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Course Title
Advanced Characterization Techniques

Lecture-07

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So today we are going to first talk about in Situ experiments in TM then I will go on to this scanning electro-microscopy experiments.

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Like in situ scanning electro-microscopy and the electron backs got a refraction that is called EVST those kind of advanced characterization tools in the CM so let us first discuss about the in situ TEM we know that in situ experiments are very popular event from the very beginning of transmission electro-microscopy when first time the high voltage electro-microscopy TEM are in

1960's people started look at the motions of this locations inside the material in fact people started looking at the experiments on different kinds of solid state phase transformations .

In the materials while looking at in a high voltage microscope but things have changed over the time scale now a days we can even do in situ experiments inside a normal TEM we do not high voltage electro-microscopy the reason the high voltage electro-microscopes are used those days is because of the use of the thicker samples because we know that the wave in a material in a electron transparent thin sample.

Will completely different than a thick sample which you normally do experiment in the lab so that is why to simulate the exact conditions people used to use think thin foils or thicker foils actually not thin foil thicker foils inside the TEM columns and then study difference kinds of dynamic way we are of the material by in this forms to heat or strain or deformation or even cooling.

So because of that different kinds of TEM sample holders where prepared samples holders which can do experiments during hearting or is basically consisting of a heater or consisting farness inside the holder and then we can load the sample inside this holder and heat up to see what is happening in the temple and record it in a video device similar sample can be cooled down using a cooling stage holder within the transmission electro-microscope with column and this cooling can be done using nitrogen.

So therefore depending on the type of stimuli we want to use inside the holder you can have different kind of holders that is at is of the costs of the microscopy because you need different kinds of holders one can actually used the strain or the deformation also as on the stimuli so that you can indeed a particular post of the sample you can strain even by applying a load the soil kind of loading.

But most popular one for the in situ behavior of material during application of the load or stimuli like strain is the nano indention inside TEM column so that we are going to discuss some of these experiments today so dynamic behavior or material during a particular stimuli has become a very important because of advent of the nano crystalline material this is one kind of experience in situ experiment people do in the literature other kinds of in situ experiments which people do is.

The in situ growth that is suppose you want to growth indium phosphate nano crystal or you want to grow a some semi conductor crystals inside a microscope column so there are groups working in this world where microscope can be modified in such a way then one can deposits this crystals or the seats of the crystal and in the presence of the different kinds of vapor of the elements how this crystal grow.

At the revolution of the electro-microscope can be studied very easily in the using in situ microscopic techniques and this is as become very popular in different groups and the worlds where one can actually look at different in situ experiments different growth behavior of the materials at the resolution of these at the list of the electro-microscope and the inform information regarding the growth behavior this material and correlate with the existing theories.

So I cannot talk about all these things in this lecture because of the time constrain but what I am going to do is I would show going to show you some examples of those kind of experiments which can be done inside test TME column and obtain information during the experiments now a days with the advent of very good imaging devices like CCD cameras in the electro-microscope one can actually take video of the whole dynamics experiments in terms of digital imaging and then analysis the video later on.

To arrive at different kinds of conclusions from the experiments so compared to previous days where the videos where very what is called low resolutions now even images can be obtained the same resolution as the microscope provides us and then analysis to the best possible way so first of all let me just tell you the heating experiment.

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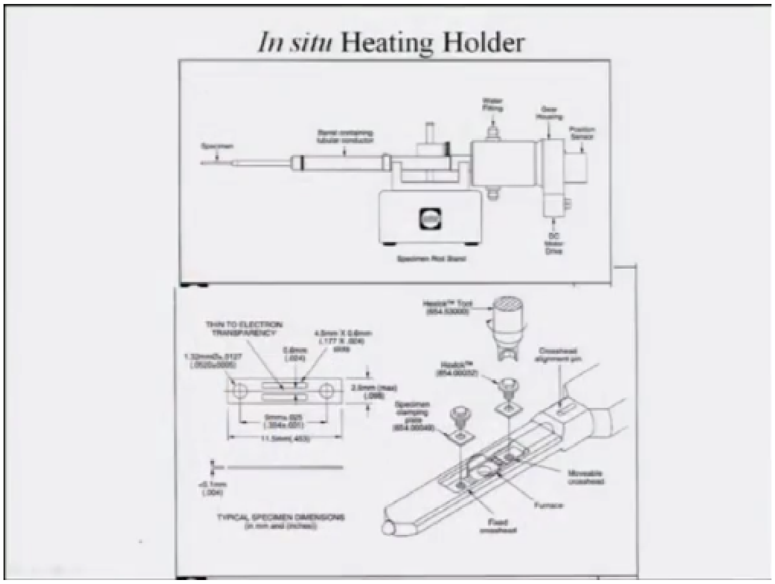

Normally heating experiment is done if see heating holder first thing you understand from this experiment is that TME mean same all the thing where changing is the holder so of you have different sets of holder one can run the same experiments different kinds of experiments actually inside a anything available in this world you do not need to have test full set up for this TEM so most importantly the high resolution microscope where the pole piece gab is very small there normally people do not allow these kind of holders to be inserted in because the holders are normally thicker.

So if the gab pole pitch gab is small then this holders may not be inserted properly and can create damage to the pole pitch or any other detectors they represents during heating or cooling or straining so therefore normally the microscopes comes TEM can be used except the high resolution configure microscope in fact people have reports that titian can also be used to doing the high situ experiments. So here I am showing such a holder which is.

