Course Name- Nanophotonics, Plasmonics and Metamaterials

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Week-12

Lecture -36

Hello students, welcome to lecture 36 of this online course on Nanophotonics, Plasmonics and Metamaterials. So, in this final lecture of the course we will discuss about different nano characterization techniques. So, here is the lecture outline, we will look into some basic optics behind different nano characterization techniques and then we will specifically look into the techniques of electron microscopy. We will see the difference between scanning electron microscopy SEM and TEM transmission electron microscopy. We will look into the scenarios where they are applicable, what are their resolution ok, all these aspects. Then we will also look into the techniques of scanning probe microscopy and we will go into the details of scanning tunneling microscopy, surface profiling and atomic force microscopy and also near field scanning optical microscopy and so on.

Lecture Outline

- Basic Optics
- Electron microscopy
  - Scanning electron microscopy
  - Transmission electron microscopy
- Scanning probe techniques
  - Scanning tunneling microscope
  - Surface profiling and the AFM
  - Near-field scanning optical microscopy
- Summary

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And then we will kind of summarize all the different characterization techniques we have studied in this lecture and that will conclude this. So, if we look into the basics of nano characterization methods, it means how do we actually see microscale objects. So, the first thing that will come to your mind from your school days will be a compound

microscope. So, this is how a compound microscope looks like ok.

And a compound microscope is mainly used for studying the structural details of cells, tissues and sections of organs. So, it has got different components as you can see here there is eyepiece, this is the body tube, there is a coarse adjustment knob and fine adjustment knob, this is the revolving nose piece ok and this is the objective, you can keep your sample over here ok, this is the condenser and then this is the mirror base ok. So, this is how a compound microscope looks like. Now, if you can see the working principle of this compound microscope, you will see that there are basically two lenses helping you in this particular microscopy. The lens near the object is called objective lens and this actually forms a real inverted magnified image of the object.

Basic Optics: Geometric optics using compound microscope

- The compound microscope is mainly used for studying the structural details of cells, tissues, or sections of organs.
- The lens nearest the object, called the objective, forms a real, inverted, magnified image of the object.
- This serves as the object for the second lens, the eyepiece, which produces the final image.
- The final image is enlarged and virtual.
- For magnification,

$$m_0 = \frac{h'}{h} \quad \text{Using} \quad \tan\beta = \frac{h}{f_0} = \frac{h'}{L}$$

We get, $m_0 = \frac{h'}{h} = \frac{L}{f_0}$ where L is the distance between second focal point of the objective and first focal point of eyepiece.

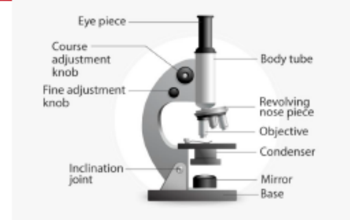

Figure: A compound microscope

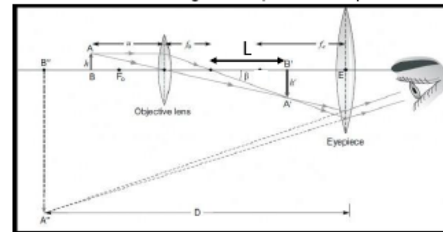

Figure: Working principle of compound microscope

Source: https://byjus.com/physics/compound-microscope/

So, this is the object that you are trying to see AB with height h ok and this objective lens will form a real inverted image ok and that is also magnified and the height will be h prime and this image is called A prime B prime ok. And this will basically serve as an object for the second lens which is the eyepiece ok and this eyepiece will produce the final image which is the much larger magnified version ok and you can see the final magnified image is A double prime B double prime and it is really enlarged and it is a virtual one. So, for magnification you can say the magnification factor for the objective lens is m_o that is h'/h and if you use if this is the angle beta ok you can say tan beta equals h over f_0 where f_0 is this focal length ok, h is the height of the object and that will be equal to h prime over L. So, L is basically this particular length ok. So, it is the distance between the second focal point of the objective and the first focal point of the eyepiece lens ok.

Basic Optics: Geometric optics using compound microscope

- If the final image A'' B'' is formed at the near point.

  then Angular magnification,

$$m_e = 1 + \frac{D}{f_e}$$

- When the final image is formed at infinity, the angular magnification due to the eyepiece is given by

$$m_e = \frac{D}{f_e}$$

- Thus, the total magnification when the image is formed at infinity, is

$$m = m_0 m_e = \frac{L}{f_0}\frac{D}{f_e}$$

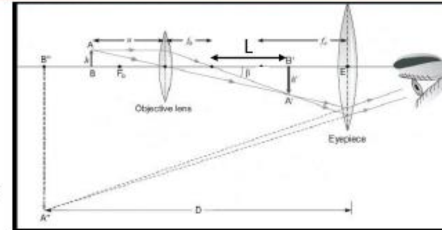

Figure: Working principle of compound microscope

Source: https://byjus.com/physics/compound-microscope/

So, you can actually obtain mo in terms of h prime over h and that can be taken in terms of L over f naught. So, that is how you obtain the magnification factor of the objective lens. Now, if you look into the final image that is A prime B prime it is getting obtained from the eyepiece. So, that particular lens also has got a amplification factor or magnification factor. So, me can be written as $m_e = 1 + \frac{D}{f_e}$ ok.

Basic Optics: Wave propagation and diffraction

- To get a quantitative understanding of imaging and feature resolution, we should know the basics of wave equation.

- We use the Helmholtz form of the wave equation

$$\nabla^2 E(\vec{r}, \omega) + k_0^2 E(\vec{r}, \omega) = 0 \longrightarrow \text{Eqn (1)}$$

  Where, $(k_0 = \omega / c)$

- Let's consider an optical beam propagating close to the z-axis.

- When the transverse coordinates are Fourier transformed, we get

$$E(k_x, k_y, z, \omega) = \frac{1}{(2\pi)^2} \iint dxdy e^{-i(k_x x + k_y y)} E(x, y, z, \omega)$$

  Equation (1) becomes

$$\frac{d^2}{dz^2} E(k_x, k_y, z, \omega) + (k_0^2 - k_x^2 - k_y^2) E(k_x, k_y, z, \omega) = 0, \longrightarrow \text{Eqn (2)}$$

So, the D is this particular distance over that is the distance of the final image that is formed from the eyepiece ok. And when the final image is formed at infinity ok, the angular magnification due to the eyepiece can be simply given as D over fe.

Basic Optics: Wave propagation and diffraction

- Equation (2) can be solved for two counterpropagating plane waves.
- The solution we seek is propagating along the positive z-axis, which is given by

$$E(k_x, k_y, z, \omega) = E(k_x, k_y, 0, \omega)e^{i\sqrt{(k_0^2-k_x^2-k_y^2)}z}$$

- For a linear system for which the input is at z = 0, the spatial frequency (k_x, k_y) response function is

$$H(k_x, k_y, z) = \frac{E(k_x, k_y, z, \omega)}{E(k_x, k_y, 0, \omega)} = e^{i\sqrt{(k_0^2-k_x^2-k_y^2)}z}$$

this represents the plane-wave decomposition of light beam propagation a distance z in space.

- The relation between the spatial expressions for the electric field and the response function is

$$E(x, y, z, \omega) = \iint dk_x dk_y e^{i(k_x x+k_y y)} E(k_x, k_y, 0, \omega) H(k_x, k_y, z).$$

So, you can ignore this 1 plus thing 1 can be ignored because this is very large ok. And the total magnification when the image is formed at infinity ok can be written as m that is the overall magnification factor that is mo times me that is L over f naught times D over f e. So, with that you are able to find out the overall magnification factor of this compound microscope.

