**Techniques of Material Characterization**
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**Lecture – 18**
**Modes of TEM**

Welcome to this NPTEL online certification course on techniques of materials characterization. So, we are now in week 4 module 4 and we were discussing about transmission electron microscopy. So, till now we have discussed how contrast generates in TEM transmission electron microscope, amplitude contrast, phase contrast and various sources of contrast generation like mass thickness contrast.

And how those different contrast mass thickness contrast are superimposed and how the bright field imaging mode works and then we discussed about another contrast formation mechanism that is atomic number contrast. All of those discussions was mostly based on our previous discussion about the interaction electron material interaction and about interaction cross section and so on and so forth.

And today we will be going to discuss about some other modes of TEM operating the transmission electron microscope we have briefly started in the last class dark field mode and today we will be continuing from there.

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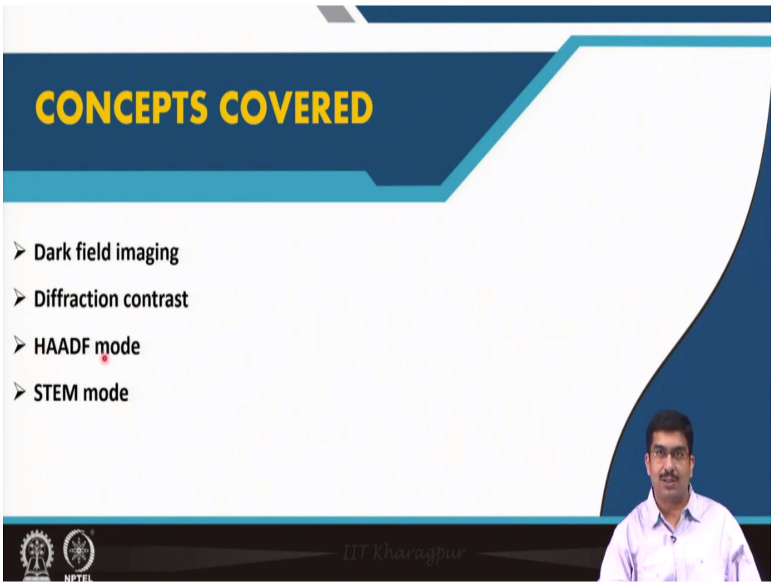

So, today we will be discussing of course about dark field imaging and then a special contrast formation mechanism that we have not discussed yet diffraction contrast and we will discuss about two other modes of working with transmission electron microscope HAADF mode which is a variation of dark field mode and STEM mode that is another very important mode for high resolution TEM work.

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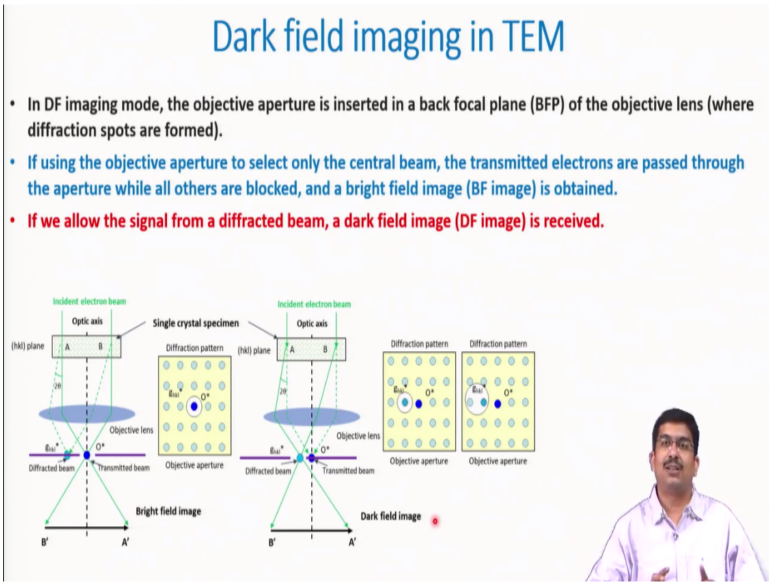


So, dark field imaging this we discussed in the last class, but again we can just begin from here. So, in dark field imaging as I said that in bright field imaging what you do is that you just choose the direct beam for forming an image and there the contrast forms whether the direct beam from various regions how or what number of electrons are present in the direct beam there and in other words that how much or what amount of electron.