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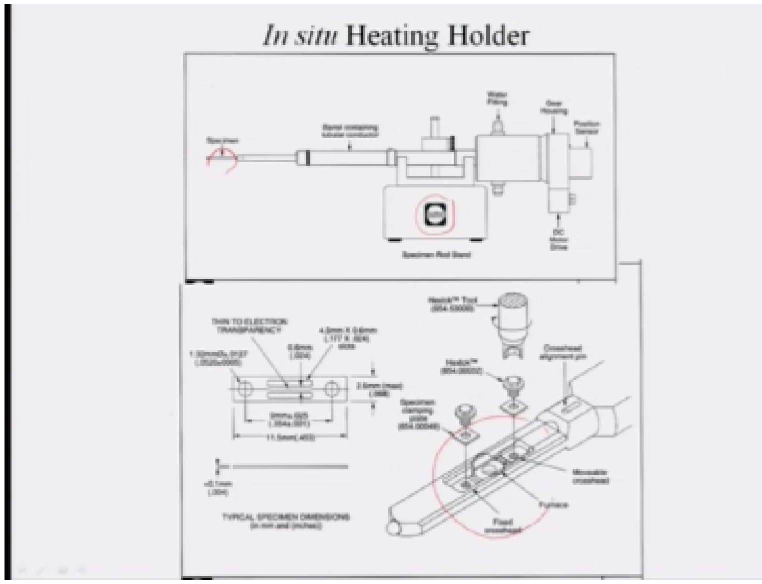

Which is basically provided by the company called get in corporation this is this slide basically of the cortesy to catten in corporations and you can see that this holder has basically for simple specimen loading set up here which can be seen in zoomed of we were where there is a finest the specimen holding positions and then this sample is basically inserted it screw down and obviously one is to use different kinds of spacer so that sample during hitting will not get well dead and with the holder and this.

Holder can be cool down using water inside this Furness felling water to this pointers and it can be tilted a single term or double did more both are both can be the features of sample and depending the type of Furness is use you can go up to maximum temperature up to some cases 15 added de cells yes, so it allows us to hit a sample to different temperature depending on our mean, so that as I said different kinds of holder available in this world are then get on many other manufactures which are preparing.

These holders so this are very generic so I will not going to go detail of this holder type I will show you some experiment this experiment this particular one is taken from my own pre this so were I worked on Iran Ge thin films.

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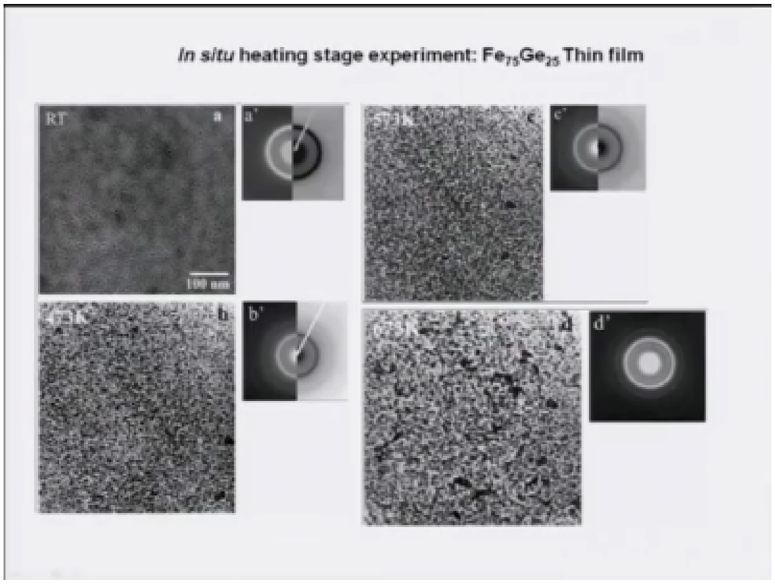

And as you can see at if I deposed a thin flames using basically deposition like lesser abbreviation here the structure is basically I got first what can be obtain from this diffraction part on this very broad among first ring and this corresponds to that this thin films are force in nature, so basically one will like to know that will happen to the among first thin flame when it is heated or rather one will like to know stimulatory of the thin flames this can be done, is easily in a heating volt of the one I have shown you.

We just insert the sample inside it heating holder and heated up as I heated up from room temperature to about 473k that means 20^0C as see there is a distinct change and the bade filed micro up correspondingly electron microscopic the diffraction pattern gets a little bit modified but not clearly same, but there are black regions increasing in this micro where as compare to the room temperature one, as you keep on heating 270 to 573k there are peaceable change is happening but most visible change 6 place at 673k.

But you started seeing this black crystal why they are quite big as you want to remember this is 100n bar so this crystal should be approximately about 20 to 30n and similarly the diffraction pattern gets changing you started seeing in this part a rings here they are so you started seeing the crystals coming in the picture.

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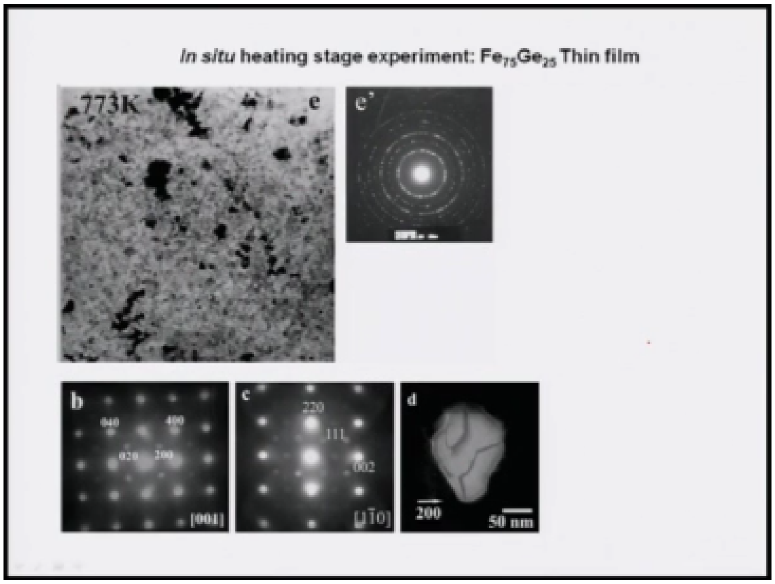

And as you see it alone and this temperature 773k that is approximately 5000C temperature, so you see this whole thin film has been crystallize and this ring diffraction patters source the dual nature, so therefore if you want to do such kind of experiments in the inside a TM column we need to have a to take the diffraction patters at the along with the bright filed or dark field images, so once this thin film is fully crystallize at 773k it can cool down by switching of the Furness.

