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NPTEL
NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING

Tutorial-6
Materials Characterization
Quantitative metallography

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Hello everyone welcome to this material characterization online course organized by NPTEL we have been seeing in last few classes the tutorial sessions of optical and other characterization techniques in today's class I would like to start a tutorial session on a scanning of microscopy I hope you all have gone through the theoretical lectures of this scanning electron microscopy where we have discussed in input in detained the principles of scanning electron microscopy the physics behind it and theory is also the instrument of details and image formation mechanisms and so on.

So in order to just recollect and realize how it is done in an typical laboratory experiment we have also demonstrated how to graph the image in a scanning electron microscopy in this class what I will do is I present couple of case studies where we will just show some of the images taken from the Cm and then try to understand how to interpret this and some of the do and downs of a things which you when perform scanning electron microscopy so as we discussed in the theoretical classes also a you have to be very clear before you start your experiments you have to clearly defined your objectives what exactly you want from this.

Microscopy session so that is primarily in important factors and for example if you just want whether you want to just look at the surface propography or you would like to examine the fracture surface what kind of a fracture surface or you are interested and looking at the second phase of distribution and so on for example even if you are interested in looking at the porosity

level or the coating thickness or a cross section sectional study so the objectives have to be first fixed clearly before you really perform the experiments then you can without wasting the time you can just go to the microscope and then look at what you dealing once is without wasting much of the time okay but before that we should also know how to prepare the samples for example I have just demonstrated in this course and various types if sample preparation techniques.

And depending upon whether it is an optical microscopy or scanning electron microscopy or even a transitional microscopy or the and so on and it also various with specimen to a specimen or sample to sample or material to material the sample preparation technique also very as you have clearly evidence in the video demonstration so today I will do is I will just present.

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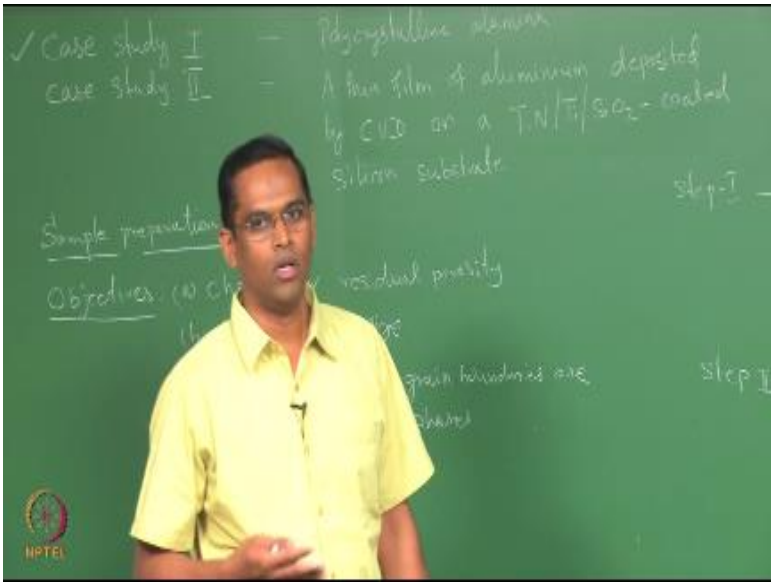

So what I am going to a present today is two case studies the first one is a poly crystalline alumina and then the second one is a thin film of aluminum deposited by chemical vapor deposition on a Titanium nitrite Titanium and silicon dioxide coated silicon substrate so you have silicon substrate and over which you have the coating of this and over which your

depositing the aluminum so how to examine these kind of materials in a ACM typical ACM okay so the first point is ample preparation how to prepare the sample.

First we take the case study one will take the poly crystalline alumina so what are the step for this sample preparation and then objectives so first we will say objectives what are the objectives in this case the objective is so like mentioned in the case study you have to be clear about what are the objectives in this case study we are interested in checking the residual porosity and determine the grain size and establish that grain boundaries are free from second phases so let us see how we will examine these things in this case study.

And the CM is one of the best techniques to look at the residual porosity in a material and now look at the sample preparation steps how to prepare the sample so we can prepare the samples by two methods either you follow the step one where you do the mechanical polishing down to a some micro meter diamond grid polish followed by thermal itching to form grain bounding groups so normally any itching activity will produce a grain boundary groups on polishing surface so in this case you do a thermal itching and then you have the a polished surface ready for the examination or you the second way of doing this thing is.

