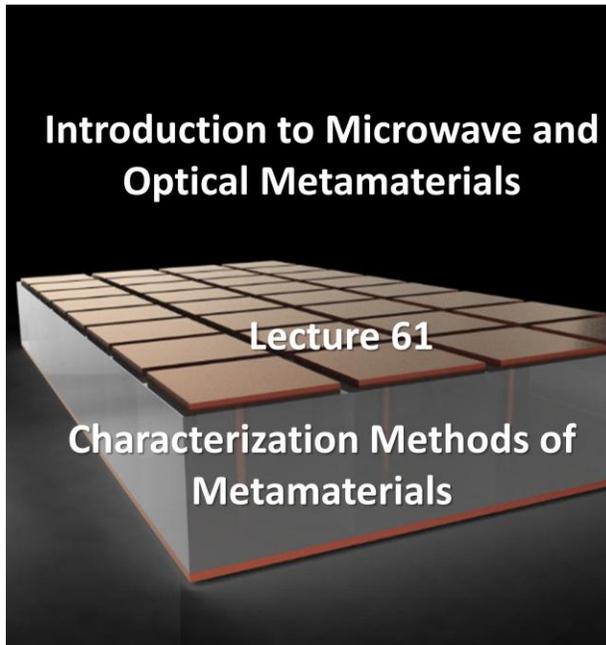


Course Name: Introduction to Microwave and Optical Metamaterials
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Week-12
Lecture-61

Lec 61: Characterization Methods of Metamaterials



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Hello everyone, welcome to Lecture 61 of the online course on Introduction to Microwave and Optical Metamaterials. In this final lecture of the course, we will look into the characterization methods for metamaterials.

Lecture Outline

- Importance of Metamaterials Characterization
- Typical Objectives of Metamaterial Characterization
- Data Processing to Characterize Metamaterial
- Characterization Methods for Microwave Metamaterial
 - ✓ Free-Space Methods
 - ✓ Waveguide-Based Methods
 - ✓ Resonant Cavity Methods
- Characterization Methods for Optical Metamaterial
 - ✓ Scanning Electron Microscopy (SEM)
 - ✓ Transmission Electron Microscopy (TEM)
 - ✓ Atomic Force Microscope (AFM)
 - ✓ UV/VIS/NIR Spectroscopy
 - ✓ Ellipsometry



So here is the lecture outline; we will briefly discuss the importance of metamaterial characterization. We will look into some typical objectives of metamaterial characterization, and data processing that can be used for this purpose. And then we will see some characterization methods for microwave metamaterials such as free-space methods, waveguide-based methods, and resonant cavity methods.

And then finally, we look into some characterization methods for optical metamaterials that is scanning electron microscopy SEM, Transmission electron microscopy, which is TEM, atomic force microscopy, which will be AFM, and Then UV-visible NIR spectroscopy and finally ellipsometry.

Importance of Metamaterials Characterization

- Metamaterials characterization means measuring and extracting the electromagnetic properties of a metamaterial sample in specific frequency range.
- Because metamaterials are engineered structures, you must verify that the fabricated sample actually shows the intended effective permittivity (ϵ_{eff}), permeability (μ_{eff}), or surface impedance etc.
- Why characterization is essential
 - **Verification:** To confirm that a fabricated metamaterial behaves as predicted by simulations.
 - **Performance evaluation:** To measure key parameters like permittivity, permeability, and refractive index.
 - **Troubleshooting:** To identify and correct discrepancies between simulated and measured results.
 - **Application development:** To ensure the metamaterial is fit for its intended use (e.g., antennas, cloaks, filters).

Now, why is it important to do the metamaterial characterization? So, when I say characterization, it basically means measuring and extracting the electromagnetic properties of a metamaterial sample in a given frequency range. Now, because metamaterials are engineered structures you must verify that the fabricated sample actually shows the intended effective permittivity and effective permeability or the surface impedance.

So, why characterization is essential? First of all, it gives you verification, which means you can confirm that the fabricated metamaterial behaves as predicted by simulations. Then you can do the performance evaluation that means you can measure the key parameters like permittivity, permeability and the refractive index of that material. And also, you can do troubleshooting that means if there is something wrong in the design you can identify and you know the correct those discrepancies between the simulated and the measured results. And finally, it can take you to application development, which means you can ensure that the metamaterial is fit for its intended use that is for some antenna, cloaking or filter applications it is working fine and then you can go and confirm that the material is perfect for this application.

So, what are the typical objectives of metamaterial characterization that aim to prove that the fabricated structure delivers the designed electromagnetic response, ensuring stability and tunability under real conditions.

Typical Objectives of Metamaterial Characterization

- Metamaterial characterization aims to prove that fabricated structure delivers the designed electromagnetic response, ensure stability and tunability under real conditions.
- So this material can be confidently used in antennas, sensors, absorbers, imaging, stealth, or other advanced electromagnetic applications.

