ISBN: 978-93-94570-41-2

6. Characterization and Microscopy

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Abstract:

The Center for Electron Microscopy and Analysis (CEMAS) is at the center of this endeavor. For both industry and academics, CEMAS is the leading center for materials characterization. The Center combines multidisciplinary skills to enhance our characterization capabilities, drive synergy, and push the envelope of what is achievable in electron microscopy. This work discusses characterisation methods for examining the physical structure, including atomic force microscopy (AFM), transmission electron microscopy (TEM), and scanning electron microscopy (SEM).

A polished surface will only provide accurate information about a given specimen if all preparation stages and steps are taken into account, followed, and carefully and professionally completed. It can be concluded that microscopy as a technique will continue to play a critical role during decision-making with a good quality optical microscope.

The course "Scanning Electron / Ion / Probe Microscopy in Materials Characterization" will give students, engineers, technicians, and researchers (material and biological scientists), in-depth knowledge of three different microscopy techniques. The surface morphology of solid materials (mainly) at the nanoscale range can be obtained using any one of these three scanning microscopy techniques. We shall talk about microscopy and characterization in this essay.

Keywords:

Electron, Microscopy, Materials, Characterization, Scanning, Optical, Microscope, Images, Materials Science, Atoms, Light, Elements, Polymer.

6.1 Introduction:

6.1.1 Microscopy Characterization:

The Microscopy Characterization facility is made up of a number of microscopy tools that are useful for a wide range of biomedical applications. These tools enable researchers to collect images and conduct structural studies on everything from individual molecules to living nanoscale cells.

In terms of materials science, characterization refers to the comprehensive and all-encompassing process of probing and measuring a material's structure and properties. Without it, no scientific understanding of engineering materials could be established. It is an essential procedure in the field of materials research.

The term's application is frequently ambiguous; for example, some definitions restrict the term's use to methods that examine the microscopic structure and characteristics of materials, whereas others apply it to any process of materials analysis, including macroscopic methods like mechanical testing, thermal analysis, and density calculation.

From angstroms, where individual atoms and chemical bonds can be seen, to centimeters, where coarse grain patterns in metals can be seen, the scale of the structures seen in materials characterization spans from these. [1]

One of the main techniques for material characterisation and scientific inquiry in general is microscopy, which refers to the use of microscopes to analyze various materials and surfaces. A variety of techniques are used by microscopes to produce enlarged images of objects and surfaces.

There are numerous varieties of microscopes, and they can be categorized in various ways. One way is to explain how an instrument interacts with a sample and generates images by either sending an electron or light beam through the sample in its optical path, detecting photon emissions from the sample, or using a probe to scan across and a short distance from the sample's surface. The optical microscope, which is the most popular and the first to be created, employs lenses to refract visible light that has passed through a thinly sectioned sample to create an observable image. The electron microscope (both the transmission electron microscopy and the scanning electron microscope), the fluorescence microscope, and other scanning probe microscope types are further important types of microscopes. [2]

6.1.2 Optical Microscopy (OM):

OM is a very adaptable imaging method that is frequently used to investigate the kinetics and behavior of crystallization in polymeric materials. When it comes to polymeric materials, OM is used to investigate the crystal formation of polymeric matrices caused by clay. The degree of dispersion of nanoparticles, mostly carbon-based nanofillers like carbon nanotubes, graphene, and graphite oxide, in immiscible polymer mix nanocomposites in the molten state of polymer matrices can also be studied using OM.

A class of characterization methods known as microscopy explores and maps the surface and sub-surface structure of a material. These methods can collect information on a sample's structure on a variety of length scales using photons, electrons, ions, or physical cantilever probes. Typical instances of microscopy methods include:

- Optical microscopy
- Scanning electron microscopy (SEM)
- Transmission electron microscopy (TEM)
- Field ion microscopy (FIM)
- Scanning probe microscopy (SPM)
- Atomic force microscopy (AFM)
- Scanning tunneling microscopy (STM
- X-ray diffraction topography (XRT) [3]