And then can be looked into the microscopic again and as you look into the microscope again for the individual crystals which as form like this one such a bigger one we can actually obtained very nicely the micro diffractions patterns from this crystals and so they are indeed dual theory order in this case DU3 is a particular are nomenclature used to depict kind of ordering presents as in fact one can actually show that there anti phase domains since in the particles by doing this experiments.

So what you understand from this experiment is that just by simply hitting a thin film inside it microscope column with this heating holder can provide a, so much of information's similarly one can actually do routinely this is again taken from routinely the hitting and cooling experiments for the melt disorder we can study of the by physic or two phase nanoparticles which you routinely do during as part of around this as activities.

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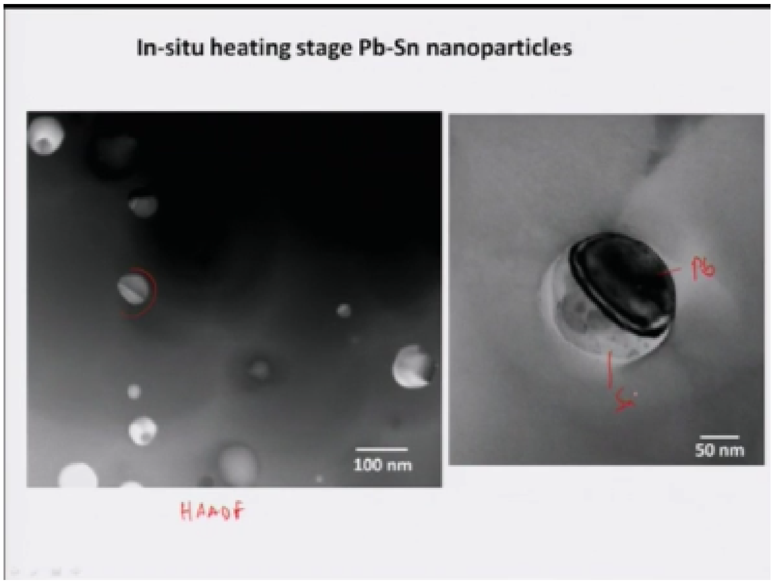

What I this is one I have shown you in a very first lecture of this course are what you see here is basically this two phase nanoparticles present in the hard if this is basically hard of image high angle or duck film image and in the bright film image you can see that this is basically late and this is a thin crystals in a one single particle and obviously has a metal sign this one will like to know how stable has particles where there any interaction within the laid thin parts during heating out or cooling also from the high temperature to lower temperature.

And as you know let in mails about 3D cell system pictures are there for heating in not to be done it to the very high temperatures, so this can be done very easily in any hitting holder provided that heating holder can precise heat in the temperature, so this is this snap sort of pictures what happens is again taken from our my own or own wok form of research group if we align a micro the particle is such way.

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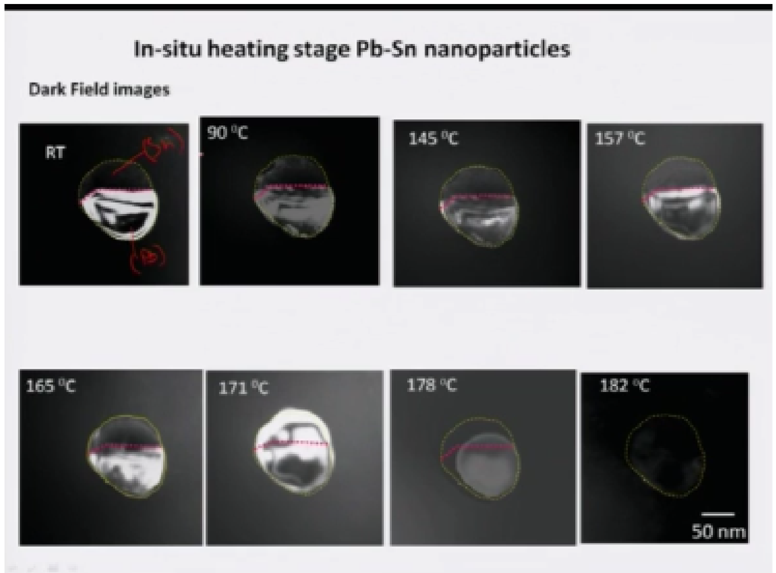

The light gets basically illuminated during duck field and tin is basically not oriented properly, so that they duck field can be obtained as you heat from the room temperature to our 91 41 47 to even 1820^{0}C temperature as you see there is a pro change in the particle during heating so from room temperature to 157^{0}C temperature there is not much except will the ball fraction of the light is getting change, but as you heated 171^{0}C whole particle with our full light solutions and then at so on 70^{0}C temperatures starts.

Melts down and find electron 182^{0}C temperature if you melt this get in more in fully therefore this kind of particles which are embedded inside of matrix can be easily you know studied by insitu heating microscopy and lot of experimental information's are results can be obtained from there we can get information and stability of the particles and they be here of the particles and do the heating at even microscopy resolution level.

This provides the information regarding the alloying at the nano scale so this is one such experiment which people do routinely now – a- days inside a microscopic specific column.

