



PHYSICAL CHEMISTRY OF NANOSTRUCTURED SYSTEMS

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LECTURE No. 7



BASIC METHODS OF STUDYING NANOSTRUCTURED MATERIALS



INTRODUCTION

- ❑ The importance of the studying methods of nanostructured materials.
- ❑ Various techniques for detecting, measuring and characterizing. No method is the “best”
- ❑ The key parameters of physical characterization.

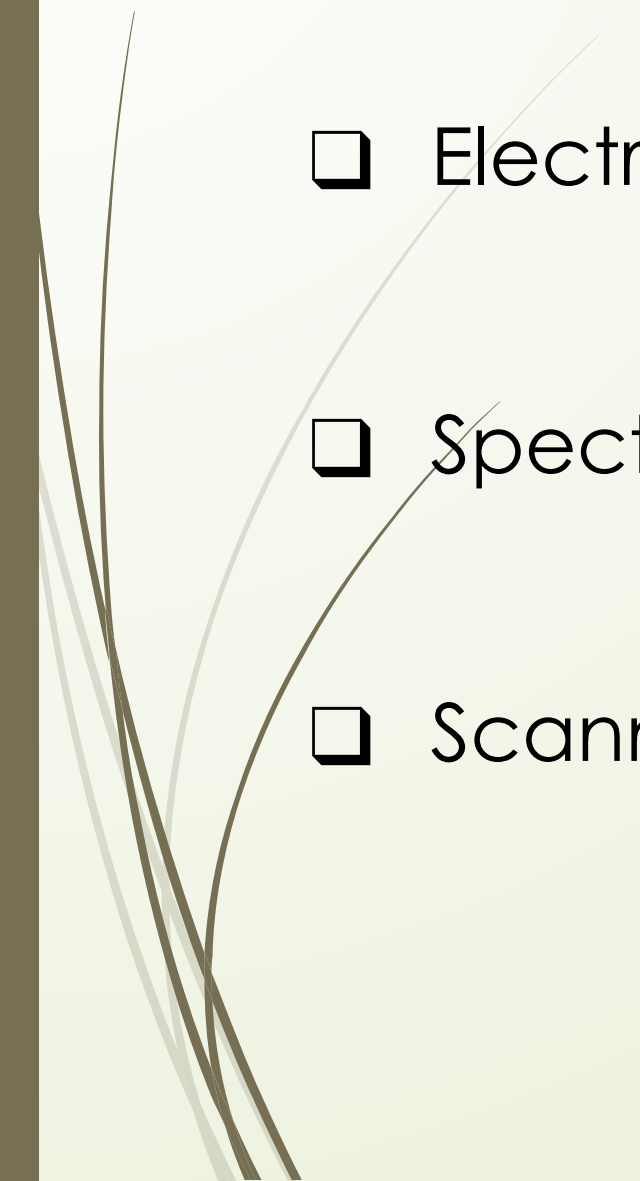


OBJECTIVES

- To describe how to characterize nanomaterials.