6.1.3 Comparing Image Formation in Light and Electron Microscopes:

The light microscope (left), the transmission electron microscope (middle), and the scanning electron microscope (right) are three common types of microscopes used for materials characterization. Figure 6.1 depicts schematic cross-sections of imaging modes for each of these three microscope types. Common components include a condenser lens above the specimen to focus the light or electron beam and an illumination source, such as an electron cannon or a source of visible light. The SEM is built differently from a light microscope and a TEM after the condenser lens. In the latter two, a static, nearly parallel beam of illumination is projected onto a viewing point or onto a camera for recording, with the objective lens that creates the image placed below the specimen. The method by which the picture is formed and projected for viewing and recording is the same as that of the light microscope, despite the TEM incorporating some additional lenses.

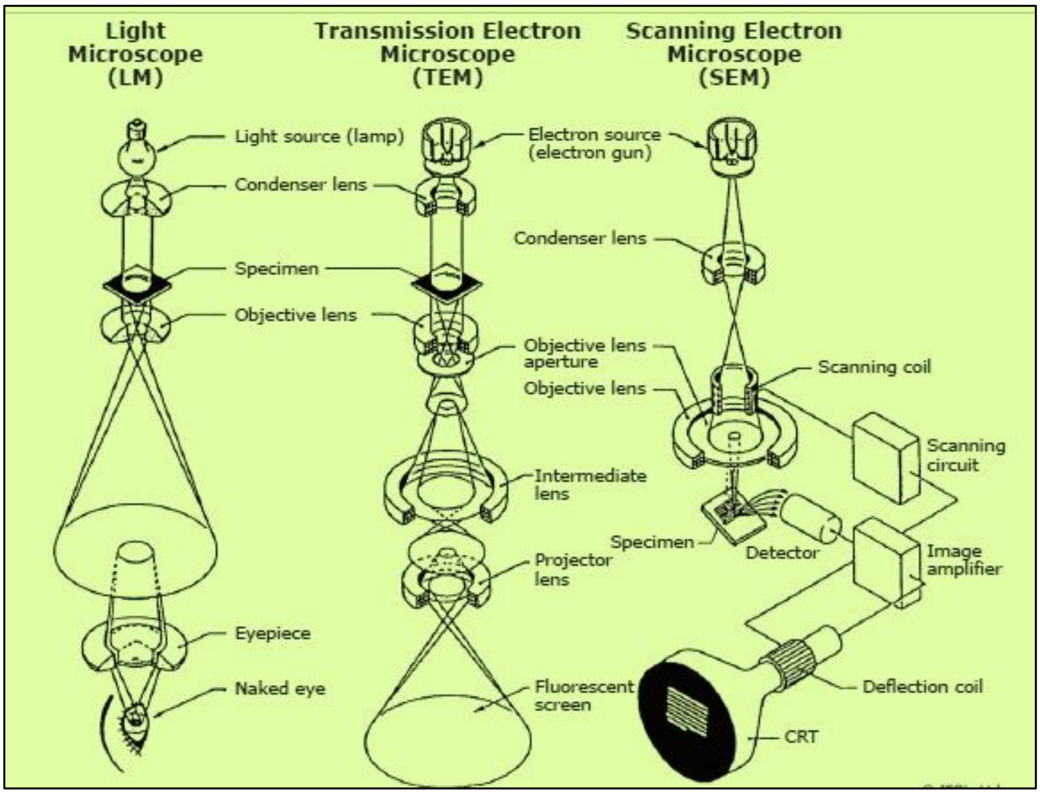

Figure 6.1: shows cross-sections of the imaging modes in the light microscope, TEM, and SEM schematically. [4]

In material science, characterization is a crucial area. For a better understanding, it refers to figuring out the material's physical and chemical properties. The performance of the material can then be optimized using this high level of knowledge. Characterization procedures are frequently employed in membrane research to verify the excellence and purity of the manufactured membranes. Characterization methods are very effective instruments for analyzing membrane performance and researching membrane breakdown. The interaction between the polymer and the fillers can be better understood thanks to characterization techniques.

A. Electron Microscopy:

Each analytical approach used in electron microscopy uses an electron beam as a source to bombard the sample being studied. This is what distinguishes electron microscopy from other types of analysis.