- Measurement and Verification of Electromagnetic Properties like:
 - **Scattering parameters (S-parameters):** Measure transmission coefficient (S_{21}) and reflection coefficient (S_{11}) to determine resonances, bandwidth, and loss.
 - **Effective parameters:** Retrieve effective $\epsilon(\omega)$, $\mu(\omega)$, and refractive index $n(\omega)$ from S-parameters.
 - **Resonance Identification:** Locate electric/magnetic dipole, toroidal, or plasmonic resonances that produce negative-index or epsilon-near-zero (ENZ) behavior.
 - **Dispersion & anisotropy:** Determine how phase velocity and impedance vary with frequency and direction.
 - **Surface properties:** For metasurfaces or absorbers, extract reflection phase, surface impedance, and absorption coefficient.



IIT Guwahati



Source: Schroder, Dieter K. *Semiconductor material and device characterization*. John Wiley & Sons, 2015.

So, this material can then be used confidently in antennas, sensors, absorbers, imaging, stealth, or any other advanced electromagnetic application. So, measurement and verification of electromagnetic properties such as scattering parameters (S parameters) are important. So, you can measure the transmission coefficient, which is basically the S_{21} parameter, and the reflection coefficient, which is the S_{11} parameter and this allows you to determine the resonance bandwidth and the loss of this metamaterial. Next, you can also derive and obtain the effective permittivity, permeability, and also the refractive index, which are all functions of frequencies from the S-parameters. Then you can also identify the resonance, which means you can locate the electric or magnetic dipole toroidal or plasmonic resonances that can produce a negative index or epsilon-near-zero behavior.

Dispersion and anisotropy: So, you can determine how the phase velocity and the impedance vary with frequency and direction. Also, you have surface properties for meta-absorbers, metamaterials, or absorbers. You can extract the reflection phase, surface impedance, and absorption coefficient, right. With these typical objectives of metamaterial characterization, you obtain the data. and that is why the data processing will be required that can finally characterize the metamaterials.

Data Processing to Characterize Metamaterial

➤ After retrieval of S-parameters from various methods; perform following steps:

1. Pre-Processing of Measured S-Parameters: Phase unwrapping & reference plane correction

- Remove artificial phase jumps ($\pm 2\pi$) from the measured S_{11} and S_{21} .
- Shift the reference planes to the physical faces of the sample to eliminate fixture or connector delays.

2. Nicolson-Ross-Weir (NRW) retrieval or related algorithms (use the corrected S-parameters) to compute:

- Material's complex refractive index $n(\omega)$ and impedance $z(\omega)$:

$$n(\omega) = \frac{1}{k_0 d} \cos^{-1} \left[\frac{1 - S_{11}^2 + S_{21}^2}{2S_{21}} \right] \quad \& \quad z(\omega) = \sqrt{\frac{(1 + S_{11})^2 - S_{21}^2}{(1 - S_{11})^2 - S_{21}^2}}$$

Here k_0 is free-space wavenumber and d is sample thickness.

3. Then from n and z , calculate: $\epsilon_{\text{eff}} = n/z$ and $\mu_{\text{eff}} = nz$.

4. For metamaterial absorbers, compute absorption $A = 1 - |S_{11}|^2 - |S_{21}|^2$.



Source: Schroder, Dieter K. *Semiconductor material and device characterization*. John Wiley & Sons, 2015.

So, after retrieving the S parameters from the different methods, you have to follow the following steps, okay? So, as you know, first you need to do the pre-processing of the measured S parameters.

Which includes phase unwrapping and reference plane correction. So, that is basically that you remove the artificial phase jumps, which are like plus or minus 2 pi, from the measured S11 and S21 parameters. Okay, and then for the reference plane correction, you have to shift the reference plane to the physical faces of the sample. That will eliminate the fixture and connector delays. After that, you can focus on Nicolson-Ross-Weir (NRW) retrieval or any other related algorithms That can be used to correct the S-parameters to compute the material's complex refractive index, that is n as a function of omega (ω) and impedance that is z is a function of omega (ω).

So, $n(\omega)$ is given by this: $\frac{1}{k_0 d} \cos^{-1} \left[\frac{1 - S_{11}^2 + S_{21}^2}{2S_{21}} \right]$;

as you can see, it is a function of the thickness of the material S11 and the S21 parameters, same here as well it is dependent on the S 11 and S 21 parameter the impedance ok. So, here you can see that k_0 is basically the free space wave number, and as I mentioned, d is the sample thickness. And from this impedance z , you can also find out what the effective permittivity (ϵ_{eff}) is, which is given by n/z . And mu effective (μ_{eff}), which is the effective permeability, will be $\mu_{\text{eff}} = nz$.

Now, once you know the reflection, or you can say reflectance, and the transmittance.

You can also find out what the absorbance or the overall absorption by this absorber is, which can be given as: $A = 1 - |S_{11}|^2 - |S_{21}|^2$, okay.

