Surface Engineering of Nanomaterials

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Lecture - 28
Evaporation Processes

Hi. In this particular lecture we are going to discuss about the Evaporation Process for generation of the thin films on to the substrate. In our last lecture we have discussed about the different types of sputtering techniques, why we are doing the sputtering, what is the physics behind the sputtering and what is the method are useful for different coating materials, and different target materials. So, in this particular topic we are going to discuss about that different types of evaporation process by which we can evaporate that target materials and directly it can deposit on to the substrate material.

So, before going to start first let us know that what is evaporations.

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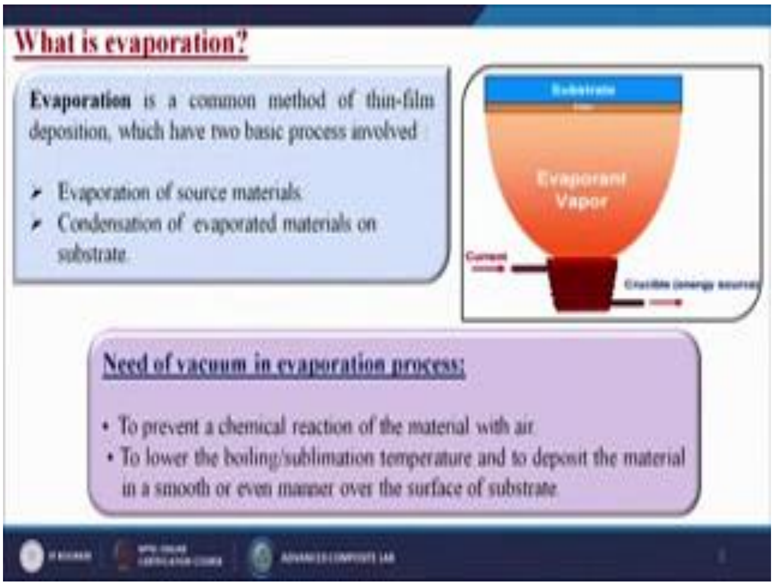

What is evaporation?

Evaporation is a common method of thin-film deposition, which have two basic process involved :

- Evaporation of source materials
- Condensation of evaporated materials on substrate

Need of vacuum in evaporation process:

- To prevent a chemical reaction of the material with air
- To lower the boiling/sublimation temperature and to deposit the material in a smooth or even manner over the surface of substrate

So, evaporations is a common method of thin film deposition which are two basic process involved: first one is called the evaporations of the source materials, whatever the target materials we are using or rather whatever the materials we are going to coat onto a substrate material. And the second one is that condensation of evaporated materials on the substrate. So, first we are agitating the target materials so that ions or maybe the molecules can comes from the target materials and how they are going to do the nucleation's or maybe the growth onto the substrate materials. So, these are the two main aspects for the evaporation techniques.

So, need of the vacuum in evaporation process generally we are doing this process in to some vacuum: to prevent the chemical reactions of the material with air, to lower the boiling or sublimation temperature and to deposit the material in a smooth or even manner over the surface of the substrate. Yes of course, because when we are generating the vapor that vapor can absorb the oxygen's or maybe the moisture so that the coating will not be proper. Not only that, it can increase the temperature in that particular material. So, just to reduce these things we are doing this experiment into some closed chambers or maybe into the vacuum chamber.

So, right hand side figure here we are giving a simple example where we are using the crucible, we are due to the current applied energy, we are heating that materials and that materials is generating certain kind of vapor and that vapor is directly depositing onto the substrate materials and it is forming a film on top of that.

First we are going to discuss about the vacuum evaporations, because as I already discussed in my last slide that generally we are doing this evaporation process into some vacuum environment. So, that is why it is known as the vacuum evaporations. So, what are the physics behind it?

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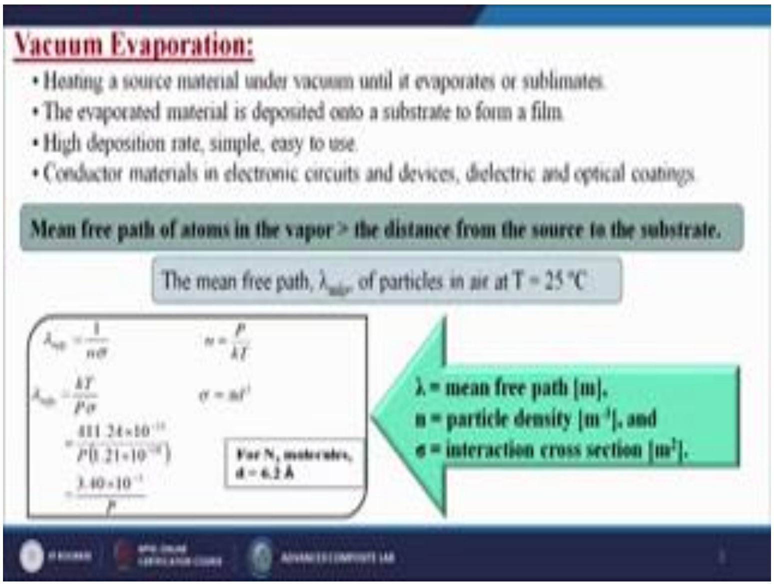

Vacuum Evaporation:

- Heating a source material under vacuum until it evaporates or sublimates
- The evaporated material is deposited onto a substrate to form a film
- High deposition rate, simple, easy to use
- Conductor materials in electronic circuits and devices, dielectric and optical coatings

Mean free path of atoms in the vapor > the distance from the source to the substrate.

