Techniques of Materials Characterization
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Lecture – 11
Basic Components of Electron Microscope

Welcome everyone to this NPTEL online certification course on techniques of materials characterization, and we are in week 3 that is we are in module 3 and this week we will be discussing about some general concepts of electron microscopy. So, by now we have finished optical microscopy. First week we began with some very generalized concepts of microscopy.

So like resolution magnification, depth of field, depth of focus, then aberrations; all of these things which are equally applicable to an optical microscope as well as an electron microscope. Then the next week we moved on to optical microscopes where we discussed first about image formation, how an image forms in optical microscope. Then we discussed about the basic components of optical microscopes like objective lens, like light source, like condenser lenses and so on.

And then we discussed about the various modes of optical microscopy like brightfield, darkfield, reflected, transmitted then phase contrast microscopy, interference contrast, polarized microscopy, fluorescence microscopy and so on. Now it is time we move on to another type of microscopy altogether and that is called the electron microscopy, which is possibly one of the most celebrated microscopy techniques for the last entire century I would say.

It is almost 100 years of discovery of electron microscope, around 90 years for sure. And this electron microscopy, the development of electron microscopy is quite concurrent with the advancement of science in various fields and you will realize why the great potential of electron microscopy, when I will be discussing then you will realize that almost all parts of science and technology all kinds of research get benefited out of this electron microscopy.

So, this week will completely dedicate on general electron microscopy concepts, we will not make any differentiation between various types of electron microscopes. And then from next week onward possibly we will be starting with some specific type of electron microscopy. We will be discussing here in this week some concepts which are equally applicable to any kind of electron microscope.

So, the first lecture today we will be discussing about the basic components of an electron microscope which are common again to any kind of, any type of electron microscope these components are very much common in nature.

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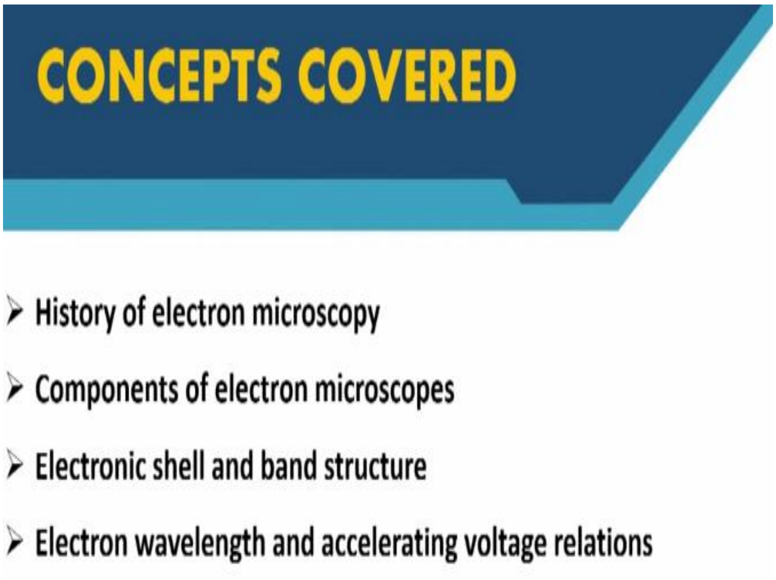

And like always, we will be discussing first about the history of electron microscopy. And then we will be discussing some of the common components of electron microscopes. And then there will be a discussion about the electronics shell and band structure and I will come what is the importance of that. And finally, we will be discussing about the relationship between electron wavelength and accelerating voltage that is under which the electrons are traveling.

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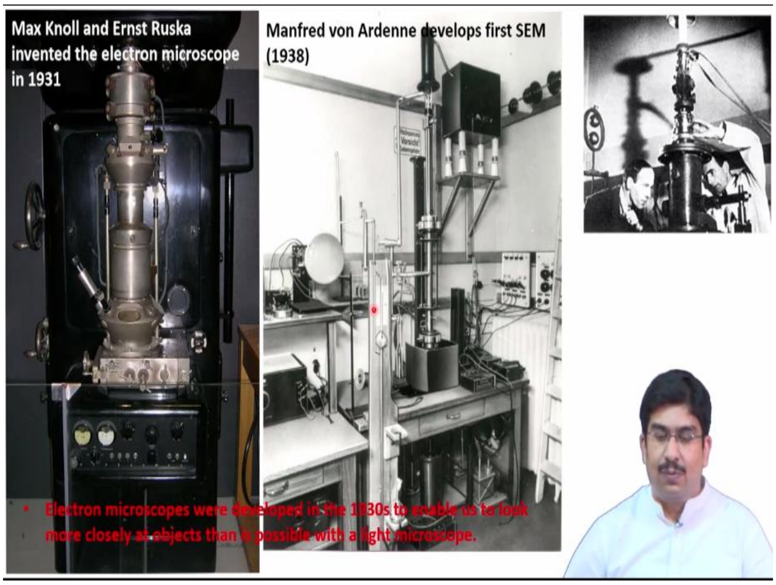