So, with that understanding, let us now go into the details of some characterization methods for microwave metamaterials.



Characterization Methods for Microwave Metamaterial



So, first you need to understand what the key instruments required for microwave metamaterial characterization are.

Key Instruments for Microwave Metamaterial Characterization

- **Vector Network Analyzer (VNA):**
 - Core instrument for sweeping the microwave band and measuring S-parameters (S_{11}, S_{21}) with high accuracy.
 - **Measurement Setup:** Calibration Standards
 - ✓ TRL (Thru-Reflect-Line): preferred for waveguides and on-wafer measurements.
 - ✓ SOLT (Short-Open-Load-Thru): common for coaxial connectors and bench setups.
 - ✓ Purpose: removes systematic errors (mismatch, delay, loss) before measurement.
- **Antenna / Fixture Options:**
 - Standard-gain horns for free-space or quasi-optical testing.
 - Waveguide flanges or custom sample holders for guided-wave measurements.



The first thing you will require is a VNA (Vector Network Analyzer).

It is a core instrument for sweeping the microwave band and measuring the S parameters, namely S11 and S21, with high accuracy. So, the measurement setup you have for the calibration standards, and then there is a TRL that is through the reflect line. These are basically preferred for waveguides and on-wafer measurements. There are also other setups, something like a calibration standard, such as SOLT, which stands for short open through load. Sorry, short open load through SOLT, okay? So, this is basically the calibration for coaxial connectors and bench setups.

So, why do you need to do this, which basically removes the systematic errors, mismatch, delay, and loss before the measurement. and then what are the antenna fixture options?

You can use standard gain horns for free-space or quasi-optical testing. You can use waveguide flanges or custom sample holders for guided wave measurements.

So, here we will first discuss the free-space methods.

Free-Space Methods

- **Free-Space Methods:** These techniques involve placing the metamaterial sample between transmitting and receiving antennas in free space.
- This methods characterize metamaterials without physical contact by measuring electromagnetic waves transmitted or reflected by the sample between two antennas.
- By analyzing the scattering parameters, or S-parameters (**transmission and reflection coefficients**), obtained from a vector network analyzer, material properties like complex permittivity and permeability are extracted using algorithms.
- This makes it ideal for broadband, non-contact, and non-destructive measurements in various environments.

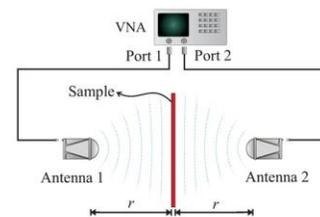


Figure: Schematic of Free space measurement Set-up

So, in the free space methods, this technique basically involves placing the metamaterial sample. Between the transmitting and receiving antennas, as you can see here, okay.

This is antenna 1 and antenna 2; the sample is placed in between, okay, in the free space. So, this method characterizes metamaterials without physical contact. By measuring the

electromagnetic wave that is transmitted or reflected by the sample placed between these two antennas. So, this is your VNA right. So, the two ports are connected to both antennas.

Now by analyzing the scattering parameters, or S-parameters, that basically give Your transmission and reflection coefficient are okay, which you can obtain from the VNA. Metamaterial properties, like complex permittivity and permeability, are basically extracted using algorithms. This makes a deal for broadband, non-contact, and non-destructive measurements in various environments.

Now, how does the free space measurement works? So, first you set up in that case what you do you put a planar sample of the material between the transmitting and this is the transmitting and this is the receiving antenna which are basically horn antennas.

Free-Space Methods

➤ How it Works

1. **Setup:** A planar sample of the material is placed between a transmitting and a receiving antenna, which are usually horn antennas.
2. **Measurement:** The setup, including antennas and the material under test (MUT), is connected to a vector network analyzer (VNA) to measure the S-parameters.
3. **Reference Measurement:** A reference measurement of the antennas and cables without the material is taken for normalization purposes.
4. **Data Extraction:** The S-parameters of the MUT are calculated by subtracting the reference measurement from the measurement with the material in place.
5. **Material Properties Calculation:** Algorithms are then used to extract the electromagnetic properties, such as complex permittivity and permeability, from these S-parameters.

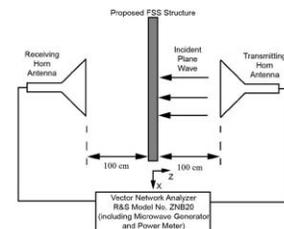


Figure: Schematic of Free space measurement Set-up

And then the setup, including antennas and the material under test, is basically connected to the VNA that basically gives you the measurement of the S-parameters. Now you also do a reference measurement, so the reference measurement of antennas and the cables is done without the material. that is basically that measurement is done for the normalization purpose. So, data extraction, how it works? the S parameter of this material and the test are calculated by subtracting the reference measurement from the measurement with the material in place. So, how material properties calculation works is that there are algorithms that can extract the electromagnetic properties.

