DEPOSITION AND CHARACTERIZATION OF MICROSTRUCTURED SILICA LAYER ON GLASS FOR LIGHT EXTRACTION APPLICATION IN LED

A DISSERTATION

Submitted in partial fulfillment of the

requirement for the award of the degree

of

MASTER OF TECHNOLOGY

in

SOLID STATE ELECTRONIC MATERIALS

by

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MAY, 2016

CERTIFICATE

This is to certify that the Dissertation work entitled, **“Design DEPOSITION AND CHARACTERIZATION OF MICROSTRUCTURED SILICA LAYER ON GLASS FOR LIGHT EXTRACTION APPLICATION IN LED”** submitted by **Mr. BRAJ BHUSHAN KUMAR** to Indian Institute of Technology, Roorkee, is a record of bonafide Project work carried out by him under my supervision and guidance and is worthy of consideration for the award of the degree of Master of Technology in **“Solid State Electronic Materials”**.

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DECLARATION

I, **BRAJ BHUSHAN KUMAR,** a bonafide student of M.Tech. in **DEPARTMENT OF PHYSICS**, **INDIAN INSTITUTE OF TECHNOLOGY ROORKEE** would like to declare that the dissertation entitled **"DEPOSITION AND CHARACTERISATION OF MICROSTRUCTURED SILICA LAYER ON GLASS FOR LIGHT EXTRACTION APPLICATION IN LED "** submitted by me in partial fulfillment of the requirements for the award of the Degree of **MASTER OF TECHNOLOGY** in **2016** is my original work.

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ABSTRACT

Light emitting diodes (LEDs) are of keen interest as their energy saving property is attracting several researchers because of enormous energy crisis we are facing. Organic light emitting diode (OLED) have attracted much attention due to their advantage such as low power consumption, high internal quantum efficiency, high brightness, high contrast and low cost than those of inorganic or semiconductor based LEDs. But only 20% of the generated photons in organic layer are extracted out from the device. Thus, light extraction efficiency improvement is required before it can make any significant impact.

The primary aim of this thesis is to enhance the light extraction efficiency of LEDs/OLEDs. To increase the light extraction efficiency, we have introduced microstructured silica layer between glass substrate and indium tin oxide (ITO). By introducing silica layer between glass and ITO, more light can be extracted from the device. As silica layer is porous, more light can couple from high index layer of OLED into the glass substrate. We have studied the effect of microstructured silica layer deposited on glass substrate. Surface morphology of the prepared samples was done by AFM and FESEM. Transmission of prepared samples was carried out by UV-Vis spectrophotometer. Enhancement in transmission was seen with the prepared samples.

We have also tried to fabricate gratings on the microstructured silica film. For fabricating gratings on the silica film, nanoimprint lithography is employed. We have studied the effect of gratings on silica film. We have also studied the optical characteristics of gratings on silica film.

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Chapter 1

Introduction

The cost of lighting devices is increasing and hence there is need to obtain high energy efficient technology. This need has made its way for various technological advancement. For lighting alone more than $37 billion is spent by the united state annually. Solid state lighting through the use of light emitting diodes (LEDs) is one of the long term strategies adopted to reduce the energy consumption. Less energy efficient lighting devices such as incandescent and fluorescent lamps is replaced by disruptive technology like light emitting diode.

The light emitting diode has grown much in last 25 years. It has grown from a laboratory curiosity to a broadly used light source for signaling applications. In 1992 $2.5 billion worth of LED-based components were shipped to original equipment manufacturers and LED production reached a level of approximately 25 billion chips.

Light emitting diodes (LEDs) has brought revolution in the lighting market. It is a kind of solid-state lighting (SSL). In terms of energy efficiency, versatility, life time and colour quality LEDs is superior from many conventional lighting technologies. As there is increase in cost competitiveness in lighting technologies, there is beginning in a variety of lighting application to successfully compete. By it is estimated that LED lighting will cover over 75 percent of all lighting sales. As a consequence there will be primary energy savings of 3.4 quads in an annual.

1.1 Light emitting diode

Light Emitting Diodes are nowadays used in many applications and are replacing traditional incandescent bulbs (bulbs which operate on the principle of thermionic emission that is electrons are emitted from a heated filament). There are various advantages of these diodes like they have lower turn on voltages (they consume less power).These diodes are 3 times more efficient than the normal bulbs.

They are nothing but a simple p-n junction diode working in forward bias. We know that in forward bias the diode supports the flow of majority carriers. It works on the principle of Electroluminescence i.e. electrical energy is converted into light energy. The battery forces the electrons into the semiconductor if the semiconductor used is a direct band gap semiconductor. It has been found that direct band gap materials with band gap varying from 1.63-3.26ev are used

Which emit radiation in some sort whose range lies in the visible spectrum which ranges from 380nm-760 nm. That is of various colours like red, yellow, orange, blue, green etc. which are of single wavelength, there are also multicoloured LED's light emitting diodes in which 2 diodes of different band gap materials are arranged antiparallelly and then the biasing is done quickly so rapidly that the colour changes quickly.

Incoherent light is produced from LEDs, incoherent means that there is no any phase relation between the emitted photons. (photons which are fundamental unit of light waves.).
Mainly highly doped p and n regions are taken in this case. Most of the commercial LED's also employ this type of arrangement.

Now taking an unbiased diode p-n$^+$ junction in which V_0 is the barrier voltage which prevents further diffusion of electrons to p side and holes to n side.

Fig 1.1 shows the pn+ energy band diagram. The left figure shows the energy band diagram for unbiased pn+ junction. In general, the potential barrier penetrate more in the less doped region. Hence depletion region penetrates more into the p-side. There is potential energy gap from conduction band edge of n-type to the conduction band edge of p-side, which is called built-in potential, V_0. This built in potential blocks the excess free electrons on the n+ side to diffuse into the p side.

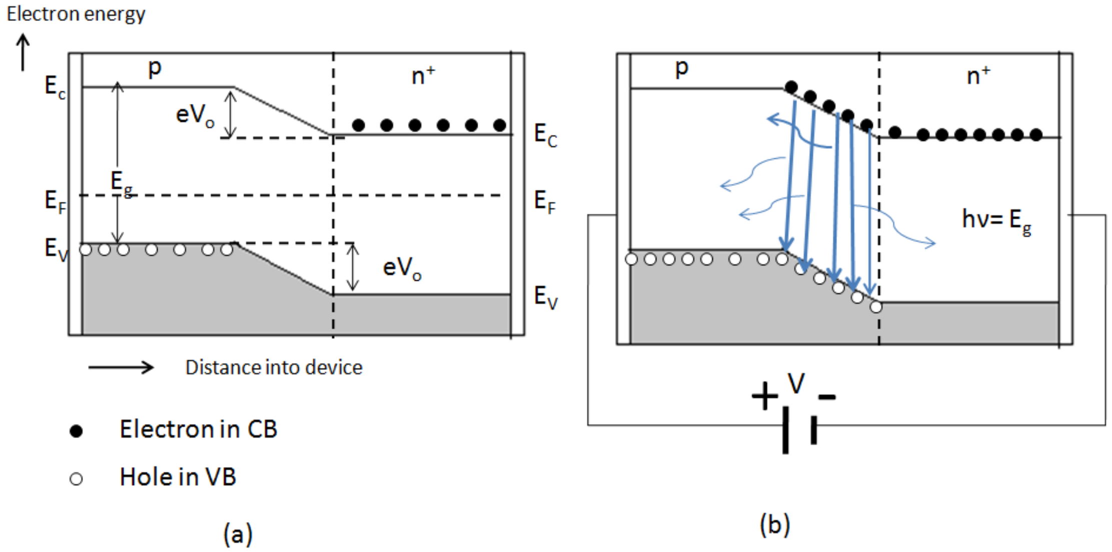


Figure 1.1: p-n+ Junction under Unbiased and biased conditions.

Now as a positive voltage is applied across the diode the built-in potential decreases by V volts. Due to this more and more majority carriers diffuse towards their opposite bulk regions where they become excess minority carriers. The electrons and the holes recombine in the active region/depletion region to produce incoherent light. This light produced is spontaneous in nature.

As n-side is heavily doped, the electron contributes mainly in current flow and current flow is very less due to holes. As the positive voltage is applied the electrons in the n-side and the holes in the p-side crosses the depletion region and goes to opposite side. In the opposite side the charge carriers becomes minority carriers. Hence it is called minority carrier injection.

This effect of luminescence is called as injection electroluminescence. Now structure is to be designed in such a way that minimum number of photons are reabsorbed within the semiconductor also the surface is made curved so that most of the photons are emitted outwards.

There are two ways by which recombination take place in semiconductor:

- Direct recombination
- Indirect recombination

1.1.1 Direct Recombination

Direct band gap materials show direct recombination. In this type of materials, the maximum energy of the valance band level and the minimum energy of the conduction band level coincide. Fig 1.2 shows the E-K plot of a direct band gap material. As the maximum energy level of valance band and minimum energy level of conduction band coincide, it has maximum

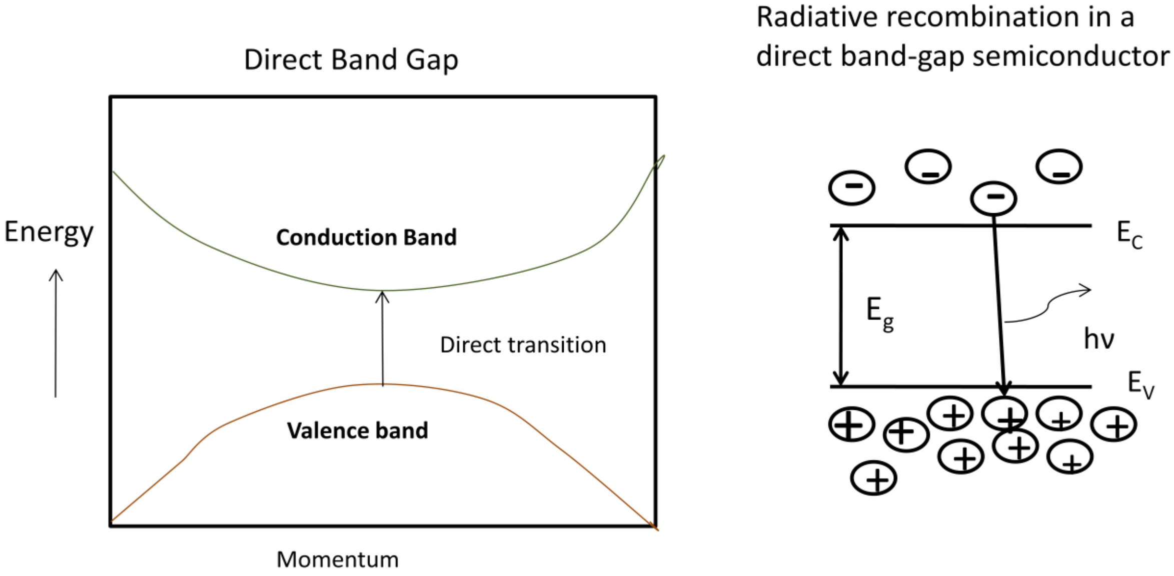


Figure 1.2: Direct Bandgap and direct Recombination.

chance that holes present at the top of the valance band is filled with free electrons present at the bottom of the conduction band. In this recombination process, there is no change in momentum. As electrons recombine directly with the holes the energy released in recombination process is entirely used in photon emission. Hence the energy of the system remains conserved. It is known as direct band recombination. GaAs/GaInP are direct band gap materials.

1.1.2 Indirect Recombination

In this type of materials, the maximum energy level of valance band is shifted by a k-vector relative to the minimum energy level of the conduction band. So for the electrons to recombine or move to the valence band there needs to be a change in the crystal momentum. So the transition results in phonon emission which are fundamental unit of mechanical vibrations. The recombination results in photon as well as phonon emission. So the probability of the recombination is very less in this case. So in these materials, additional dopants(impurities) atoms are added. Or some may be present intrinsically which temporarily captures the electron.