What number of electrons are lost due to scattering effect from the direct beam. So, that was forming the contrast there. So, in dark field what we do is that we choose the scattered beam for imaging. So, that scattered beam now works like a direct beam here that is the beam we used for imaging, we stop the direct beam somehow. Usually, what we do is that bring an aperture to the back focal plane of the objective lens, objective aperture and there we stopped the direct beam.

Once we allow direct beam only and stop all the scattered beam it is a bright field imaging and once we bring that aperture and stop the bright field direct beam and only allow the scattered beam then it is a dark field imaging and in specialty in dark field imaging is that we can bring or we can allow selected kind of selected beams or not we can choose between various different scattered beams we can choose by bringing that aperture, by placing that aperture.

So, usually dark field imaging in order to do the dark field imaging the first thing we derive is this diffraction pattern what we will be discussing in subsequent classes then it will become much more clearer. So, we will first derive this diffraction patterns and from the diffraction pattern we can either choose for bright field mode we can choose the direct beam or for the dark field mode we can choose any of those scattered beams.

And once we do that we will be getting a electron beam which is scattered and then that beam will be used for image formation. So, any change in that beam depending upon the features, depending upon the specimen condition any change in the scattered beam now will be the source of contrast generation.

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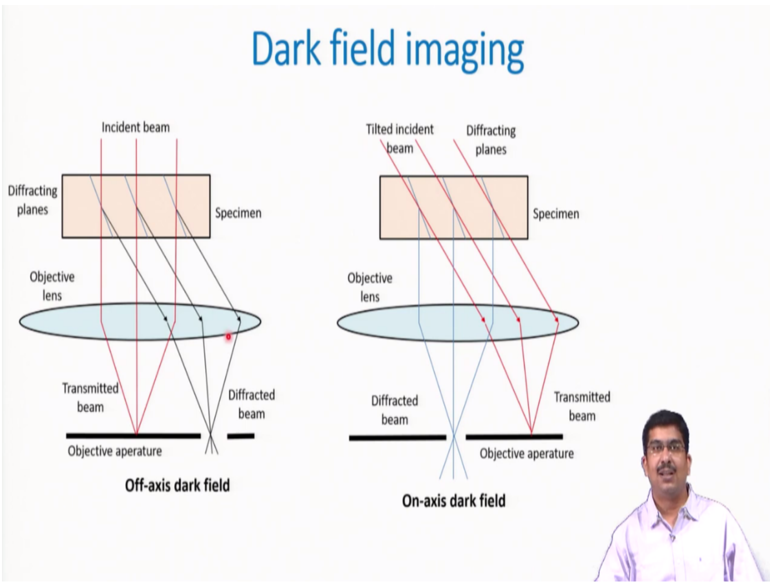


So, there are two ways we can basically do dark field imaging and these two are called off-axis dark field and on-axis dark field. So, what do we do basically in this? So, in off-axis dark beam the incident beam is parallel to or incident beam is perpendicular parallel to the optic axis that is perpendicular to the specimen and it remains always like that. So, what we get is after passing through the objective lens the direct beam is still.

Before reaching the objective lens the direct beam is parallel to the optic axis and gets focused on the optic axis by the objective lens.  Diffracted beam of course since it is scattered and scattered by a particular angle now that beam will be having an angle with the optic axis. So, it is not parallel with the optic axis and when it passes through the objective lens again it will be focused on the back focal plane.

And we will be using that beam only after that focusing it basically becomes or getting focus on the optic axis itself or parallel to the optical axis. So, we bring an objective aperture stop this direct beam and only allow the diffracted beam. So, in this case the difference is the main characteristics is the incident beam is always parallel to the optic axis not the diffracted beam. Instead what we can do in on-axis dark field mode is that we can make the beam, incident beam tilted with respect to the optic axis. So, optic axis is always like this.

