Techniques of Materials Characterization
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Lecture – 25
Application of Electron Diffraction

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. And we are in module 5 that is week 5 and we were discussing about transmission electron microscopy. So far in the last two weeks, we have covered the imaging, different type of imaging modes, different sources of contrast generation in transmission electron microscope.

And of course, then we were discussing about electron diffraction little over a week. And we have discussed about first the sources of diffraction, how it happens, Bragg's law, reciprocal lattice, Ewald sphere construction. And then we also have seen different aspects of solving an index diffraction pattern, so indexing electron diffraction pattern, what we need to know in order to solve an electron diffraction pattern or what the information that we can derive out of this electron diffraction pattern and so on.

So, in the last class we had a very lengthy discussion about how to index exact procedure and how to index this electron diffraction patterns. And today we will be discussing about the application of electron diffraction pattern. Though I have written application of electron diffraction pattern, but in some sense these are general application of transmission electron microscope for different type of metallurgical materials problems.

Of course, the possibilities are endless. So, you can do depending on your nature of a problem, the transmission electron microscope can come very handy and you can use it for various purposes. Here I just try to list some very basic application areas where transmission electron microscope is very popular and most often they are used for these kinds of purposes. So, we will of course continue.

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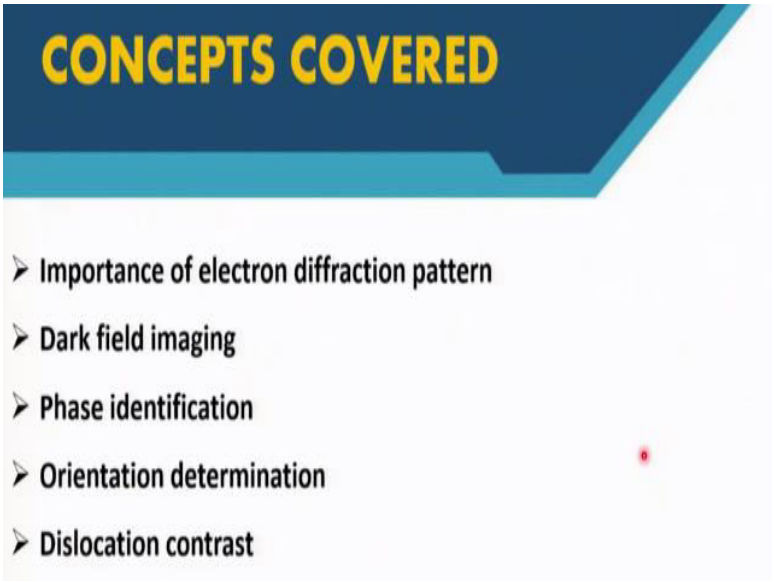

We will start with the importance of electron diffraction pattern. Then we will take some few specific examples like dark field, imaging, phase identification, orientation determination and finally we will talk about one very special technique that is called dislocation contrast. So, dislocation contrast is a very important part of TEM both for imaging as well as for the diffraction or it is related to diffraction only this contrast generation mechanism.

But little different than the typical diffraction contrast that we see in dark field mode. And we will see how it can be useful for the study of dislocation, something to do with a dislocation, we will discuss about that.

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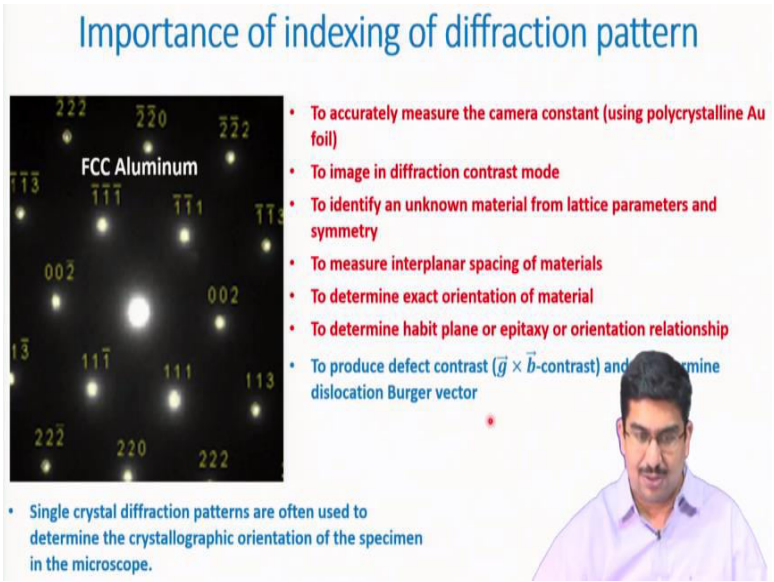

So, first of all of course the importance of indexing of diffraction pattern. Here we have named a few of the areas, but as I said the possibilities are endless. And you can use it as when you require the electron diffraction pattern or as a general or more generally the transmission electron microscope. So, this already we discussed to accurately measure the camera constant using polycrystalline gold film.

