Techniques of Material Characterization
Prof. Shibayan Roy
Department of Materials Science Center
Indian Institute of Technology – Kharagpur

Lecture – 17
Modes of TEM (BF & DF)

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. We are now in fourth week module 4 and we were discussing about transmission electron microscopy. So, in the last class we discussed about the basic imaging of contrast formation in transmission electron microscopy how the fluorescent screen the detector basically how the contrast generates there.

And what is called amplitude contrast, what is called phase contrast and so on. So, what we understand is that in transmission electron microscope mostly where we use a very thin specimen where we tend to have elastic interaction mostly elastic interaction and in that case it is primarily phase contrast that is what at least at high magnification it will be primarily phase contrast.

The phase changes after scattering, because of the scattering, the phase of the electrons that changes and in the interference this is what produced difference between different regions of the specimen. So, today we will be continuing that discussion and we will be discussing about various different modes at least two important modes of doing transmission electron microscope. One is called bright field mode, one is called dark field mode very similar to optical microscope and then there will be some other modes as well which we will discuss in the next class.

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So, as I said the topics covered in this lecture will be bright field transmission electron microscope and in that we will be discussing something called mass thickness contrast and then atomic number contrast and then we will be discussing about dark field imaging and in dark field imaging there will be another special type of contrast we will discuss that is called diffraction contrast.

But basically what you should understand and remember is that all this sources of contrast equally applicable in dark field and bright field imaging. So, this just like what we discussed during optical microscopy I told that reflected and transmitted lights are two modes, two configurations and all other different ways of image formation like phase contrast microscopy.

Fluorescent microscopy, interference contrast microscopy all of these were contrast enhancing mode which is equally applicable to a reflected mode microscope and transmitted microscopy. Same thing here that bright field and dark field are two different ways of imaging and these general contrast formation mechanisms are valid for both of them, but how what is the change that we will be seeing.

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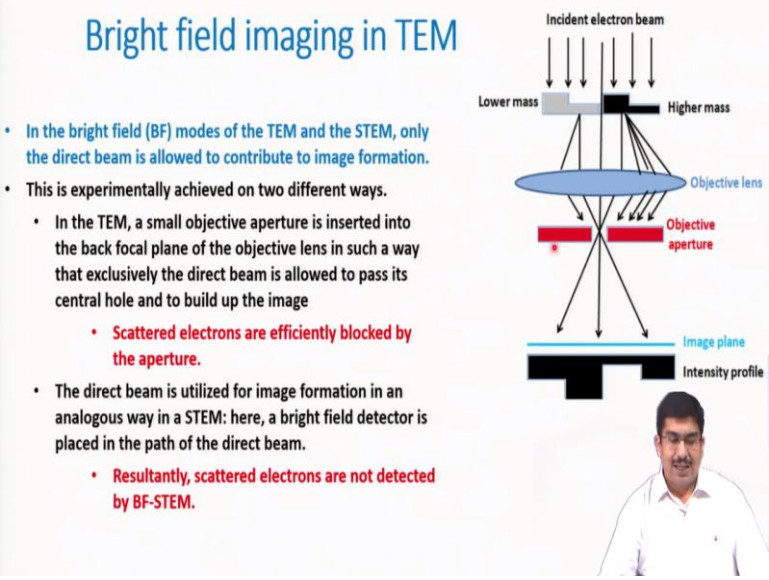

So, first thing is of course the bright field imaging and as the name suggests the bright field imaging means what we do is we take the direct beam. So, if you imagine that you have an incident electron beam which passes through the material and after that it goes through the objective lens. So, we bring what we do is that we bring an objective aperture and only allow the direct beam to pass through.

And direct beam to hit the fluorescent screen that is what we do it here. We stop all other kinds of beam mostly we stop all other diffracted beams within a reasonable limit of course because even with the direct beam also there will be some amount of scattered beam will be mixed with it because we cannot have very small aperture due to the intensity problem we need adequate intensity in order to form the image in the first place.

But as much as possible we will be just using the direct beam close to the optical axis those beams we will be using for this mode bright field imaging mode. So, this is done the bright field imaging mode is done either you can bring a small objective aperture which will basically block all the scattered electrons and in the process it will just allow the direct beam exclusively it will allow the direct beam through the central hole and that will form the image that is one way.

