Bapatla Engineering College :: Bapatla

IV/IV B. Tech (Regular) DEGREE EXAMINATION

Month & year : november, 2022

Semester: Seventh

Subject code: **18ECD31**

Subject Name : Introduction to Nano Science & Nanotechnology

SCHEME & SOLUTIONS

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18ECD31

Hall Ticket Number:

IV/IV B.Tech (Regular/Supplementary) DEGREE EXAMINATION

November,2022 **Electronics and Communication Engineerin**

Seventh Semester **Introduction to Nano science and Nanotechnolog**

Time: Three Hours **Maximum:** 50 Mark

Answer Question No. 1 Compulsorily. (10X1 = 10 Marks

*Answer **ANY ONE** question from each Unit.* (4X10=40 Marks

1.	a)	Define the term Nano.	CO1	L1	
	b)	Recall three dimensional quantum confinement.	CO1	L2	
	c)	What are nanorobots?	CO2	L2	
	d)	What is meant by atomic layer deposition?	CO2	L1	
	e)	Mention any two applications of Nano materials.	CO2	L2	
	f)	State Nanotechnology.	CO1	L1	
	g)	Define bragg's law	CO3	L3	
	h)	How does the Scaning electron microscope scan the sample?	CO3	L1	
	i)	Define Fullerence.	CO4	L1	
	j)	Mention two applications of graphene.	CO4	L2	

Unit - I

2.	a)	Discuss about various applications of Nano materials.	CO1	L2	5M
	b)	Obtain an expression for Density of states (DOS) in a Q1D, Q2D solid	CO1	L3	5M

(OR)

3.	a)	Discuss in detail about particle in a one-dimensional box	CO1	L1	5M
	b)	What is the physical significance of wave function?	CO1	L3	5M

Unit - II

4.	a)	Discuss about X-ray lithography technique.	CO2	L1	5M
	b)	Explain briefly how nano materials are prepared using 'Sol-gel techniques'	CO2	L3	5M

(OR)

5.	a)	Discuss about atomic layer deposition (ALD) technique used for the preparation of nano materials.	CO2	L3	5M
	b)	Discuss in detail about sol-gel method.	CO2	L3	5M

Unit - III

6.	a)	Discuss about the working of STM with the help of a neat diagram.	CO3	L2	**5M**
	b)	Write a short. Notes on U-V visible spectroscopy.	CO3	L4	**5M**

(OR)

7	a)	Discuss in detail about SEM technique for the characterization of nano materials.	CO3	L1	
	b)	Discuss about the X-ray diffraction method for characterization of Nano materials.	CO3	L1	**5M**

Unit - IV

8.	a)	Distinguish between single walled nano tube and multi walled nano tube.	CO4	L2	**5M**
	b)	Briefly discuss about various applications of CNTs	CO4	L3	**5M**

(OR)

9.	a)	Discuss about CNFED	CO4	L2	**5M**
	b)	Discuss about CNT based Bio-chemical sensor	CO4	L2	**5M**

ECE….SEVENTH SEMESTER NOV 2022

INTRODUCTION TO NANOSCIENCE AND NANOTECHNOLOGY

1(A) Define the term Nano.

Nano means **10^{-9}**.

(B) Recall three dimensional quantum confinement.

The energy levels are not continuous as a bul due to confinement of the electronic wave function to the physical dimentions of the particles, this is called quantum confinement

$$\psi(x,y,z) = \sqrt{\frac{8}{L_xL_yL_z}}\sin\left(\frac{n_x\pi x}{L_x}\right)\sin\left(\frac{n_y\pi y}{L_y}\right)\sin\left(\frac{n_z\pi z}{L_z}\right)$$

$$E_n = \frac{\pi^2\hbar^2}{2m}\left(\frac{n_x^2}{L_x^2}+\frac{n_y^2}{L_y^2}+\frac{n_z^2}{L_z^2}\right)$$

(C) What are Nano robots?

Nanoid robotics, or for short, nanorobotics or nanobotics, is an emerging technology field creating machines or robots whose components are at or near the scale of a nanometer.

(D) What is meant by atomic layer deposition?

- Atomic layer deposition (ALD) is a newly introduced technology.
- It is a modified CVD process by which we can go for pristine control over the growth mechanism.

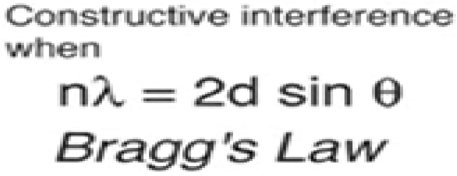
(E) Mention any two applications of Nano materials.

- Energy
- Biomedicine
- Environment
- Food
- Textile.

(F) State Nanotechnology.

Nanotechnology is design, fabrication and application of nanostructures or nanomaterials which are in the order of 1$\mathbf{10}^{-9}$.

(G) Define Bragg’s law.



Constructive interference when

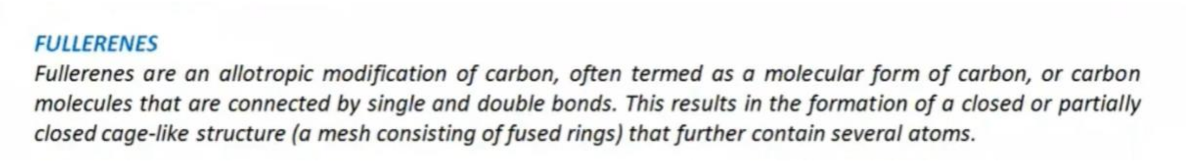
$$n\lambda = 2d \sin \theta$$

Bragg's Law

(H) How does the Scaning electron microscope scan the sample?

RASTER SCANNING

(I) Define Fullerence.



FULLERENES

Fullerenes are an allotropic modification of carbon, often termed as a molecular form of carbon, or carbon molecules that are connected by single and double bonds. This results in the formation of a closed or partially closed cage-like structure (a mesh consisting of fused rings) that further contain several atoms.

(J) Mention two applications of grapheme.

Water filtration

Solar cells

Air conditioners

Drug delivery

UNIT -1

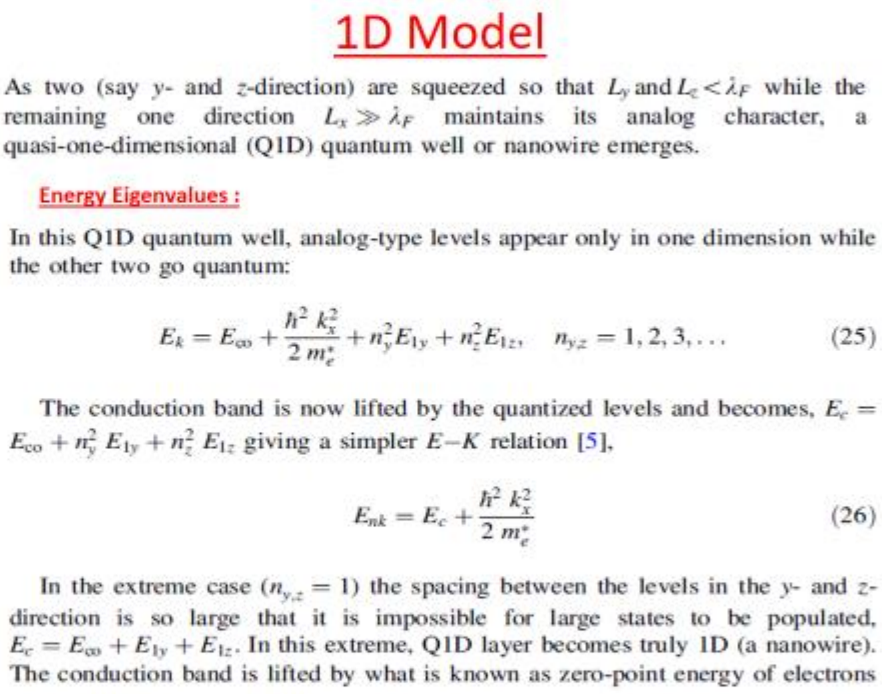
2(a) applications of Nanomaterials

- Nanomaterials can occur naturally, be created as the by-products of combustion reactions, or be produced purposefully through engineering to perform a specialized function.
- Due to the ability to generate the materials in a particular way to play a specific role, the use of nanomaterials spans across a wide variety of industries, from healthcare and cosmetics to environmental preservation and air purification.
- The healthcare field, for example, utilizes nanomaterials in a variety of ways, with one major use being drug delivery.
- One example of this process is whereby nanoparticles are being developed to assist the transportation of chemotherapy drugs directly to cancerous growths, as well as to deliver drugs to areas of arteries that are damaged in order to fight cardiovascular disease.
- Carbon nanotubes are also being developed in order to be used in processes such as the addition of antibodies to the nanotubes to create bacteria sensors.

- In aerospace, carbon nanotubes can be used in the morphing of aircraft wings.
- The nanotubes are used in a composite form to bend in response to the application of an electric voltage.
- Elsewhere, environmental preservation processes make use of nanomaterials too - in this case, nanowires. Applications are being developed to use the nanowires - zinc oxide nanowires - in flexible solar cells as well as to play a role in the treatment of polluted water.
- In the cosmetics industry, mineral nanoparticles – such as titanium oxide – are used in sunscreen, due to the poor stability that conventional chemical UV protection offers in the long-term.
- The sports industry has been producing baseball bats that have been made with carbon nanotubes, making the bats lighter and therefore improving their performance.
- Further use of nanomaterials in this industry can be identified in the use of antimicrobial nanotechnology in items such as the towels and mats used by sportspeople, in order to prevent illnesses caused by bacteria.
- Nanomaterials have also been developed for use in the military. One example is the use of mobile pigment nanoparticles being used to produce a better form of camouflage, through injection of the particles into the material of soldiers’ uniforms.
- Additionally, the military have developed sensor systems using nanomaterials, such as titanium dioxide, that can detect biological agents.
- The use of nano-titanium dioxide also extends to use in coatings to form self-cleaning surfaces, such as those of plastic garden chairs.

- A sealed film of water is created on the coating, and any dirt dissolves in the film, after which the next shower will remove the dirt and essentially clean the chairs.

2(b) DOS in Q1D,Q2D solid



1D Model

As two (say y- and z-direction) are squeezed so that L_y and $L_z < \lambda_F$ while the remaining one direction $L_x \gg \lambda_F$ maintains its analog character, a quasi-one-dimensional (Q1D) quantum well or nanowire emerges.

Energy Eigenvalues :

In this Q1D quantum well, analog-type levels appear only in one dimension while the other two go quantum:

$$E_k = E_{co} + \frac{\hbar^2 k_x^2}{2\, m_e^*} + n_y^2 E_{1y} + n_z^2 E_{1z}, \quad n_{yz} = 1, 2, 3, \ldots \tag{25}$$

The conduction band is now lifted by the quantized levels and becomes, $E_c = E_{co} + n_y^2\, E_{1y} + n_z^2\, E_{1z}$ giving a simpler $E{-}K$ relation [5],

$$E_{nk} = E_c + \frac{\hbar^2 k_x^2}{2\, m_e^*} \tag{26}$$

In the extreme case ($n_{y,z} = 1$) the spacing between the levels in the y- and z-direction is so large that it is impossible for large states to be populated, $E_c = E_{co} + E_{1y} + E_{1z}$. In this extreme, Q1D layer becomes truly 1D (a nanowire). The conduction band is lifted by what is known as zero-point energy of electrons

$E_{1y} + E_{1z}$. Similarly, the valence band $E_v = E_{vo} - E_{1hy} - E_{1hz}$ drops by zero-point energy of the holes $E_{1hy} + E_{1hz}$ [5].

- **Density of States (DOS):**

Following the same pattern as before (with spin 2 factor) and noting that $\Delta k_x = 2k_x$ (positive and negative direction), the DOS $D_{1e(h)}(E)$ for electrons and for holes is given by

$$D_{1e}(E) \equiv \frac{\Delta n_x}{L_x dE} = \frac{1}{\pi}\left(\frac{2m_e^*}{\hbar^2}\right)^{1/2}(E - E_c)^{-1/2}$$
$$D_{1h}(E) \equiv \frac{\Delta n_x}{L_x dE} = \frac{1}{\pi}\left(\frac{2m_h^*}{\hbar^2}\right)^{1/2}(E_h - E)^{-1/2} \tag{27}$$

The factor of 2 is due to spin and the other factor of 2 is for $\pm k_x$. Numerically, for a semiconductor, it can be put in the form [5]

$$D_1(E) = 4.07 \times 10^{18}\,\mathrm{J}^{-1/2}\,\mathrm{m}^{-1}\left(\frac{m^*}{m_o}\right)^{1/2}(E_J - E_c)^{-1/2}$$
$$= 1.63 \times 10^9\left(\frac{m^*}{m_o}\right)^{1/2}\mathrm{eV}^{-1/2}\,\mathrm{m}^{-1}(E_{\mathrm{eV}} - E_c)^{-1/2} \tag{28}$$

2D Model

In a quasi-two-dimensional (Q2D) quantum well, one direction (say z-direction), for example, is below the De Broglie wavelength ($L_z < \lambda_F$) while the other two $L_{x,y} \gg \lambda_F$ maintain their bulky character. In this Q2D quantum well, analog-type levels appear only in two dimensions while third dimension is quantized.