So if you do that index, if you index the diffraction pattern you can know this camera constant and from there again further you can calibrate the machine. And you can further go ahead and do this indexing for any other kind of diffraction pattern. Of course, you can also use this diffraction patterns for imaging diffraction contrast mode in dark field imaging, we will discuss that. And these two are basically almost related.

This to identify an unknown material from lattice parameters and symmetry of diffraction pattern. So, to identify phase identification by electron diffraction pattern and also from there if you know the phase then you can possibly get interplanar spacing for lattice parameters for any known or unknown materials. Also, you can use it for determining the exact orientation of a material, we will discuss about that. And an extension of this is to determine the habit plane or epitaxy.

We have discussed in the last class how this electron diffraction pattern can be very helpful in determining the change in the single crystal to polycrystal and then it can help you to identify the nature of epitaxy, nature of the habit plane and orientation relationship between various phases. And finally, of course to produce defect contrast that is g cross b contrast that is what we call and determine the dislocation Burgers vector. This is something that is very unique and we will discuss about this today.

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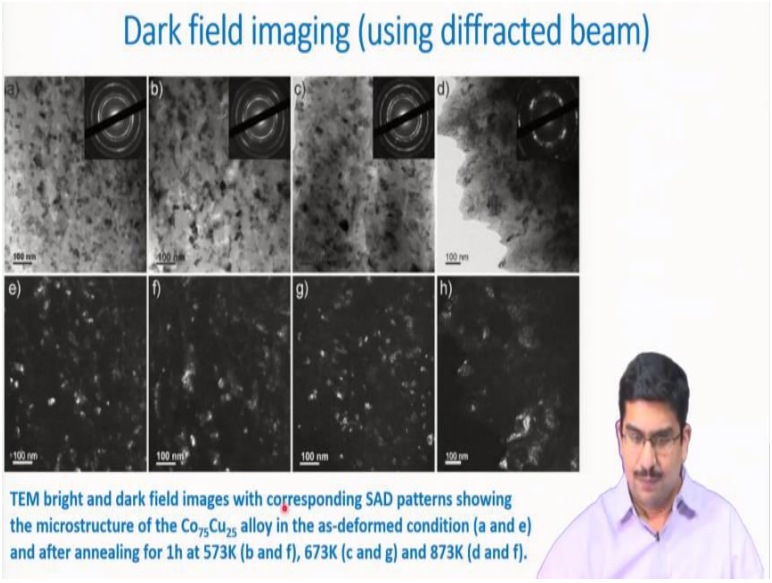

So, first thing of course dark field imaging and in dark field imaging even know that either we can use the entire scattered beam or we can selectively use the diffracted beam and then try to image certain features of exact orientation, we can determine their orientation as well. For example, if you see this structure, so these are the bright field images and correspondingly these are the dark field images.

But some speciality is this dark field images here we use different part of the diffracted beam corresponding to different spots corresponding, some cases the beams are coming from one particular atomic plane we are choosing let us say first order beam of 111 plane, sometimes we can choose the first order beam from 200 players so on and so forth; the possibilities are endless. And we can get these different kinds of dark field images.

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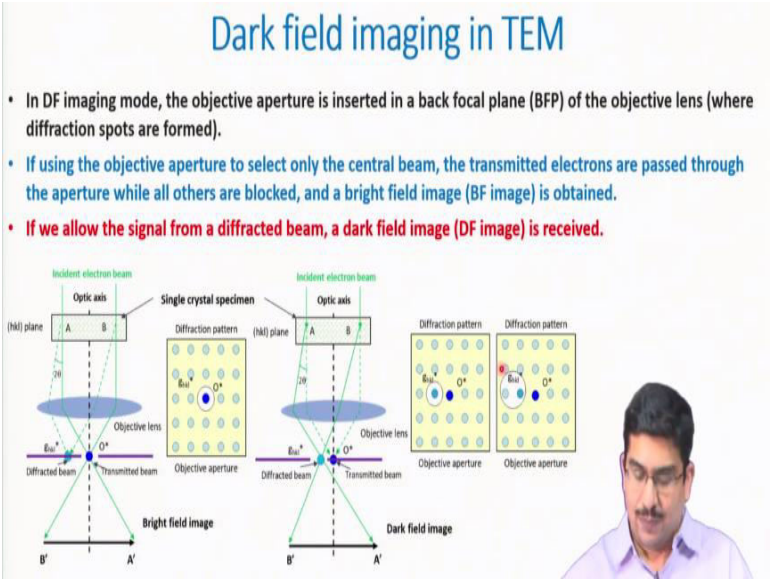

So, the way we do it we already discussed and that time I told that we can possibly come back to this one, this method of dark field imaging in transmission electron microscope after we discuss the electron diffraction pattern, this will make much more sense. So, exactly the same thing. Now, as we all know that diffraction, here dark field imaging can be done by using a suitable aperture.