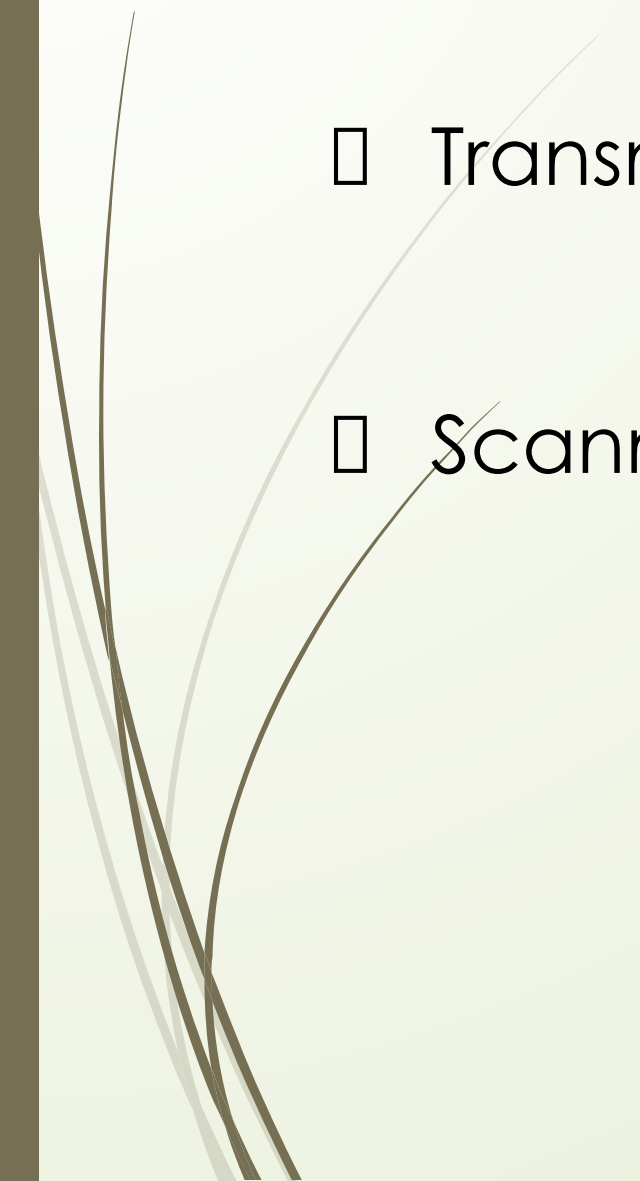


OUTLINE

- ❑ Electronic Microscopy
 - ❑ Spectral methods of research
 - ❑ Scanning Probe Test Methods
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Electronic Microscopy

- Transmission Electron Microscope (TEM)
 - Scanning Electron Microscope (SEM)
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Electronic Microscopy

□ Transmission Electron Microscope (TEM)

- ✓ Measures: particle size and characterization.
- ✓ Sample preparation: $< 1\mu\text{g}$ thin film and stable under an electron beam and a high vacuum.
- ✓ Sample preparation is difficult (thin sample on a support grid).
- ✓ Time consuming and costly.
- ✓ Sensitivity: down to 1 nm.



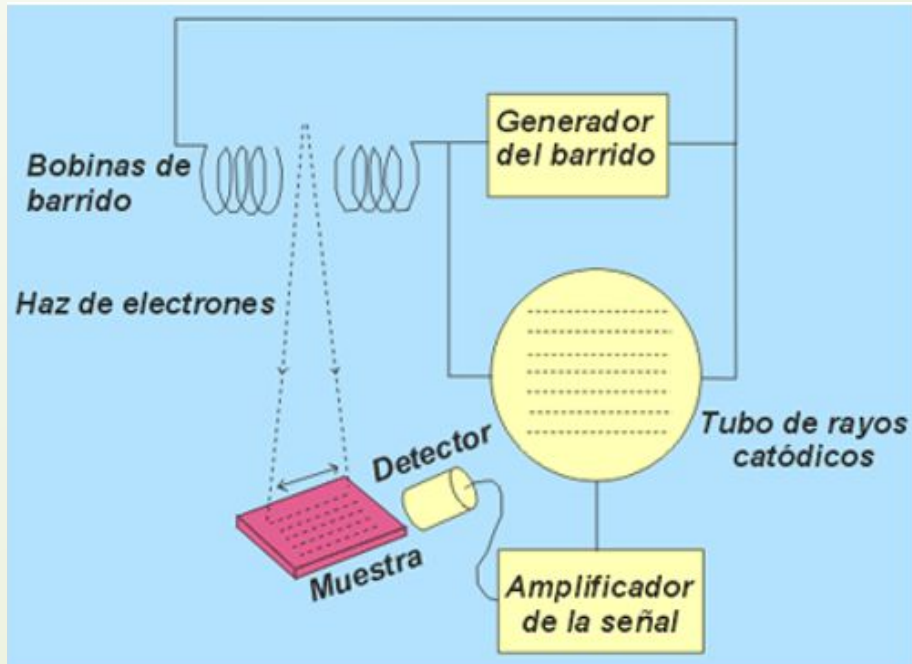
Electronic Microscopy

- Scanning Electron Microscope (SEM)
 - ✓ Measures particle size and characterization.
 - ✓ Sample: conductive or sputter coated.
 - ✓ Easier to prepare than TEM.
 - ✓ Samples mounted on a stub of metal with adhesive, coated with 40 - 60 nm of metal such as Gold/Palladium.
 - ✓ Sensitivity: down to 1 nm.