Either you break this some suitable mechanical specimen so that we can examine the fracture surface so there also we can find out the kind of features we are interested so that is another way of doing is so remember alumina is in insulator so we need to take care of the conductivity of see if you recall the lectures to examine the electron under electron microscopy the material should be conductive. So we need to make the surface suitable conductive enough before we try to examine the samples. so what we do

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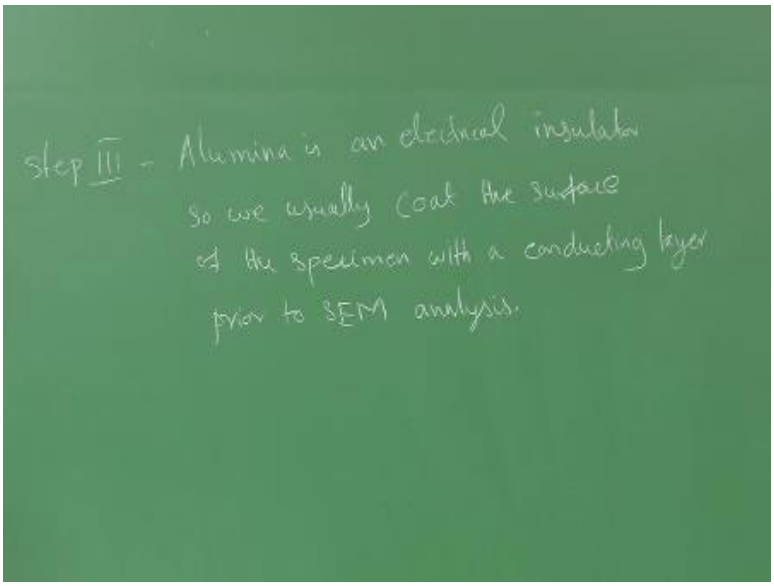

So what we do so we can use any of the conducting coating on the subsequent surface before the SCM analysis taken up so these are the important steps you have to consider before the go to the SCM analysis so I will repeat quickly we are now looking at the polyester alumina and raisin so the first step is the I mean the things which we have to keep in mind is you should have clear objectives and you should know how the samples suitably for examination.

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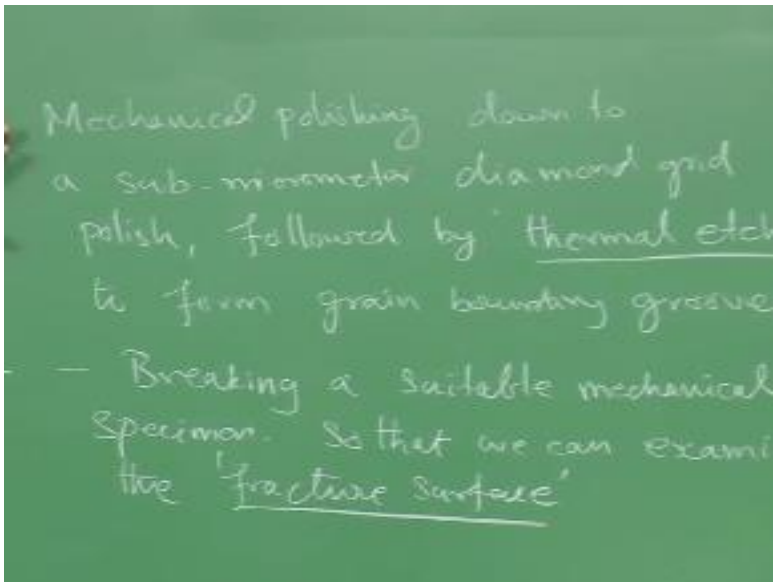

So in this case the objectives are examine the residual porosity determine the grain size establish the grain boundaries or free from, second phases and you can prepare the sample in both ways to look at these objectives the first one is very conventional standard one mechanical polish down to a sub micrometer diamond grid polish followed by thermal etching to form grain boundary groove.

Or you can break a suitable mechanical specimens so that you can also analyze the fractise surface there also you can get these kinds of information. I mean you can examine these objectives on the surface as well as polished surface and you come to polishing alumina is lightly heated we need to coat the surface with suitable conducting layer whichever is available in your laboratory you can use either a gold coating or carbon coating etc.

