Techniques of Materials Characterization
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Lecture – 15
Electron-Material Interaction - Continued

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. And we are currently in module 3, week 3 and we were discussing about general concepts of electron microscopy. So, we have discussed so far about the history of electron microscopy, then the relationship between the electron wavelength and accelerating voltage and then electron production, electron generation from different type of electron guns like thermionic guns, field emission guns and so on.

Then we discussed about electromagnetic lenses, scanning coils, and then how aberrations are related to the electron source and electromagnetic lenses. And then we have discussed about the electron-material interaction and that is what we are continuing from there. So, in this lecture also we will be discussing about electron-material interaction.

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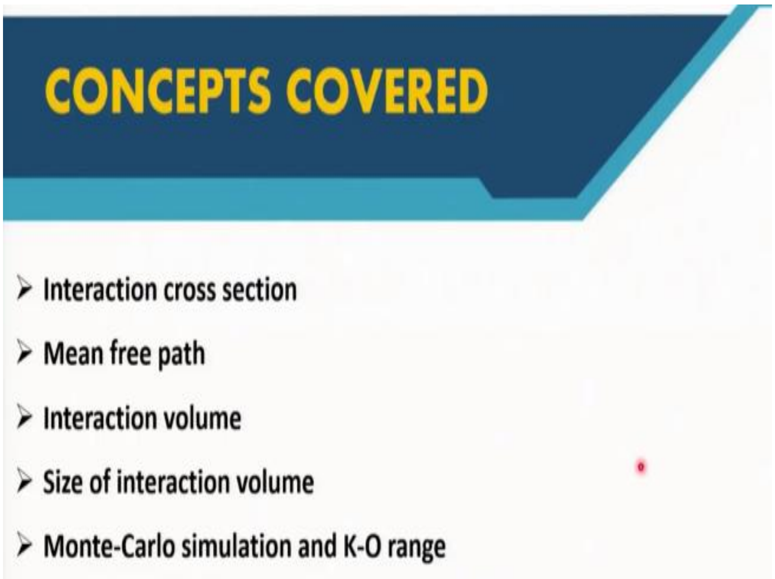

So, today's topics will be on interaction cross section, mean free path, interaction volume and size of the interaction volume. And finally if time permits, we will discuss about Monte-Carlo simulation and K-O range.

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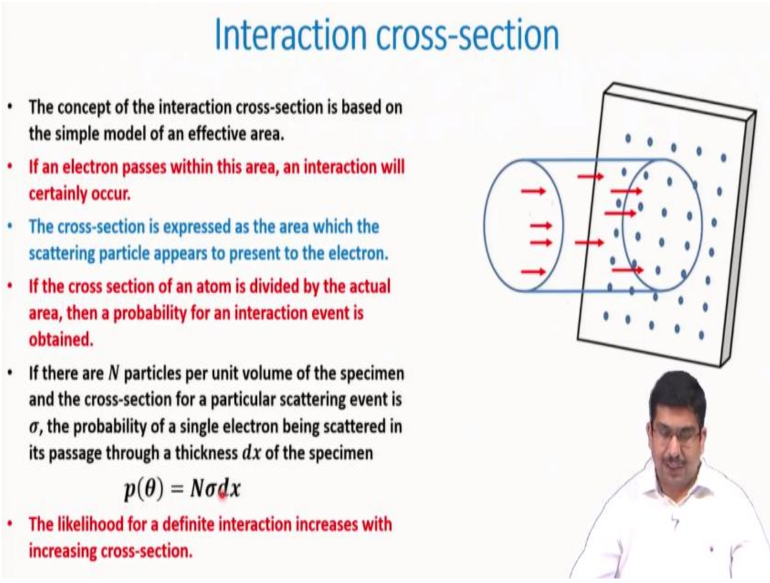

So, in the last class I just got introduced you to the concept of this electron-material interaction and the last topic was the probability of electron-material interaction and I said there the probability can be expressed in two different terms. So, basically what the probability means is that you have an electron beam like this, which contains lots of electrons and number of electrons are there. And then you have this material which is here and the material contains the atoms.

So, if the electrons fall on this material, then what is the probability or how to express the probability that these electrons will interact with this material? The interaction can be elastic interaction and or inelastic interaction does not matter, but what is the probability of these electrons being interacted or electrons interacting with the atoms which are present not only this surface but also on the surfaces which is in the thickness direction.

So, how to express this issue? And we discussed that this we can do it by two terms, one is called the interaction cross section. So, interaction cross section is basically meant as an effective area. So, what is that effective area? What is so special about the effective area? Basically, this is an area within the space within the atom or scattering centers, in this case the atoms are the scattering centers.

So, within that area if an electron passes through, then certainly there will be some kind of an interaction, elastic and inelastic does not matter. There will be some interaction definitely. That means that within that cross section, the probability of having an interaction is 100% within that cross section. So that cross section or that effective area is interaction cross-section and how do we get it?

Basically, if the total cross section of an atom or scattering center whatever is causing the interaction, if the total cross section of that atom is divided by this actual area that interaction cross section, then we can get the probability of any interaction event. Now just understand this; if electron passes through this area interaction cross section probability is 100%, scattering probability is 100%.