The mean free path, λ_{air}, of particles in air at T = 25 °C

$$\lambda_{air} = \frac{1}{n\sigma} \qquad n = \frac{P}{kT}$$

$$\lambda_{air} = \frac{kT}{P\sigma} \qquad \sigma = \pi d^2$$

$$= \frac{411.24\times10^{-23}}{P(1.21\times10^{-18})}$$

$$= \frac{3.40\times10^{-3}}{P}$$

For N_2 molecules, d = 6.2 Å

λ = mean free path [m],
n = particle density [m^{-3}], and
σ = interaction cross section [m^2].

Heating a source material under vacuum until it evaporates or sublimates, the evaporated materials is deposited onto a substrate to form a film, high deposition rate simple and easy to use, conductor materials in electronic circuits and devices dielectric and optical coatings can be done by these vacuum evaporations.

So now, the thing is that I am having the target materials, I am giving the heating on to that target materials so that it can vaporized, right; so when this will from some vaporize some atoms or maybe the molecules which can these molecules or atoms can fly up to certain distance. If that distance will not be covered it will not properly deposited onto the material substrate. So, based on that generally we are calculating the mean free path of atoms in the vapor should be more than the distance from the source to the substrate because, suppose from I am standing over here I am the target material I want to reach up to the target, my target maybe 1 feet from my standing positions, but if my mean free path will be less than that so what will happen I will not directly touch with the substrate materials. So, my mean path will be more than the distance in between the substrate and to the target then only I can go and I can directly heat that substrate and I can deposit on to the substrate material.

So, the mean free path generally we are calling it as a lambda m f p of particles in air at t is equal to 25 degree centigrade. So, here we have given one examples where we are using the nitrogen molecules and the diameter is 6.2 angstrom. So, we are using the mean free path the formula is 1 n by sigma; where n is equal to capital P by small k and capital T. So, lambda is mean free path in meter, n is the particle density per meter cube and sigma is the interaction cross sections per meter square. So, by calculating these equations we can get that what is the mean free path so that my nitrogen molecule can directly come and deposit onto my substrate.

Next we are going to discuss about the evaporation rate and the vapor pressure, because now the thing is that I am agitating that material but how much energy I have to put onto that material so that it can really certain kind of atoms or molecules. So, that depends upon the film thickness that depends upon the time actually for doing the coating onto that material.

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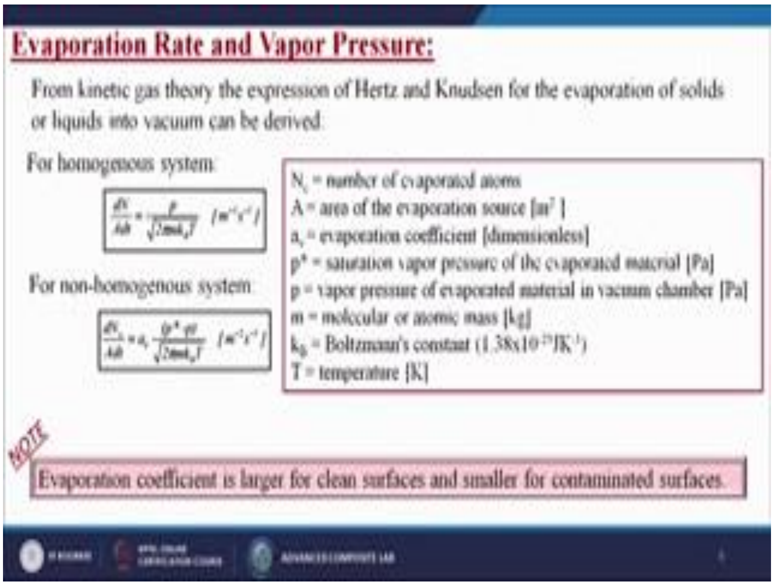

Evaporation Rate and Vapor Pressure:

From kinetic gas theory the expression of Hertz and Knudsen for the evaporation of solids or liquids into vacuum can be derived:

For homogenous system:

$$\frac{dN_e}{A\,dt} = \frac{p}{\sqrt{2\pi m k_B T}} \quad [m^{-2}s^{-1}]$$

For non-homogenous system:

$$\frac{dN_e}{A\,dt} = a_v \frac{(p^* - p)}{\sqrt{2\pi m k_B T}} \quad [m^{-2}s^{-1}]$$

N_e = number of evaporated atoms
A = area of the evaporation source [m^2]
a_v = evaporation coefficient [dimensionless]
p^* = saturation vapor pressure of the evaporated material [Pa]
p = vapor pressure of evaporated material in vacuum chamber [Pa]
m = molecular or atomic mass [kg]
k_B = Boltzmann's constant ($1.38 \times 10^{-23} JK^{-1}$)
T = temperature [K]

NOTE

Evaporation coefficient is larger for clean surfaces and smaller for contaminated surfaces.

So, just to know the evaporation rate and how much vapor pressure we have to follow certain gas theory who in expressions which has been invented by two scientists named Hertz and Knudsen for the evaporation of solids or liquids into vacuum can be derived. So, before going to say this one I can say that we can take two types of sources; either I can take the solid sources which directly I will give the heat it will form into the liquid then it will generate the vapor or maybe I can take the liquid samples directly from the liquid it can generate the vapor.

So, for these two systems; one is called the homogeneous systems and for non-homogeneous systems they have derived two different equations. So, for homogeneous systems they have derived d n by the a d t is equal to p by root over 2 pi m k b capital T into m to the power minus 2 and h to the power minus 1. And for non-homogeneous systems rest of the things is same only they have multiplied it by the a v and it is p prime minus the p. So here, n e is nothing but the number of the evaporated atoms, a is the area of the evaporation source in meter square, a v is the evaporation coefficient it is dimensionless, p prime saturation vapor pressure of the evaporated materials that is in Pascal, small p vapor pressure of evaporated material in vacuum chamber that is Pascal, m is the molecular or atomic mass in kg, k b is nothing but the Boltzmann constant that is1.38 into 10 to the power minus 23 joule per Kelvin, and t is the temperature in the Kelvin.

