Solar Photovoltaics: Fundamental Technology and Application

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Lecture - 39
Electron Microscopy in Solar Photovoltaics

Welcome everyone to our Solar Photovoltaics course. Today we have 8th week and 4th module. So in the last few lectures we have discussed about the vacuum technology. We have seen what is the need for a vacuum in a system and how we can create the vacuum and also we have seen how to measure the vacuum and if we have a leak in our system how to detect that leak.

Now I have said in the last lecture that in addition to this vacuum technology another important characterization tool which we will be using again and again as far as the solar cell is concerned is the advanced imaging technique. Now whenever we talk about the imaging we know about the optical microscopic based imaging things so that we have commonly used everyone has used that, but optical microscope has some resolution issues.

It has some issues in terms of the resolution which does not come from the instrument, but that comes from the fundamental diffraction limits of the optics. So to circumvent that people have been working for designing a new modalities of the imaging system which has led to the different varieties of the scanning probe microscopic system where the electron beam instead of a optics beam or a photon beam used as a probe beam to look at the sample.

Now out of the different scanning probe system scanning electron microscope or SEM is commonly used for characterizing the morphology and the features of the materials. So today we will discuss about some of the parts of the some of the working principle of the SEM and how we can use the SEM to characterize a solar cell. Before we go for the SEM so as I said that in an SEM we use an electron beam.

So we go back to our very early lectures where we have said that just like photon can be represented by a wave similarly an electron can also be represented by a particle. So for example we have seen that a photon beam which is a particle right so that can be represented by a wave or in other words like electromagnetic wave that can be represented by a particle

and similarly an electron beam which is a particle that can be represented by a wave which is the de Broglie wave or matter wave.

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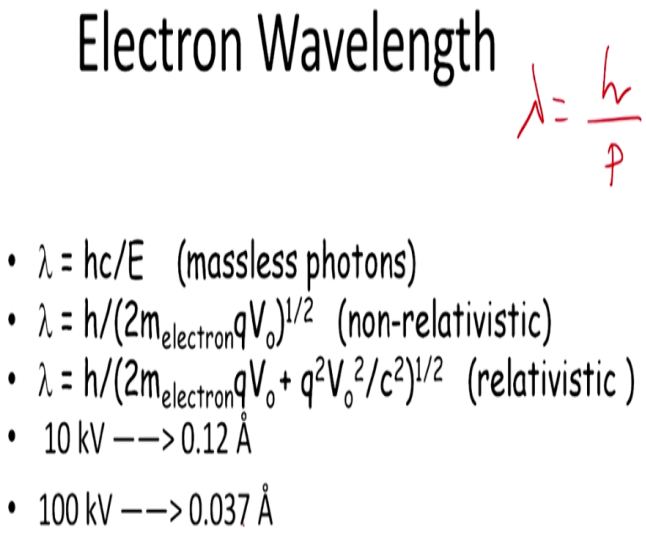

And we have learnt that this de Broglie lambda that= h/p where h is the Planck's constant and p is the momentum of the particle. So this lambda can also be written as hc/E in the case of massless photon and lambda you can write it as h/2m where m is the mass of the electron into q the charge of the electron times the V0 the applied potential difference times whole 1/2 this is for the non relativistic case.

For relativistic case we have to do the relativistic correction so lambda=h/2m electron q0V0+ q square V0 square/ c square to the power whole 1/2 that is for relativistic case. Now 10 kilovolts that corresponds to 0.12 angstrom and 100 kilovolts that corresponds to 0.037 angstrom. So depending upon whatever the resolution we are looking for so we use that kind of excitation potential for the electron beam.

