Nanotechnology Course/ Ph-457 Lecture 6

Chapter 3:
Nanomaterials Characterization

By

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Nanomaterials Characterization

- **Structural Properties (XRD) to find crystallite size**

X-ray diffraction (XRD) is an effective technique for verifying the crystalline structure of materials. It identifies crystalline substances with crystal domains above **3-5 nm**. It is utilized to characterize the bulk crystal structure and chemical phase composition.

- The atomic planes of a crystal cause an incident beam of X-rays to interfere with one another as they leave the crystal. The phenomenon is called X-ray diffraction.
- The X-ray diffraction pattern of a pure substance is, therefore, like a fingerprint of the substance. It is based on the scattering of X-rays by crystals.

H.W/ How is X-ray production?

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- **Why is X-Ray important**

1- Determine the orientation of a single crystal or grain

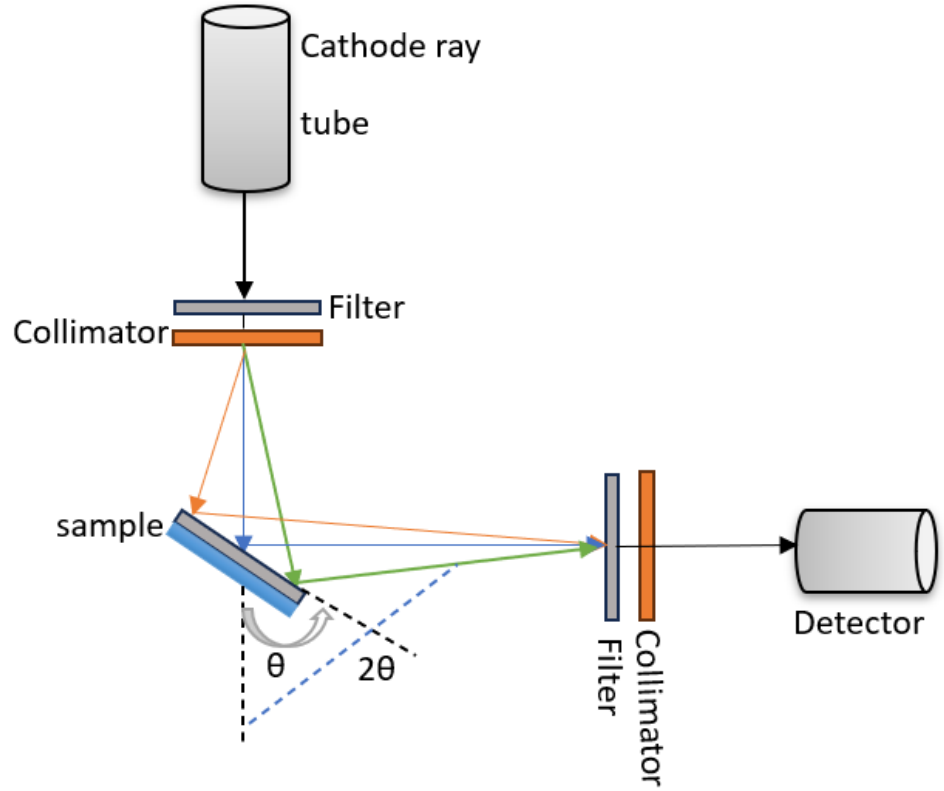
2- find the crystal structure of an unknown material