The electron beam interacts with matter to produce a variety of signals that can be used for imaging as well as for studying a material's topographical, crystallographic, and structural properties. These properties can all be correlated with the material's chemical, electrical, optical, and luminescence properties. Scanning electron microscopy, electron probe microanalysis, and transmission/scanning transmission electron microscopy are some of the methods we employ. X-ray diffraction is used in addition to these microscopy techniques. [5]

Transmission electron microscopy (TEM) and scanning electron microscopy (SEM), in addition to light optical microscopy, are particularly significant. Targeted investigation of important sample areas is also possible using the focused ion beam (FIB) technique on the SEM. Both the crystallographic orientation (EBSD) and chemical composition (EDX) can be ascertained through a corresponding analysis. For the high-resolution analysis of surface features, an atomic force microscope and a shared laser scanning microscope are also available. The phases that exist in the materials can be examined using the X-ray diffractometers that are readily available (for example, crystal structure, lattice parameters, texture, etc.). Up to 1100°C can be investigated in a heating chamber.

These target fracture surfaces are primarily characterized using optical and scanning electron microscopy. The MEE team has gained proficiency in operating.

- (relatively) low magnification/resolution optical microscopes.
- high magnification, and ultra-high resolution scanning electron microscopes. [6]

B. Light Microscopy:

An essential tool for characterizing particles and materials is **light microscopy** analysis. It detects small objects using visible light. When using a light microscope, it is possible to observe samples up to 1,000 times closer than normal with little to no sample preparation. A sample can be seen in its current state while taking note of attributes like color, transparency, and morphology.

Types of Light Microscopy:

- Polarized light microscopy (PLM) - PLM may identify optical characteristics that specifically distinguish particular chemical phases.
- Phase contrast microscopy (PCM).
- Microscopy using bright field and dark field reflected light.
- Stereo microscopy.
- The sample is irradiated by ultraviolet light during fluorescence microscopy, which causes some phases to emit visible light. The sample is identified using the generated spectrum.