So, first let us discuss about the history of electron microscopy as I said that we are almost close to a century of the discovery of electron microscopy. And if any person has to be credited for the discovery of electron microscopes is basically these two people Max Knoll and Ernst Ruska, who have literally invented electron microscopy in 1931. So, the story goes like this.

Ernst Ruska was a Ph.D. student from TU Berlin and he was developing this TM microscope to help in his research, electron microscope. And finally, this becomes his Ph.D. thesis, this development of the electron microscope and he basically invented the transmission electron microscope, first various concept of transmission electron microscope and this he did when he was just a Ph. D. student.

Max Knoll on the other hand side was an instrumentation specialist with developing instruments and prototypes. And he was in a company Philips company which has now become Siemens. So, he was in Philips and he did completely the instrumental part, instrumentation side of this electron microscopes. And this is the first prototype made by both of them.

The first working prototype of a transmission electron microscope that they have made way back in 1931. So, we are exactly 90 years from the discovery of electron microscopes. The next big leap in the field of development of electron microscope was again sometime in 1938. At that time another gentleman and Dutchman called Manfred von Ardenne he develops basically the first prototype of SEM, scanning electron microscope.

So, the difference is Ruska developed it for transmission mode, he needed an electron transparent specimen. And for many specimens it was difficult to make the electron transparent by thinning down with the available techniques at that time. So, for a long time people were looking for some technique which can give them the similar kind of electron microscopy image for opaque specimen and that first discovery was first success came by this person M V Ardenne's development on SEM.

But at that time also this SEM was working almost similar principle as this transmission electron microscope and it was working at a very high voltage, only difference between these two is that in this case the sample was electron transparent, in this case the sample was opaque. So, that is the main difference between these two working microscopes at that time. So, that is how the first electron microscope was developed.

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Charles Oatley begins SEM development at Cambridge University, UK (1948)

Cambridge Scientific Instruments releases the first commercial SEM (1965)

And then the next big breakthrough come through this gentleman, Charles Oatley, who was a professor at Cambridge University and he got a project to develop an SEM and he was basically credited for many of the signal generation, many of those sources of signal which today we use for generating SEM images. He is credited for the discovery of many of them and of course the instrumentation part through a project which almost went over two decades that he was working.

The project was given continuously, he was funded from federal sources, he was funded from industries company or these electron microscopy manufacturing companies, all of them were funding him and he was developing this. And finally when it was developed, they came up with a company and that company called Cambridge Scientific Instruments who first released the commercial SEM way back in 1965.

Now, what is the importance of this commercial SEM? Before that, so you may be wondering that the first prototype was developed all in 30s and then the first commercial one came on the in the 60s and so on. Point is up to that time whatever a CM or TM was developed, they were mostly kept or they were built there itself. Some groups from some universities, some labs they were developing it for their own purpose and they were keeping it, they were using it. Nobody developed it for a general.

There was not like you just go to a company and ask that I want a CM or TM or such and such specification and they will do it for you. It was not possible up to that time till 1965 when Cambridge Scientific Instrument releases their first commercial SEM. Now it made everyone could take advantage of this SEM. Everyone you can just order it to this company and they will send you a commercial SEM and you can install it in your place.

And then you can also take advantage of this electron microscope. So that was another big year and of course there are many advancements that follows which we will go through sometimes over that, I did not give you a complete timeline of this. If you are really interested, there are some microscopy manufacturing, most importantly I can talk about is Zeiss, and they have a very nice poster.

I did not provide the link here, but you can check in Google and you put the Zeiss and the history of microscopy and you can get a very nice poster and that complete timeline is given how the discovery of microscopy happens right from 1930 all the way to the present day, various milestones. So, I request all of you all the students to go through that poster and you can order one that is the best part. You can also order one such printed poster for yourself, just please goes to go through this.