As I mentioned, the complex permittivity and permeability from these S parameters.

Then you have waveguide-based methods.

Waveguide-Based Methods

➤ Waveguide-based methods for metamaterial characterization:

- It involves measuring the scattering parameters (S-parameters) of a metamaterial sample placed within a waveguide, typically a rectangular or circular waveguide.
- This measurement of S_{11}/S_{21} is done with a Vector Network Analyzer (VNA); connected via waveguide.
- By analyzing the change in the transmitted and reflected waves within the waveguide, it is then used to determine the metamaterial's properties.
- This enables the design of devices like miniaturized waveguides or filters by controlling wave propagation and absorption.
- This method is highly accurate but is limited to specific frequency bands.

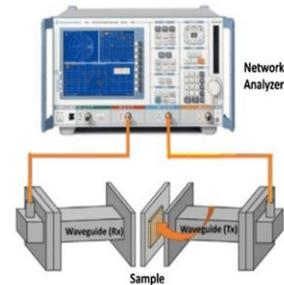


Figure: Schematic of Waveguide-Based measurement Set-up

So, waveguide-based methods for metamaterial characterization basically involve measuring the scattering parameters. The S parameters of a metamaterial sample placed within a waveguide, typically a rectangular or circular waveguide. So, here is a schematic of the waveguide-based measurement setup.

So, this is the network analyzer, and this is the waveguide that is behaving with the transmitter. This is the one with the receiver, and then you put the sample in between, right? So, this measurement of S_{11} and S_{21} done with the VNA is also connected via waveguide. So, by analyzing the changes in the transmitted and reflected waves within the waveguide, it can basically determine the metamaterials properties. So, this enables the design of the device like miniaturized waveguides or filters ok by controlling the wave propagation and absorption. So, this particular method is highly accurate, but it is specifically limited to certain or small frequency bands.

So, what are the key principles and methods in this case?

Waveguide-Based Methods

➤ Key Principles and Methods:

▪ **Scattering Parameters (S-Parameters):**

- At the core of the technique is the measurement of S-parameters, which describe how a material scatters electromagnetic waves.

▪ **Waveguide Insertion:**

- A sample of the metamaterial is inserted into a waveguide, such as a rectangular or TEM transmission line, which acts as the guiding structure for the electromagnetic waves.

▪ **Parameter Retrieval:**

- Algorithms are then used to extract the effective permittivity (ϵ) and permeability (μ) of the metamaterial from the measured S-parameters.

▪ **Frequency Dependence:**

- The measurements are taken over a range of frequencies to characterize how the metamaterial's electromagnetic properties (including its magnetic susceptibility and dispersion) change with frequency.

We are basically measuring S-parameters, right, the scattering parameters. So, these scattering parameters describe how a material basically scatters the electromagnetic waves. So, when a sample of a metamaterial is inserted into a waveguide, such as a rectangular or TEM transmission line, it acts as a guiding structure for electromagnetic waves. Now, you can use an algorithm to extract your parameters. That is the effective permittivity and permeability of the metamaterials derived from the measured S parameters.

Now, as you know that these properties are also frequency dependent. So, the measurements have typically been taken over a range of frequencies that could characterize. How the metamaterials' electromagnetic properties, such as magnetic susceptibility and dispersion, will change with frequency.

Next, we will look into another method that is the resonant cavity method.

Resonant Cavity Methods

- Resonant cavity methods characterize metamaterials by placing the material sample inside a resonant cavity.
- The resulting changes in the cavity's resonant frequency and Q-factor are measured.
- By comparing these measured changes to full-wave simulations of the cavity with the material, the metamaterial's complex permittivity and permeability can be extracted:
 - Which provides insight into its electromagnetic properties and dispersion relations.
- This method gives a very high degree of accuracy for characterizing the material's properties at a single frequency.

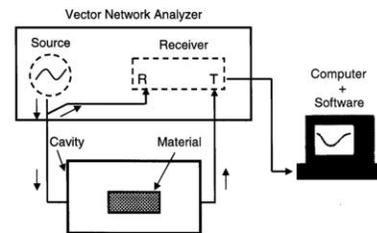


Figure: Schematic of a resonant cavity method (R = reflected power, T = transmitted power).

So, this method characterizes metamaterials by placing the metamaterial or the material sample inside a resonant cavity.

So, this is the schematic of the resonant cavity method; this is the cavity, this is the material, and then you have a source. You have the receiver, and then you have a computer and software. So, this is basically coming from the VNA. So, you actually measure the parameters, okay. So, what you see here is basically the change in the cavities' resonant frequency and the Q factor.

So, you compare these measured changes to the full-wave simulations of the cavity with the material. The materials' complex permittivity and permeability can then be extracted. Okay. So, the change is basically obtained here, and then you get an understanding of the electromagnetic property and the dispersion relation. So, this method is found to be very accurate for characterizing material properties at a single frequency, because it works on the principle of resonance and there is high quality factor involved.

