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

## Lecture – 40 Imaging in SEM Continued

Welcome everyone to this NPTEL online course on techniques of materials characterization. We are in week 8 we are discussing scanning electron microscopy and possibly this is the last lecture on scanning electron microscopy. We were discussing about imaging in SEM in the last class we discussed how topographical images works and what generates contrast and then we were discussing about the topographical images captured in secondary electron mode.

# (Refer Slide Time: 00:54)



And today we will be discussing about the topographic imaging on backscattered electron mode using the backscattered electrons and then how we can use the backscattered electrons for compositional mode I mean like compositional contrast generation or compositional images using backscattered electrons and then we will see a specialized or more recent approach of using backscattered electron.

That is a quantitative compositional analysis with backscattered signals and finally we will see how the mixing of backscattered electrons and SE images, topographic mixing or compositional images how it can be done.

### (Refer Slide Time: 01:34)



So, as we discussed in the last class topographic imaging can also be done with backscattered electron because the yield of backscattered electron just like secondary electrons the yield of backscattered electron is sensitive to the specimen tilt, but there is with a but that in case of secondary electrons the detector does not come in between. So, detector more or less or for all practical purpose the ET detector secondary detector is independent of the sample surface roughness.

So, it does not depend on the trajectory of the secondary electron where the secondary electron is generated, but the backscattered electrons since they are having a directionality their yield is very much sensitive to the direction the detector itself or the detector means the annular the solid state annular detector the detector also is sensitive to the specimen tilt. So, this is a very important difference.

And with the difference if we use a multi element backscattered detector that is why we use a multi element backscattered detector. We can play around with this topographic contrast and we will see how. So, if we now compare the SE images and backscattered images basically any rough surface in the same surface the backscattered electron will be able to produce more shadows this may not be so obvious here.

If you look at the secondary electron image and this backscattered electron image this may not be so obvious, but if you see this image compare this backscattered electron image and this secondary electron image it becomes very clear that here you can generate selectively you can generate the shadow effect or you can generate much more contrast in a selective way.

So, when we use all <u>5-4</u> segments. So, this is the annular backscattered detector which is in and around this kept in and around this specimen this electron beam and imagine that this is the specimen and you have this four different elements here. Now, now if all four elements are active that means they are all capturing the backscattered electrons then what we capture is this backscattered electron image with very few shadows.

And it highlights I mean the contrast generation or the highlights of the difference in contrast between various regions is not that much as the secondary electron because we are only depending on the sample surface roughness here whatever the sample surface difference or roughness difference related to that the backscattered electron yield is changing. The detector basically is not really coming into play.

The effect of detector the effect of sample tilting on the detector is not coming here at all. So, detector all four elements are on just simply the backscattered electron yield what is happening let us say from this region and this region the backscattered electrons the difference between them is the only source of contrast production. This element or this detector does not come into play.

But when we use one segment only one segment of this detector let us imagine that any of the segment. Let us say we are using only this segment so this segment has a solid angle of collection from here which is very, very depending on the sample surface roughness. So, it will be able to capture let us say imagine that possibly from this facets only it will be able to capture the signal and not from this facets.

The way it is placed and the way or the solid angle that angle that it has with the solid angle of collection whatever BSE is coming within the solid angle of collection that is basically those BSEs which are produced only from this region because BSE has a directionality and that directionality cannot be altered unlike secondary electron they are of very small energy and that is why this metal cage is able to bring all the secondary electrons whatever be their trajectory that cannot be done with the backscattered electron.

So, whatever backscattered electrons is let us say coming from these region this detector is able to see that. This region is possibly some other elements is possibly able to see this regions better than this regions. So, if all four are open this effect is neutralized that is why all the facets are equally visible and if I stop one of them I can selectively make the contrast depending on the surface roughness.

So, backscattered electrons are much more flexible in case or in generating topographic image.

(Refer Slide Time: 06:12)



This will be more clear if we compare this similar to the secondary electron and comparing the light microscope with secondary electron images or the way secondary topography images are generated with SE if we do that same thing for BSE electrons then we can find the closed strong analogy. So, we have these different facets just like here let us imagine that we have different, different facets here.