Energy Eigenvalues :

The total energy for an electron is the sum of energies of one quantized and two unquantized levels [5].

$$E_k = E_{co} + \frac{\hbar^2(k_x^2 + k_y^2)}{2\,m_e^*} + n_z^2 E_{1z} = E_c + \frac{\hbar^2(k_x^2 + k_y^2)}{2\,m_e^*} \tag{22}$$

with

$$E_c = E_{co} + n_z^2 E_{1z}, \quad n_z = 1, 2, 3, \ldots$$

In the extreme case ($n_z = 1$) the spacing between the levels in the z-direction is so large that it is impossible for large states to be populated, $E_c = E_{co} + E_{1z}$. Q2D layer becomes truly 2D in this extreme. The conduction band is lifted by zero-point energy E_{1z} (minimum energy in a quantum wave). Similarly, the valence band $E_v = E_{vo} - E_{1hz}$ drops by zero-point energy E_{1hz} of holes.

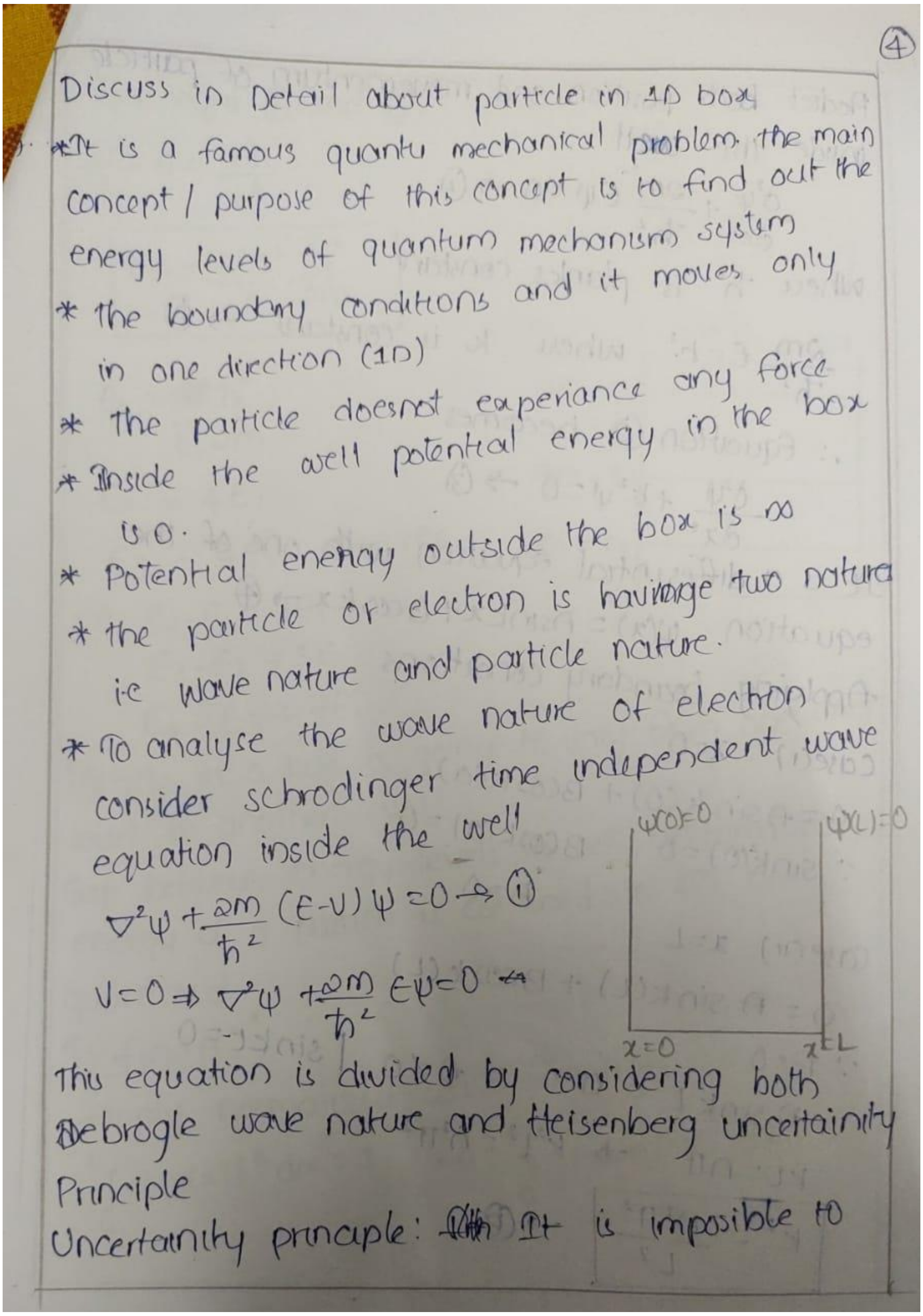
Density of States (DOS)

Following the same pattern as before (with spin 2 factor) and noting that $\Delta k_x \Delta k_y = kdkd\theta$ in circular coordinates with $\theta = 0 \rightarrow 2\pi$ (going full circle), the DOS $D_{2e(h)}$ for electrons e (or for holes h) is given by [5]

$$D_{2e(h)}(E) \equiv \frac{\Delta n_x \Delta n_y}{L_x L_y dE} = \frac{2\pi}{(2\pi)^2}\left(\frac{2m^*_{e(h)}}{\hbar^2}\right) = \frac{m^*_{e(h)}}{\pi\hbar^2} \tag{23}$$

DOS in this case are the number of quantum states per unit area per unit energy. These can be numerically shown to lead to

$$D_{2e(h)}(E) = \frac{m^*_{e(h)}}{m_o} \times 2.6 \times 10^{37}\,\mathrm{J}^{-1}\,\mathrm{m}^{-2} = \frac{m^*_{e(h)}}{m_o} \times 4.17 \times 10^{18}\,\mathrm{eV}^{-1}\,\mathrm{m}^{-2} \tag{24}$$



Discuss in Detail about particle in 1D box

* It is a famous quantu mechanical problem the main concept / purpose of this concept is to find out the energy levels of quantum mechanism system
* the boundary conditions and it moves only in one direction (1D)
* The particle doesnot experiance any force in the box
* Inside the well potential energy is 0.
* Potential energy outside the box is ∞
* the particle or electron is havinge two natura i.e wave nature and particle nature.
* To analyse the wave nature of electron consider schrodinger time independent wave equation inside the well

$\psi(0)=0$ $\psi(L)=0$

$x=0$ $x=L$

$$\nabla^2\psi + \frac{2m}{\hbar^2}(E-V)\psi = 0 \rightarrow ①$$

$$V=0 \Rightarrow \nabla^2\psi + \frac{2m}{\hbar^2}E\psi = 0 \leftarrow$$

This equation is divided by considering both Debrogle wave nature and Heisenberg uncertainity Principle

Uncertainity principle: It is imposible to

3(a) particle in one dimensional box

Predict both position and movementum of particle inside the well

$$\frac{\partial^2\psi}{\partial x^2} + \frac{2m}{\hbar^2}E\psi = 0 \rightarrow ②$$

where $\hbar^2$ is planks constant

$$\frac{2m}{\hbar^2}E = k^2 \quad \text{where } k \text{ is constant} \rightarrow Ⓐ$$

∴ Equation ② becomes

$$\frac{\partial^2\psi}{\partial x^2} + k^2\psi = 0 \rightarrow ③$$

It is a differential equation with one of the equation $\psi(x) = A\sin kx + B\cos kx \rightarrow ④$

Applying boundary conditions

case(i) $x=0$

$0 = A\sin k(0) + B\cos k(0)$

$\because \sin k(0) = 0 \quad B\cos k(0) = 0$

$B = 0$

case(ii) $x=L$

$0 = A\sin k(L) + B\cos k(L)$

$\because B=0 \qquad \because A \neq 0 \mid \sin kL = 0$

$A\sin kL = 0$

$kL = n\pi \Rightarrow k^2L^2 = n^2\pi^2$

$$\boxed{k^2 = \frac{n^2\pi^2}{L^2}} \rightarrow Ⓑ$$

from eqns Ⓐ and Ⓑ

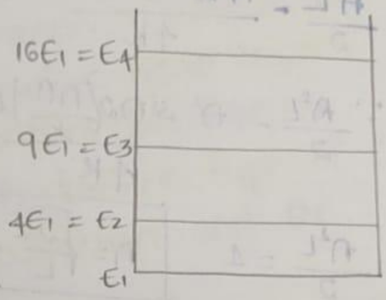
$$\frac{2m}{\hbar}E = \frac{n^2\pi^2}{L^2}$$

$$\therefore \boxed{E_n = \frac{n^2\pi^2}{L^2}\frac{\hbar^2}{2m}}$$

$$E_1 = \frac{\pi^2\hbar^2}{L^2 2m}$$

$$E_2 = 4E_1$$

$$E_3 = 9E_1 \qquad E_n = n^2E_1$$



$$\Rightarrow E_2 - E_1 = 3E_1$$

$$E_3 - E_2 = 5E_1$$

$$E_4 - E_3 = 7E_1$$

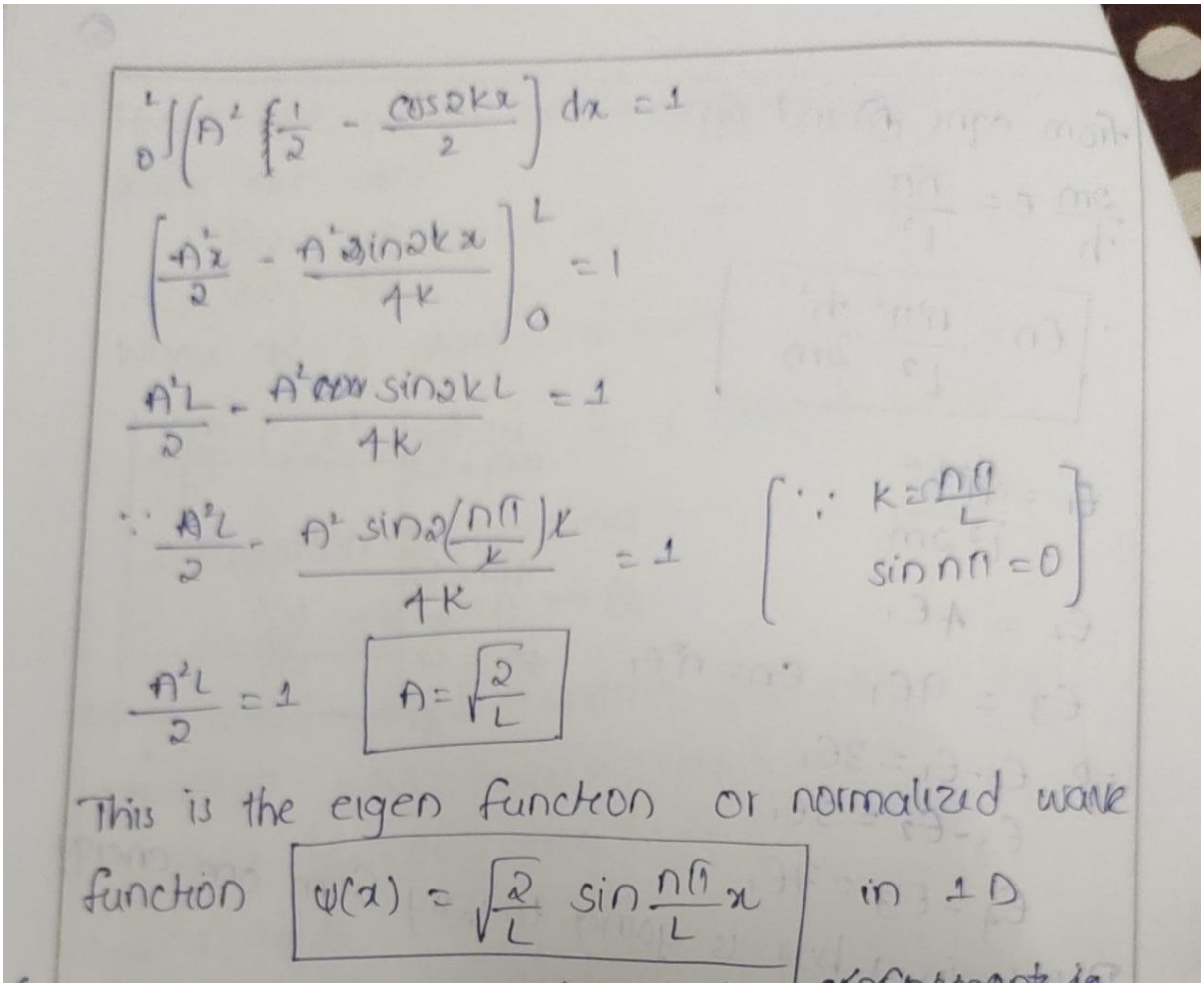
Particle in a box is going to vary from one energy level to another level