You can bring that aperture and you can either close all the diffracted beam, you can just allow the direct beam, in that case you are getting a bright field image and then you can try to bring the aperture or tilt the beam whatever. If you are on axis, off axis, dark field depends on that. But let us say we are on off axis dark field mode, so what we can do is that we can bring that same aperture, now we can close the direct beam completely and allow a selected diffracted beam.

And this selection usually is done by seeing the diffraction pattern. So that means you must identify the diffraction pattern, you must take an SAD pattern, let us say SAD pattern or a complete ring pattern whatever it is, let us say it is a single crystal or it is an SAD pattern, so we get a spot pattern here. And what we do is that we first just allow the direct dream for the bright field image and then we tend to select one of the diffracted beams.

So, we tend to select the beam corresponding to one of these spots, either we can choose this one or certain cases we can also try to choose multiple number of diffracted beams so on and so forth. So, the possibilities are endless. So in modern days, it is all software

related. So, basically you see the diffraction pattern, you take your cursor, you select that beam, and automatically the objective aperture will go and select that part of the diffracted beam.

It will stop the direct beam and it will just allow that part of the diffracted beam or you can allow multiple diffracted beams whatever, depends on the aperture size, depends on your choice.

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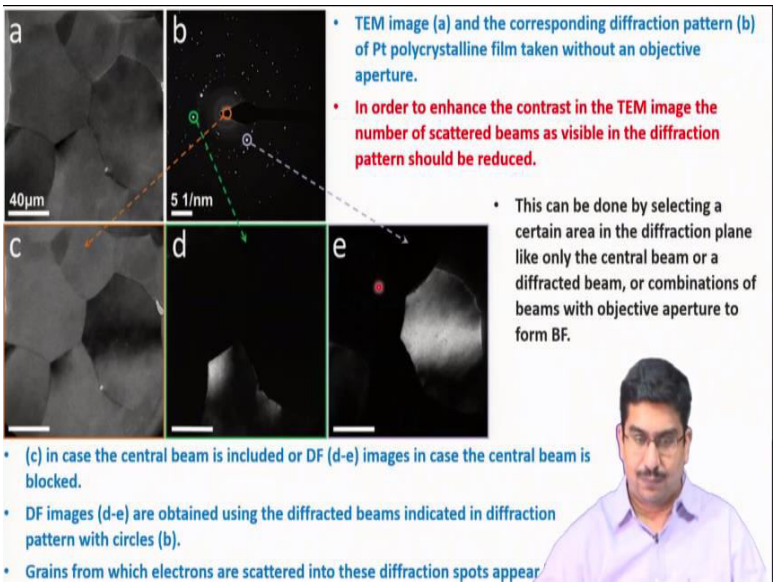

If you do that, this also we have discussed, then this kind of beautiful things you can do. You have this bright field image. You have this corresponding diffraction pattern. Basically, it is a spotty pattern; it is not a spot pattern, neither it is a ring pattern. So spotty pattern that is because of the orientation of many crystallites which are here contributing to this ring.

So, you can correspondingly choose some different rings here and from there you can identify these beams or these grains out here. So, if you see that you are in case one case you are taking this green dot, corresponding to this green dot here you are imaging one single grain and then if you choose some other dot here some other diffracted beam, then you are able to selectively see this grain.

So, not only you can image them, you can also know exactly which part of the diffraction pattern is contributed by which grain that means we will come to that, you can basically identify the orientation of these grains as well in the microstructure. So,

that is a very good nice thing that you can do by taking advantage of this electron diffraction pattern and then use it for imaging from this diffraction contrast mode.

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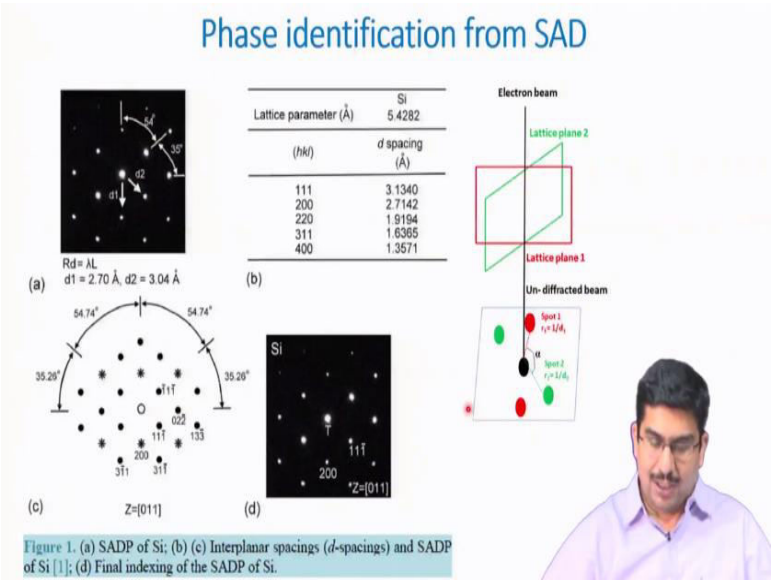

Figure 1. (a) SADP of Si; (b) (c) Interplanar spacings (*d*-spacings) and SADP of Si [1]; (d) Final indexing of the SADP of Si.