Now, the total atom area divided by this interaction cross section is the probability for a scattering event, total scattering event because that interaction cross section area will control how much scattering is possible. So, now how to express this in mathematical terms? Basically, if we have n number of particles per unit volume of the specimen and this interaction cross section or rather the cross section for any particular scattering event is sigma.

Then the probability of a single electron being scattered during its passage through a thickness dx of the specimen the thickness which contains the same number of electrons or a number of atoms, the probability can be expressed by this relationship p theta equals N sigma dx. So, here the sigma is interaction cross section, N is the number of particles and dx is the thickness through which these electrons are passing through.

So, the first thing we can notice is that this probability definitely increases if interaction cross section increases. The more cross section more such area any atom or any scattering center has, the better chances or better higher probability for that atom to scatter the incoming electrons that is first thing that we can make out of this.

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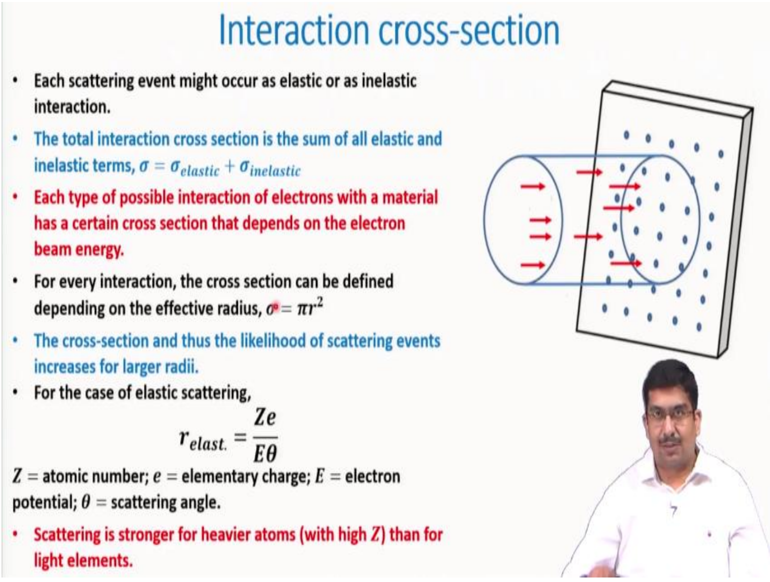

Now, each of these scattering as I already said can be elastic or inelastic does not matter, both are possible. So, the total interaction cross section the sigma can be expressed as a linear summation of this elastic interaction and inelastic interaction, probability for elastic interaction plus probability for inelastic interaction. That means the total interaction cross-section is the sum of interaction cross-section for elastic interaction and interaction cross section for inelastic interactions.

And both this sigma elastic and sigma inelastic this depends on the energy of the electrons, the incoming electrons. What will be the cross section for elastic whether elastic will be higher than inelastic or so, everything will depend on the energy of this electrons and we will see that dependence of energy in a minute. Also, this interaction cross-section if we assume that this is a spherical area, any assumption related to spheres is always good.

My students sometime asked me that why spherical and I said spherical assumptions are good because then you have to even though if it is not really very close to the actual situation, any spherical assumption any area anything if we consider it sphere then we have to deal with only one parameter that is its radius. If we talk about something like a rectangular area or a square are, then we have more than one parameter to discuss.

If it is rectangle, then we have to consider two different parameters, the length and the width and so on and so forth. Square also we have to then consider the angle and so on. But radius means only one, no angle, no other dimension, only the radius we can consider. So, that is the advantage of considering anything as a square just like even in atomic area, we consider atoms as a circle and so on.

But, if we do that if we consider this, then the interaction cross-section can be expressed by certain effective area and then what we can understand is that this effective area basically will control the interaction. So, if the effective area is large, the interaction area will also be correspondingly large and it is in square terms. So, any change in effective area will be really changing the interaction cross-section, will be reflected in a larger change in the interaction cross-section.

So if we now see the possibility for elastic interaction, then this radius for elastic interaction this will depend on these four terms. The first one Z is the atomic number, e is the charge of the electrons of course, then capital E is the electron potential or the accelerating energy of that electron fine and theta is obviously the scattering angle. So, the first thing we can make out from there is if Z increases that means if we have a heavier atom, then of course r will increase.

And that will increase the likelihood for this, that will increase the interaction cross-section meaning that will increase of course the probability of having a scattering. So, scattering will be much stronger for a heavier atom or atoms with a higher or elements with a higher atomic number that is the first thing we can understand from this concept.