Gap between energy levels in not uniform so energy of a particle is confined in 1D box is quantized

To find A:

Apply normalized wave function

$$\int_0^L |\psi(x)|^2 dx = 1 \Rightarrow \int_0^L |A\sin kx|^2 dx = 1$$



$$\int_0^L \left(A^2 \left[\frac{1}{2} - \frac{\cos 2kx}{2}\right] dx = 1$$

$$\left[\frac{A^2 x}{2} - \frac{A^2 \sin 2kx}{4k}\right]_0^L = 1$$

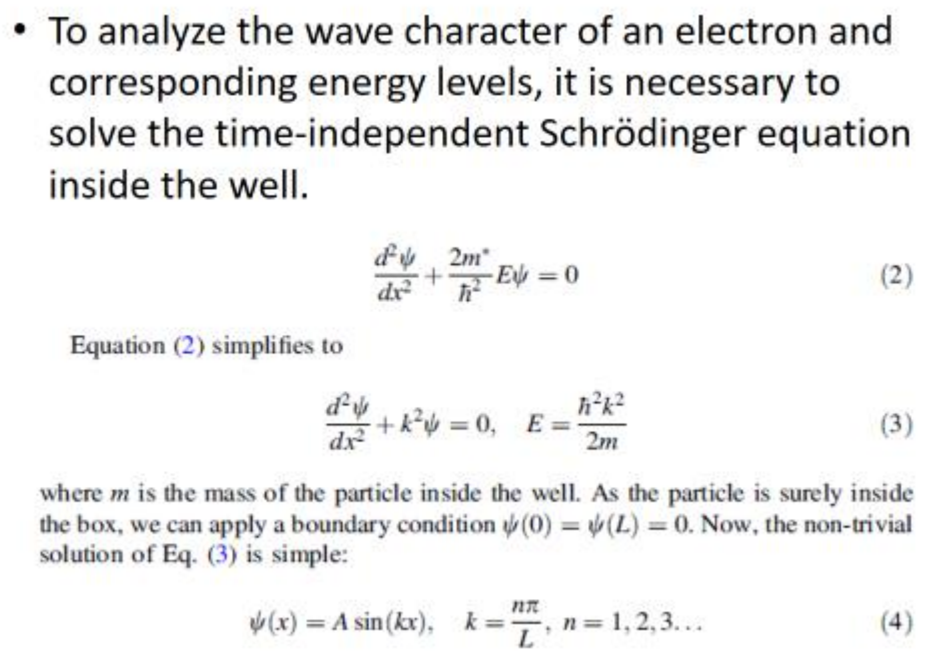
$$\frac{A^2 L}{2} - \frac{A^2 \cancel{\cos} \sin 2kL}{4k} = 1$$

$$\because \frac{A^2 L}{2} - \frac{A^2 \sin 2\left(\frac{n\pi}{L}\right)L}{4k} = 1 \qquad \left[\because k = \frac{n\pi}{L},\ \sin n\pi = 0\right]$$

$$\frac{A^2 L}{2} = 1 \qquad \boxed{A = \sqrt{\frac{2}{L}}}$$

This is the eigen function or normalized wave function $\boxed{\psi(x) = \sqrt{\frac{2}{L}} \sin \frac{n\pi}{L} x}$ in 1D

3(b) physical significance of wave function



- To analyze the wave character of an electron and corresponding energy levels, it is necessary to solve the time-independent Schrödinger equation inside the well.

$$\frac{d^2\psi}{dx^2} + \frac{2m^*}{\hbar^2} E\psi = 0 \tag{2}$$

Equation (2) simplifies to

$$\frac{d^2\psi}{dx^2} + k^2\psi = 0, \quad E = \frac{\hbar^2 k^2}{2m} \tag{3}$$

where m is the mass of the particle inside the well. As the particle is surely inside the box, we can apply a boundary condition $\psi(0) = \psi(L) = 0$. Now, the non-trivial solution of Eq. (3) is simple:

$$\psi(x) = A\sin(kx), \quad k = \frac{n\pi}{L},\ n = 1, 2, 3\ldots \tag{4}$$

The quantum condition in Eq. (4) when substituted in the energy $E = \hbar^2 k^2 / 2m$ expression of Eq. (3) gives the quantized energy levels. Applying the normalization condition, one can extract the value of constant A.

$$\int_0^L \psi * \psi dx = 1 \Rightarrow A = \sqrt{\frac{2}{L}} \tag{5}$$

Including the value of A, the normalized wavefunctions (also known as eigenfunctions) and quantized energy values (also known as eigenvalues) are then given by

$$\psi(x) = \sqrt{\frac{2}{L}} \sin\left(\frac{n\pi x}{L}\right), \quad E_n = n^2 \frac{\pi^2 \hbar^2}{2mL^2} = n^2 E_1 \tag{6}$$

Assuming an electron inside the box, Fig. 2 gives the quantized wavefunctions in (a) and the description of the energy levels in (b) for a quantum well of 10 nm length ($L_x = 10\,\text{nm}$). The energy spacing increases as energy moves higher. On the other hand, the levels tend to come closer together in a hydrogen atom. The spectrum indicated is thus inverted spectrum of that of the hydrogen level.

In case of a 3D box, electron wavefunctions are obtained by multiplying independent contributions coming from each dimension. For a 3D box, the complete time-independent solution and energy eigenvalues look like,

$$\psi(x, y, z) = \sqrt{\frac{8}{L_x L_y L_z}} \sin\left(\frac{n_x \pi x}{L_x}\right) \sin\left(\frac{n_y \pi y}{L_y}\right) \sin\left(\frac{n_z \pi z}{L_z}\right) \tag{7}$$

$$E_n = \frac{\pi^2 \hbar^2}{2m} \left(\frac{n_x^2}{L_x^2} + \frac{n_y^2}{L_y^2} + \frac{n_z^2}{L_z^2}\right) \tag{8}$$

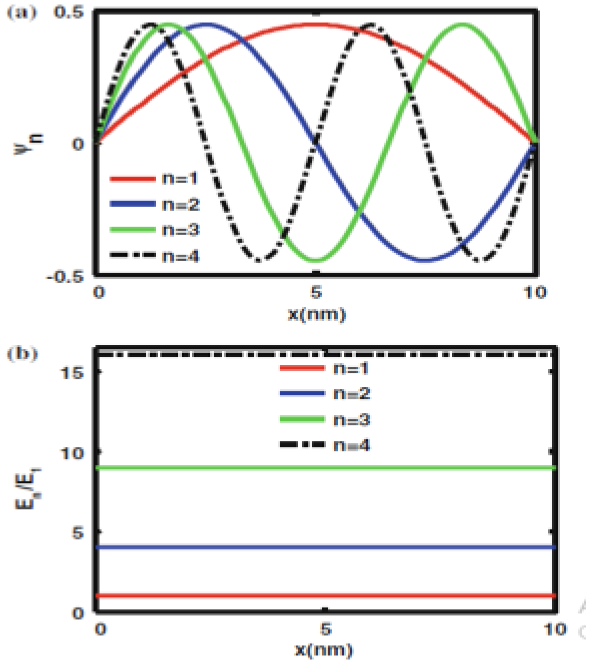
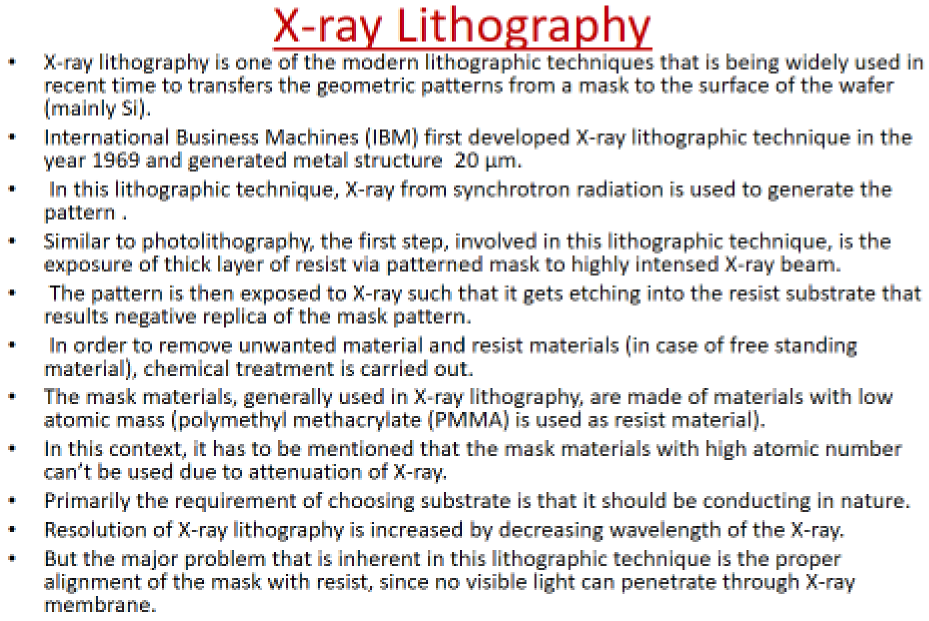


Fig. 2 **a** Electron wavefunctions in a quantum well of 10 nm length, **b** Normalized discrete energy levels inside a quantum well

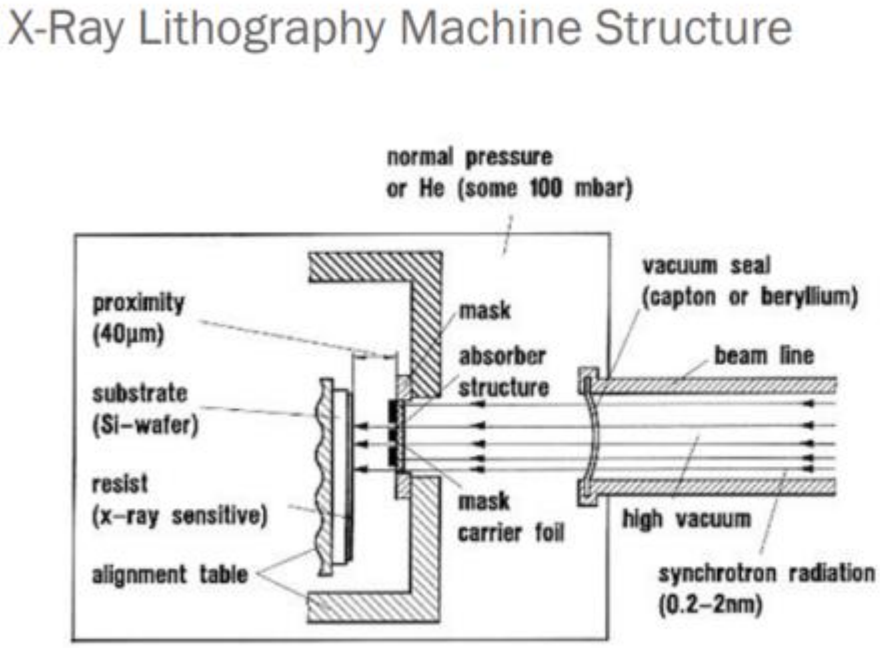
UNIT-2



X-ray Lithography

- X-ray lithography is one of the modern lithographic techniques that is being widely used in recent time to transfers the geometric patterns from a mask to the surface of the wafer (mainly Si).
- International Business Machines (IBM) first developed X-ray lithographic technique in the year 1969 and generated metal structure 20 µm.
- In this lithographic technique, X-ray from synchrotron radiation is used to generate the pattern .
- Similar to photolithography, the first step, involved in this lithographic technique, is the exposure of thick layer of resist via patterned mask to highly intensed X-ray beam.
- The pattern is then exposed to X-ray such that it gets etching into the resist substrate that results negative replica of the mask pattern.
- In order to remove unwanted material and resist materials (in case of free standing material), chemical treatment is carried out.
- The mask materials, generally used in X-ray lithography, are made of materials with low atomic mass (polymethyl methacrylate (PMMA) is used as resist material).
- In this context, it has to be mentioned that the mask materials with high atomic number can't be used due to attenuation of X-ray.
- Primarily the requirement of choosing substrate is that it should be conducting in nature.
- Resolution of X-ray lithography is increased by decreasing wavelength of the X-ray.
- But the major problem that is inherent in this lithographic technique is the proper alignment of the mask with resist, since no visible light can penetrate through X-ray membrane.

4(a) X-ray lithography technique.