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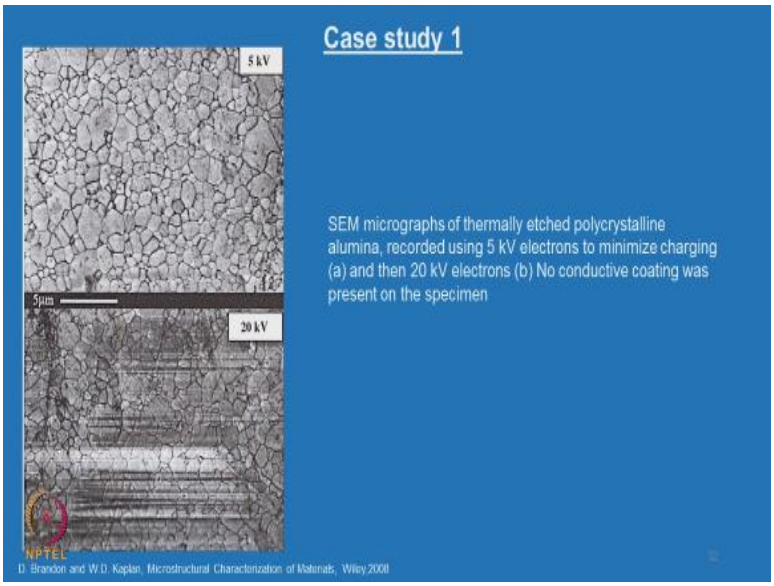

Normally people use gold coating so the next thing is I just go to the slide and I want you to look at this image there are two images a and b there are SEM microscope of thermally polished etched polycrystalline alumina, recorded using 5kv electrons to minimize charging and then 20 kv electron you have the you can see the difference in image quality.

So here in this image there is no conducting coating was under the specimen so you can see that charging is talking place so what is that you make out of it this two micro graph one is taken at low kv and high kv you can see that high kv does not help much in getting the good images so all that you have to remember you have to go back and recollect what all been discussed in theory classes.

I just mentioned in the image forming mechanisms in SEM you have form an image primary by the secondary electron which is coming from the immediate surface I also mentioned that you have SC 1 SC 2 SC 3 For example the secondary electron which comes from the immediate surface primarily all SC 1 secondary electron one category.

There are some electrons which is escape from the sub surface from the detector there are called the secondary electron 2 there are some stray electrons which is heat the column there are the categorizes SC 3 the interactions of this SC electrons is limiting to image quality remember if you recalling the secondary electron detector is kept on the side of the electron column in fact the back stray electron is just fitted on the hole piece sample the secondary electrons always kept on the side so the collection efficiency is always you know can be shade out from some of the detector positions and that is why these days you also have in column lines or inlines detector to form an image.

And then here what we are looking at why I am bringing this detector system when you have the in lines detector which can collect the secondary electron effectively and the reason for in this particular image what you are seeing because most of the secondary electron is hind rate properly or interacting more with the SC2 SC3 SC1 high voltage does not improve the image quality because it also second electron which is coming from the secondary surface quite of it.

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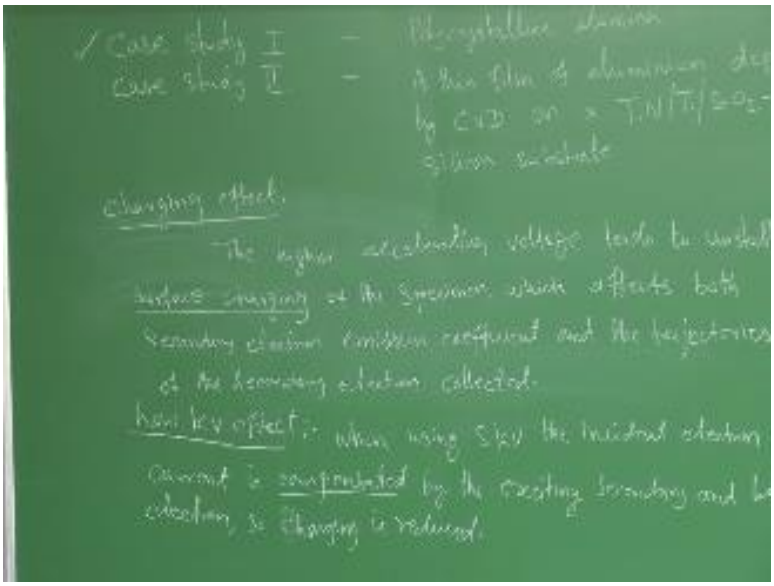

Because the lower kv is better way of using this so we writing out some of this observations so that you will forget these things why we are getting charger just charging effect because so why

we you produce the back stray when you use the higher kv in this case when you use higher accelerating voltage that leads to unable surface charging of the specimen which affects the both first is secondary electron emission coefficient that means the secondary electron which is coming from the surface itself is affected getting affected and then the trajectories of the secondary electron recollected, that is why I just said that if you recall or look at the video again the secondary electron detector is kept in the side to the, with respect to the specimen.