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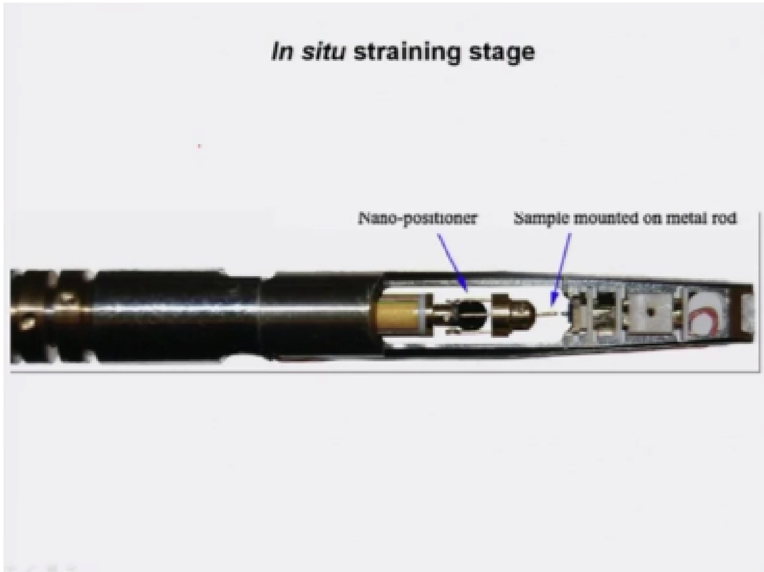

Rather kind of stage another kind of where other holder used in the real microscope experiments you know experiment is called straining stage this one is taken from high citron tanks to them. So there is a holder where actually one can actually do nano indecent test inside the transmission electron column what you see is a basically the sample mounting stage and this is the in and tar and this is position.

So once you mound the sample in side on this rod and them it is substitute electron toss point obviously and then apply a load to this in enter will go and hit the sample and then we can actually get information regard in the information behavior of the sample.

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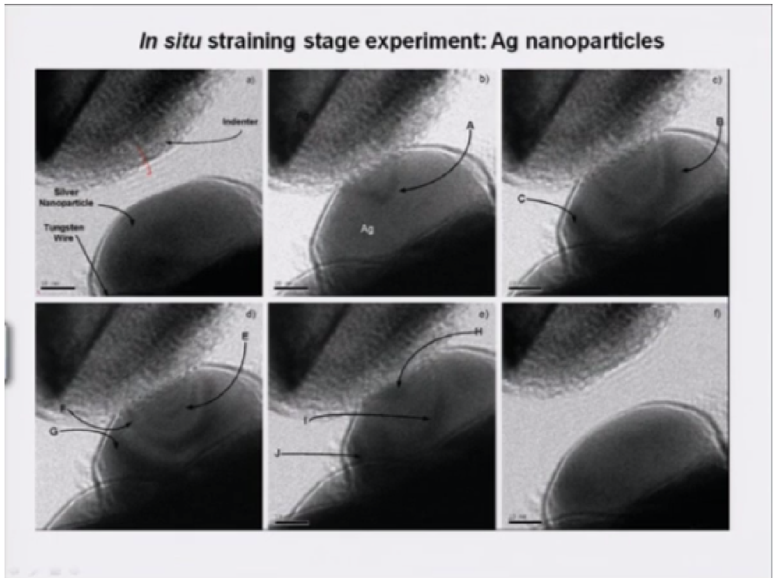

To show one such again from our own work if we take a silver nanoparticle and bring the indenter at the close to the particle what are the change is happens, as you see indenter is basically looking not a like sharp indenter at the microscopy level this is remember this is 10nm bar so the and this resolution level this is looks like a flat or round rather and that is come close to the particles gets quizzed as you see their particle gets quizzed and once the indenter is taken out see it is lives behinds of permanent information in the silver part nanoparticle.

This is again can we done or anything you can do one any kind of material just putting inside the microscope column and then just deformity using this straining stage. So in sort I just wanted to show you in the last ten minutes are so that what can for different experiments can be done using this what is called it is easy to studies, obviously there are problems while doing the in situ experiments in a transmitter electron microscopy column we cannot neglect the effect of electron during heating of the sample or dealing in fact phase transformation of the different phases there in the experiments that is cannot be neglected.

So therefore there is all is an effect on this electron beam higher electron beam affecting the beam where are the material during this easy two experiments. Then there are problems of what is call the resolution problems of booing them booing the material and during the experiments many cases some cases what happens, while you are viewing the sample then expiry heating stage or cooling stage experiments sample guess contaminated so much because of the material present around in the sample to be do not get any information at all.

So these are the caveats you must remember once you are doing the experiments and once you are analyzing the experiment results from this in situ experiments. So next thing which we are going to discuss or basically we are going to switch over from now onwards from tm to the SEM characterization techniques.

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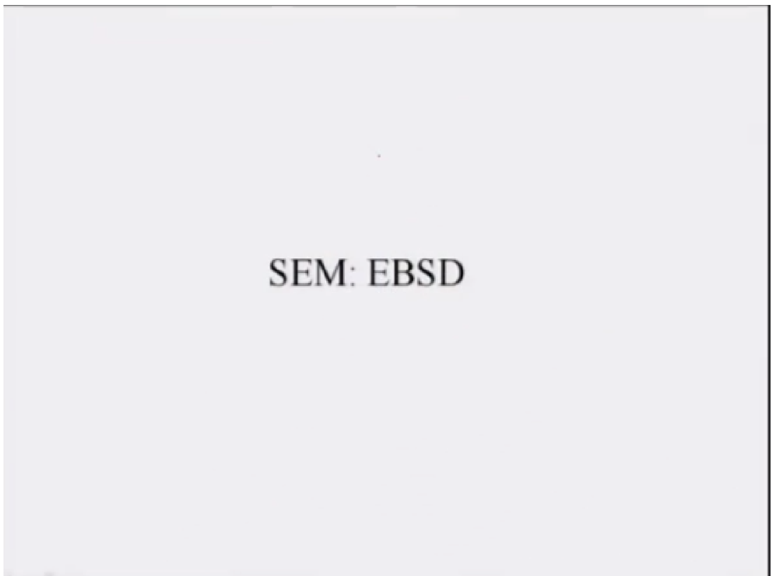

As I told you in the last class, so the scanning electron microscope which is said routinely used by all kind of users because of the EBSD is to use for characterizing the micro structure of the different kinds of samples can be used for advance characterization also, nowadays scanning electron microscopes are available at a very high resolutions and also with the very high beam current also because of that lot of experimental studies can be done which can provide us information regarding this sample behavior in the microscope in the real processing conditions.

