

# Elemental & Chemical Composition

## Energy Dispersive X-ray Spectroscopy

### (EDX/EDS)

- Attached with SEM/TEM
- Gives elemental composition
- Semi-quantitative

## X-ray Photoelectron Spectroscopy (XPS)

- Surface elemental composition
- Oxidation states
- Chemical bonding
- Example:

Ti  $2p_{3/2}$  peak  $\rightarrow$   $Ti^{4+}$  confirms  $TiO_2$



# Energy Dispersive X-ray Spectroscopy (EDX/EDS)

Energy Dispersive X-ray Spectroscopy (EDX or EDS) is an **elemental analysis technique** commonly coupled with **SEM and TEM** that identifies and estimates the **chemical composition of materials**. In nanomaterials research, EDX is widely used to:

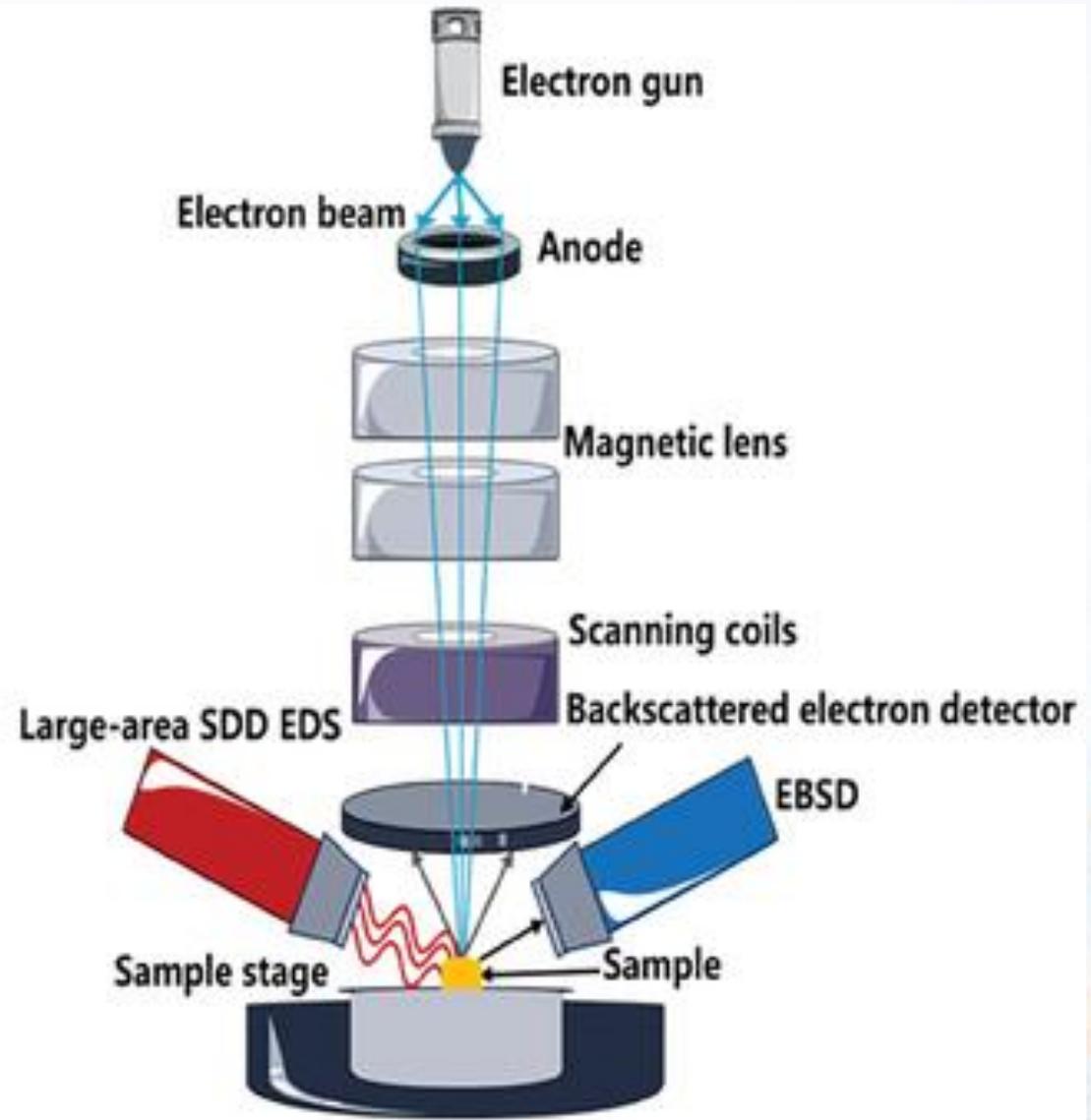
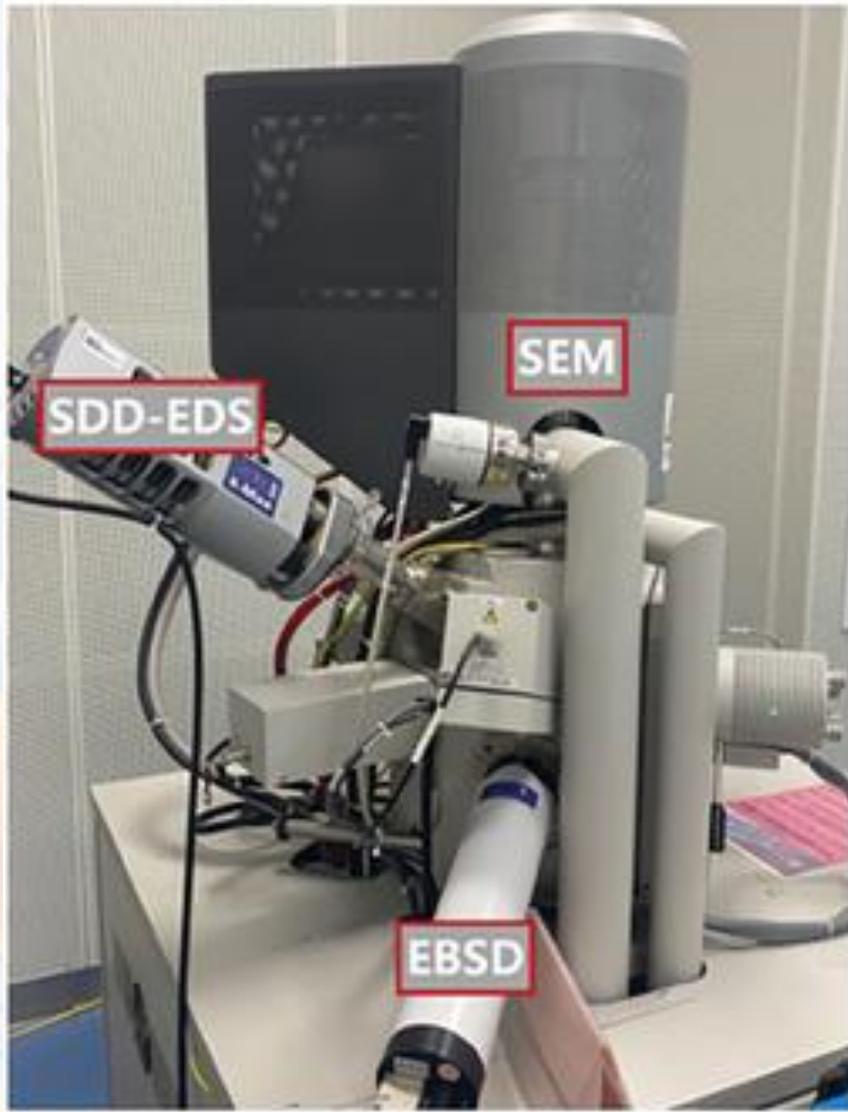
- Confirm presence of desired elements
- Detect impurities
- Verify doping
- Perform elemental mapping



# Basic Principle of EDX

- EDX is based on the interaction between a **high-energy electron beam** and atoms of the sample. It can be stepwise broken as:
  1. Electron beam strikes sample
  2. Inner shell electron is ejected
  3. Outer shell electron falls into vacancy
  4. Energy difference is released as **X-ray photon**
  5. X-ray energy is characteristic of the element
  6. Since each element has a unique electronic structure, the emitted X-rays have **unique energies**, acting as fingerprints.