Basic Optics: Wave propagation and diffraction

- The scalar electric field is expressed in spatial coordinates using the convolution form

$$E(x, y, z, \omega) = \iint dx' dy' E(x', y', 0, \omega) G(x - x', y - y', z).$$

- The propagator $G(x - x', y - y', z)$ is the Fourier transform of the response function (k_x, k_y, z).
- It is also referred to as the spatial impulse response function:

$$G(x, y, z) = \iint dk_x dk_y H(k_x, k_y, z) e^{i(k_x x+k_y y)} \longrightarrow \text{Eqn (3)}$$

- The evaluation of Eqn (3) yields the result

$$G(x, y, z) = -\frac{ik_0}{2\pi\sqrt{z^2+\rho^2}} \cdot \frac{z}{\sqrt{z^2+\rho^2}} e^{ik_0\sqrt{z^2+\rho^2}} \left(1 + \frac{i}{k_0\sqrt{z^2+\rho^2}}\right) \quad \text{Where } \left(\rho = \sqrt{x^2+y^2}\right)$$

- The second term is called the obliquity factor and is expressed as a cosine function $\left(\cos\Theta = \frac{z}{\sqrt{z^2+\rho^2}}\right)$, where Θ is the off–axis angle.

Now, there is another important parameter in magnification is the resolution ok. So, what is the minimum resolution or the minimum size that you can see ok. So, increasing magnification of objects with light has a limit because of how waves of light basically behave. So, when you make things really big with a microscope and they get close in size to the wavelength, they will start looking blurry ok. So, Ernst Abbe he figured out

that there is a limit to how small you can see things with a microscope ok.

Basic Optics: Wave propagation and diffraction

- For small off-axis angles $\rho \ll z$, the paraxial approximation can be used for the propagator, yielding

$$G_p(x,y,z) = -\frac{\mathrm{i}k_0}{2\pi z}\mathrm{e}^{\mathrm{i}(k_0 z - k_0\rho^2/(2z))}$$

- The corresponding paraxial spatial frequency response function is

$$H_\mathrm{p}(k_x,k_y,z) = \mathrm{e}^{\mathrm{i}k_0 z}\mathrm{e}^{-\mathrm{i}\kappa^2 z/2k_0} \quad \text{Where } \left(\kappa = \sqrt{k_x^2+k_y^2}\right)$$

- H_p describes a spatial translation of a paraxial field.
- The spatial coordinate expression for the electric field is

$$E(x,y,z,\omega) = \iint \mathrm{d}k_x \mathrm{d}k_y \mathrm{e}^{\mathrm{i}(k_x x + k_y y)} E(k_x,k_y,0,\omega) H_\mathrm{p}(k_x,k_y,z) \longrightarrow \text{Eqn (4)}$$

This is the Fresnel diffraction formula

And it is determined by something called the resolvable size of a feature. So, that is the resolution ok and it is given $\mathcal{R}_\mathbf{A} = \frac{\lambda}{2n_\mathrm{e}\sin\alpha}$. So, here you can see that alpha is this particular angle ok of the incident light. So, you can see that this is the incident light ok and this is the object plane, this is the aperture ok and lambda is the wavelength of light and n e sin alpha is basically the numerical aperture of the lens.

Basic Optics: Gaussian beams

- A focused Gaussian beam shape at z = 0 is expressed as

$$E(x,y,0,\omega) = E_0 \mathrm{e}^{-r^2/2w_0^2}$$

where w_0 is the Gaussian beam width at z=0.

- Applying the Fourier transform, the plane-wave decomposition of the Gaussian function is

$$E(k_x,k_y,0,\omega) = \frac{w_0^2}{2\pi}E_0\mathrm{e}^{-\kappa^2 w_0^2/2}$$

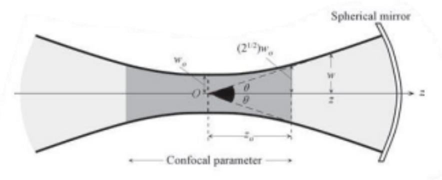

Figure: Schematic of a Gaussian beam. The region z_o is called the Rayleigh range and also the depth of focus.

- Applying this expression in Eqn (4), the integral is

$$E(x,y,z,\omega) = \iint \mathrm{d}k_x \mathrm{d}k_y \mathrm{e}^{\mathrm{i}(k_x x + k_y y)} \frac{w_0^2}{2\pi}E_0\mathrm{e}^{-\kappa^2 w_0^2/2} H_\mathrm{p}(k_x,k_y,z)$$

- The integral is evaluated with the explicit result

$$E(x,y,z,\omega) = \frac{w_0^2}{2\pi(w_0^2 + \mathrm{i}\,z/k_0)}\mathrm{e}^{\mathrm{i}k_0 z}E_0\mathrm{e}^{-r^2/2(w_0^2+\mathrm{i}z/k_0)}$$

Source: Kasap, Safa O. Optoelectronics and photonics. Pearson Education UK, 2013.

So, you can think of 2 different objects ok like this they produce an overlapping diffraction pattern like this when you are having a tiny aperture circular aperture ok. And the Rayleigh criteria for the diffraction limit to resolution states that 2 images are just

resolvable when the centre of the diffraction pattern of one is directly over the first minima of the diffraction pattern of the other. It means these 2 object can be resolved as 2 different objects when the maximum of one is exactly at the first minima of the other.

Basic Optics: Gaussian beams

- The result can be recast into the following form:

$$E(x,y,z,\omega) = \frac{w_0^2}{w(z)^2} e^{i\varphi} e^{ik_0 z} E_0 e^{-r^2\left(\frac{1}{w(z)^2}+\frac{ik_0}{R(z)}\right)/2}$$

Where
$$w(z)^2 = w_0^2\sqrt{1+(z/z_0)^2},$$
$$R(z) = \frac{(z^2+z_0^2)}{z}.$$

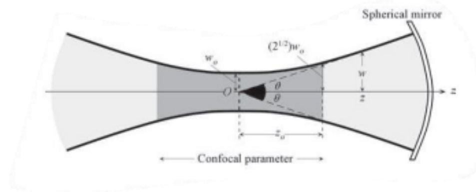

Figure: Schematic of a Gaussian beam. The **region z_o** is called the Rayleigh range and also the depth of focus.

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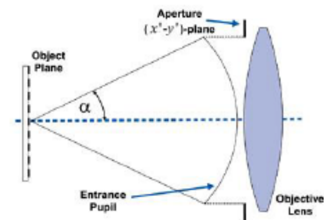
Source: Kasap, Safa O. Optoelectronics and photonics. Pearson Education UK, 2013.

Basic Optics: Resolution

- Increasing magnification of objects with light has a limit because of how waves of light behave.
- When you make things really big with a microscope, and they get close in size to the wavelength of light, they start to look blurry.
- A scientist named Ernst Abbe figured out that there's a limit to how small you can see things with a microscope, and it's determined by something called the "resolvable size of a feature", given by

$$\mathcal{R}_A = \frac{\lambda}{2n_e \sin\alpha}$$

where λ is the wavelength of light in vacuum and α is the incident angle of light



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Source: https://spie.org/publications/fg06_p16_na?SSO=1

So, for this curve this is the first minima and this maxima and this first minima they are overlapping similarly here also it is overlapping. So, these 2 objects are basically resolvable and if you think of the angle that these 2 objects make ok if you think in terms of angle you can say that 2 point objects are just resolvable if they are separated by this particular angle. So, what is the angle you can see here that is 1.22 lambda by d. So, if theta is just this you can see those 2 different objects. What is lambda here? Lambda is

the wavelength of light and d is the diameter of the aperture or lens mirror with which you are basically observing the 2 objects. Now can we try to find out this is the angle ok that we understood now can we relate it to the separation between the 2 objects ok.