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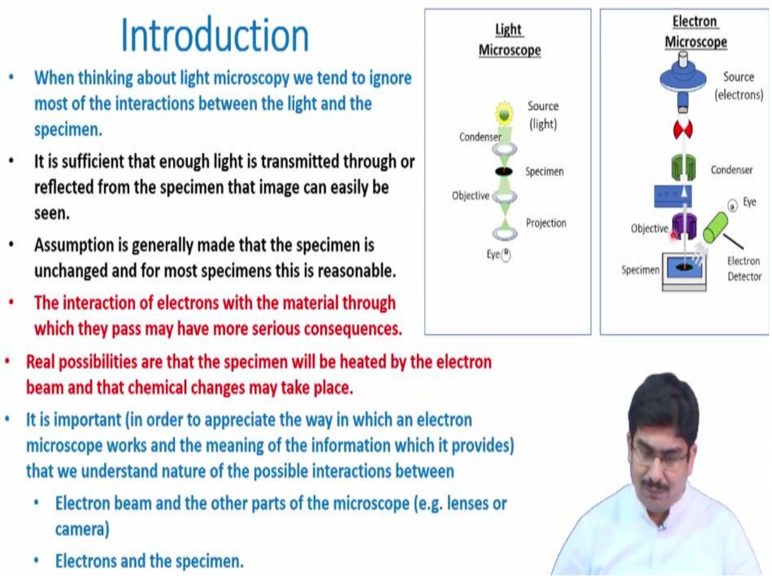

Now, let us have a very quick introduction of electron microscopy before we move on to various aspects of electron microscope. And first thing we need to understand is basically we have discussed this the difference between electron microscope and optical microscope time and again in the last two weeks when we were discussing about mostly with optical microscope.

Now, let us see the major difference the most important significant difference between the light microscope and electron microscope in the way they work, the working principle. So basically, when we discuss about light microscope here all we have a light source, condenser lens, specimen in transparent mode, objective lens and a project system, finally you see it with your eye.

So, this is what all light microscopy needs. Here, we tend to neglect one single thing that is how the light interacts with the specimen that is what that one thing we tend to neglect. What we are interested is that how the diffraction things happen over on the light if we are in bright field or the diffraction happens and how after passing through the light the direct beam forms, the entire background intensity and the diffracted beam goes and modifies it as per the specimen details that is it.

We do not consider how the light interacts with this specimen. Was there any change in the energy of the light, whether there is a change any change or a new type of signal is generated or not, nothing we do not, we completely neglect this. Of course in some methods, some contrast enhancement methods, we do consider a few things for example like phase contrast microscopy.

We do consider okay what is the change of phase there as a change of refractive index or for that matter you can consider that even in fluorescence we consider interaction between the fluorophores and the fluorescence the signal that is going. But by and large, we do not pay that much of an importance on this fact how light interacts with the specimen. The light is transmitting through the specimen, transmitting and reflecting that is enough for us.

But the electrons being a charged particle, their interaction with this material, any material when they pass through when they heated gets reflected that is very important and that is the heart of any electron microscope that is what makes the entire components, the entire working principle, everything of any electron microscope. It is all about how the electrons are interacting with the specimens.

So the first possibility of course is that the specimen gets heated up because, we will discuss about this a little later, when the electrons hit the specimen, most of the energy from these electrons goes in hitting as a form of hitting. So, this is something called inelastic interaction, we will discuss about that. But most of the energy of this incoming electron finally ends up in hitting the specimen.

So, that is one major difference between the light microscopy and electron microscopy. The electrons really interact with the specimen and generates some effect. Other than this hitting effect, there are some other things happen. There are some secondary signals that is generated and so on and there are some more changes happen both in the electron side as well as in the specimen side from which we finally get various sources of imaging.

So, all of these things need to be considered. So, most important to consider for an electron microscopy when we discuss about electron microscopy first thing is the electron beam how is it forming and the interaction of the electron beam with other parts of the microscopes. For example, lenses, camera and so on; so all the components basically.

And second part we have to understand is how the electrons are interacting with the specimen in order to understand the image that is formed from this electron microscopy. So, these are the two important things that needs to be considered and that needs to be discussed for any kind of electron microscopy to understand electron microscopy there and that is exactly what we will be doing in this week.