A slightly different method is used in case of STEM which is scanning transmission electron microscope there the beam is made to raster over the specimen for a better resolution and here a bright field detector is placed in the path of the direct beam, method is basically almost

the same there also there is an objective aperture is introduced and here also the scattered beam is restricted by the objective aperture.

So, in both the method we just use the direct beam for imaging that is why it is called bright field imaging mode.

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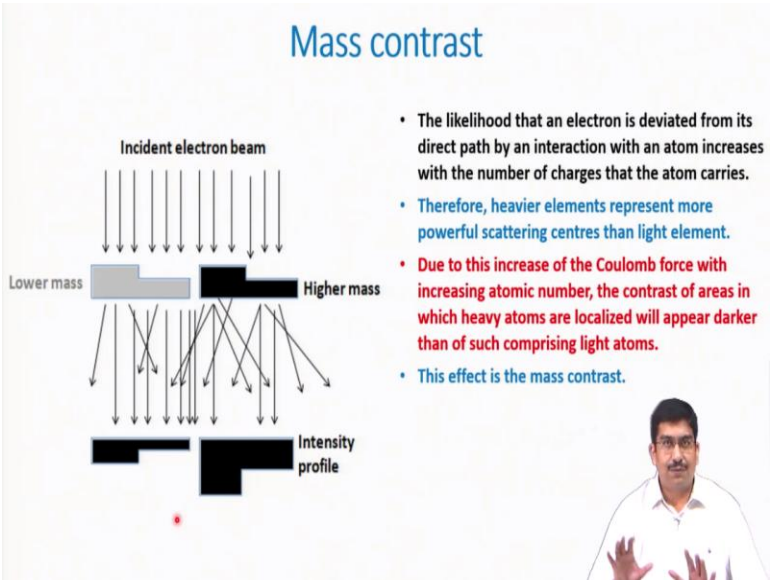

And this is possibly what when you start a TEM if nothing is selected this is by default this will be mode of imaging, the bright field mode. Now what forms the contrast? As I already said that we are discussing purely based on phase contrast here amplitude contrast we are not considering because that is mostly coming out of inelastic scattering where at least in this the context of this contrast formation in whatever we are discussing now.

And in the next few classes it will be purely elastic interaction no change of amplitude mostly it is phase change. So, in that case what we will have is the incident electron beam which is of same amplitude coherent beam, very, very coherent beam and it hits the specimen. Now what happens is the specimen of course will undergo some elastic interaction and scattering will happen depending upon the electron cloud.

And the positively charged nucleus they will scatter, they will change the direction that will be a phase change. What we can take it as a or we can imagine this that from the direct beam if some beams or some electrons are getting scattered. So, they are deviated from the path of the direct beam. So, if I now capture the direct beam I can imagine that whatever number I started here.

Let us say I started with 100% here and in the scattering process 10%, 20% is lost, lost in the sense their path is changed they are now scattered beam and the objective aperture just restricts them. We can imagine in that way also then I start with 100%, but I ultimately get 90%, 10% is lost in scattering. So, what I have to see is what influences this scattering, what are the features in the specimen that can influence the scattering process.

So, in this final event how many of those scattered electrons will be lost because of the some certain characteristics in the sample itself that is what I have to check here that is the first source of contrast formation. So, what we can think is that the mass of the material that will dictate the mass or density of the material will dictate mass, density, atomic number whatever you can say that we will dictate the number of or if I have this difference in the specimen.

In the material I have a difference in mass because of the density, because of the atomic number whatever I have a difference between different regions then that can be utilized as a source of contrast. How, that means if something has a more mass something is more denser then on the path of this direct beam it will produce that part of the specimen will produce more number of scattering centers.

That means more number of elastic interactions I can imagine that more amount of electrons will be lost from the direct beam corresponding to that path that region compared to some other region let us say we have so now that is what it is shown we have lower mass a region next to it there is a region of higher mass. What will happen that higher mass region will have more number of such scattering centers.

Interaction cross section will increase and in that process more amount of scattered beam will be produced and more amount of electrons will be lost from the direct beam compared to some regions which is lower mass. Finally, what will happen, this direct beam if I take it somewhere over here and use it for imaging. Here in the higher mass region I am having a direct beam which is much weaker than this lower mass region where scattering is less.