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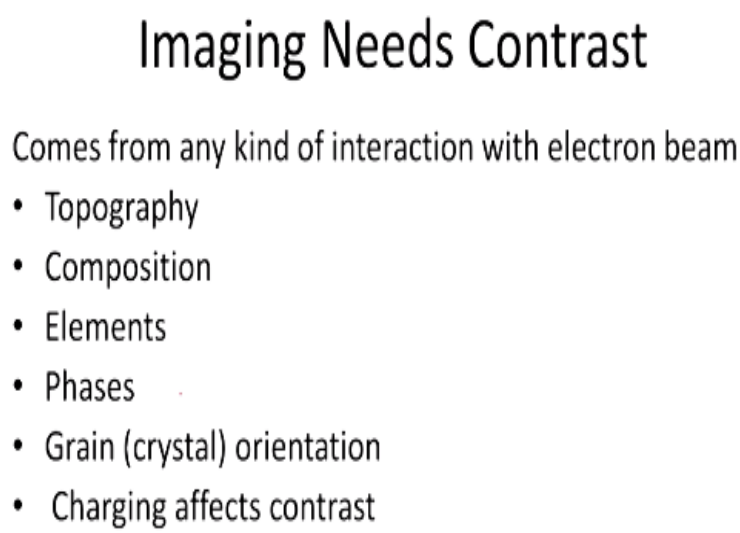

Now Imaging Needs Contrast so if I have to have a very good image we need a contrast comes from any kind of interaction with electron beam. It can be topography, it can be composition, it can be elements, it can be phase, it can be grain or crystal orientation or even charging that affects the contrast. So the topography of any image so topography means the top features or the textures of the image that also needs a good contrast.

The composition like let us say I have a perovskite material a common organic, inorganic Perovskite CH3NH3 led iodide. So the composition also affects the contrast. Elements what kind of materials we are using that also affects the contrast. Phase so what is the phase of the system that also affect the contrast. Grain orientation whether the crystal grains are oriented parallely or in a particular fashion or their random fashion so that also affects the contrast.

And also the charging affect if it is an organic material there is a possibility of charging it if it a inorganic material the charging possibility is less. So those all things actually changed the contrast. So that that means those all things can change the quality of the image. What are the advantage of the SEM over optical microscopy? Now to begin with so in the optical microscope we use a optical beam and there the source of the light is photon beam.

And what happens like either if you use an upright microscope or an inverted microscope so light beam comes and they interact with the sample. Interaction means that they transmit through the sample and we get the inverted image of that object.

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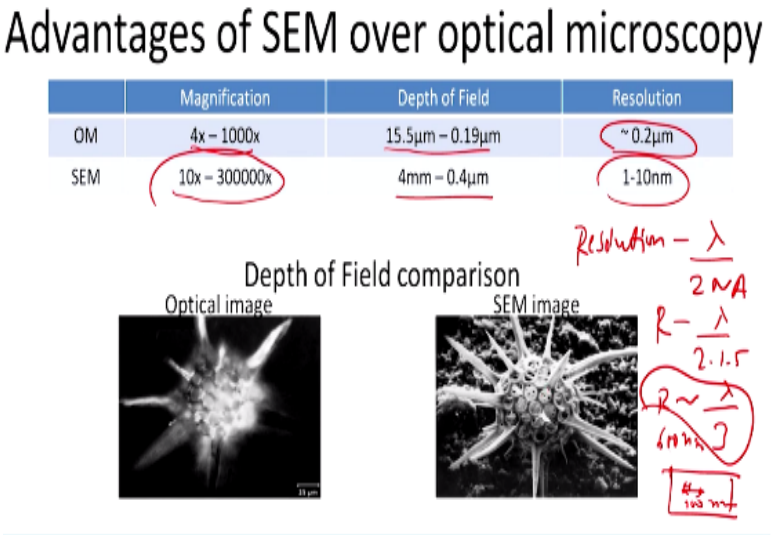

Now the magnification in an optical microscope which is written here as OM what is usually 4x to 1000x. Although 1000 x magnification optical microscope is very, very difficult and rare. Usually the normal inverted microscope fluorescence microscope we use it for 4 different light cues and with that 4 different magnifications of 10x, 20x, 40x and 100x in the case of 100x we also use the oil.