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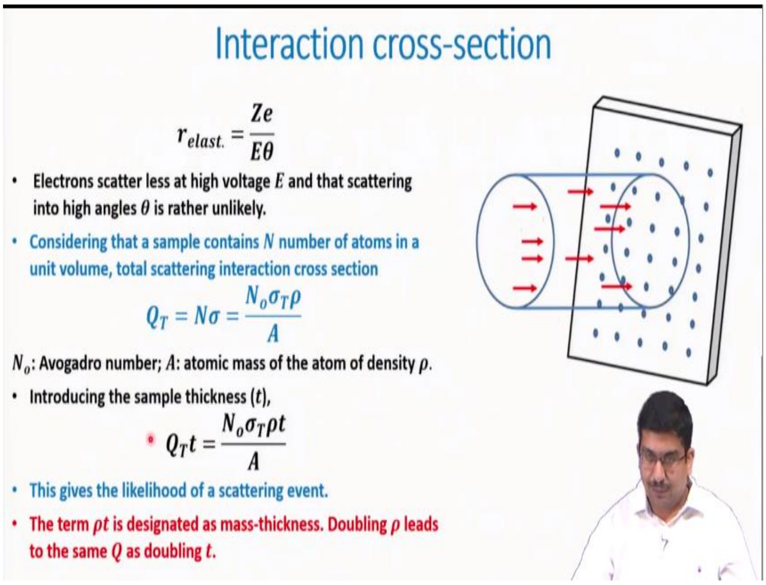

Again, we can also see that it depends this r depends inversely on the electron energy. That means if we have a higher electron energy, then this r will be less, correspondingly interaction cross-section will be less. This is something that we discussed in the last class as well that if the electrons are of very high energy, very high velocity, then they will just undergo, they will just penetrate.

They will pass through without having any scattering by the nucleus, either the electron cloud or the positively charged nucleus. They will not have enough time for these scattering centers to influence those highly charged high energy electrons that is what. So, here we are expressing the same thing, but now with the concept of interaction cross-section. That is one and second thing what we discuss is the high angle scattering.

If θ is very large, then r is correspondingly low, interaction cross-section is low. That means this probability for high angle scattering again is very unlikely because high angle scattering remember high angle scattering is mostly done by the positively charged nucleus, which is a very tiny fraction of the entire atom. So that is why high angle scattering probability interaction cross-section is very low.

Now what we can consider is the Q_T in a specimen in a sample of unit dimension. If that unit dimension contains say for example n number of atoms and each of these atoms has a contraction cross-section of σ, then this total interaction cross-section for the entire specimen will be $Q_T = N\sigma$. So, this we can express again by this relationship where we can introduce Avogadro number, atomic mass, density and so on.

And we can express it in this way $Q_T = (N_0\ \sigma_T \rho)/A$. Again, what we can do is that we can introduce another quantity here, we can just multiply it with the thickness. Now, we are considering not an area, but in the thickness direction as well the total volume interaction volume. Then we will see that there is one term that comes over ρt and that ρt is basically what is the specimen property, materials property, this ρt, mass and or density and the thickness of the material.

So, what does it mean is that if these two ρ and t either together or individually if they are changed that will impact this interaction cross-section interaction volume as such. This term is called mass-thickness and we will see more about it when we discuss about transmission electron microscope, image formation contrast formation there. Basically, what it says is that this mass thickness works together.

If we double this ρ then that will produce the same effect as doubling the t on Q that is what. So, these two terms come together and called mass-thickness and these are characteristics of the material which is there or the material which is basically working as an interaction with which electrons are interacting fine.

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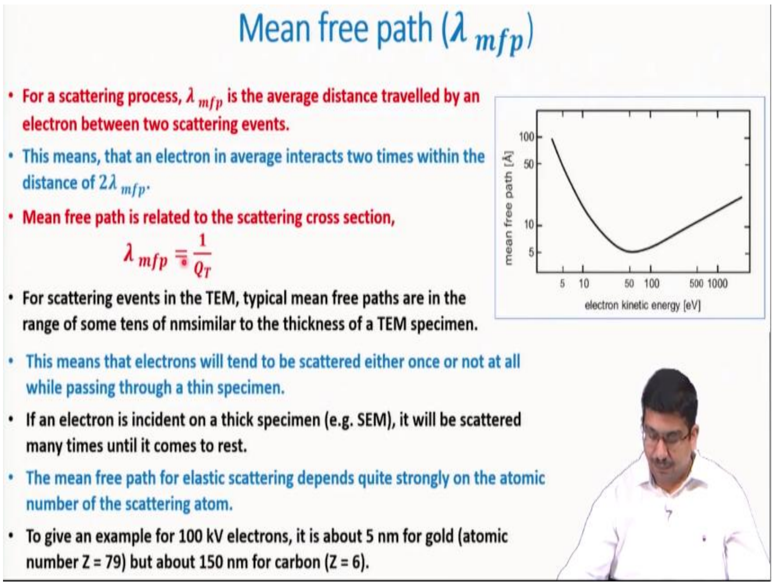

The next concept about again on the probabilities mean free path. Now, what is mean free path? We express this as λ_{mfp}. So, this λ_{mfp} is basically the average distance traveled by an electron between two scattering events. Now, this is a little confusing. My students always confuse this. This is the distance traveled between two scattering events, not necessarily the distance traveled between the scattering centers that is the difference.

That means, the minimum number of such interaction that you can have over a distance of $2\lambda_{mfp}$ is two electrons will interact, in average it will interact two times within a distance of 2λ. That does not mean that within 2λ you have most of them think that it is 3 that within 2λ distances first you have a scattering, then another in λ distance another scattering, then another scattering another λ distance.