Optic axis is always perpendicular to the specimen. So, in this case we are tiling the incident beam in such a way that the diffracted beam now becomes parallel to the optic axis after it comes from the specimen. So, this tilting is not an arbitrary tilting this tilting also is in relation to the angle of scattering or angle of diffraction. So, in this case what happens is that the incident beam or the direct beam is not parallel to the optic axis.

Rather the diffracted beam becomes parallel to the optic axis and finally we get the diffracted beam again here and we use it for imaging. So, in this entire process what happens is that you do not need to do anything with the objective aperture. So, the objective aperture remains the same. So, you can use it like just for bright field imaging. So, once you are interested in bright field imaging keep this objective same aperture.

You just make the beam to come parallel to the optic axis you get the bright field imaging then your direct beam will be parallel to optic axis and then you will be using that for bright field imaging then in order to go to the dark field you do not anything with the objective aperture, do not change it, do not move it you just make the beam tilted or in other words normally what we do is that we do not tilt the beam we tilt the specimen.

So, we tilt the specimen the beam remains still like parallel to the optic axis, but we sometimes we just tilt the specimen in a way that this diffracted beam after diffraction it becomes parallel to the optic axis. So, that is what the difference and what the different ways of doing the on-axis dark field either you can tilt the beam or you can tilt the specimen so that the diffracted beams becomes parallel to the optic axis you do not do anything with the aperture.

In this case the beam neither you tilt the beam not you tilt the specimen you just use an aperture in order to stop the direct beam and capture the diffracted beam. So, these are the two different modes of doing dark field imaging and as I said dark field imaging is something to do with choosing the dark field beam which beam you are interested to use for imaging and there is a reason why we will go through that.

So, you tend to choose a particular diffracted beam it is not any arbitrary beam normally when we go for a dark field mode we tend to choose the diffracted beam and that choice is made out of this diffraction pattern. So, sometimes the aperture is not helpful for choosing some (()) (08:46) obscure beam some particular beam which is somewhere very less intensity or at a very angle or so on.

So, those cases we have to switch to something like on-axis dark field mode the aperture is not enough to give us this selected or the diffracted beam that we want, the aperture is not able to do it then we have to possibly go to on axis dark field mode and vice-versa. Sometimes we choose on-axis dark field mode and then because this tilting in the specimen tilting inside the beam is also very, very limited.

So as the incident beam tilting that is also very, very limited so then we have to go for off-axis dark field mode. So, there is a switch between this two in order to choose a particular diffracted beam for dark field mode.

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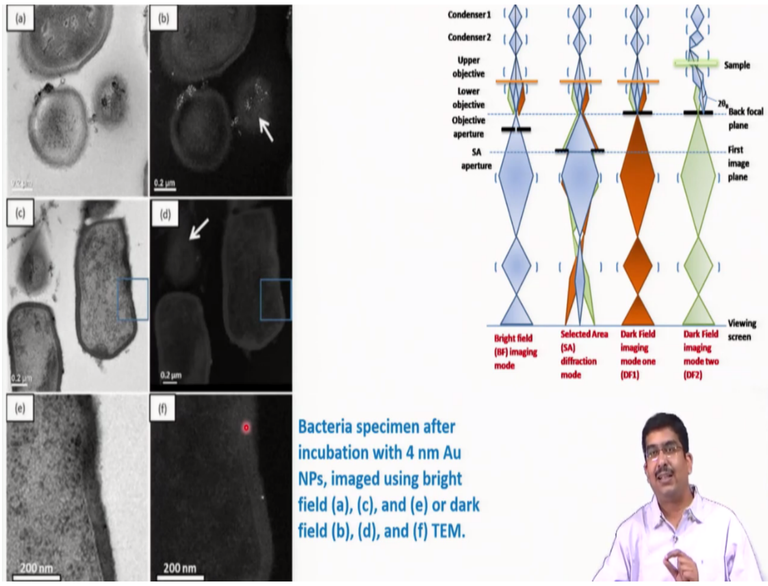


Now, in the dark field mode how the contrast forms? Basically here also the same mass thickness contrast and atomic number contrast is valid. In fact the dark field mode just like optical microscope it is a contrast enhancing mode. What you are doing here is first thing bright field image you are using direct beam so the intensity is very high. So, there the difference between different features is not so prominent may not be so much.