Electronic Microscopy

□ Scanning Electron Microscope (SEM)

- ✓ Principle: The SEM is based on the interaction of the electron beam with the specimen surface.

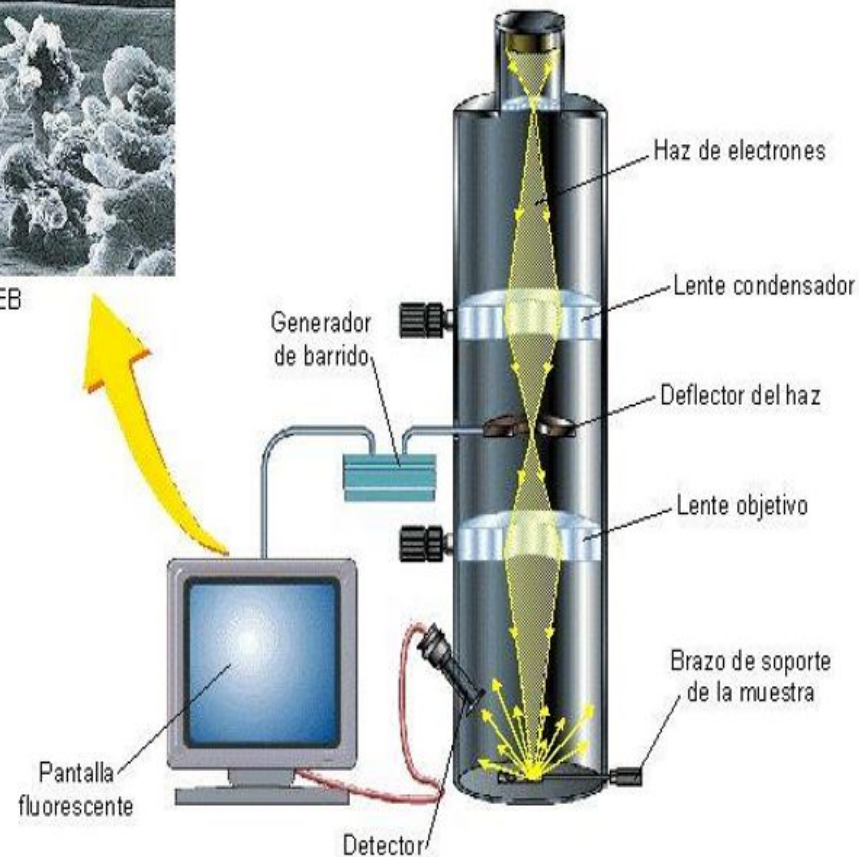


Electronic Microscopy

□ Scanning Electron Microscope (SEM)



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Spectral methods of research

- Electronic Auger Spectroscopy (AES)
 - ✓ Analytical technique used specifically in the study of surfaces.
 - ✓ Based on the energy analysis of secondary Auger electrons.

Spectral methods of research

- Secondary ion mass spectroscopy
 - ✓ Analyze the composition of solid surfaces and thin films by sputtering the surface of the specimen with an ion beam and collecting and analyzing ejected secondary ions.
 - ✓ High sensitivity and allows determining all chemical elements, including hydrogen and helium.

Spectral methods of research

□ Laser microprobe analysis

- ✓ It uses a focused laser for microanalysis.
- ✓ It employs local ionization by a pulsed laser and subsequent mass analysis of the generated ions.
- ✓ The resulting ions generated by this laser are then analyzed with mass spectrometry to give composition, concentration, and in the case of organic molecules structural information.
- ✓ Disadvantage: rather low accuracy in determining the quantitative content of elements.

Scanning Probe Test Methods

- ❖ The most widely used in the field of nanomaterials and nanotechnology.
- ❖ The main idea is to use a device for reading information from the surface of the material being studied.
- ❖ In most cases, a diamond needle with a tip radius of about 10 nm is used as the working body of the probe.