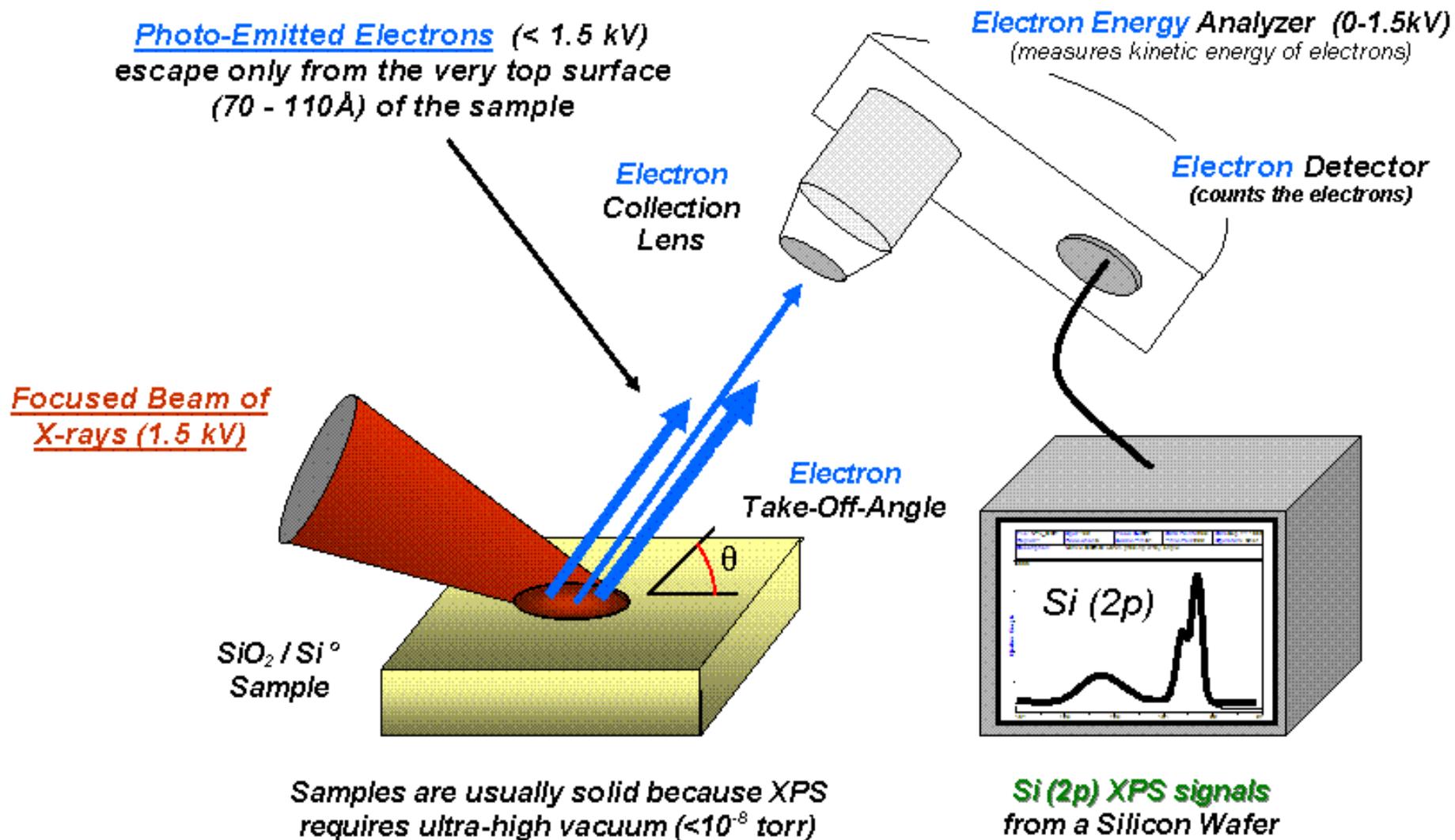


# X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

X-ray Photoelectron Spectroscopy (XPS), also known as **Electron Spectroscopy for Chemical Analysis (ESCA)**, is a **surface-sensitive analytical technique** used to determine:

- Elemental composition
- Chemical states (oxidation states)
- Surface stoichiometry
- Electronic environment of atoms
- XPS analyzes only the **top 1–10 nm** of a material's surface, making it extremely valuable for **nanomaterials**, where surface atoms dominate properties.