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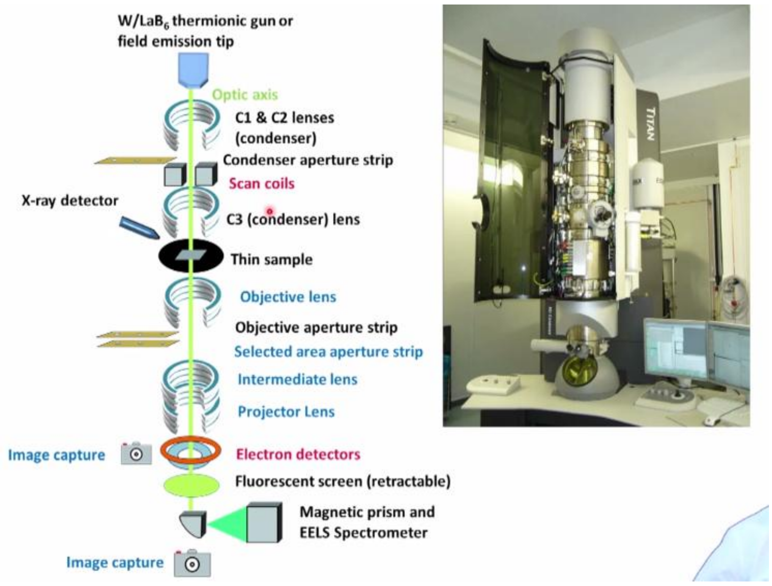

So, I have shown you these prototypes developed by Ernst Ruska and Max Knoll the first prototype of TM. So, from there and now we have come all the way to a very complex design for this electron microscope. This is one of the very complex electron microscopes called Titan made by FEI and this you can show how so many such a tall machine and so many different components are there.

This usually almost used to be a two-storey building, not less than that, I am not exaggerating. This one sometimes it can be of that at least a single storey building for sure. This one the height of this from the base or rather from this here up to here and depends on the detectors also, what kind of detector. So if you are adding more number of detectors, more capabilities, it can be even more complex as well.

But whatever it is basically if you take a cut cross section of these machines, what you will be seeing is basically these various components here. It will have a gun, we will come to that. It will have a gun, here some sorts of electron, and then you will be having a lens, you will have aperture of course. Then what you will have is specimen. If it is

TM transmission electron microscope you will have a specimen which is electron transparent.

So, you will have this condenser lenses on this side on the source side and then on specimen side on the imaging side, you will have the objective lenses, objective apertures then you will have a lot of intermediate lenses and finally you will have a projection screen on that screen the image will form. And in between you can have various other types of detectors.

For example, here you can have an X-ray detector, characteristic X-ray detector which we know as EDS, energy dispersive spectroscopy. And then you can have a CCD camera here to capture this fluorescent screen, this image of this which is forming on the fluorescent screen. You can also have some other detector called EELS, electron energy loss spectroscopy detector here.

And you can also have this another very special type of detector which we will discuss when we discuss about image formation in electron microscope. This is called high angle. In transmission electron microscope this is called high angle annular diffraction, the head of detector. So, these various types of detectors you can add here.

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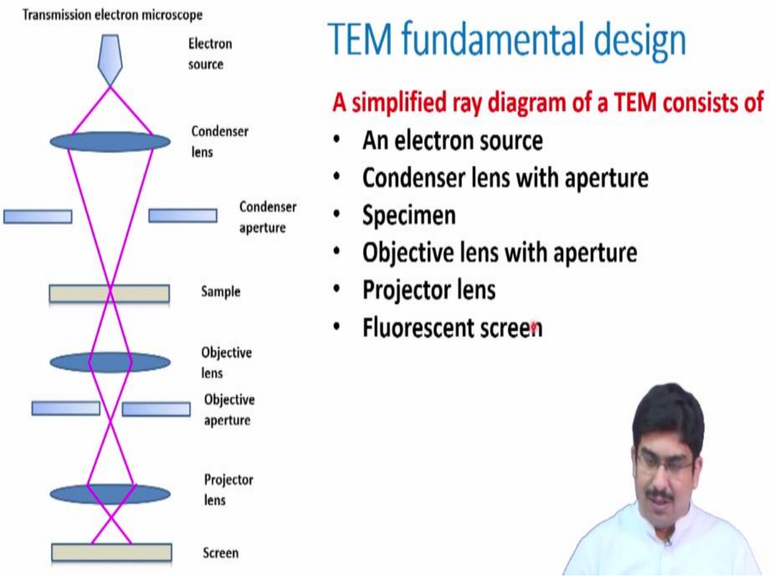

But the basic construction of an electron microscope still remains the same just like an any optical microscope, the same lens system. So, you have the electron source. Just like light source, you have electron source. You have the condenser lens system, condenser aperture which basically cause this light to focus on your specimen. After it passes through the specimen, it has to go through the objective lenses and an objective aperture.