Of course, you can also do phase identification from selected area diffraction that is something that we were discussing. So, if you have let us say, I have taken an example of silicon. So, silicon is generally used as a wafer, silicon wafer for the electronic industry, packaging industry, semiconductor industry and many cases they tend to dope it or otherwise it may have some impurities and that may change the lattice parameter from the standard one.

And by solving the diffraction pattern, you can actually identify the d spacing. So, what you are doing you know lambda, you know sine theta and you are trying to find out the d by looking at this pattern. So you know that you need to know the your material a prior, you need to know the chemical identity of your material a priori. So you know it is silicon and you know lambda, you know sine theta, and what you do?

You know the camera constant, all of these things you know and you then solve this diffraction pattern. You measure the same thing r 1, r 2 all of these and from there you accurately measure this d spacing and from there either you can identify the material or if it is known material you can get the lattice parameter.

So, this this is a most possibly most common example. Of course, you have to be careful about all the previous class whatever the precautionary measures that we have discussed, you must be aware of all of these things.

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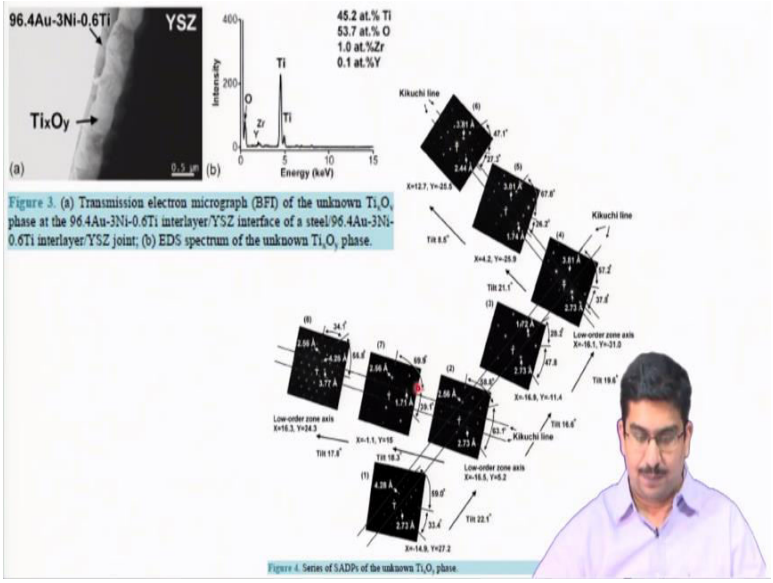

Figure 3. (a) Transmission electron micrograph (BFI) of the unknown Ti_xO_y phase at the 96.4Au-3Ni-0.6Ti interlayer/YSZ interface of a steel/96.4Au-3Ni-0.6Ti interlayer/YSZ joint; (b) EDS spectrum of the unknown Ti_xO_y phase.

Figure 4. Series of SADPs of the unknown Ti_xO_y phase.

Life becomes of course a little more complicated when you do not know anything about the material or either its chemical identity nor its crystallography nothing. Then solving this identity of this material new materials or to know about their crystal structure is extremely difficult. I just tried to show here one example and this is a very typical example, many of you may have faced this one.

So, whenever you are trying to bond a ceramic with a metal, so they do not go and particularly if it is for high temperature application. For example, this is YSZ yttria stabilized zirconia, you are trying to melt it or trying to bond it with steel. With a steel substrate you are trying to put a ceramic coating usually this is why if there is a thermal barrier coating improves the thermal capability of any components, most likely it is, I do not know exactly, but most likely it looks like it is a turbine component most likely.

So, you want to increase the temperature capability of this component of the steel and for that you want to coat it, but the problem is that this metallic part and the ceramic part they have completely different thermal expansion coefficient. So, if there is a thermal cycle that goes on this may very easily undergo a thermal shock and this too because of the difference in thermal expansion coefficient, the ceramic layer, the coating can very easily peel off that can disintegrate from the metal.

So, because there is not really a very nice bonding or close bonding, tight bonding formed between the ceramic and the metal part. In order to negotiate this problem what people used to do is that they used to put another coating which is known as bond coats. So, these bond coats have intermediate thermal expansion coefficient between the metals one side and ceramic one side.

And in order to make it even better what they do is that they try to grow from this metallic this bond coat, there is another intermediate layer which is in-situ grown and it is known as the thermally grown oxide layer. So here once side thermally grown oxide layer is shown, which is basically titanium and titanium where the oxygen diffuses and it reacts with oxygen and forms this titanium oxide.

But the point is that here this titanium oxide phase is nonstoichiometric and its composition, its crystal structure, everything changes over this length. So, in order to determine what exactly is the crystal structure here or their chemical nature or the phase, it is extremely difficult because you do not have any guidelines. You do not know, you can have this chemical identity check first.