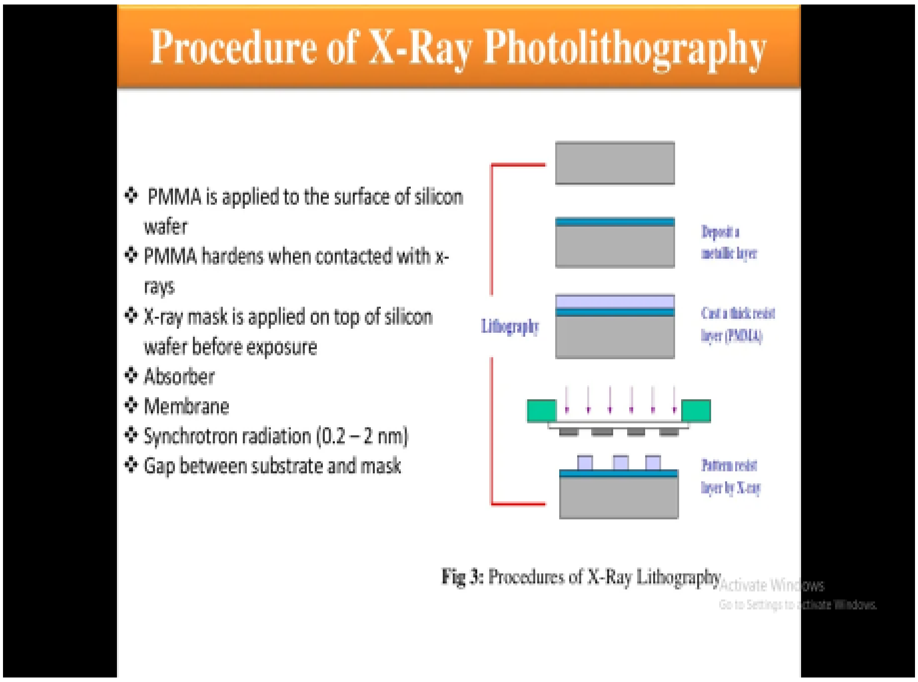


X-Ray Lithography Machine Structure

<u>Components</u>

- Absorber – reduce scattering of X-rays
- Membrane – allows X-rays to travel through

- Vacuum- to isolate the X-ray from interferences



Procedure of X-Ray Photolithography

- PMMA is applied to the surface of silicon wafer
- PMMA hardens when contacted with x-rays
- X-ray mask is applied on top of silicon wafer before exposure
- Absorber
- Membrane
- Synchrotron radiation (0.2 – 2 nm)
- Gap between substrate and mask

Fig 3: Procedures of X-Ray Lithography

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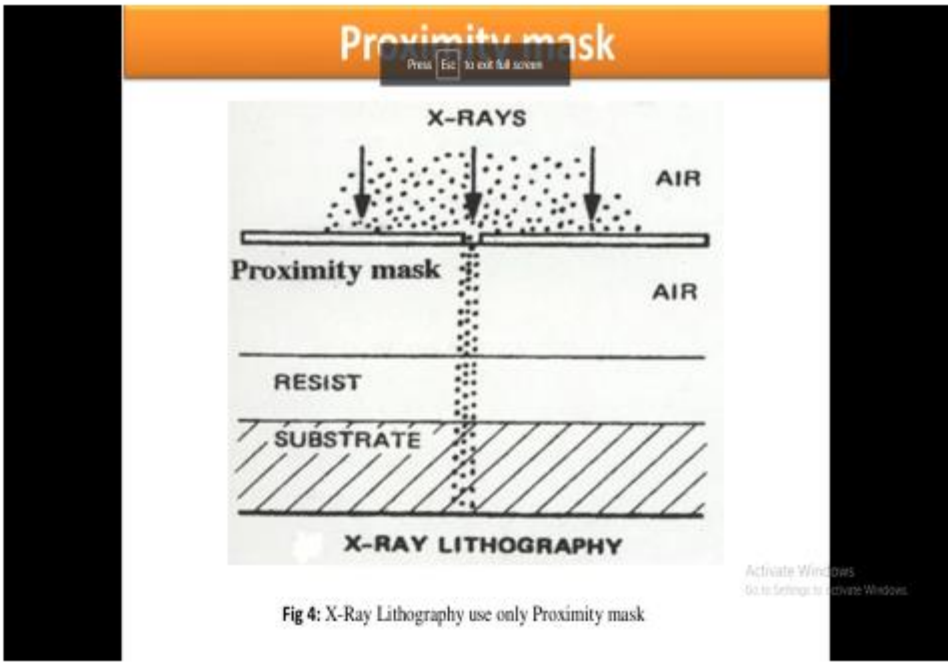

Proximity mask

Fig 4: X-Ray Lithography use only Proximity mask

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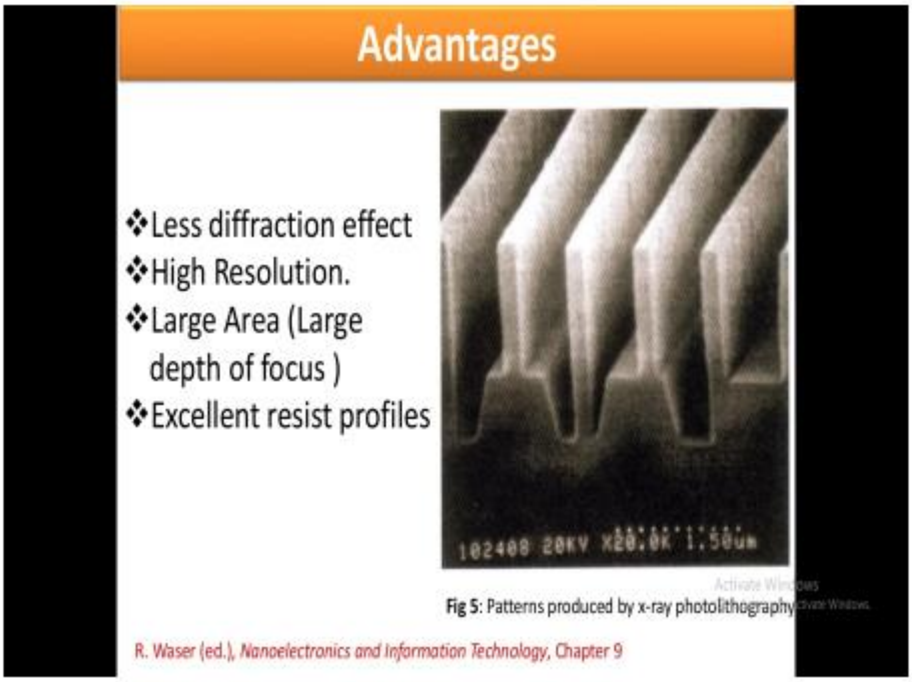

Advantages

❖Less diffraction effect
❖High Resolution.
❖Large Area (Large depth of focus)
❖Excellent resist profiles

Fig 5: Patterns produced by x-ray photolithography

R. Waser (ed.), *Nanoelectronics and Information Technology*, Chapter 9

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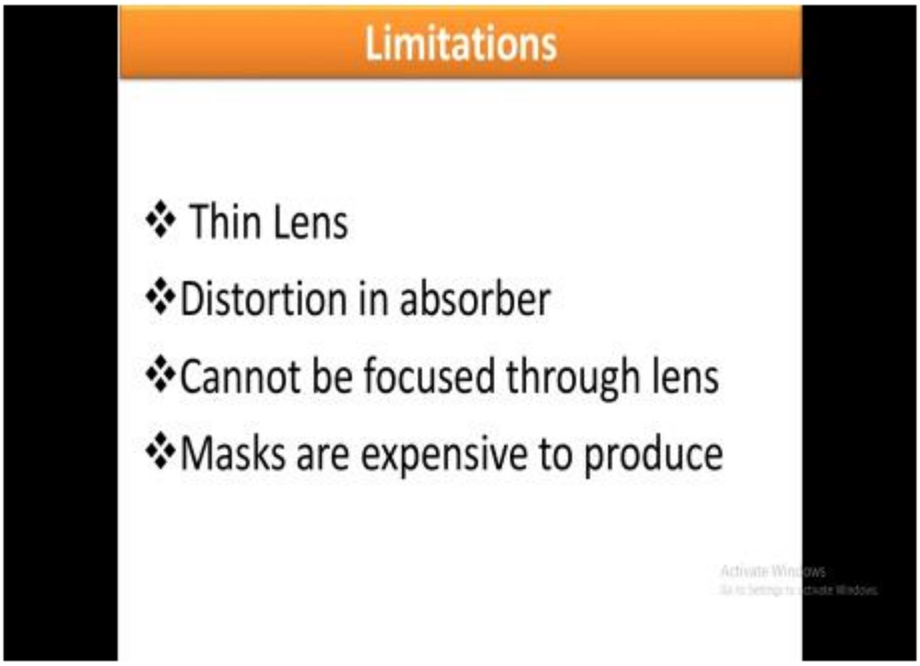

Limitations

❖ Thin Lens
❖Distortion in absorber
❖Cannot be focused through lens
❖Masks are expensive to produce

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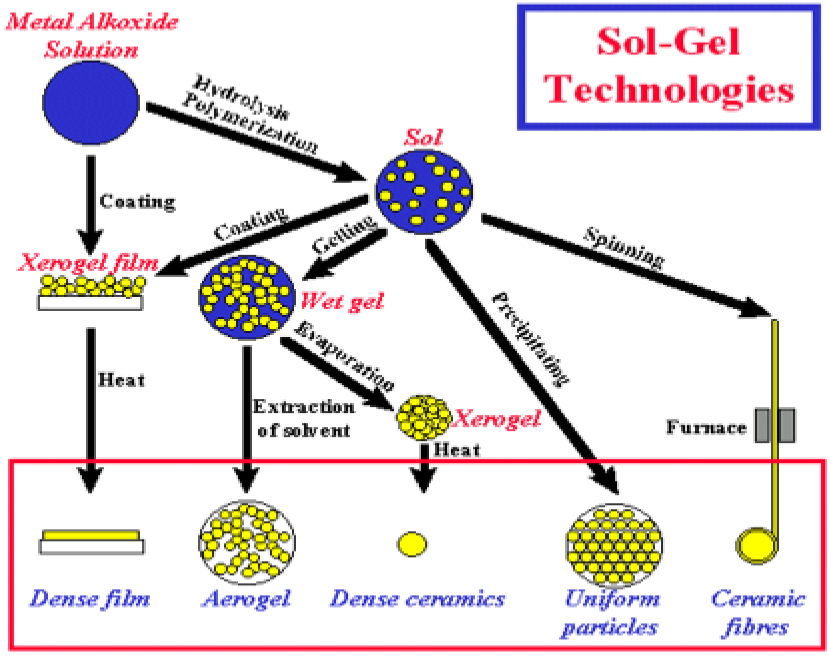
- X-Ray Lithography: Applications
- X-ray lithography is primarily used in nanolithography
- 15 nm optical resolution
- Utilizes short wavelength of 1 nm
- Simple: Requires no lenses
- Allows for small feature size

4(b)sol-gel techniques

Sol Gel

- Sols are dispersions of colloidal particles typically sized 1-100 nm in a liquid.
- Gels are interconnected, rigid networks with pores of sub-micrometer dimensions and polymeric chains whose average length is greater than a micron.
- The sol-gel process is a name given to the hydrolysis and poly-condensation of inorganic compounds to form ceramic materials



- non-hydrolytic sol–gel process exists in nature that does not require any solvent.
- Sol–gel processes sometimes are also categorized into alkoxides and non-alkoxide groups according to volatility of the precursor material.
- Metal alkoxide is one of the best precursor materials and those are being widely used, because most of the metals are capable of forming metal alkoxides with general chemical formula M(RO)x, M and R represent the metal and alkyl group, x is the valence state of the metal.
- In order to obtain homogeneous coating or monodispersed nanoparticle using sol–gel process, homogeneous gel without precipitation is required.
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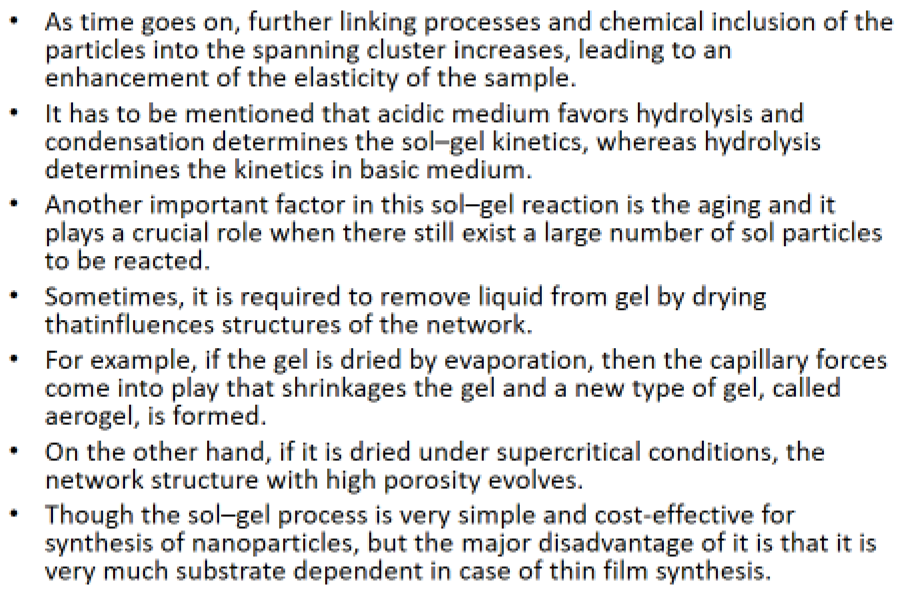
- Sometimes, additives are used to increase the time of gelation to increase the stability of sol–gel derived product.