So EBSD is one such but before I discuss EBSD I will like to give you some idea of the scanning electron micro scope because you might have for gotten from your basic characterization course so do you just next five minutes or so to just give you some basic idea about the electron microscope then I move on to the EBSD. So scanning electron microscope is basically very simple as compare to transmission electron microscope it is a very simple microscope in the same side number which places a small and the accelerating voltage is pretty low.

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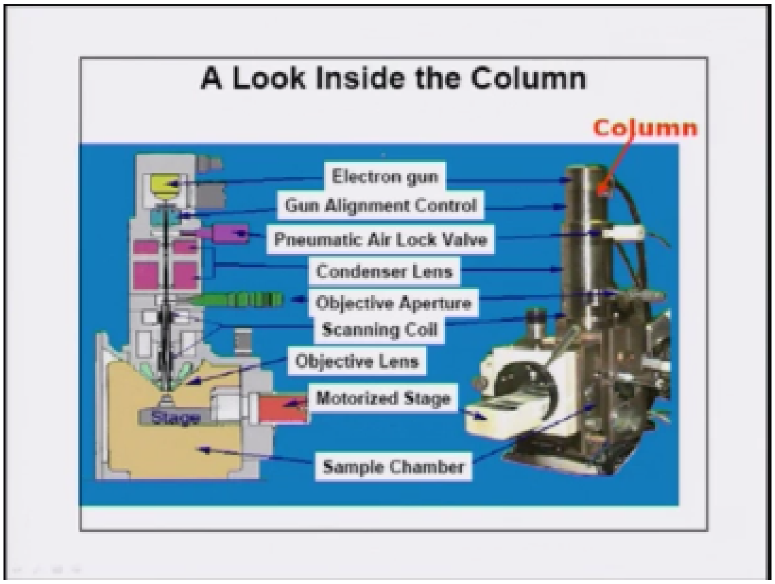

Compare 200 or 300 k volts we are going to continuous from 20 to 40 k volts and not only that the imaging basically imaging is totally different as compare to transmission electron microscope here. So what you see basically is electron guns source which can be either a tan stein or lab six or FEG the fill a machine gun which is normally used nowadays and then this is basically aligned a focus are there be using a lens assembly the condenser lenses.

And then there is a objective lens which resort which actually focus is the lens electron beam or the sample, and then you can are a scanning coil have sitting which will alive you to raster the beam on the sample sub phase and then obtain the images at the same scan rate as the raster speed. And there is obviously this is sample chamber in a electron microscope and there you can fit lot of things as I discuss you to the during the electron beams falls on a sample it interacts the sample and generates all kinds of information's for a sample starting from the second electron beam to back scared electron beam to the exit is x rays or maybe you know adder information like RG electrons everything is possible to obtain.

Depending on the type of detector uses can electron microscopic and actually use this signals to obtain image is and this images can be further studied, so normally in a scanning electron microscope what you basically it is nothing but a TV as usual look at it.

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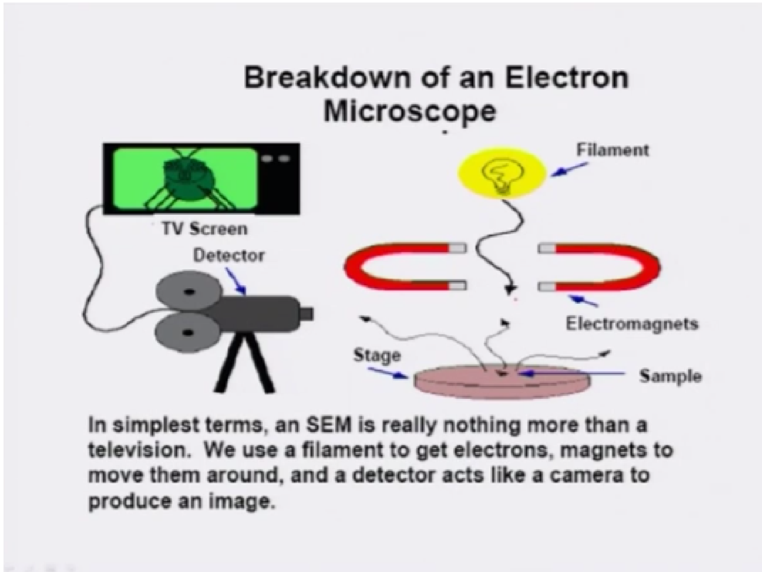

That is what I can say you so there is a filament which gives electrons and there is a sample electron falls interaction a sample and obviously the electron beam can be focused by using electro magnets which just nothing but the objective lens and then after this interaction of the electrons which arte the sample we generate signal the signals can be process using different kind of detectors.

Normally one can use second electron detectors or bask electron detectors form the normal imaging purposes which are mounted here ordinate a sample or a one can actually use r rays tool detect the different kind of elements presents and quantify them, this is a very simple one as compare to the tm.