You know it is titanium it is oxygen, but it is nonstoichiometric and then to solve their diffraction pattern and to get an idea about the exact phase stoichiometry and to get the crystal structure it is really a tough job. But of course, people do that and this gives fantastic result, but this is one example of identifying the phases.

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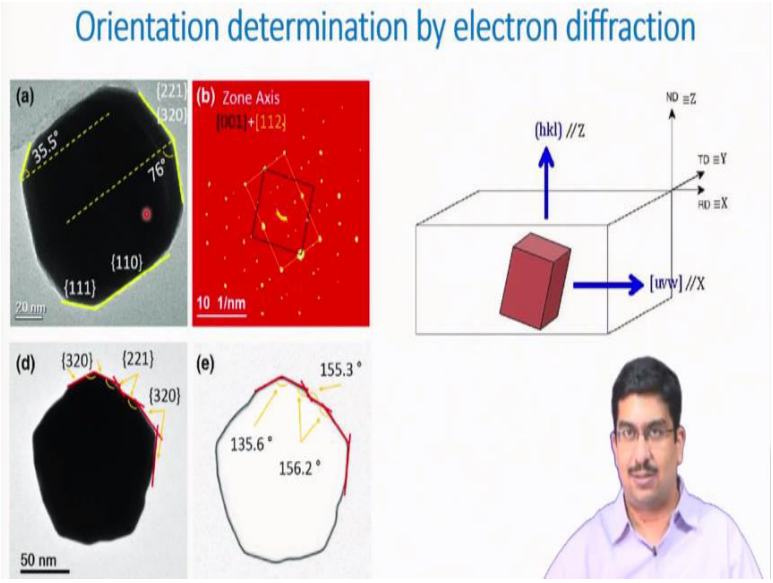

Next orientation determination by electron diffraction. Of course, orientation, what is orientation, what is called crystallographic orientation and all that is a subject in itself, we are not going into this. Roughly what we can call is it is if you have a specimen a bulk specimen of material or a process, if you have this specimen axis that is what we call it the specimen axis here.

So, let us say you have a rectangular bar of this, a bar of rectangular cross section. So, there are three orthogonal directions. You have this X, Y and Z directions here which are if it is a rolled plate typically called rolling direction, normal direction and transverse direction and then the crystallography direction. So, you have let us say this is a cube crystal, this is a let us say a unit cell, so, you have certain crystallographic directions.

Now, what we try to find out is that which one, what is the direction that is lying along this normal direction or Z direction and what is the direction of that is lying along this RD or rolling direction or X direction. So, that interrelation is basically the orientation, roughly you can call it is the orientation. The interrelation between these crystallographic directions and this bulk specimen direction this is the orientation.

Now, this orientation determination by electron diffraction is very useful here because you know by virtue of electron diffraction, basically only the planes which are parallel to the specimen normal or in other words the planes zone axis is always normal or always parallel to the specimen axis. That means if you understand this that means your hkl, if you imagine this is the zone axis this is automatically parallel to normal direction.

So, if you take an electron diffraction pattern, you determine the zone axis the way we discussed in last class from Kikuchi diffraction pattern or normal selected area diffraction pattern, you determine the zone axis and by that what you can get is at least one direction, you can know which is parallel to the specimen normal, you can determine one. Now, what happens is if this is a rectangular piece and rectangular cross-section, so you have two important directions here.

And imagine that this is a circular cross section, in that case both RD, TD all of them are same, you have only one ND direction that is important. In that case, you can very safely determine the crystallographic orientation of this material and you can by tilting or by other means by using crystallography you can of course determine this RD that is direction parallel to rolling direction all of that.

But basically, you can determine the direction or determine the orientation of this material simply by electron diffraction and not to mention the advantage of this is that you can determine this in a very small length scale. That means in a microstructure you want to know the orientation of a particular phase, then you can do that by using a selected area diffraction pattern.

So, you can choose an aperture which is exactly of the size or even smaller than the size of that particular feature and you can identify the orientation, what is the zone axis and what is the orientation of that feature. And that is exactly one example I have shown that this is one type of precipitate which is here and you can see that the precipitate has certain interfaces, which again looks like faceted interfaces that is what we call it.

Faceted interface and that means these interfaces have a particular orientation. So, by solving this diffraction pattern not only you know the zone axis, but also you can identify these different phases here, how this precipitate basically grow, what angle it makes. This is essentially like a single crystal. So, this single crystal you know that this is possibly the 111 plane, this is 110 plane and so on and between their angles and all of this.

You can determine how the precipitate grows, what are the facets, what is their orientation everything you can identify just by simply solving this selected area diffraction patterns.