So, if I star with 100% of electrons here due to scattering let us say in this higher mass region 20% is lost, 20% is scattered and in lower mass region 10% is scattered. So, ultimately here if I capture the direct beam I will get 80% of the direct beam here I will get 90% of the direct

beam and since this is a phase contrast. So finally I can imagine that the amplitude or correspondingly the intensity when those electrons hit the fluorescent screen.

These regions if I capture direct beam again this regions there will be less number of electrons finally hitting the screen and this region there will be more number of electrons finally hitting the screen. So, ultimately this regions will appear in darker contrast, lower mass regions will appear in brighter contrast that is how the contrast first source of contrast will be purely because of the mass density atomic number difference and that is the first effect and this is called the mass contrast effect.

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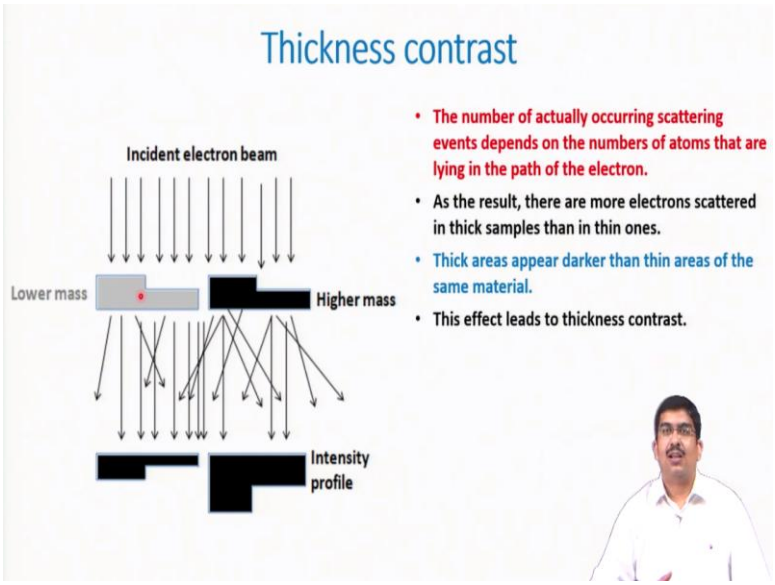

Now along with the mass contrast effect we can also imagine some other kind of contrast basically the same concepts, but that comes purely now because of the thickness of the material. So, let us imagine that the density may be same so I am just considering let us say higher mass or lower mass region same region, but some place it has higher thickness, some place it has lower thickness.

So, I can imagine that having same density this higher or wherever the thickness is more that region will have more number of scattering centers compared to this region which is having less number of scattering center the density is the same, but the number is varying here, scattering center number is varying purely because of thickness. So, same effect will happen this regions more number of scattering center.

More amount of scattering then much more weakening of the direct beam compared to regions which are thinner correspondingly in the image what we will get this regions which are thicker they will have darker intensity compared to this regions which are thinner. So as such for mass this regions will be darker. So even with darker also that dark contrast compared to this one what we will have certain regions will be even far more darker compared to thinner regions.

Same effect will happen here also in these lower mass regions. So on top of the mass thickness or mass contrast now we will be having a contrast coming out of purely thickness and that is called thickness contrast.

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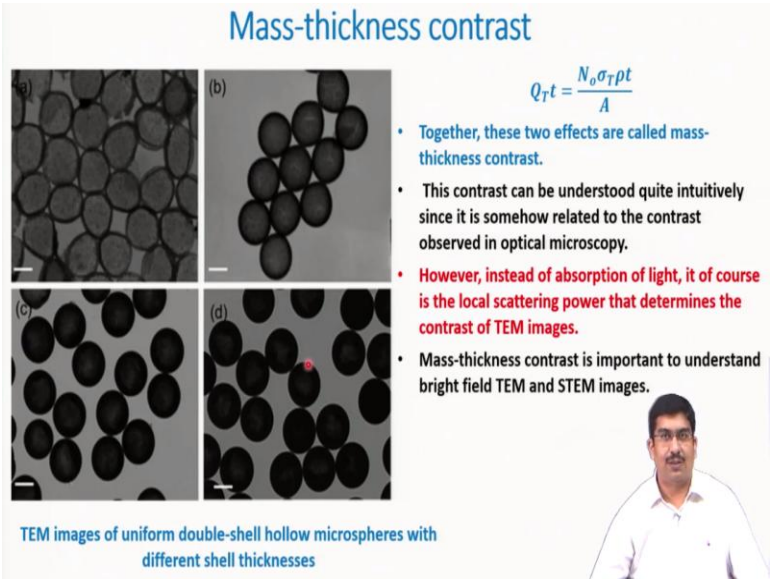