But going to 1000x is sometimes very difficult although it is possible. Whereas in a scanning electron microscope which is an example of an electron microscope the magnification is 10x to 3 lakh x so you can go to really large magnification. The depth of the field here is 15 to 0.19 micrometer in the case of optical microscope, but in the case of SEM it is 4 millimeter to 0.4 micron. So the depth of the field is also very low.

The resolution of an optical microscope is 0.2 micrometer whereas the resolution of an SEM is only 1 to 10 nanometer. So the resolution of any optical microscope if I write it as resolution that is lambda/2 into numerical aperture where lambda is the wave length of incident light. Now if I even use the very best quality film the numerical aperture let us say we get 1.5. So that means the resolutions R that is lambda/2 into 1.5 or R= lambda/3. So what is resolution?

Resolution is a capability to resolve the 2 point image. So let us say I have an image like this so how good or how closely I can separate these 2 points that gives the resolution of the system. Now let us say I have a feature in a scale of 100 nanometers. So this distance within these 2 points is 100 nanometer and I am looking at this system with a light of wavelength

600 nanometer.

Now according to this formula using the light beam of 600 nanometer the maximum separation between the 2 points which we can observe is 600/3 that is 200 nanometer. So that means if I have to look a features in a scale of 100 nanometer that I cannot do it by using a 600 nanometer light and look that it is not a problem of the light or it is not a problem of the optical system, it is not a problem of the length or not how we have organized our optics.

It is actually a limit diffraction limit of the systems beyond which it is not possible to resolve 2 components in an image, but if you use electron beam instead of light beam as a source then it is even possible to go to somewhat around 10 nanometer scale. Now this is only possible when you consider the electron not as a particle, but as a wave. Now that was the trick of the quantum theory.

In quantum theory, in contrast of the classical theory a particle like electron is considered as a wave and we say that we represent the electron as a wave function and we define the probability. We said that the probability of the electron in a particular space is more feasible or more realistic way to define its position rather than telling that electron is at a particular position at a definite position right.

So when we consider the electron as a wave then it was possible to use the electron beam as a source of the excitation beam and in electron microscope this is exactly what it has been done. Here instead of a light beam we use the electron beam as a source of the excitation beam. Now just like in an optical microscope light beam comes and that interacts with the sample substrate.

Here also electron beams comes and interacts with the sample substrate. Now the depth of the field comparison if we do an optical image and SEM image let us look at this features. This is a image of a virus if you look under the optical microscope you cannot resolve each and every component here because of the diffraction limit, but if you look it under an electron microscope you can see each and every features.

Now let us say for a solar cell devices, energy materials I made a perovskite thin films and I need to see where the grains are uniform whether it is continuous and large. So and the grain

size we know it is let us say 80 to 100 nanometer. So obviously by using this optical microscope I will not be able to get a good image of this perovskite grains, but if we use a scanning electron microscope then I can know what is my grain size.

What is the composition in the grain even I can optimize, I can do the grain engineering or I can do the morphology optimization to find out a proper morphology so that the Photophysics is also supported. So that is the importance of the SEM in the Solar Photovoltaics.

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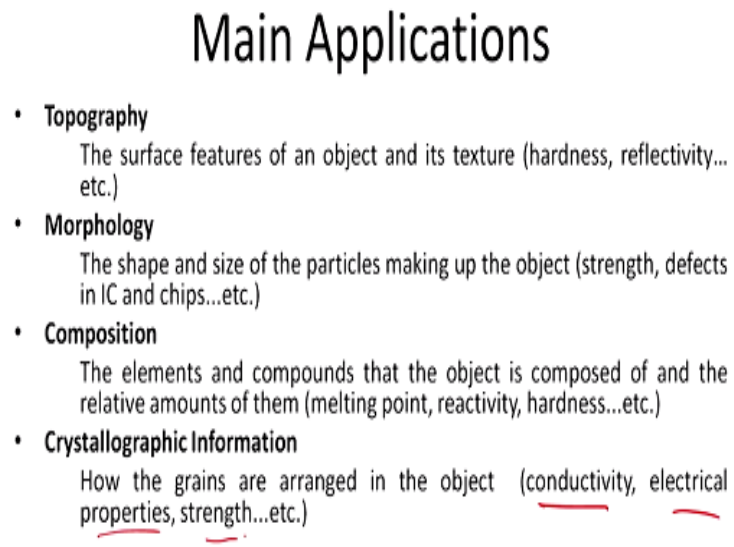

