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NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING

Tutorial-7
Materials Characterization

Quantitative Characterization

Dr.S. Sankaran
Associate Professor
Department of Metallurgical and Materials Engineering
IIT Madras
Email: sankaran@iitm.ac.in

Hello welcome to this material characterization online course organized by NPTEL in the last couple of classes we are taking a scanning electron microscopy screed studies in the form of tutorial classes I would like to continue in this class also the same case studies what we have seen so far in the last couple of classes we have taken some of the examples like alumina how to polish them and how to proceed with the fracture analysis.

In all these cases I just emphasis fact that when you go for this kind of characterization techniques which involves lot more time and precision. It is better to have a Connecticut objectives well defined before you enter the lab and some of the objectives I have just shown in the case studies for example if you look at this alumina we were interested in

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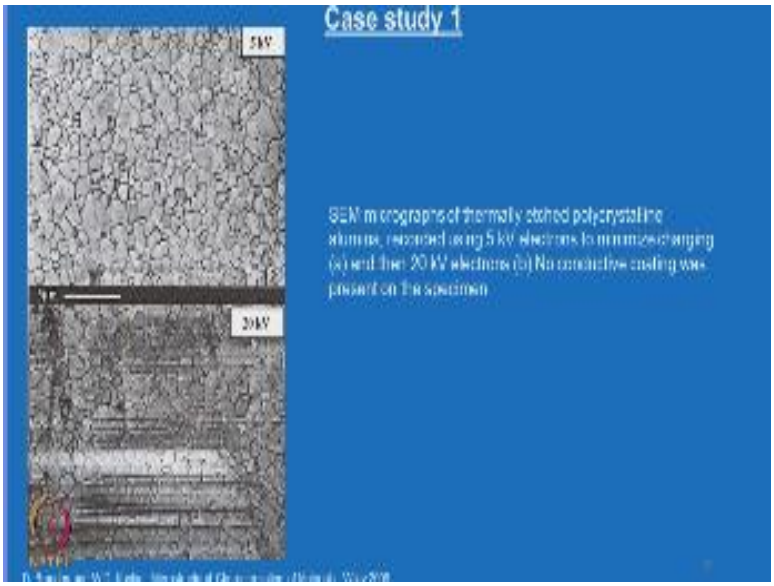

Looking at the drain sighs and grain boundaries and then whether they gain bounties are free from the second phase.

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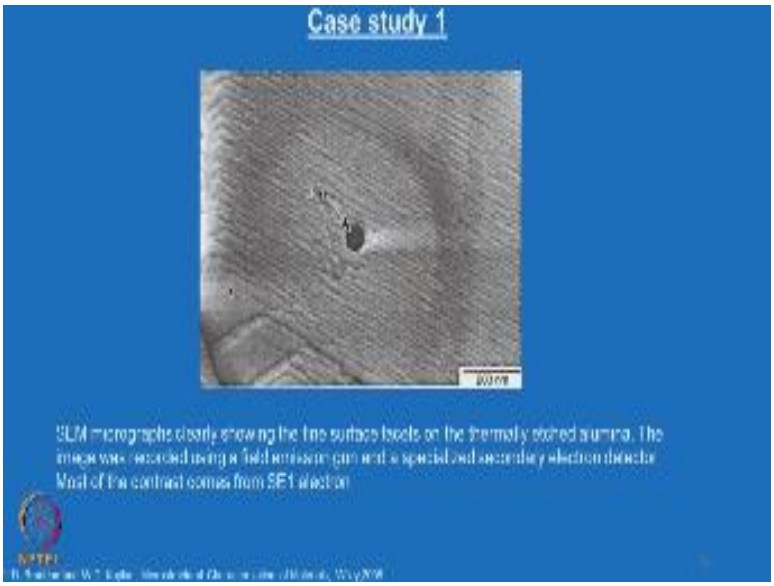