So, if we know all these value easily we can calculate that what is the evaporation rate and vapor pressure for the homogeneous medium and for the non-homogeneous medium. So here, one is the vital point we have to keep in our mind is that the evaporation coefficient is larger, for the clean surface and smaller to the contaminated surface. Of course, because the clean surface means easily that it can heat the target materials, it can evaporate, and it can form the vapor; but if we are having certain kind of contaminants or maybe some kind of impurities so that the heat not directly reach to the target material so that the evaporation rate will be slower.

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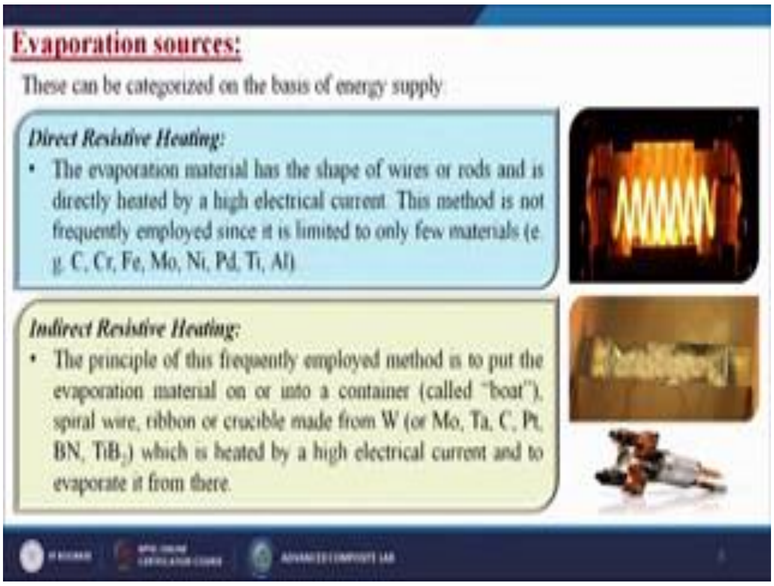

Evaporation sources:

These can be categorized on the basis of energy supply

Direct Resistive Heating:

- The evaporation material has the shape of wires or rods and is directly heated by a high electrical current. This method is not frequently employed since it is limited to only few materials (e.g. C, Cr, Fe, Mo, Ni, Pd, Ti, Al)

Indirect Resistive Heating:

- The principle of this frequently employed method is to put the evaporation material on or into a container (called "boat"), spiral wire, ribbon or crucible made from W (or Mo, Ta, C, Pt, BN, TiB_2) which is heated by a high electrical current and to evaporate it from there.

Next, depending upon the evaporation sources also we can divide it into several parts. So, there can be categorized on the basis of energy supplies; that means, what type of heat we are going to use or rather what type of source of heat we are going to use to agitate that target materials. So, first one is called the direct resistive heating. In that particular case the evaporation materials has the shape of wires or rods and is directly heated by a high electrical current. Here, you can see the structure of this source like a filament types of source, it is made of some different materials like carbon chromium, maybe iron, molybdenum, nickel, palladium, titanium, and aluminum. It is in the spiral into that structure directly we are putting the electric current so that it can be heated up and it can generate the heat energy.

Next one is called the indirect resistive heating. The principle of this frequently employed method is put the evaporation material on or into a container is nothing but a crucible generally in our technical term we are calling it as a boat that boat maybe spiral wire ribbon or crucible made from tungsten or sometimes we can use the molybdenum, tantalum, carbon, platinum, boron nitride, titanium boride, which is heated by a high electrical current and to evaporate it from there. So, either the source can directly come or maybe that source can directly come into the boat and then it can heat the material. So that is why it is called the in directive resistive heating.

Next one is called the inductive heating. So, by the induction methods we are generating the heat energy which heat energy we are using to heat the target material.

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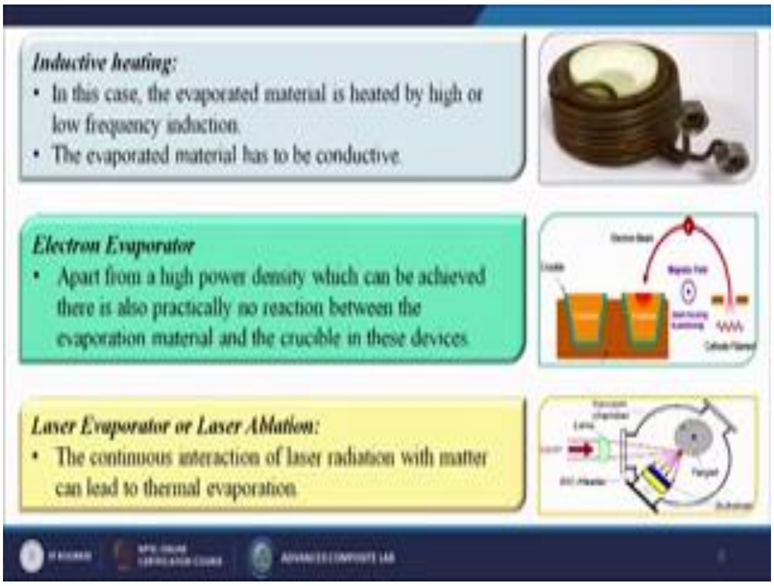

Inductive heating:

- In this case, the evaporated material is heated by high or low frequency induction.
- The evaporated material has to be conductive.

Electron Evaporator

- Apart from a high power density which can be achieved there is also practically no reaction between the evaporation material and the crucible in these devices.