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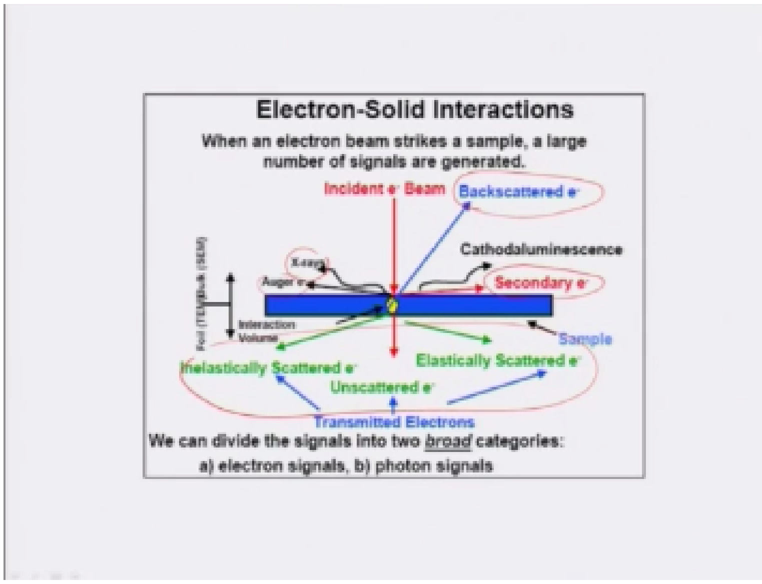

As I shoed this is picture slide I have showed in the beginning of this discussion on the microscopic techniques as a electron forms on a sample in generates all kind of signals you can have a secondary electrons or you can have a backscatter electrons which are based under you can have as x rays or you can have Ager electrons or you can have a Cathodaluminescence on the coming from the tops of the sample one of the electron is falling.

On the other hand if the electron passes through it generates other kinds of signals which are not use for the SEM normally which are this kind of signals are use in TM which we have discussed you could have elastically scattered electron or un scattered electron so many other things possible, which we have discussed in the transmitted electron microscopic. As obviously as you see there are electron signals and proton signals coming from the samples.

And in a scan electronic microscope electron signal and the signal electron and back or other electron beams also both these beams they are used image whether the x rays which are basically photons they can be used for determining the element compositions also. So therefore basically is a using the signal and plotting this signal on a TV screen that says cm scanning electron microscope.

They have seen a scanning electron microscope but rarely you will find people or very are you will find the idea that scanning electron microscope can be also used to obtain crystal formations. Normally our basic understanding is that we can obtain all kind of crystal information at a fraction contrast in a terminology microscope, but in a scanning electron microscope also we can get lot of this information provided we know that how to obtain this information how to use that.

That is what is related to discovery of electron vast scatter diffraction if we look back in the history it is come from the channeling patterns, which normally people have seen in the long back but only in 1990s they are going to computers and identifying the processing of the signals we could see a rapid change in scatter material using this technique so many of this microscope as detectors are actually come up in 1990s and later.

Previously people used to do all kinds some kinds of diffraction study in scanning electron microscope they are very limited so therefore this is again I have shown it here how the electrons comes into the picture because Backscattered electrons.

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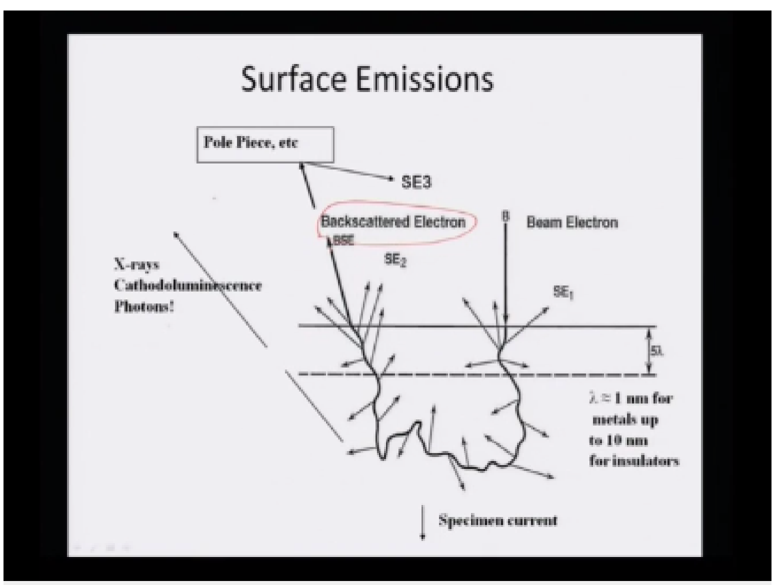

Very important for the image study, so as the electron falls it just goes in the sample and the secondary electrons but it can actually get scattered and come out. Many times people says Backscattered electrons is same as which is fallen of and then getting scattered back, that is why this is called Backscattered electrons in the scanning electron microscope and this Backscattered electrons actually having very high energy more than actually 50 to even higher.

So these Backscattered electrons can be then obtained and as they say the carry the channel information which you will see right now.

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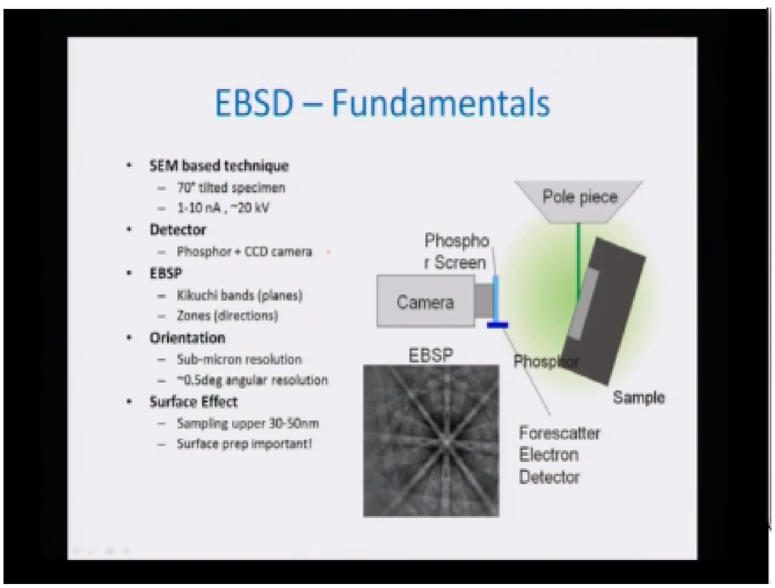

So first let me just go to the board and tell you what actually happens or what is unique, unique to the Backscattered image is the information related to the crystallographic nature of sample? So origin of this contrast.