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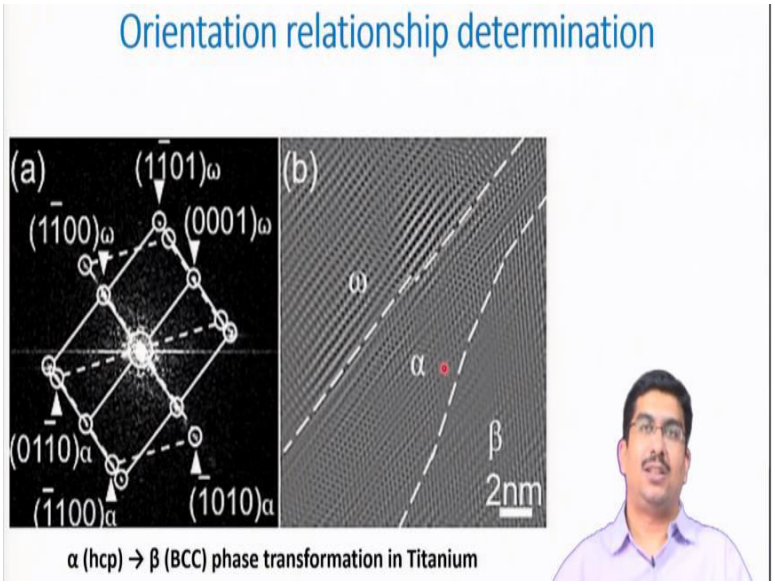

One step further from there, you can obviously determine the orientation relationship, we have discussed about that in the context of titanium 6 aluminum 4 vanadium where you have this alpha phase, hcp alpha phase next to BCC beta phase and by taking either a selected area diffraction pattern from individual phases with the same zone axis and then you can superimpose them.

And without changing the zone axis basically you can take a selected area diffraction from this and from this and then you can superimpose, you can try to find out what is the relative direction, which directions are parallel between these two phases and how much relative rotation is needed and all such things and that is the way you can find out or otherwise the way we did it in the last class.

You can take a selected area diffraction pattern from this interface region, where both the phases will contribute. And there also by solving for both phases, you can identify the orientation relationship between these two phases.

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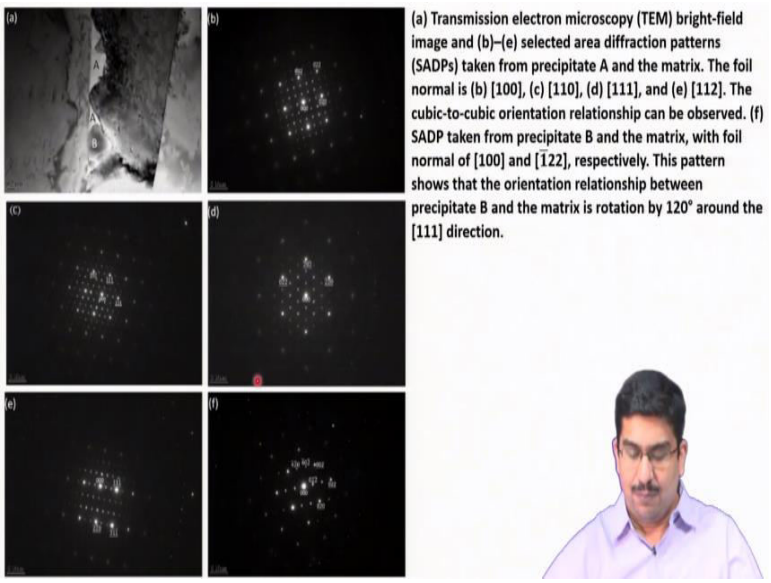

And at times as I said it comes very handy because of the inherent scale length scale at which this electron diffraction patterns are determined by selected area diffraction. So, for example, this is a steel specimen and within the steel specimen, you have many different types of precipitates. So, steels usually have a very complex microstructure where you have like carbides or nitrides, microalloying agents, microalloying elements which forms various type of precipitates.

So, now if you want to know the orientation of those precipitates and more importantly if you want to know the orientation relationship of those precipitate with respect to the matrix or the orientation relationship between them, what you can do you can take again selected area diffraction pattern from various phases, different phases, and then try to solve it and from there you can determine the orientation relationship. So, that is how you are using this electron diffraction for solving various type of materials problems.

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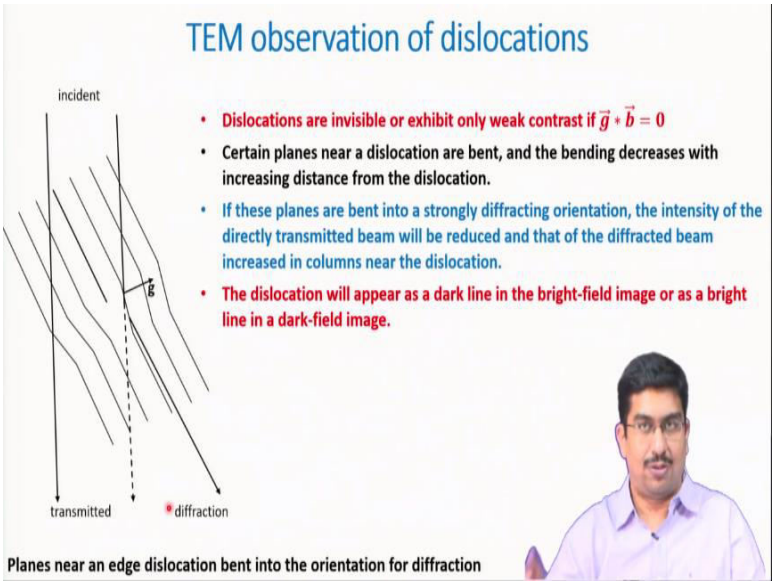

The last part of it with that we will also close our discussion on transmission electron microscope is the observation of dislocations. Now, dislocations I am not discussing much. I hope you already have an idea about what is called dislocations. So, dislocations basically the way we can visualize them at least for edge dislocations is a missing atomic plane. So, if you look at here these are atomic planes and we can imagine that this atomic plane continues up to here and after that it misses out.