3-Measure the average spacing between layers or rows of atoms

4- measure the size, shape, and internal stress of small crystalline regions

❑ How diffraction work

- **Basic components of X-ray diffractometers**



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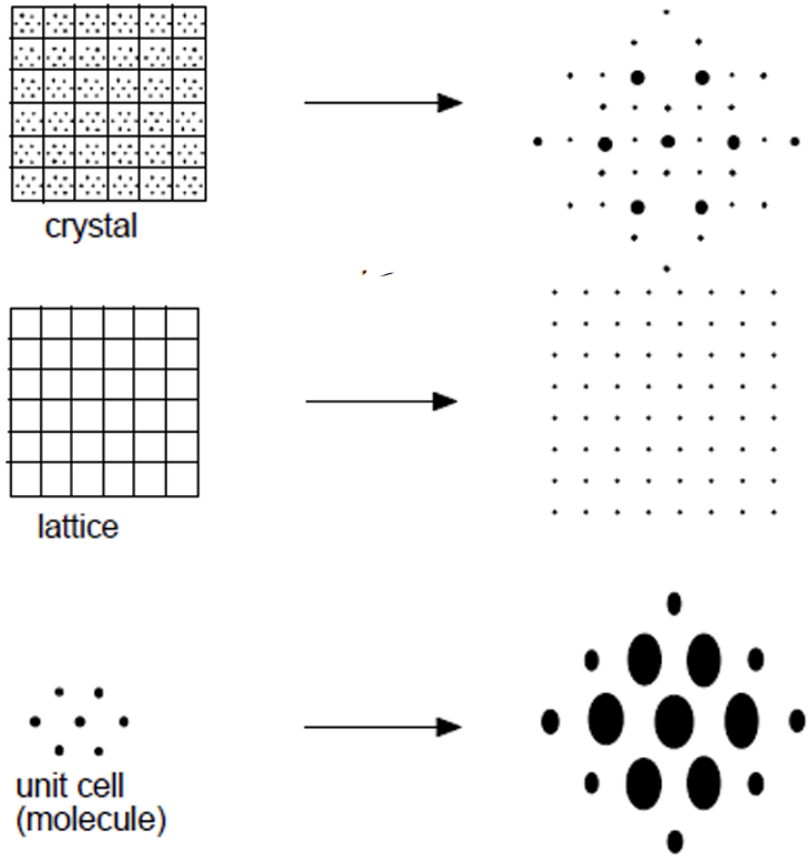
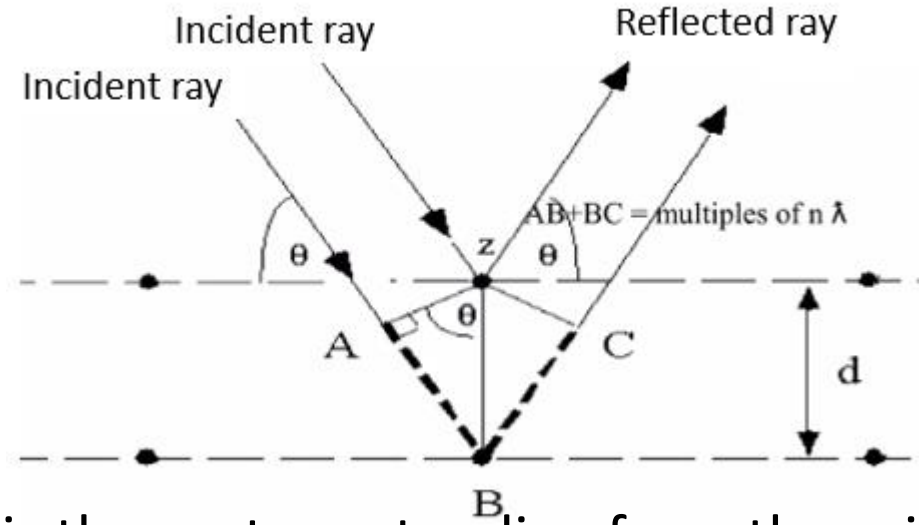
- **How diffraction work**



- **How diffraction works (Bragg low)**

$$\lambda = 2d_{hkl}sin\theta$$

*hkl is the miller indices



$n\lambda = AB + BC$

$AB = BC$

$n\lambda = 2AB$

$\sin\theta = AB/d$

$AB = d\sin\theta$

$n\lambda = 2d\sin\theta$

$\lambda = 2d\sin\theta$

d_{hkl} is the vector extending from the origin to the plane (hkl) and is normal to (hkl)

the vector d_{hkl} is used in Bragg law to determine where diffraction peaks will be observed.