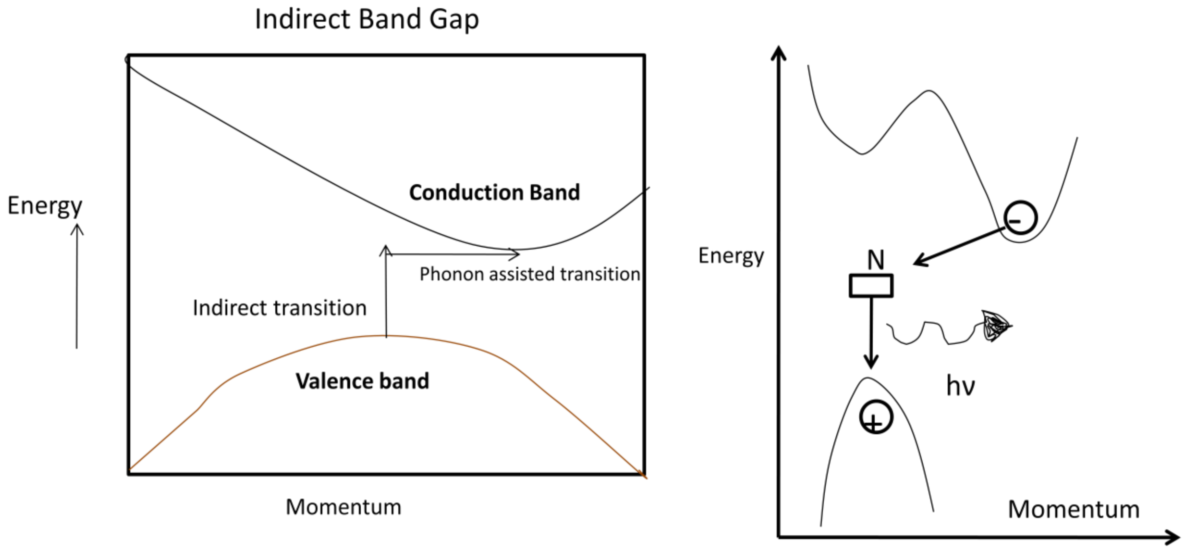


Figure 1.3: Indirect Bandgap and non-radiative recombination.

be emitting phonons. These donor sites act as local trapping centers. Figure 1.3 depicts the E(energy)-k(wave vector) plot of an indirect band gap semiconductor material (left) and also shows nitrogen working as recombination center (right). This recombination center represents the donor impurity state

The wavelength of the radiation light emitted, and therefore the colour depends upon the band gap energy of the material and is approximately equal to the energy gap of the semiconductor material. The following equations are given as:-

$$hv=Eg$$

here h is the plank's constant and its value is 6.626 $*10^{-34}$ J/sec, E_g is the energy gap of the material and v is the frequency of light emitted.

1.2 Oraganic light emitting diode (OLED)

OLED stands for organic light emitting diode. As in LED semiconductor material is used for electroluminescence, here in OLED organic material is used in place of semiconductor material. OLED is the part of organic optoelectronic devices. Other organic optoelectronic device includes organic resonant tunneling diodes, organic photodetectors, organic photovoltaic cells, and organic phototransistors. These organic devices have formed tremendous area of researc interest in physics and chemistry. Organic materials in electroluminescent devices are of much interest because of their potential application in flat panel devices and attractive characteristics, properties and features. An OLED, similar to LED, is a device emits light when an external voltage is applied across it. There basically two categories of OLED devices: first one is those made of organic molecules and the other one are those made of organic polymers. There are some unique properties of OLEDs. They are flexible, lightweight, colour tune ability and transparent. These properties make OLED an ideal modern light source. There are many benefits of using OLED technology which made interest in OLED. it operates in emissive mode (which does not require backlighting). it provides a wide viewing angle. It needs a low operating voltage (less than 5V). It provides reduction in production cost and flexible display and also provides light emission for all visible range. OLED displays are made of component devices. These component devices contain organic electroluminescent material. organic electroluminescent materials emit light when electricity is applied across it. An OLED is solid state lighting device. The thickness OLED is in the range of 100 to 500 nanometers.

The structure of OLED comprises a conducting layer and an emissive layer. These two layers are sandwiched between two electrodes and whole is deposited on a substrate. organic plastic molecules forms the conducting layer. This layer transports holes form the anode. Organic compound forms the emissive layer. it transports electrons from cathode. this layer also emits light when electricity is applied. In organic layer there is delocalization of pie electrons. This delocalization is caused by conjugation in organic molecule in part or all over organic molecule. Therefore the conduction layer varies from a conductor to an insulator. The injected electron-hole pair recombines at the interface of conducting and emissive layer efficiently and results in electroluminescence. Thus OLEDs are bipolar devices. Simultaneous supply of both electrons and holes to the electroluminescent material are required. In this OLED there are two layers: at the organic-electrode layer interface, the conduction band of organic compound (that is LUMO) receives electrons from the cathode and the valance band of the organic compound (that is HOMO) receives holes from anode.

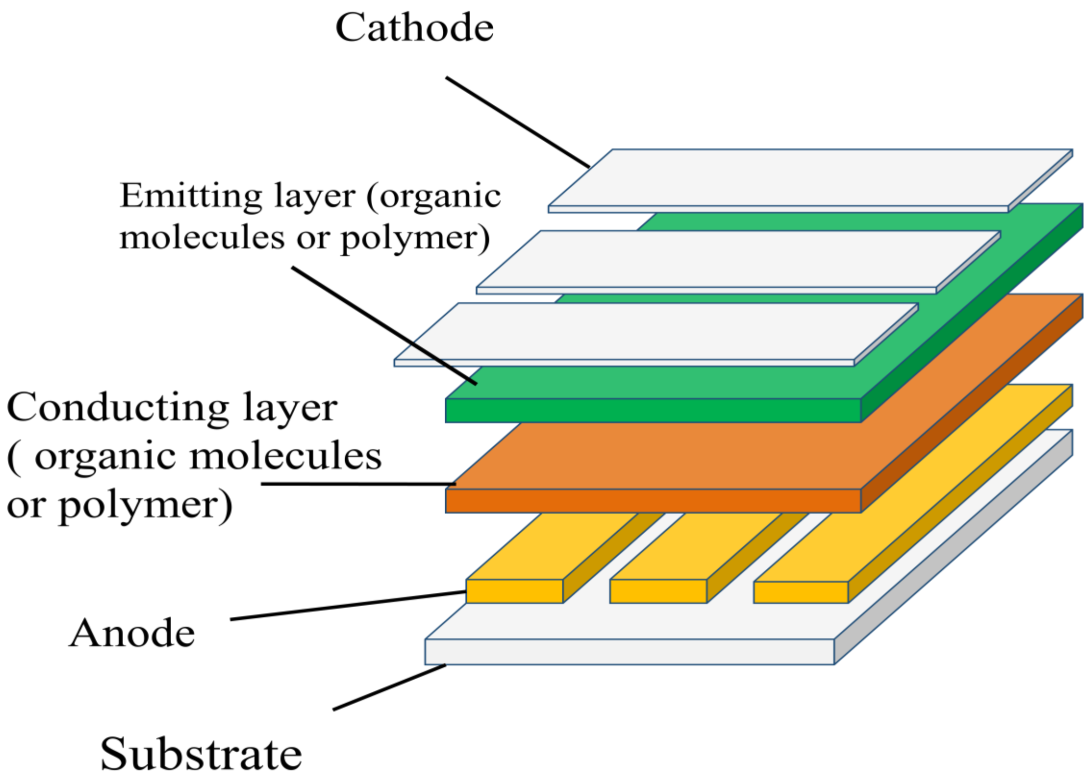


Figure 1.4**:** Fundamental Structure of OLED.

In OLED made of three layers: electron-transport layer (ETL) and hole-transport layer (HTL) are introduced in place of conductive layer. These two layers are more effective than one conductive layer. Now when a positive voltage is applied across OLED, that is anode is more positive than cathode, the HOMO of HTL receives holes from the anode and the LUMO of ETL receives electrons from the cathode. The oppositely charged electrode attracts the injected electrons and holes when an external electric field is applied which results in a hopping transport regime. In hopping transport charge jumps in series from on molecule to other molecule. now consider organic emissive layer(EML), localization of a hole and electron on the same molecule and which are also spatially close, some of them form exciton(a bound state of hole and electron) by recombination: this is an excited energy state of a localized electron-pair. Using photoemissive mechanism some of this exciton relaxes and hence cause spontaneous emission of light as they decay radiatively to the ground. There are some cases when localization of exciton may be on an exciplex(excited complex) or an excimer(excited dimer).there is also thermal relaxation using non-radiative mechanism which occur but it is undesired generally. After recombination, light is produced due to release of energy and for proper emission of light at least on electrode must be transparent. thus it results in a crispy and very bright display consuming less power in comparison to LCD and LED.

In the year 1987, the scientists from KODAK also introduced a double layer organic deposition technique which is biased at lower voltages. At the lower voltages they produce an attractive luminous efficiency..

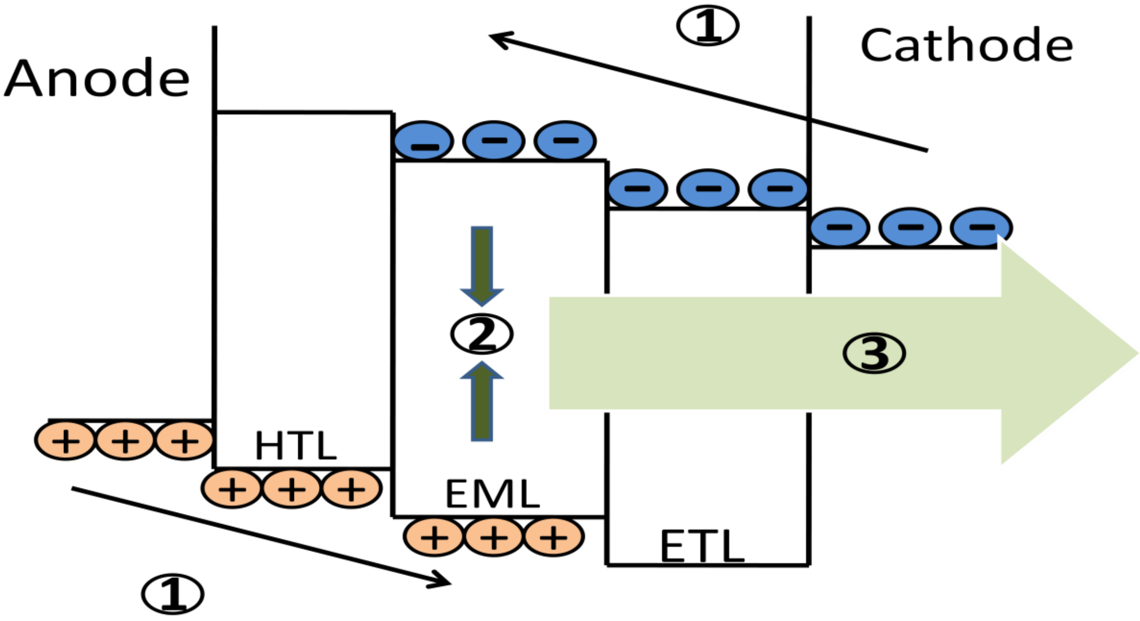


Figure 1.5: Scheme of the electroluminescence mechanism in an OLED.

(1) electrons in colour (blue) and the holes in the colour (yellow) they are injected from the respective electrodes into the Organic Emissive Layer(OEL).

(2) The formation of an electron and a hole pair and an exciton (green.)

(3) The photon is emitted while emitting radiatively recombining.

Although it is known that smaller molecules emit light of higher intensity i.e. brighter in light, for this the Scientists had to deposit the substrates into the vacuum region by (an expensive process of manufacturing known as vaccum deposition process.) In the year 1990, some of the researchers have produced the first ever polymer light emitting diode which is made by using a conjugated polymer like poly-para-phenylene-Vinylene(PPVL)which is given by (Figure 1.6a), which is still one of the so far /widely used polymer. Then A further boost for the polymers based devices came to arena when the emission was due to the emissions from the polyfluorenes given by (Figure 1.6b). So As of present these kind of polymers exhibit one of the most promising characteristics for its commercial applications. The devices based on these kind of devices are the polymers which now have the efficiencies excessive than 20lm/W and the efficiencies which are in excess to 20 lm/W and which are broadly classified as being comparable to the inorganic type of LEDs (Light emitting diodes)that are based on III-V semiconductors in the terms of efficiencies and brightness. Now these polymers can be made cheap /less expensive and can be drawn into also large sheets, so that they are more suitable for screen displays, large Screen displays.. Besides, this the polymer OLEDs are pretty efficient and require a relatively tiny amount of power for the amount of the light produced.

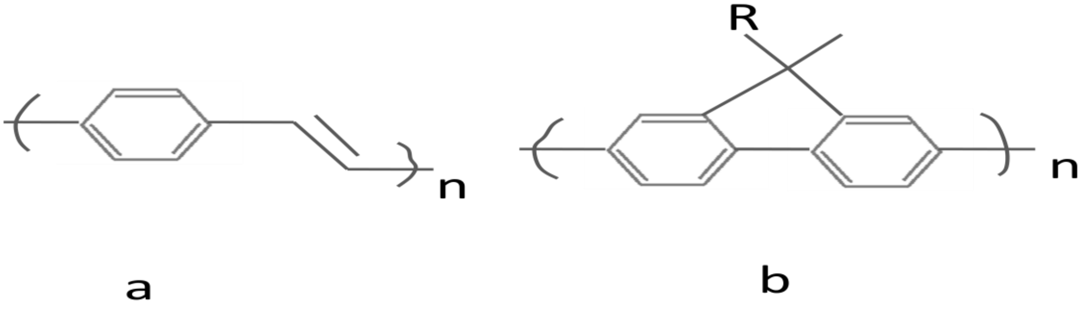


Figure 1.6**:** Mostly used polymers to produce electroluminescence in OLEDs.