So some of the main application of the SEM is the topography. The surface features of an object and its textures like hardness, reflectivity etcetera. Morphology the shape and size of the particles up the object strength, defects in IC and chips etcetera. Composition the elements on the compounds that the objects is composed of and the relative amounts of them melting point reactivity hardness etcetera.

Now it is worthwhile to mention that in many times along with the SEM there is one more module is attached that is called EDS extra diffraction spectroscopy. So basically this EDS is used to do the elemental mapping so that can tell you what is the composition of the different elements inside the material and then crystallographic information how the grains are arranged in the object like conductivity, electrochemical properties, strength etcetera.

So all these properties or all of this information or the features or all of this information can be obtained by using the SEM.

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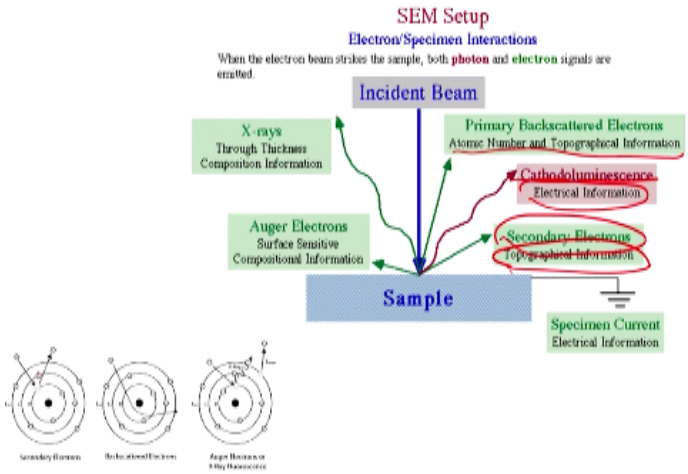
Electron Beam and Specimen Interactions



Now in an SEM setup so that an electron beam is allowed to interact with the sample specimen. Now whenever the incident beam interacts with the sample so many factors can happen so many phenomena can happen. So the SEM setup an electron or specimen interactions when happens when the electron beam strikes the sample both photons and electrons signals are emitted.

So there is lot of different possibilities is there when the electron beam is heating the sample substrate that mean the proton beam is here instead of the photon we are using an electron beam. So electron beam is interacting with the sample substrate. What is a substance substrate a layer of the atoms. Now what can happen firstly it can happen the emission of the X-rays to the thickness composition information.

So we know that x-ray can be continuous x-ray or characteristic x-ray. Continuous x-ray is a deceleration of the accelerated electrons and then the characteristic x-ray where the electron is ejected from the inner orbit. So both of this case will get an information about the sample composition then there can be Auger electrons. The surface sensitive compositional information can be obtained from there.

There can be primary backscattered electron which is the atomic number and topographical information. There can be Cathodoluminescence which gives about the electrical information and finally it can be secondary electrons also which gives the topographical information. In SEM mainly we look for the secondary electron which gives the topographical information. So for example if you look at this electron orbit this is the nucleus and the electron is waiting

around k, l and m orbits here we are just trying k l1 and l2,3 orbits.

Now whenever the electron ejected from there so then electron jumps from the inner orbit to the outer orbits and if that electron ejected then you get a secondary electrons.