- The basic sol–gel reaction involves dispersed colloidal particles having very small size such that gravitational force on them is negligible and exhibits random Brownian motion within the fluid matrix.
- The interaction among them is dominated by short-range forces like Van der Walls attraction, surface charges, and extends only for a few nanometers.
- In order to counter these attractive forces, some repulsive forces are always present there.
- Depending on the way of formation of network structure, gel is classified as colloidal and polymeric gels.
- Gel forming from colloidal particles upon aggregation and condensation are linked into a three-dimensional network surrounded by liquid phase are termed as colloidal gels.
- Theses gels are formed from sol particles and are also known as corpuscular gel.

- Polymeric gels are formed from sub-colloidal chemical units like branched macromolecules where individual particles cannot be distinguished.
- It is defined as polymeric gel because their structures are generated through repetition of one or a few elementary units.
- The solubility of the macromolecules into water or alcohol or acid medium restricts the transformation of their chains into individual particles.
- The gel corresponding to subunits of colloidal chemical directly forms from the precursor solution without the intermediate occurrence of individual particles.
- The interactions within gel, in most of cases, are covalent in nature and the gelation process is found to be reversible.
- This sol–gel process depends on the precursor material, solvent, and catalysts (if necessary).
- The precursor, consisting of metal or metalloid elements, surrounded by various ligands (if necessary), generally guides the formation of colloidal particle.
- Alkoxides are mostly used for this purpose.

- Hydrolysis and polycondensation are the two important steps, involved in the sol–gel process, those prefer water as solvent medium.
- To fasten the reaction kinetics catalysts that promote the hydrolysis of the precursors are used in some cases.
- The basic mechanism, involved in this sol–gel method to synthesize solid materials at liquid and low temperature, is polymerization process that establishes metal–OH–metal or metal–O–metal bridges between metallic atoms of the precursor materials.
- As a result, colloidal particles are formed.
- The next step involves the linking of these colloidal particles to build a three-dimensional network structure within solvent.
- The step of gelation arises when linking process enhances to an extent such that a giant spanning cluster is formed.
- At this point, the gel possesses high viscosity with negligible elasticity.



- As time goes on, further linking processes and chemical inclusion of the particles into the spanning cluster increases, leading to an enhancement of the elasticity of the sample.
- It has to be mentioned that acidic medium favors hydrolysis and condensation determines the sol–gel kinetics, whereas hydrolysis determines the kinetics in basic medium.
- Another important factor in this sol–gel reaction is the aging and it plays a crucial role when there still exist a large number of sol particles to be reacted.
- Sometimes, it is required to remove liquid from gel by drying thatinfluences structures of the network.
- For example, if the gel is dried by evaporation, then the capillary forces come into play that shrinkages the gel and a new type of gel, called aerogel, is formed.
- On the other hand, if it is dried under supercritical conditions, the network structure with high porosity evolves.
- Though the sol–gel process is very simple and cost-effective for synthesis of nanoparticles, but the major disadvantage of it is that it is very much substrate dependent in case of thin film synthesis.

5(a) ALD

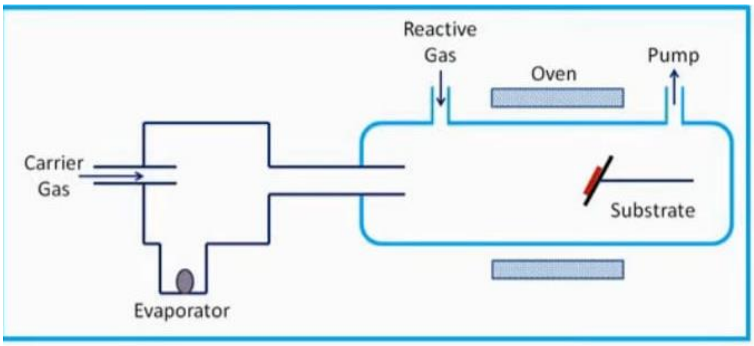
- As the name suggests, chemical vapor deposition (CVD) mainly involves chemical reactions that make it different from PVD processes .
- In CVD, the reaction occurs in gas phase giving a nonvolatile solid, which can be deposited on a suitable substrate to make it a thin film.
- The CVD includes wide varieties of reactions like pyrolysis, réduction, oxidation, disproportionation, réversible transfer, etc., depending on the activation of the CVD process, it is divided into several methods like:

- <u>Thermal activation</u>
  - In thermally activated CVD, high temperature (>900 °C) is required.
  - But in the case of organometallic components, this operates at relatively low temperature, called metal-organic CVD (MOCVD).
- <u>Plasma activation</u>

- Basically, it is low-temperature CVD, lies in the range of 300–500 °C.
- It is typically called as PECVD (plasma-enhanced CVD), where the plasma are excited by RF (100 kHz–40 MHz) or by microwave.

• Photon activation

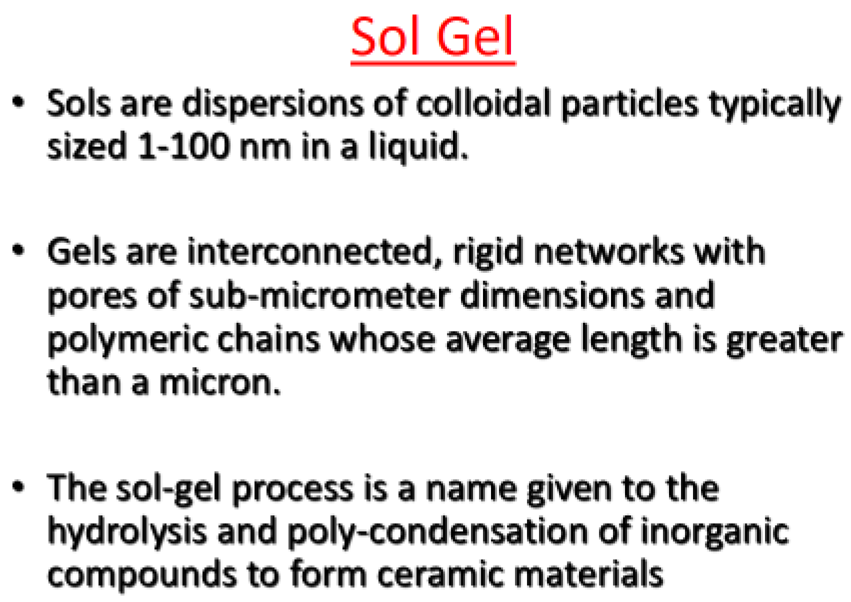
- Photon-activated CVD involves the direct activation of reactant in the presence of shortwave ultraviolet radiation.
- CVD is a very flexible technique that can be used to synthesize particular solid from different precursors and by varying the concentration of them.
- Also by varying the precursor composition and temperature from the same precursors we can have different films.
- Another interesting characteristic of CVD is that various morphologies can be generated using this method, CVD does not require ultrahigh vacuum chambers.
- Though CVD is a nonequilibrium process, we have to go for equilibrium analysis to understand its intricacy.
- Actually, the reaction is governed by first and second law of thermodynamics that predict the feasibility of the reactions, its kinetics and controls the energies associated with it.
- The geometry of the reactor along with the chamber pressure also plays dominant roles.
- For most CVD system the pressure remains at 0.01 atmospheres, but in a special case, called low-pressure CVD, pressure lies in the range of*0.5–1 Torr.
- There are many types of CVD reactors.
- We can broadly divide them into hot wall and cold wall.

- Hot wall reactors are tubular in shape and resistive heating has been used all around.
- in cold wall reactors, inductive heating has been given to the substrate by graphite susceptors but wall remains cold with the help of air/water flow.
- Atomic layer deposition (ALD) is a newly introduced technology.
- It is a modified CVD process by which we can go for pristine control over the growth mechanism.
- It has a self-limiting growth nature, which cannot be done in any other CVD processes.
- 

- That means we can create mono or few layer (2D/nearly 2D) films with the help of ALD.
- ALD mechanism is a combination of vapor phase self-assembly and surface reaction where the surface is activated by chemical reaction .
- The precursor molecules are introduced in the chamber where they react with the activated surface and form chemical bond with the substrate.
- The precursor molecules do not react with each other so that a monolayer forms.
- If we further activate the newly formed monolayer, it will act as base and we may have more than one layer.

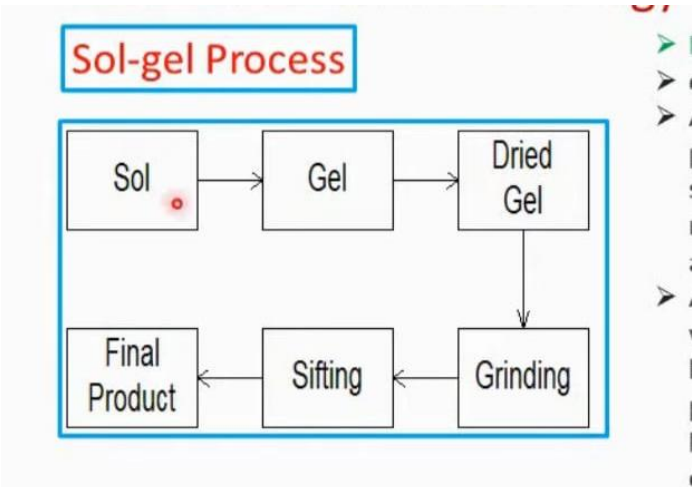
- Depending on the precursor, complex films can be easily generated by this technique.

5(b) sol gel method



Sol Gel

- Sols are dispersions of colloidal particles typically sized 1-100 nm in a liquid.
- Gels are interconnected, rigid networks with pores of sub-micrometer dimensions and polymeric chains whose average length is greater than a micron.
- The sol-gel process is a name given to the hydrolysis and poly-condensation of inorganic compounds to form ceramic materials



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- non-hydrolytic sol–gel process exists in nature that does not require any solvent.
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- Metal alkoxide is one of the best precursor materials and those are being widely used, because most of the metals are capable of forming metal alkoxides with general chemical formula M(RO)x, M and R represent the metal and alkyl group, x is the valence state of the metal.
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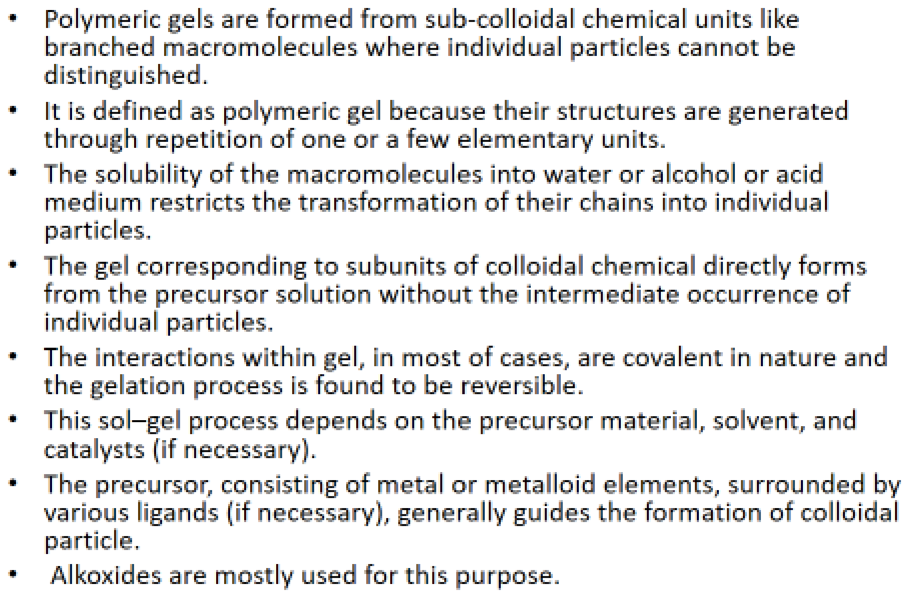