So, the accuracy of measurement at a particular frequency in this cavity method is very high, very good, or very accurate.

So, how it works?

Resonant Cavity Methods

➤ How the Method Works:

- **Cavity Design:** A resonant cavity, typically a rectangular or cylindrical metal conductor, is designed to confine electromagnetic waves.
- **Sample Insertion:** A small sample of the metamaterial is inserted into cavity.
- **Measurement:** A network analyzer is used to measure the transmission and reflection characteristics of the cavity with sample. This provides the resonant frequency and Q-factor for the perturbed cavity.
- **Simulation & Extraction:** Simulation calculates expected resonant frequency and Q-factor based on the assumed properties.
 - The simulation parameters (specifically, the material's complex permittivity and permeability) are then varied to minimize difference between the simulated and measured values.
- **Characterization:** Once the discrepancy is within acceptable tolerance, the final varied complex permittivity and permeability are adopted as characteristic parameters of the metamaterial under test.

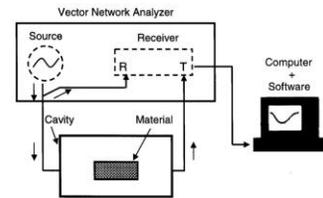


Figure: Schematic of a resonant cavity method (R = reflected power, T = transmitted power).

A cavity resonant cavity typically in the shape of a rectangular or cylindrical metal conductor is basically defined to confine the electromagnetic waves. And then a small sample of the metal material will be inserted into the cavity. And then you put a network analyzer to measure the transmission and reflection characteristics of the cavity with the sample. So, this basically provides the resonance frequency and the Q factor of the perturbed cavity.

Perturb means the cavity is disturbed in the presence of this metamaterial, okay. And then finally, you do the simulation to calculate the expected resonant frequency and Q factor based on the assumed properties, right? So, the simulation parameters, such as complex permittivity and permeability, can be varied to minimize the difference between the simulated and the measured values and that is how you obtain the closed gas of the value. So, once the discrepancy is within acceptable tolerance, the final varied complex permittivity and permeability can be adopted as the characteristic parameter of this metamaterial under test and this is how the method works.

So, with that, we move on to the characterization methods of optical metamaterials now.



Characterization Methods for Optical Metamaterial



So, those were for microwave metamaterials. Here in the optical metamaterials, you use scanning electron microscopy (SEM), which is popularly used for high-resolution surface imaging.

Scanning Electron Microscopy (SEM)

➤ **Overview:**

- Scanning electron microscopy (SEM) is a method for high resolution surface imaging
- It is a great way to obtain information about a sample's surface topography and composition in industries such as microelectronics, semiconductor, medical devices.

▪ **Benefits of SEM testing include:**

- Digital image resolution as low as 15 nanometers.
- Magnification for all imaging is calibrated to a traceable standard (Image analysis for coating thicknesses, grain size determinations and particle sizing can be applied to the saved images)
- Qualitative elemental analysis, standard-less quantitative analysis, x-ray line scans and mapping.



Figure: A typical scanning electron microscopy (SEM) device



Source: <https://www.innovatechlabs.com/materials-analysis-electron-microscopy/>

This is how a typical SEM device looks. So, it is a great way to obtain information about the samples' surface topography and composition in industries. Such as microelectronics, semiconductors, and medical devices, they do a lot of the same. So, the benefits of SEM testing will include that you can obtain a digital image of your metamaterial with a very high resolution of up to 15 nanometers. Okay. The magnification of all imaging is

basically calibrated to a traceable standard.

So, you can do the image analysis for coating thickness, grain size determination, and particle sizing; those can be applied to the saved images. Qualitative elemental analysis, standard less quantitative analysis, X-ray line scans, and mapping can also be done.

Next is transmission electron microscopy.

Transmission Electron Microscopy (TEM)

➤ Overview:

- Transmission electron microscopy (TEM) is an analytical technique used to visualize the smallest structures in matter.
- Unlike optical microscopes, which rely on light in the visible spectrum, TEM can reveal stunning detail at the atomic scale by magnifying nanometer structures up to 50 million times.
- This is because electrons can have a significantly shorter wavelength (about 100,000 times smaller) than that of visible light when accelerated through a strong electromagnetic field, thus increasing the microscope resolution by several orders of magnitude.
- To form a TEM image, a high energy electron beam is accelerated through an extremely thin “electron transparent” sample, typically thinner than 100 nm.



Figure: A typical transmission electron microscopy (TEM)

So, this is another analytical technique that can be used to visualize the smallest structure in the matter, okay. So, unlike the optical microscopes which basically rely on the light in the visible spectrum, TEM, this is how a typical TEM looks like.