If you can stop the intensity, if you can reduce the overall intensity then the difference also will become prominent. So, that is what we followed for optical microscope same thing we follow here as a contrast enhancing method and in this case also mass thickness contrast and the atomic number contrast will contribute, but in a negative sense then the direct beam or bright field mode.

So, in bright field mode what happens is that the amount of scattering decides what will be the contrast. So, if some region is scattering more either because of the higher mass or higher thickness or maybe higher atomic number whatever. So, if some region is scattering more than in those amount of electrons are lost from the direct beam that will appear in darker contrast.

In this case in dark field mode the same method now the diffracted beam or scattered beam is used for imaging. So, those regions which are scattering more that means either mass, higher thickness or because of higher atomic number those regions are scattering more. So, more number of electrons will be available from those regions. So, ultimately what will happen those regions will appear in brighter contrast in the dark field model.

In the regular dark field mode if you do not choose a particular dark field beam or so then this is how the contrast will form. So, exactly in a reverse sense to the bright field mode if some feature is dark in bright field it will appear brighter in dark field and vice-versa, but since as I said it is a contrast enhancing method. So, of course this case the difference will be much more prominent.

So, this is what it is shown in bright field mode what you do is you use basically the direct beam and then selected area diffraction again dark field mode you can use the diffracted beam. So, you basically use the diffracted one of the diffracted beam you can choose this diffracted beam which one you want just by moving this aperture you can choose which one you want to use.

Now, if you look this images which are taken from the same specimen a biological sample and in bright field mode here and the dark field mode here. So as you see this regions so some regions like here. So, this is coming out to be mass thickness contrast here in this case and this also has a atomic number source of atomic number contrast because it has a nano particles of gold.

So, here let us discuss that first atomic number contrast. So, this high atomic number contrast material nanoparticles will appear dark we have already discussed that because of high level of scattering the same particles now in dark field mode will appear as bright. The difference that you will notice is that in this case this is since this area will appear very, very bright there is nothing to scatter these are almost empty regions no scattering.

So, you are using dark field direct beam that is why these regions are very bright. Here those regions will be completely dark because you do not have any signal there and that is why this atomic number contrast is much more prominent compared to this bright field mode here. Second thing is the mass thickness contrast is also coming out here that this cell region this cell wall regions or rather we can see it here somewhere over here it is much more prominent.

This cell wall regions are appearing darker because more scattering. In this case the cell wall regions also appears darker of course comparatively, but that contrast mechanism is much more reduced in case of dark field mode. You are mostly getting this from atomic number contrast and that is the difference that is the best thing about the dark field mode why you need to go to dark field mode because again as I said contrast enhancement.

You are stopping other or you are selectively stop other contrast forming method. So, this mass thickness contrast in atomic number contrast is always superimposed in case of bright field mode. In dark field mode selectively you can deconvolute this different types of contrast mechanism where the superimposition of mass thickness contrast and atomic number contrast will sometime can be avoided that is the best part of it.

So, for example, you are able to see this dot coming out of pure atomic number contrast which you are not able to see here because of the superimposition of mass thickness contrast. So that is why we sometimes need to go dark field and what I advice personally to all my students is you should always check any image, you are capturing on bright field mode you should always go to a dark field mode corresponding dark field mode and try and capture that.

And then you will be able to see this different features which possibly are not so much prominent in a bright field mode, the same advice is given to anyone who is watching this NPTEL course and will try their hands on transmission electron microscope just try to look at the dark field modes. When you are taking a bright field mode just check the dark field mode as well because you may get, you may miss many of these contrast.

Many of these features because of poor contrast or superimposition of contrast in bright field mode. So, this is how the images are forming in dark field mode the same atomic number contrast, mass thickness contrast everything the sense of contrast is exactly reversed from bright field mode and second thing is that the contrast enhancement is much better here and the deconvolution of different contrast forming mechanism is or are possible in this dark field mode that is the advantage of it.