Together these two are called mass thickness contrast. Now, if you go back to this equation which was giving you the interaction cross section so this was the total interaction cross section and we were introducing the sample thickness so that we can get the complete three dimensional interaction cross volume or cross section or cross section extended to three dimensions that is what we can imagine.

That will be depending on this rho t which is the mass thickness contrast. So, this is the same effect now coming here because of the mass and thickness difference we will have a source of contrast in the final imaging which is called mass thickness contrast in this material and in bright field material, but the same thing can happen in dark field which we will discuss later when we discuss about dark field imaging.

So this is the first source or inherent source of contrast formation in case of a transmission electron microscopy. The first thing you will get is the mass thickness contrast in your material. So, now the same thing we can imagine for like in optical microscope also, but in case of an optical microscope the local scattering power will be very low and there the same effect basically the same effect happens there because of the absorption of light, because of the higher mass and higher thickness there it will sort of introduce more absorption of light in case of light signal.

More absorption from higher mass and higher thickness regions that is why ultimately in the final if we capture the direct light in the bright field those regions in the optical microscopy also they will appear in darker contrast in this case of transmission electron microscopy this will purely come because of scattering not because of absorption because of the scattering same effect.

Now, if you understand or if you see this images which is shown here. So, this images is from a double shell hollow microsphere. So, these are small tiny spheres the inside of this it is a hollow it is like inside there is nothing and what we have is this cell walls here. So, if you look at here definitely the cell walls are having higher mass compared to this inside region both higher mass and possibly even higher thickness also compared to inside region.

So, if I capture the direct beam if I just now think about the direct beam the direct beam that passes through this regions which is of lower mass and lower thickness both less amount of scattering will happen from this regions and of course this if I capture the direct beam for imaging this regions will appear in brighter contrast compared to the cell wall regions which will have much more thickness and mass both.

So, finally they will appear in darker contrast. So, here it is purely mass thickness contrast. Now, if the cell wall thickness is increasing if you look at here this is progressively the cell wall thickness is increasing and this final one is cell wall is almost completed it is not a hollow microsphere any longer it is almost like a solid particle. In this case you will see that the entire region is almost giving you the same contrast.

There is no such two different contrasts from this material and it is continuously diminishing here. So, here; it is a complete dark contrast which is coming and if you look at this dark

background. So, now this dark background is also completely here you can imagine that there is nothing in this regions that is why this also appears in a much brighter contrast and in the final case you will only have these two regions.

One which does not have any material so low mass and thickness brighter region this is where there is higher mass and thickness so in bright field contrast. So, this mass thickness contrast this is how you will see that mass thickness contrast is progressively changing with increase in the thickness and mass, with increase in the cell wall thickness so that is how you can understand. So, this is very inherent contrast formation mechanism in case of TEM.

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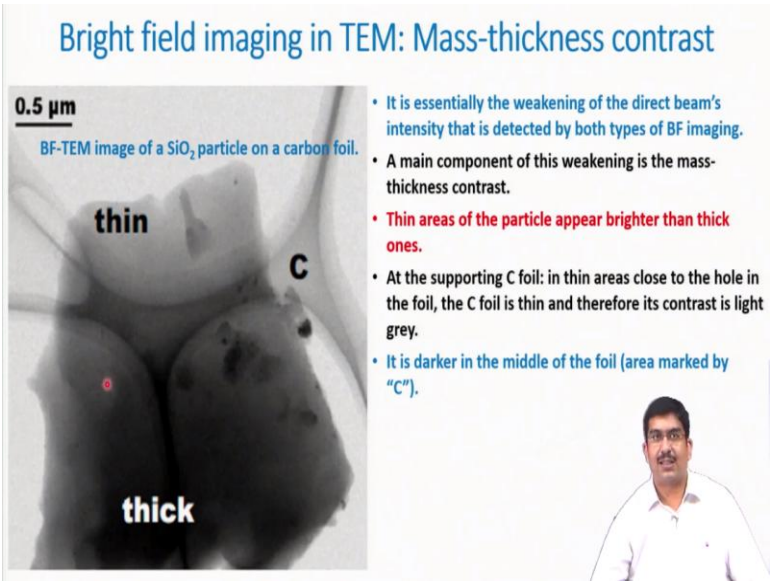