In an OLED a single organic emitter is sandwiched between a transparent anode and a metallic cathode. by using all current materials it is difficult to produce high luminescence quantum yield using a single organic emitter. Hence it is difficult to achieve OLED with single-layer configuration which is efficient and it gives poor efficiency. It also has high driving voltage. The external quantum effieciency has been improved by using two layers of different organic materials. And by using two layers of different organic materials driving voltage has also decreased. The fluorescent OLEDs showed upto 8% efficiency when we add hole and electron blocking layers. These blocking layers force the charge carriers to recombine within the bulk. The phosphorescent OLEDs showed up to 19% efficiency when we used these blocking layers, here the triplet excitons also converts into light. Organic materials of interest in OLEDs which are highly phosphorescent or fluorescent tend to have either p-type (hole transport) or n-type (electron transport) charge transport characteristics. The type of organic molecule determines the color of the light in the emissive layer. To make colour displays several types of organic films on the same OLED is placed by manufacturers. The amount of electrical current determines the intensity or brightness of the light. The more is current, the brighter the light. The pursuit for efficient and long lasting OLEDs has seen significant effort in four fields of development: materials, device structure, optical engineering and electrical engineering.

Like all other light emitting devices quantum efficiency is also defined for organic light emitting diode. The quantum efficiency of OLED is divided into two classes: internal quantum efficiency and external quantum efficiency.

The internal quantum efficiency is defined as the ratio of number of photon generation by the device to the number of electron-hole pair injected. It is denoted by η_{int}. in the emitting layer the number of electron-hole pair determines the efficiency of OLED, which is obvious that more is the number of recombinations more is the efficiency of the OLED or better will be the efficiency. On the theory basis even if all the holes and electrons excite to the excited state, the internal quantum efficiency will be less than 50%. As only a singlet excited state participate in the light emission it causes limitation on IQE. The triplet states and the singlet states forms the excited states of the conjugated polymers. Instead of emitting the light a triplet state will emit heat and heat produced degrades the device. For pretty small molecules, there consists of about 25% singlet states in number, which results in a lower efficiency of power than that of in conjugated polymer. The strong spin-orbit coupling due to introduction of heavy metal ions such as Ir, Pt and Os into emitter molecules mixes the excited triplet and singlet states and this results in an emission. This emission is known as the disallowed phosphorescence. Thus the rest of 75% of triplet spin state is emissive in nature. So, an internal quantum efficiency level of 100% is achievable in principle. The basic of physical mechanisms responsible for light emission in organic light emitting diode is understood with the help of external quantum efficiency.

The Internal Quantum Efficiency here IQE and the lifetime here have undergone dramatic /drastic improvements since the first reports of nearly 100% internal quantum efficiency levels IQE levels for the organic fluoroscent OLEDs(Organic light emitting diodes) and the devices with the phosphorescent- emitting materials. Here however the majority portion of the light is generated in an emitting layer is confined within the transparent electrodes and the glass substrates due to the pretty large difference in the values of the refractive indices of both the layers ($n_{electrode}$=1.9 and n_{glass}=1.5).These are the refractive indices of the electrode and the glass. Thereby as explained by the classical ray theory following the (Snell's law), this difference in the refractive indices hence produces a very low output-coupling efficiency (η_{out}) which is around 20%,hence the efficiency level here is expressed by the ratio of the surface emission to all the emitted light. Then the remaining of the 80% of the photons emitted are trapped into the organic and the substrate modes. This low efficiency of the output coupling is here a major limitation on the high efficiency of the OLEDs. Thus, here there are the two major challenges here helps in the attainment of a highly EQE (external Quantum Efficiency), hence to fabricate an OLED structure with enhanced quantum efficiency and thus to investigate the related factors and the mechanisms here that will affect the light output coupling characteristics.

It was reported that nearly 100% internal quantum efficiency levels had been achieved for devices with phosphorescent-emitting materials and organic fluorescent OLEDs. Because of the large difference in refractive index of ITO (n=1.9) and that of glass (n=1.5) majority of light generated in an emitting layer remains confined. From the classical theory of ray optics a low out-coupling efficiency (η_{out}) is produced due to this difference in refractive index of around 20%. The rest of 80% of photons remains trapped in substrate and organic modes. Hence for attainment of high external quantum efficiency there are two major challenges: to find out the mechanisms and factors that affect the light out-coupling characteristics and to design an OLED structure with enhanced ηout.

1.2.1 The external quantum efficiency of OLEDs

1.2.1.1 Fundamental principle of the OLED

The external quantum efficiency, η_{ext}, is defined as:

$$\eta_{ext}=\frac{number\ of photons\ emitted\ from\ the\ device}{number\ of\ electrons\ and\ holes\ injected\ into\ the\ molecular\ layers}$$

The efficiency is expressed as

$$\eta=\Upsilon.\chi.\eta_r\ .\eta_{out}=\eta_{int}.\eta_{out}$$

η_r= photoluminescent quantum efficiency of the organic material

η_{out}= light out-coupling efficiency

The EQE (External Quantum Efficiency) here is given by the multiplication of charge carrier balance factor, γ; the excitonic spin factor, χ. The multiplication of the charge balance factor, the photoluminescent quantum efficiency and the exciton spin factor these are often summarized as the IQE (internal Quantum Efficiency), η_{int}.

The probability of an electron meeting a hole and forming exciton is termed as charge carrier balance factor. On the contrary unbalanced charge which do not form exciton their mobility gives either the electrons or holes travelling through the whole device with high probability and such behavior renders a low internal quantum efficiency and a large leakage current. Using the charg injection layer, the charge balance factor can be optimized. LiF, MgF_2, BaF_2, MgO, and $CsCO_3$ forms the EIL materials in the cathode region. HIL materials in the anode region generally consists of transparent transition metal oxides, particularly NiO, V_2O_5, CuO, MoO_3, and WO_3. A charge balance factor of nearly 100% is produced by using EIL and HIL.

The spin statistics of the formed excitons is represented by the exciton spin factor. A singlet-to-triplet ratio of 1:3 is present in OLEDs because the electrons and holes have a random spin distribution. Only singlets can decay to a ground state when fluorescent emitters are used because the relaxation of the triplets into a ground tate is spin forbidden. However, to introduce strong spin-orbit coupling if phosphorescent emitters are used the quantum mechanical selection rules are relaxed and triplets can also produce an χ value of 100%.

The probability of a radiative recombination of excitons is represented by the photoluminescence quantum efficiency. It is the ratio of number of emitted photons to the number of generated excitons.

$$\eta_r = \frac{K_r}{K_r + K_{nr}} = \frac{number\ of\ emitted\ photons}{number\ of\ generated\ excitons}$$

κ_r = Rate constant of radiative decay of the exciton

κ_{nr} = Rate constant of non-radiative decay of the exciton

An η_r value is nearly 100% can be achieved easily with very high-purity organic semiconducting materials.

The exciton spin factor, the charge balance factor and the photoluminescence efficiency gives the internal quantum efficiency. The internal quantum efficiency of phosphorescent OLEDs can reach 100% if the photoluminescence quantum efficiency and the charge balance factor both are 100%. But the generated light within the device may not couple-out through the transparent substrate of which there is some probability. Hence the external quantum efficiency should be reduced due to this out-coupling loss and its value cannot excess 20%.

1.2.1.2 Output coupling efficiency of the OLEDs

The waveguide mode, the electrode absorption, the substrate mode and the surface plasmon polaritons (SPPs) cause the out-coupling losses. About 40% of the generated light is dissipated as a non-radiative emission in the cathode or anode region or as scattered light. The structure of OLEDs consist of an organic layer (n=1.8) sandwiched between a reflective metal contact and a transparent electrode, such as indium tin oxide (ITO, n=1.9) on a glass substrate (n=1.5). the total internal reflection phenomenon can occur at the ITO-glass interface and the glass-air interface by the application of Snell’s law. Thus, the substrate-air interface (substrate mode, 23%) contains a significant portion of generated light. Similarly ITO-glass interface (waveguide mode, 15%) contains a significant portion of light generated. About 4% of light is absorbed at the electrodes. Thus there is limitation on the light extraction efficiency to only 18% by using conventional planar layered structured of OLEDs. Hence the task of attaining a high external quantum efficiency in OLEDs is hindered by low out-coupling efficiency. So the external quantum efficiency of phosphorescent OLEDs is only 18% whereas it has an internal quantum efficiency of 100%. Due to the small exciton spin factor ($\chi = 25\%$) the same external quantum efficiency dropped to 4.5% for fluorescent OLEDs. For the purpose of extracting the light that is confined within the OLED structure many device structure has been proposed.

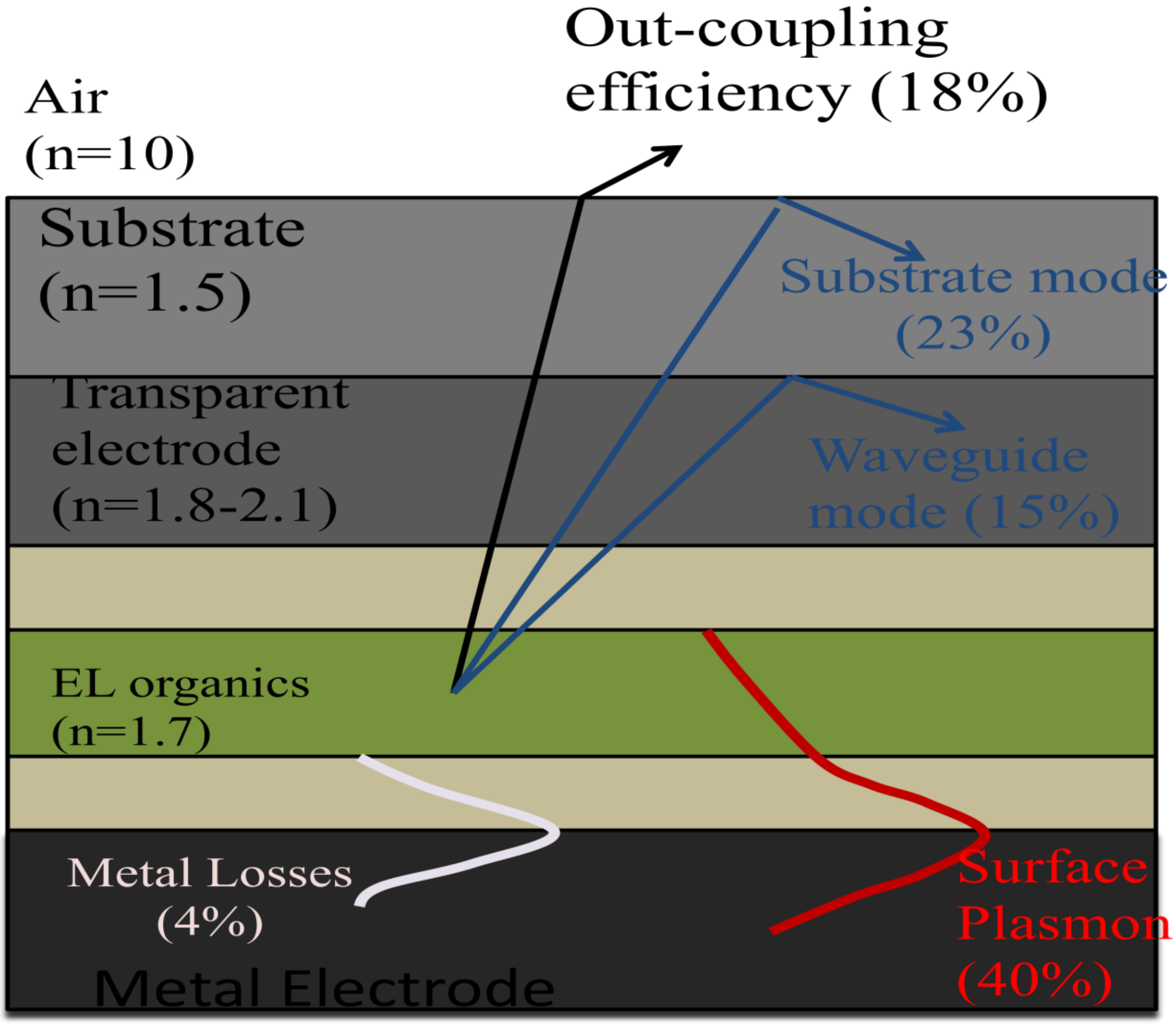


Fig. 1.7: Various kinds of light out-coupling losses in normal structure OLEDs.