The TEM basically reveals stunning details at the atomic scale by magnifying nanometer structures up to 50 million times. So, this is because the electrons They have sufficiently shorter wavelength which is like 100,000 times smaller than that of the visible light When accelerated through a strong electromagnetic field. So, that basically increases the microscope's resolution by several orders of magnitude. So, to form a TM image, a high-energy electron beam is accelerated through an extremely thin, electron-transparent sample thinner than 100 nanometers, okay. So, that can give you all details about a structure.

Another method that is used is called AFM, which stands for atomic force microscopy.

Atomic Force Microscope (AFM)

➤ Overview:

- AFM provides three-dimensional topographic imaging at sub-nanometer resolution in air, liquid, or vacuum, without extensive sample preparation.
- Compared with electron microscopes (SEM/TEM), AFM offers direct height measurement, lower cost, smaller size, and flexible operating environments.
- Thus; it is a versatile, cost-effective technique for nanoscale 3D characterization of chemical, mechanical, electrical, and magnetic properties.

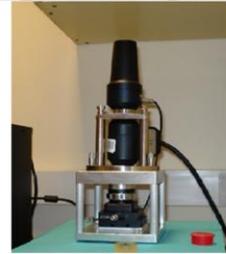


Figure: A typical atomic force microscope (AFM)

▪ Operating Modes:

- Contact mode: Tip maintains direct contact; suited for moderate roughness but may damage soft surfaces.
- Non-contact mode: Tip vibrates close to the surface without touching; ideal for delicate samples or fluid environments.
- Tapping mode: Tip intermittently contacts the surface, reducing lateral forces and preserving soft materials.

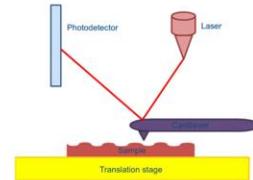


Figure: Schematic representation of AFM

Now this can basically give you a three-dimensional topographic image. At sub-nanometer resolution in air, liquid, or vacuum without extensive sample preparation. So, this is how a typical AFM microscope looks, and this is the schematic representation of AFM.

You have a photodetector, a laser, and a probe that goes very close to the surface, and you can actually image. So, this is a sample. placed on a translational stage. If you compare AFM with the two electron microscopes, SEM and TEM, you can see that AFM basically offers direct height measurement.

Lower cost, smaller size, and flexible operation. Thus, it is a versatile, cost-effective technique for nanoscale 3D characterization of chemical, mechanical, electrical, and magnetic properties. So, what are the operating modes at the AFM? So, there is a contact mode. So, here the tip will basically maintain direct contact. So, that is suitable for moderate roughness, but it may damage the soft surfaces.

So, the other one will be the non-contact mode. The tip vibrates close to the surface without touching it. So, this is ideal for delicate samples and fluid environments. And then there is tapping mode. So, it basically intermittently contacts the surface, reducing the lateral force, and that can help preserve the soft materials.

Atomic Force Microscope (AFM)

Working Principle:

- A sharp cantilever tip scans the surface while forces (van der Waals, repulsive/attractive) between tip and sample cause cantilever deflection, detected by a laser-photodiode system.
- Piezoelectric scanners move the sample with nanometer precision.
- Cantilever stiffness and tip sharpness are critical for resolution and sample protection.

Key Applications:

- Surface property measurement: Quantifies roughness, scratches, and microstructural defects.
- Damage characterization: Maps slip bands, persistent slip markings, and fatigue-induced surface features for crack initiation analysis.
- Surface treatment assessment: Evaluates nanocrystalline layers and residual stress after processes like ultrasonic nanocrystal surface modification.

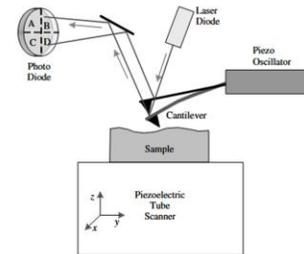


Figure: Schematic representation of AFM

So, here is the working principle of the AFM. You have a sharp cantilever tip, okay that scans the surface while forces such as the van der Waals it can be attractive or repulsive okay between the tip and the sample cause the cantilever deflection okay which is basically detected by the laser photodiode kind of system. So, a piezoelectric scanner basically moves the sample with nanoscale precision. So, this is the piezo oscillator, this is the laser diode, and this is your photodiode. So, the cantilever stiffness and the tip sharpness are both very critical for the resolution and sample protection.

What are the key applications? So, surface property measurements like it can quantify the roughness, scratches, and microstructural defects, okay. It can characterize the damage that it can map the slip bands, persistent slip markings and fatigue induced surface features for crack initiation analysis. Also, it can evaluate the nanocrystalline layers and residual stress after processes such as ultrasonic nano-crystallized surface modification. So, it can also basically assess surface treatment.

So, here is a table that compares AFM to other microscopy techniques.

Comparison of AFM with other microscopy techniques

- Table below compares AFM with SEM and TEM, highlighting AFM's clear advantages, while figure further shows that AFM has a much lower capital cost than other surface measurement tools.