Again another example that you can see here the bright field TEM image of a silica particles sitting on a carbon foil. Okay, And this is the carbon foil first of all. So, carbon foil definitely this does not have any silica on to it. So, this region is obviously of lower mass and lower thickness that is why these regions are the brightest number one first of all this carbon regions is white, white bright here.

The next thing of course you will have this region where a thickness is much higher not only the mass the thickness is much higher in this region compared to this kind of this regions. So, these regions thicker regions will have much darker contrast because again the same reason, more scattering and more amount of electron is lost from the direct beam. So, this region appears in darker contrast thicker regions and these thinner regions appear in much brighter contrast. So, again it is showing more or less a pure thickness mass thickness contrast.

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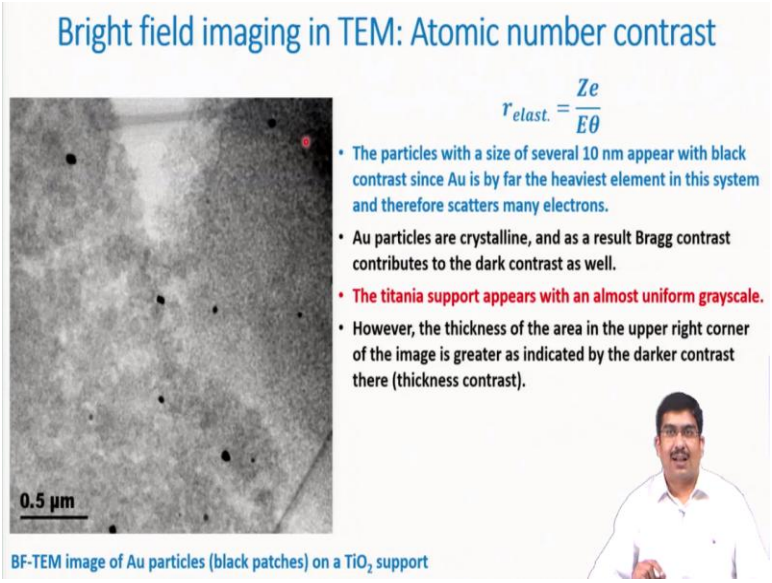

The next source of contrast comes because of atomic number difference. So, this also we have discussed when we were discussing about interaction cross section. So, interaction cross section remember this equation where the size of the interaction cross section strongly depend is directly proportional to this atomic number it also is proportional to this energy of the electrons, but for that case as we already assume that the energy remains the same for all the electrons which are coming.

So, here the interaction cross section is directly related to the atomic number. So that means what happens is that the regions which are having higher atomic number regions will present higher interaction cross section, interaction possibility will be much higher scattering possibility will increase for higher atomic number regions within the specimen that means those regions there will be more number of scattered electrons that will be lost from the direct beam.

So, if I now capture the direct beam what will happen is that the higher atomic number regions will appear in darker contrast because they scatter more that is it compared to regions which are having lower atomic number this is purely atomic number, this is nothing to do charge center or so. This is purely from the atomic number difference this contrast is coming.

For example you can see this regions these two regions which is a bright field TEM image for the gold particles on the Titania support the same one that we were discussing I think in the last class about what we were discussing about the contrast purely how contrast happens. So,

in this case first contrast thing you will notice is this that there are some regions which are completely dark which looks like a particle something like a particle this is complete.

There are darker regions here also I am not going into that we will come back to this, but there are definitely certain regions which looks like a particle and those particles are definitely in very dark contrast and this dark contrast comes because of the atomic number. Gold is having much higher atomic number compared to the Titania which is everywhere else the Titania.