Chapter 2

Simulation Work

2.1 Why simulate OLEDs/LEDs

- Light generated inside organic light emitting diodes (OLEDs) and planar light emitting diodes (LEDs) remains trapped inside due to total internal reflection (TIR) and eventually get absorbed in the material. Hence OLEDs and LEDs do not have optimal quantum efficiency.
- The quantum efficiency of these devices can be increased by micro and nano scale patterning.
- Calculation of the effect of these patterns is a challenge.
- Simulation gives the opportunity to cheaply and quickly test ideas, optimize designs and solve problems.

2.2 FDTD method

One of the most popular algorithm in electromagnetics is finite-difference time-domain method (FDTD). It is state-of-the-art method for solving maxwell's equation. As a direct time and space solution, all type of problems in photonics and electromagnetic offered by FDTD to the user. Maxwell's equations solution is accomplished by approximating the partial differential equations by finite differences, both in time and in space. In order to obtain an evolution of the fields in time, we use the time derivatives of the coupled Maxwell's curl equations. From its current distribution at time t and from the spatial derivation of the magnetic field distribution at an intermediate point in time t+Δt/2, the electric field distribution for the next time step t+Δt is derived. From the magnetic field distribution at time t-Δt/2 and a spatial derivation of the electric field distribution at time t, the latter has been calculated. Hence, the field evolve by alternately calculating the new electric and magnetic field distributions. The solution are more robust because both fields are used instead of only one of them. It allows to approximate both the space and time derivatives using the unique spatial discretisation scheme via so-called Yee cells but central-difference finite difference that are second-order accurate. In general, the strength of this approach relies on the fact that as long as the computational domain can be stored within the available computer memory and the spatial grid is dense enough almost arbitrary structure can be calculated. On simulation run yields the response of the system through a frequency analysis of the temporal field evolution within abroad frequency range since the method is time domain based.

2.3 OLED design

Photonic crystal (PC) patterns in two dimensional slab InGaAs light-emittng structures has previously been used for increasing the extraction efficiency. Similarly, PC structure can be used in OLED devices. The photons confined in the high-index guided mode account for up to 50% of the total emitted energy. We plan to couple these photons with external mode. It is given that only 20% of the total photons drains into direct transmission mode, significant improvement in the total light extraction into the air is expected. To maintain the image quality required in display devices, it is important to extract the photons that is close to the light-emitting region from the high index layer.

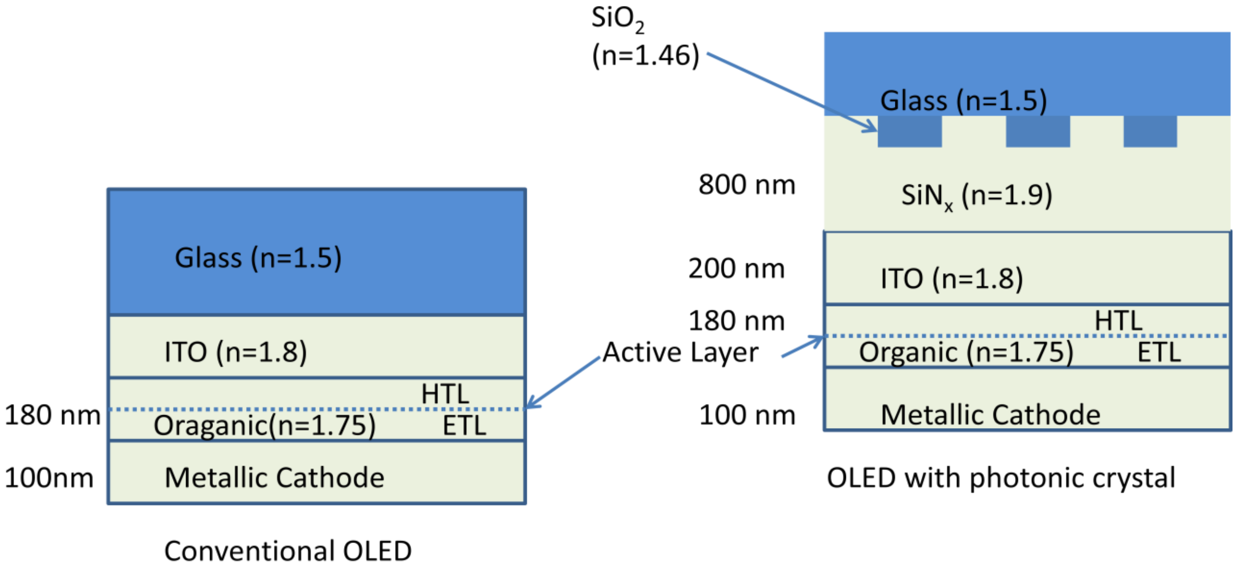
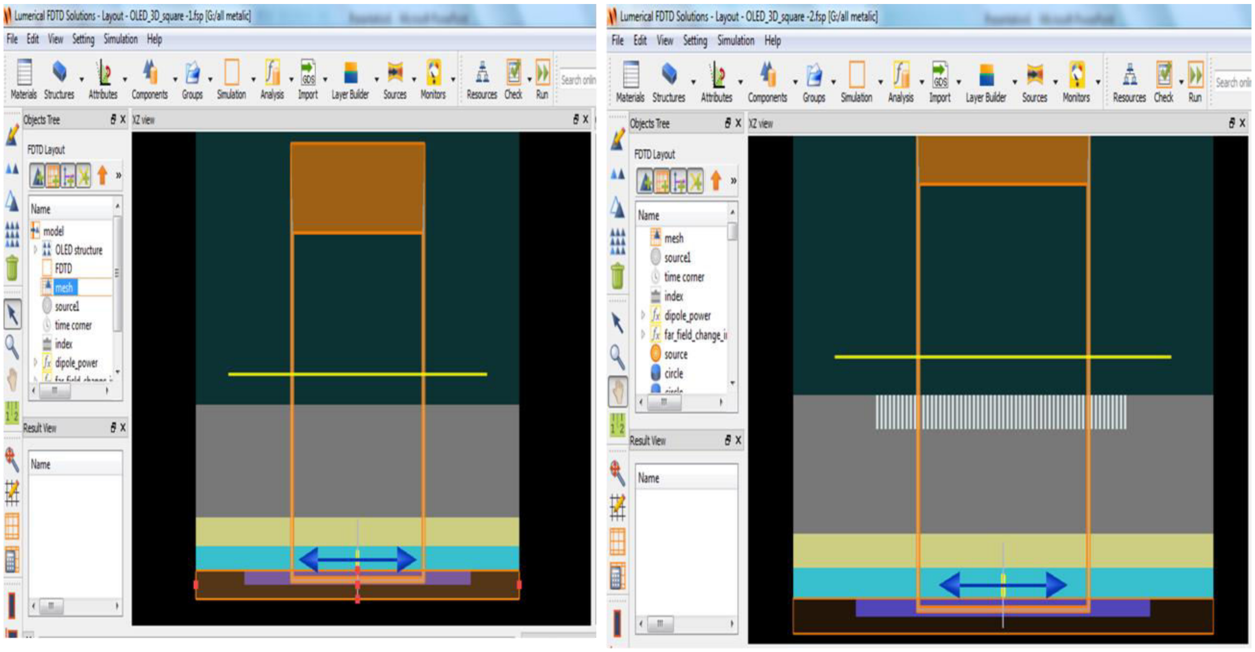


Fig 2.1: OLED desing with photonic crystal modified in conventional OLED

To study the emission characteristics of the OLED shown schematically in Fig. 2.1, we employed the finite difference time-domain FDTD method. For OLED devices in which the electron-transporting layer (ETL) is only a few tens of nanometers distant from a metallic reflector and in which the layer structure is complex, the FDTD method has been shown to be very effective.

For the horizontal dipole source ($d_{x,y}$) and the vertical dipole source (d_z) the radiation profiles were investigated by varying the distance D_a between the metallic cathode and the active layer. Most of the radiation from the d_z dipoles cannot escape the glass into the air since the radiation is emitted below the critical angle. Hence, we focus on the in-plane dipoles d_x and d_y . note that constructive interference is expected when the ETL thickness Da is l/(4n_{ETL}) since the image dipole of $d_{x,y}$ induced by the metallic cathode is out of phase with the original $d_{x,y}$. there is enhancement of the vertical radiation when D_a('80 nm) satisfies the condition for constructive interference. We then spatially integrated the outcoupled radiation power over several values of the viewing angle 90° ± 40°, and confirmed that the optimized ETL layer thickness is about 80 nm. The thickness of the ETL layer and the position of the active layer are critical to the design of the OLED structure.

The photonic crystal pattern in OLED devices enhance the extraction efficiency. This enhancement is related to three factors: the depth of the pattern (d), the lattice constant (L) and the size of the rod.



Conventional OLED

OLED with photonic crystal

Fig 2.2: FDTD simulation image of conventional OLED and OLED with photonic crystal

Fig 2.2 shows the FDTD simulation image of OLED with photonic crystal (right). In the simulation there are five layer: at the bottom metallic layer, then organic layer , then ITO layer, then SiNx layer and at the top glass layer. The thickness of metallic layer is 100 nm. The thickness of organic layer is 180 nm. The thickness of ITO layer is 200 nm. The thickness of SiNx is 800 nm. The photonic crystal which is made of silica material is embedded in SiNx layer. The radius of photonic crystal rod is 0.3L. We have placed perfect reflectors at the domain boundaries. We temporally integrated the energy extracted up to the average time needed into the air for photon to reach the boundar of the pixel. For isotropic light emission the *dx*,*y*,*z* dipoles are distributed evenly in the active layer.

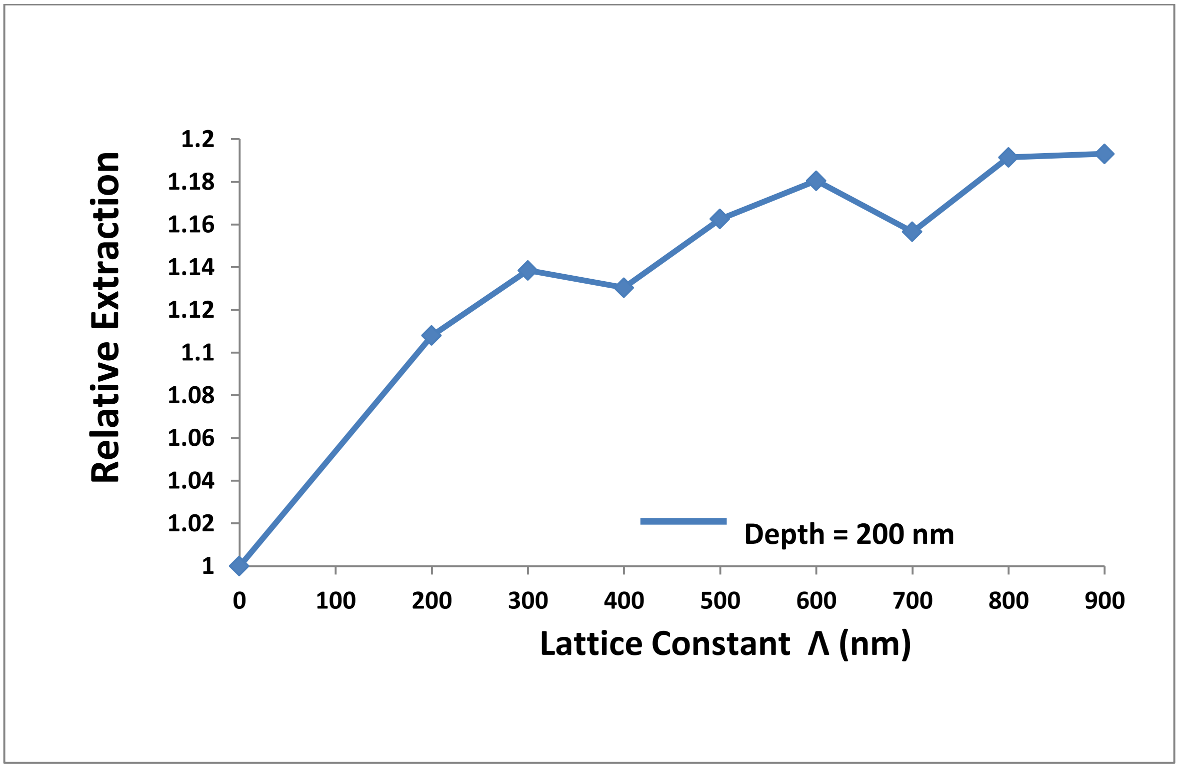


Fig 2.3: Relative extraction efficiency with lattice constant of photonic crystal rod and changing radius of the rod

Photonic crystal LED is two dimensional. The radius of the photonics crystal rod depends on the lattice of photonic crystal. Fig 2.3 shows the relative extraction efficiency of OLED with photonic crystal for different lattice constant of photonic crystal and different radius of the rod. As we increase the lattice constant and the radius of the rod the extraction efficiency increases up to a certain value after that it starts decreasing. The graph is plotted for the depth of photonic crystal rod of 200 nm.