Laser Evaporator or Laser Ablation:

- The continuous interaction of laser radiation with matter can lead to thermal evaporation.

So in this case the evaporated materials are heated by high or low frequency inductions. The evaporated material has to be conductive.

Next we can generally use the electron beam evaporator. Apart from a high power density which can be achieved there is also electrically no reaction between the evaporation material and the crucible in these devices. If we remember in our last presentations I have discussed about the magnetron sputtering, in where we have used the magnetic as well as the electrical energy to generate the sputtering techniques. So here also we are doing the same thing, here we are having that electron and then we are generating the magnetic field in this particular case so that it can direct the electron beam to directly fall upon the evaporate materials and the evaporate materials will be heated up and it will generate the vapor.

And last one is called a laser evaporator or the laser ablations. From the name itself we can understand that here we are using the laser energy to heat our source material. The continuous interaction of laser radiations with matter can lead to thermal evaporations. So here, we are using the laser, the laser is directly heating the target, then from the target some ions or molecules is directly coming and it is depositing onto the material or maybe the substrate.

Now, we are going to discuss why evaporation techniques are preferred many times, what is the logic behind it? Because nowadays we can heard that people are using these kinds of techniques for several applications for numerous applications this is the easiest method then this is the very inexpensive methods and not only that it can be done very quickly.

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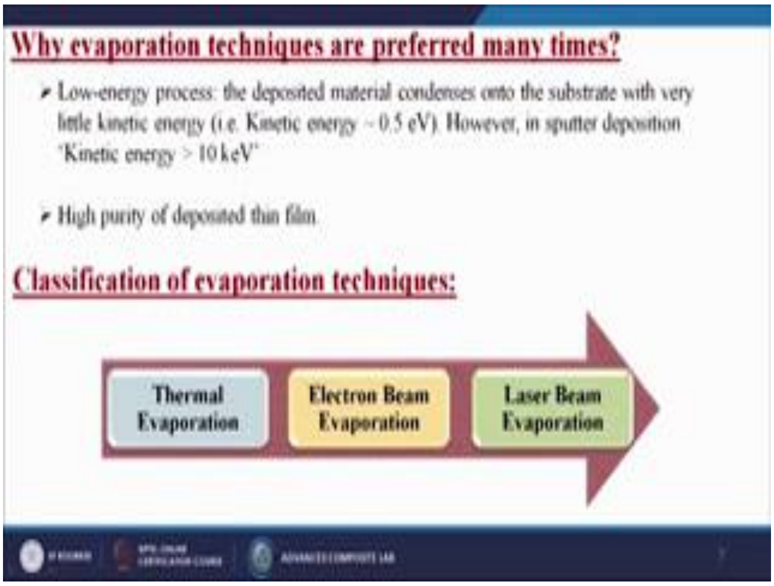

Why evaporation techniques are preferred many times?

- Low-energy process: the deposited material condenses onto the substrate with very little kinetic energy (i.e. Kinetic energy ~ 0.5 eV). However, in sputter deposition 'Kinetic energy > 10 keV'
- High purity of deposited thin film.

Classification of evaporation techniques:

So, low energy process first of all the deposited material condenses onto the substrate with very little kinetic energy. Generally that kinetic energy varies around 0.5 electron volt. However, in sputter deposition kinetic energy more than 10 kilo electron volt; high purity of deposited thin film. Classification of evaporation techniques: generally thermal evaporations, electron beam evaporations, and the laser beam evaporations.

So here, generally the evaporation techniques can be done by these three techniques: First one is called the thermal evaporation techniques. So, in these particular process the transition mate target material from solid to gaseous state is carried by means of brick of inter atomic bonds between atoms on the surface itself.

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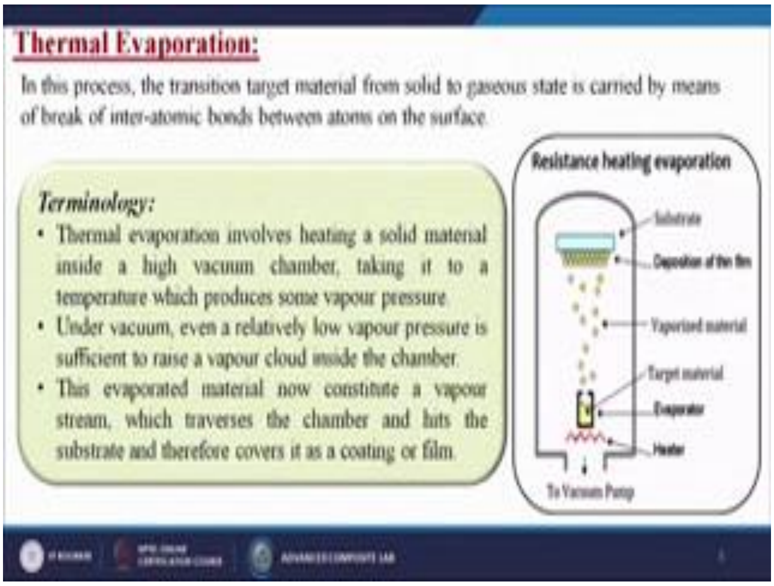

Thermal Evaporation:

In this process, the transition target material from solid to gaseous state is carried by means of break of inter-atomic bonds between atoms on the surface.

Terminology:

- Thermal evaporation involves heating a solid material inside a high vacuum chamber, taking it to a temperature which produces some vapour pressure.
- Under vacuum, even a relatively low vapour pressure is sufficient to raise a vapour cloud inside the chamber.
- This evaporated material now constitute a vapour stream, which traverses the chamber and hits the substrate and therefore covers it as a coating or film.