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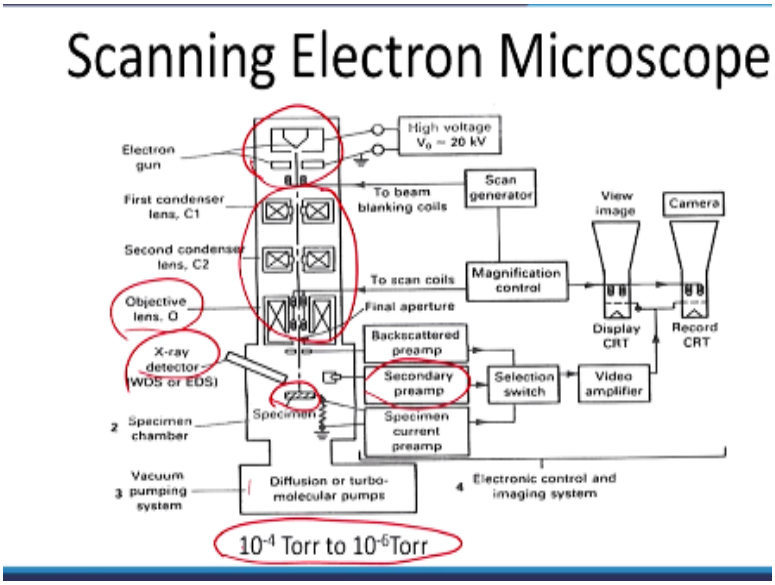

So the real schematic diagram of a scanning electron microscope is very, very complex. As you can see from this figure we have an electron gun here so this is the electron gun which emits the electron beam this one. Now in an optical microscope we use lens to collimate the beam. Now in an electron microscope we cannot use the lens to collimate the beam. Here we use electromagnetic coil and this electromagnetic coils serves as a lens and they actually collimate the beam.

So the first condenser lens C1 so that helps to collimate the beam and the second condenser lens C2 that further helps to collimate the beam and finally the collimated beam falls on an objective lens O which is situated here and which is also connected to a scanning coil and this scanning coil is connected to a magnification control unit and the magnification control unit is connected to a scan generator.

So basically the scan generator do the scanning and a beam blanking coils is attached to the scan generators. In the electron gun we apply a high voltage like 20 kilowatt below the objective lens there are x-ray detectors in the case of EDS and then there are different detector there, but first there are the specimen the sample substrate and this is the specimen chamber which is stayed in a very ultra high vacuum.

And these are the pumping system vacuum pumping system diffusion of the turbo-molecular pumps. We said that when I do the SEM we usually maintain a very high vacuum right because the electron is allowing the interaction of the scattering with another atomic layers. So basically we have to make sure there is nothing else inside the chambers. Now the detector they can be different things.

Now basically we are looking for the secondary electrons so that is why we have a secondary preamplifier and then we have the backscattered preamplifier and then we have a specimen current preamplifier. Now these are all connected to a selection switches which is connected to a video preamplifier and finally it goes to the display CRT and record CRT which is displayed in the camera.

So if we look the whole picture so basically there are 3 to 4 main components is there. One is the electron gun which emits the electron at high voltage at 20 kilovolt then there are condenser lens and objectives these are the optical components which helps to collimate the beam, collimated beam falls on the specimen substrate and whatever the electron is ejected that is detected by the detector.

It can be an x-ray detector or it can be a secondary electron detector and by doing some magnification we collect the image by an image acquisition system it is shown in the camera. So there are 4 parts in there. One is the electron gun, another is the optics, another is the specimen sample chamber where you keep the specimen under the vacuum and then there are some detectors is there.

And all the system is kept in a high vacuum 10 to the power – 4 Torr to 10 to the power -6 Torr. So that was the circuit diagram of an SEM.