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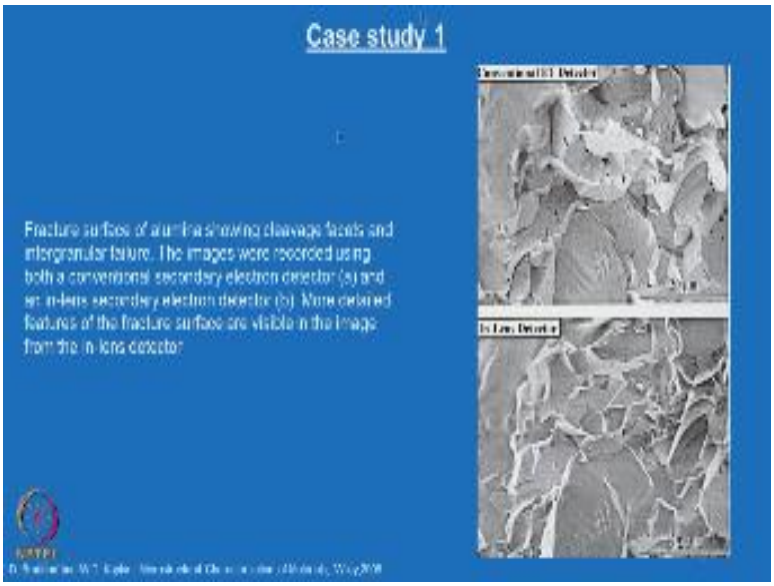

Particles and soon and then I also just demonstrated this micrograph which is a such a surface and then which revealing the inter granular fracture and of course I also mentioned the difference between these two is because nothing to do with the sample but the detector system which has got up in England detector in this case that is why you can see they better contrast in this surface.

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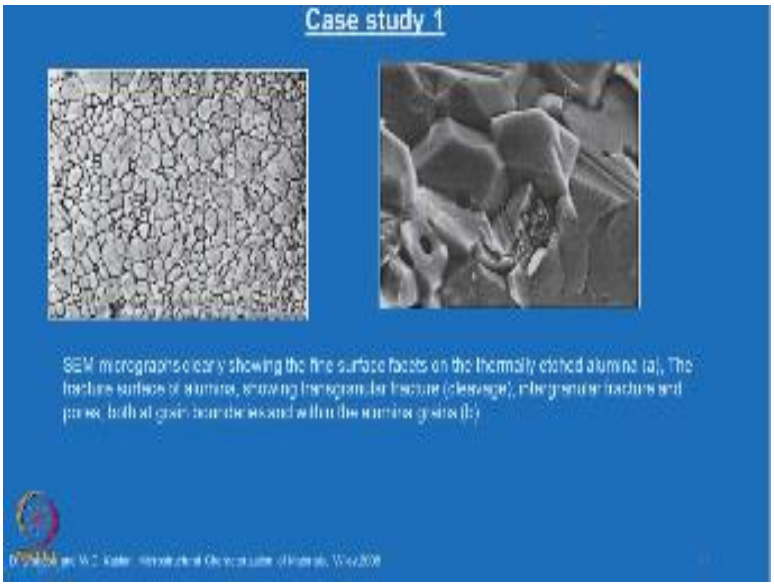

Then I showed you the fracture surface of this the thermally etched alumina where you throw granular and inter granular and then kind of a poor inside the material and this is how we discussed as a case one case study one.

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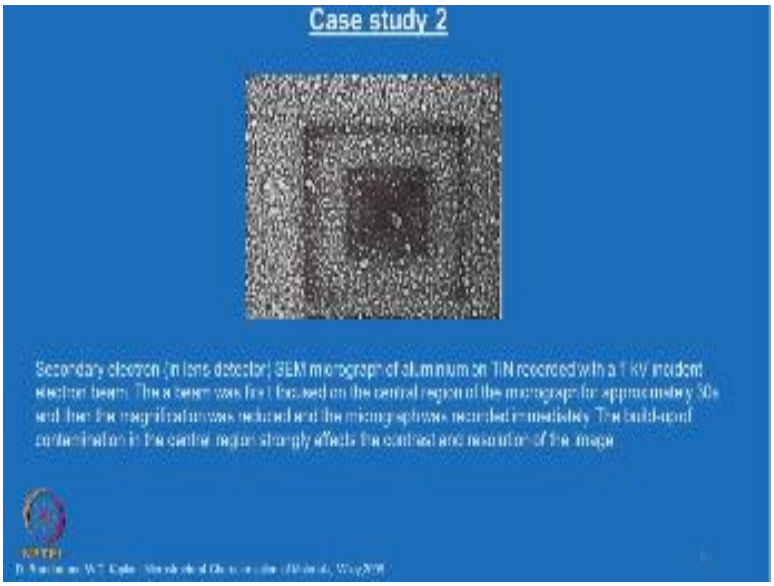

And then we also looked at this micrograph where I showed these micrograph of aluminum on titanium nitride with one kb incident electron beam then I just try to explain the reason between the difference in contrast in the center and then you can see that the other rectangular region around it and then the periphery region so you can see that a tear cat contrast difference between the outer region compared to inner region.