Basic Optics: Resolution

- The *Rayleigh criterion* for the diffraction limit to resolution states that: " two images are just resolvable when the center of the diffraction pattern of one is directly over the first minimum of the diffraction pattern of the other".
- Two point objects are just resolvable if they are separated by the angle

$$\theta = 1.22\frac{\lambda}{D}$$

where λ is the wavelength of light (or other electromagnetic radiation) and D is the diameter of the aperture, lens, mirror, etc., with which the two objects are observed.

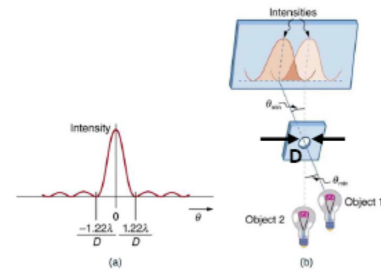

Figure: (a) Graph of intensity of the diffraction pattern for a circular aperture (b) Two point objects produce overlapping diffraction patterns.

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Source: https://openstax.org/books/college-physics-2e/pages/27-6-limits-of-resolution-the-rayleigh-criterion=1

So, here is an this particular figure a that shows the lens and 2 objects which are basically separated by distance x. Now according to Rayleigh criteria the resolution is possible when minimum angular separation is $\theta = 1.22\frac{\lambda}{D}$. And this angle can also be written as arc over the radius and that is x over d. So, that way you can correlate the minimum separation between the 2 objects which can be resolved using this particular lens. The numerical aperture here is basically a measure of the ability of the lens to gather the light and resolve fine details. So, if you see that the angle subtended by the lens at its focus is basically theta and theta is same as 2 alpha.

Basic Optics: Resolution

- Figure (a) shows a lens and two object separated by distance x.
- According to the Rayleigh criterion, resolution is possible when the minimum angular separation is

$$\theta = 1.22\frac{\lambda}{D} = \frac{x}{d}$$

- The Numerical Aperture (NA) here is a measure of the ability of the lens to gather light and resolve fine detail.
- The angle subtended by the lens at its focus is defined to be θ=2α.
- From the figure (b) and using the small angle approximation, we can write

$$\sin\alpha = \frac{D/2}{d} = \frac{D}{2d}$$

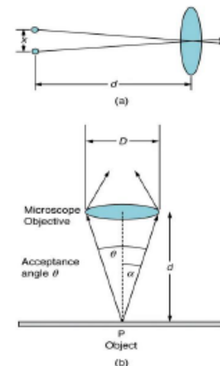

Figure: (a) Two points separated by at distance x and a positioned a distance d away from the objective (b) Terms and symbols used in discussion of resolving power for a lens and an object at point P.

So, the lens is basically accepting light at this angle theta this particular cone. So, from this particular figure b you can use small angle approximation and write sine alpha equals capital D by 2 over this distance d. So, you can write sinα = D/2d. So, small d is basically the distance of the object from the lens and d is basically the size of lens. So, that way you can also find out what is the numerical aperture for this lens.

NA is basically n times sine alpha which you have seen previously. n is basically the refractive index of the medium between the objective lens and the object which is placed at point P. So, whatever is the refractive index of this media that will go as n and sine alpha is this alpha is this half angle. So, that way you can correlate now what is your x. x can be the separation between the two objects which are resolved.

Basic Optics: Resolution

- The NA for a lens is NA = nsinα, where n is the index of refraction of the medium between the objective lens and the object at point P.
- From this definition for NA, we can see that the resolution is given by

$$x = 1.22\frac{\lambda d}{D} = 1.22\frac{\lambda}{2\sin\alpha} = 0.61\frac{\lambda n}{NA} \longrightarrow \text{Eqn (5)}$$

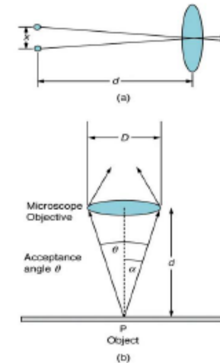

Figure: (a) Two points separated by at distance x and a positioned a distance d away from the objective (b) Terms and symbols used in discussion of resolving power for a lens and an object at point P.

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Source: https://openstax.org/books/college-physics-2e/pages/27-6-limits-of-resolution-the-rayleigh-criterion=1

So, x =λd/D. So, from that you can write $1.22\frac{\lambda}{2\sin\alpha}$ and that can be like simplified to $0.61\frac{\lambda n}{NA}$. So, that way for a given lens if NA is known refractive index of the medium in which you are imaging that is known and the wavelength is known you can actually find out the minimum distance that has to be there between the two objects to be identified separately or they should be resolved if that is the minimum separation between the two objects.

Basic Optics: Depth of field

- The depth of field is a sharpness criterion for how well objects within a certain longitudinal range will be in focus.
- Figure shows a simple illustration of the depth of field.
- Light focused by a lens and the NA is defined by D/2f, where D is the clear aperture diameter of the lens.
- The focal plane is indicated by a vertical dashed line.
- A cylinder of radius $\mathcal{R}_R$ is drawn around the focus and intersects the boundary rays.
- The length of the cylinder is the depth of field. By geometrical considerations, the depth of field is given as

$$D_f = 1.22\,\lambda/\text{NA}^2$$

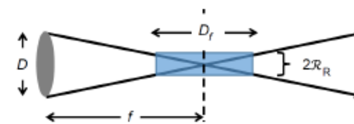

Figure: Diagram for the determination of the depth of field.

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So, this is the final answer you can obtain that this is the resolution of that particular lens. x equals 0.61 lambda times n times NA. Another important parameter for imaging will be the depth of field. Now the depth of field is basically is the sharpness criteria for

how well objects within a certain longitudinal range will be in focus.

So, in this particular figure it gives you an idea about this depth of field. So, here you can see light focuses by a lens at this particular focal point f and the numerical aperture is defined by D over 2 f. So, D is basically the clear aperture of the lens and you can see this is basically the focal plane the vertical dashed line. Now if you draw a cylinder of radius RR around this point. So, this is intersecting the boundaries and the length of the cylinder is basically the depth of field Df.

And if you do the geometrical consideration you can find out the value of Df to be 1.22 lambda over NA square. So, within this particular length the object will be in focus outside that it will be out of focus. So, these are important parameters through which you can actually tell how whether your microscope will be able to resolve the kind of features that you are looking for. Because when you are doing some nanofabrication or like fabrication of nanophotonic structures, metamaterial, metasurfaces you already know the element size and their periodicity.

So, depending on that you can actually decide which microscope what wavelength you should be using for seeing those features which you have fabricated. So, with that we move on to the next topic which is electron microscopy. And we will discuss two most popular electron microscopy techniques that is scanning electron microscopy and transmission electron microscopy. So, electron microscopy the name itself suggest that we are basically using electrons instead of light. And why we need that to examine tiny things on a nanoscale.

Because we understood from the compound microscope equations that the resolution is basically limited by lambda right. So, you can say it is lambda by 2 NA. So, lambda for optical frequency is very high. It is like 400 to 780 nanometer. So, the minimum size that you can resolve is also in several hundreds of millimeter or tens of millimeter.

