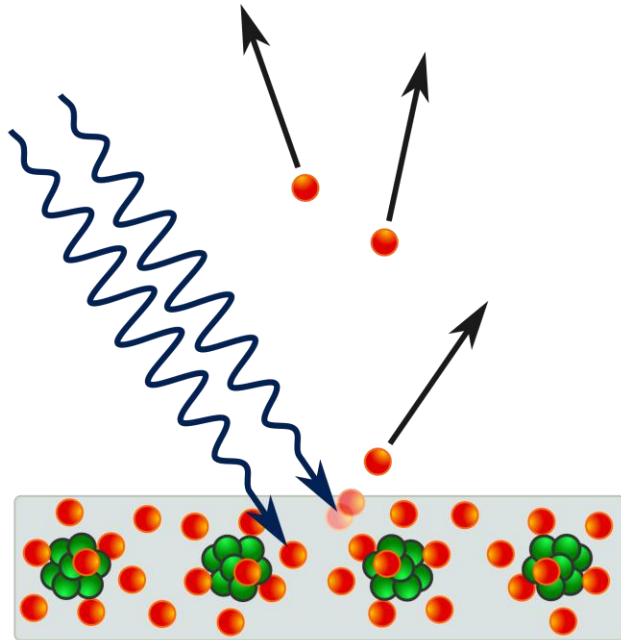
# Secondary radiation



Absorption process: EM wave  $\xrightarrow{\Delta E}$  sample

| $\Delta E$   | Secondary radiation              |
|--------------|----------------------------------|
| meV – 1.5 eV | Heat & vibrations                |
| 1.5 – 6 eV   | Fluorescence and phosphorescence |
| 6 – 2000 eV  | Electrons (photoelectric effect) |

# The photoelectric effect



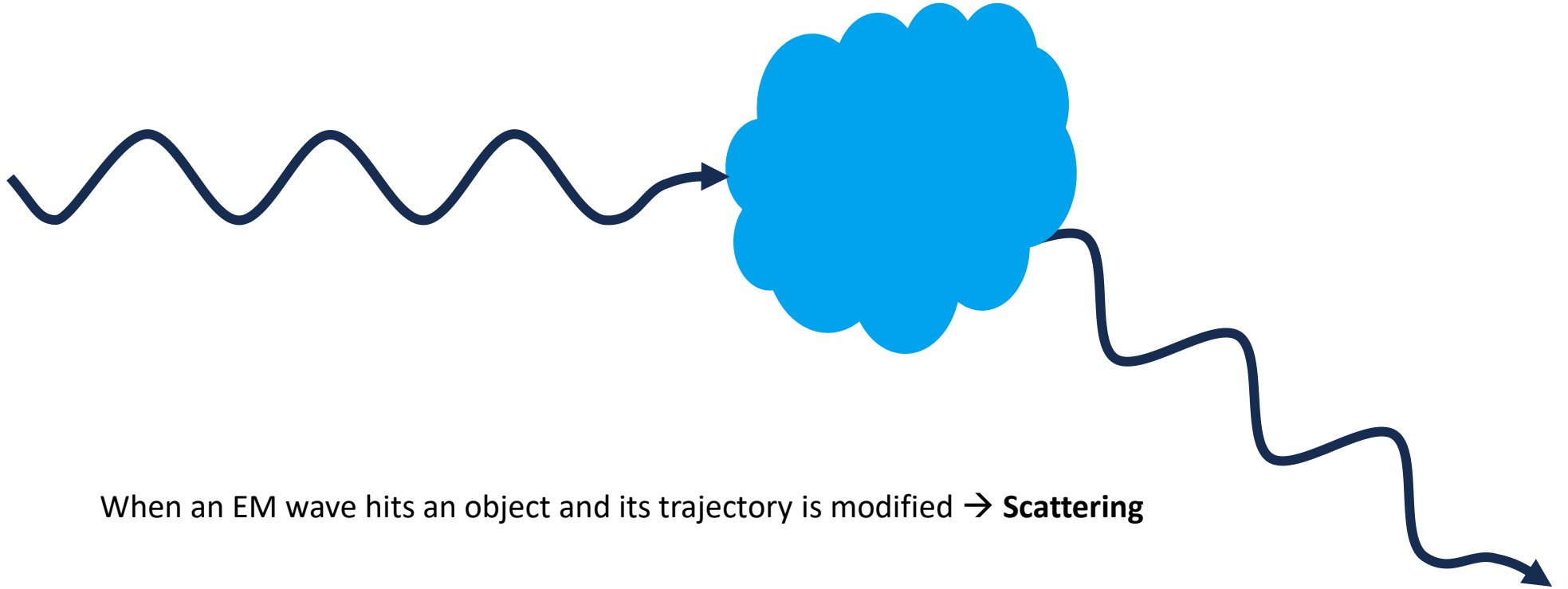
- When EM radiation shines on material, **emission of  $e^-$**
- Process occurs above specific frequencies
- Photons must have **particle-like behavior**
- Work function  $w$ : minimum energy required to remove an electron from the surface