And I said that the reason for becoming this particular rectangular region becoming dark because of the contamination so if you expose this electron beam I a being sensitive specimen like this for a more time then the built up of contamination is inevitable and that is why it is very important that you do the recording very fast so you have to take that precaution and then why this contamination is forming and the reason for the sample to be clean that is something you have to be very I mean clearly keep it in mind. I am in order to do that let me write some of the few important points.

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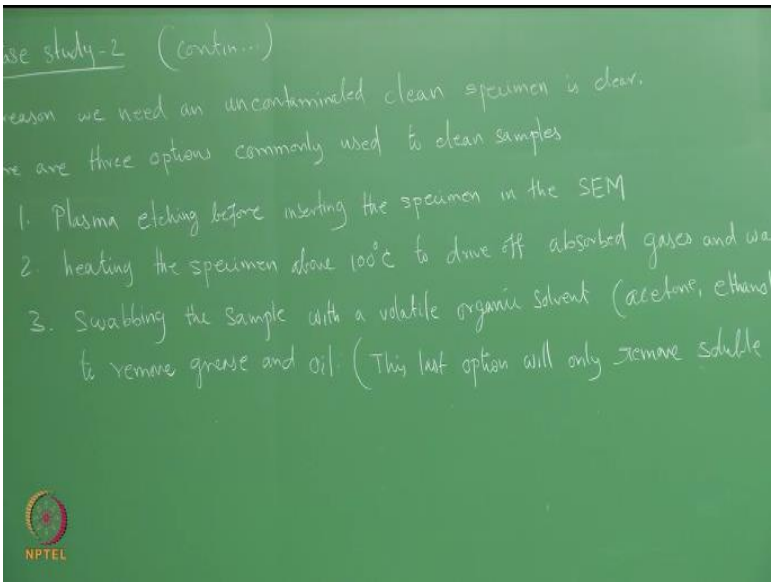

You so what I have written here is that if you look at the image what I have shown on these slides we are talking about the contrast difference between the center region in the periphery region and then we said that it is because of the build-up of contamination and then so the reason we need an uncontaminated clean specimen is very clear from this case study there are three options commonly used to clean samples.

The first one is a plasma etching before inserting the specimen in the seam the second option would be heating the specimen above 100 degree centigrade to drive off absorb gases and water and the third one else swabbing the sample with a volatile organic solvent it could be acetone ethanol or methanol to remove the grease and oil of course the this option will only remove the soluble contamination.

So you need to follow any one of the previous two to remove the other contaminants so if you follow this thing in the sample preparation you can reduce the build-up of contamination rapidly so before you grab the image so in spite of that what you are seeing in the case study even up to the you know thermal etching is done you see that the longer exposure of the electron beam on the specimen lead to contaminants build up on the sample.

So now we will look at the cross-section view of the same samples in the next study so what that you are seeing on the screen.

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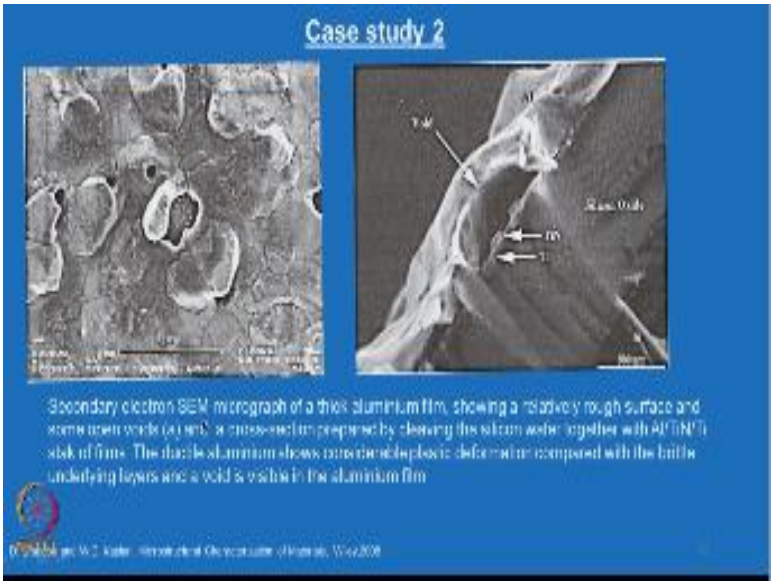

Is the secondary electron seam micrographs of the ticks aluminum film remember the one of the objectives of this case study was to look at the initial deposition of aluminum and its morphology so the earlier one what we have seen is the lower magnification and then there you could see the grain boundaries and so on and here you see that this is a higher magnification image and then you see that much more details are visible you can see the porosity very nicely miscible and you have the very big pores.