Electron microscopy

- To examine tiny things on a nanoscale, we can use electrons instead of light.
- Electrons, which we usually think of as tiny particles, can also behave like waves according to quantum physics.
- We express this using an equation: $p = h/\lambda$, where p represents momentum, h is Planck's constant, and λ is the electron's wavelength.
- When we apply high voltages to speed up electrons, their wavelengths become much smaller than the wavelengths of light used in regular optical microscopes.
- This is helpful because smaller wavelengths can reveal tinier details.
- The energy of these accelerated electrons is determined by the voltage we apply.
- We calculate this energy using the formula $K = eV$, where K is the kinetic energy, e is the electron's charge, and V is the voltage.

So, if you want to go below that you should use electrons instead of photons. So, electrons which we usually think of as the tiny particles can also behave like waves according to point of physics. And if we express this using an equation that p equals h by lambda where p is basically the momentum, h is Planck's constant you can find out what is the wavelength of that particular electron. And when we use high voltage to speed up electrons their energy increases or the momentum increases ok. So, their wavelength becomes much more smaller as compared to the wavelength of light and that those are used in regular optical microscopes.

So, electron microscopy thus helps us look at very very tiny details or tinier details ok because of their smaller wavelengths. And the energy of this accelerated electrons can be determined by the voltage that has been applied. So, you can use this particular formula for the energy that K equals eV. So, K is basically the kinetic energy is the electron charge and V is the voltage that you are applying. Now this is how a typical high resolution SEM system looks like SEM is scanning electron microscopy right or you can simply call scanning electron microscope the instrument is called electron microscope ok.

So, SEM is a kind of electron microscope that uses a fine beam of focused electrons to scan a samples surface. So, what it does it basically records information about the interaction between the electrons and the samples and thus it creates a magnified image. So, you can magnify an image up to 2 million times using SEM and that is really really good. SEM images can give you insight into the samples topography and its elemental composition like what are different elements are present and it captures a 3D black and white image of thin or thick samples ok. So, this is you can use SEM for thin or thick

samples ok.

Electron microscopy: Scanning electron microscopy

- At the top of the column(figure a), electrons are emitted from a source and accelerated to a small aperture by a potential difference V.
- The primary electron beam reaches the sample to produce reflected (ballistic) backscattered electrons and secondary electrons.
- Secondary electrons are emitted from the surface after scattering of the primary beam under the surface.
- After the primary beam is scattered within the sample, its energy is imparted to excite atomically bound electrons in the material.

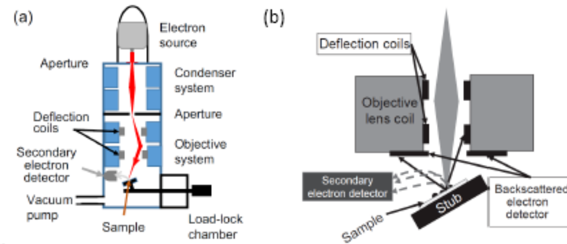

Figure: (a) SEM illustration with major elements. (b) Primary electrons incident on the sample surface

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And the size of the sample is limited only by the size of the electron microscope chamber. So, there is no technological limitation on the size it is basically the limitation by the size of the chamber fine. Now this particular figure on the right it shows an illustration of how the SEM machine looks like. So, at the top of the column you can see there is an electron source. So, electron is basically emitted from here and it is accelerated to a small aperture by the potential difference V and you can see that the primary electrons beam reaches the sample.

Electron microscopy: Scanning electron microscopy

- At each inelastic collision, the primary electrons slow down while at the same time ionizing the atoms around them.
- The primary electrons can also knock out inner core electrons from the atoms, which will result in characteristic X-ray emission from the surface.
- The X rays, have energy spectra that can be used to determine the abundance of atomic constituents near the sample's surface.
- The backscattered electrons also provide quantitative information about the elements in the material.

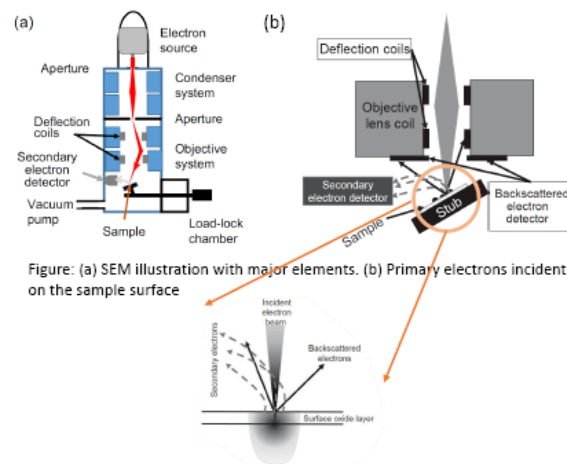

Figure: (a) SEM illustration with major elements. (b) Primary electrons incident on the sample surface

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This is where the sample is kept to produce. So, primary electrons will come and hit this and they will produce reflected or you can say ballistic back scattered electrons and

secondary electrons. So, secondary electrons will be basically emitted from the surface after scattering of the primary beam under the surface. And after the primary beam is scattered within the sample its energy is imparted to excite atomically bound electrons in the material.

And this will be done under vacuum. So, there is a vacuum pump as you can see and at each inelastic collision the primary electrons slow down while the same time ionizing the atoms around them. So, this is the mechanism that is happening and the primary electrons can also knock out the inner core electrons from the atoms which will result in the characteristic X-ray emission from the surface and that is how you are able to get the elemental signature. The X-rays which have energy spectra that can be used to determine the abundance of atomic constituents near the samples surface. And as it if you look into this part what is happening here you can see that this is the direction of incident electron beam, this is basically the back scattered electrons and these are the secondary electrons. And this back scattered electrons also provide quantitative information about the elements in the material.

Now here is some example of SEM images this is an image of an ant and this is an image of a photonic crystal waveguide. So, this is the length scale bar it is 2 micron here. So, you can see that they can display significant depth of field. You can actually see the structure very well. And this deep focus capability in SEM is a result of the electron beams used they are having a very small numerical aperture and that is why you can see such beautiful depth deep focus.

Electron microscopy: Scanning electron microscopy

- SEM (Scanning Electron Microscope) images have a remarkable characteristic: they can display a significant depth of field.
- This deep focus capability in SEM images is a result of the electron beams used having a small Numerical Aperture (NA).
- At low magnification shown for the specimen from nature in Figure (a), the details of the eye are clear and the antenna and leg in the foreground are equally in sharp focus.
- At higher magnification in Figure (b), the nanoscale holes are clearly imaged and they have a sharp appearance over many periods.

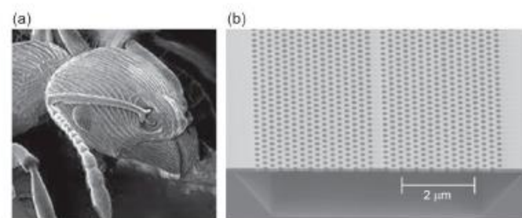

Figure: Examples of SEM images. (a) An image of an ant (b) An image of a photonic crystal waveguide.

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A low magnification shown for the sample from nature so like this is from for an ant as I told. Here you can see the details of the eyes are also very clear ok and the antenna and

the legs they are equally in sharp focus ok. And if you look into this figure that is this particular photonic crystal here you can see at higher magnification this nanoscale holes are also clearly imaged and they have a very sharp appearance over many many periods. So, that is how you are actually able to see your structure very well with SEM. Now there is another method that is also very popularly used which is called transmission electron microscopy.