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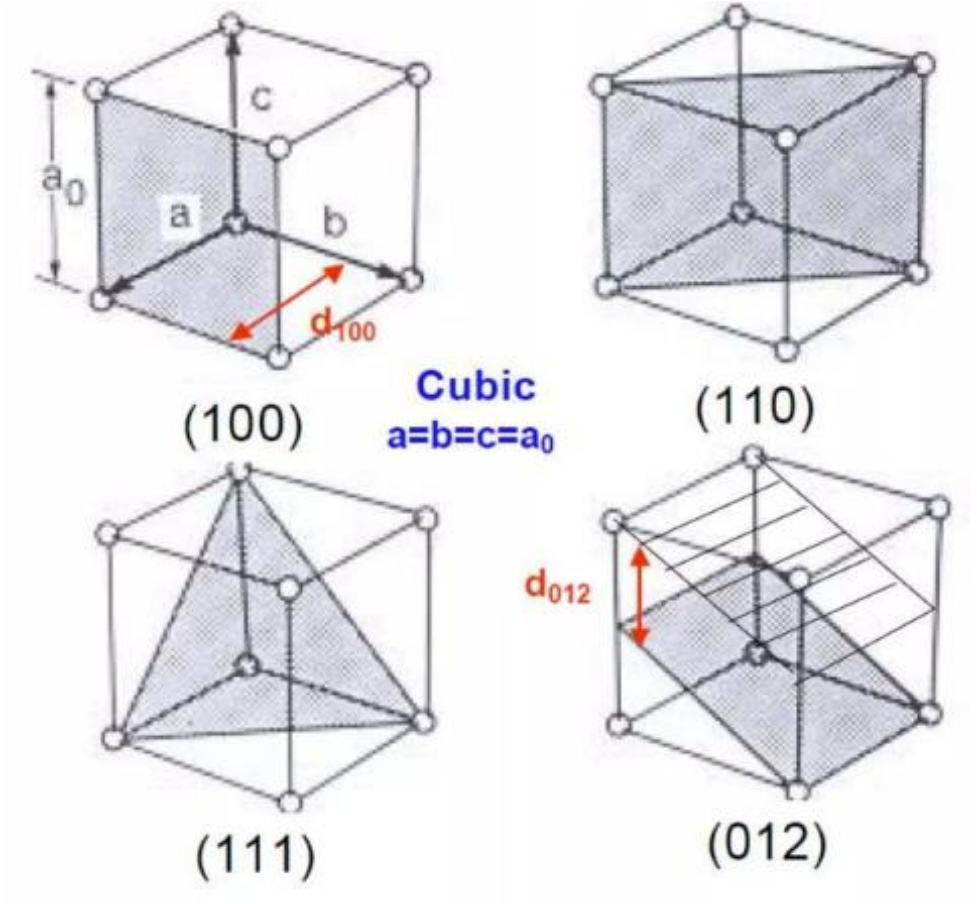
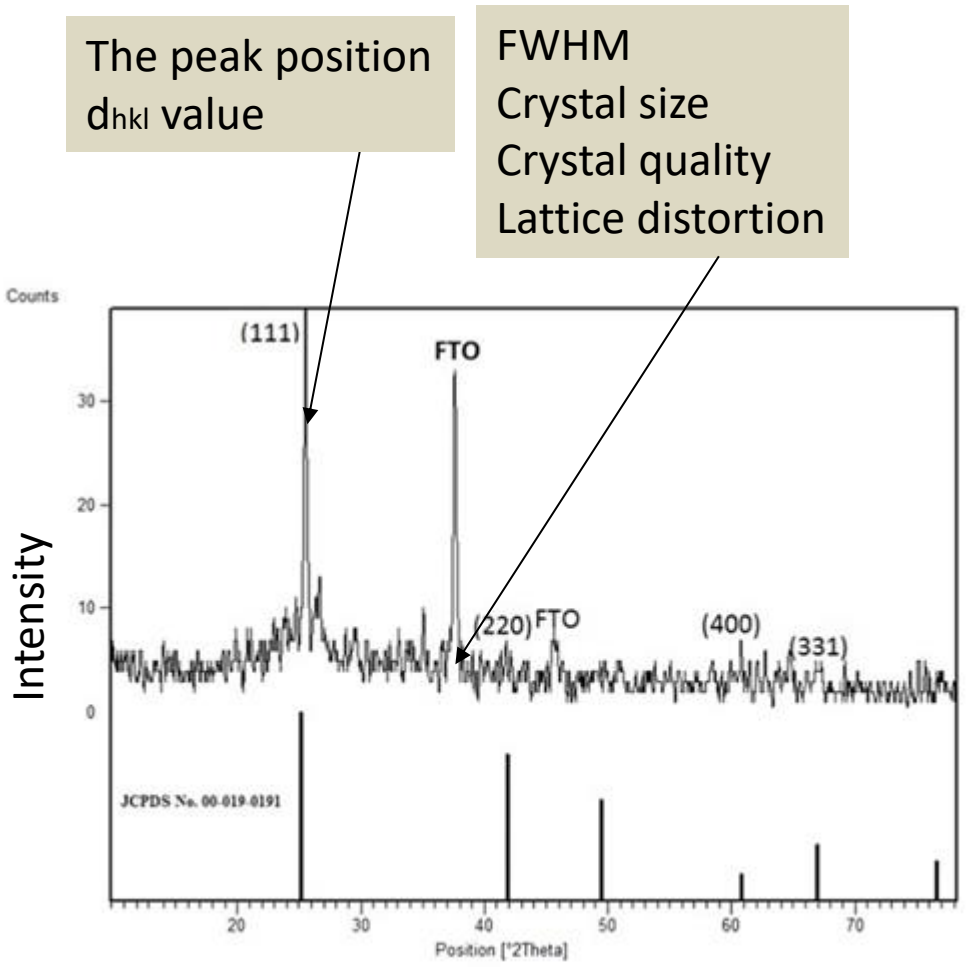
- Review of the Millar indices