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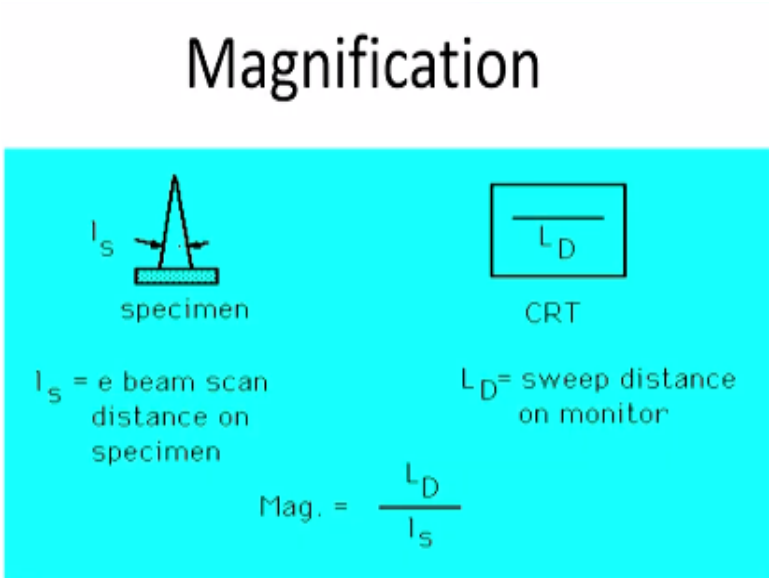

And in terms of the magnification we also gain a lot. For examples let us say IS is the beam which is falling on the specimen IS is the electron beams and it is the scan distance on the specimen and LD is the sweep distance on the monitor we are using a CRT. So LD is the sweep distance and IS is the electron beam scan distance from the objective to the sample specimen substrate. Most of the case it is kept at 80 millimeter. Now the magnification is defined as LD/IS. So the sweep distance/e beam scan distance.

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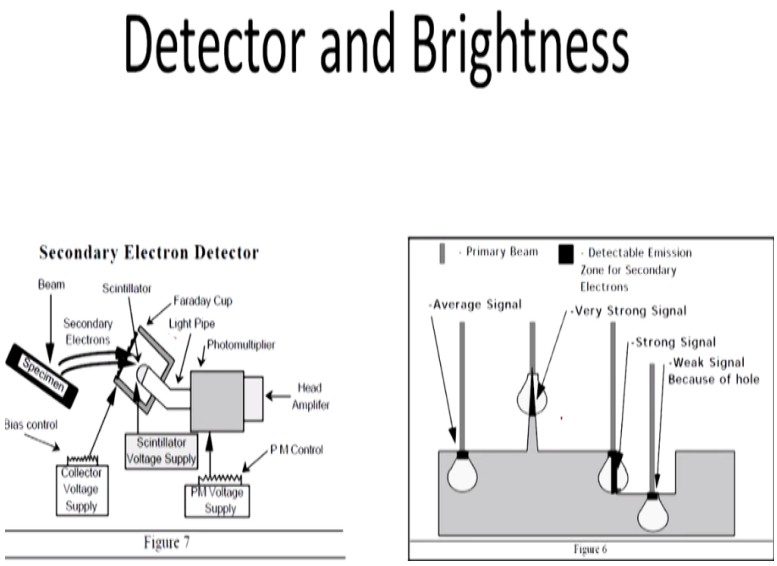

What about detectors and brightness. So there are lot of detectors are there, but mainly we are talking about the secondary electron detector. So you look at this figure like this is the specimen sample substrate on which the beam is falling now the secondary electron is ejected there now we put a scintillator. The scintillator collects the secondary electrons and that has been kept in a faraday cup.

And there are light pipe which is connected to a PMT or photomultiplier tubes and this PMT tube is biased by a PMT voltage supply and there is a scintillator voltage supply which bias the scintillator and whatever the light comes from the scintillator that goes to this PMT and the PMT is connected finally to the image acquisition systems. So basically the beams falls on the sample specimen.

It ejects the electron, the electron is collected by the scintillator and then it enters to the light pipe finally it goes to the PMT and the PMT is connected to the image acquisition systems. Now in this figure we are showing it here what will happen if we put a primary beam and detectable emission zone for secondary electrons. So for an average signal so this is the distance the beam can penetrate.