Finally, there will be a projector lens which will be forming an image on the screen. In this case, it is fluorescent screen. Basically, what happens is that electrons when they hit that screen it generates a fluorescence effect. That means it generates a light basically. So, if I look at now the simplified ray diagram as I was saying, I have an electron source, condenser lens, specimen, objective lens, projector lens, screen exactly the same kind of configuration like an optical microscope.

This is sometimes really very surprising that the most I would say in the bottom, bottom most part of microscopy, optical microscopy resembles the highest or most complex of the microscopes, transmission electron microscopy. So, fundamentally they are related because ultimately both of them are kind of image formation device, and any image formation device if they will have these basic components.

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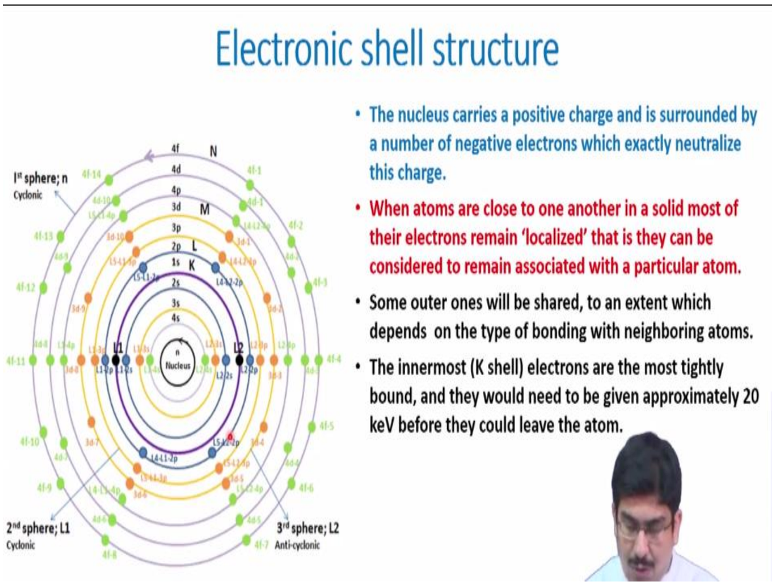

Now in order to understand how this electron source and how this electron material interaction happened, we must very briefly discuss about electronic cell structure. We will not go through very details of it or anything, whatever is important for us for the context of electron microscopy we will discuss about that. So, what we know is that nucleus is carrying positive charge and the rest of the electrons are in this shell, various different shells.

They are circling around these heavy nuclei and the charge is balanced exactly. The number of electrons is exactly same as the number of protons within the nucleus that is it. Now, when these electrons are very close to the nucleus that is the electrons which are possibly there what we name is K shell or L, M, N all those shells and they have a subshell as well, one is 2S something like that. All this electronic configuration which we possibly used to do in your +2, at least I used to do in my +2.

And so electrons when they are very close to the nucleus, that means when they are within this K, L or such shell, they are very tightly bonded to the electron. So, this energy or this binding between the electrons in these orbital electrons and the nucleus strongly depends on the distance because it is ultimately columbic interaction. And there the K shell electrons or L shell electrons are very tightly bonded with the nucleus, whereas the electrons which are far away from the nucleus they are very loosely bonded.

And those far away electrons are basically those electrons which takes part in bonding, when they are bonding means these electrons are shared between various atoms, whereas these electrons stay close to the nucleus and stays as I can say that the atoms keep them as their own, whereas they are ready to share the outer electrons that is what. Outer electrons are not that strongly bonded.