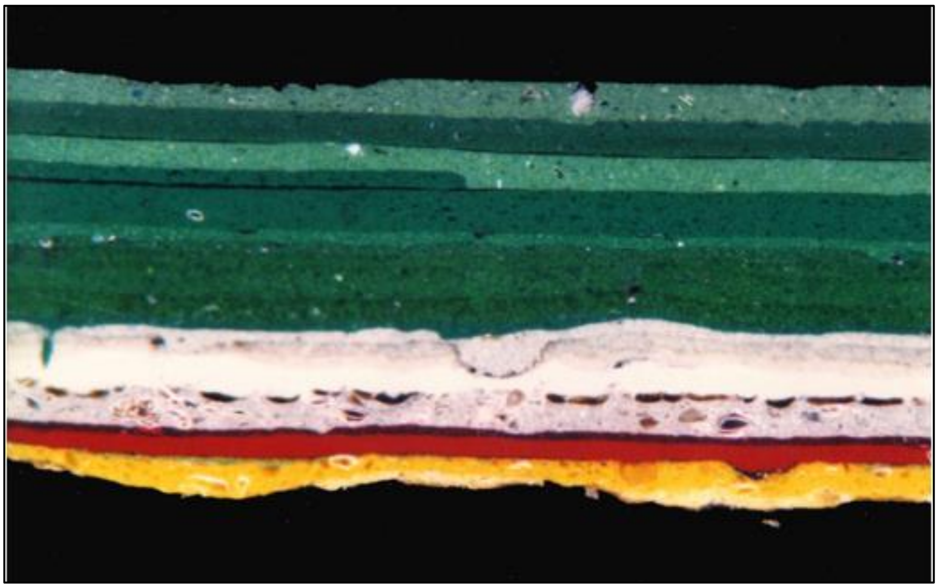

Figure 6.2: A cross section of paint chips for light microscopy examination

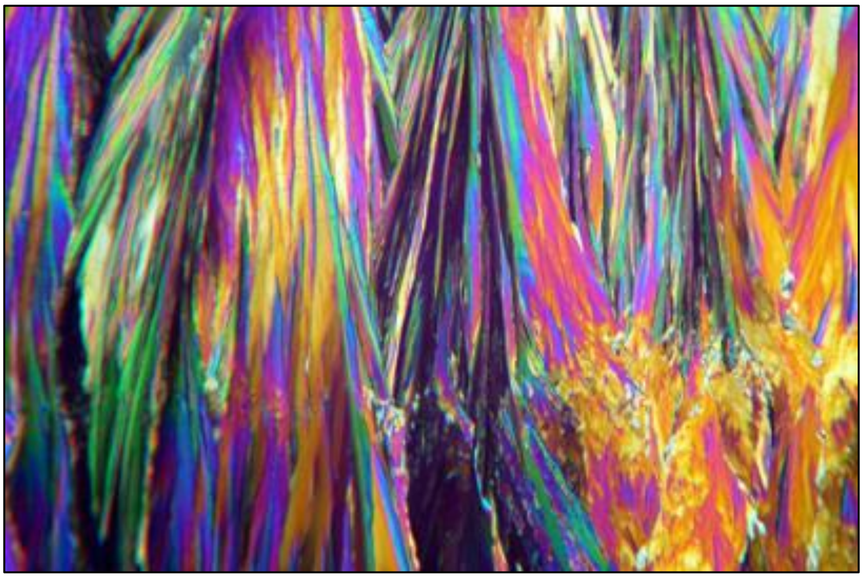

Figure 6.3: Organic Crystal Fusion Preparation

The principal tool used by scientists and engineers to analyze the microstructure of materials is light or optical microscopy. The use of a light microscope to examine the microstructure of materials dates back to the 1880s.

Since that time, metallurgists have extensively used light microscopy to study metallic materials. For metallurgists, light microscopy evolved into a distinct discipline known as metallography. The fundamental methods created by metallography can also be utilized to examine ceramics and polymers in addition to metals. [7]

A. Optical Principles:

- **Image Formation:**

The generation of images, magnification, and resolution are all aspects of the optical principles of microscopes. The behavior of a light path in a compound light microscope, as shown in Figure 6.4, can be used to illustrate image generation. At position A, a specimen (object) is positioned between one and two focal lengths away from an objective lens. A magnified inverted picture is created when light rays from the object focus at location B after initially converging at the objective lens.

The second lens (projector lens) further converges the light rays from the picture to create the final, magnified image of the item at C.

Figure 6.4's light path creates the actual image at C, which is different from what our eyes view, on a screen or camera film. On a screen, only an actual image may be created and captured for photography. [8]

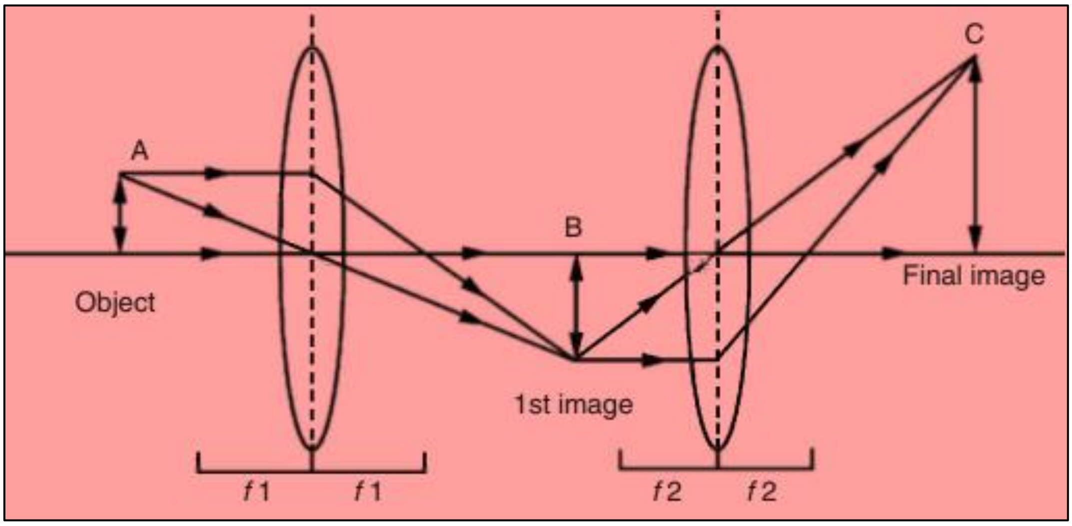

Figure 6.4: Principles of magnification in a microscope.