$$E_k = h\nu - w - E_b$$

Einstein, 1905  
Nobel prize in 1921

# Scattering

---

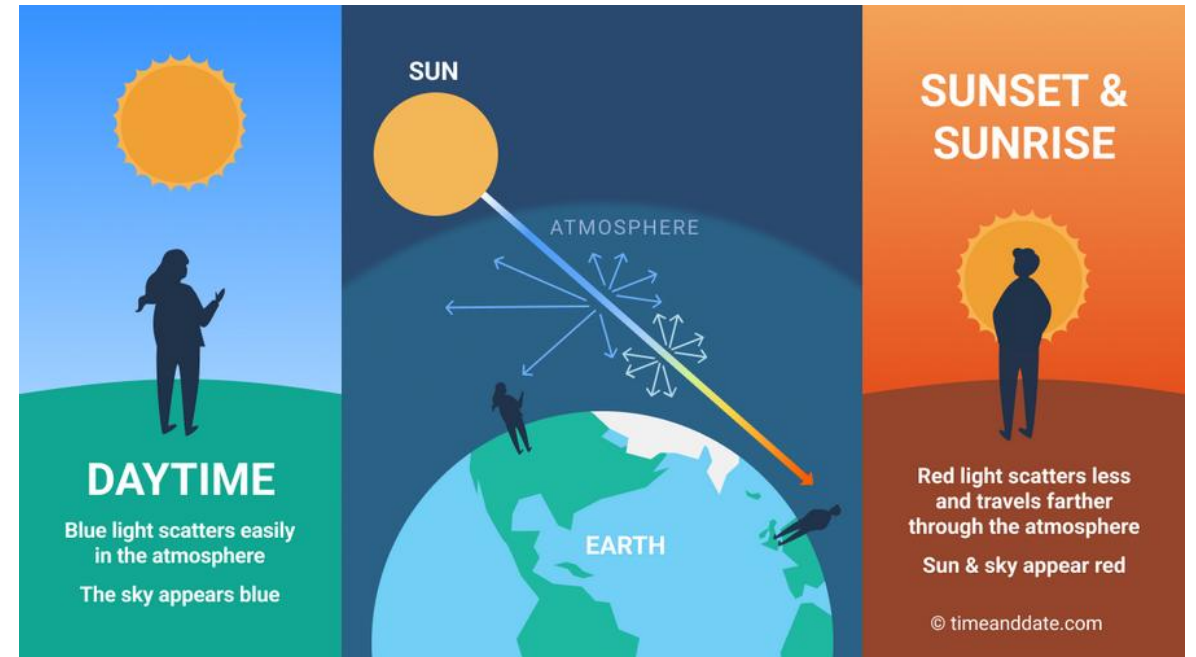


When an EM wave hits an object and its trajectory is modified → **Scattering**

Scattering can occur without exchange of energy (*elastic*) or with exchange of energy (*inelastic*)

If the object (or array of objects) has a size similar to  $\lambda$  → **Diffraction** (distinct ordered patterns)

# Scattering



$$I_{scat} \propto \frac{1}{\lambda^4}$$

The shorter the wavelength, the higher the scattering intensity