The backscattered electrons and what we can now imagine that the lights are not falling from any arbitrary angle the lights are very much monochromatic and the lights are all falling, all coming parallely very much parallel light and this parallel light is falling on this sample inherently which is rough. So, different facets now will generate different amount of light which is parallel itself and finally is reaching to our human eye.

So, this sample roughness just because the lights that is falling here are all parallel and that is finally that will generate some kind of contrast. It may so happen that this C facets the lights

the parallel lights coming and hitting the C facets and then depending on the specimens angle with respect to this parallel light the reflected light will not be parallel or will not reach to the eye human eye here our eye here.

Whereas this B facets also maybe the same it is not reaching to here at all C facets it is reaching very less and only possibly the A facets are able to produce this parallel beams which are directly reaching to our eye here exactly the same situation if we imagine the backscattered electrons. Now imagine this is the electron that is falling on different, different facets.

And only the electrons which are generated backscattered electrons which are generated from A facets they are able to reach to this detector here not this backscattered electrons which are generated from B or C facets because they are completely going in other direction. This detector has a solid angle of collection and within that only the backscattered electrons coming from A facet are able to reach here.

So, this is why the backscattered electrons this is how it is different from the secondary electron detection the backscattered electron here the detectors are also sensitive the angle the detector has with this facets is also very, very important here. So, when we imagine this and bring an analogy with this and the multi element detector backscattered detector this is basically same as if we are using different light source.

So, not only one light source we are using different light source so let us imagine that we have a light source here, we have a light source here and all of them are sending parallel beams at different, different angle. And finally we are seeing it here so when all four or four light sources are on then we will be able to see all of the facets. When each selectively switch if off then we will be able to see selective facets from here.

Same thing here once element of detector maybe we have placed one here, one here and so on. So, that means when all four elements are on we are able to see all the facets when only selective one of them is on we are able to see only one selective facets that is it. So, that is how this multi element detectors are very, very effective in modulating the topographic contrast.

### (Refer Slide Time: 09:51)

- A multi-element backscattered electron detector may also be used to enhance the topographic image from a specimen which is almost flat.
- The elements A and B, being on opposite sides of the optic axis, will receive different topographic signals from the specimen.
- If the signals from these elements are subtracted from each other, then we will obtain no contrast from flat
  regions of the specimen, but contrast from any topographic features will be enhanced.



And now we will see how. So this first thing the multi element detectors can be used to enhance topographic images selectively even when the specimen is very flat and we will see next few slides we will see that these very flat surfaces are actually needed for compositional contrast. So, what happens is many a times we produce specimens in a way that they are very, very flat.

And they do not show any kind of topographic contrast, but in case we need to see them in the backscattered electron in topographic contrast mode then we can enhance this contrast just be selectively using the multi element detector. For example let us imagine that these detectors has two elements. We are just considering two, but actually you can consider four elements.

So, we are considering A and B two elements and they are kept at two different sides of this. So, if you imagine this is something like this one and this one. They are kept at two different sides of the optic axis. So, these two as I just explained they will be seeing different sides of these specimen, different, different features, different tilting of the specimen will produce backscattered electron which will be reaching.

Some of them will reach here and some of them will reach this part. Now what if we do is that this sub signals can be subtracted or artificially we can do it in the detectors. So, finally the current that they generate the solid state detector the current that they generate from this PN junction layers here. So, the current that is generated that we can use it to subtract, add and so on.

We can just put some logic gates there and whatever way we want to modify these signals or we want to play with the signal we can do that. If we do this something then we can change the contrast. So, thus imagine that these are the four different cases from these four different elements. So, when we are using only one element and this is the four different so these are four different elements.

So, when we are switching on any one of these elements let us say this is coming from one set of detector from one side of the optic axis. So, we are able to see that star as a projected one at a higher depth and when we are using let us say this set of detectors on the other side of the optic axis we are now able to see this star as a depressed region so at a lower height and if we just imagine that if we are doing a subtraction or we are doing an addition for example.