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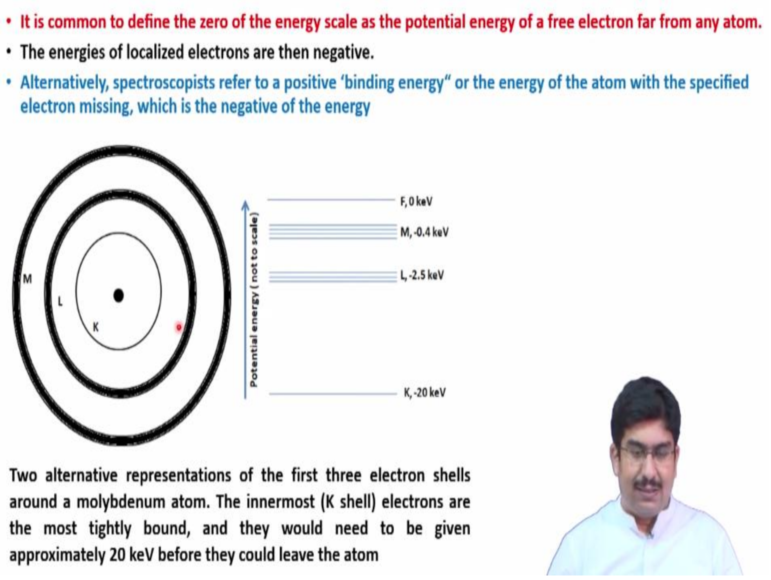

So if I now see the energy scale in terms of an energy scale then what we can do is that we can define the zero energy as for a free electron which is far off from the atom. So if we consider any electron which is very far from the electron, we can consider their potential energy as 0. Compared to that all other energies will be -10 and as you can see the K shell electrons will have the highest amount of energy in negative, –20 kV usually value safely you can think of for most of the metals and most of the elements. So, this is how the energetics is defined.

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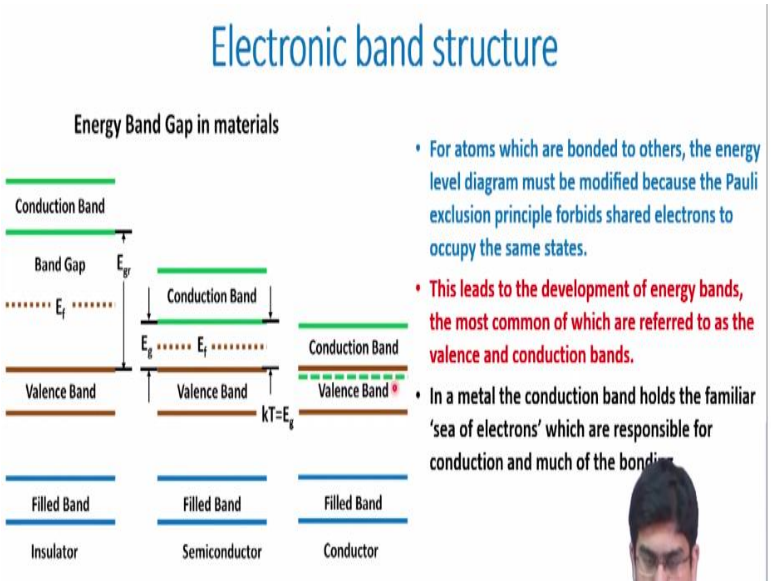

And now what happens is that because of these outer electrons which are shared between each other, those electrons finally go and make something known as the conduction band structure in the material. So, this electronic band structure again I think everyone knows about this, most of you at least know about this that there are various band structures where the electrons are shared in the bonding, the electrons are shared.

And Pauli's exclusion principle forbids shared electrons to occupy the same state. Because of that this band structure is introduced concept and what we have is conduction band, valence band and then filled band. And it is basically the energy difference between the conduction band and the valence band, which differentiate between an insulator, semiconductor and conductor.

So, in case of a conductor, the valence band and conduction band almost overlap and that means there is a huge abundance of electrons available within the conduction band for these metals, mostly metallic specimen conductors.

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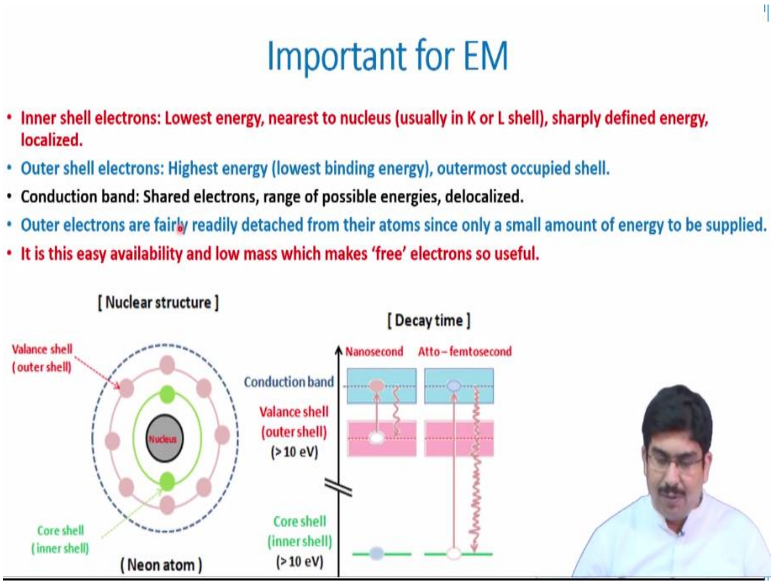