What happens is that these dislocations are invisible depends; invisible or visible depends on the bright field or dark field whatever field we are seeing or they are invisible or exhibit very weak contrast in bright-field mode if g cross b where g is the diffraction vector, remember we discussed about the diffraction vector that is the zone axis and b is the Burgers vector for dislocation.

Again, I am not going into what is Burgers vector and all. Burgers vector basically represents the dislocation. It gives the amount or magnitude of that dislocation and sometimes it is direction as well, depends on the edge dislocation and screw dislocation. So, Burgers vector defines a dislocation. Burgers vector and line vector, these two things, line vector is basically the direction of the dislocation; these two defines a dislocation.

So, Burgers vector determination is very important in the field of dislocation. People who study dislocations, they want to know Burgers vector all the time. Now, this is the condition where if g cross b = 0 that means those dislocations are diffracting. Now, how

this happens? So, as I said that dislocations are defects, line defects basically, where an atomic plane is missing. So, as this schematic is showing and really it happens.

We can see it in high resolution microscope, I will show you one such image. Then the atomic planes are bend particularly this place which is called the core of the dislocation. So, near to the core of the dislocation, the atomic planes are kind of bent and if you go further away this bending is reduced. So, this is a very localized effect this atomic bending, a clear bending of atomic planes.

Now, what happens is because of the bending the d spacing changes there atomic planes. So, whatever is the d spacing between these atomic planes here somewhere further away from the dislocation that is definitely changes somewhere very close to the dislocation core. Most likely it increases here because an atomic plane is missing and that will affect the diffraction phenomena because if you remember according to Bragg's law diffraction strongly depends on the interatomic spacing.

So, if your lambda is constant, your sine theta is constant, if you change the d or rather the lambda is constant if you change the d, your sine theta is going to change. So, some things, some crystal; now if you imagine that some crystal is oriented on certain planes if this is let us say 111 plane, then if you have the zone axis such that now 111 planes are diffracting, they are satisfying the diffraction condition close to the dislocation or near to the core of the dislocation since the d spacing is different.

That means those are not satisfying the diffraction condition and these are not in diffracted or these will not produce any intensity for the diffracted beam or vice versa on the other way around you can imagine that if some parts are not diffracting or its zone axis is such that these are not diffracting, the rest of other planes are not diffracting close to the dislocation that may satisfy the diffraction condition and for that the g cross b = 0.

If g cross b = 0 that means those dislocations are in diffraction condition and dislocations will diffract. So, if they diffract strongly, then what will happen is that if you just now capture the direct beam for bright-field image those regions from the diffracted regions or from the dislocation regions, now there is a strong diffraction effect. So, those areas there will be a loss in the intensity of the direct beam.

So, those areas will appear very dark line or black line in bright-field mode and vice versa correspondingly it will appear a bright line in dark-field image. If you choose that particular diffracted beam from dislocations, then in the dark-field mode, it will appear like a bright image. So, that is how dislocations can be imaged using this dislocation contrast under transmission electron microscope.

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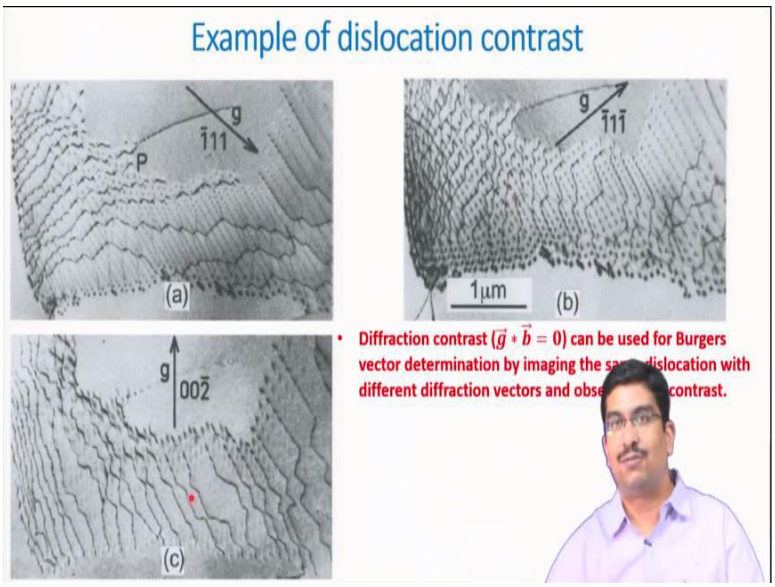

And that is what is very important with people who works with dislocations, for them seeing the dislocations is, more than seeing using this diffraction contrast you can determine because you know the g, you know the diffraction vector and from there you can identify the Burgers vector. So, this is another advantage that not only you can visualize these dislocations here.