Here, from this particular figure you can understand that we are having certain kind of crucible type of things in which we are putting our target materials, then simply we are giving the heat by the heater and the whole process we are doing in to some vacuum pump, then it is evaporating that vapor is directly going and it is depositing on to the substrate by layer by layer techniques.

So, terminology is that; thermal evaporation involves heating a solid material inside high vacuum chamber taking it to temperature which produces some vapor pressure. Under vacuum even a relatively low vapor pressure is sufficient to raise the vapor cloud inside the chamber. This evaporated material now considers a vapor stream which transfers the chamber and hits the substrate, and therefore covered it as a coating of film. First we are generating the single single molecules then the all molecules will be form a cloud then when the pressure will be more then that will act as a stream, a stream of total vapor pressure vapor pressure can come and directly hit onto the substrate material.

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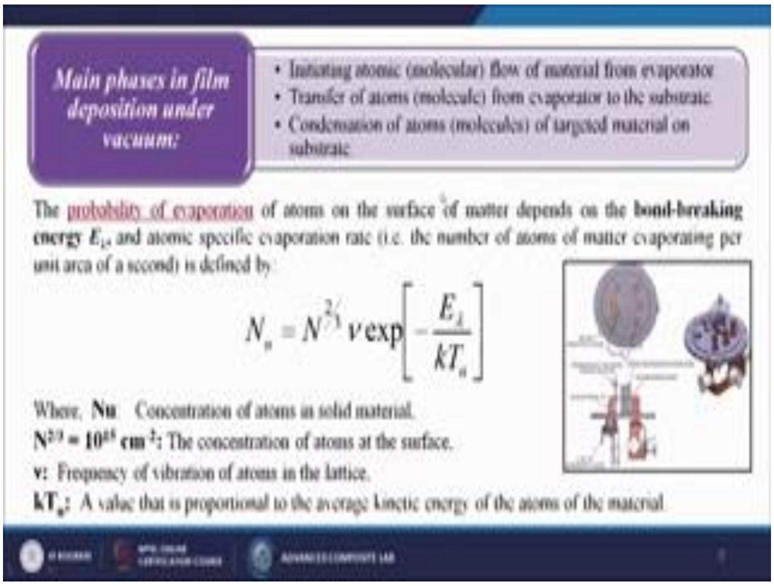

Main phases in film deposition under vacuum:

- Initiating atomic (molecular) flow of material from evaporator
- Transfer of atoms (molecule) from evaporator to the substrate
- Condensation of atoms (molecules) of targeted material on substrate

The probability of evaporation of atoms on the surface of matter depends on the **bond-breaking energy E_λ**, and atomic specific evaporation rate (i.e. the number of atoms of matter evaporating per unit area of a second) is defined by

$$N_u = N^{2/3} v \exp\left[-\frac{E_\lambda}{kT_u}\right]$$

Where, **Nu** Concentration of atoms in solid material.

$N^{2/3} = 10^{15}$ cm^{-2}: The concentration of atoms at the surface.

v: Frequency of vibration of atoms in the lattice.

kT_u: A value that is proportional to the average kinetic energy of the atoms of the material

A main phase in film deposition under vacuum is that: initiating atomic or maybe the molecular flow of materials from evaporator, transfer of atoms molecule from evaporator to substrate, conditions of atoms of the target material on the substrate. So here are the three; first one that you have to generate that molecule, then second one is that you have to take that molecule from target to the subs substrate, and third one is the deposition of these molecules on to the substrate surface.

Next, the probability of evaporations of atoms on the surface of matter depends on the bond breaking energy E lambda and atomic specific evaporation rate that is the number of atoms of metal evaporating per unit area of second is defined; N u is equal to N to the power two third v exponentially minus E lambda by k T u. Where, N u is the concentration of atoms in solid materials, N two third is equal to 10 to the power 15 centimeter per centimeter square the concentration of atoms at the surface, v is equal to frequency of vibration of atoms in the lattice, k T u is nothing but the value that is proportional to the average kinetic energy of the atoms of the material.

Simple thing is that; suppose I am holding a material, so some source heat is coming and then it is trying to taking these molecules or maybe atoms from my hand itself, so whatever the source it is coming is energy should be more than the energy by which I am holding these materials. So, if that energy will be more then only it is possible to take these molecules from my hand itself. So, that is known as the bond breaking energy, because each and every molecules is attached by some kind of bonds or maybe some kind of force generally we are calling it is a Van der Waals force. So, whatever the energy I am applying to release the molecules that energy should be higher than the bonding energy or maybe the more Van der Waals force of that particular target material so that the ions or maybe the molecules can release from particular target material.

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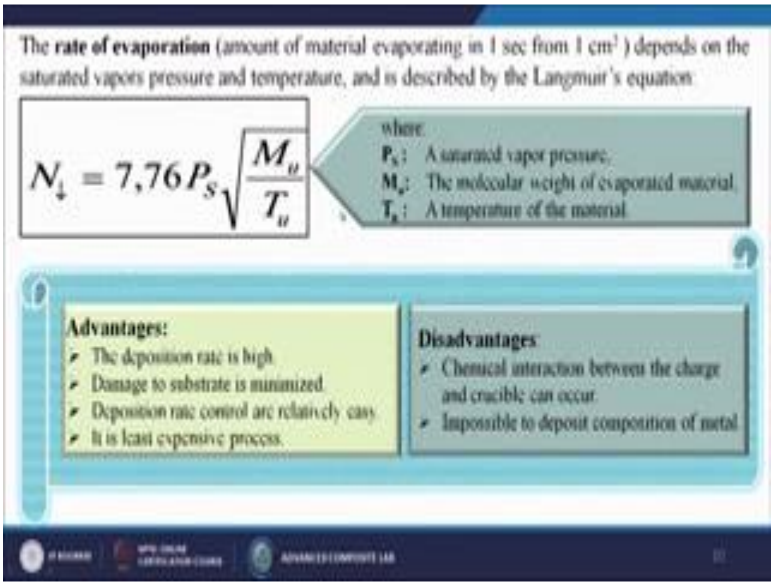

The **rate of evaporation** (amount of material evaporating in 1 sec from 1 cm^2) depends on the saturated vapors pressure and temperature, and is described by the Langmuir's equation:

$$N_{\downarrow} = 7{,}76 P_S \sqrt{\frac{M_u}{T_u}}$$

where:

P_s : A saturated vapor pressure;

M_u: The molecular weight of evaporated material

T_u : A temperature of the material

Advantages:

- The deposition rate is high
- Damage to substrate is minimized
- Deposition rate control are relatively easy
- It is least expensive process.