Now, for why we are discussing this in context of electron microscope that is because this inner shell electrons means this filled band here somewhere very K shell, L shell those electrons, those electrons obviously have lowest energy negative, means highest binding energy you can say that they are most tightly bonded with the nucleus highest binding energy.

So, these electrons are nearest to nucleus and they are having very sharply defined energies. The electrons which are there in K, L those shells they have a very sharply defined, their energy is very much I would say defined. So, they have certain amount of energy which is very much so. You move from one atom to another atom and one state to another state their energy will not change.

So, these are called the localized electrons. Outer shell electrons on the other hand, they have lowest binding energy because they are farther away. And they are also usually in the outermost occupied shells, they do not have very sharply defined energies and they are not localized, they are delocalized electrons. They take part in bonding, they take part in conduction, all sorts of such phenomena.

Now, conduction band of course is made out of this outer shell electrons, these shared electrons are there and they have a range of possible energies and delocalized almost. These outer electrons are fairly readily can be detached from the atoms since only a small amount of energy is needed. The binding energy, they are far from nucleus their binding energy is very low, so they can be very easily taken out form these atoms.

And this free electron, so called free electrons are very useful for producing electron. So, materials can work as, so these electrons can work as a source for electron beam to generate the electron beam. So, outer shell electrons, conduction band electrons those electrons can produce the electron beam that is why they are important in case of an electron microscopy.

Whereas the inner shell electrons are responsible for an electron interaction for signal generation, which we will discuss in a little later. So, for signal generation inner shell electrons, sharply defined energy; for electron beam generation what we need is outer electrons. These are all important for distinction between these two types of electrons.

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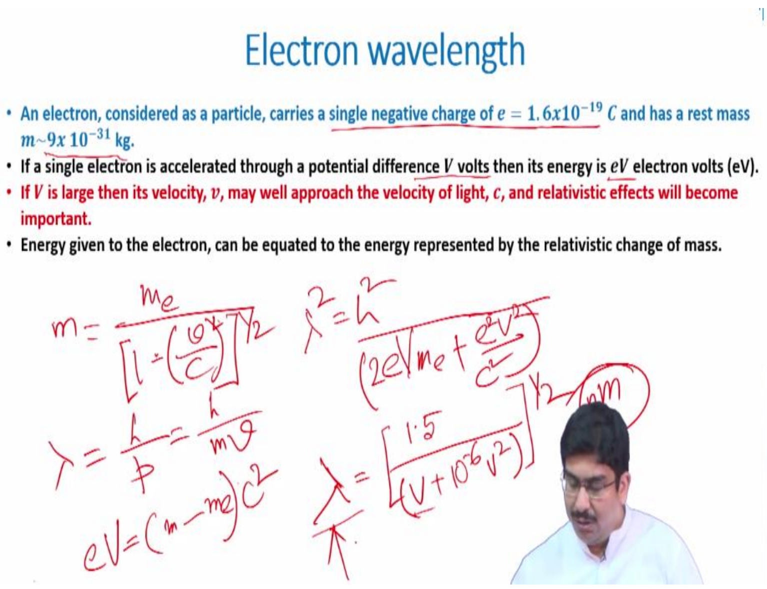

Now, we will be doing a little work here and this is to find out the relationship between the electrons and the acceleration voltage under which, so now we are assuming that these outer electrons have come out from the material, from the atoms, somehow we have taken them out and we have applied an acceleration voltage for these electrons which are coming out from the material.

Now, what will be the wavelength of these electrons that is what we are going to calculate, the relationship between the accelerating voltage and the electron wavelength. So, in this case you know electrons have dual characteristics and we will use both here. What we can imagine is that an electron now is considered to be a particle and they carries a single negative charge of this much, this coulomb $1.6*10^{-19}$ C.