Electron microscopy: Transmission electron microscopy

- TEM works on the same principle as an optical microscope.
- The side-by-side analogy between the two is shown in Figure.
- To allow electrons to pass through a sample in a Transmission Electron Microscope (TEM), the sample needs to be incredibly thin, typically around 50 nanometers thick.
- In TEM, scientists often prepare specimens by using a microtome, a specialized tool, to slice very thin sheets from their samples.
- These sheets are then placed on a grid that conducts electricity.

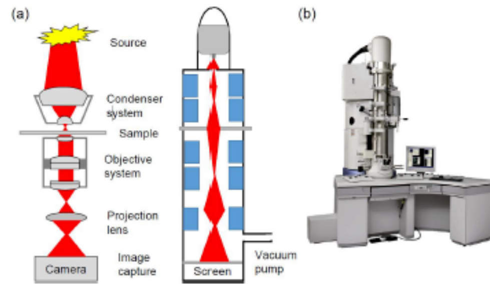

Figure : (a) Comparison of the elements of an optical microscope and a TEM instrument. (b) Image of a TEM instrument.

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So, here you can see that the sample is basically placed here ok and TEM basically works in the same principle of an optical microscope. So, you can actually see the side by side analogy of the two ok. So, this is an optical microscope and this is a TEM and this is how things work. So, you have the sample you have the objective system here you have projection lens here and then finally, you are capturing the image and this is where the source is fine. So, this is typically how a TEM instrument will look like in your nanotechnology center.

Electron microscopy: Transmission electron microscopy

- The process of making the sample even thinner depends on the material's properties
- It involve methods like diamond cutting, ultrasonic cutting, mechanical thinning, grinding, or ion milling.
- Preparing samples for TEM while preserving their tiny features is a challenging task that demands careful attention to each step of the process.
- The sample is placed after the condenser lens, where illumination covers the region to be imaged.
- The electron beam passes through the sample undeflected, and the objective and projector lens areas magnify the image as in a compound microscope.

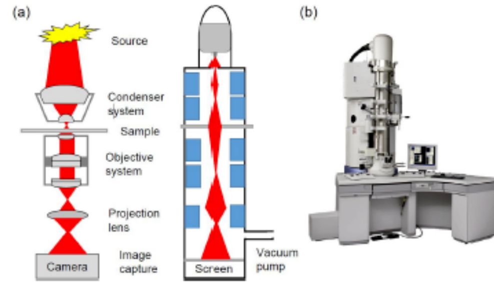

Figure : (a) Comparison of the elements of an optical microscope and a TEM instrument. (b) Image of a TEM instrument.

So, you have electron source here. So, it allows the electrons to be generated and then you need a sample, but in this case there is a catch the sample has to be thin ok. It should be very thin typically 50 nanometers ok. So, that there is transmission through this sample ok. So, in TEM scientists often prepare specimens by using a microtome which is a specialized tool that can slice very thin sheets from their samples and this sheets are then placed on a grid that conducts electricity and the process of making the sample even thinner depends on the materials properties. So, every material you cannot actually make very thin slices.

You can actually use different methods like diamond cutting, ultrasonic cutting, mechanical thinning, grinding, ion milling and all these things. So, preparing the sample for TEM is very important because you have to preserve the tiny features as well as you have to make this very thin. So, that you can images through a TEM. So, sample will be placed after the condenser lens ok and the illumination covers the region that is to be imaged and once the electron beam passes the sample ok and deflected then there will be this objective and projectile lens systems. They basically magnify the image as you actually do it in a compound microscope.

Electron microscopy: Transmission electron microscopy

- To see how well TEM can image, consider a 100-keV electron beam for which the de-Broglie wavelength is $\lambda = 3.89$ pm
- The Rayleigh resolution and depth of field, using eqn (5) for the electron beam with an NA of 0.01 is $\mathcal{R}_R = 0.24$ nm
- The high-resolution TEM images of silver nanoparticles in figure demonstrate the atomic-level detail that is possible to resolve with this instrument.
- The individual atomic sites are captured in these pictures.

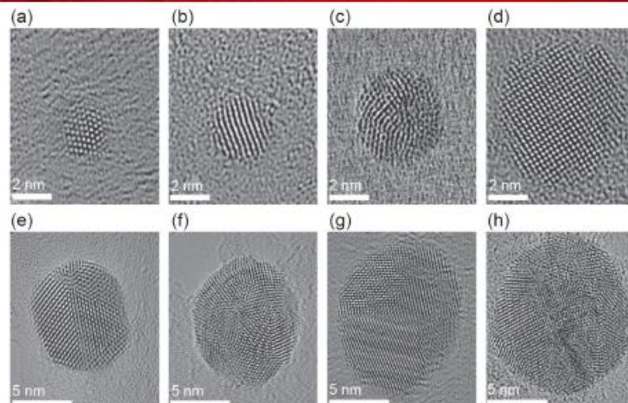

Figure : TEM images of silver nanoparticles.

So, the principle is very similar to an optical compound microscope, but here instead of light you are using electron beam. So, here are some examples of TEM images ok. So, these are TEM images of silver nanoparticles. Here the diameter of the particle is increasing from 2 to 3, 4.5, 6, 7.5, 9, 10.5 and 12 nanometer ok, but you can see the very fine details that you can obtain from TEM images ok. What is been used here a 100 keV electron beam is been used which has got a de Broglie wavelength of 3.89 picometer and that is why you are able to see such fine details of the nanoparticle structure ok.

Scanning probe techniques: Scanning tunneling microscope

- Scanning Tunneling Microscope (STM) is a high-resolution imaging technique that can see things at an atomic scale (about 0.1 nanometers).
- It works by using a metal tip that can be very precisely placed close to the surface being studied, and it has electronics to move the tip around the area of interest.
- STM achieves such incredible resolution because it relies on electronic quantum tunneling current, which is incredibly sensitive to tiny changes in how far the tip is from the surface.
- STM tips can be made extremely sharp, even just one atom wide, allowing for precise positioning.
- It uses piezoelectric materials that respond to voltage by changing in size at the nanometer scale.
- These materials help control the distance between the tip and the sample with incredible precision, down to a fraction of a nanometer.

So, the Rayleigh resolution and the depth of field for this electron beam with a numerical aperture of 0.01 is 0.24 nanometer ok. It is like 2.4 angstrom. So, you are actually able

to see atomic scale ok. So, they are very very high resolution right and the high resolution TEM image of the silver nanoparticles also gives you the idea of the atomic details and it tells us that the power of this particular microscope in terms of resolving fine details. So, the individual atomic sites are captured in this particular images. So, you can see carefully and see where the atoms are sitting ok. So, we understood that there are two popular systems for imaging one is SEM another is TEM.

So, this one is the cartoon showing the setup for SEM and here you can see the sample is at the bottom there is no restriction on the sample size it can be thin it can be thick ok and you are basically capturing the x-ray and the secondary electrons ok. So, the electron beam is basically focused and then you are capturing those and these are the back scattered electrons which are detected here and in TEM you put the sample on the top ok. So, you are basically measuring in the transmission mode right and this part is like the objective lens and the projector or intermediate lens ok. These are basically functioning the same way an optical microscope works.

Scanning probe techniques: Scanning tunneling microscope

- The tip is held by a piezoelectric sleeve that is voltage controlled to differential displacements of better than 1 nm.
- The voltage controls move the tip with nanometer accuracy to a spatial coordinate (x, y, z).
- There is an applied voltage between the tip and the sample to drive a tunneling current between them.
- As the tip is raster scanned across the surface (x, y), the height of the tip (z) is adjusted by changing the applied voltage on the piezoelectric actuator to keep the tunneling current constant.
- The voltage applied to adjust the tip height is recorded at each position (x, y), and the result is processed to render a three-dimensional image of the surface.