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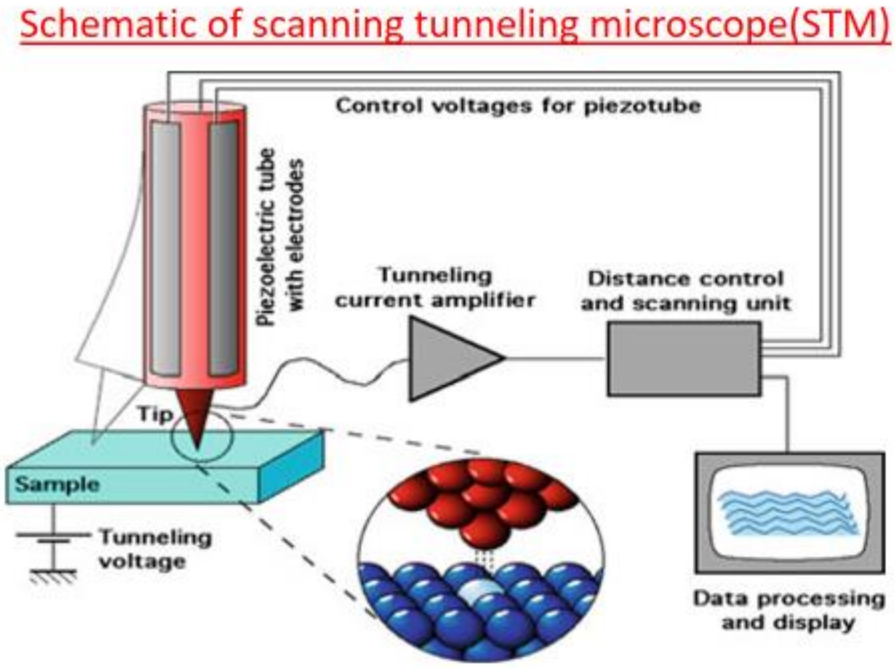
UNIT-3

6(a) STM



Schematic of scanning tunneling microscope(STM)

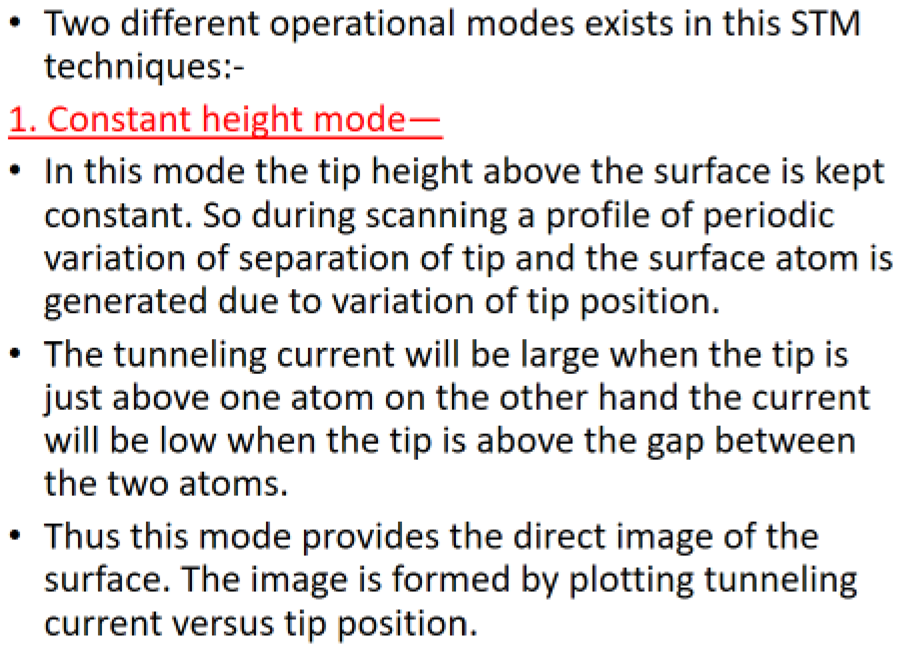
4.Scanning Tunneling Microscopy(STM)

- It was invented by Binning and Rohrer in the year 1981 has been proven to be a sophisticated and advanced characterization tools to investigate electronic structure of solid state nanostructure materials .
- In recent time, it becomes also possible to image surface and local electronic structure associated with it.
- Basic principle inherent in this technique is the mapping of the surface by tunneling current between surface and STM.
- STM consists of sharp probing tip, one piezoelectric scanning system, positioning system and electronic control unit.
- Piezoelectric system is used to control both vertical and lateral movement of the scanning tip.
- The process is very much sensitive to vibration; therefore the entire measurement must be vibration free.
- The purpose of the coarse positioning system is to control the tunneling current.

- Electronic control system is generally used to measure the tunneling current and to control scanner with the help of a feedback circuit.
- The feedback circuit takes the output signal from the current pre-amp, and then it compares with the signal level in present value.
- The final step involves the response feedback voltage depending on the user-defined feedback parameters.
- Feedback voltage is sent through an analog to digital converter to the high voltage amplifier that magnifies the input signal to control the piezo scanner.
- Current flows between sample and tip electrode when the gap between them is of the order of several A^0 and a bias voltage is applied.

- The tunneling probability of the electrons depends on the separation between tip and sample surface and the corresponding tunneling current (I) α e^{-2kz} , where z is the thickness of the insulator and $k = \sqrt{2}\, m(V\text{-}E)/ h$
- where m = effective mass of electron,
- V = Electrostatic potential of the potential barrier (insulator),
- E = the energy of electron,
- h = the Planck's constant.

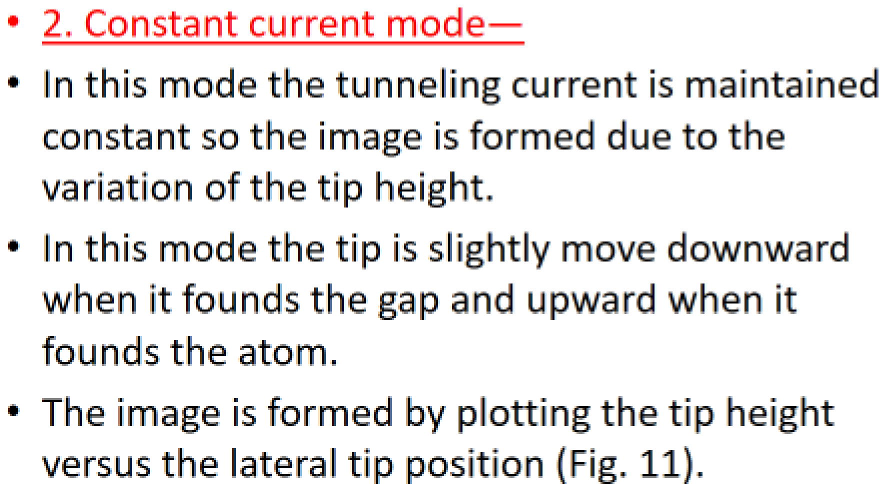
- If there is 0.1 nm variation in the gap results a change in current by an order of magnitude, hence STM possesses high tip sensitivity.
- The atomic resolution of the image along X–Y direction is 0.01 nm and 0.002 nm along Z direction.
- From differential conductance (dI/dV), the average DOS of the sample can be calculated.
- The tip is made up of metal or metal alloys like tungsten or platinum– iridium and it is mounted on a three dimensional array of piezoelectric material and it should be very sharp (Fig. 10).



- Two different operational modes exists in this STM techniques:-

1. Constant height mode—

- In this mode the tip height above the surface is kept constant. So during scanning a profile of periodic variation of separation of tip and the surface atom is generated due to variation of tip position.
- The tunneling current will be large when the tip is just above one atom on the other hand the current will be low when the tip is above the gap between the two atoms.
- Thus this mode provides the direct image of the surface. The image is formed by plotting tunneling current versus tip position.



- 2. Constant current mode—
- In this mode the tunneling current is maintained constant so the image is formed due to the variation of the tip height.
- In this mode the tip is slightly move downward when it founds the gap and upward when it founds the atom.
- The image is formed by plotting the tip height versus the lateral tip position (Fig. 11).

6(b) UV visible spectroscopy

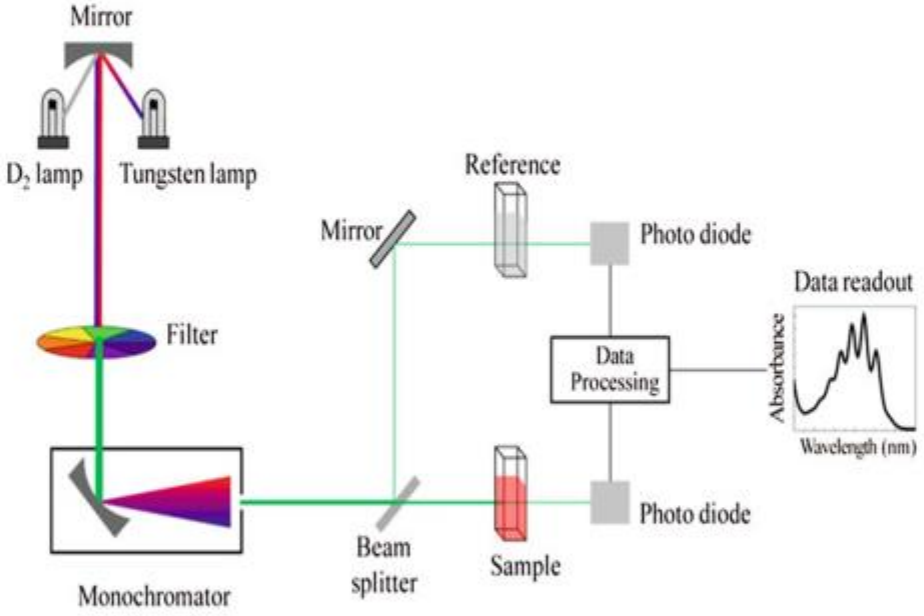
UV-visible spectrophotometer

- Ultraviolet-visible (UV-Vis) spectroscopy is a widely used technique in many areas of science ranging from bacterial culturing, drug identification and nucleic acid purity checks and quantitation, to quality control in the beverage industry and chemical research& analysis of nanoparticles.
- UV/Visible spectroscopy is a technique used to quantify the light that is absorbed and scattered by a sample.
- A sample is placed between a light source and a photo detector, and the intensity of a beam of UV/visible light is measured before and after passing through the sample.
- These measurements are compared at each wavelength to quantify the sample's wavelength dependent extinction spectrum. (extinction is defined as the sum of absorbed and scattered light).
- The data is typically plotted as extinction as a function of wavelength.
- Each spectrum is background corrected using a buffer blank to guarantee that spectral features from the buffer are not included in the sample extinction spectrum.

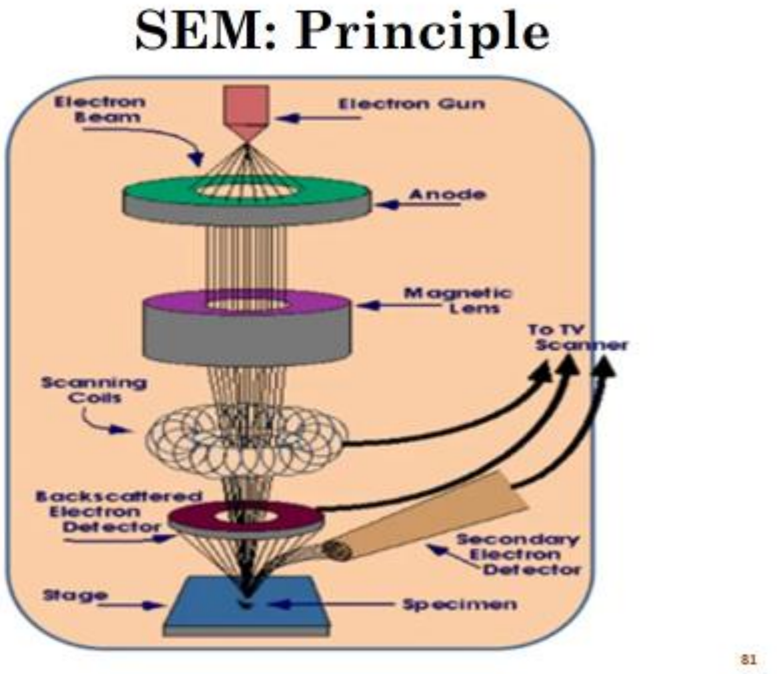
- UV-Vis spectroscopy is an analytical technique that measures the amount of discrete wavelengths of UV or visible light that are absorbed by or transmitted through a sample in comparison to a reference or blank sample.
- This property is influenced by the sample(nano material) composition, potentially providing information on what is in the sample and at what concentration.
- Light has a certain amount of energy which is inversely proportional to its wavelength.
- Shorter wavelengths of light carry more energy and longer wavelengths carry less energy.
- A specific amount of energy is needed to promote electrons in a substance to a higher energy state which we can detect as absorption.
- Electrons in different bonding environments in a substance require a different specific amount of energy to promote the electrons to a higher energy state.
- This is why the absorption of light occurs for different wavelengths in different substances.
- UV light has wavelengths shorter than that of visible light to approximately 100 nm. Therefore, light can be described by its wavelength, which can be useful in UV-Vis spectroscopy to analyze or identify different substances by locating the specific wavelengths corresponding to maximum absorbance

- Gold and silver plasmonic nanoparticles have optical properties that are sensitive to size, shape, concentration, agglomeration state, and refractive index near the nanoparticle surface.
- This makes UV/Visible spectroscopy a valuable tool for identifying, characterizing, and studying nanomaterials.
- The measured spectrum can be compared to the predicted spectrum based on numerical models.
- Non-plasmonic nanoparticles also have size and concentration dependent optical properties, however, their spectrum is not as sensitive to the dispersion properties as plasmonic nanoparticles.
- At nano Composix, UV-Visible analysis is performed with an Agilent 8453 single beam diode array spectrometer which collects spectra from 200-1100 nm using a slit width of 1 nm.
- Deuterium and tungsten lamps are used to provide illumination across the ultraviolet, visible, and near-infrared electromagnetic spectrum.
- Spectra can be collected from samples as small as 60-100 µL using a microcell with a path length of 1 cm.