Disadvantages:

- Chemical interaction between the charge and crucible can occur.
- Impossible to deposit composition of metal

The rate of evaporations, amount of material evaporation in 1 second from 1 centimeter square depends on the saturated vapor pressure and temperature and is described by the Langmuir's equations that is nothing but n is equal to 776 P s root over M u by T u. Where, P s a saturated vapor pressure, M u the molecular weight of evaporated material, T u a temperature of the material

So, what are the advantages for this particular process? The first one is the deposition rate is too high, damage of substrate is minimized, deposition rate control are relatively easy, it depends upon how much energy you are going to put so that the vaporization speed rather vaporization amount it can be controllable and it is least expensive process. What are the disadvantages? Chemical interaction between the charge and the crucible can occur impossible to deposit composition of metals. So, these all are the disadvantages for these particular techniques.

Next one we are going to discuss about the electron beam evaporation process. From the name itself we can understand that we are using certain kind of electron beam or maybe the source of electron to heat the target materials by which it can generate the vapor and that vapor can be deposited onto the substrate.

So, what are the procedures to generate an electron beam?

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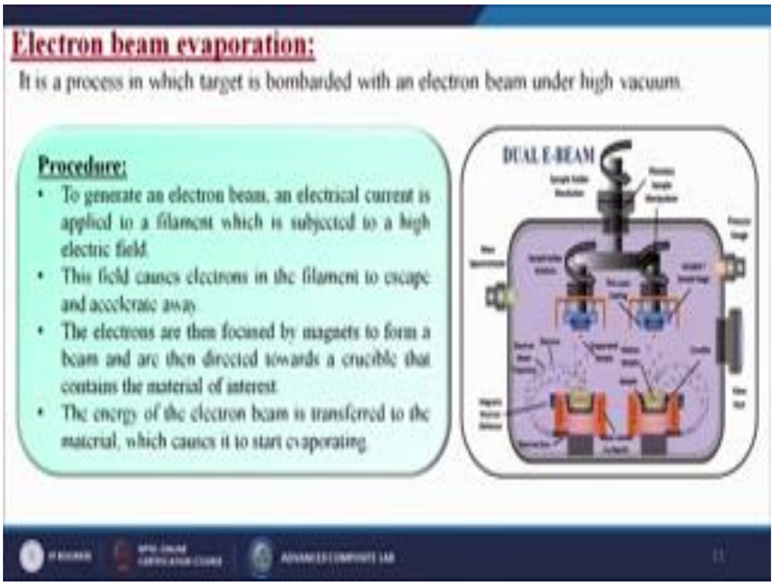

Electron beam evaporation:

It is a process in which target is bombarded with an electron beam under high vacuum.

Procedure:

- To generate an electron beam, an electrical current is applied to a filament which is subjected to a high electric field.
- This field causes electrons in the filament to escape and accelerate away.
- The electrons are then focused by magnets to form a beam and are then directed towards a crucible that contains the material of interest.
- The energy of the electron beam is transferred to the material, which causes it to start evaporating.

An electron current is applied to a filament which is subjected to a high electric field. The field causes electrons in the filament to escape and accelerate away. The electrons are then focused by magnets to form a beam and are then directed towards a crucible that contains a material of interest. The energy of the electron beam is transferred to the material which causes it to start evaporating. So, from this particular figure we can understand that we are having some kind of crucibles there we are generating the electron gun, then we are using the magnets, then through that magnets that electron beam is directly falling onto the crucible materials, it is making the vapor of that target material and then that vapor is directly going and it is depositing on to the substrate itself.

So, to generate the electron beam generally we are using an electric current to a filament which is subjected to a high electric field. So, we are generating the electron that electron through the magnetic field directly it is falling onto the crucible, it is heating up the target materials, vaporization is taking place that vapor is directly depositing on to the source material.

So, many metals such as aluminum will melt fast and then start evaporating well ceramics will sublimate. The thing is that it depends upon the melting temperature of that particular material. In that particular case aluminum melting temperature is low that is why it vaporized very fast.

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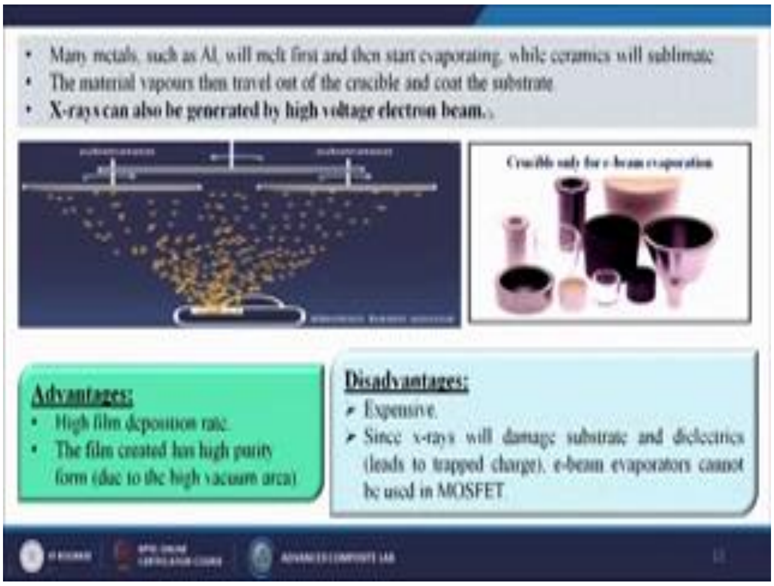