So, these regions more scattering will happen the direct beam will be weakened when we capture the direct beam less number of electrons are hitting the screen those areas appear completely dark. Now the problem is on top of this if you just look at this Titania support here this regions ideally if it is pure atomic contrast. Ideally, this should have produced an uniform grayscale contrast, but that is not happening even in this regions you will have certain regions which is very bright, certain regions which is very, very dark.

And in these dark regions you will see that there is one tiny gold particle here. Now this dark and bright contrast is coming because of mass thickness contrast. So, these regions are very thin or almost negligible. It is almost at the edge of the specimens where a hole is possibly produced and these regions is therefore producing no scattering it is not offering any scattering from the direct beam this regions will appear in a very bright contrast.

These regions possibly are much more darker and those darker regions will produce of course much more thickness, higher thickness and they will definitely produce very dark contrast here. So, it is sometimes very difficult to identify or to separate the atomic number contrast from the mass thickness contrast. So, as I said mass thickness contrast is inherent it will be just there in the material depends on because if not mass at least thickness will definitely vary.

Even whatever technique you used to produce the TEM sample then still you will have some difference in the thickness. So, it will definitely show some thickness contrast that will inherently be there and at times the thickness contrast will be almost as strong as the atomic number contrast and it will be very, very difficult to find out what exactly is producing this contrast.

And that is why many a times I tell this to my students many a times the students and more of that we will discuss when we discuss about other sources of contrast generation. Many a times I tell them that; so many different source of contrast formation is merged in the TEM sample that it is nearly impossible to understand TEM images off line. If you do not know exactly what is there.

So, this is best understood when you are sitting or when you are doing the transmission that imaging then you know exactly what is forming or is your contrast you know the sample condition, you know what are the chemical composition of the samples all of this things if you know then possibly you can differentiate between atomic number contrast and thickness contrast.

If you just look at an image if you show it to someone it is very difficult or almost nearly impossible to find out that exactly what source of contrast generation is operating here is active here. So, that is why when you take a TEM image then you should be very sure that exactly what is producing the contrast right there otherwise afterwards it is very difficult to find out like this you can imagine this case.

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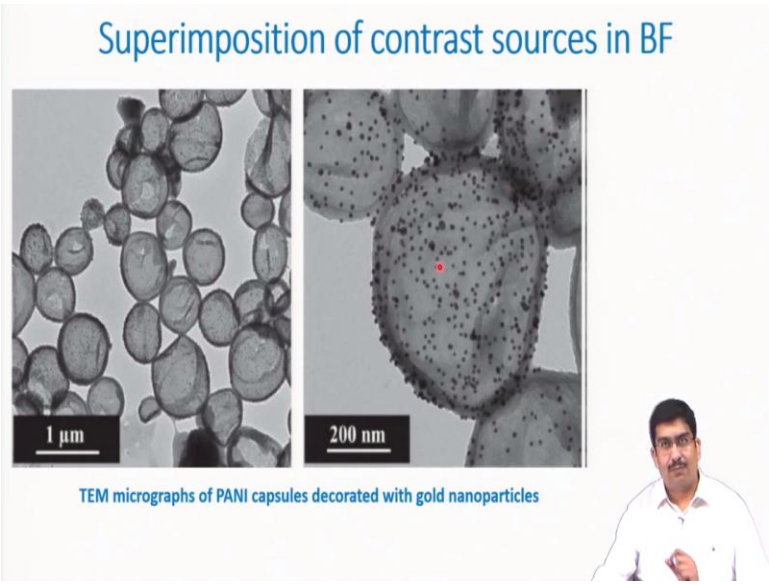

So, more of this as I said superimposition of contrast sources in bright field so this is a TEM micrograph for again a particles or hollow microspheres, but this time with gold nanoparticles inside them. Now, if you look at this images you will see that even in low magnification and high magnification you have the small dots block dot complete black dots and those complete

black dots obviously belongs to gold because that is purely coming out of atomic number contrast gold is scattering more in the direct beam.