Just we are adding this signal with this signal what will happen is now this star will exactly fill in this place so the sample surface will be completely flat. If we add this one and this one then again the same thing the sample surface will be completely flat. If we just on the other hand if we just add these two, <u>this these</u> one and <u>these this</u> one we add then this or this star will be seen at a much higher height because now this adding this regions pixel by pixel I am adding these signals.

So, the topographic contrast will be enhanced exactly the same thing is done here just by added these two elements we are adding it here and these will increase the topographic contrast for this features out here. So, that is how we can enhance the topographic feature just by selectively playing with the signals from this multi element backscattered detector. **(Refer Slide Time: 13:32)** 



And this mode of operation is particularly useful when there are other source of contrast formation like compositional contrast which we will be going to discuss now. So, if there are compositional contrast or something called crystallographic contrast that is also there we are not discussing that, that is coming because of the orientation difference of different, different features.

And that also generates different type of backscattered signal that is another source of contrast generation in backscattered mode, but we are not going into that. So just imagine that we have the pure compositional contrast and that case the topographic contrast is basically somehow subdued we can even enhance that in some cases just by modifying or just by playing with this multi element backscattered detector.

And that is if you just compare this backscattered electron topography signal and the compositional signal so this has a very strong compositional contrast and that is because of that the topographic contrast in some sense is subdued and these are captured with no modification all four elements are basically here all four elements are on. So, this is a pure natural topography contrast nothing else has been done.

But this compositional contrast is you can see is so strong that this topographic contrast here is not that strong at least it is not that strong as strong as the scanning electron the same area when it is topographic images is captured in secondary electron mode it is not as strong as that. So, this can be enhanced here just by playing with that multi element detector. The problem of backscattered electron topographic imaging mode or backscattered electron compared to secondary electron is <u>badt\_that</u> even though this modifications all these good things can be done.

The inherent problem is backscattered electron the sampling volume the larger sampling volume and interaction volume of backscattered electron which is restricting its special spatial resolution in the first place compared to secondary electron. So, that is why topographic imaging in backscattered electron mode at a higher magnification is somewhat problematic. It cannot give adequate special spatial resolution.

And so lower magnification low magnification backscattered mode works very good for topographic imaging, but at high magnification it is always secondary electron topographic mode secondary electron which is very much preferred because of this problem of larger interaction volume and lower special spatial resolution.

(Refer Slide Time: 16:01)



Now we are going to compositional images using backscattered electrons. So, backscattered electron yield we know it depends strongly depends on the sample composition or it not only from only from surface topography, but also from the composition and this is the way remember the backscattered electrons are elastically scattered electron that is the only elastically scattered signals coming in SEM.

And it strongly depends on the elastic interaction and out of elastic interaction also backscattered electron is a special one because they are the electrons incoming electrons are scattered by the positively charged electrons not the electron cloud. So, positively charged nucleus is causing the backscattered electron. So, heavier the nucleus more is the backscattered yield as simple as this.

That is how the atomic number basically affects the backscattered electron yield in the first place and that is what it is shown here if we compare secondary electron versus backscattered electron the electron yield for backscattered electrons is very sensitive to atomic number and after a certain atomic number nearly 40 and all the backscattered electron yield almost increase and then finally it saturates of course.

And secondary electrons it increases slightly, but not much on the other hand secondary electron yield is very sensitive to accelerating voltage backscattered electrons for practical purpose or for the kind of accelerating voltage we use typically in SEM backscattered electrons are not that sensitive to accelerating voltage of that is used unlike secondary electron.

So, if we check this dependence of backscattered electron yield or backscattered coefficient eta that basically varies with atomic number in this way and if we imagine that it is independent of the accelerating voltage then eta varies with atomic number in this. So, it is basically a series and we are just considering the first three terms of that series it is a cubic and up to the cubic term we consider.