- **Anatomic of the diffraction pattern**



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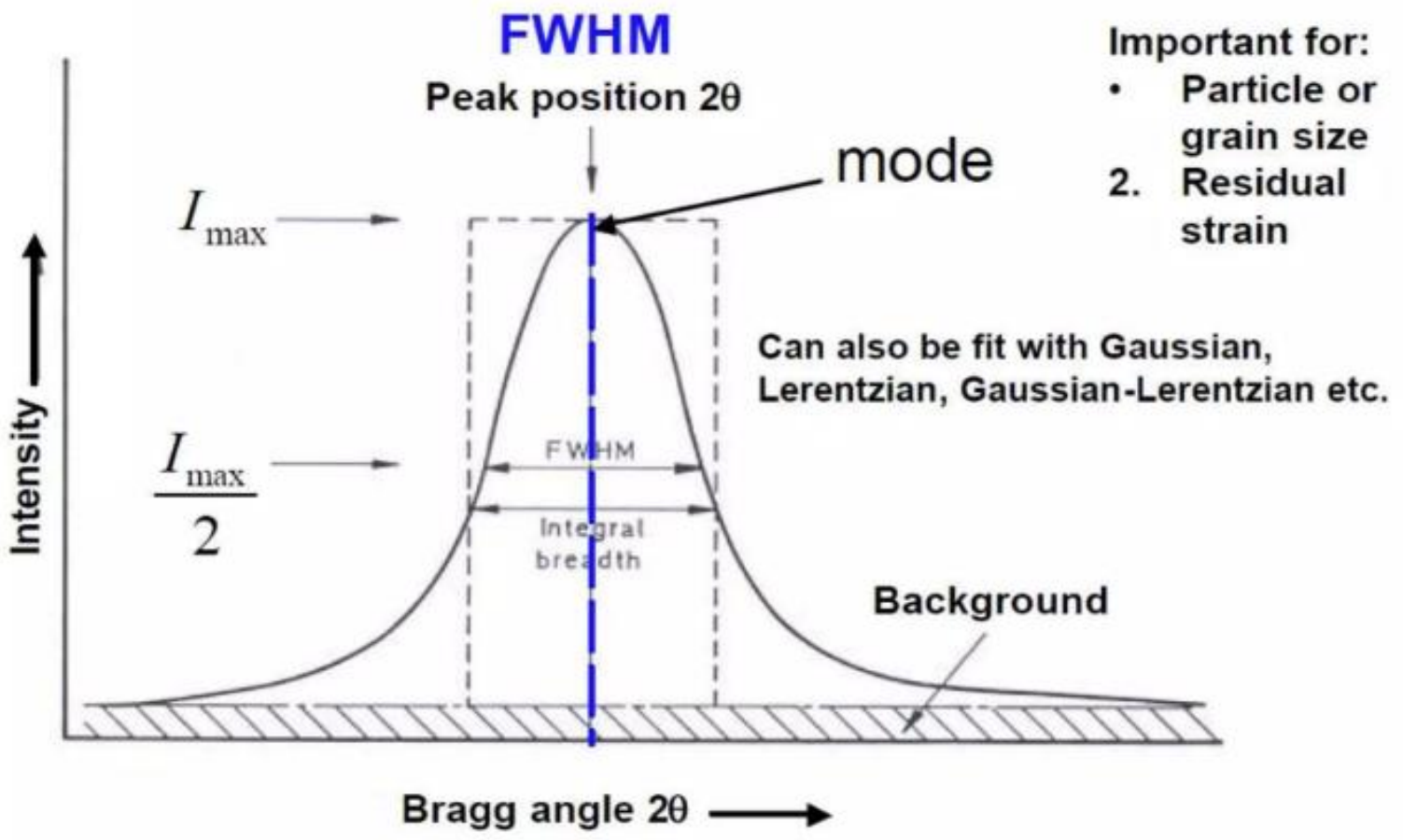
- **Full-width at half maximum (FWHM)**



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- **Calculated average crystallite size**

❖ Ideally, a Bragg diffraction peak is a line without width. Diffraction from a crystal specimen results in a peak characterized by a specific width, referred to as peak broadening.

❖ The peak width depends on the size of the crystals. Peak width is inversely related to crystal size; peak width increases with decreasing crystal particle size.

- **Calculated average crystallite size (Cs)**

The average crystallite size (Cs) can be determined by Scherrer eq.

$$C_s = \frac{K\boldsymbol{\lambda}}{\beta cos\theta}$$

where K, β, λ, and θ are the incident Scherer constant is 0.9, the FWHM

diffraction peak, X-ray wavelength (0.154 nm), and the angle between the incident beam and crystal plane respectively.

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- **scanning electron microscopy (SEM) of the morphology study**

In scanning electron microscopy (SEM), an electron beam is directed toward the specimen instead of a light beam, as in the case of an optical microscope. A highly concentrated electron beam is shot at the device's top from an electron gun. The two main electron gun types are field emission guns, which generate a strong electric field that rips electrons from the atom, and thermionic guns, which heat the filament until the electrons stream away.

- The SEM scans the surface of the sample with high-energy electron beams. Thus, SEM differs from conventional light microscopes as they use light waves to create a magnified image. In SEM, when the electron beam strikes the specimen surface, it interacts with the surface.