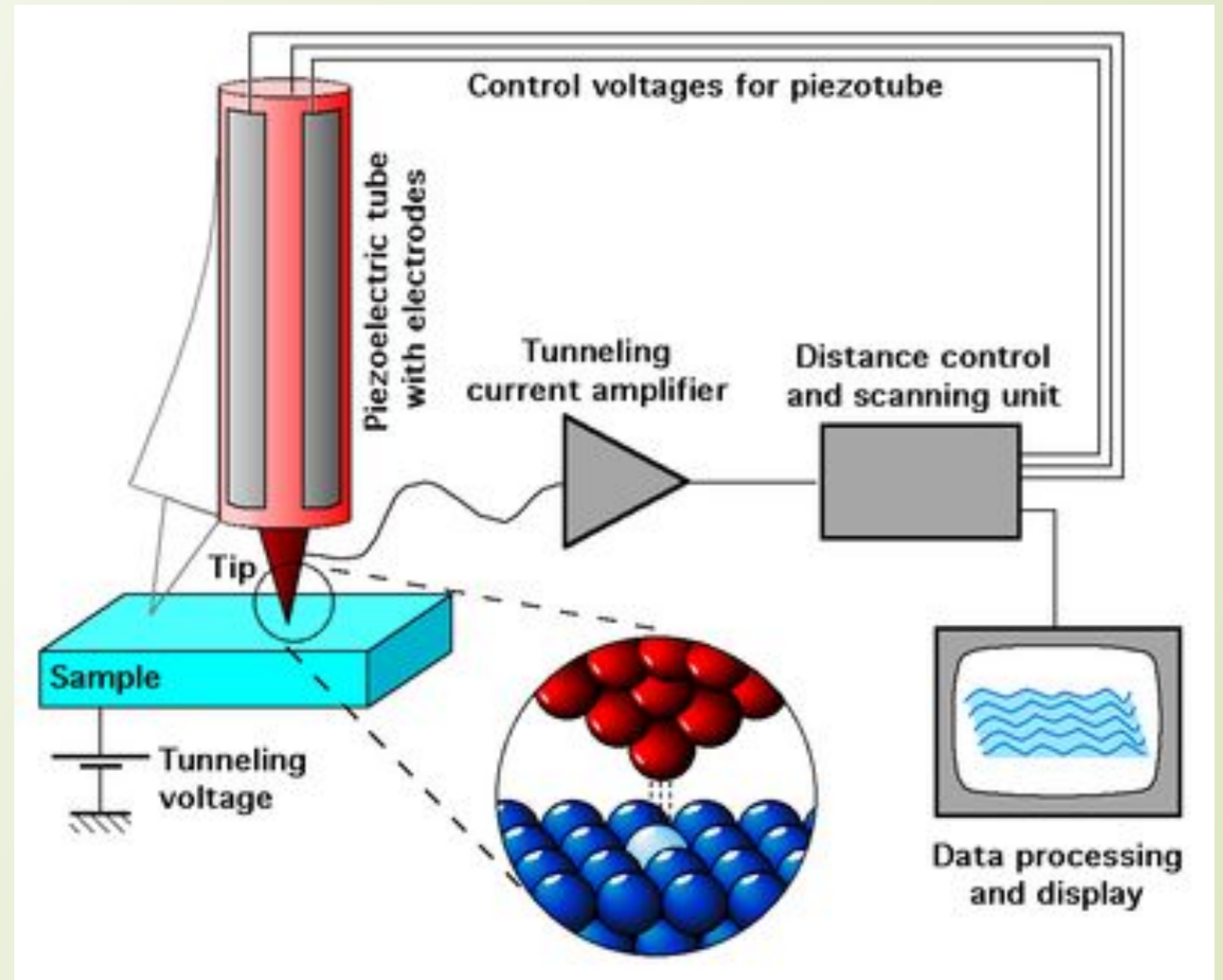
Scanning Probe Test Methods

- ❖ The cost and size of probe microscopes are usually much lower than those of electronic microscopes.
- ❖ The presence of vacuum is not required.
- ❖ Research materials can be very diverse, including insulators, semiconductors, biological objects.