So the secondary electron trajectory itself is getting affected by this charging so you get a very poor quality image, so how the lower Kv is able to produce good image because you will write low Kv effect, you write. So when you use a lower Kv in this particular case 5Kv the incident current is compensated by the exciting the secondary and back scattered electron so charging is reduced.

So you have to remember so whatever the incident current which is falling on the surface has to be nicely compensated by the emission of secondary and back scattered electron so net charging is reduced so that is the point, so if you have the higher Kv then this is not happening the more and more and electron will go and bombard on the surface which will promote the charging effect which indirectly will have at the image quality so that is what you have realize effect of using higher and lower Kv on the image quality.

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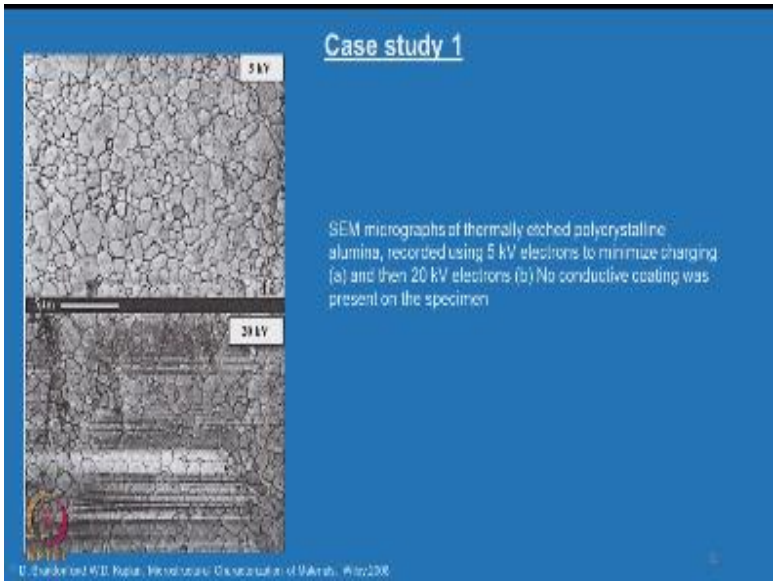

So this is the secondary electro image what you are seeing and then.

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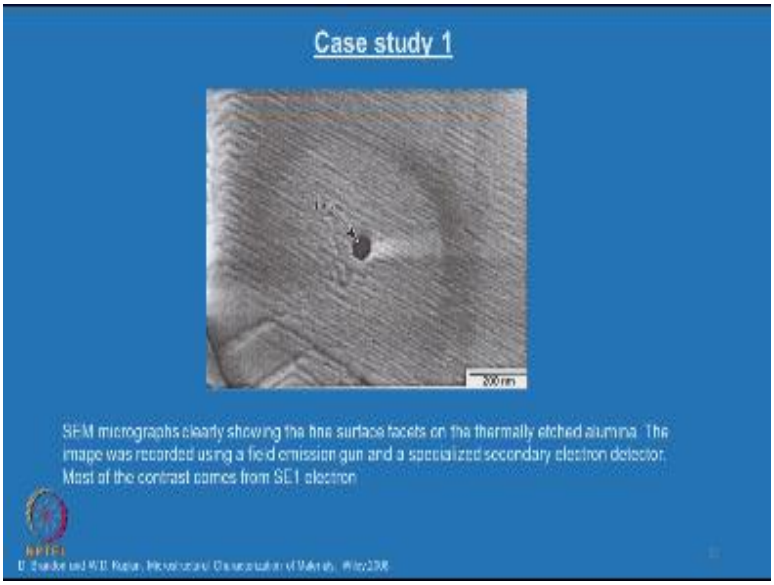

You can also now see that then SEM micrograph clearly showing the fine surface facets on a thermally edged alumina. The image was recorded using a field emission gun and a specialized secondary electron detector. So most of the contrast comes from SE1 electron, so you can look at the micron bar is about 200 nanometers you are able to resolve this nicely. So if you want to write something on this specimen you can write so.