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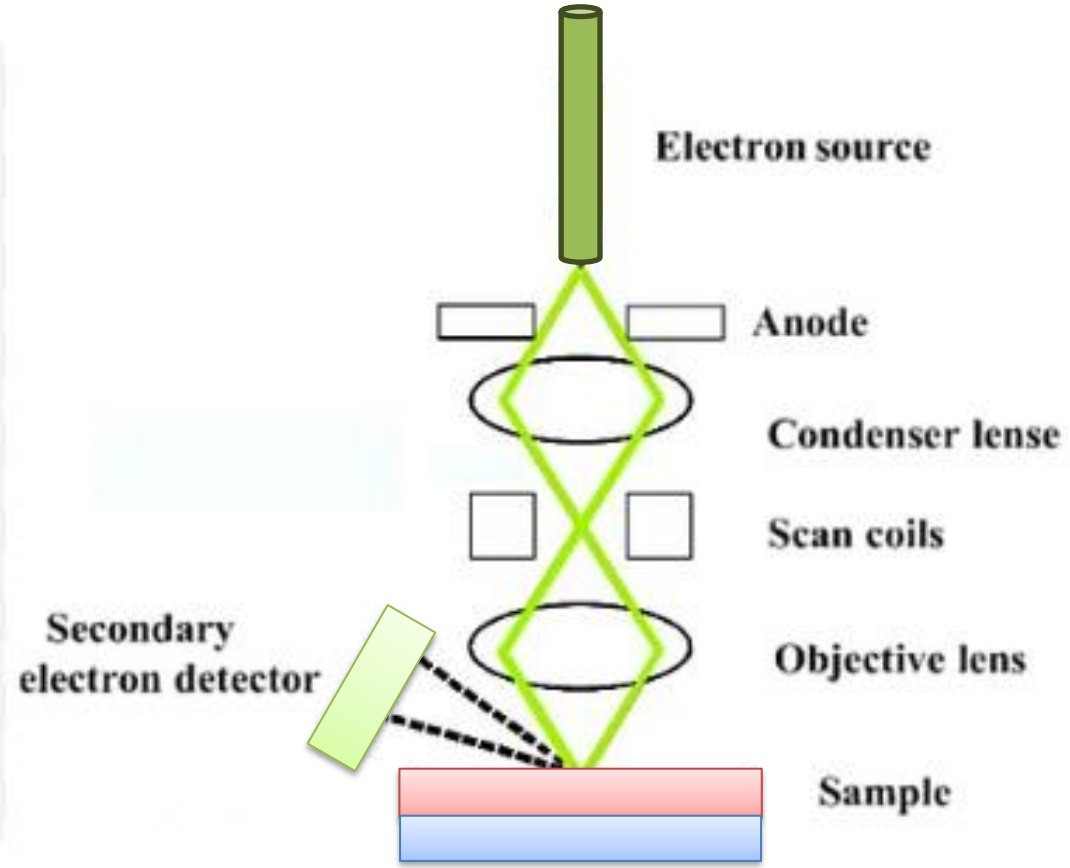
- The electron column has the scanning coils, and the electron beam is passed through them to the final lens. This deflects the beam vertically and horizontally to perform raster scanning over the surface's rectangular area. Electronic devices detect and amplify signals, displaying them as images on a cathode ray tube.



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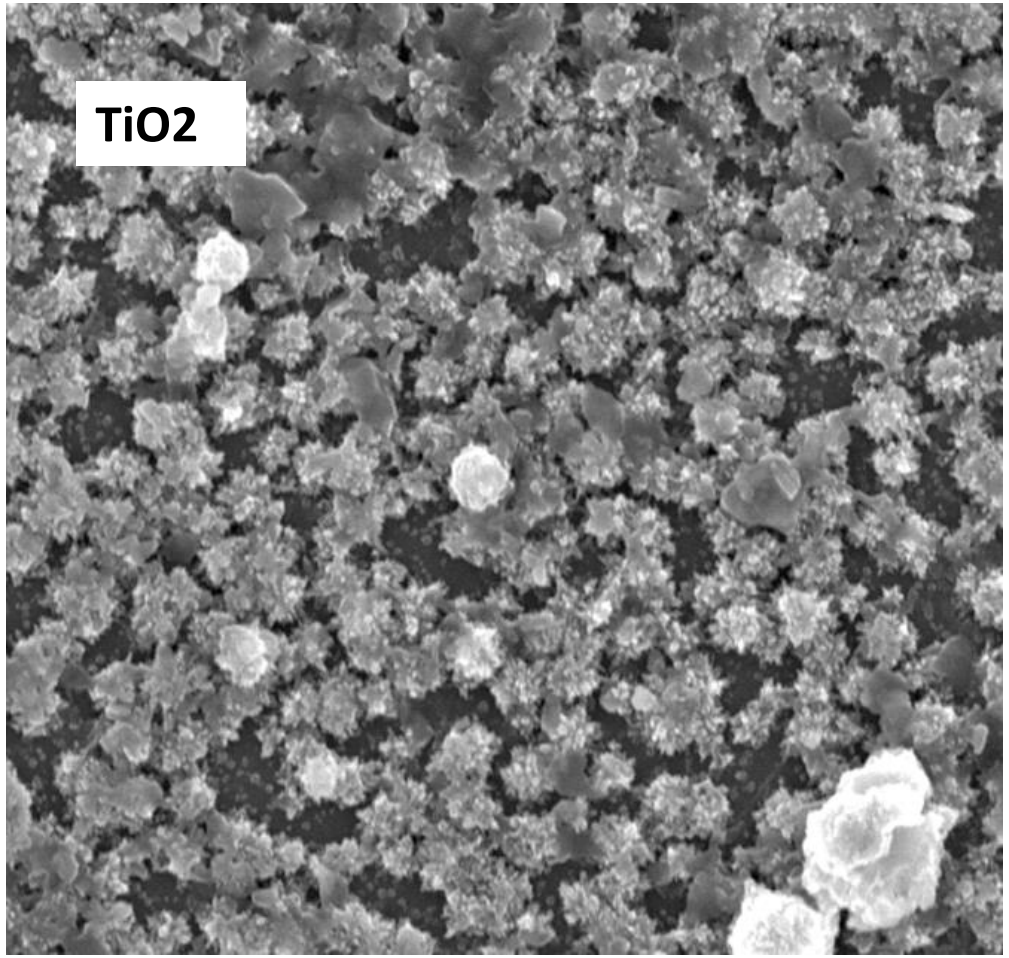