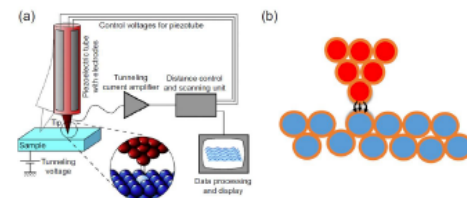

Figure: (a) Schematic of the STM apparatus (b) Caricature of the tip-sample interaction

Now I need to know that which one I should use in which case. So, in that particular position it is worth comparing this to electron microscopy techniques. So, if you compare SEM and TEM in terms of electron stream SEM has got fine focused beam whereas TEM has got broad beam. Image taken in SEM it can be a topological or surface image whereas TEM can see the internal structure. Resolution wise SEM is lower and TEM is much higher. So, in TEM it can go up to 2 million times for SEM whereas TEM can go up to 50 million ok and the images that you can see in SEM is 3D you have seen that and the photonic crystal that is 3D, but TEM actually gives you 2D.

Sample thickness again I have discussed SEM can be thick or thin does not matter, but

in TEM it should be very thin typically less than 50 nanometer. So, SEM does not penetrate the sample, but in TEM it does penetrate the sample. So, there are sample restrictions in TEM more whereas SEM there are less restrictions. In TEM sample preparation is needed SEM less preparation will be needed. So, obviously one things are in more details and there is more care to be taken for TEM.

TEM is more expensive as compared to SEM. However SEM will be faster, TEM will be slower because TEM actually magnifies much more and it goes into much finer details. So, if you look at the operation SEM is easier to use, TEM is much more complicated and it requires proper training. This also requires training, but this is more requires training. So, TEM is widely used to characterize nanomaterials at the atomic and nanoscale levels. It provides the information about the size, shape, crystal structure, defect and composition of nanomaterials.

This is crucial for the development of nanotechnology, material science and study of nanoparticles, nanotubes, nanowires, etc. There are other types of techniques for imaging which are also known as scanning probe techniques. So, now we will look into some of those techniques. The first one is scanning tunneling microscope, then we will look at surface profiling and AFM and finally we will look into near field scanning optical microscopy. So, scanning tunneling microscopy or STM is a high resolution imaging technique that can see things at an atomic scale.

Scanning probe techniques: Scanning tunneling microscope

- A tip interaction with the sample surface is illustrated in Figure .
- The electron cloud around the atoms is represented as its skin.
- The electron wave functions extend into the space between the tip and the sample and a current passes.
- A supertip is made when there is a single atom at the tip.
- This atom precisely defines the position of the tip and exquisite information on the local electronic states on the sample is obtained.

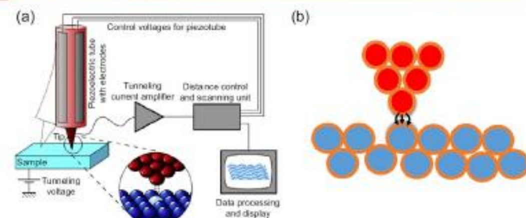

Figure: (a) Schematic of the STM apparatus (b) Caricature of the tipe-sample interaction

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So, we are talking about 0.1 nanometer scale. It works by using a metal tip that can be very precisely placed close to the surface which is under study and it has electronics to move the tip around the area of interest. So, STM achieves such incredible resolution that it relies on electronic quantum tunneling current which is incredibly sensible or

sensitive to the tiny changes in half of the tip is from the surface. So, the STM tip can be made extremely sharp and when I say extremely sharp it is like just one atom wide, ok. So, that it allows precise positioning and it has to be there because the resolution you are talking is in terms of 0.1 nanometer and it uses piezoelectric material that responds to voltage by changing in size at nanometer scale.

We look into a diagram and then things will be clear. So, these materials help control the distance between the tip and the sample with incredible precision down to a fraction of nanometer. So, this is how the STM setup looks like. So, this is the tip I was talking about. So, if you zoom it you will see that there is the tip is basically very very sharp only one atom at the tip end and this is the surface that you are studying. So, if you zoom then you can see that there is basically tunneling current and that is what you are basically measuring.

So, you can see that there is control voltages for the piezo tube that actually does the alignment, ok. So, this is a piezoelectric tube with electrodes and this is the tip, ok and this is the sample that you are scanning. So, the tip is held by a piezoelectric sleeve as you can see here that is voltage control to differential displacement of better than 1 nanometer. And the voltage control actually helps you move the tip with nanometer accuracy in the spatial coordinates like x, y, z. So, there is an applied voltage between the tip and the sample to drive the tunneling current between them and as the tip is raster scanned across the surface x, y, ok.

The height of the tip, ok, z is adjusted by changing the applied voltage on the piezoelectric actuator to keep the tunneling current constant. So, this is what is the, this is how you get an idea of the height, ok, of the surface because you have to adjust the height of the tip to make sure that the same current is maintained throughout. So, the voltage applied is just is then adjusted to the tip height and it is recorded at each position x, y. So, for every x, y location means on the at any point on the surface of the sample you will get the height of that particular sample, ok.

Scanning probe techniques: Scanning tunneling microscope

- The quantum mechanical action of the tip-sample interaction is illustrated in figure.
- A barrier is drawn between two materials in figure (a)
- The electron wave impinging on the barrier from the left side is largely reflected, but has some extension in and through the barrier.
- Each material has a work function defined as the minimum energy for an electron to escape from the material.
- For metals, the minimum is from the Fermi level to the vacuum states.

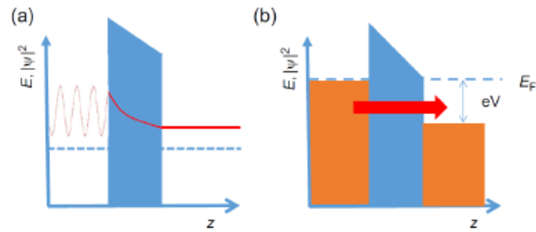

Figure:(a) The tunneling of the wave function through the barrier for an energy below the barrier (b) An applied voltage drives current across the tunneling gap from the filled electron states in metal 1 to the empty electron states in metal 2.

So, that is how you are able to produce a 3D image of the surface. Now, a tip interaction is it can be seen here, ok. And the electron cloud around the atoms is represented by its skin, you can see here. The electron wave function extends into the space between the tip and the sample and a current will pass that is the quantum tunneling current. And here a super tip is used which is a single atom at the tip of the, there is a single atom at the tip, right. And this atom precisely defines the position of the tip and the exquisite information of the local electronic states on the sample can be obtained.

So, this is one example of a famous and interesting image with atomic scale resolution that has been obtained using this technique. So, this is a coral made of 48 iron atoms placed on a copper 111 surface. So, you can actually see inside the coral the electronic density has a specially periodic oscillations and they reveal the true quantum nature of the electronic density, ok. So, scanning tunneling microscope basically works on the principle of quantum tunneling.

So, you can actually understand that from this particular diagram. So, the quantum mechanical action of the tip sample interaction can be understood from this. So, the barrier is drawn between the two materials, ok. So, these are the two materials and you can see the electron wave impinging on the barrier on the left side is largely reflected. But it somehow now extends into the barrier and it goes through the barrier, ok. So, each material has a work function which is defined as a minimum energy for the electron to escape the material. And if you take metal, for metals the minimum is from the Fermi level to the vacuum states, right. So, you can see that here. So, if you look carefully, so this shows the tunneling of the wave function through the barrier for an energy which is

lower than the barrier. So, this is the height of the barrier.