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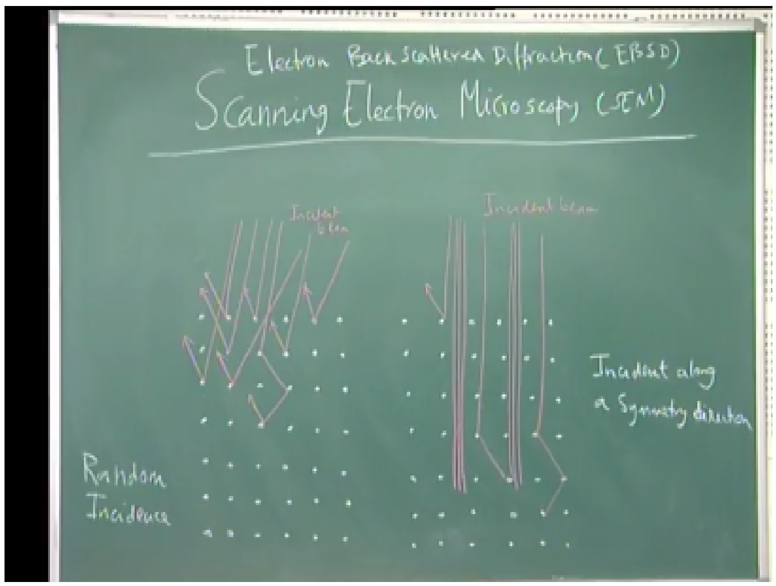

Can be easily obtained from these two pictures, let us suppose these dots white dots are actually atoms in a crystal and then electron beams are falling on the sample. In the 1st left hand side of the picture is to see which I am just putting my finger my hands on where the electrons are falling randomly. If the electrons are falling randomly and can get scattered by the atom or the nucleus and then they can produce Backscattered electrons. So if that is the case the Backscattered is very high because we are generating large number of Backscattered electrons by this way. On the other hand if suppose you have this sample or the crystal oriented very nicely and the electron is falling along the symmetric direction of the crystal.

So what will happen in that case is that it will tend to channel within the plane that is what we have shown, you can see electron beams are channeling through this and this because of these Backscattered yield are very low because all the electrons are channel through, very less electron beams will be scattered and produce Backscattered electrons. So therefore all though this is the physically what people think that the region or the origin of the channeling in a basically crystal.

Now there is no way of confirming it or doing an experiment and see that this is what the normal concept or the idea we have. Now suppose if the angle up incident actually of the electrons beam and the crystal can be varied at a you know from the random incidence to a particular direction similar to direction of the crystal it can be varied then what will happen is that we can modulate the Backscattered electrons yield at different angles corresponding to symmetric excise of the crystals.

And if we do that if we just vary this electron incident angle from this random to the symmetric directions then we can actually modulate the electron density the intensity of the Backscattered electrons or actually yield Backscattered electrons and get what is known as channeling patterns and this is what I have shown you here.

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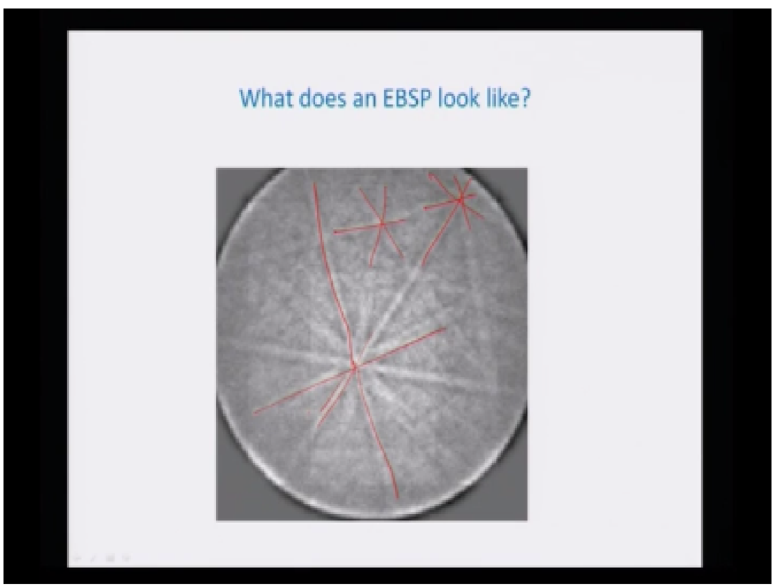

Let me show you one such pattern you can clearly see that the channeling pattern looks like bands okay, they are actually bands like this and this bands are actually meeting at a certain points, this one is taken from pure iron crystals which is taken from our own work and these bands are actually some cases meets like this, those of few we have done they are actually similar to the bands in the electro microscopic.

In fact oraganing of these bands can be traced back to the kikuchi nature that is because of the in lasses scattering of the electron beams. Now obviously then if that is the case then this can be related with basically we can get information regarding the crystal, the atomic spacing. So angular width of this band is basically this one is approximately twice the appropriate bi angle for the given lad spacing and electron wavelength.

So that accelerating voltage is specifying can be reduced how the measure width after the angular scale of the pattern obtained most importantly w3hat you understand from the basic thing which I am showing I am going to discuss in detail about how this permission obtain and how this analysis is done.

So on this is this contain both the space and the angular information basic information is spacing nothing but they are the position of the crystal from or micro structure on higher you getting this patterns and angular information means one the crystal is self how this spans are related to the original crystal both this information are inbuilt within this patterns so what I am going to do it in next five minutes time is that is to show you how this are actually obtained.