Chapter 3

Experimental Work

3.1 Fabrication design

My goal is to increase the light extracting from glass in OLED. To meet this purpose I introduce microstructured silica layer between glass and ITO. Fig 1 shows the design of fabrication in which I have deposited microstructured silica on glass and then ITO is deposited on top of microstructured silica.

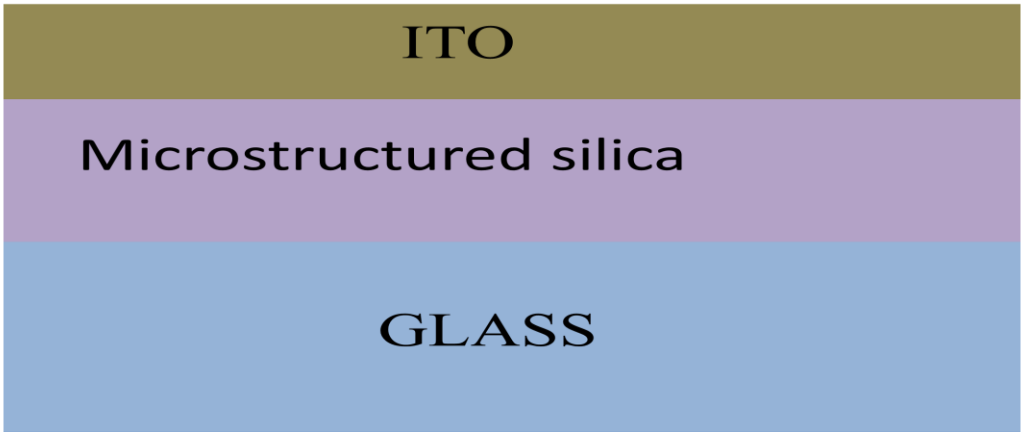


Fig 3.1: Microstructured silica is deposited on glass and then ITO is deposited on microstructured silica.

To fabricate the design first I have to prepare silica using sol-gel method.

3.2 Sol-gel method

Sol-gel method is : "Formation of an oxide network through polycondensation reactions of a molecular precursor in a liquid."

A sol is a stable dispersion of colloidal particles or polymers in a solvent. The particles may be amorphous or crystalline. An aerosol is particles in a gas phase, while a sol is particles in a liquid.

A gel consists of a three dimensional continuous network, which encloses a liquid phase, In a colloidal gel, the network is built from agglomeration of colloidal particles. In a polymer gel the particles have a polymeric sub-structure made by aggregates of sub-colloidal particles.

Generally, the sol particles may interact by van der Waals forces or hydrogen bonds. A gel may also be formed from linking polymer chains. In most gel systems used for materials synthesis, the interactions are of a covalent nature and the gel process is irreversible. The gelation process may be reversible if other interactions are involved.

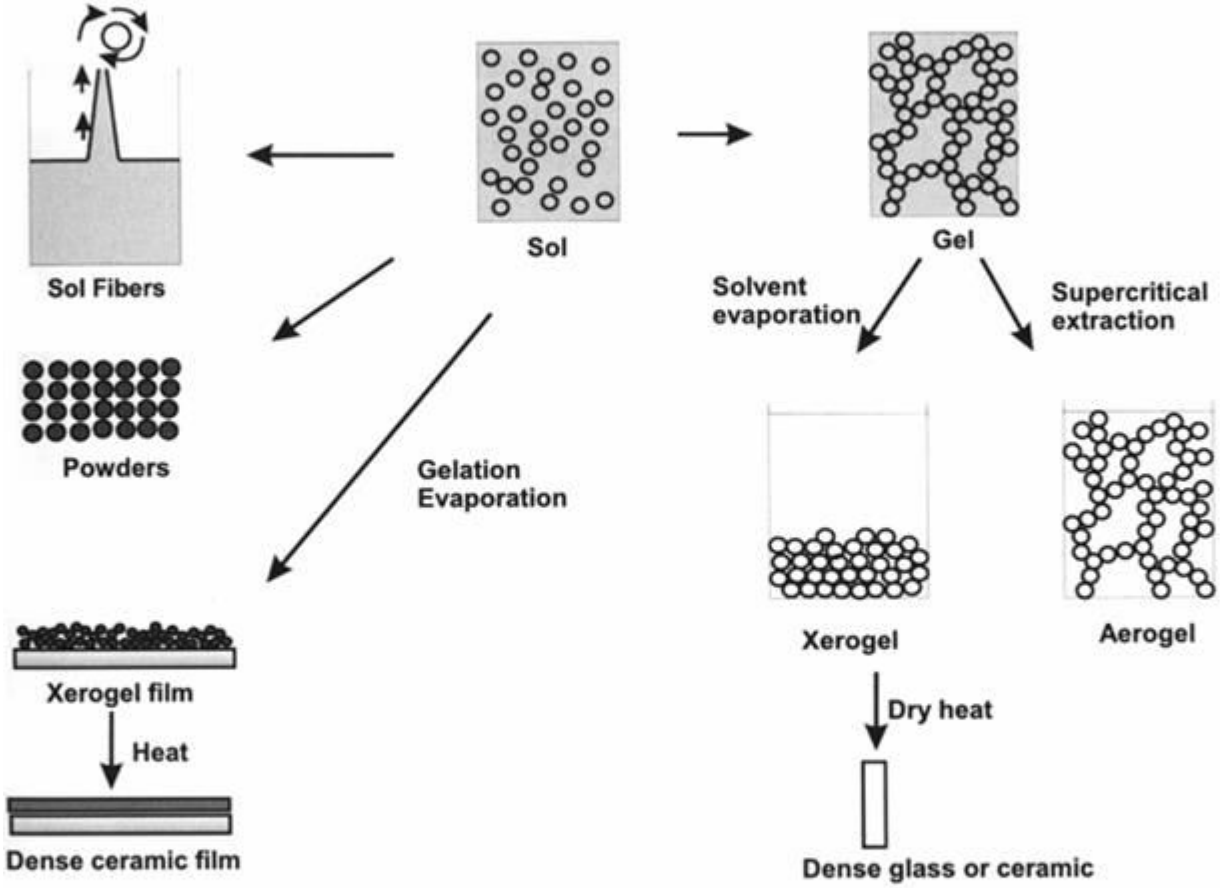


Fig 3.2: Sol-gel process.(Source Ref.[4])

Sol-gel synthesis may be used to prepare materials with a variety of shapes, such as porous structures, thin fibers, dense powders and thin films. If the gel is dried by evaporation, then the capillary forces will result in shrinkage, the gel network will collapse, and a xerogel is formed. If drying is performed under supercritical conditions, the network structure may be retained and a gel with large pores may be formed. This is called an aerogel.

3.2.1 Microstructured silica preparation using sol-gel method

Materials required: TetraEthylOrthoSilicate(TEOS), Ethyl Alcohol, Ammonium Hydroxide.

Method:

Take two beakers. In beaker 1 take 1.7 ml TEOS and then add 11 ml ethyl alcohol drop by drop to it. In beaker 2 take 15.7 ml ethyl alcohol and add 0.25 ml ammonium hydroxide drop by drop. Stir it continuously for 5-10 mins. Now add beaker 2 contents to beaker 1 drop by drop.

Keep this solution on hot plate at 250 rpm and ambient temperature for around 2 hours. And then keep the solution for overnight.

3.2.2 Deposition of prepared silica on glass

3.2.2.1 Spin coating method

Spin coating process involves depositing a small puddle of a fluid material on the center of the substrate and then spun the substrate at high speed.

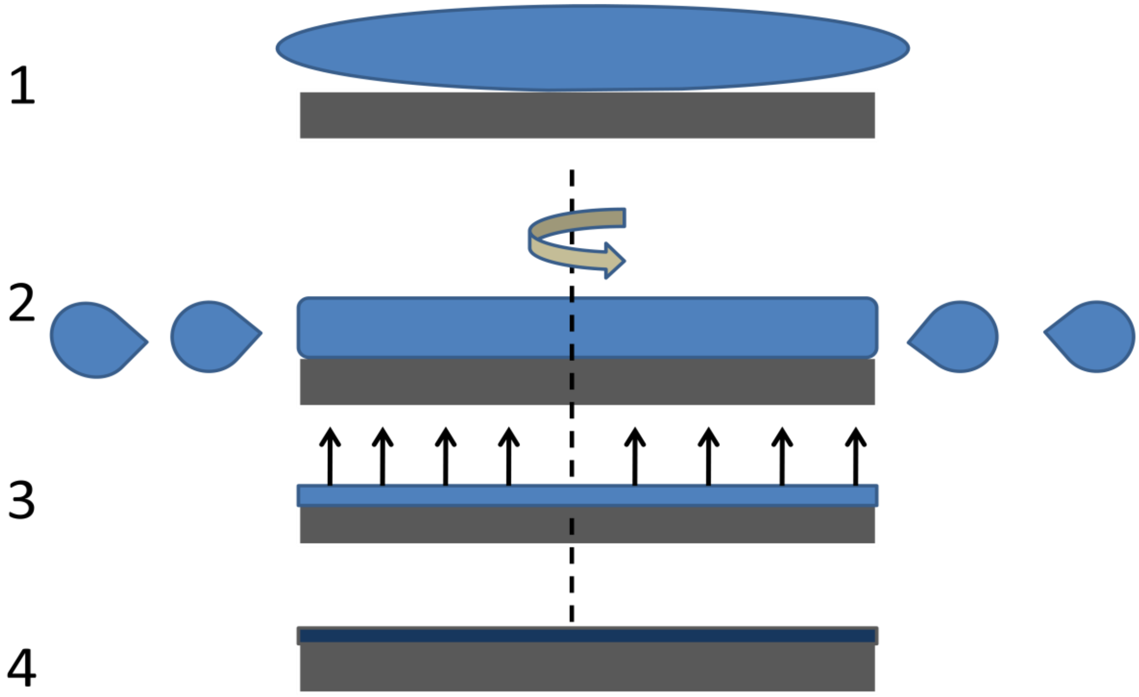


Fig 3.3: Example of spin coating a small molecule in solution using a static dispense.

- First the substrate is coated in the ink containing the molecules dissolved in a solvent (1).
- Then substrate is rotated at high speed and the majority of the ink is flung off the side (2).

- Airflow the dries the majority of the solvent leaving a plasticized film(3) befor the film fully dries to just leave the molecules on the surface (4).

The rotation of the substrate at high speed (usually >10 rotations per second = 600 rpm) means that the centripetal force combined with the surface tension of the solution pulls the liquid coating into an even covering. During this time the solvent then evaporates to leave the desired materia on the substrate in an even covering.

3.2.2.2 Silica deposition on glass

Silica prepared using sol-gel process is in liquid form. To deposit the silica on glass we use spin coating method. Here I use glass as a substrate material and silica as solution. I put the glass in the spin coater and press on the vacuum pump. The spin coater is set at 2500 rpm and glass rotates for 15 seconds. Final film thickness depends on the nature of the fluid material and the parameters chosen for the spin process. A high speed spin step to thin the fluid and vice- versa.

Now silica deposited on glass sample is fully dried on hot plate. The sample is left on the hot plate around 90 degree Celsius temperature for 2-3 hours.