- Many metals, such as Al, will melt first and then start evaporating, while ceramics will sublimate
- The material vapours then travel out of the crucible and coat the substrate
- **X-rays can also be generated by high voltage electron beam.**

Crucible only for e-beam evaporation

Advantages:
- High film deposition rate.
- The film created has high purity form (due to the high vacuum area)

Disadvantages:
- Expensive.
- Since x-rays will damage substrate and dielectrics (leads to trapped charge), e-beam evaporators cannot be used in MOSFET

The material vapors then travel out to the crucible and coat the substrate. X-rays can also be generated by high voltage electron beam. So here, this is the electron beam source actually we are using it is targeting, and the beauty of these things is that if your source substrate material will be small you can go for two to three materials simultaneously. So, the coating can be done of these two to three materials at a time. Crucible only for e beam operations, generally the crucible is looking like this.

The advantage of this process is: high film deposition rate, the film created has high purity form due to the high vacuum area because we are using the high vacuum for this particular process. What are the disadvantages? First one it is the expensive process, because we have to first generate the electron, for generating the electron we needs some kind of materials filaments then it can be heated up and the maybe generally tungsten filament we are going to use so that is also the very very expensive one so that is why the whole process is too expensive. Since, X-rays will damage substrate and dielectric leads to trap charge e-beam evaporators cannot be used in the MOSFETs; some kind of electronics purpose we cannot use these kind of process.

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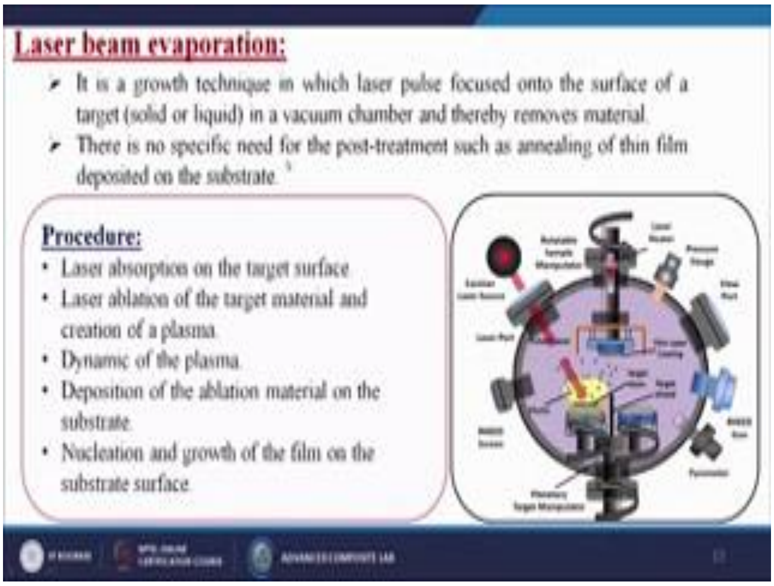

Laser beam evaporation:

- It is a growth technique in which laser pulse focused onto the surface of a target (solid or liquid) in a vacuum chamber and thereby removes material.
- There is no specific need for the post-treatment such as annealing of thin film deposited on the substrate.

Procedure:

- Laser absorption on the target surface
- Laser ablation of the target material and creation of a plasma
- Dynamic of the plasma
- Deposition of the ablation material on the substrate
- Nucleation and growth of the film on the substrate surface

Next one is called laser beam evaporations. So, it is a high growth technique in which laser pulse focused onto the surface of a target solid or liquid in a vacuum chamber and thereby removes the material is the simplest method. Simple I am having the target material, I am using the laser either the laser is the continuous laser or maybe the pulsed laser, that laser is directly falling and it is heating the target materials due to that the high temperature generation is taking place, and the vaporization is taking place and that vapor is directly going and depositing onto the substrate material.

What is the procedure? Laser absorption on the target surface: laser ablation of the target material and creation of plasma, dynamic of the plasma deposition of the ablation materials on the substrate, nucleation and growth of the film on the substrate surface. So here, you can see that the laser is coming directly heating on to the target materials, then target materials is vaporizing and then directly it is going on to the substrate material. And here this is the total experimental setup design where there is some viewpoint so that you can see that whether the target material is heating directly by the laser or not, whether the evaporation is taking place or not, whether the direct material is depositing on to the substrate materials or not.

And not only that, here we are having the parameter some kind of probe where we can see, where we can change our target materials. Suppose here this is the rotations so you can put one or more target materials at a time so that you can do the coatings of by layer by layer method on to the substrate itself.