Table: Comparison of attributes of various surface characterization tools

Parameter	SEM	TEM	AFM
Measurement environment	Vacuum	Vacuum	Air, water, gas, vacuum, etc.
Surface height	Not possible	Not possible	Possible
Measurement dimension	2D	2D	3D
Size of equipment	Large	Large	Very small
Cost	Expensive	Expensive	Cheap
Usage	Skilled operator needed	Skilled operator needed	Easy to use
Measurement speed	Fast	Fast	Slow

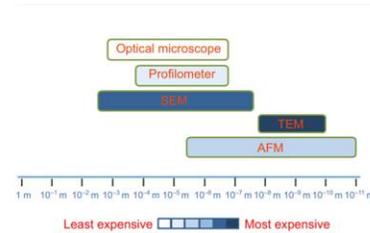


Figure: Comparison of capital cost of various surface measurement tools

So, you can see SEM, TEM, and AFM in this table. So, the measurement environment for TEM and SEM is vacuum, whereas AFM can be used in air, water, gas, and vacuum. Surface height measurement is only possible in an AFM. So, it is basically giving you 3D, whereas the other two give you 2D, okay. The size of the sample in SEM and TEM can be large, but in AFM, it is very small. However, it is very cheap, whereas TEM and SEM are expensive; they also require very skilled operators, but AFM is easy to use.

Also, SEM and TEM are fast methods; the measurement is fast, but AFM is slow. So, this is a capital cost of various surface measurement tools. So, you can see the darker one was the most expensive. So, TEM, the one that gives the highest resolution, is the costliest one; AFM and optical microscopy are much cheaper, okay.

The other methods for characterizing the reflectance or absorption spectrum in the UV-visible.

UV/VIS/NIR Spectroscopy

➤ Overview:

- UV/VIS/NIR spectroscopy is a non-destructive, non-contact optical characterization technique.
- It measures the light absorbed or reflected by a sample across the ultraviolet (UV), visible (Vis), and near-infrared (NIR) regions of the electromagnetic spectrum (approximately 175 to 3,300 nm)
- It is used to determine the optical properties (transmittance, reflectance and absorbance) of metamaterials.
- It can be applied to characterize semiconductor materials, coatings, glass and many other research & manufacturing materials.
- This technique analyzes electronic and vibrational transitions within molecules, providing information on a sample's composition, concentration, optical properties, and molecular structure.



Figure: Perkin Elmer Lambda 1050 Spectrophotometer

The infrared range is this spectroscopy: UV, visible, and NIR spectroscopy. Which is a non-destructive, non-contact optical characterization technique. It basically measures the light absorbed or reflected by a sample across the UV, visible, and infrared regions of the electromagnetic spectrum. that ranges from 175 to 3300 nanometers. So, it is used to determine the optical properties: transmittance, reflectance, and absorbance of the metamaterials.

It can be applied to characterize semiconductor materials, coatings, glass, and many other research and manufacturing materials. And this technique analyzes the electronic and vibrational transitions in molecules. Providing information on a sample's composition, concentration, optical properties, and molecular structure.

So, this is how it works?

UV/VIS/NIR Spectroscopy

➤ How it Works:

1. Light Source: A broadband light source emits light across the UV-Vis-NIR spectrum.

2. Sample Interaction: The light interacts with the sample, causing either absorption or reflection at specific wavelengths.

3. Electronic/Vibrational Transitions: In the UV-Vis regions, energy from light promotes electrons from highest occupied molecular orbital (HOMO) to lowest unoccupied molecular orbital (LUMO).

- In the NIR region, the technique probes vibrational overtone and combination bands, offering insights into molecular structure.

4. Detection: A detector measures the amount of light that is transmitted through the sample or reflected from its surface.

5. Spectrum Generation: The data collected is used to generate a spectrum, which shows the relationship between the intensity of absorbed or reflected light and its corresponding wavelength.

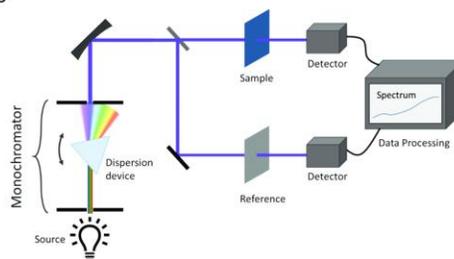


Figure: Schematic of UV-Vis-NIR spectrophotometer

You have a light source that is typically a broadband source, emitting light across the UV, visible, and NIR spectrum.

Then you put your sample over here, okay? So, the light is basically interacting with the sample, okay? causing the light to either get absorbed or reflected at particular wavelengths. And in the UV-visible region, the energy of light can promote the electrons in the material to occupy higher energy levels. Higher energy orbitals. So, you can actually send the electrons from the highest occupied molecular orbital, which is the HOMO. You can send them to LUMO, which is the lowest unoccupied molecular orbital, okay.