3.3 Characterization

3.3.1 UV spectrophotometry

UV spectrophotometer is used for transmittance measurement. This technique is based on absorption of light. The sample is exposed to a beam of light. The sample absorbs light and

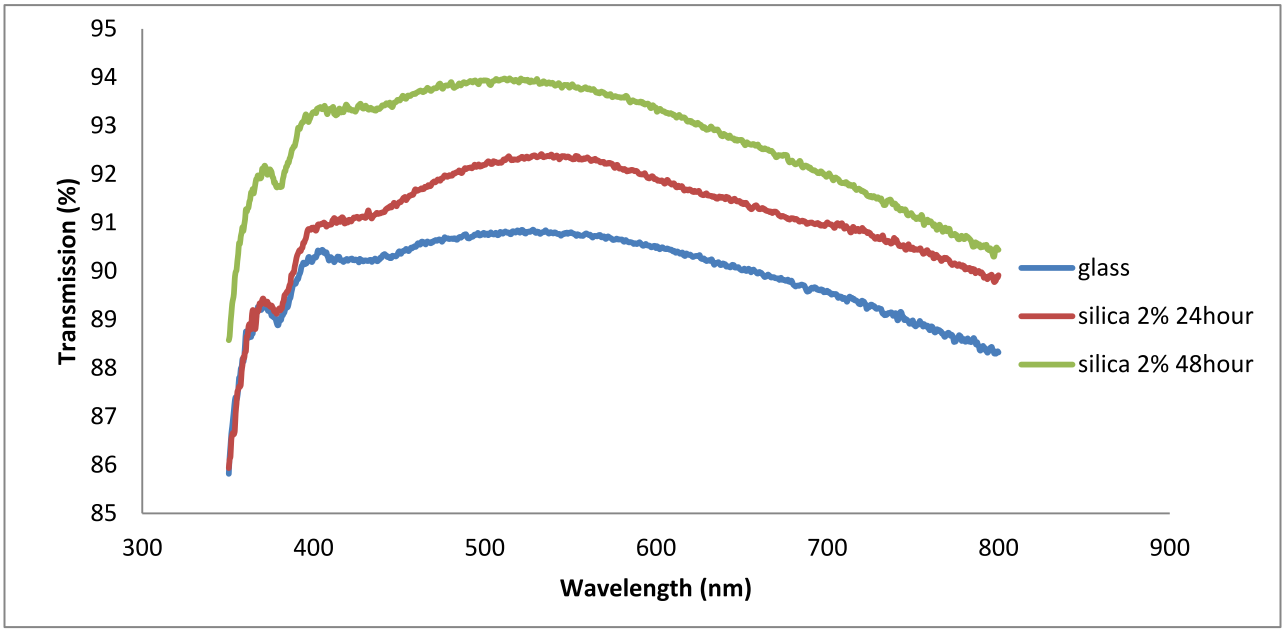


Fig 3.4: transmittance of silica on glass compared with plain glass

instrument measures transmitted light. Concentration of the analyte is proportional to the amount of light absorbed.

Fig 3.4 shows transmittance of silica sample compared with glass. In this figure green curve shows transmittance of silica sample in which silica solution is left for aging for 48 hours. Red curve shows transmittance of silica sample in which silica solution is left for aging for 24 hours.

Both green and red curve shows transmittance greater than glass (blue curve). So it shows that deposition of silica particle on glass enhance the light extraction from glass.

3.3.2 Morphology

3.3.2.1 Field emission scanning electron microscope (FESEM)

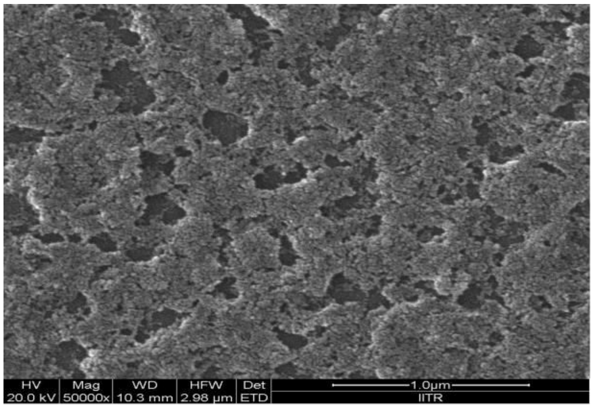
The FESEM is a state-of-the-art electron microscope. The new field-emitter technology and advanced Gemini column allow for ultra-high resolution electron imaging, over x1,000,000 (>10nm at 1kV) for a some samples. At such low voltages, non-conductive samples are easily imaged without coating.

The VP mode runs the sample chamber at high pressure (20-40Pa) which assisting in charge-compensation of the sample. This allows insulating samples to be analyzed and imaged without coating.

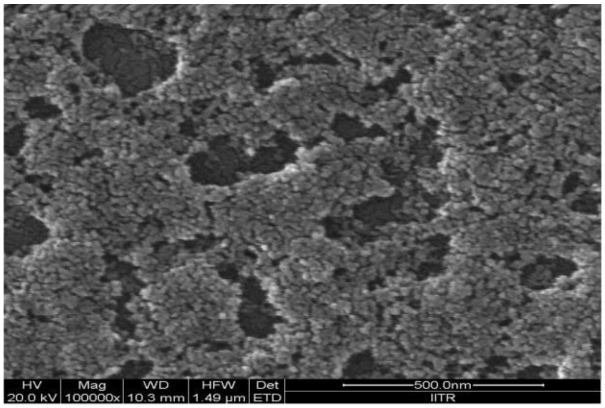
Fig 3.5: FESEM apparatus in IIT Roorkee

The EDS system detects X-rays emitted from the sample as a result of the high-energy electron beam penetrating into the sample. X-ray spectra can be collected and analyzed, yielding quantitative elemental information about the sample. A "standardless" routine is utilized, which yields an accuracy of 1-2% and sensitivities for some elements down to 0.1 weight percent. Lines can and x-ray maps can also be generated.

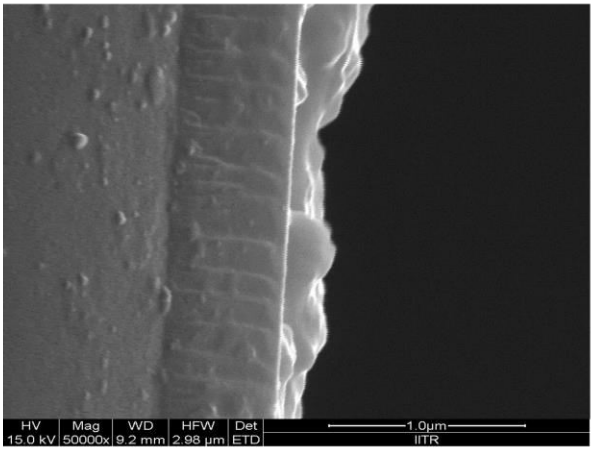
The Electron Backscattered Diffraction (EBSD) system utilizes a fluorescent screen to capture the electrons diffracted from a crystalline sample. The diffraction pattern is characteristic of the crystal structure and orientation of the sample region from which is was generated. With the aid of very sophisticated software and multiple crystallographic databases, crystal structures can be identified and oriented. For an expertly-polished sample, mapping of orientations, stresses, morphologies, etc. are also possible.



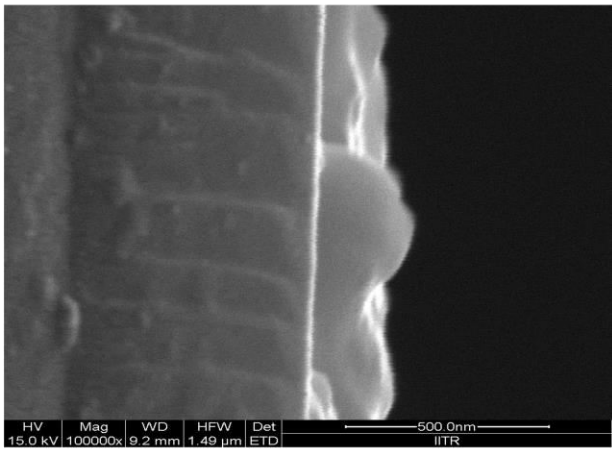
FESEM IMAGE 1



FESEM IMAGE 2



FESEM IMAGE 3



FESEM IMAGE 4

Fig 3.6: Different FESEM image of silica on glass

fig 3.6 shows the FESEM image of silica on glass. FESEM image1 and image2 shows surface structure of the film. FESEM image1 has taken at 1 micron scale and FESEM image2 has taken at 5oo nm scale. FESEM image3 and image4 shows the thickness of the film. As from the image it is clear that surface of the silica film is non-uniform, the thickness of the film varies from 150 nm to 250 nm.

3.3.2.2 Atomic force microscope (AFM)

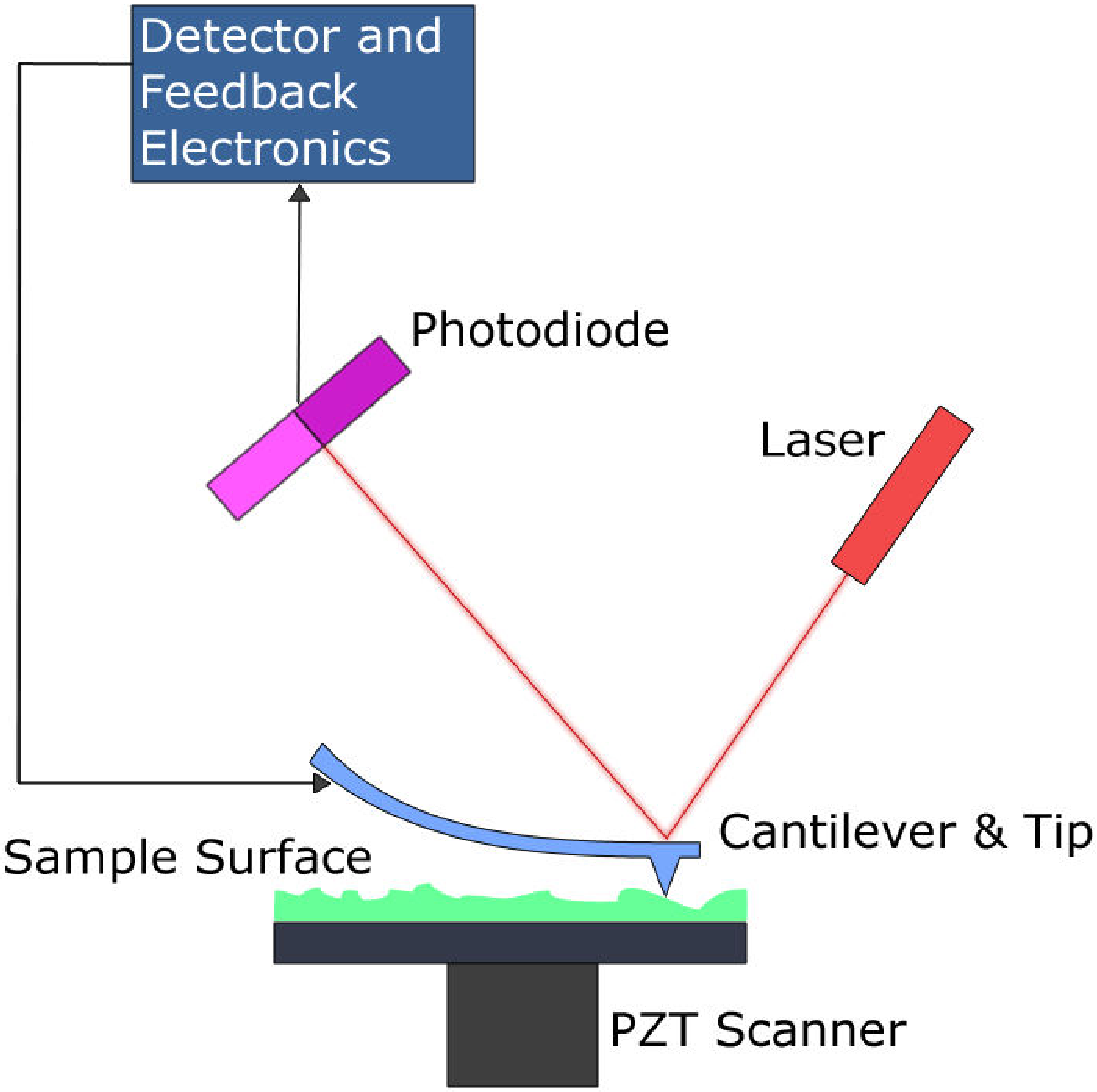
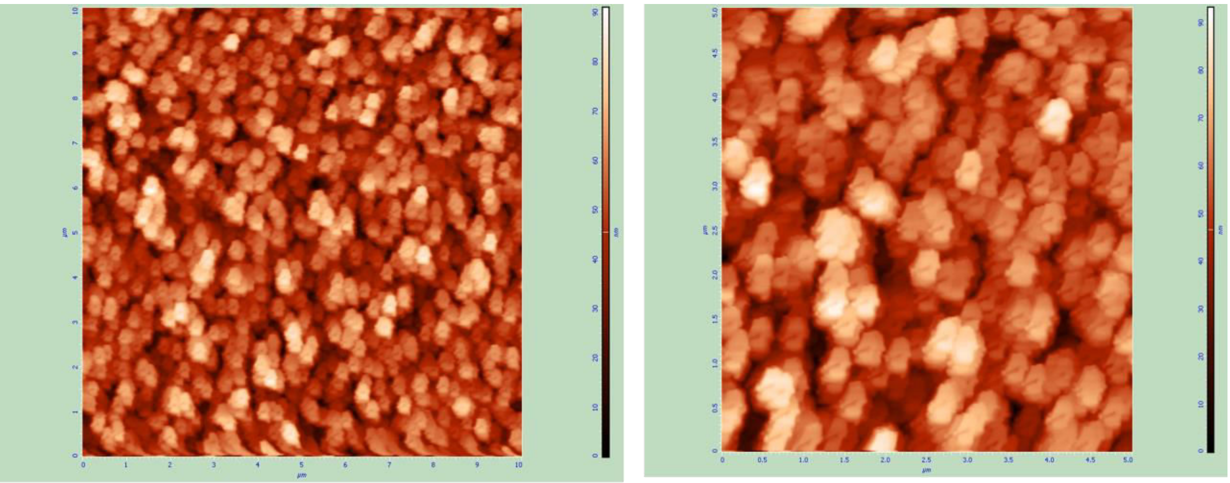


Fig 3.7: Schematic of AFM. (Source Ref[7])

Atomic Force Microscopes (AFMs) are devices that measure the surface topography of a sample on a nanometer/micrometer scale and turn those measurements into an image. The basic principle behind the AFM is based on the interaction between a probe (a sharp tip attached to a cantilever) and the atomic surface of the sample. The forces on the tip can be attractive or repulsive and cause the tip to deflect due to a change in these forces. This deflection is detected by the reflection of a laser beam shone on the back surface of the cantilever.