And it is relatively a rough surface and some open voids like this and the same specimen across-section view is shown in this second image and again it is prepared by cleaving the silicon wafer together with aluminum titanium nitride titanium stack of films the ductile aluminum shows a considerable plastic deformation compared with the brittle underlying layers and your widest visible in the aluminum film.

So this is silicon oxide and then you can see that the first layer the contrast is not very bright here but nevertheless user you will be able to distinguish between these two the first layer is where I have put the cursor shows a titanium the second layer is titanium nitride and then you have the void formation here and this is adulating aluminum you can see that deform layers of aluminum particles I mean aluminum coating I would say is sitting on this layer.

So you get a clear picture of these aluminum layers and they on the coating and the kind of defect it forms during the coating is also clearly analyzed doing this kind of case I mean the ACM examination so that is another example where you can use this if you more clearly I will buy some salient features of this.

So whatever we have seen on the slide on the coated aluminum what we have seen is say be the final sticks aluminum film has got a different mythology and seam shows that open voids have formed during the deposition process these voids are process defects which could affect the optical and other properties of these materials. So that is how one can you know interpret the secondary electron image in ACM in this case study similarly we have just looked at the cross sectional I mean view of this material at where we looked at the thickness of a titanium coating tighten my trade coating and then finally the aluminum coating.

So from this case study and we have now clearly demonstrated how the SEM analysis is being used and then the information is taken out for the appropriate interpretation and usefulness so i will now just move on to the other important use of the cesium in this case study called microanalysis seethe micro analysis can be done in two or three methods depending upon the detection capability so one of the very common use of this micro analysis is done by energy dispersion spectroscopy as spectrometer which is an attachment to an ACM or a TM and so on.
So if you look at the slide we have taken two examples in this case.

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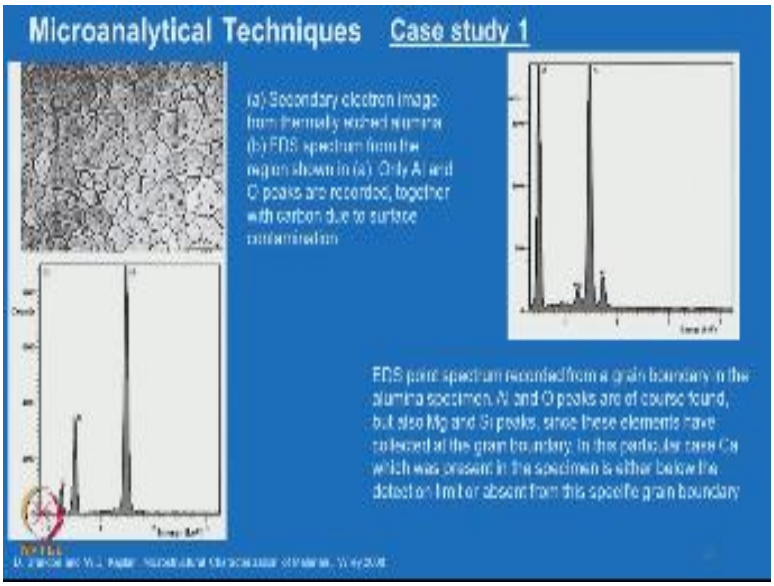

The image what you are seeing is the same alumina a second electron image thermally etched alumina and the media spectrum from the region is shown in a only aluminum and oxygen pigs are recorded together with the carbon used to be surface contamination please remember when you do this microanalysis you just compare the corresponding theoretical basis what I have given in the lectures in a theory class.

Where I have all clearly showed the detection limit of Ed that and that clearly shows that in order to obtain any useful information using EDS you need to have that particular element to be present in the sample sufficiently beyond that controlled quantity the hideous cannot detect so that is first and fore most important you have to realize before you go for this analysis whether you have the elements sufficiently present in the sample.