So, in 2λ distance you have three such scattering. No, you have three scattering centers, but the scattering event, minimum number of scattering events can be 2λ where you start let us say from the middle of two atoms, then you move on half you get one scattering, you move on another full you get another scattering right, and then you can move another λ, λ/2.

So, together you will move 2λ and you get two scattering events, so that is what, do not get confused. Anyway, so mean free path again is related to the scattering cross section, interaction cross-section by this relationship $1/Q_T$, so inverse relationship and that is very much justified. If you have a very high interaction cross-section, λ_{mfp} will be less, meaning electrons will be undergoing scattering quite frequently, very frequently.

Interaction cross-section is very high or all the atoms total atoms then number of scattering centers will also be very high and then electrons will be continuously getting scattered. So, distance between scattering events will also be very less. Now, this mean free path concept is very important for transmission electron microscope, why because in transmission electron microscope what we will discuss later, we tend to keep the number of interactions as minimum as possible.

So, this is another reason. So already we have seen or already I have said that in order to make something electron transparent which is necessary for transmission electron microscope you have to thin it down to a very small level where a very small thickness is needed, almost in the μm range. But what will decide that thickness? This λ_{mfp} will be the deciding factor.

You tend to reduce or you tend to restrict the number of interactions to as minimal as possible, maybe single scattering or at the most plural scattering and mostly elastic interaction because the problem is that if there is a multiple scattering, then it will be a mixed elastic and inelastic scattering. If you want to restrict only within elastic scattering, then you should restrict with one single scattering or at the max plural scattering.

So, that means the mean free path should be off the order of thickness of this TEM specimen that is the relationship. So, if you λ_{mfp} is exactly the same as the thickness, you will be having only one scattering, only one pure scattering single scattering and most likely that will be elastic interaction that is what is desirable and that is how you can imagine the TEM specimen thickness is decided.

Of course, in reality TEM specimen thickness normally is bigger than the λ_{mfp}, so electrons undergo not a single scattering, multiple scattering or plural scattering. I would not say multiple scattering, plural scattering they undergoes. For a thick specimen anyway the λ_{mfp} will be like the specimen is definitely opaque, right, we know it, it is not electron transfer, it does not matter that whatever the thickness in relationship to the λ_{mfp}.

Of course, λ_{mfp} it depends on interaction cross-section the similar concept, so here also if the Z increases then λ_{mfp} will be decreasing, right. So, for example if you can see that λ_{mfp} = 5 nm for gold which is very heavy element with Z = 79 and it will be only around 150 nm for carbon which is very light element.

That is because you have more number of scattering centers now and more amount of, so the heavier elements means you have more interaction cross section more, the scattering can be more, much more, much frequent and definitely according to this relationship that will reduce λ_{mfp} as well.

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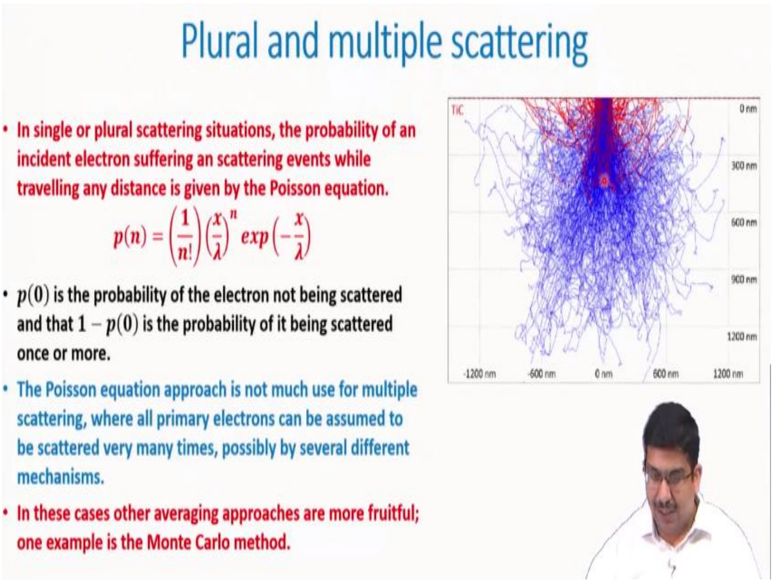

Now, this is what I said to single scattering is desirable. If you do not have that, then if you have plural or multiple scattering; in case you have a plural scattering, you can express the probability of having such scattering event by this relationship again p(n) =

$(1/n!)(x/\lambda)^n \exp(-x/\lambda)$. If the thickness is x, if the distance is x, you will have this much probability for a plural scattering.

Of course, p(0) here is the probability of the electron not being scattered at all, if there is no scattering at all, p(1) of course will be single scattering and so on and so forth. Now, this Poisson equation is not valid after a couple of interactions. So, you will find that if we use this equation and try to predict the path of the electrons, you will find that after a couple of interaction, does not matter elastic or inelastic.