AFM image (10 micron) AFM image (5 micron)

Fig 3.8: AFM image of silica film

Fig 3.8 shows the AFM image of silica film at 10 micron scale (left) and at 5 micron scale (right). It is clear from the image that silica is in particle form. The size of the silica particle is not uniform. Fig 3.9 shows the histogram of silica particle size for 10 micron AFM image. From figure most of the silica particle has size between 50 nm to 60 nm. Fig 3.10 shows histogram of silica particle size for 5 micron image. From figure again most of the silica particle has size between 50 nm to 60 nm. Histogram for both 10 microm AFM image and 5 micron AFM image shows same particle size behavior, hence the data is consistent for both image.

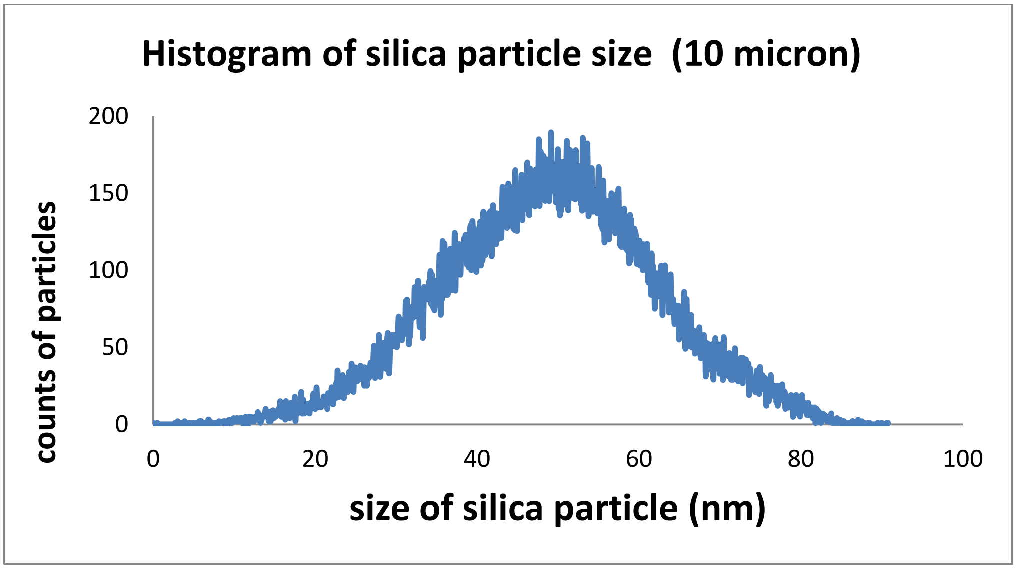


Fig 3.9: Histogram of silica particle size for 10 micron image (left in fig 3.8)

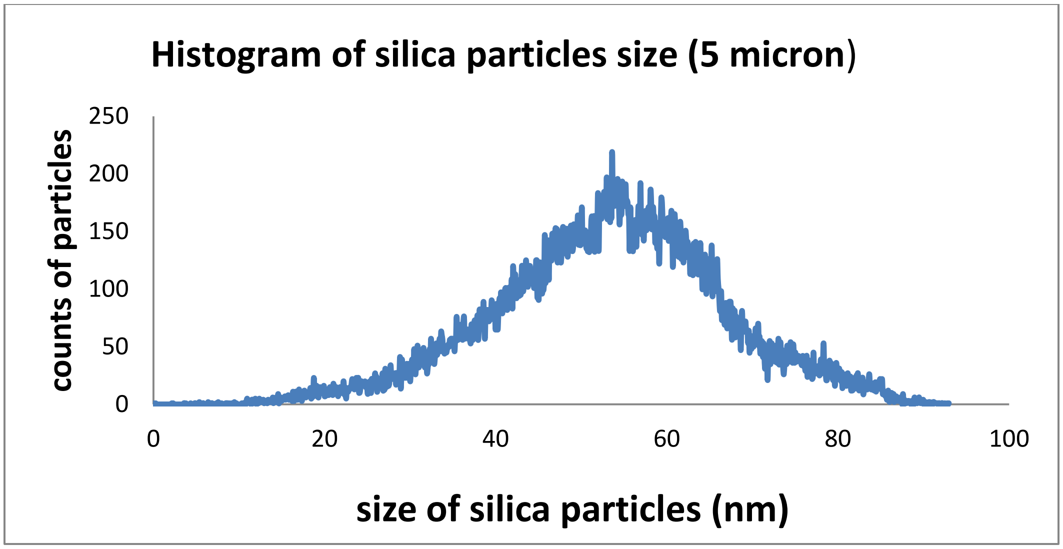


Fig 3.10: Histogram of silica particle size for 5 micron image (right in fig 3.8)

3.4 Preparing grating on silica film

Till now I studied the behaviour of silica film on glass. I deposited silica film on glass and studied optical characteristics and surface morphology using FESEM and AFM method. All my work is dedicated to enhance the light extracted from the glass. First I deposited silica film on glass and noticed enhancement in light extracted from the glass. Now I want prepare micro grating on the silica film and study the characteristics of the micro grating on silica film. For preparing micro grating on silica film first I have to prepare PDMS stamp and then using nanoimprint lithography prepare the grating on the silica film.

3.4.1 Preparing PDMS stamp

An elastomeric stamp is the key element of soft lithography. It is usually prepared by replica molding by casting the liquid pre-polymer of an elastomer over a master that has patterned relief structure on its surface.

PDMS ia an optically transparent material. It is cured by an organometallic crosslinking reaction. It has ability to reproduce surface features. The polymer is cured in contact with the pits on a CD or DVD or with optical transform slides. To know the perfection of the original feature copied we use diffraction of laser beam. The PDMS is flexible in nature so the surface features imprinted into the elastomer can also be distorted mechanically. The changing spacings can be monitored by diffraction.

MATERIALS:

Sylgard 184 elastomer (consists of pre-polymer and curing agent)

Dessicator

An oven capable of reaching 70 degee celcius.

Master that has patterned relief structure (CD/DVD).

Plastic cup, plastic fork, sharp scalpel, petri dish.

METHOD:

Dispensing the viscous liquid can be messy. Cover the work surface and the balance with aluminum foil. Take 10 g of Sylgard polymer base in a large weighing boat using plastic spoon. Now add 1 g of curing agent using a dropper.

Now mix the PDMS component thoroughly. It will create bubbles in mixture.

The optical qualities of cured PDMS degrade due to bubbles. Hence before curing bubbles should be removed. The mixture is kept in the dessicator to remove the bubbles.

Optical Transform Slide used as a lithography master

An optical transform film has emulsion side (side containing raised arrays). The smooth surface of the film will sharply reflect room lights. Whereas the emulsion side of film will give more diffuse reflection. Peel open the white plastic slide mount, pick up the film by its edges, and place the film in a weighing boat with the emulsion side facing upward.

Slowly pour the uncured PDMS mix on the film placed into mold assembly.

Place the mold into the oven at 130°C for 20 min or 90°C for 30 min. Now remove it from the oven and leave it to cool. Now gently remove film and the plastic sheet.

Recordable CD used as a lithography master

Small feature pattern can be made using CD-R disk. Using scissors cut out small section.

Using sharp object peel off the aluminium foil. We can use either the polycarbonate support or the foil as the lithography master. Pour the mixture on to the film in a petri dish. Place the petri dish into the oven at 130°C for 20 min or 90°C for 30 min. Now remove the petri dish from the oven and leave it to cool. Now gently take out the foil from the cured PDMS.

Recordable DVD used as a lithography master

Small feature pattern can also be made using DVD-R disk. Using sharp object peel off the two polymer layers. We can use either the polycarbonate support or the foil as the lithography master. Pour the mixture on to the foil in a petri dish. Place the petri dish into the oven at 130°C for 20 min or 90°C for 30 min. Now remove the petri dish from the oven and leave it to cool. Now gently take out the foil from the cured PDMS.

3.4.2 Nanoimprint lithography

Nanoimprint lithography (NIL) technology is based on the hot embossing lithography, using patterned mold (metal pattern) as a master mask. As nanoimprint lithography is capable of transferring microstructures with low-cost, its application to semiconductors, storage devices, and optical devices is expected. Toppan is developing various types of master molds, utilizing its minute fabrication technologies developed for photomasks and compact disc stampers.

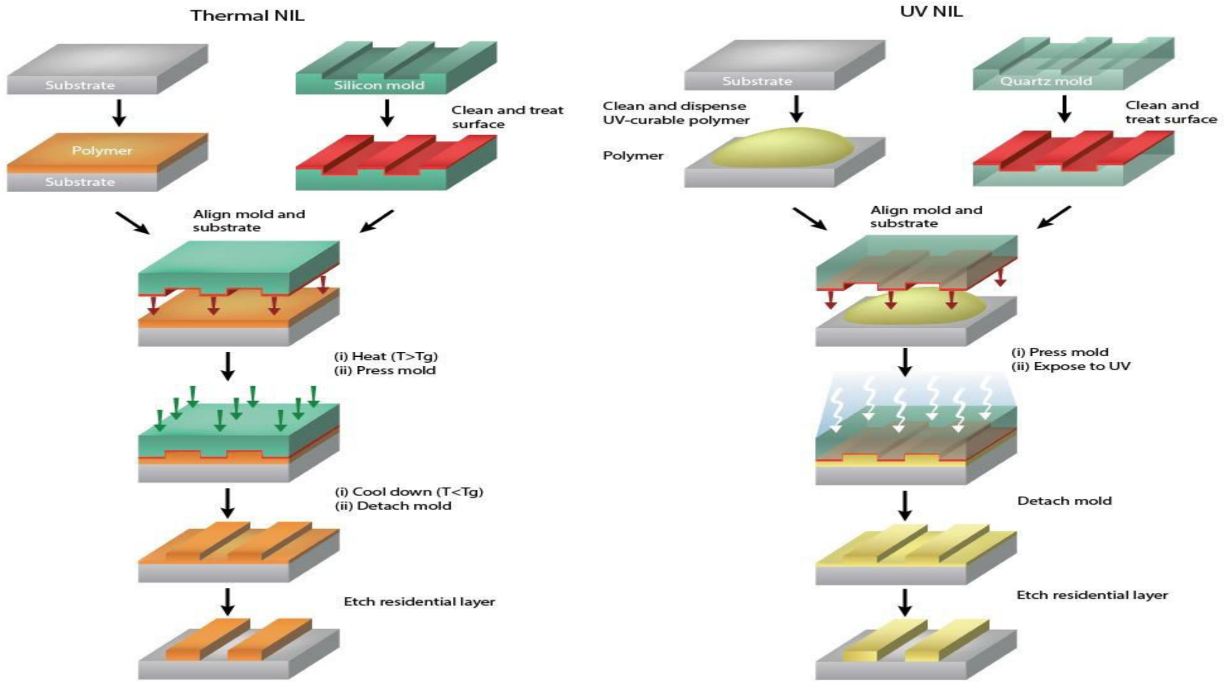


Fig 3.11: Nanoimprint lithography process.(Source Ref. [8])

Nanoimprint lithography provides wide range of linewidth resolutions. The line width resolution can range from several hundred micron to about 5 nm. Nanoimprint lithography can proceed either a thermal or UV curing protocol, depending on the type of polymer used.