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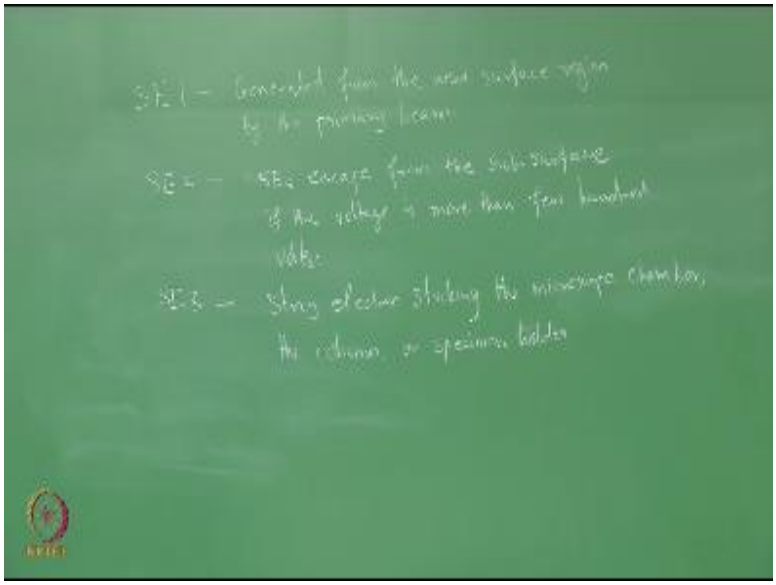

So what I have written here is nothing but what we have already discussed in the theory classes, you have the SE1 which is generated from the near surface region by the primary beam which is primarily responsible for obtaining a surface topography you can just go through the lecture again and then you have some SE2 which are coming from the sub surface then again escape from the detector and this is primarily observed when the voltage is more than few hundred volts and there is something called SE3 they are of stray electron striking the chamber or column or a specimen holder which again you know deviates from the trajectories.

So these things will contribute to the image quality so the one which you are seeing on the slide is the.

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You know is recorded at the field emission using field emission gun primarily by the SE1 electron contrast so very fine contrast.

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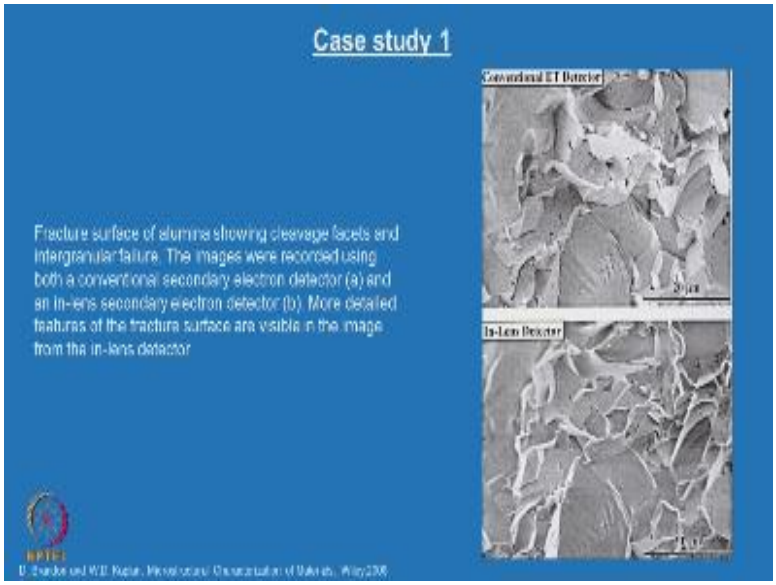

Then we will see that the same thing same specimen but it is a fracture surface what you are now seeing is fracture surface of alumina showing a cleavage facets and the intergranular failure. The images were recorded using both a conventional secondary electron detector that is a, and the in lens secondary electron detector b. more detailed features of the fracture surface are visible in the image from the in lens detector.

So what I am try to say by this images see look at this images let us understand what is this, the conventional ET detector captures you can see that facets all fracture surface and you have the intergranular fracture you are able to see, but the contrast is better here when you have the in lens detector. So what is just mentioned few minutes back if you have the in lens detector it could be angular type what it does is the all the secondary electron which is coming from the surface, immediate surface are effectively collected.