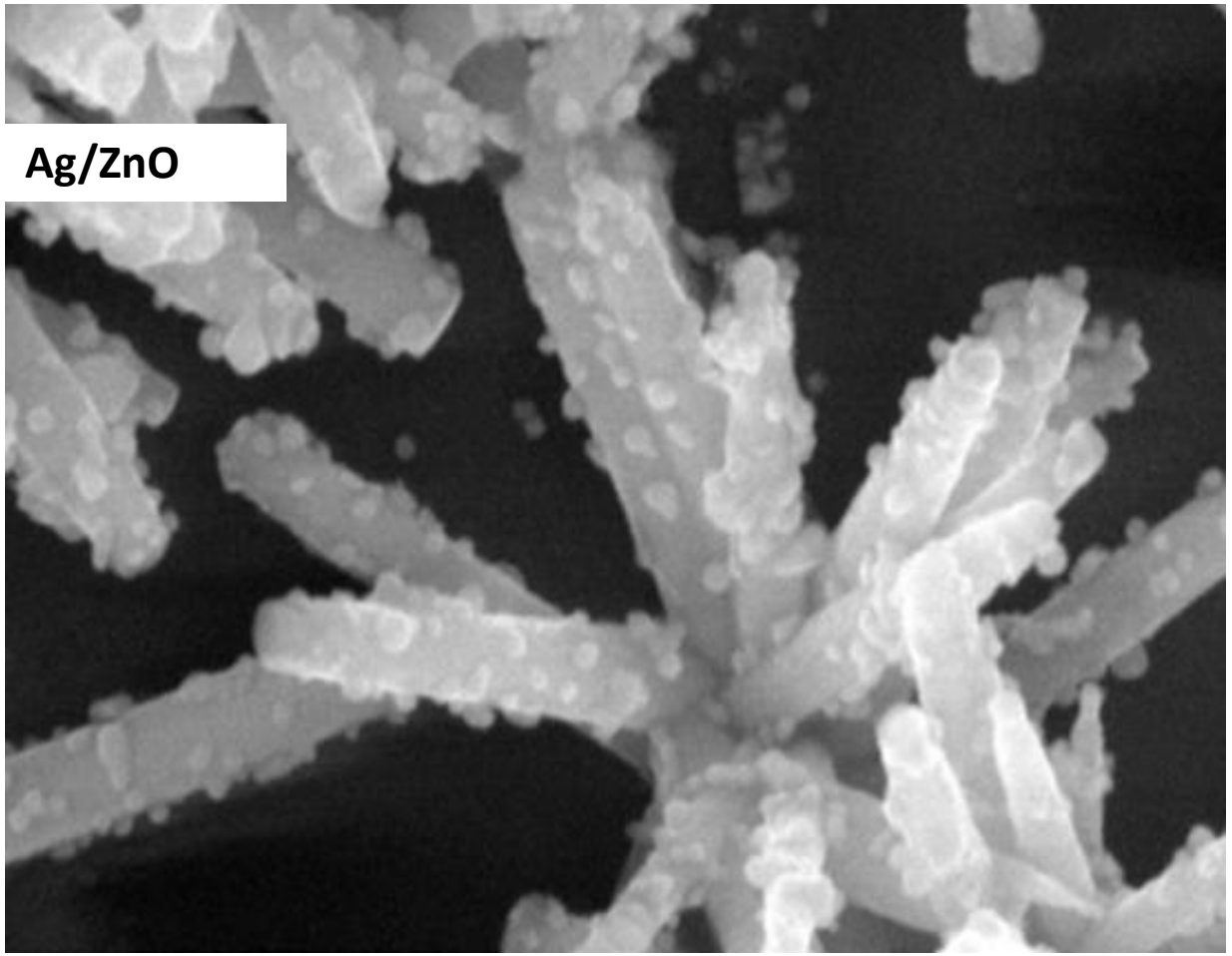

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- **TRANSMISSION ELECTRON MICROSCOPY (TEM)**

The contrast in TEM images is not the same as the contrast in light microscope images. Diffraction is what happens when the electron beam hits the object instead of absorption. The strength of the diffraction changes depending on how the plane is positioned in relation to the electron beam. At some angles from the axis, the electron beam is heavily bent, but at other angles, it passes through.