Different categories can be used to categorize microscopes. One classification is based on the element that interacts with the sample to produce the image, such as light or photons (for optical microscopes), electrons (for electron microscopes), or a probe (for scanning probe microscopes).

As an alternative, microscopes can be categorized according to whether they analyze the sample all at once (wide field optical microscopes and transmission electron microscopes) or via a scanning point (confocal optical microscopes, scanning electron microscopes, and scanning probe microscopes). In optical microscopes, electromagnetic waves are used; in electron microscopes, electron beams are used.

The wavelength of the radiation used to scan the sample in these microscopes determines their resolution; shorter wavelengths provide higher resolution. [9]

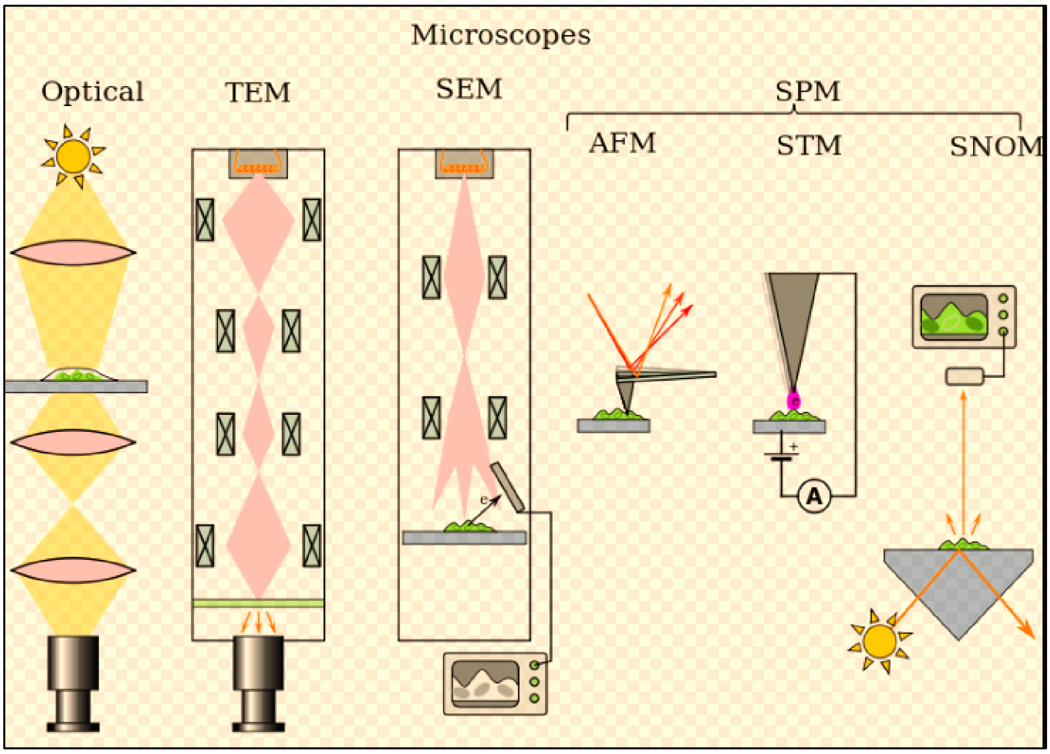

Figure 6.5: Different types of microscopes are depicted by the fundamentals of their beam pathways.

Using lenses to focus a beam of light or electrons onto the sample, scanning optical and electron microscopes, such as the confocal microscope and scanning electron microscope, examine the signals produced by the beam's interactions with the sample.

After that, the point is moved over the sample to examine a rectangle area. The data from scanning a physically small sample area is displayed on a comparatively big screen to magnify the image. The resolution limit of these microscopes is the same as that of broad field optical, probe, and electron microscopes.