Rather than by a secondary electron detector in a conventional ET detector is kept at the side to the specimen, so the electron trajectory is have you know little deviated interested to be really tilted and specimen has to be tilt should be effective enough so that all the electron projector is moving towards the ET detector. But if you have inlands detector you do not have that problem it will effectively try to collect as much as the second electron emit from the unit surface. So the

collection is re-effective then the contrast is much better that is what you're evidencing here. If you have the inlands detector you have the better feature I mean the better contrast.

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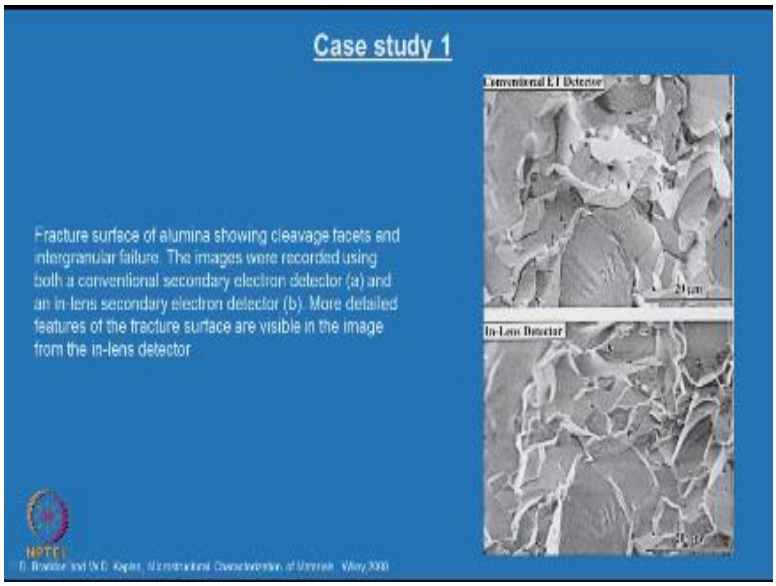

You are getting or also seeing much more details you can see that the details which are there in the corner aside is much more better than the you have the inland detector. That is because the deduction capability is high and also if you remember all these again as a secondary electron image, and this is how it is. It is in interpreted so you should have some idea about why this two different contrast is appearing on the same specimen and same fracture surface. But you're able to deduct this features which are interested.

Say for example the objective which we have just narrated about the inter-gradual and fracture surface. And we want t understand the grain boundary whether it is free of secondary phases or not and you can clearly see such analysis. So we will quickly go to the next one is basically.

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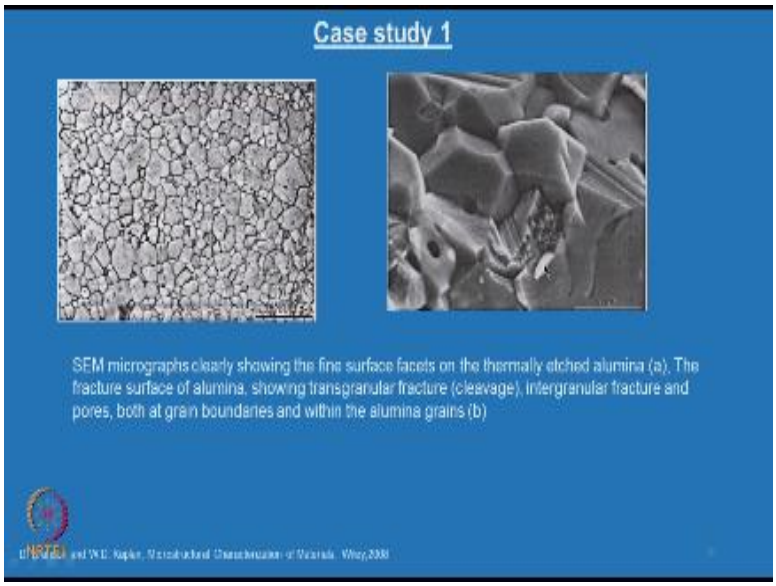

Looking at the fracture surface much more closely the same secondary electron micrograph as I have shown here. This again thermally etched alumina the fracture surface of the alumina will be shown on the right hand side. So in the transgranular fracture on the right hand side, so in the transgranular fracture this image all this faces there are all the result of P rays transgranular fracture. And then you have the grain boundary fracture you can see here these are all inter-granular fracture. And also we can see the entire pole here nicely the pores.