If I have a very strong signal, then the distance will become more and more. If it is a strong signal it can goes until this point if it is a weak signal because of the hole it can only penetrate a small distance.

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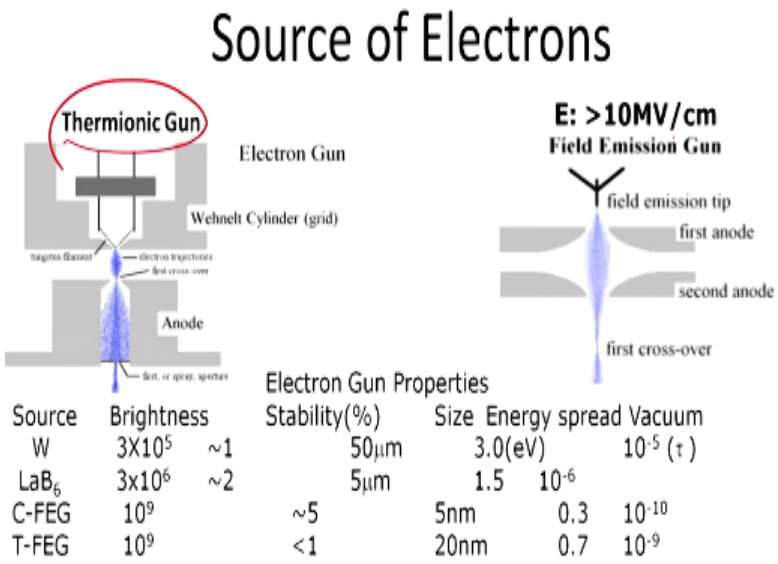

Now what can be the source of electrons the source of the electrons are called thermionic guns because they emits the electrons by heating the coils by hitting the filament so that is why they are called thermionic gun okay they are electron gun. So they have been placed in a cylinder called Wehnelt cylinder or grid and there are the anodes filament is there and some of the parameters for the source and brightness.

For example, if you use tungsten the brightness is 3*10 to the power 5. If we use Lanthanum boride or Lanthanum B6 this is 3*10 to the power 6. Similarly, for C-FEG and tungsten-FEG it is 10 to the power 9. So electron gun it is usually represented in terms of the stability and also in terms of the size energy spread vacuum which is tabulated in this table. Now in most of the cases the field emission gun that has a energy more than 10 millivolt per centimeter and if we use an organic sample we have to tune the voltage of the electron gun.

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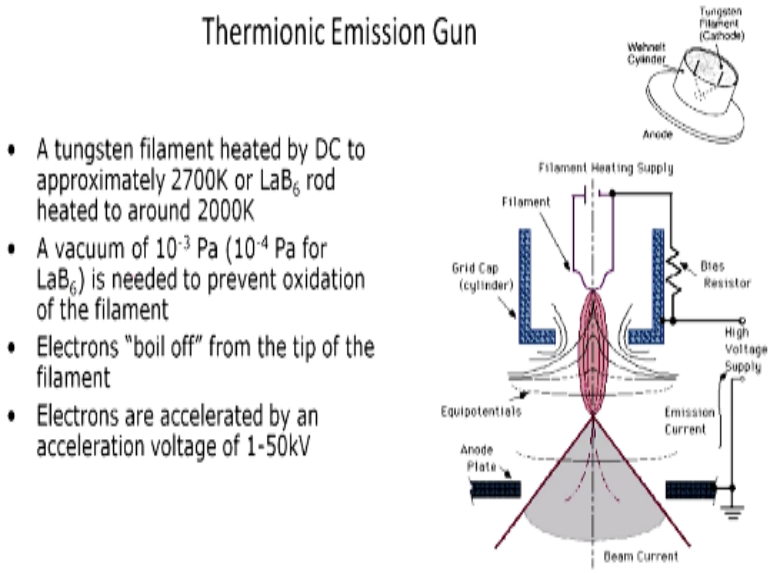

So here we are showing the thermionic emission gun as you can see this is a filament and this is the filament heating supply and the whole filament is put in a grid cap cylinder and there is biasing is happening to the filament supply. Now the electron is ejected and it goes to the electrode plate. This is the 2 electrode plate and which is kept under a voltage difference with respect to the cathode and whatever the electron that comes they goes in this solid angle.