So, those regions electron is less in the direct beam and finally that is giving a black contrast or dark contrast completely. Now the other regions you will see that they will if pure atomic number contrast they should appear brighter, but even within them also there is a difference. So, you have these cells walls which are much darker than inside that. So, most you can see in fact four different type of contrast which area which is just the support there is nothing.

So, this area is the brightest then the next regions is here which are less compared to this regions and possibly having lesser thickness also just slightly thicker than this so slightly having some mass than that. So, these are little more darker than this region the support regions and then the next regions you see is this cell walls and those cell walls is now having more mass and more thickness.

So, they are darker than this region or this region and finally you have these gold nanoparticles which are again completely dark. Now, if I zoom this out and sometimes I do that for my students and I ask them do you see a difference in contrast between this gold nanoparticles if you look at here you will possibly see that even within the gold nanoparticles you have little difference in the contrast that is coming again from the mass thickness contrast.

So, even the gold nanoparticles are not having a uniform thickness that is why they appear very dark, but within the darkness you can possibly; if you look very carefully you can find out this mass thickness contrast as well. So, mass thickness contrast is very inherent that is the primary sources of contrast formation in case of transmission electron microscopy on top of that all other kinds of contrast formation mechanisms work on top of that. So always please remember this.

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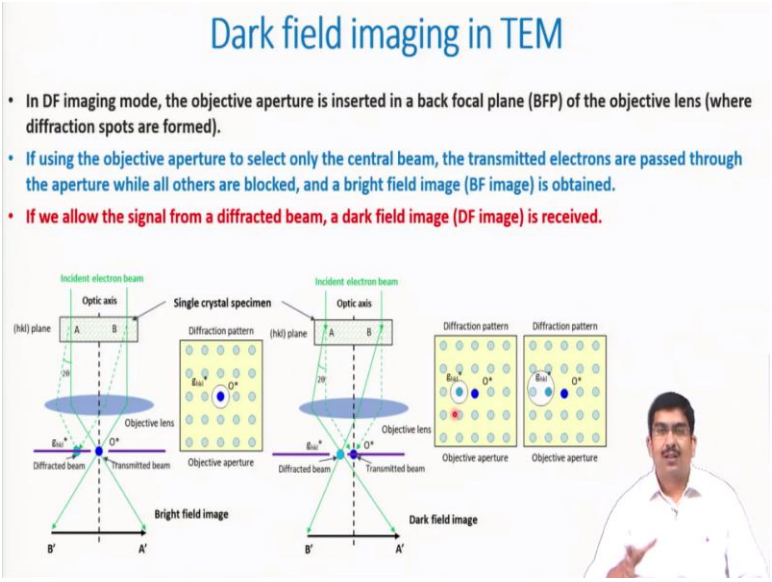

So, the next method is dark field imaging in transmission electron microscope. In dark field imaging as the name suggests and a similar you can draw an analogy like the optical microscope. What you do in dark field imaging basically you bring an aperture in the back focal plane of the objective lens where basically you get the diffraction spot if you bring an imaging if you image this back focal plane of this objective lens.

You will be basically able to see the diffraction spots, all the diffracted beams. So, now what you do is that basically instead of the direct beam when you use a direct beam when the objective of aperture is just allowing the direct beam. So, if you look at this diffraction pattern for that example if you imagine that you are just imaging this back focal plane. So, what we will have is the central beam which is the direct beam.

And then you will be having all this; diffracted beam. Diffraction is a special type of scattering for that just for now just understand this the diffraction is a part of the scattering where constructive interference happens along a certain scattering angle that much is enough for now. We will have a discussion about this later. So, this is the direct beam and these are the diffracted beams here.

So, when your objective aperture is selecting the direct beam then it is a bright field image. When it is selecting a diffracted beam other than direct beam any other beam other than direct beam then it is a dark field imaging. So, the aperture now what it is doing is that it is stopping the direct beam here and it is just capturing this diffracted beam and that can by any diffracted beam.

In the entire diffraction pattern it can be any one of them and all of them are called dark field imaging. So, that is the difference between bright field imaging and dark field imaging. It is purely which of these beams you are using for imaging whether you are using the direct beam that is the bright field imaging, whether you are using a diffracted beam which is dark field imaging.

So, we will stop here today and more about the dark field imaging and the contrast formation in dark field imaging and so on we will be discussing in the next class. Thank you.