The optical microscope is the most popular and oldest form of microscope. This optical device has one or more lenses that expand the image of a sample that is put in the focal plane. Refractive glass, occasionally acrylic or quartz, is used in optical microscopes to focus light on the eye or another light detector.

The same principles govern how mirror-based optical microscopes function. Assuming visible light, the typical magnification of a light microscope is up to 1,250×, with a theoretical resolution limit of around 0.250 micrometers or 250 nanometers. This caps the amount of feasible magnification at ~1,500×.

This magnification may be exceeded by specialized methods (such as scanning confocal microscopy and Vertigo SMI), but the resolution is diffraction restricted.

The spatial resolution of the optical microscope can be enhanced by using shorter wavelengths of light, such as ultraviolet, as well as tools like the near-field scanning optical microscope. [10]

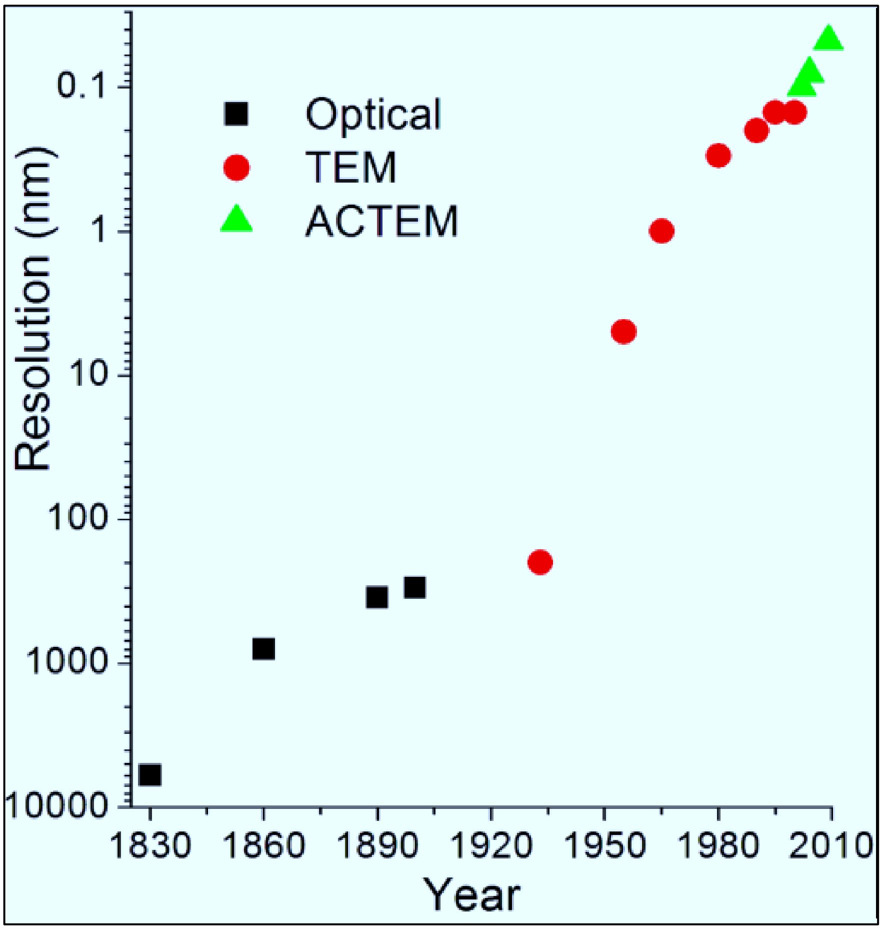

Figure 6.6: illustrates the development of spatial resolution attained using optical, transmission (TEM), and aberration-corrected electron microscopes (ACTEM).