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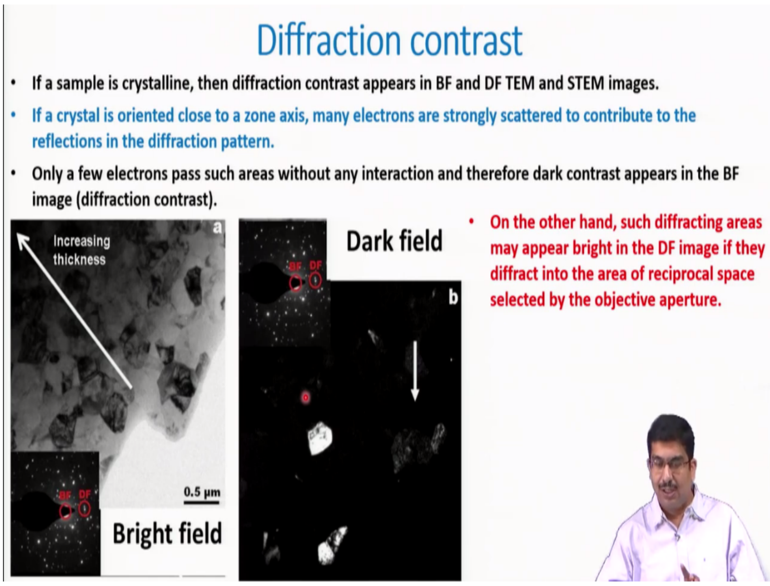


So now another very typical contrast forming mechanism that is very, very important in case of dark field mode, but it is also there is bright field mode of course that is called diffraction contrast and for the time being let us imagine or or let us just understand the diffraction is a special type of scattering we will discussing diffraction within our next class or next to next class.

The diffraction is a special type of scattering happening along certain particular angle or certain particular directions and in that direction the scattered beams get constructive interference. They undergo constructive interference and that is why the scattered beams are very strong on those compared to all of that not as strong as direct beam I am not saying that, but out of all the scattered beams those particular beams along certain particular directions they are quite intense.

So those are the diffracted beams or diffraction condition and this typically happens from a crystalline materials. So, if your specimen is crystalline then sometimes you can get this scattering in form of diffraction and then what will happen is that there will be another source of contrast that will appear in the bright field mode as well as in the dark field mode. How it happens is that basically when a crystal is oriented close to its zone axis we do not need to think what is zone axis and at this moment.

Just imagine that something is happening to the crystal it is oriented favorably so that scattering is happening there in form of diffraction. So, those crystal certain crystals not every crystal in this crystalline material are undergoing diffraction only certain crystals are undergoing diffraction. If that happens then those crystals or if it is a polycrystalline material let us imagine this is a polycrystalline material is basically zirconia this is a polycrystalline material.

And in this polycrystalline materials the grains or entities which have different crystalline orientation. So, not every grains here have the same orientation only certain grains are satisfying the diffraction conditions so from those grains we are getting a very strong diffracted beam. Scattering is very strong there on those grains because of the virtue of their orientation.

If that happens then those grains will be diffracting much more than grains which are in the nearby regions and this is for a single phase material so no chance of atomic number contrast. Now this grains if I take the direct beam and form the image the bright field image what will happen is that those grains will appear in darker contrast because of the diffraction lots of beam or lot of electrons are lost from the direct beam because of the diffraction the special type of scattering.

So, those compared to the grains which are nearby which are simply scattering not diffracting. So, they will appear like with a brighter contrast than the other kinds of grains which will appear in a very strong dark contrast and this kind of contrast you can see again it will superimpose with the mass thickness contrast we will come to that. Now, if you see the corresponding dark field image.

And in this case if you are somehow able to choose that diffracted beam, the particular beam which is coming out from this grains these grains which are diffracting. If you are able to choose it from the diffraction corresponding diffraction pattern so you take a diffraction pattern of this entire material here, entire specimen here and you are somehow able to how you do it will come to that.

Somehow you are able to find that particular diffracted beam which is coming out from this grains. If you are able to use that beam for imaging now it is a dark field image, but now you will be able to see only those grains which are satisfying the diffraction conditions. You will not be seeing any other grains because the diffracted or scattered beams from them are somehow stopped by this objective aperture.