Let us imagine all elastic interaction even after that this equation will fail to predict the path of the electrons. And that is the boundary where you are entering from a plural scattering to a multiple scattering. So, this probability, this Poisson equation based probability for a plural scattering is purely for a couple of number of scattering plural scattering events. When it fails, then you are entering in the multiple scattering range.

So, this is how you can differentiate between these two. And for multiple scattering, you have much better you have to use some averaging approach approaches and the most useful one or most well celebrated method is Monte-Carlo method, we will discuss about that. This is one example of the Monte-Carlo method where all these lines are tracing the trajectory of the electrons and electrons undergoing multiple number of elastic and inelastic scattering.

And finally, whatever the trajectory, so each of these lines is basically finding the trajectory or tracing the trajectory of the electron that is how Monte-Carlo simulation is usually done. The different colors are for different types of electrons. I will come to discuss more about this multiple scattering and Monte-Carlo simulation.

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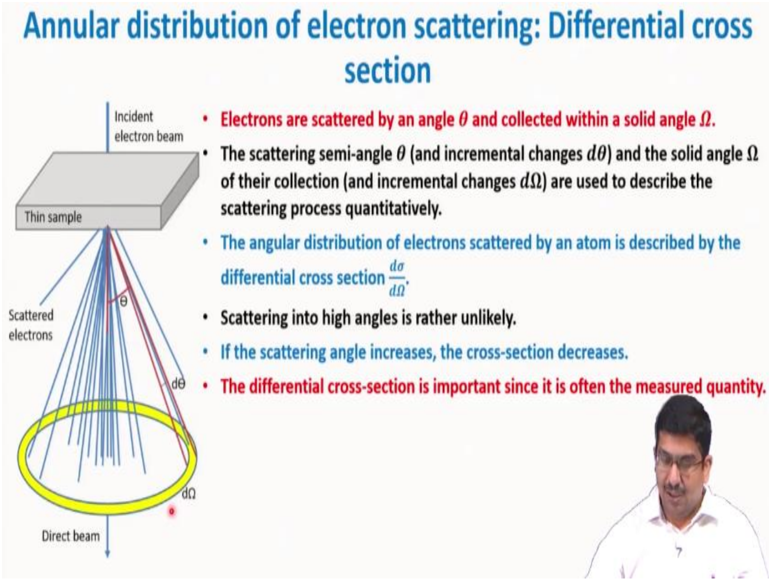

Now, another important topic that we need to discuss issues is called the annular distribution of electron scattering, differential cross section. So, differential cross sections this is a real quantity, just understand the difference. Differential cross section is different from all the interaction cross section in terms that this is a measurable quantity. This is not something like a probability.

This is not an imaginary or concept that gives you finally the probability. It is something that you can measure. Now, how it is defined? Basically, electrons are scattered by an angle theta and collected within a solid angle sigma. So, if you keep a detector here, somewhere over here if you keep a detector, then this theta and in terms of d theta that is the incremental change basically in dθ.

So, these amounts of electrons you can collect within a solid angle of sigma with again an incremental change of d sigma. So, d sigma is the range over which you can collect this theta and the plus minus d theta amounts of scattered electrons. So, then the angular distribution of this electron scattered by an atom you can describe this by this differential cross section which is expressed as d sigma by d capital sigma.

This sigma is the interaction cross section again. So, this is the differential cross section. Now, out of this what you can understand is that number one scattering in high angles is very unlikely. So, if the scattering angle increases first thing, scattering angle increases of course this cross section also decreases. So, differential cross section decreases and this is a measured quantity, you can measure this d sigma.

You can measure the sigma solid angle and d sigma that change both you can measure by putting a detector. This is a measured quantity. So, from there what you can basically get is d theta and you know the scattering angle, you know the solid angle over which you are measuring, you can make an idea about the interaction cross section and mean free path that is the importance of this differential cross section.

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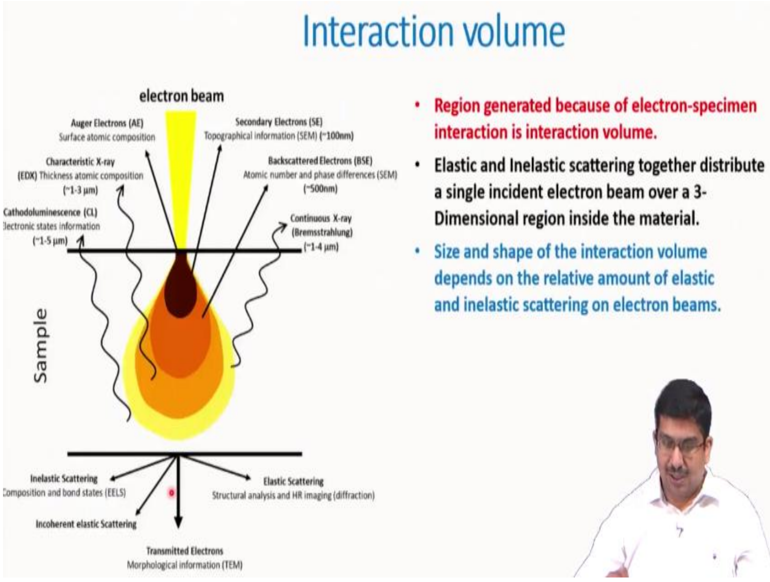

Now, we are coming to another way of expressing this electron material interaction and that is interaction volume. Now, again I am saying this that this interaction volume concept is a real thing. It is not an imaginary or for something like it is not used. It is not a concept to calculate the probability like interaction cross section. This is a real quantity interaction volume, which is an experimentally observed, we will see that.