But since these entire phenomena is extremely crystallographic it is related to the crystallography, so you can determine the Burgers vector of the dislocations as well. And that is a big thing in the study of dislocation. So, if you look at these three different cases, so here you change the g vector and you are basically seeing, g vector is in both of them are 111 family, but exactly not 111, so different particular planes the g different direction the zone axes.

You are basically imaging different type of dislocations here. If you change the zone axis, different dislocations and now dislocations are forming on different atomic planes and direction, so with different Burgers vector. So, now what happens is you are able to

image the dislocations at different zone axis, not only that since you know the zone axis what you can determine is the Burgers vector of those different dislocations.

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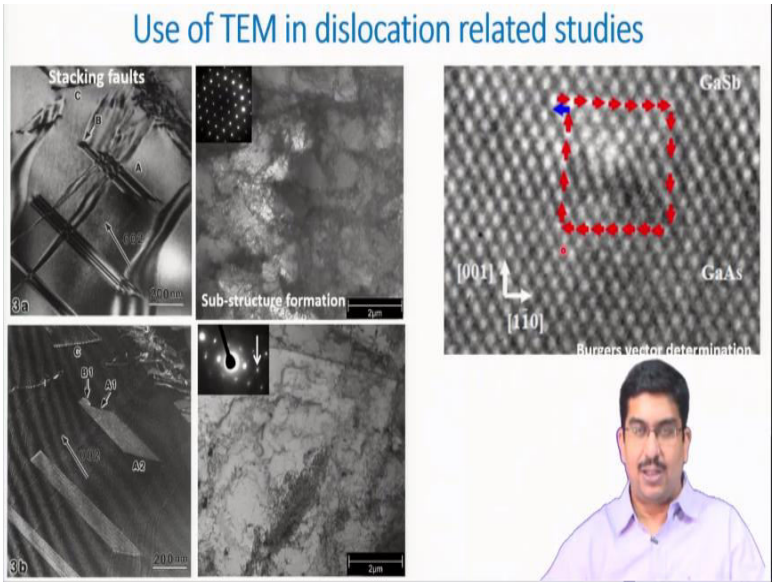

This is extremely important in the study of dislocation field. Whichever way people produce dislocation, dislocations are important like if you deform a material, you will get dislocations, even sometimes I used to get students who are coming with thin films and all, there you have misfit dislocations for example. One such example I have shown, I will come to that, but more than anywhere you can use this dislocation contrast and you can find out the dislocation.

So, dislocation contrast in TEM is possibly the only way to directly see the dislocations and directly you can determine the Burgers vector. All other methods of determining Burgers vector of dislocations is indirect, only in the TEM possibly you are getting that and you will be able to see the dislocations and you will be able to get their Burgers vector so that TEM is very important.

In fact, in 50s when this condition was determined and people started getting the dislocation image, so many of the theories before the dislocations, most of the theory of dislocations were derived on paper and after 50s when transmission electron microscope becomes powerful it was able to see the dislocations and determine Burgers vector, then those theories were proved right.

And you can go ahead with one step further with dislocation you can determine the nature of something called stacking faults. And stacking faults is again a combination, it is a dislocation phenomenon. Stacking faults are basically regions where the stacking sequence change and both sides of stacking faults we call something like call partial dislocation, they are bounded by partial dislocation.

So, you can determine those partials, you can see those dislocations, so they also produce pretty much similar dislocation contrast. From there you can find out if you know zone axis you can know the nature of this stacking fault. Even you can see under the deformation condition, when we deform a material very high extent these dislocations form some kind of structure which we call sub-structure formation.

And then there are some metallurgical processes like dynamic recovery, dynamic recrystallization, and so on. And those processes form typical dislocation structures, which again you can see it in transmission electron microscope for that and if you choose your zone axis properly, you can even identify the nature of the dislocations there. Finally, this is one nice example what I was telling that you can determine the Burgers vector in a high-resolution image.

Where you are actually visualizing, you are actually imaging the atomic columns, you can try to find out the missing atomic plane and you can actually find out if you look at here the atomic planes are really bent. So, there is a dislocation which is present here and this is for typical semiconducting material gallium arsenide and gallium antimonide. And here just by drawing this Burgers circuit, you can determine the Burgers vector of this dislocation.

This is called a misfit dislocation. So, this is the implication, one of the very big implication of transmission electron microscope in the study of dislocation. So, with this, we are closing our discussion about transmission electron microscope here and in the next class or next week onwards, we will be moving to another really important electron microscopy technique that is scanning electron microscope. And till then, goodbye.