So please refer those slides where I have given the detection limits for any addax that is energy dispersive spectroscopic analysis where I have given the different case studies as well so in this particular case if you see that you be the spectral meters I mean the spectrum the accounts energy I mean number of counts versus energy spectrum shows only oxygen and aluminum along with the carbon though the carbon is no intentionally added it is coming from the surface

contamination and the other spectrum which is taken on the same sample shows a peak of oxygen magnesium aluminum and silicon so you should wonder why the difference between these two.

So let us read this caption first EDS point spectrum recorded from a grain boundary in the religion of specimen aluminum and oxygen pikes are of course found but also magnesium and silicon pikes sinks the element these elements have collected at the grain boundary in this particular case calcium which was present in the specimen is either below the detection limit are absent from this specific grain boundary.

So the first and most foremost important point to note here is the fastest spectrum is recorded on the gray the second spectrum is recorded from the grain boundary so you can see that in grain boundary you are seeing additionally a magnesium silicon so there are two things you can infer from this result that is either the magnesium and silicon could be a segregation but to make that conclusion you need to have a theoretical background about the solubility limits of this magnesium and silicon aluminum that is one that is confirmed then you can conclude this is segregation otherwise you cannot conclude as a segregation.

You can only say that magnesium and silicon present in the grain boundary maybe in the large quantities as compared to the inside the grain where it may be there magnesium and silicon may be there but it may be beyond the detection capability of this EDS red curve that is how you have to interpret but if you want to say that it is a segregation then you need to have a solubility data and then say that it is segregation that is another issue.

So similar point can be taken on the calcium observation calcium peak observe shin it could be same argument it could be a segregation or the quantity of calcium on the grain boundary may be a higher than D inside the drain so that is another way of looking at this spectrum and how to interpret those remember as I mentioned in the TD class the detection capabilities of energy dispersion spectrometer is inferior to the other similar technique called wavelength dispersive spectroscopy.

So where you can have much more resolution of this analysis so you can do the similar analysis if you have the attachment with the nation if you are interested in looking at higher resolution I mean EDS spectrum are microanalysis it is always better to have the WDS rather than addax but addax has got its own advantage that means EDS spectroscopy has got much more advantage listen it is quite fast and you can quickly find out the result as compared to the WDS which I have already shown in the daily theoretical analysis. So if you look at the other case studies like this.

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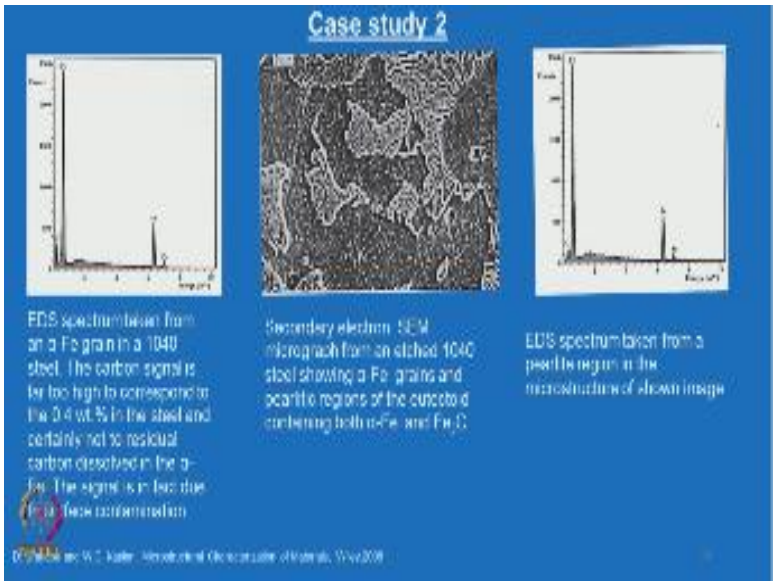

What I have shown is the a micrograph of a steel which shows a two-phase micrograph micro structure which is alpha iron and then Fe 3 C so this is in metallurgy this is called if this colony is called the par alight which is which consists of alpha iron and the fe3 p the black region is alpha iron is called the ferrite so this spectrum the first spectrum is recorded in alpha iron brain in a 1040 steel this is a plain carbon steel with 0 point for weight percent of carbon.