So a tungsten filament heated by DC to approximately 2700 kelvin or LaB6 rod heated to around 2,000 kelvin. A vacuum of 10 to the power -3 pascal 10 to the power -4 pascal for LaB6 is needed to prevent oxidation of the filament. Electrons boil off from the tip of the filament, electrons are accelerated by an acceleration voltage of 1 to 50 kilovolt. So basically we use a tungsten filament or LaB6 filament needs a high vacuum.

Now if we heat the filaments electrons are ejected and there is a potential difference between the anode and the filament which collect the electron and electron start ejected in a solid angle and the electron comes to the condenser lens and the condenser lens will collimate the electron beam.

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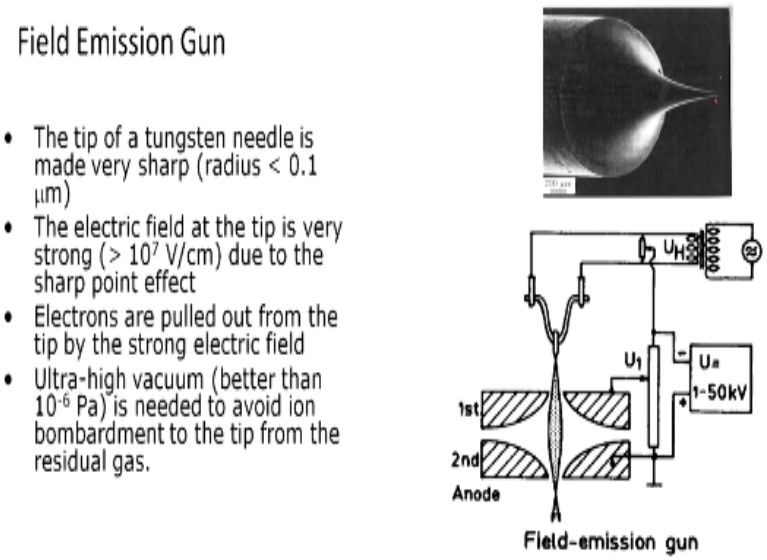

In a field emission gun the tip of a tungsten needle is made very, very sharp radius of less than 0.1 micrometer. The electric field at the tip is very, very strong so if you make this kind of tip the electric field at this tip will be very, very sharp this is about more than 10 to the power 7 volt per centimeter due to the sharp point effect. Electrons are pulled out from the tip by a strong electric field and ultra high vacuum better than 10 to the power -6 pascal is need to avoid ion bombardment to the tip from the residual gas.

Now there can be gas inside this chamber since there is a very strong electric field at the top of this tip so there is a possibility of the bombardment of the gas with the tip. So to avoid that we use ultra high vacuum in a field emission gun. So field emission gun is more sophisticated than a normal emission gun or normal thermo emission gun. So today we have discussed with you about the working principle of the scanning electron microscope.

And the different component of a scanning electron microscope and as I have said that SEM or scanning electron microscope is commonly used to know the morphology of the systems. Now since we have learn that morphology of any kind of solar cell is very, very important to optimize the efficiency as the morphology is directly related to the photophysics of the system.

That is why whenever we make any thin film before making we devise first we look it under the SEM to look its morphology. So in today's lecture we have discussed about what is the need for the SEM in a solar cell device and what are the different components of the SEM

system or the scanning electron microscope. In the next lecture, we will discuss about one important techniques of the solar cell characterization that is impedance spectroscopy based characterization.

And we will see that apart from some standard characterization this impedance spectroscopy technique has emerged as a very new technique for the characterization of the electrical parameters of the solar cell. Thank you so much.