It is basically a series form that eta depends with the atomic number. So, this is the relationship basically between the backscattered electron coefficient with the atomic number and that is exactly what is plotted here schematically, but that is exactly what is plotted here this relationship is basically plotted in this case. Now one more thing we have to understand here.

So, this is showing here that if we have the same element basically the same phase then this yield of backscattered electrons here is exactly the same. If the accelerating voltage is not changed the atomic number remains the same and the yield of the backscattered electron is exactly the same. Problem happens is that when you have a phase multi element phase. So, you have two different element sitting next to each other and having different atomic number.

So, one of these atomic number which is the higher atomic number that will have larger interaction volume and that will generate more number of backscattered electron compared to the lighter element and this is where I am showing the primary beam is possibly smaller than this phase, but let us imagine that this is entire phase and the primary electron beam is hitting both of these two elements here or both of these two regions the primary beam is hitting.

Then the backscattered electron yield from any phase multi element phase that we can calculate simply from rule of mixture and using the weight fraction although there will be certain little bit dependence of this backscattered electron yield with the density how dense this thing, but we can for all practical purpose we can neglect that and we can imagine that the backscattered electron yield from any phase that is the complete eta phase is given by eta A into w plus eta B into 1 minus w.

W is the weight fraction of these two different advantages that is how we basically calculate the backscattered electron yield for any phase multi element phase.

### (Refer Slide Time: 20:04)



From this what we can calculate now is the contrast that is generated. So, if we imagine that we have two different regions one region is having this with a certain eta 1 that is backscattered electron yield of eta 1 something is here. And this is another phase which is having another kind of backscattered electron yield eta 2 then the contrast between these two phases we know this contrast comes from the signal.

We have derived all of these in our third lecture how it depends on the natural contrast, depends on the signal strength and so on. So, simply from those relationship we can find out that this contrast here is now (eta 1 - eta 2) / eta 1 as simple as this. So, this is the contrast that is generated between these two phases which are just next to each other. Let us imagine two different pixel so one pixel is containing one phase eta 1.

And another pixel is containing another phase eta 2 may be these two phases can be multi element in that case the eta 1 and eta 2 will be further calculated by this rule of mixture here, but in general let us imagine that the contrast is coming and contrast is expressed in terms of backscattered electron yield in this way. Now what happens is that let us imagine I have this two adjacent phases or two elements single phase or element whatever.

The two adjacent phases have very little difference in their atomic number. In that case they will have a very less difference in the contrast. So a very less contrast between them that is typically between 1% to 5%. So, if I have two adjacent elements next to each other. Let us say I imagine the transitional element, iron, nickel, (()) (21:51) cobalt and if I have iron and then I have nickel then this contrast that is coming out from them because of this elemental difference is hardly 1% to 5%.

And in case of an alloy various phases in an alloy different, different if I have multi element phases next to each other and the elements are very close to each other then it is even lesser that means what that means the inherent contrast in the compositional mode when I use the BSE signal and inherent contrast that is generated by this compositional difference atomic number difference is very less compared to topographic contrast where the topographic contrast is natural contrast from the signal strength.

It does not matter whether the signal is generated because of the topography difference that means because of the specimen tilt or because of the composition atomic number difference the detector does not know any of the detectors all it knows is that how many electrons or how many BSE electrons in this case, how many backscattered electrons are finally coming and hitting.

Whether the backscattered electron is generated because of the atomic number difference in two adjacent pixels or because of the sample roughness in the two adjacent pixel it does not depend on that. So, what we just now discussed is that the compositional effect the atomic number difference that effect or that can lead to much poorer contrast finally in the detector than this contrast produced due to specimen tilting.

So, somehow this we must understand and this is again this is where the multi element detectors are coming very handy. So, there we have just now we have seen that how to selectively suppress here we have seen that how selectively we have suppress the topographic contrast and this will come very handy if we want to only have compositional contrast. We can suppress the topographic contrast and we can just keep or just retain the compositional contrast which is inherently very poor.