6.1.4 Electron Microscope:

The two main categories of electron microscopes are TEMs (transmission electron microscopes) and SEMs (scanning electron microscopes). To focus an electron beam with a high energy on a sample, they both have a set of electromagnetic and electrostatic lenses. Similar to basic optical microscopy, a TEM involves the passage of electrons through the material. Since most materials heavily scatter electrons, this calls for meticulous sample preparation. The samples also need to be very thin (less than 100 nm) for the electrons to travel through them. [11]

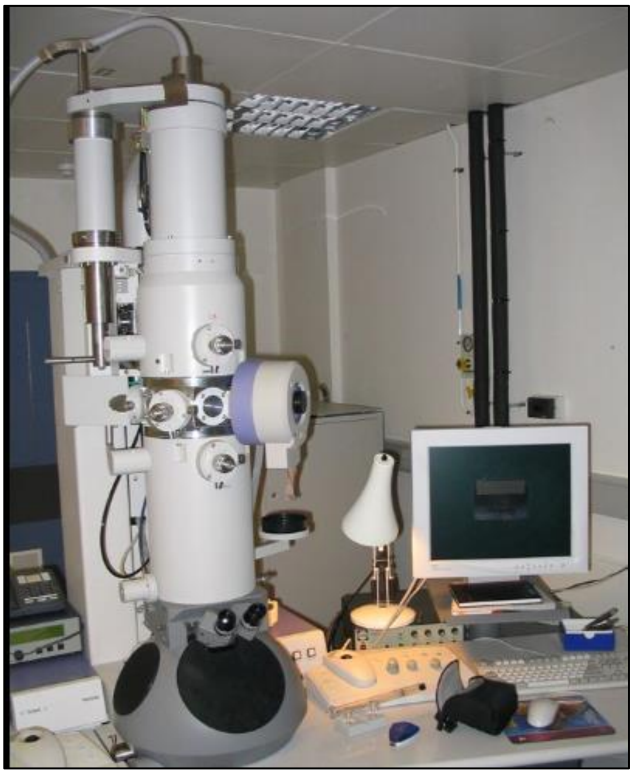

Figure 6.7: Modern transmission electron microscope

6.2 Scanning Tunneling Microscopy:

Quantum tunneling is the foundation of the STM. A bias (voltage differential) provided between the two can cause electrons to tunnel through the space between them when a conducting tip is brought extremely close to the surface being studied.

The local density of states (LDOS) of the sample, applied voltage, and tip position all influence the tunneling current that results. Information is gathered by keeping an eye on the current as the tip scans the surface and is typically shown as an image.

STM can be difficult to use since it needs electronics of the highest caliber, surfaces that are impeccably clean and steady, sharp tips, and superb vibration control. [12]

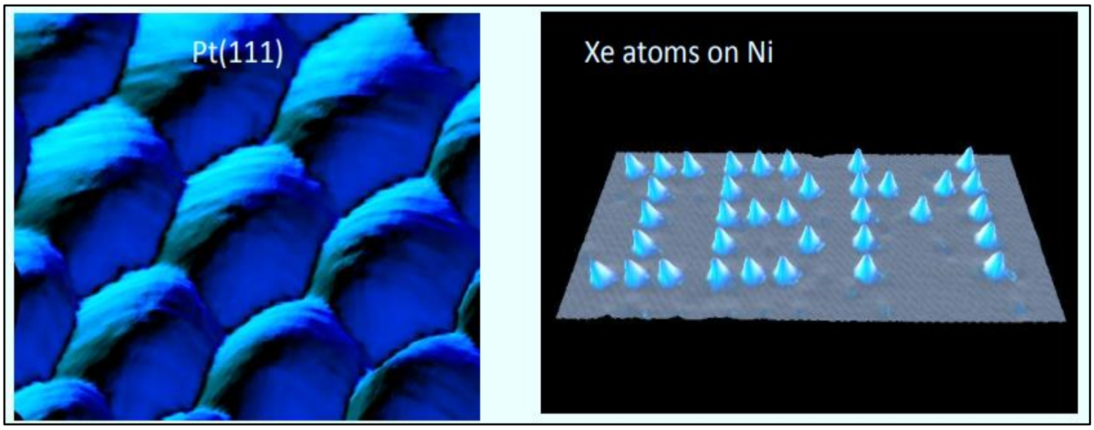

Figure 6.8: Scanning Tunneling Microscopy

6.2.1 Microscope Components:

In comparison to a low-power or dissection microscope, a compound microscope has higher magnification capabilities.

It is employed to examine minute specimens, such as cell structures, which are not visible at lower magnification levels.