And they have a rest mass of $9*10^{-31}$kg okay. And we are accelerating a single electron through a potential difference of V volts then the energy of those electrons is obviously eV electron volts. What happens is that often this velocity of electrons is very high and they will approach, they can reach the velocity of light and whenever such things happen, we know that we have to introduce relativistic effects in here.

So if we do that, then we can possibly write this rest mass of the electrons we can write it as this $m = m_e/[(1 - v/c^2)]^{0.5}$. So, this is how after relativistic corrections the mass of electron that becomes. Now if we consider the electrons as a wave, what we can do is that we can get the $\lambda = h/p$ where p is the momentum of course and this comes out to be again and I can again write this the h/mv.

So that is it, that is the De Broglie relationship we know and h is of course the Planck's constant here. Again, what we can do is that the energy that is given to the electrons from this potential difference that under which the electrons are accelerating, so this energy given to the electrons we can equate it to the energy represented by the relativistic change of mass. That means what we can write is $eV = (m - m_e)c^2$.

So, that is how the electrons are given this match of energy under the potential difference and that is creating this relativistic change in mass. If I combined all of this, then what I can get is a relationship of this kind $\lambda^2 = h^2/[2eV\, m_e + e^2\, v^2/c^2]$ okay. And if I simplify, if I put the values of all of these that e, if I put values of Planck's constant, if I put value of m_e rest mass, if I put the value of speed of light, ultimately the relationship comes down to be of this sort.

$\lambda = [1.5/(v + 10^{-6}v^2)]^{0.5}$ and this is in nm, this is important the unit of this, please remember this one. So, this is how I have got now the relationship between the accelerating voltage of this here this V the accelerating voltage and the wavelength of the electron which is passing through that kind of a potential difference, right.

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Wavelength-accelerating voltage correlation

- **Wavelength of the electron depends on the potential difference, or accelerating voltage.**
- **At the accelerating voltages which are most useful for electron microscopy, ($2x10^4$ V upwards) the electrons are accelerated to a velocity which is a significant fraction of the velocity of light and relativistic effects are quite important.**

Table 2.2 Electron Wavelengths

V (kV)	Wavelength λ (nm) Uncorrected	Wavelength λ (nm) Relativistically corrected
20	0.0086	0.0086
40	0.0061	0.0060
60	0.0050	0.0049
80	0.0043	0.0042
100	0.0039	0.0037
200	0.0027	0.0025
300	0.0022	0.0020
400	0.0019	0.0016
500	0.0017	0.0014
1000	0.0012	0.0009

$c = 2.998*10^8\ ms^{-1}$
$e = 1.602*10^{-19}\ C$
$h = 6.62*10^{-39}\ Js$
$m_e = 9.108*10^{-31}\ kg$

So, now if we put some values for V, what is happening here. So, I am putting this wavelength of the electron and I am putting some typical values here. So, if we now look at this wavelength of the electron that will depend on the potential difference or accelerating voltage and this is how we can calculate this, the V, we are applying from 20 to all the way to 1000 kV.

And the wavelength this is without any relativistic correction and this is for relativistically corrected. And these are the kinds of values that we can use for c, e, h or m e. Now, typically on electron microscope in transmission mode is used this kind of an accelerating voltage from 100 to 300 kV typically, whereas an SEM scanning electron microscope uses something like 20 to 30 kV of light.

One thing you can notice number one if you are working with this kind of range, then the difference between uncorrelated, uncorrected and relativistically corrected value is quite significant basically. By the way if you are working somewhere up to 500, I mean imagine that you have an electron microscope and such electron microscopes are increasingly becoming popular, very high kV electron microscopes.

There basically if you do not apply this relativistic correction, this value, this point 0.0017 what it is happening is that these values are coming or sometimes exceeding the value of c, value of light, velocity of light which is not possible according to Einstein's general theory of relativity. So, for those cases, those kinds of an acceleration voltage, you must apply the relativistic correction.

And from there you can get this wavelength of the electrons and the significance of calculating the wavelength of electrons you will realize this when we discuss about a phenomenon called diffraction. There you will realize what is, why or this numbers or the spread why it is so important for to calculate and to get an idea about this lambda value here.

So, we will stop here and in the next class we will be going through some more about these basic components of electron microscopy and then there will take some specific components and we will discuss about their significance in electron microscope. So, till then thank you and goodbye.