Scanning probe techniques: Surface profiling and the AFM

- Surface profiling is a technique used to measure and characterize the topography or surface features of an object or material.
- One of the tools commonly used for surface profiling is the Atomic Force Microscope (AFM).
- The AFM is a descendant of the profilometer and the STM, which represents a significant advance in metrology.
- The pickup of the profilometer comprises the stylus, stylus holder mechanism, transducer, and any signal conditioning associated with the transducer.
- The pickup is driven by a gear box, which draws the stylus over a surface at a constant speed.

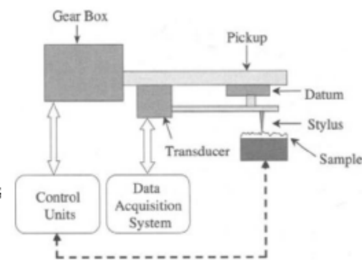

Figure: The schematics of a stylus profilometer

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Source: https://www.sciencedirect.com/topics/physics-and-astronomy/profilometers

This is a case when a voltage is being applied, ok, across the tunneling gap. So, what happens in that case? The tunneling happens from the filled electron states in metal 1 to the empty electron states in metal 2, this one. So, in this case also the, the barrier is having a higher height than the tube metal energy levels. So, with that we can look into the next one. The next method is called surface profiling and atomic force microscopy or AFM. So, here is a image of AFM atomic force microscopy and this is the schematic of a stylus profilometer.

So, when I say surface profiling, it is basically a technique that is used to measure and characterize the topography or the surface of a object or material. So, the common tool that is used for doing that is called atomic force microscopy, ok. So, this is basically a descendant of the profilometer and the STM which represents a significant advance in metrology. So, here what happens? You can see that there is a stylus, ok, that picks up the details from the sample.

So, the pickup of the profilometer, this part is called the pickup, ok. The pickup comprises of a stylus, then a stylus holder mechanism, a transducer and any single conditional circuit that is attached to the transducer. And this pickup is driven by a gearbox which draws the stylus over the sample at a constant speed. Why you need this? You need this to scan the sample. So, when the sample is scanned across the surface, the z-axis displacement of the stylus is basically sensed by the transducer, ok.

Scanning probe techniques: Scanning tunneling microscope

- As the system is scanned across a sample, the z-axis displacements of the stylus are sensed by the transducer.
- The transducer converts linearly the mechanical motion to the electrical signal.
- The signal, after being magnified by an electronic amplifier, is collected by a data acquisition system to generate the surface profile.
- The cantilever is attached to a piezoelectric scanner that controls the (x, y, z) position of the tip (figure b).

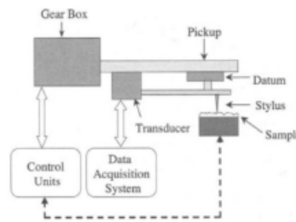

Figure (a): The schematics of a stylus profilometer

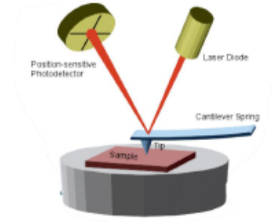

Figure (b) : A schematic diagram of the essential elements of an AFM.

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And you can actually see it bit more details from here. So, it is a bit of cantilever kind of arrangement. And the transducer will help you convert this mechanical motion to electrical signals. And the signals will then be magnified by an electrical amplifier. And you will use a data acquisition system to generate the profile of this particular surface. And the cantilever that is attached to the microelectronic scanner that controls the xyz position of the tip as you can see here, ok.

So, this is the tip of the cantilever. This is the sample, ok. And you can scan it. So, when you shine some light on this depending on the depth of the cantilever, ok, the reflected beam will be directed in a different direction, ok. So, that can be received by this position sensitive photo detector. So, that is also called a quad detector, ok. So, deflection of this cantilever as it moves across the surface is measured by this laser system and a quad detector.

Scanning probe techniques: Scanning tunneling microscope

- Deflection of the cantilever as it is moved across the surface is measured by a laser system and a quad-detector.
- The tip interacts with the surface via the van der Waals force.
- This has a repulsive action in the near field and attractive action at longer separations
- In the repulsive force region, the tip has a strong interaction with the surface.

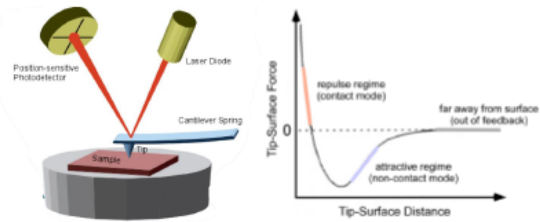

Figure (a) : A schematic diagram of the essential elements of an AFM.

Figure (b) : A van der Waals force curve.

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So, this is what you do it in AFM, atomic force microscopy. And the tip interacts with the surface and there is van der Waals force. And this force is basically repulsive in nature when it is near field and it becomes attractive at longer separation, ok. So, that is how you are basically scanning the surface. And this is what is the tip surface force.

So, you can see it is repulsive in the near field and then it goes into the attractive region. So, that is where it is in non-contact mode and it becomes positive. So, that is how atomic force microscopy or AFM images can give you the profile surface profile of a particular sample. You can also, there is another technique called near field scanning optical microscopy or NSOM. So, that is also an optical imaging technique. It also utilizes a sharp tip which will scan the sample surface while maintaining a constant height through the use of piezoelectric steering element, ok.

And it allows to achieve high resolution optical imaging at the nanoscale. So, this figure shows you the essential elements of an NSOM instrument. You have a laser, you have a fiber tip here, this is a sample, then you have the collection optics and this is the detector. And this is a close up of the fiber tip with light that is scattering through this particular sample and then there is the collection optics, ok. So, NSOM employs the laser source as you can see and that eliminates the sample through an optical fiber. And the fiber tip is basically tapered as you can see here and it is coated with metal that enables the light to pass through a sub wavelength aperture like this, ok.

Scanning probe techniques: Near-field scanning optical microscopy

- Near-field Scanning Optical Microscopy (NSOM), is an optical imaging technique.
- NSOM utilizes a sharp tip, which scans the sample's surface while maintaining a constant height through the use of piezoelectric steering elements.
- This allows it to achieve high-resolution optical imaging at the nanoscale.
- NSOM employs a laser source that illuminates the sample through an optical fiber.
- The fiber's end is tapered and coated with metal, enabling light to pass through a subwavelength aperture at the tip.

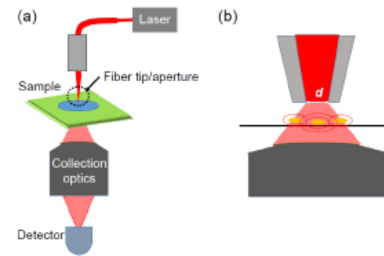

Figure :(a) The essential elements of an NSOM instrument. (b) A close-up of the fiber tip with light scattering features and collection optics below the surface.

And that is happening at the tip. Now when you have precise control of the fibers tip position, ok, you can scan a particular location and that is done by a piezoelectric element that is not shown here. The detector will measure the changes in the scattered light that is resulting from the tiny, surface features. If there are more bumps, there will be more scattering, if it is a plane surface, there will be less scattering. And that is how, the amount of light being collected by the detector will change depending on the surface roughness. The size of the aperture which is typically much smaller than the wavelength, it determines the ultimate resolution of this NSOM.