So, this basically for the organic ones ok normally you can have for the semiconductors you can basically send it from the highest level of the valence band to the lowest unoccupied energy level in the conduction band, right? So, in the NIR region, the technique provides vibrational overtone and combination bands that offer insights into the molecular structure, right? So, you also need a detector that measures the amount of light that is getting transmitted through the sample. Okay, you also have another detector that can measure the reflectance, right? And finally, you can generate the spectrum. where you are basically collecting the data, doing the data processing, and You show the relationship between the intensity of the absorbed and the reflected light according to different wavelengths.

Finally, we will look into another method called ellipsometry. Spectroscopic ellipsometry is basically a non-destructive, non-contact, and non-invasive optical method.

That is based on the change in the polarization of the state as it is reflected obliquely from a thin film sample.

Ellipsometry

➤ Overview:

- Spectroscopic ellipsometry is a non-destructive, non-contact, and non-invasive optical technique which is based on the change in the polarization state of light as it is reflected obliquely from a thin film sample.
- Ellipsometry is an optical technique for investigating the dielectric properties (complex refractive index or dielectric function) of thin films.
- It uses a model based approach to determine thin film, interface, and surface roughness thicknesses, as well as optical properties for thin films ranging in thickness from a few angstrom to several tens of microns.

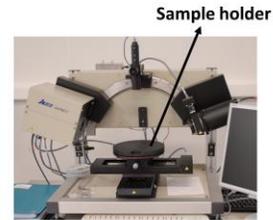


Figure: A typical Ellipsometric device

▪ Applications:

- **Thin Films & Coatings:** Determine thickness, refractive index, and extinction coefficient for microelectronics, photovoltaics, OLEDs, and optical coatings.
- **Metamaterials:** Characterize size-dependent optical properties and plasmonic behavior.
- **Real-Time/In-Situ Monitoring:** Track growth rates and property changes during deposition.

Now, why do you require this? This ellipsometry is an optical technique that can investigate the dielectric properties. such as the complex refractive index or dielectric function of thin films. It uses a model-based approach to determine thin film interface and surface roughness thickness. as well as the optical properties of the thin films ranging in thickness from a few angstroms to several tens of microns.

So, what are the applications? You can use them for thin films and coatings to determine the thickness, refractive index, and extinction coefficient. Which are very important for microelectronics, photovoltaics, OLEDs, and optical coatings. You can use them for metamaterials; you can characterize the size-dependent optical properties and the plasmonic behavior. You can also do real-time or in-situ monitoring, which means you can track the growth rate and property changes.

That is happening during the deposition.

So, this is how it works?

Ellipsometry

➤ How it Works:

- 1. Polarized Light:** Ellipsometry starts with polarized light, which has a specific orientation of its electric field.
- 2. Interaction with Sample:** This polarized light reflects off or transmits through the sample.
- 3. Polarization Change:** The interaction causes a change in the light's polarization state. This change is measured by a detector.
- 4. Psi and Delta:** The measured changes are represented by two values, Psi (Ψ) and Delta (Δ), which describe the relative and absolute changes in the light's amplitude and phase.
- 5. Model Fitting:** These measured values (Ψ and Δ) are then compared to a theoretical model.
 - By adjusting the model parameters (like film thickness and refractive index), the best fit between the model and the experimental data is found, allowing for accurate material characterization.

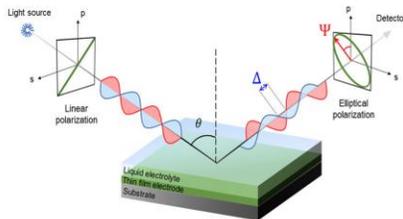


Figure: Schematic of Ellipsometry Set-up

you send your elliptic meter, which basically starts with polarized light that has a specific orientation of the electric field. And then it interacts with the sample; so, this polarized light is basically reflected off or transmitted through the sample depending on that type. And then there could be a polarization change, okay, that you can see here, and the interaction causes. This change in this polarization state is basically measured by the detector.

So, you are sending a linear polarization, and because of this, you are getting a phase shift between the two polarizations, which are orthogonal, and then when they add up, you get elliptical polarization, right? So, what is happening is that the measured change is basically represented by two values, psi and delta. That basically can describe the relative and absolute change in the light's amplitude and phase. So, this is psi (Ψ), and this is delta (Δ) that you see here.

And then you can do a model fitting where these measured values of Ψ and Δ can be compared to the theoretical model. And by adjusting the model parameters, such as film thickness and refractive index, you can find the best fit that matches this with the experimental data and that will allow you an accurate material characterization. So, with that we understood different methods for characterization of optical metamaterials previously we have seen the characterization methods for microwave metamaterials, and that basically concludes our discussion in this course.



So, I thank all of you for your attention and participation in this course. So, this will be all from my side. If you have any queries regarding this course or any of this lecture, please drop an email to this email address mentioning the course name and the lecture number on the subject line. Thank you very much. Thank you.