And most of the time if I keep all four elements if I keep them like here if I keep all four elements on and then this topographic contrast usually dominates. Here it is not very clear it is because the compositional difference here between the two phases is very, very high. One phase contains titanium plus aluminum one phase contains titanium plus vanadium. So, vanadium is much higher atomic number compared to aluminum.

That is why this specimen this titanium 6 aluminium 4vanadium here is giving very high compositional contrast, but this is very unlikely if we have two elements which are very close next to each other like in steel normally in steel if we add other transitional metals the compositional contrast is very less and that is why the topographic contrast may be poor here, but the compositional contrast is very good in this case.

Otherwise but normally we have to do something and we have to enhance this. Other thing we can do in order to enhance the compositional imaging is basically we can try to remove any kind of topographic contrast source means we can try to remove the sample roughness. We can make the sample surface very, very flat and generally are next if you want to take good images, good compositional contrast.

BSE images in compositional contrast or good compositional contrast we should use them very much flat after polishing without etching that means we should reduce any source which will generate sample roughness which will increase the sample to specimen tilt and which will give you topographic contrast.

(Refer Slide Time: 25:32)

- Although we may be able to detect two phases in a specimen, we may not be able to do so with very good spatial resolution, because the ultimate resolution of the instrument depends on the contrast.
- Having calculated the atomic number contrast, we can calculate the spatial resolution of the two phases from minimum probe diameter and critical beam current.
- This should be considered as a degradation of the resolution, beyond that of 0.1 µm due to the limitations of the sampling volume.
- For phases with similar atomic numbers, the resolution is poor.
- Manufacturers of backscattered detectors will usually quote the atomic number difference that their
  equipment will detect, e.g. 0.1. but, without resolution under these conditions, this claim is not very
  meaningful.

Phase I	ZI	Phase 2	Z2	η	η <sub>2</sub>	Contrast %	Resolution Degradation (nm)
AI	13	Mg	12	0.153	0.141	7.6	19
Al	13	Cu	29	0.153	0.304	49.4	5
AI	13	Pt	78	0.153	0.485	68.4	4
Cu	29	Zn	30	0.304	0.310	2.3	47
α-brass	29.4	β-brass	29.5	0.305	0.306	0.2	264

Now, what we can do is that although we can be able to detect two phases in the specimen we may not be able to do so with very good special resolution because remember the natural contrast also depends on the probe size or the beam current we have discussed this in lecture 3 that how the natural contrast depends on the signal strength and from there we have derived many different other sources which affects this contrast natural contrast.

Like for example the probe current the size of the probe the pixel size and so many things frames time and so on. In compositional mode in order to generate if we have two elements which are giving very poor natural contrast in order to improve that contrast further we have to basically increase the probe size. We have to increase the number of electrons, signal strength has to be increased in the first place in order to reduce this or in order to enhance the compositional contrast.

So, that means this process of getting a good compositional contrast at times also reduce the <u>special\_spatial</u> resolution of the specimen inherently because contrast finally all of them contributes to contrast. This compositional mode the atomic number difference that contributes to the contrast and similarly the probe current and all other things that also contribute or that also basically controls the contrast here.

And from there if we just see this now table here if we have let us say aluminum and next to it we have magnesium, aluminum is the first phase and magnesium is the next phase we see that atomic number difference between these two phases is very less and that is why this backscattered electron yield this also is very less or the difference between them or the backscattered electron yield for aluminum is very close to the backscattered electron yield of magnesium.

So, this difference is very less and finally this will lead to a very low contrast only 7.6 and from here if you look something else for example if you go for aluminum and platinum huge difference in the atomic number and that will affect hugely on their yield backscattered of course platinum will have a very high backscattered electron yield because that is a heavier element.

And finally that will lead to very good contrast here and copper and zinc again they are very close to each other. So, the contrast between them is very, very less and an affect of that alpha brass versus beta brass. Brass is basically an alloy of copper and zinc and because of their very similar chemical composition they have almost inseparable atomic number contrast.