Components for both the structural and optical parts of a compound microscope. The head, arm, and base are the three basic structural parts.

- The optical components found in the microscope's upper section are found in the body or head.
- The arm joins and supports the microscope's base and head. It is also used to transport the microscope.

- The base of the microscope contains the illuminator as well as supporting the microscope.
- The microscope's optical section consists of:

a. Eyepiece
b. Eye tube
c. Objective lenses
d. Nosepiece
e. Adjustment knobs
f. Stage
g. Illuminator
h. Condenser and condenser focus knob
i. Diaphragm

6.2.2 Functions of Microscope:

A microscope's main use is to examine biological samples. Magnification and resolution are the only two ideas that a microscope relies on to operate.

Simply put, magnification is the microscope's capacity to enlarge the image. While the resolution affects one's ability to analyze minute details.

The two types of microscopes that are most frequently used in classrooms for educational purposes are compound and dissection microscopes.

6.2.3 Functions of compound microscope:

- It makes the research of bacteria and viruses simpler.
- They are employed in pathology labs to facilitate simple disease diagnosis.
- They are additionally employed in forensic labs to recognize human fingerprints.

Simple Microscope with Parts Name:

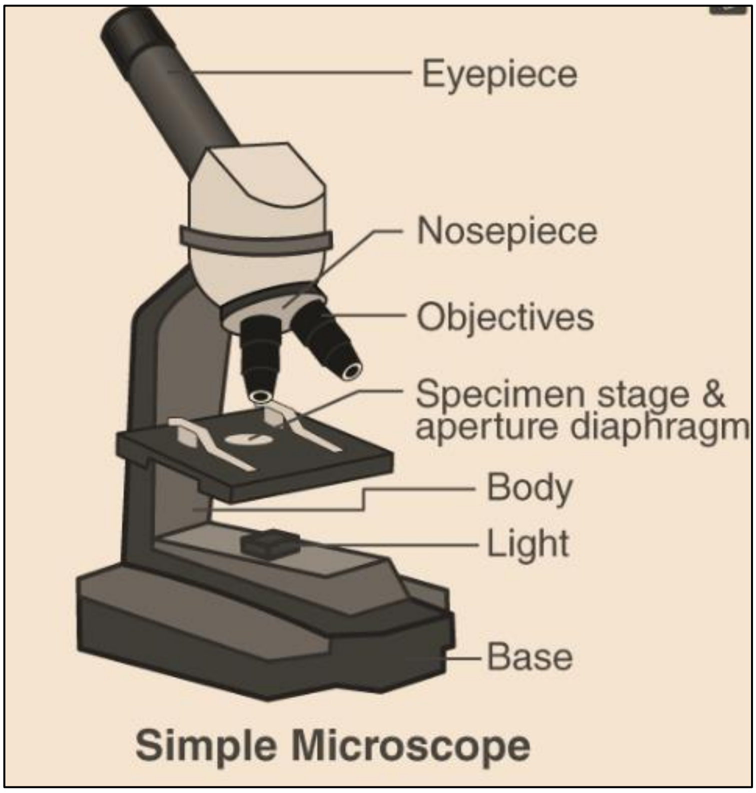

Figure 6.9: Simple Microscope with Parts Name

A rudimentary light microscope with only one lens is referred to as a simple microscope. Simple microscopes don't have a condenser component. They use natural light and fewer hooks and knobs for adjusting things.

Compound microscopes, on the other hand, feature two adjustment knobs: fine and coarse. Additionally, they have a greater magnification than a standard microscope. [13]

6.3 Conclusion:

At sub-nanometer resolution, several types of functional characteristics are now investigated. Most of the time, single scalar integers are used to represent attributes like resistivity, conductivity, surface potential, and charge density.

The enormous variety of specialized microscopes and related techniques that are currently available for materials characterization only scratches the surface of what may be done with microscopes at the two extremes of the resolution scale. microscopy's enormous capacity for data collection and its crucial role in the characterization and advancement of today's high-performance materials. The topics will include preparation of various types of samples, evaluation of findings/data, and basic concepts of these approaches as well as many parameters that affect image quality.

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