2.Schematic of UV-visible spectrophotometer



7(a) SEM



SEM: Principle

Characterization by Scanning Electron Microscope

- Scanning electron microscope (SEM)/field emission scanning electron microscope (FESEM) is a microscope to visualize very small topographic details of the sample surface.
- Researchers in nanoscience, material science, biology, chemistry and physics employs this technique to observe structures that may be as small as 1 nm (=billion of a millimeter).
- Electrons, liberated from a field emission source, are allowed to pass through high vacuum column in the presence of a voltage gradient (typically ~30 kV).
- These beams of electrons are focused by electronic lenses, called electromagnetic lenses (combination of electric field and magnetic field), to produce a narrow scanning beam of electron having diameter 1–10 nm (called electron probe).
- In conventional SEM/FESEM, the electron probe is used to scan the specimen horizontally in two perpendicular directions. This is generally carried out by two sawtooth wave generators.

- In conventional SEM, the electron probe is used to scan the specimen horizontally in two perpendicular directions.
- This is generally carried out by two sawtooth wave generators.
- Scanning rate along one direction is much greater than that of other.
- During scanning along one direction (say x direction), the electron probe moves in a straight line (known as line scan), after reaching the end point it is again deflected back along x-axis as quickly as possible.
- In this subsequent time, probe moves slightly along other direction (say y direction).
- Again the probe moves along x-direction.
- The entire procedure is known as raster scanning that includes the beam to cover a rectangular area on the specimen.
- When these high energetic electrons enter (called primary electron) into the solid, they suffer from both elastic and inelastic scattering.

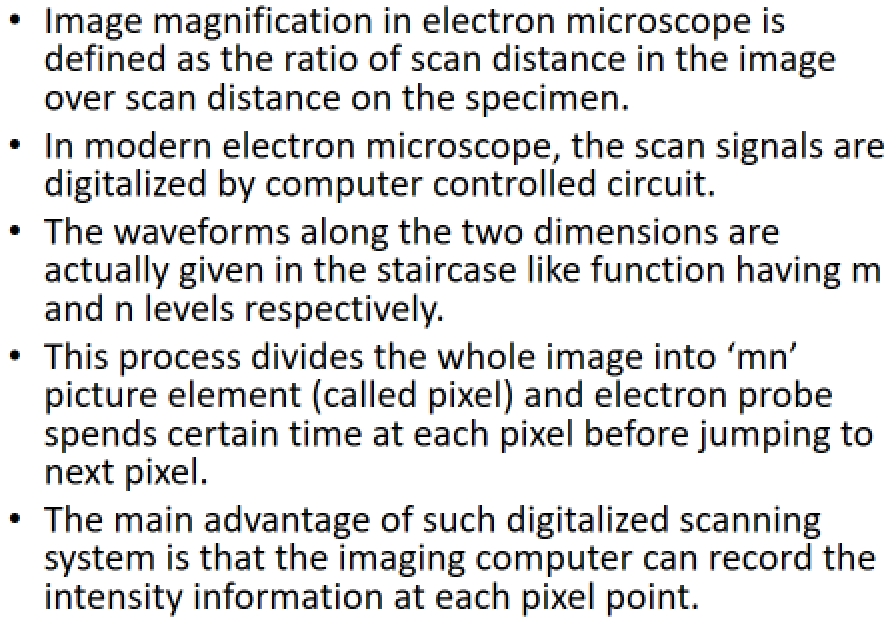
- The energy lost by primary electron during inelastic scattering is gained by atomic electrons.
- If this energy is sufficient for them, they escape from atom and travel freely through the solid specimen (called secondary electron).
- Secondary electrons escape from the sample into vacuum if they are being generated very close to the sample surface within some depth, known as escape depth.
- Since escape depth is the characteristic property of the material, the secondary electrons carries the signature of the surface structure (topography) of the specimen, rather than underlying structure.
- In electron microscope, signal from secondary electrons are collected by the photomultiplier tube for imaging purpose.

- Output of the two probe generators and image are synchronized to a display device i.e., there exists one-to-one correspondence between probe generators and signal of secondary electrons.
- The contrast in the images arises from the variation of number of secondary electron, collected at photomultiplier tube.
- In this context, it has to be mentioned that the following conditions as given by James Clerk Maxwell must be satisfied between object and image:

(a) for each object there should be one image point.

(b) geometrically the object and image must be similar.

(c) for any planer object perpendicular to the optic axis, corresponding image must be planer and perpendicular to the optic axis.

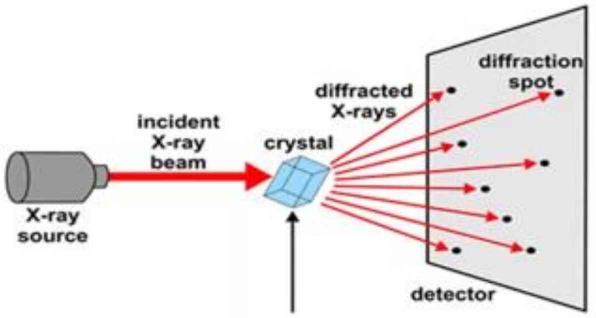


- Image magnification in electron microscope is defined as the ratio of scan distance in the image over scan distance on the specimen.
- In modern electron microscope, the scan signals are digitalized by computer controlled circuit.
- The waveforms along the two dimensions are actually given in the staircase like function having m and n levels respectively.
- This process divides the whole image into ‘mn’ picture element (called pixel) and electron probe spends certain time at each pixel before jumping to next pixel.
- The main advantage of such digitalized scanning system is that the imaging computer can record the intensity information at each pixel point.

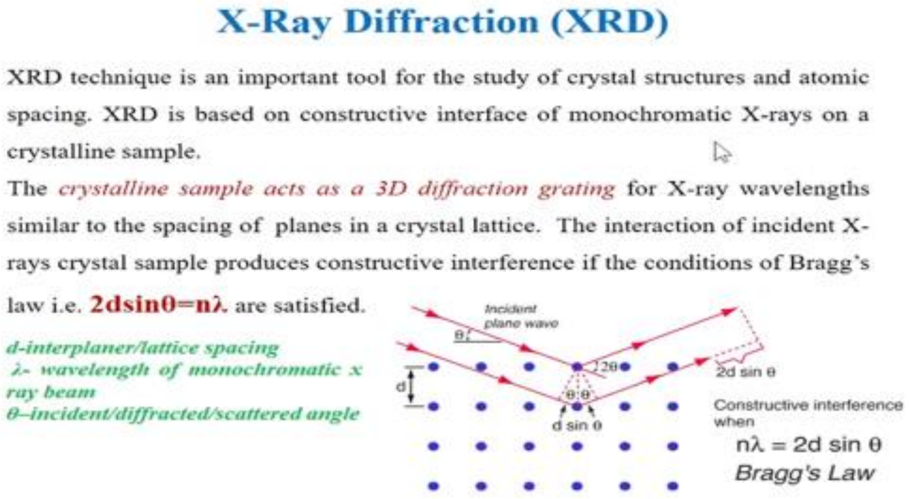
7(b) X ray diffraction

Characterization by X-ray Diffraction

- We know from basic knowledge of optics that diffraction occurs when electromagnetic radiations are incident on any periodic arrangement of obstacles having its periodicity comparable to the wavelength of the incident radiation.
- Crystalline materials are characterized by periodic arrangements of atoms.
- We may consider those periodic arrangements as plane through them. Since wavelength of X-ray (characteristic X-ray is used) is comparable to that of interplanar spacing of crystalline material, so diffraction effects can be observed. This diffraction phenomenon is generally used to determine fine structure of materials.
- This phenomenon is also used for chemical analysis, stress determination, particle size calculation etc.



Bijelic, A. & Rompel, A. ChemTexts (2018) 4: 10. https://doi.org/10.1007/s40828-018-0064-1



X-Ray Diffraction (XRD)

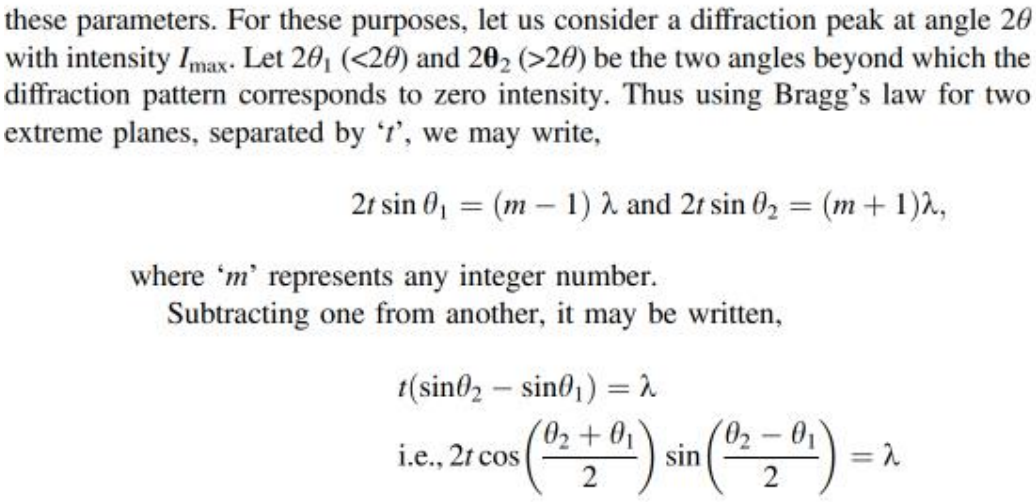
XRD technique is an important tool for the study of crystal structures and atomic spacing. XRD is based on constructive interface of monochromatic X-rays on a crystalline sample.

The *crystalline sample acts as a 3D diffraction grating* for X-ray wavelengths similar to the spacing of planes in a crystal lattice. The interaction of incident X-rays crystal sample produces constructive interference if the conditions of Bragg's law i.e. **$2d\sin\theta=n\lambda$** are satisfied.

d-interplaner/lattice spacing
λ- wavelength of monochromatic x ray beam
θ–incident/diffracted/scattered angle

- X-rays, reflected from two successive planes, possesses a path difference of 2d sin θ, where d and θ represent interplanar spacing and angle of the radiation with sample surface.
- If this path difference is equal to an integral multiple of λ (wavelength of the incident X-ray), then they will interfere constructively resulting maximum intensity, otherwise their interference is destructive giving zero intensity.
- The condition of interference maxima i.e., $2d \sin \theta = n\lambda$ ('n' is any integer) is known as Bragg's law.
- The basic principle, involved in this experimental technique, is the measurement of intensity as a function of θ. Experimentally, intensity is measured as function of 2θ i.e., angle between incident and reflected X-rays

- Presence of different peaks is attributed to the satisfaction of Bragg's law for different planes.
- Intensity of the peaks depends on the number of electrons within atom and on the type and arrangement of atoms within unit cell of the crystalline materials.
- Actually the plane that will correspond diffraction maxima is determined by atomic form factor and structure factor.
- One main objective of this technique is phase identification. It is well known that each crystalline material possesses characteristic interplanar spacings.
- Phase identification is carried out by comparing the experimentally measured interplanar spacing with their standard value. Instead of δ-like pattern of diffraction peak in ideal case, its experimental pattern possesses finite width.
- There are several factors such as finite size of particle, strain etc. responsible for such finite width. Thus we may use this finite width to determine these parameters.



these parameters. For these purposes, let us consider a diffraction peak at angle 2θ with intensity $I_{\max}$. Let $2\theta_1$ ($<2\theta$) and $2\boldsymbol{\theta}_2$ ($>2\theta$) be the two angles beyond which the diffraction pattern corresponds to zero intensity. Thus using Bragg's law for two extreme planes, separated by 't', we may write,

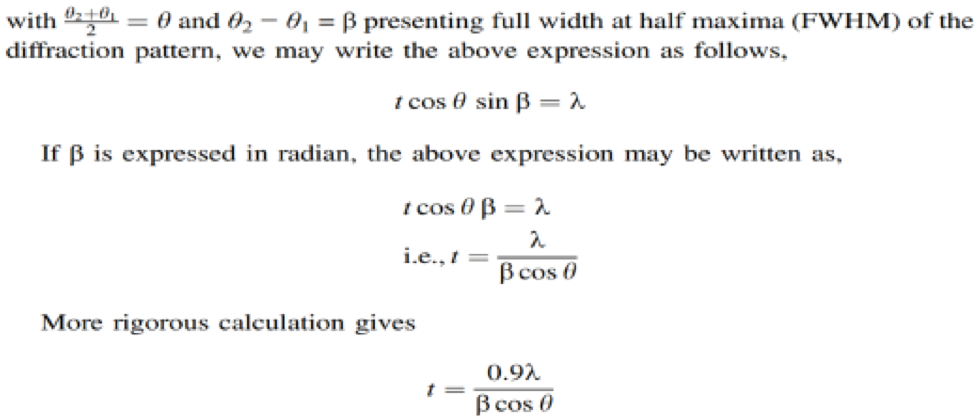
$$2t\sin\theta_1 = (m-1)\,\lambda \text{ and } 2t\sin\theta_2 = (m+1)\lambda,$$

where 'm' represents any integer number.