So what the way you can define this is basically it is a region, TEM or SM does not matter, within the specimen region over which this electron specimen interaction is happening that is what the interaction volume that is it. This interaction volume will contain both elastic as well as inelastic scattering and this is a three-dimensional quantity. It is a three-dimensional region, 3D region inside the material and the size and shape of this interaction volume this is very important.

This depends on the relative amount of elastic and inelastic scattering on the electron beams so that will dictate the size and shape of this interaction volume. And if you measure this, as I said this is experimentally observed, so if you can somehow predict this you can basically go back and you can find out the relative probability of elastic interaction and elastic interaction to certain extent.

So, this is one of this example of interaction cross section which occurs mostly in case of scanning electron microscope because of the inelastic scattering events. And already we have discussed about this various type of signals that is generated; X-ray signals, different types of electrons, Auger electrons, secondary electrons, characteristic x-rays, cathodoluminescence and so on.

What you can see is that they are produced from different depth as well as the width different depth means this side in the Z direction as well as from a different size of this. So, all of these different signals have their own interaction volume. So, if we know this exactly from whichever depth they are produced, we know what size of the interaction volume.

And ultimately what does this interaction volume do is that even if you are beam size is this much, they will finally decide your beam size from the region from which this entire specimen or entire signal is coming. So, this is very important to calculate finally your resolution in your material particularly in scanning mode because your beam may be falling over here and you are thinking that this is the beam size, this is a spot size.

So, this is what the final resolution is and then next you are putting a beam again here, but actually it is the interaction volume over which the signal is generated. So, if you are putting a beam just next to it, then there will be certain amount of overlapping between the interaction volumes. Of course, you will get a duplicacy in this signal and that will not be the resolution, your resolution will be much higher than that.

And particularly you have to put this beam in such a way that this interaction volumes do not really overlap, then only you can distinguish certain features here, over here these two features can be distinguished. If the signals are overlap, you will not be able to distinguish two features. That is what the primary importance of interaction volume. And as I said the different type of signals have their different interaction volume.

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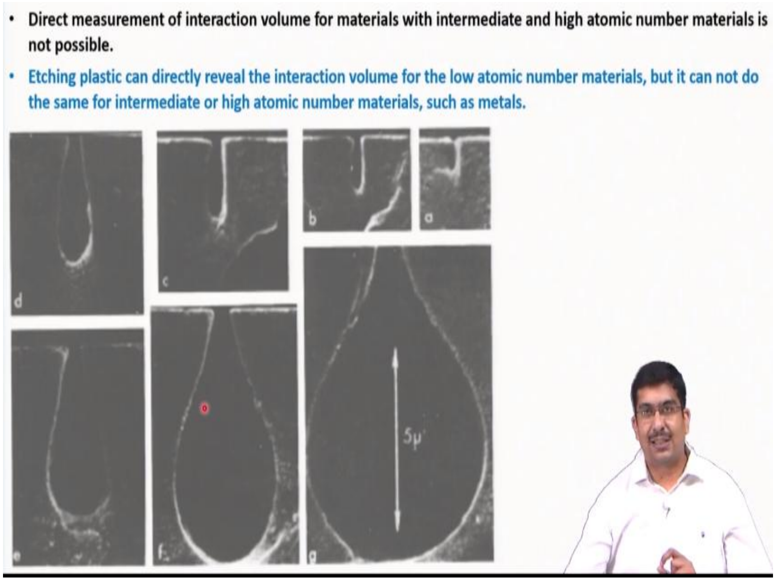

So, now as I said that this one interaction volume is definitely some measurable quantity. You can see it, you can observe it; but unfortunately for heavier elements, high atomic number elements like ceramics, like metal, you cannot see it really. You cannot directly observe the interaction volume from the specimen, neither before nor after the interaction. I mean the when the electron beam falls on that after that if you see, you would not be able to see.

But for certain materials very low atomic hammer materials, for example some polymeric materials containing carbon, you can very well see this kind of these regions which basically tells you from which these signals are generated because remember I told you that most of this finally inelastic interaction ends up in heating the specimen. And in this heating process what happens is that carbon just burns out.

And wherever this interaction happens, inelastic interaction happens, the heat is produced from there and those regions the carbon is evaporated and that will tell you the interaction volume size and shape of this interaction volume. Now, one thing you can notice here that these are basically different if we go from this side to this side and finally to decide this is basically giving a higher and higher electron energy, acceleration voltage.

One thing you can notice that the size may be changing, size of this interaction volume is changing both in the depth side as well as in the spleen in the cross section, both these directions size is changing, but the shape is not changing. Shape remains quite the same and that is what the speciality of interaction volume. So, this schematic it is not drawn arbitrarily, it is drawn based on this kind of observation that is why I said interaction volume is a real thing.