Scanning probe techniques: Near-field scanning optical microscopy

- Precise control of the fiber tip's position is achieved using piezoelectric elements.
- The detector measures changes in scattered light resulting from tiny surface features.
- The size of the aperture, which is much smaller than the wavelength, determines the ultimate resolution of the NSOM.
- Figure (b) shows that scattered light passing through the sample is gathered by a highly sensitive detector.
- The size of the smallest aperture is chosen based on a trade-off between achieving better resolution and maintaining a sufficient signal-to-noise ratio.

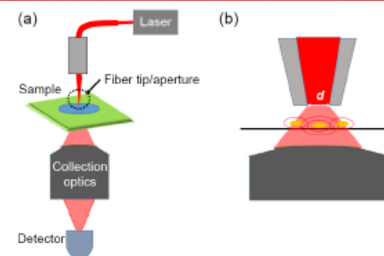

Figure :(a) The essential elements of an NSOM instrument. (b) A close-up of the fiber tip with light scattering features and collection optics below the surface.

So here you can see clearly what we are talking about the scattered light. So when the light passes through the sample, it is scattered and it is detected by this very sensitive

detector. And the size of the smallest aperture will be chosen in such a way there is a tradeoff between the resolution and your SNR, Single to Noise Ratio. So, that decides what should be the smallest aperture that you should choose for NSOM microscopy. So, smaller aperture diameters, it is clear that it can enhance resolution, but then the amount of light that will be detected by the detector will be very less.

So it you need a some value of SNR, Signal to Noise Ratio to detect a proper signal, ok. So if the detected light is very very feeble, then you will not be able to differentiate it from the noise. So it should be much higher than the noise flow. So that limits the dimension of the aperture, ok. So that will depend, that will decide the aperture diameter.

Now NSOM also works in different operational mode as you can see three different modes are shown here. So the first one is basically a transmission mode where the light will be passing through the small aperture of the tapered fiber and it scattered from the sample and it is getting detected on the other side, ok. So you are basically using a far field collection optics to collect this particular light, right. In sample B, this is basically illuminated based on total internal reflection like this, ok. So, you are using evanescent wave illumination from below, ok and you have tapered fiber collection.

Scanning probe techniques: Near-field scanning optical microscopy

- Smaller aperture diameters can enhance resolution but reduce the amount of light that can pass through the tiny aperture.
- NSOMs are used in several different operational modes.
- Figure (a), is a transmission mode with light passing through the small aperture of a tapered fiber
- The scattered light from the surface or near-surface volume irregularities is collected in the far field by optical means.
- In Figure (b), the sample is illuminated from below at an angle that exceeds the total internal reflection
- Here, the scattered light is collected through the small aperture at the end of the tapered fiber.

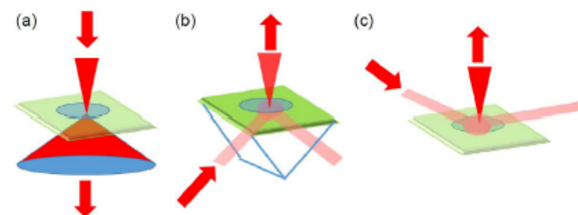

Figure : Three NSOM measurement modes: (a) fiber transmission with far-field collection optics, (b) evanescent wave illumination from below with tapered fiber collection, and (c) reflected light illumination with tapered fiber collection.

So this is how you are basically shining the light and this is how you are collecting it, ok. Here the scattered light is basically collected through the small aperture at the end of the tapered fiber. And the fiber must be in the near field for location sensitive detection of the scattered light. So this is the requirement for this particular setup. And the third one shows the reflection based measurement.

So the reflected light illumination is shown and you are using a tapered fiber for

collection. So you are illuminating the light from above, ok and a near field tip is placed to collect the scattered light from the local irregularities on the surface. So with that we can now compare the three different scanning probe techniques that we have studied. One is STM, another is AFM. So, this is atomic force, this is scanning tunneling microscopy and this is NSOM, near field scanning optical microscopy.

So as you can understand the principle for STM is quantum mechanical tunneling effect. For AFM it is basically the atomic force between the tip and the sample and for NSOM it is the near field interaction between the tip and the sample. Now what are the imaging method? It is topographic for STM where the constant current mode is maintained, ok. And for AFM also it is topographic and you basically do a force spectroscopy, you measure the force across the sample, ok.

And for NSOM it is basically optical, ok. You measure the near field or in far field. Now what is the imaging capability? You can obtain surface topography and electronic structure using STM. Sometime you can get surface topography and some mechanical properties using AFM and you can see optical contrast and surface topology for NSOM. If you talk about imaging resolution STM is atomic scale, you can go below Armstrong, ok. Whereas AFM can be sub nanometer to nanometer scale and whereas NSOM will have a lateral resolution of around 6 nanometer and vertical resolution of 2 to 5 nanometer.

Scanning probe techniques: Near-field scanning optical microscopy

- The fiber must be in the near field for location-sensitive detection of the scattered light.
- In figure (c), the sample is illuminated from above by light and a near-field tip collected the scattered light from local irregularities on the surface.

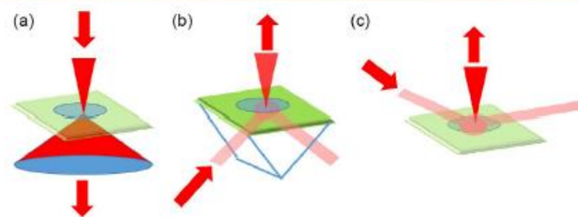

Figure : Three NSOM measurement modes: (a) fiber transmission with far-field collection optics, (b) evanescent wave illumination from below with tapered fiber collection, and (c) reflected light illumination with tapered fiber collection.

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So that has been demonstrated till now. Now what are the kind of samples each of them can handle? So STM requires the conductive and flat surface. AFM you can have wide range of samples whereas NSOM will require generally flat sample and you require that optical contrast. What are the applications? AFM is used for surface analysis at atomic

scale. You can study the electronic and molecular structure and you can also do manipulation of individual atoms that is very good, ok. And for AFM you can see the surface topology, you can map it, you can do some mechanical property measurement and biological imaging at the nanoscale.

Whereas NSOM can be used for optical imaging at with nanoscale resolution, ok. And you can use subcellular imaging in biology and you can do near field spectroscopy of nanomaterials. Now what are the limitations in each of this case? STM the drawback is that it requires only conductive samples. It cannot insulate image insulating materials, ok. And ultrahigh vacuum is needed. If you look into AFM it is also limited to surface properties and limited in terms of imaging speed and you cannot do imaging in liquid environment.

NSOM has also got some limitations. It is very short working distance and extremely shallow depth of field. The instrumentation is very complex and it requires precise alignment and it is having a very long scan time for large areas of sample. So here are the different techniques that we have understood. So let us quickly summarize the different techniques. So SEM that is scanning electron microscopy.

The applications are in material science, nanowires for gas sensing, semiconductor inspection, microchip assembly. TEM transmission electron microscopy. They are very popularly used in nanotechnology, biological and material research, forensic analysis. STM scanning tunneling microscopy is very useful for semiconductor science, electrochemistry, surface chemistry, etc. AFM on the other hand atomic force microscopy will be useful for thin film and coating and then piezoelectric and ferro materials, ferroelectric materials.

NSOM which is near field scanning optical microscopy. They are also very useful for nanotechnology research, nanophotonics, nano optics, material science and etc. So with that we come to a conclusion of this lecture as well as this course. So I hope you have enjoyed learning these concepts taught in this course. If you have got any query on anything you can drop an email to me at this particular email address mentioning MOOC on the subject line. Thank you.