You are stopping the direct beam and you are stopping all other scattered beams except for this diffracted beam that particular diffracted beam that is coming out of this grains here and then you will be able to see those grains because the intensity is highest here, the intensity of the diffracted beam is maximum and it is coming only from here maybe some other grains also you will be able to see, but at a much lower intensity.

So, this is another way of forming a contrast in the material or in the dark field mode just because of purely diffraction phenomena. So, this is called the diffraction contrast and in this case you take advantage of this diffraction pattern here and choose your right kind of beam or right kind of diffracted beam or out of all scattered beams you just choose a part of that scattered beam for dark field imaging then you will get this diffraction contrast here.

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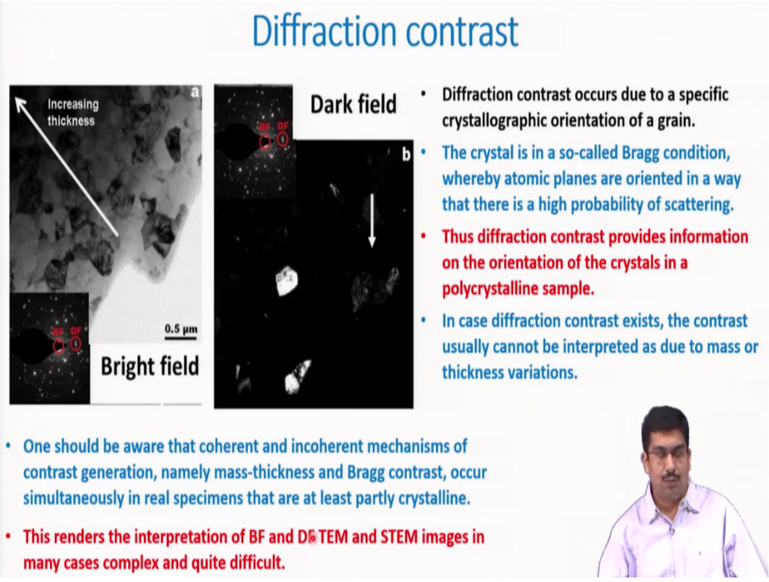


So, this diffraction contrast as I said is very, very important for forming contrast in case of a crystalline materials and there you can selectively even within the grains or within various grains you will be able to see them selectively in the dark field mode. So, you can make one more level of filtering in your diffraction or in your dark field images. So, that is why it is so important in this case.

And since all of this grains which are now diffracting and which are satisfying the diffraction condition it is very crystal if it is related to the orientation of those grains it is related to the crystallography of those grains. So, what you can finally get out of this diffraction contrast is not only you will be able to image this grains, you will be also able to get information about their orientations in the polycrystalline material.

In the polycrystalline samples you will get to know that how these grains are oriented, you will be able to image them, you will be able to know their orientation and you will be able to image any of those grains. You are seeing that there are many such grains are there which are showing these diffraction contrast in bright field mode. Now, if you go to diffraction dark field mode by selecting different, different beams here you will be able to select or you will be able to see different, different grains or not all the grains you will be seeing you will be seeing only certain particular type of grains because of their orientation.

So, that is the most important part of this diffraction contrast mode in dark field mode and even in the dark field mode you are now going and you are taking advantage of diffraction contrast. So, that is the best part of diffraction contrast in dark field mode. Of course what also you have to understand that the diffraction contrast in case the diffraction contrast exist usually that contrast cannot be interpreted by mass thickness variation.

For example here you will see that these are the regions basically the brightest region in bright field mode is coming out of there is an empty space if there is nothing basically and the thickness progressively increasing in this along this direction. So, here in this regions if it is a pure mass thickness contrast then this region should appear in a uniform grayscale and this as thickness is increasing this region should show you black darker contrast.

But that is not happening. You are getting some anomalous contrast here anomalous dark regions here in this bright field mode purely because of diffraction contrast. So, diffraction contrast this darker contrast in this regions you cannot really interpret here by this mass thickness function, but sometimes again you have to be very careful that this diffraction contrast and mass thickness contrast can be superimposed as well at times.

And that makes the life complicated that makes again bright field interpretation of bright field, dark field mode or images in dark field mode in diffraction contrast mode sometimes becomes very difficult because you never know it is always there is a possibility that mass thickness contrast will superimpose with your diffraction contrast.