Both on the grain boundary as well as within the grains so if the primary objective. Of this case study was looking at the grain size which is typically the facets sides as well as looking at the grain boundary whether it is free of a second phase particle. And also examine there is visible probity like this so we can clearly identify all of them. Let us see whether we can write on them something about these features. Okay this is nothing but a secondary electron image and all that I have described is we have already written the objective. So we do not want to write it again you have the pores and facets and inter-granular fracture so now we move on to the case study 2.

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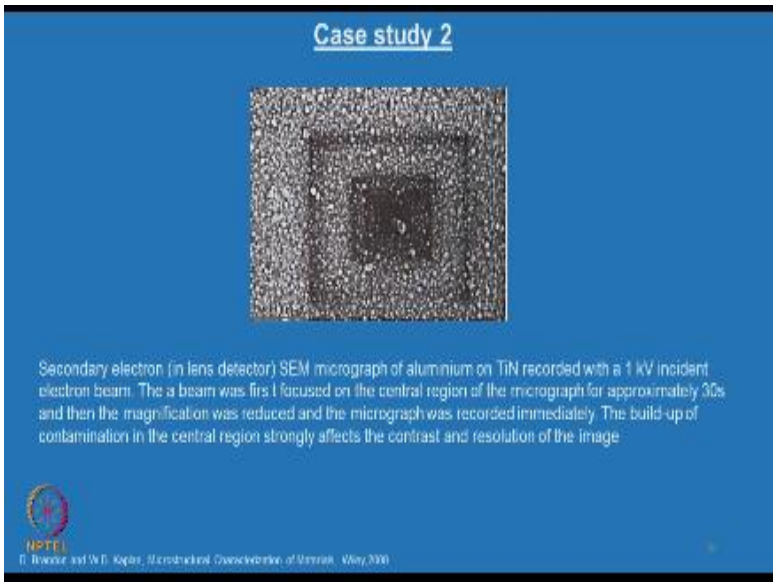

That is so this is about a thin form of aluminum deposited by CVD on a TIN TI S O2 coated silicon substitute.

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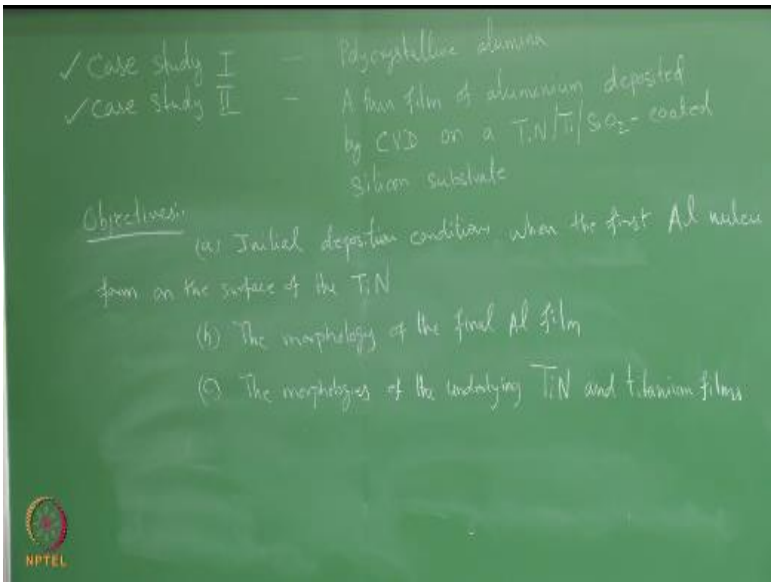

So objectives we will write, so here the second study what are the objectives. Initial deposit conditions were the first aluminum nuclei form on the surface of the TIN. And the morphology of the final aluminum fills. The morphology of the underlined titanium nitrite and titanium Phyllis. So you have titanium nitrate and titanium, so these are the objectives of this case study. So sample preparations if you look at sample preparation. Since it is metallic samples do not worry it is a conductor so directly so straight forward. And then looking at the,