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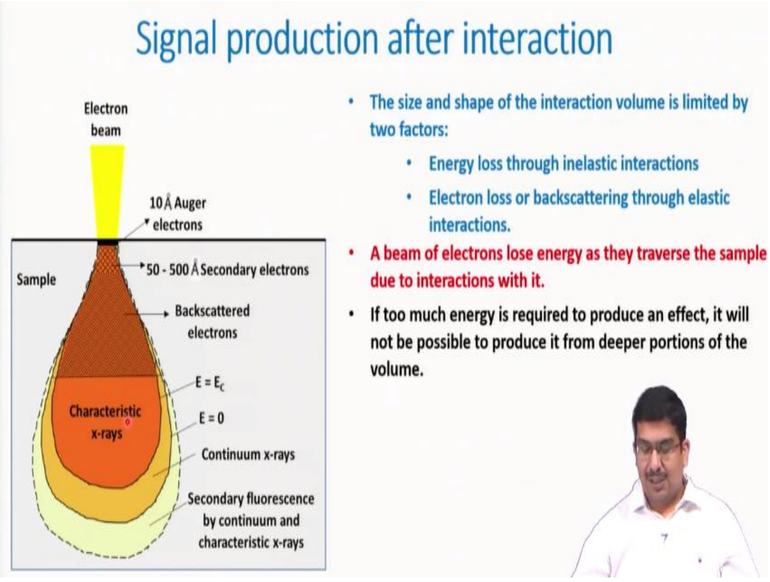

Now, we can see how those signals are produced after interaction and from which depth and so on and so forth. So, here you can see that different types of signals are generated from different depths and this is in case of scanning electron microscope in case of mostly inelastic interaction. So, Auger electrons are developed almost from the surface, a slightly higher depth is for secondary electrons, backscattered electrons even higher a depth.

X-ray signal is generated at much deeper regions of the specimen and then the continuum x-ray and the secondary fluorescence and all those signals are generated at even higher depth. Now, the size and shape of this interaction volume obviously depends on the energy loss through the inelastic interaction plus the electron loss or backscattering through elastic interaction.

So, this interaction volume basically represents both elastic and inelastic interactions here. And of course, we have already known that as this electron beam is traveling through this interaction volume it will continuously lose energy mostly by inelastic scattering. Now, if any of these effects the inelastic events, inelastic interaction any of the signal, secondary signals that is generated, if that is energy consuming if it consumes large amount of energy.

That means if too much large amount of energy is required to produce any effect, then what will happen is that it will be mostly a surface event. That means because the electron beam carries maximum energy where it falls on the specimen as it moves. As the electrons moves deeper and deeper within the specimen, it is continuously losing the energy, continuously inelastic scattering is happening.

So for example if any of those processes like secondary electron emission, this is a hugely energy consuming process. So, these secondary electrons do not produce from very deep within compared to something like characteristic x-ray that is not that energy consuming and that is why characteristic x-rays are produced from a much deeper regions within the specimen.

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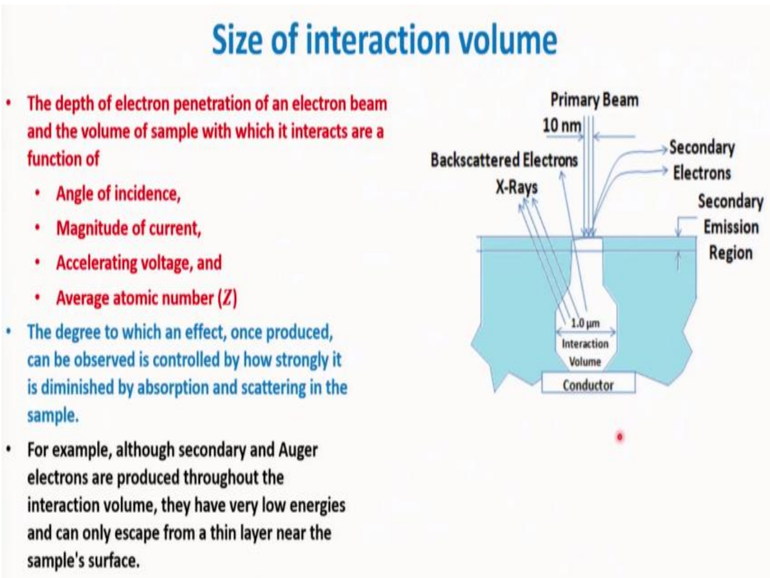

But again, the size and the shape of the interaction volume also depends on some few other factors, for example angle of incidence. So, whether the beam is falling directly at a 90^{o} angle to the surface I mean directly perpendicular way or it is falling with some angle if it is a tilted beam, so the angle of inclination that will also define or that will also affect the size of beam.

Remember the shape of the beam does not change whether it falls like this or whether it falls in this direction the shape does not change, but the size of course changes to certain extent. Then, the magnitude of beam current that is how many electrons are present within the beam, accelerating voltage that means the energy of the electrons that also depends and of course average atomic number.