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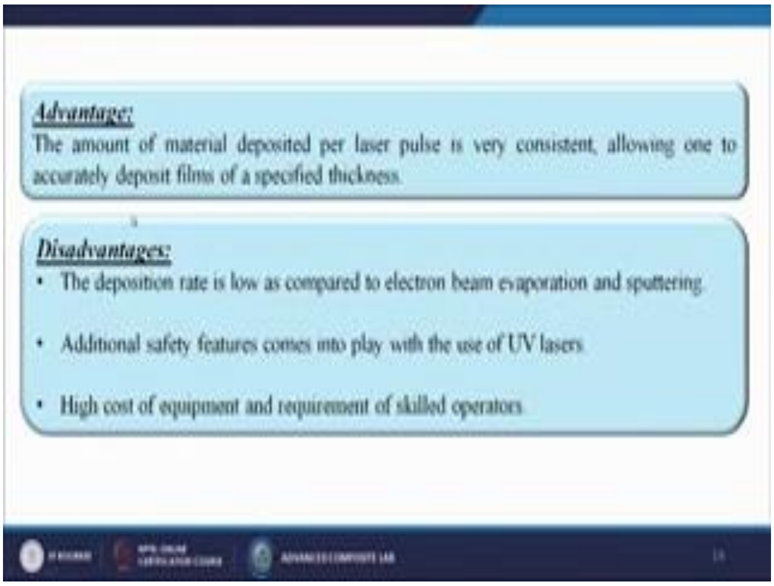

Advantage:

The amount of material deposited per laser pulse is very consistent, allowing one to accurately deposit films of a specified thickness.

Disadvantages:

- The deposition rate is low as compared to electron beam evaporation and sputtering
- Additional safety features comes into play with the use of UV lasers
- High cost of equipment and requirement of skilled operators

What is the advantage? The amount of material deposited per laser pulse is very consistent, allowing one to accurately deposit films of a specified thickness. So, generally we are using the pulsed lasers. Means one certain the laser will come then after certain time again the second laser will come so that easily we can give the specific deposition time for the particular target material.

Disadvantages: the deposition rate is low as compared to electron beam evaporations and sputtering, additional safety features comes into play with the use of UV lasers, high cost of equipment and requirement of the skilled operators. So, these all are the disadvantage for this particular case.

Now, we are going to summarize that evaporations for the sputtering, because last lecture I have given the brief description about the sputtering techniques and then in this particular lecture I am talking about the evaporation techniques.

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Evaporation vs. Sputtering:

	Material	*Deposition rate*	*Purity*	*Uniformity*	*Adhesion*	*Capital equipment*	*Thickness control*
Evaporation	Limited	~1000 atomic layer/sec	High	Difficult	Often poor	Low cost	Not easy to control
Sputtering	Almost unlimited	1 atomic layer/sec	Low	Easy over large areas	Excellent	Expensive	Control Possible

So what are the difference between the evaporation and sputtering techniques in terms of material? For evaporation the material is limited, but sputtering almost the unlimited. Deposition rate is generally more or less 1000 atomic layer per second, here the sputtering is 1 atomic layer per second. So, evaporation is much better in this particular case. Purity is very very high for the evaporation materials, but for sputtering it is low. Uniformity it is very very difficult to maintain for evaporation process, but easy over the large areas for the sputtering process; adhesions often poor for the evaporation process, but excellent in the case of sputtering. Capital equipment it is the low cost process for the evaporations sputtering it is expensive; and thickness control not easy controllable for the evaporation process, but for the sputtering it is well controllable.

So, now we have come to the last slide of this particular lecture where we are going to summarize the whole whatever I have discussed till now.

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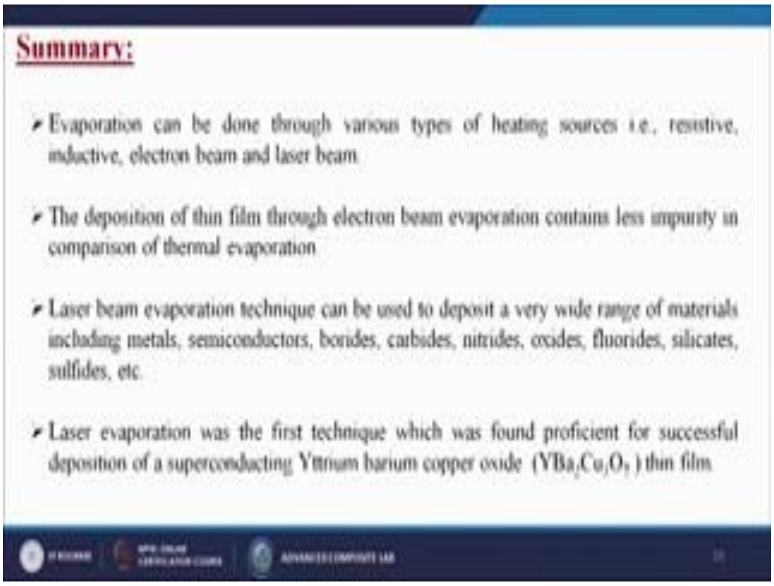

Summary:

- Evaporation can be done through various types of heating sources i.e., resistive, inductive, electron beam and laser beam.
- The deposition of thin film through electron beam evaporation contains less impurity in comparison of thermal evaporation
- Laser beam evaporation technique can be used to deposit a very wide range of materials including metals, semiconductors, borides, carbides, nitrides, oxides, fluorides, silicates, sulfides, etc.
- Laser evaporation was the first technique which was found proficient for successful deposition of a superconducting Yttrium barium copper oxide ($YBa_2Cu_3O_7$) thin film

So, evaporation can be done through various types of heating source like resistive, inductive, electron beam, and laser beam. Already we have discussed all this force into details. The deposition of thin films through electron beam evaporation contains less impurity in comparison of thermal evaporations. Laser beam evaporation techniques can be used to deposit a very wide range of materials including metals semiconductors borides, carbides, nitrides, oxide, fluoride, silicates, and sulfides because the laser the heat energy is more it can vapor any kind of target material easily.

Laser evaporation was the first technique which was found proficient for successful deposition of a superconducting yttrium, barium, copper, oxide, thin film. This is also one kind of very very wonderful fantastic materials, it is not possible by any other three methods like the resistive or maybe the inductive or maybe the electron beam only it is perceiving by the laser evaporation methods, because it needs high energy to evaporate.

Thank you.