When the polymer is thermoplastic, hot embossing or thermal nanoimprint lithography is used. The procedure is follows:

1. A mold with inverse structures is cleaned before treated in oxygen plasma. The surface is then further treated with an anti-stiction laye to facilitate detachment of the mold. Depending on the pattern complexity, the mold can be made of silicon, quartz or nickel, and may also be available commercially.
2. A substrate is coated with a layer of polymer to be imprinted.
3. The mold is aligned to be directly on top of the polymer-coated substrate.
4. Both the mold and the polymer-coated substrate are heated to a temperature above T_g of the polymer, where the polymer is fluid and amenable to the mold.
5. Imprinting is performed by pressing the mold onto the polymer film at the elevated temperature.
6. After the mold cavities are filled with a molten polymer, the temperature is lowered to below Tg of the polymer and the mold is detached.
7. The residual layer is etched away to leave behind the micro- or nanostructures of interest.

. UV NIL works only for UV-curable polymers or resists. A typical procedure is as follow:

1. The surface of the mold is cleaned and treated with an anti-stiction layer to facilitate mold detachment. Here the mold must be transparent to UV, for example quartz.
2. A prepolymer mixture of UV-curable polymer, which is less viscous, is dispensed onto substrate.
3. The mold is aligned directly on top of the prepolymer mixture.
4. The mold and substrate are passed together to fill the cavities of the mold with the prepolymer mixture.
5. The polymer is exposed to UV to cure and solidify.
6. After curing, the mold is detached from the substrate.

7. The residual layer is etched away to leave behind the micro- or nanostructures of interest.

Each process has its own advantages. UV-NIL can be performed at room temperature and at low pressure since the UV-curable polymer solution is already fluid and less viscous. Thermal NIL has less restrictions on the mold, where it can be non-transparent, and thus has lower production costs.

3.4.3 Characterization

3.4.3.1 UV spectrophotometry

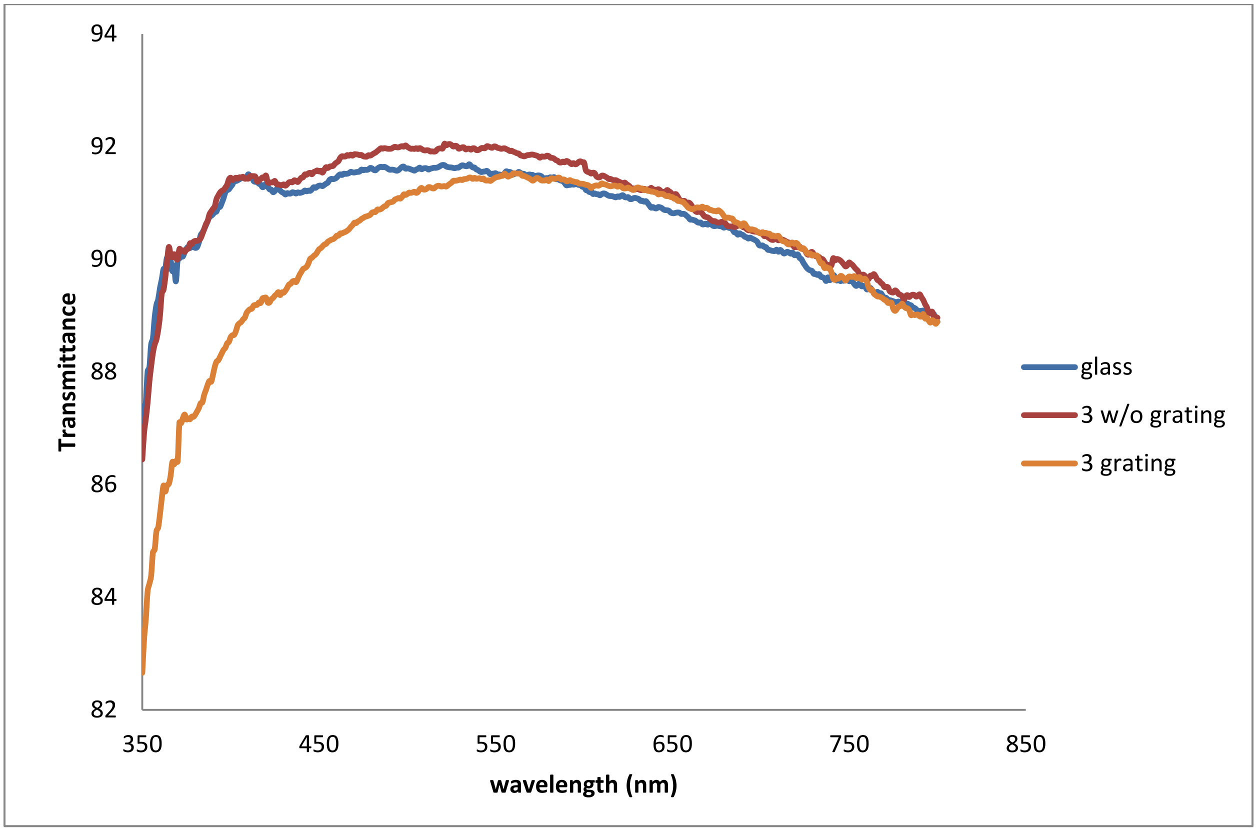


Fig 3.12: Transmittance of plain glass(blue), silica on glass(red) and grating on silica(yellow)

Transmittance is measured using UV spectrophotometer. In fig 3.11 there are three curves. These three curves are transmittance of plain glass(blue), silica film on glass(red), and gratings on silica film(yellow). As it is clear from the graph transmittance of silica layer on glass is more than that of plain glass for all wavelength. Gratings on silica film does not show increase in transmittance.

3.4.3.2 AFM and FESEM

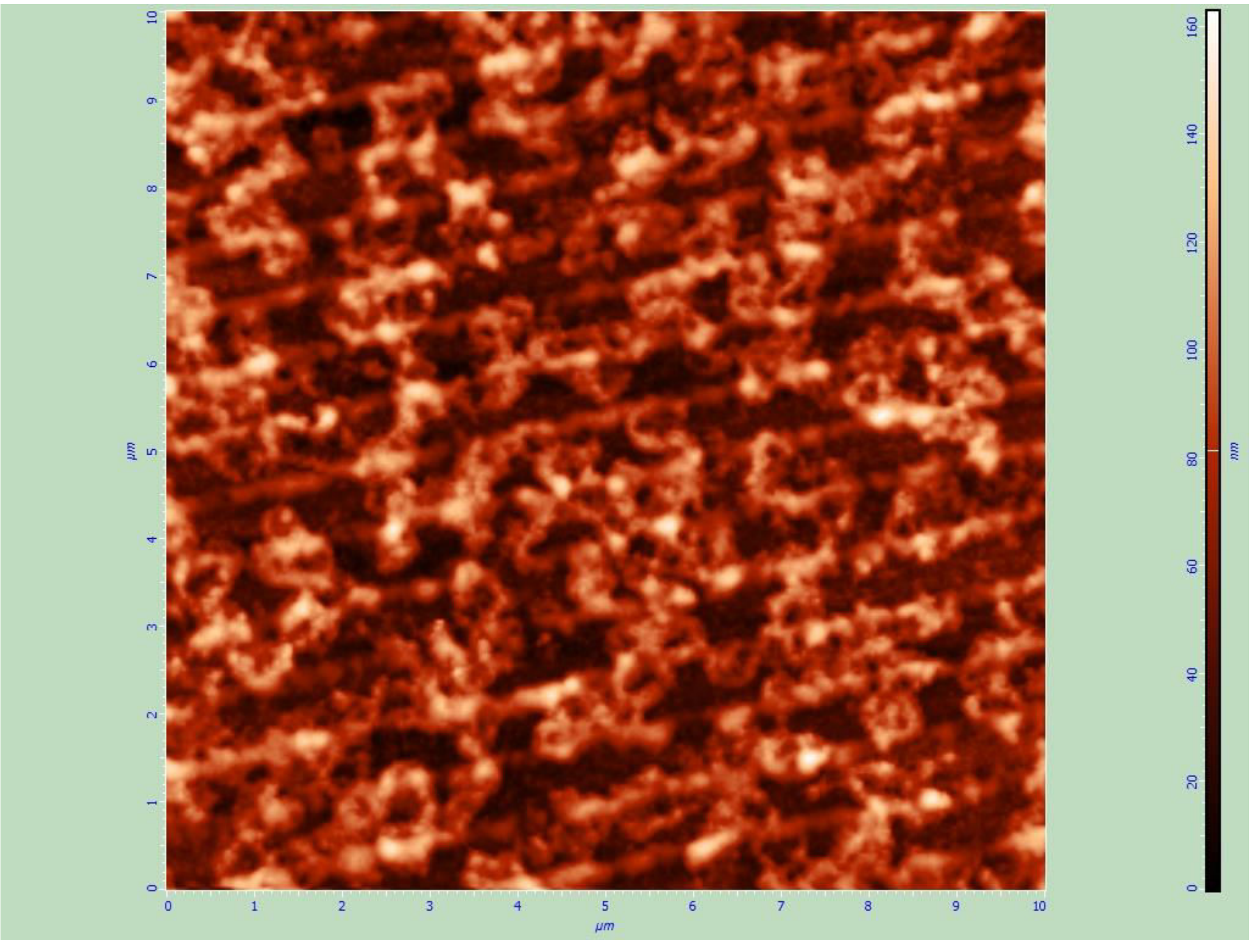
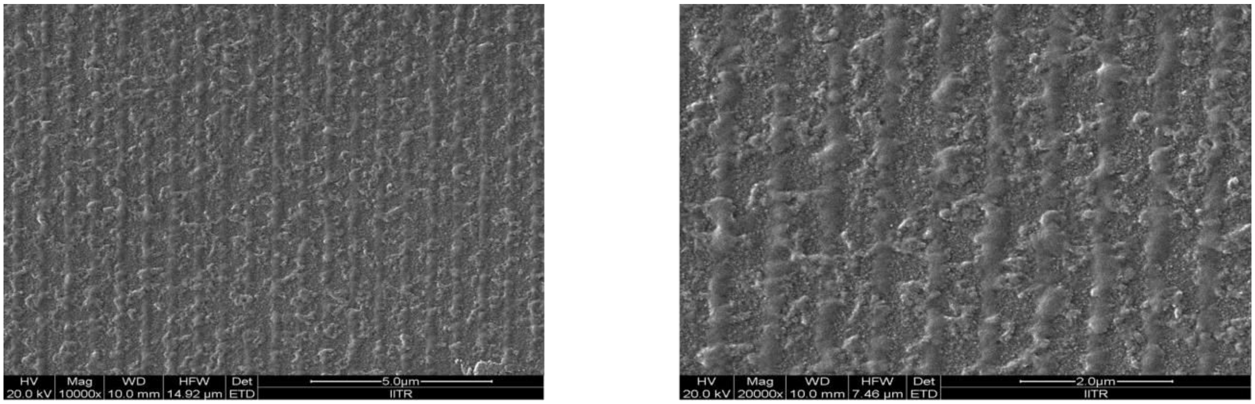


Fig 3.13: AFM surface image of gratings on silica film

Fig 3.13 shows the AFM surface image of grating on silica film. Fig 3.14 shows FESEM image of gratings on silica film. FESEM image 1 (left) is taken for 5 micron scale. FESEM image 2 (right) is taken for 2 micron scale. From FESEM we can notice the pitch of the gratings. The track pitch for the blank DVD is 0.78 micron. Hence the pitch of gratings is also around 0.78 micron.



FESEM image 1(5 micron)

FESEM image 2(2 micron)

Fig 3.14: FESEM surface images of grating on silica film

Chapter 4

Conclusion and future work

Scientists are employing many ideas to enhance the light extraction efficiency in LEDs/OLEDs. To enhance the light extraction efficiency in OLEDs I introduced microstructured silica layer between glass and ITO. To prepare microstructured silica I used sol-gel method. Then I deposited silica particle on plain glass using spin coating method. Then I measured transmittance of silica film on glass. It showed increase in transmittance of light on silica film on glass compared to transmittance of plain glass. The transmittance of plain glass comes up to 92 % at wavelength in between 550-650 nm. In the same range of wavelength the transmittance of silica film on glass comes around 94 %. We have to optimize the shape and size of silica particle so that there is more transmittance through silica film on glass. Hence there will be enhancement in light extraction efficiency.

Next I made gratings on silica film. To make gratings on silica film first I prepared PDMS stamp and then embossed the gratings on silica film using nanoimprint lithography. To prepare PDMS stamp I used DVD foil. The track pitch of DVD foil is 0.78 micron. So there is work left to find adequate methods to make gratings of different pitch. The PDMS stamp is flexible in nature so the pitch can be changed by stretching the PDMS. Nanoimprint lithography does not produce effective grating patterns on silica film. So the research should be done to find effective lithography methods to make gratings pattern.

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