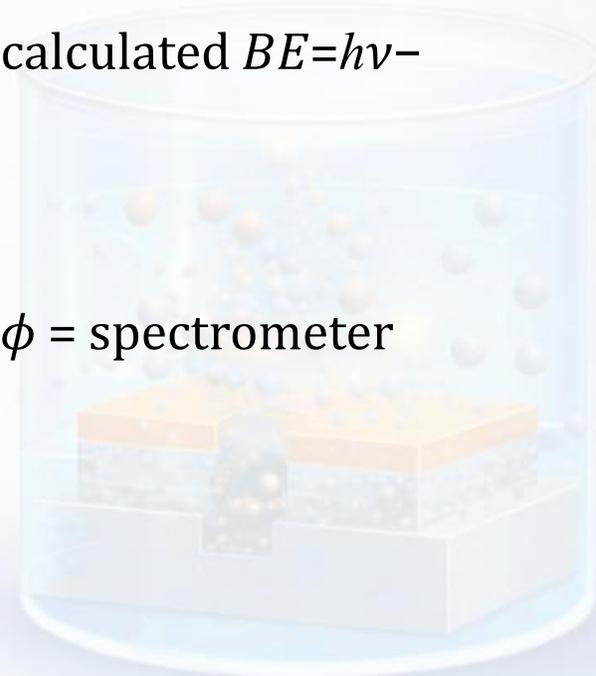


# Basic Principle of XPS

- XPS is based on the photoelectric effect.
- When a material is irradiated with monochromatic X-rays: X-ray photon strikes an atom
- Core electron is ejected
- Kinetic energy (KE) of emitted electron is measured Binding energy (BE) is calculated  $BE = h\nu -$

$$KE - \phi$$

- Where:  $h\nu$  = energy of incident X-ray, KE = kinetic energy of photoelectron,  $\phi$  = spectrometer work function
- Each element has unique binding energies, so peaks act as fingerprints.



# Why XPS Is Important for Nanomaterials

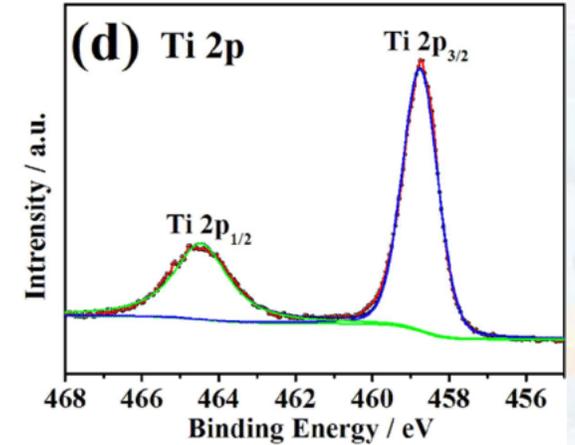
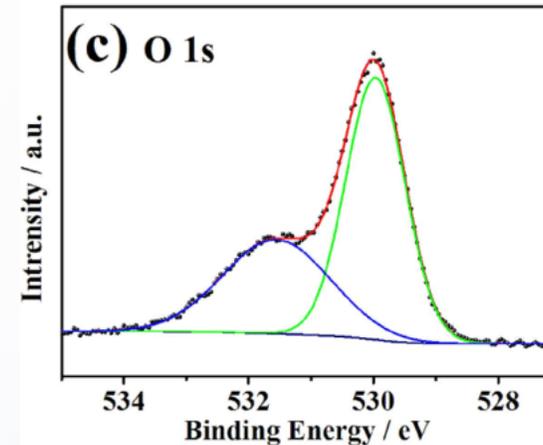
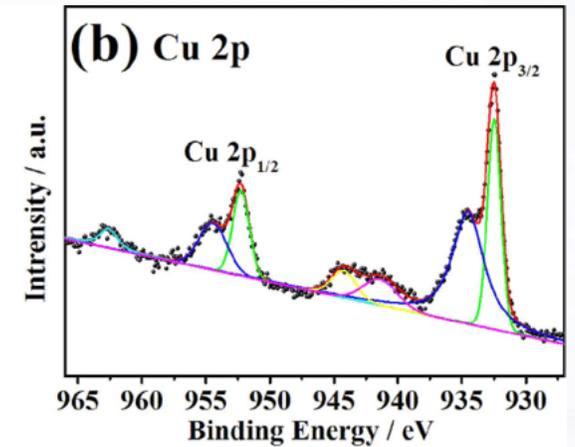
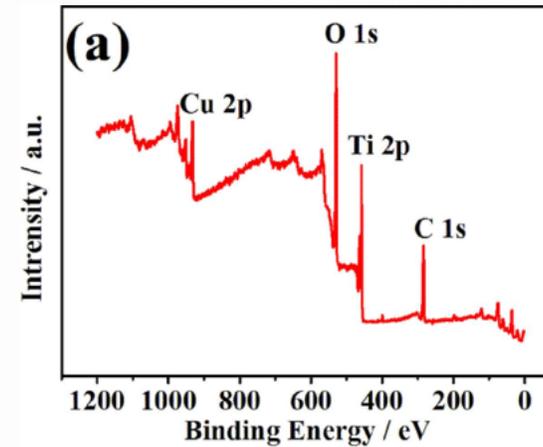
- Nanomaterials have very large **surface-to-volume ratio**
- Surface chemistry controls:
  - Catalysis
  - Photocatalysis
  - Sensing
  - Biocompatibility
- XPS directly probes surface composition and bonding → ideal technique.