- Holders for the specimen are put in place so that it can be tilted to get a specific diffraction state. Light field is the process of using electrons that have not been scattered to make a contrast picture. The aperture blocks the electrons that have been deflected, letting the electrons that have not been scattered pass through. It's possible to make a picture with the electrons that have been bent. This is called a dark field image.

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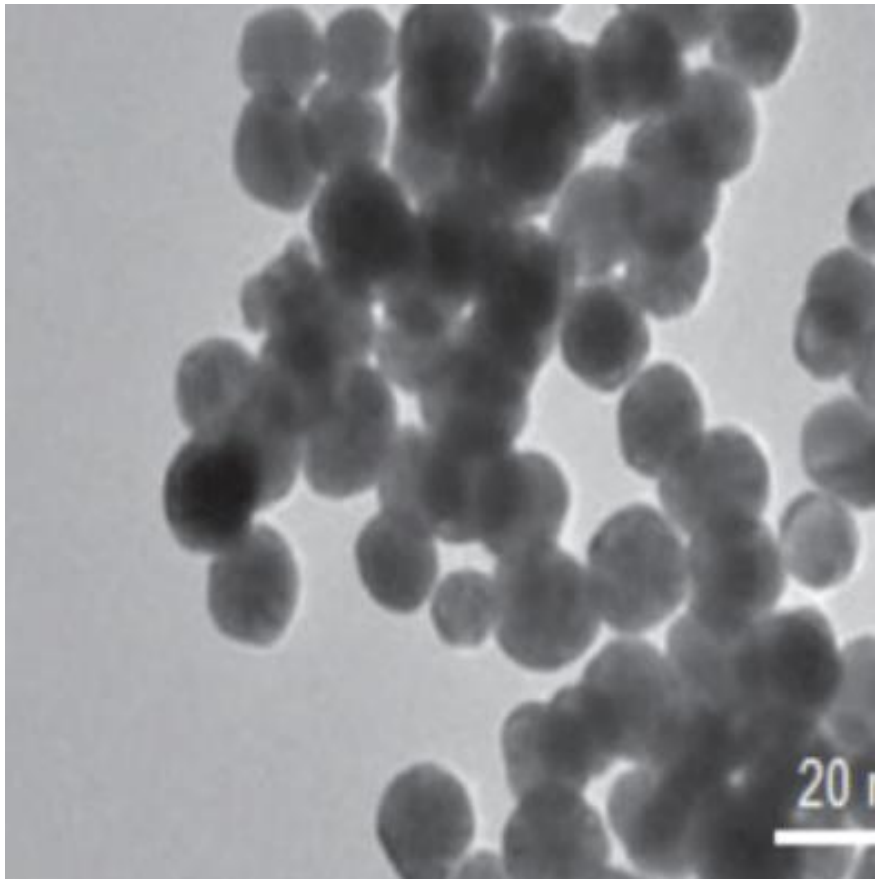