The heavier the electrons it is then the size of this one the interaction volume will also be smaller and we will see that in a minute. So, all of these factors basically affect the size and shape of that interaction volume here. Another important point is that these different signals that is produced from this interaction volume that also depends or this interaction volume for any of the secondary signals it also depends on how strong that signal is.

If the secondary signal is of low energy, then even if it is produced from deep within the specimen by certain inelastic scattering, it will not be able to come up to the surface. So, this is another reason why the Auger electrons and secondary electrons that interaction volume is almost close to the surface. They are produced very close to the surface because they are also of lower energy.

That is why even if they are produced from deep within the specimen or deep within the interaction volume, they will not be able to come up to the surface all the way and they will be all again this is reabsorbed within this interaction volume itself. So, that is another factor which controls the interaction volume for different type of signals produced.

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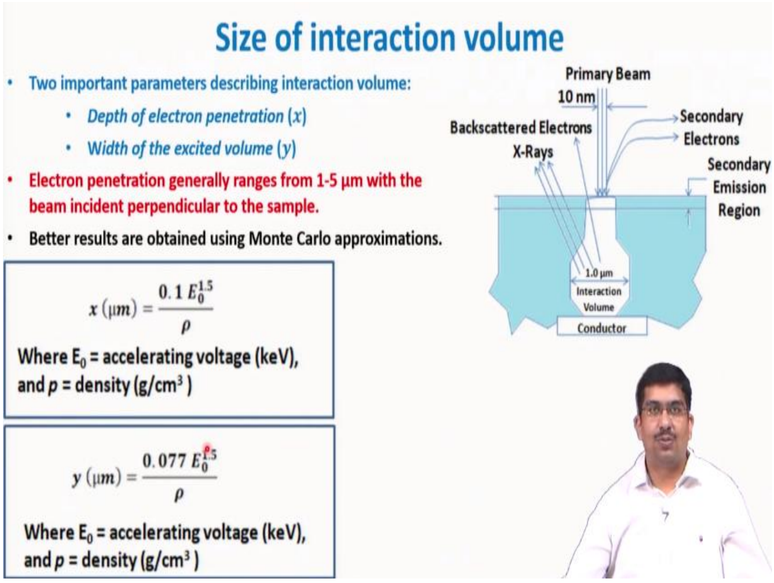

And this also we will quickly discuss the size of the interaction volume and the size of the interaction volume two things are very important here. One is a depth of electron penetration (x) that is what depth that is usually expressed by the maximum depth. So, if you have this interaction volume, so the maximum depth finally where it ends the interaction volume to the surface. So, this depth is the depth of electron penetration.

And width of the excited volume (y) or width of interaction volume means the size at the maximum where the interaction volume is maximum in the width direction in the cross section, whichever place it is maximum in size that width. So, these are the two important parameters that usually describe the electron or the size of the interaction volume. And this you can calculate from this simple equation.

I am not going how these are derived and all. So, x the depth of penetration and of course as I said it depends on the accelerating voltage or rather the energy of the electrons and also the mass, mass here represent the density or the atomic number also in some sense. So, this depends on this, again here also the depth or the width also depends on the accelerating voltage as well as the density.

And this usually electron penetration if you calculate for most of the metals, most of the elements this electron penetration this maximum depth comes out to be 1 to 5 μm. So, when the beam is perpendicular to the surface that is what the best approximation that you get.

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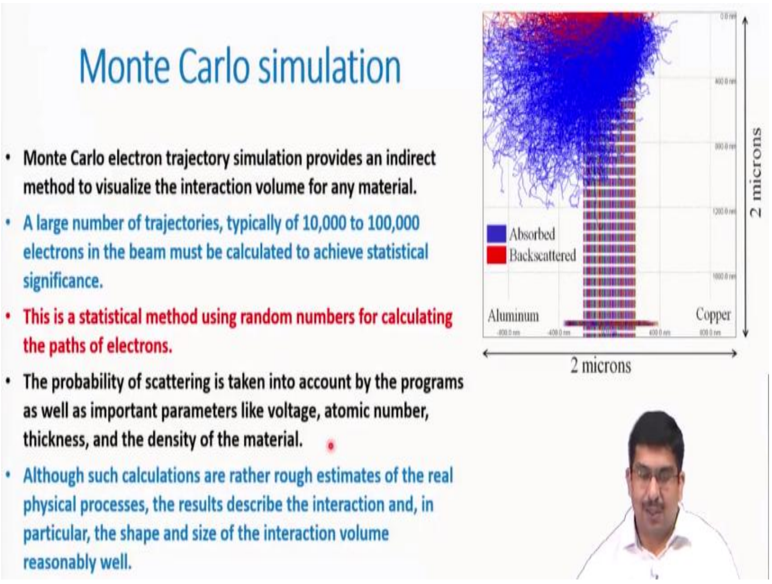

Of course, this is not very much close to the reality that we used to see, we tend to see, and that is why much better results are obtained using Monte-Carlo approximation or

Monte-Carlo simulation, which we will be discussing, but in the next class. So, for now thank you and goodbye.