# XPS Spectrum

## Intensity vs Binding Energy (eV)

- **Two Types of Spectra**
- **(A) Survey Spectrum**
- Shows all elements present
- **(B) High-Resolution Spectrum**
- Detailed peaks for specific elements
- Used for oxidation state analysis



# Applications in Nanomaterials

- ✓ Confirm composition
- ✓ Identify surface functional groups
- ✓ Detect dopants
- ✓ Study oxidation states
- ✓ Analyze catalysts
- ✓ Surface contamination detection



# Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a **vibrational spectroscopic technique** used to identify:

- Functional groups
- Chemical bonds
- Surface modifications
- Capping agents
- Molecular interactions

In nanomaterials research, FTIR is extremely important because nanoparticles possess a **large surface area**, and most chemical interactions occur at the surface.



# Basic Principle of FTIR

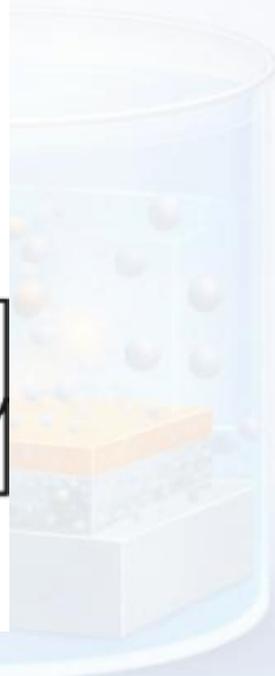
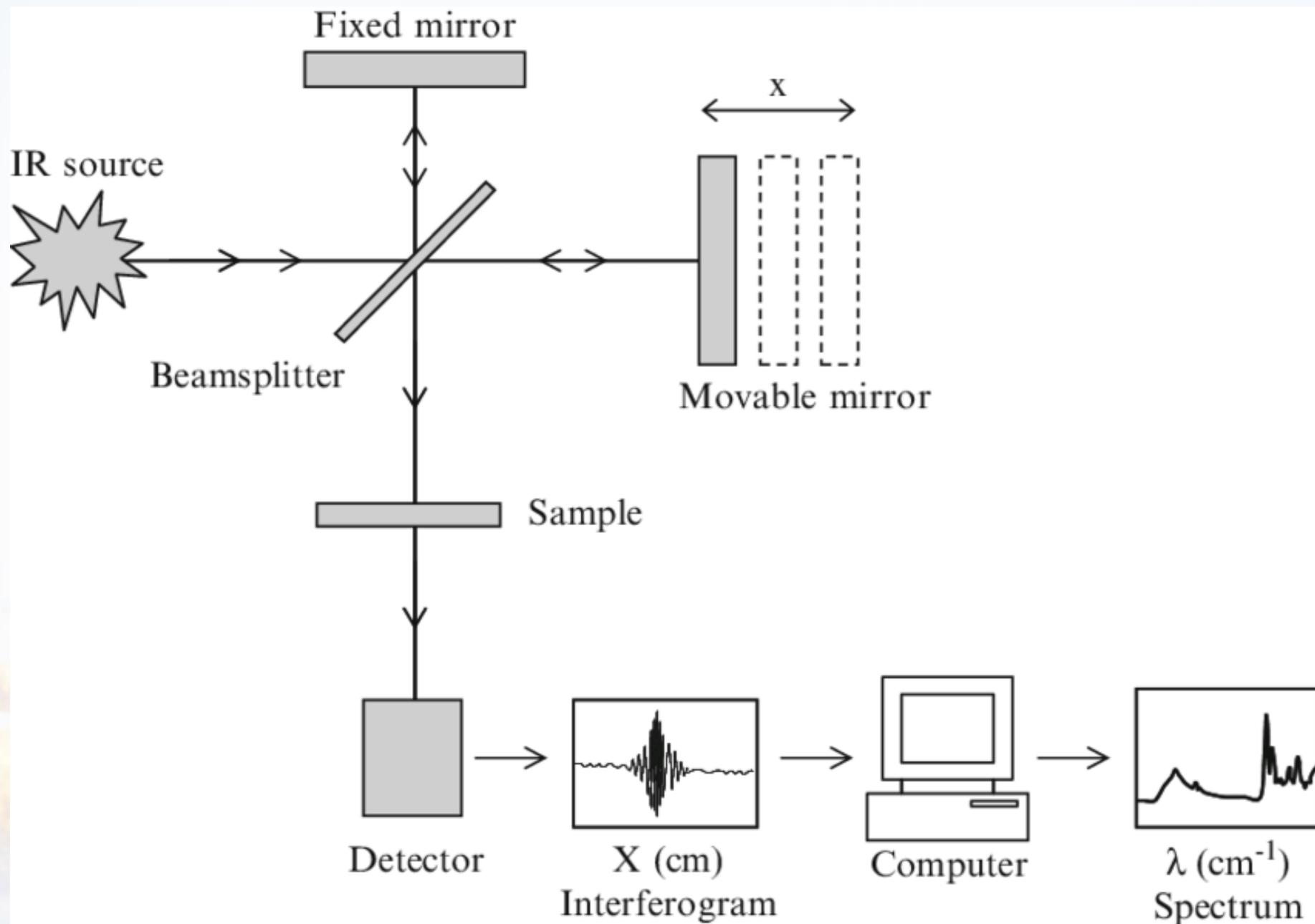
- FTIR is based on the **absorption of infrared radiation** by molecules.
- When IR radiation interacts with a molecule:
  1. Bonds vibrate (stretching or bending)
  2. If vibration causes a change in dipole moment → IR absorption occurs
- Each type of bond absorbs IR radiation at a **characteristic frequency**, producing a unique spectrum.



# Why It Is Called “Fourier Transform” Infrared

- Instead of scanning each wavelength separately:
- All wavelengths are collected **simultaneously**
- An interferogram is produced
- A mathematical **Fourier Transform** converts this into a spectrum
- This makes FTIR:
  - ✓ Faster
  - ✓ More sensitive
  - ✓ Higher signal-to-noise ratio





# Information Obtained from FTIR in Nanomaterials

- ✓ Presence of surface functional groups
- ✓ Capping or stabilizing agents
- ✓ Polymer or biomolecule coating
- ✓ Metal–oxygen bond formation
- ✓ Interaction between components in nanocomposites



## Advantages of FTIR

- ✓ Rapid
- ✓ Non-destructive
- ✓ Small sample quantity
- ✓ Simple operation
- ✓ Identifies functional groups

## Limitations

- ✗ Does not give elemental composition
- ✗ Overlapping peaks possible
- ✗ Low sensitivity for very dilute species