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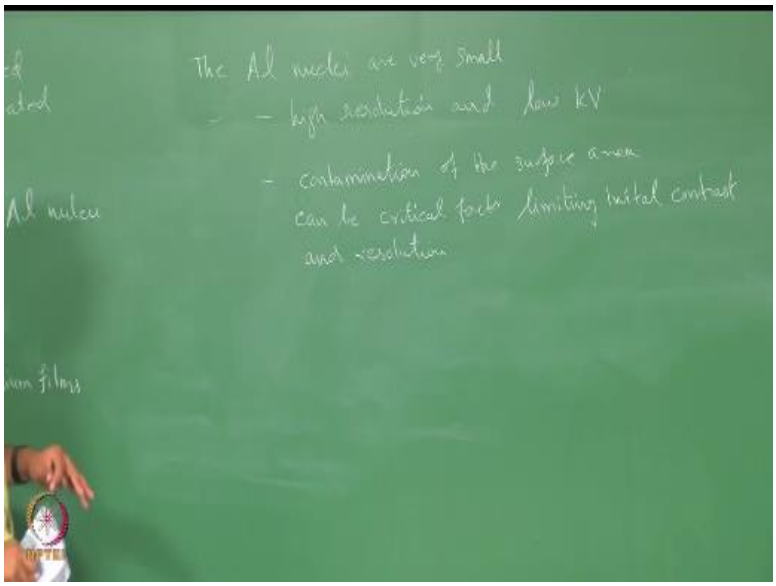

So the first objective is looking at the initial conditions of the aluminum nuclei is very it is very, very small. Since it is very small we have to keep that in mind that we require very higher solution and a low KV. In order to avoid the charging as well as the contamination, so sine we are interested in looking at the nuclei of aluminum and the after disposition. This surface contamination can be very critical which will be limit the contrast and resolution. So you might as that what about the previous case there is also it is I mean high voltage will produce there is a possibility of contamination.

But the less objective is we are looking at the surface coating which is very thin so the aluminum nucleic will be very small. So you can the contamination could be at the major critical path in this case other than the nucleic. So you have to look at this point very clearly, so what you are now seeing on the slide.

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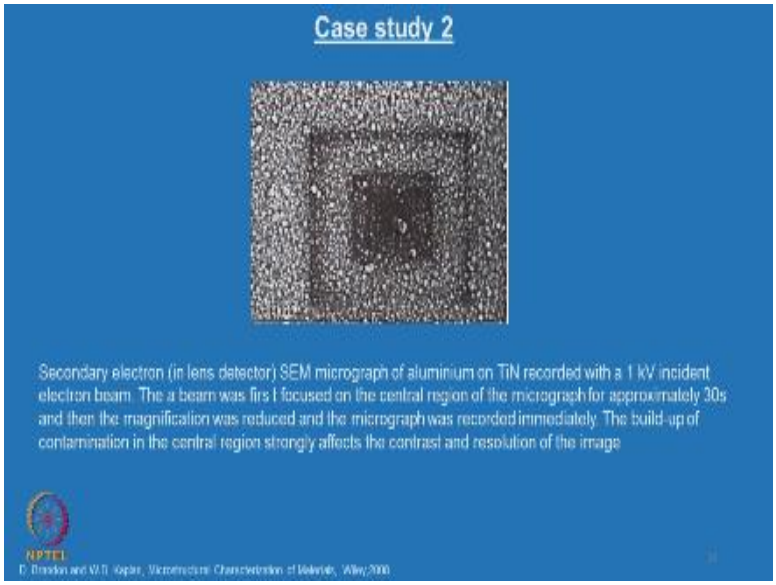

Is very interesting slide you can see that the secondary electron image is again it is taken with the in lens detector, you can see that aluminum particles are very small the coating initial nuclei on the titanium nitrate recorded with 1kV incident electron beam. Remember, it is taken with 1kV the initial beam was focused on a central region here oh the micrograph for approximately about 30 seconds and the magnification was reduced and the micrograph was recorded immediately.

The build-up of contamination in the central region strongly affects the contrast and resolution of the image, so what you are seeing is a black region it is because of the exposure over exposure of this electron beam and remember here it is 1kV is being used for 30 seconds in spite of that the contamination is so fast but then you have to reduce a magnification because you are interested in looking at the aluminum nuclei particle so the image is taken immediately.

So this is one typical case study which clearly demonstrates how the contamination can affect your resolution and contrast in SEM, so similar precautions you should take when you really want to examine such samples so this is another interesting study. So we can also now focus on the other two objectives looking at the morphology of final aluminum film and morphology of underlying titanium nitrate and titanium films probably I will take it up in the next class, thank you for your attention.

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