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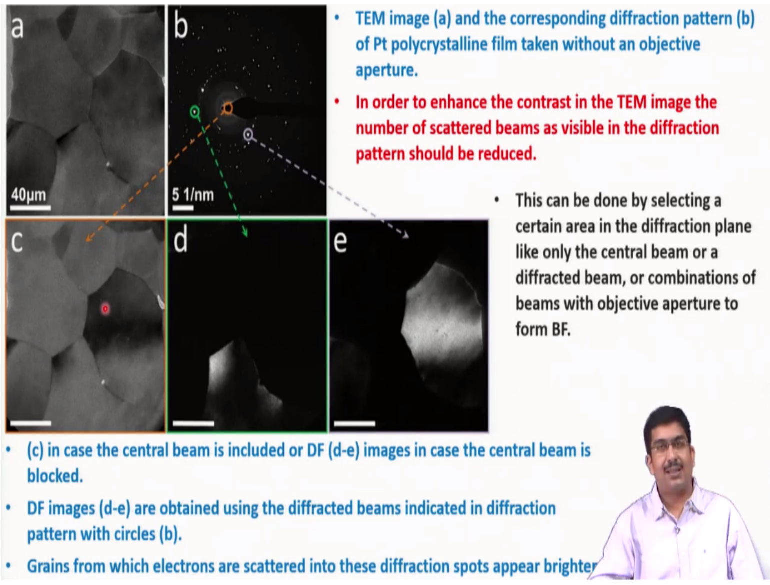


For example there is one nice example I could find out that this is an TEM image from platinum polycrystalline platinum film and so this is the bright field image corresponding first bright field image from there and you are seeing different grains here and some regions are of course very dark, some regions are comparatively brighter and you can imagine that this is possibly coming in the first place.

This is coming because of the mass thickness contrast, but of course since this is a polycrystalline material. So, maybe there is some amount of diffraction contrast always it will be there diffraction contrast for a crystalline material. So, what you can do you can take definitely again as I said you should always check the corresponding diffraction pattern. So, you take the diffraction pattern and then you use some particular type of diffracted beam to image this.

So, if you do that if you choose these two particular type of images what you can get is you can image now two particular type of grains. So, you know their orientation and you are able to see those grains individually in the dark field mode, but even within this grains if you look that you are seeing that certain regions really appear very, very dark not every region within this grain is equally intense.

Even if you are choosing the same pure if you are expecting pure diffraction contrast even with that you are having this mass thickness contrast superimpose on top of that which is restricting you to see this entire grain. So, that is some regions you should be really aware of that this is kind of always superimposed, but what makes life again as I said what makes life complicated is that this diffraction spot has certain width.

That means this grain may not be that entire region of the grain may not have the same orientation. So, that means the entire region may not be showing exactly same diffraction contrast. So, this black region may also come because this part of the grain is oriented separately differently. So that means this part also can or this black region here (()) (26:51) on this black regions here may be forming because of orientation change.

And that is why if you compare them with the bright field images normally you will be able to understand which part of the contrast because of the mass thickness contrast and which mass of the contrast is purely because of diffraction contrast. You can make an idea, but of course it is very difficult again as I said to interpret this in off line particularly in offline mode.

In online mode you can switch between this bright field mode, dark field mode and so on you can check it in diffraction if you take diffraction pattern and can choose different beams and see it and confirm it, but offline it is nearly impossible to find out exactly which one or which of this contrast forming mechanism is active in this case.