Subtracting one from another, it may be written,

$$t(\sin\theta_2 - \sin\theta_1) = \lambda$$

$$\text{i.e., } 2t\cos\left(\frac{\theta_2+\theta_1}{2}\right)\sin\left(\frac{\theta_2-\theta_1}{2}\right) = \lambda$$



with $\frac{\theta_2+\theta_1}{2} = \theta$ and $\theta_2 - \theta_1 = \beta$ presenting full width at half maxima (FWHM) of the diffraction pattern, we may write the above expression as follows,

$$t\cos\theta\,\sin\beta = \lambda$$

If β is expressed in radian, the above expression may be written as,

$$t\cos\theta\,\beta = \lambda$$

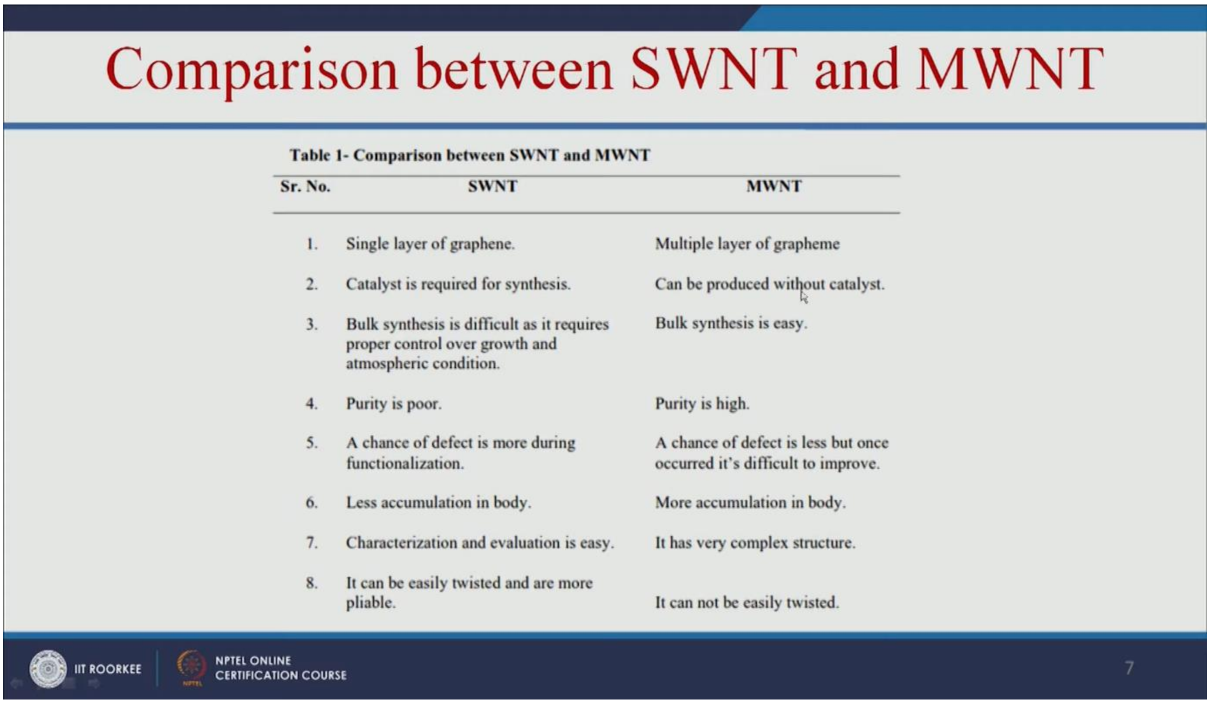
$$\text{i.e., } t = \frac{\lambda}{\beta\cos\theta}$$

More rigorous calculation gives

$$t = \frac{0.9\lambda}{\beta\cos\theta}$$

UNIT -4

8(a) SWNT vs MWNT



Comparison between SWNT and MWNT

Table 1- Comparison between SWNT and MWNT

Sr. No.	SWNT	MWNT
1.	Single layer of graphene.	Multiple layer of grapheme
2.	Catalyst is required for synthesis.	Can be produced without catalyst.
3.	Bulk synthesis is difficult as it requires proper control over growth and atmospheric condition.	Bulk synthesis is easy.
4.	Purity is poor.	Purity is high.
5.	A chance of defect is more during functionalization.	A chance of defect is less but once occurred it's difficult to improve.
6.	Less accumulation in body.	More accumulation in body.
7.	Characterization and evaluation is easy.	It has very complex structure.
8.	It can be easily twisted and are more pliable.	It can not be easily twisted.

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8(b) Applications of CNT

Medical

Thermal materials

Air filtration

Water filtration

Hydrogen storage

Energy storage

Drug delivery

Paint that cleans air

9(a) CNTFED

CNTFED

Introduction to FED:

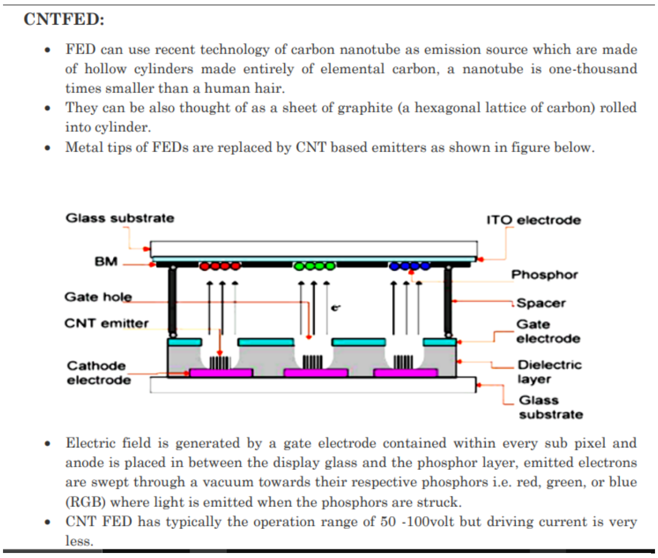
- Field emission display (FED) is a type of Flat Panel Display which is thinner, brighter, energy-efficient and cheaper display than liquid crystal display (LCD).
- FED also named as Nano Emissive Display (NED) consist of millions of accelerated electrons charged by very less voltage, compared with high voltage for large-screen, HD LCDs.
- These electron emitters are controlled by cold cathodes to generate colored light and are emitted towards phosphor plate and this phenomenon creates moving images.
- FED has a wide viewing angle and provides brightest color among plasma and other latest displays.
- At the place of CRTS carbon nanotubes (CNT) can be also used to aim electron beams on phosphor coated screen which will further enhance resolution.
- FED is very thin, less than an inch so we can hang them very easily on walls like a portrait and capable to offer 20,000-to-one contrast and can flash 240 images per second twice as many as the fastest HDTVs in the market.
- FED can be used for television, desktop, laptop etc. and has applications in medical imaging, defense and many other fields.

Field emission process:

- Emission takes place from one medium to another and this medium can be solid, liquid, air, vacuum or any dielectric.
- Field emission is, also called "cold emission" directional quantum-mechanical tunnelling which occurs due to electrostatic field.
- Thermal emission, which is the basis of CRTs requires high operating temperature, consumes much power and not energy efficient as loses due to kinetic energy disturbance.
- On the contrary, field emission extracts electrons through a large electric field without high temperature requirement.
- This resulting electron source has reduced emittance and no heat issue that's why field emitters are a "cold cathode" electron source.
- Field emission based electron sources has many application as it gives smaller spot but there most undesirable and inappreciable aspect for engineers is that they introduce electric discharge and vacuum breakdown.
- Field emission guns give higher current densities than thermionic guns.

- The idea behind FED is to offer each pixel a separate electron emitter compared to only a single one in the CRT leading to an ultra-high definition picture with unmatched clarity and no blurring.
- Carbon nanotubes can be also used as electron emitter source and the sharp point on the emitter allows for electrons to be pulled off of the nanotubes at surprisingly low voltages.
- FED employs arrays of small sharp silicon or molybdenum cones which are deposited on a substrate within an etched hole.

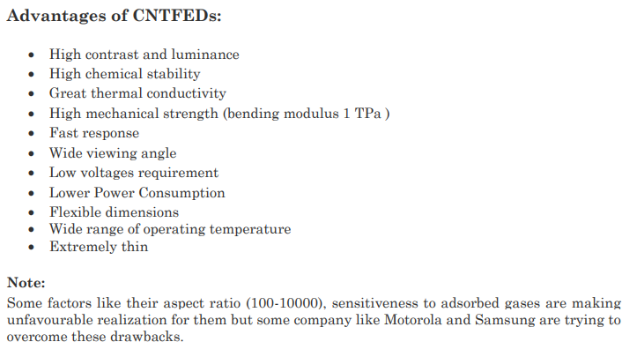
- The result is a triode structure of between a few and less than one micron in diameter, of which there are thousands per individual pixel.
- Traditional FED structure made use of many micro tips, which were blue, red or green and together form one pixel.
- As FEDs display colour sequentially, the advantages of the FED include the fact that they only produce light when the pixels are 'on', and as a result power consumption is dependent on the display content.
- Electrons leave the sharp tips with relatively low extraction voltages at the gate.
- Field emission from a flat cathode plane reduces the need for fine lithographic features and relaxes the tolerances demanded of the triode structure.



CNTFED:

- FED can use recent technology of carbon nanotube as emission source which are made of hollow cylinders made entirely of elemental carbon, a nanotube is one-thousand times smaller than a human hair.
- They can be also thought of as a sheet of graphite (a hexagonal lattice of carbon) rolled into cylinder.
- Metal tips of FEDs are replaced by CNT based emitters as shown in figure below.

- Electric field is generated by a gate electrode contained within every sub pixel and anode is placed in between the display glass and the phosphor layer, emitted electrons are swept through a vacuum towards their respective phosphors i.e. red, green, or blue (RGB) where light is emitted when the phosphors are struck.
- CNT FED has typically the operation range of 50 -100volt but driving current is very less.



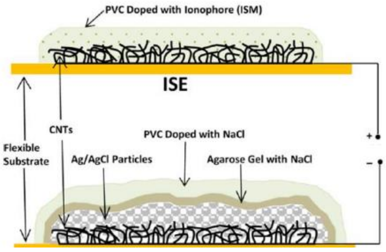
Advantages of CNTFEDs:

- High contrast and luminance
- High chemical stability
- Great thermal conductivity
- High mechanical strength (bending modulus 1 TPa)
- Fast response
- Wide viewing angle
- Low voltages requirement
- Lower Power Consumption
- Flexible dimensions
- Wide range of operating temperature
- Extremely thin

Note:
Some factors like their aspect ratio (100-10000), sensitiveness to adsorbed gases are making unfavourable realization for them but some company like Motorola and Samsung are trying to overcome these drawbacks.

9(b) CNT based Bio- chemical sensor

- Solid-state ion selective electrodes (ISEs), sensitive to Na^+ ions, were prepared by drop coating plasticized polyvinyl chloride (PVC) doped with ionophore and ion exchanger on CNT electrodes.
- The ion selective membrane (ISM) filled the intertubular spaces of the highly porous CNT film and formed an attachment that was stronger than that achieved with flat Au, Pt, or carbon electrodes.
- Concentration of the ISM solution used influenced the attachment to the CNT film, the ISM surface morphology, and the overall performance of the sensor.
- The following figure shows the CNT base ISE along with reference electrode:



- The following figure shows the steps involved in the preparation of CNT based ISEs

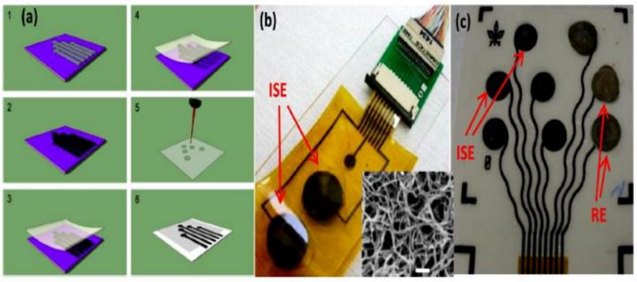


Figure 1. CNT-based flexible ISEs. (a) Fabrication flow of CNT electrodes: (1) Ni lithography on SiO_2/Si substrate, (2) CNT growth by CVD, (3,4) CNT transfer to a flexible adhesive film (5) laser cutting of a double sided adhesive film (6) aligning and bonding the double-sided adhesive film to the CNT electrode array for passivation. (b) ISEs on polyimide (Kapton) substrate. (inset) SEM image of the surface of the CNT electrodes. Scale bar is 200 nm. (c) ISEs on temporary tattoo paper.