The carbon signal is too far too high to correspond to the 0.4 going percent in the steel and certainly not to the residual carbon resolved in the alpha iron the signal is in fact due to surface contamination so you have to be very clear when you do medias analysis on a steel sample as we

are talking about this contamination you get consistently with carbon peak in medias analysis so it does not mean your example has the carbon in solution or in higher quantity and we also know that how the carbon is generated tracking of this contaminants and becomes hydrocarbons and so on.

So in this particular case the higher amount of carbon shown in the spectrum is not due to the carbon present in this steel itself so how to compare that so if you look at that other spectrum the EDS spectrum taken from a partite region in the microstructure as shown of the image that means the first spectrum is taken from this region second spectrum is taken from this region you see that here also the carbon is there. So this probably due to a carbon which is coming from this because this contains an alternate layer of alpha iron and FE3C so like that you can confirm this.

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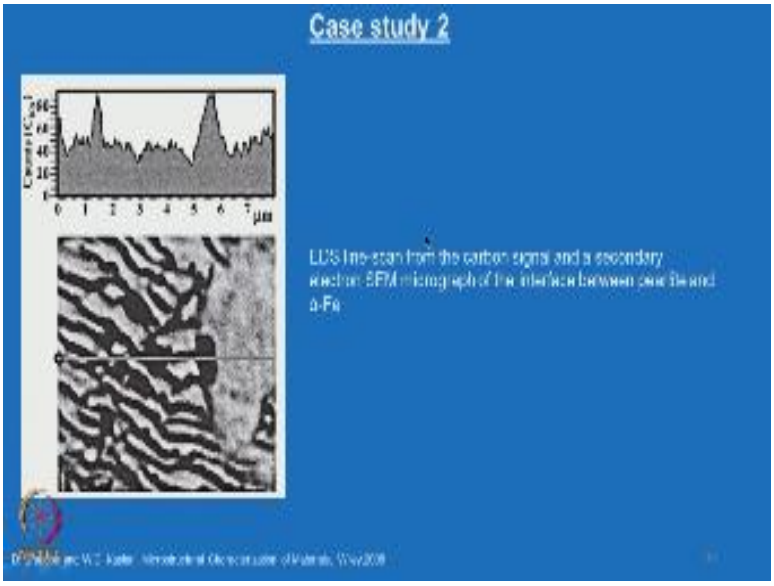

Seethe other example of this EDS analysis is shown here this is a medias line scan from a carbon signal and a secondary electron seam micrographs of interface the dream where lights and alpha red so you see that this is a scanned image taken from this is the partite colony what you are seeing here and this is the correct grain and you see that clear interface is shown here and the analysis is done across this section so called line scan.

The corresponding count is shown here that you can make a 1 is to 1correlation between the region which is covered here to the counts which are shown here so you clearly see that the as the length the scanning go through here so here you see that there is a peak a carbon peak even the micro craft does not show the presence of the carbide here but you see that the peak is there and I rest all you have a fright region and finally it goes through another peak it could be as carbide here and then this is acid.

So when you do this EDS analysis you may find some of the chemistry 11 chemical elements sorry which may not be seen in a secondary electron image like this one so in order to confirm that you need to do this analysis in two three regions are as many regions as possible to confirm the presence of identifying the elements which is present here so what I want to conclude from this session of the tutorial is first of all you have to choose what kind of a microanalysis you want to do whether you want to find out the light elements or higher atomic number elements a please understand in a light elements will not be detected from this technique.

And this technique can be used for higher atomic number elements and then also the quantity or the concentration of each element to be analyzed should be present in the system subject insufficient quantity otherwise this technique will not capture those elements and if you want a very fine I find out the traces of the elements are the elements which are present in a very small quantities you need to adopt to another technique called WDS are you have to go to a special techniques like TM and then with attachment with electron energy loss spectroscopy and so on.

So probably those spectroscopy techniques I will discuss in a in another separate and of course under where I'm like no preparing those things then if you follow that you can get to know how to handle those cases so I think with this I would like to conclude this case studies of seam so i request you to go through these case studies whether it is engaging our a microanalysis along with the theory then I will just upload some assignments you try to do that the assignment could be i will post from a micrograph and I will ask you to interpret in terms of image contrast are in terms of microanalysis and so on.

So that we give you some confidence and which again will help you in finally writing them in semester examination please remember these case studies are taken from a standard textbook I have given you a lot of references as well you go through the same and if you have a doubt you can always write to me I will just try to address those issues as much as possible thank you.

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