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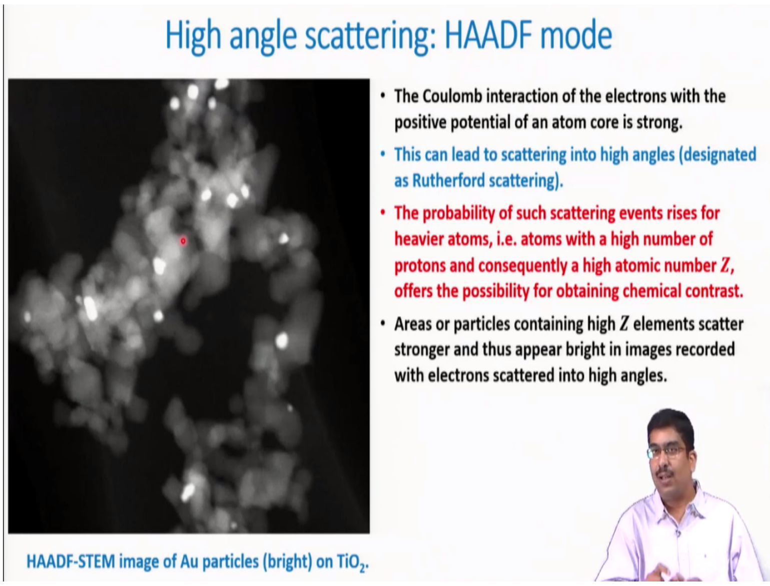


So now we will be just starting this another very important mode of imaging that is called high angle scattering or high angle annular dark field mode. So, this mode is a variation of dark field which is again for contrast enhancement and it can enhance the contrast or it can even deconvolute various type of contrast forming mass thickness contrast, it can separate out mass thickness contrast from the atomic number contrast particularly for that it is very, very important.

It is different than diffraction contrast here there is no diffraction involved for contrast forming mechanism. It is again purely mass thickness contrast and atomic number contrast, but in this case the mass thickness contrast is quite subdued and atomic number contrast is the most prominent here. Now this HAADF mode or high angle annular dark field mode works or for that matter HAADF is just a special of high angle.

It can also be ADF mode that is annular dark field mode, high angle is variation of this annular dark field mode, but I am not going in annular dark field mode because annular dark field mode is something similar to dark field what we discussed high angle annular dark field mode is a little special. So, in this case what happens that coulombic interaction between the incident electron and the positively charge nucleus is the highest.

So this leads to scattering of a high angle Rutherford scattering because of the interaction between incident electron and positively charged nucleus, high angle scattering typically happens when incident electrons are scattered by positively charged nucleus and a special case remember is the backscattering where the scattering angle is nearly 180 degree. So, electron just goes out and it just gets somewhat rebound from the positively charged electron.

We are not interested in backscattered electron we are not considering here, but we are considering real high angle scattering scattered electrons in this case compared to the direct beam the scattering angle is really in the higher side 60 degree, 70 degree or so on. So, in those cases what happens is that we already discuss this that if your atomic number is higher that means the nucleus is having more number of protons.

The nucleus is much more heavier and much more charged it is quite higher charge. So that the possibility of high angle scattered electrons will increase the number of high angle scattered electron will increase with higher atomic number. So, this is another kind of atomic number contrast that will happen between the high angle scattered electrons. So, if I have two different materials or two different regions with different elements and difference in their atomic number.

And if I capture the high angle somehow capture the high angle scattered electrons where I should expect to see a lot of contrast from between this atomic number two different number. So, if you look at this HAADF image which is actually gold particles on a titania support TiO 2. So, gold is of course very, very heavy and it is giving a high amount of high angle scattered beam.

So, if I just capture that high angle scattered beam I will be seeing the regions corresponding to gold now appears at a much higher and brighter contrast compared to the titania support all other regions is basically dark because no high angle scattering is happening from those regions there is mostly empty region only from gold regions very high, very large number of high angle scattered electrons coming out and titania regions of course is much less.

So, I should be able to see this gold particles over this titania support just if I use this high angle annular dark field mode. So as I said here also I am using scattered beam only I am using the dark field mode it is a variation of dark field mode I am still scattered beam, but a particular special type of scattered beam just like diffraction contrast. There I am using a special type of scattered beam that is particular angle the diffracted beam or the scattered beam which is sort of out of the entire scattering beam.

I am choosing a particular angle there in diffraction contrast mode only the diffracted contrast mode only the diffracted beam. In this case again out of the entire scattered beam I am choosing a particular angle for the scattered beam. A particular angular region or angular range I am choosing for imaging. So, this is another variation of dark field mode and we will continue this discussion about HAADF mode in the next few classes. So for this one for now thank you and good bye.