If we try to get any kind of atomic number contrast compositional BSE image from this alpha and beta brass it is very difficult to separate them. Now because of this what happens is if you have very good compositional contrast inherently for example if you consider aluminum and platinum where the compositional contrast itself is very high around 70% then the beam that we can use for other that also affects the contrast the beam size will be very small.

That means the resolution that we can achieve in this kind of specimen where aluminum is kept next to platinum. The beam size will be very small we can keep the beam size very small and the resolution that is degraded because of this in order to get a very good signal in order to get this compositional contrast the resolution degradation from all the calculations within. Remember the d minimum that we calculated considering the beam probe size natural probe size.

And then the spherical aberration and then diffraction and then we have seen how that is that resolution the final special resolution how it gets affected by the scan rate. So, considering all of these affects just because of compositional in order to create very good compositional contrast we have to increase the beam size, we have to increase the beam current that will further reduce the special resolution, but that degradation in the resolution is very slow if we have inherently very high contrast compositional contrast.

And if it is inherently compositional contrast is very, very small then the degradation in resolution will also be very large. So, if we have let us say the extreme example alpha brass and beta brass I want to have the inherent natural contrast they can give is 0.2 hardly less than 1 and if I want to still capture a good compositional image from BSE mode, from this specimen then the sample the probe size has to be real big and real huge and that will be possibly almost in the order of 300 nanometer almost 0.3 micron. So, its special resolution is extremely poor in this case.

(Refer Slide Time: 30:25)



So, one more we will discuss here and then we will stop and the rest of the things we will discuss in the next class. So, this is the last topic that we can discuss that is how we can play around with this multi element detector and still we can suppress the topographic image and enhance the compositional image. For example this topographic BSE image we know the detector elements let us say it is coming from out of all 4 what we can do we can subtract the signals coming from two different sides of the detectors.

If we imagine this is the detector again A and B kept on two different sides of the optic axis what we can do we can just subtract this detector signals or we can add the detector signals from the same side from two elements which are in the same side of the optic axis we can add them or we can subtract it from two elements which are in the two different side of optic axis and then what we can get we can get a better topographic contrast we have already seen that. So, this is created in that way, but the problem is this specimen this is not properly polished so these are all the slip lines these are called the polishing scratches. So, these scratches are very much visible here and this sample by default we know that it has a very good compositional contrast if we have two different phases which are not at all seen in this secondary electron topographic BSE mode.

These two phases are not at all seen here. So, we are just enhancing this topographic imaging here and we are not able to distinguish between these two phases rather we are seeing the artifacts of polishing here. From this what we can do we can add the signals from different detector elements and that will completely cancel out the topographic contrast. Remember what we discussed in here that how to reduce the compositional contrast here we can play around with this and we can simply remove the topographic.

If we add this and this we can simply remove the topographical contrast and then we can enhance this here if we can see here this there is this black spots and on the background these are coming because of compositional contrast. So, we can just enhance this compositional contrast if we add this and this we can retain only this compositional contrast and we can completely remove this topographical contrast.

This is exactly what we are doing here we are just subtracting the or adding the signals from two elements which are in the same side of the optic axis we are adding them up and that will remain all that will be just removing all of these topographic contrast and it will just keep this compositional contrast. It will be enhancing the compositional contrast which are inherently very good.

But we are not able to see them just because of the topographic contrast case that is subdividing this compositional contrast inherently. So, that way we can generate very good compositional BSE images here. And for compositional contrast imaging one last thing is that the solid angle of the detector solid angle of collection for the detector should be as large as possible because that inherently the compositional contrast the signal strength the difference in signal the contrast is very less in compositional contrast.

So, we better keep the specimen as close to the detector as possible to increase the solid angle of collection for the detection that means we should use a very low working distance if we

are interested to capture compositional BSE image. So, compositional BSE image can be very good compositional BSE image can be captured if we keep the working distance low if we make the sample surface very flat, unetched.

And then if we play around with like this detection the multi element detector signal if we suitably add and subtract we can reduce topographic contrast and we can keep only the compositional BSE contrast. So, we will stop here and rest of the few topics we will discussing in the next week. Thank you.