Scanning Probe Test Methods

□ Scanning tunneling microscope

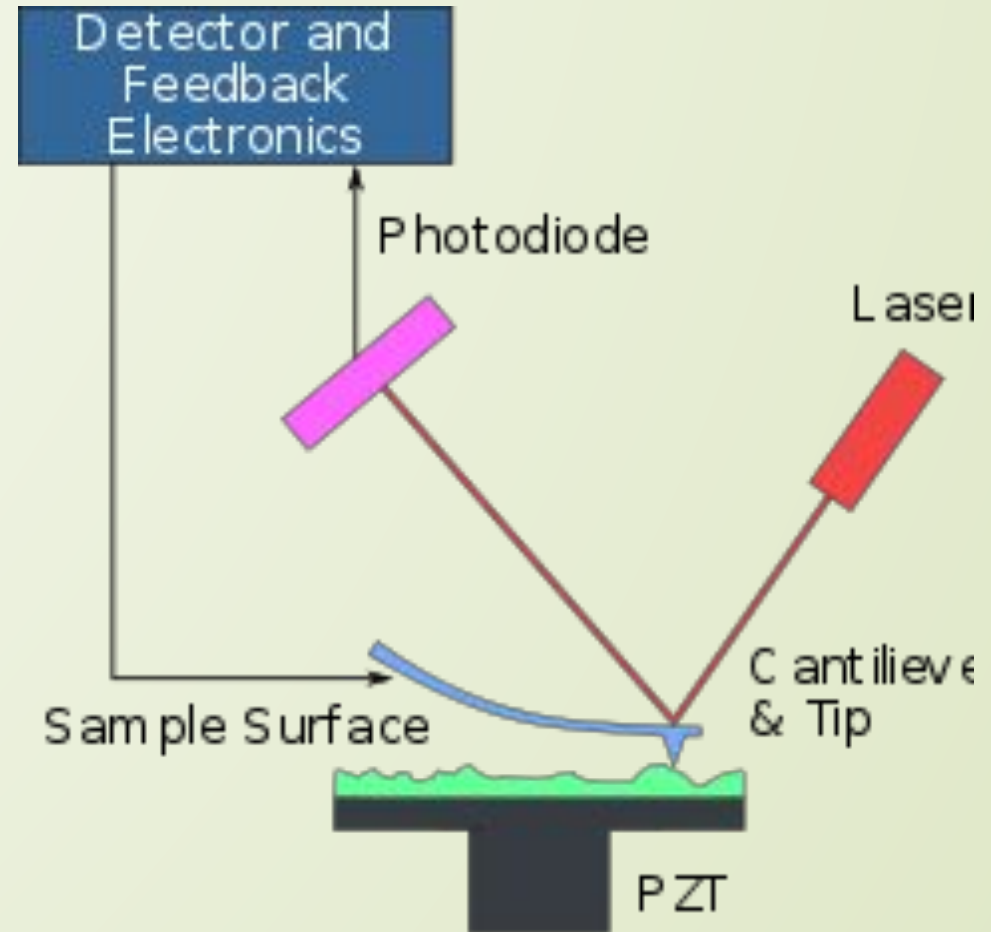
- ✓ STM is based on the concept of quantum tunneling.
- ✓ Extremely clean and stable surfaces, sharp tips, excellent vibration control, and sophisticated electronics are required.



Scanning Probe Test Methods

□ Atomic Force Microscopy (AFM)

- ✓ It is a very-high-resolution type of scanning probe microscopy (SPM).
- ✓ The AFM has three major abilities: force measurement, imaging, and manipulation.





Other techniques that can be used in the characterization of nanomaterials:

- ❑ X-Ray Diffraction (XRD)
- ❑ Dynamic light scattering (DLS)
- ❑ Nanoparticle Surface Area Monitor (NSAM)
- ❑ Condensation Particle Counter (CPC)
- ❑ Differential Mobility Analyzer
- ❑ Scanning Mobility Particle Sizer (SMPS)



Control questions

1. What are the key parameters in the physical characterization of nanostructured materials?
2. Describe the operating principle of the electronic scanning microscope.
3. Explain the importance of the use of Secondary ion mass spectroscopy.
4. Why the Scanning Probe Test Methods are the most widely used in the field of nanomaterials and nanotechnologies?



**THANK YOU FOR YOUR
ATTENTION!**