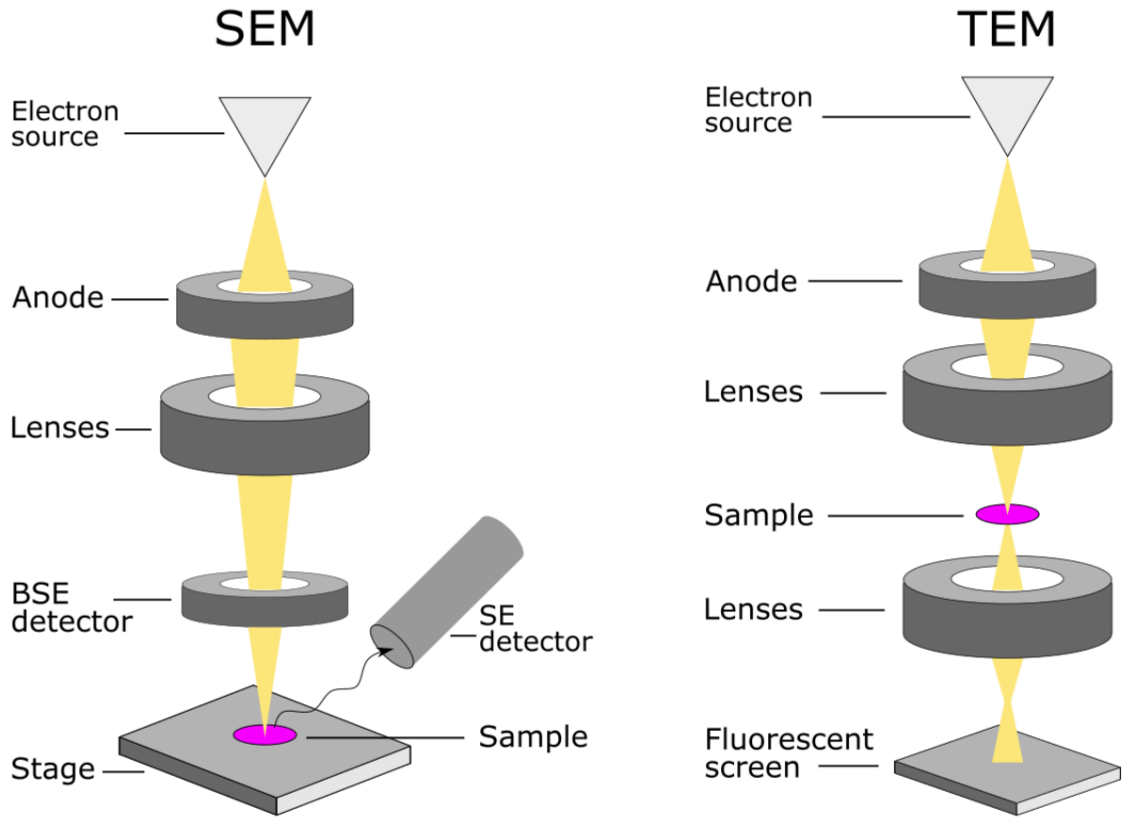

https://anapath.ch/electron-microscopy-2/

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- **ATOMIC FORCE MICROSCOPY (AFM)**

An AFM is a strong and flexible type of microscopy used to look at objects at the nanoscale level. It can take a picture of a three-dimensional landscape and give you different surface measures. With less sample preparation, AFM can make images with atomic precision and height information on the Angstrom scale. It can measure the surface roughness and see the surface structure of many materials in polymer nanocomposites. In addition, it is a nondestructive method with a high spatial precision in three dimensions.

- AFM can scan the sample's surface with an AFM probe with a sharp tip. The cantilever bends slightly toward the surface as the surface gets closer to the tip. This is because the tip and surface are attracted to each other. The cantilever is brought close to the surface so that the tip touches it, but the forces pushing against it are stronger, and it moves away from the surface.
- Laser beams measure how far the cantilever moves away from or toward the surface. When the cantilever bends, it can slightly change the direction of the mirrored beam.

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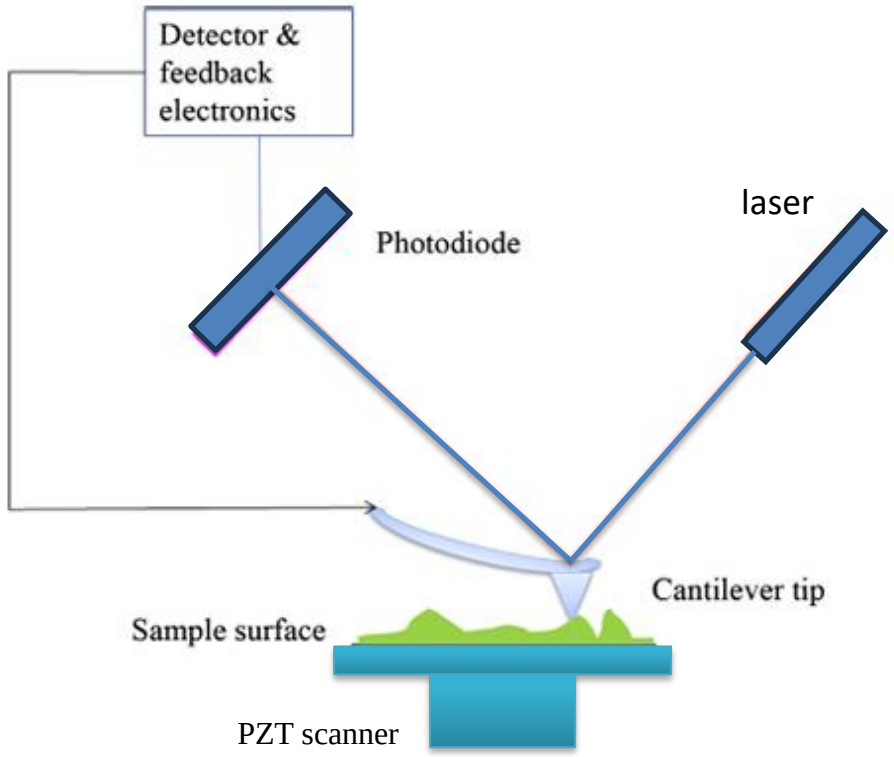
- A photosensitive photodiode will be used to track this change. This means the photodiode records how the cantilever bends when an AFM moves over a raised surface.
- By using the cantilever to scan the area of interest, a picture of the sample's surface topography is made. The photodiode measures how much the cantilever bends, and the features on the sample surface affect this. Controlling the tip height above the surface keeps the laser in the same place, which lets AFM make an accurate topographic model of the surface features.

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