And then I will go back to the microscope I will actually take you to the real microscope which will have in our IIT Kanpur campus and I will show you how this are obtained while doing an experiments and then I will show you how this can be proceed also so first let me just show some kind of muscles inside the scanning electron microscope so what is then in a normal scanning electron microscope is that the sample is the reason it is triggered is to very high angle 70 degree.

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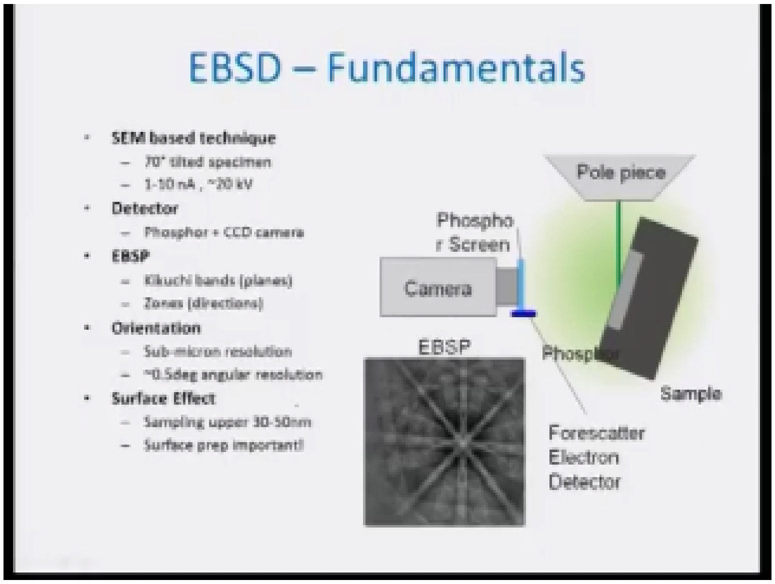

Because it can be increased the detector the Pascal detector can be met close by the close of electrons from which generated coming across sample surface will be allowed or will be going to the detector march larger fashion so can be increased normally the we use fake or the columns like the figures like film guns in the electron microscope for the beam intensity is quite high as a detector has basically a phosphor screen or we can use CCD camera.

And this patterns which are called electron basket at diffraction patterns are electron basket pattern either EBSP patterns or pattern so they actually contains bands as I told just now or obviously they will be alive on a certain journal axis pattern which I have shown you there and they contain information regarding the orientation of the crystals with the angular relations of the 0.5 degrees.

And obviously there will be a other surface like sampling can be cannot be done if the crystal lies less than above 50 nanometers because then there will be a overlapping of the information and there is obviously surfacing permission surface sample permission is very important because there is a oxide layer surface we really do not get any information from the actual crystals because beam will fall of the oxide layer and produce first like.

But there is nothing but useless thing in a EBSD so this is what is shown pole piece here this is nothing but the pole piece of the objective lens the sample is which is titled very high angle approximately 17 degrees or more in this case and then there is a camera within the phosphor scale sitting here and the basket electron comes and fall on this and produce channel in panel like then one which is shown here.

So that is how we get information now we have a we have very large number of crystal in sample one can actually obtain this information from each point of the crystal and then process it this is how actually experiments are done but remember that the sample is to very clean just to be electro poles many cases to obtain oxide free surface layer so that very good quality first can be obtained okay how actually it works

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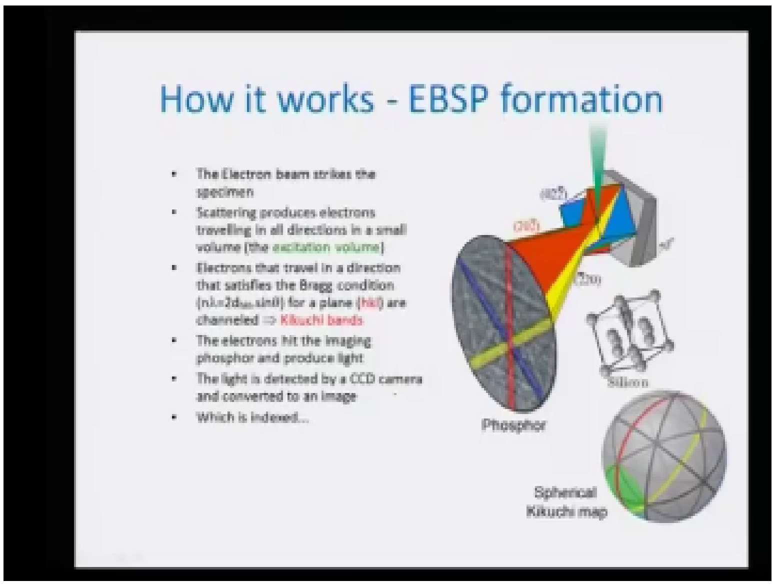

It is very again not very complex like many other trans electro techniques so we have a crystal oriented like this what I am showing you is silicone crystal here diamond cubic structure and then the electro wave falls like an crystals it falls on a particular plane or set of planes are and scattering of the lector wave happens from this from the like this and the scattering of electron travels in all directions in a small volume and electro that travel in directions that satisfies the bricks law let us say $\lambda=2d\sin\theta$ for a particular plane our channel.

And these actually lives to the permission kikuchi maps electrons hit the imaging phosphorus skin and produce a light as we known the triangle electron microscope is also used us with a phosphorous skin on which the electron falls and creates the light so light can be deducted by CCTV camera and the CCTV camera can be used to convert to this image and this patterns future obtained routinely can then be even tested by knowing.

The crystal structure all the material and then by knowing that we can obtain how is crystal is very intelligent to the sample the real advantage of this kind of techniques is that combined with TM is that here actually we can get information from very large crystals or large number of crystals in the micro structure because this beam can allow us to see large number of crystals.

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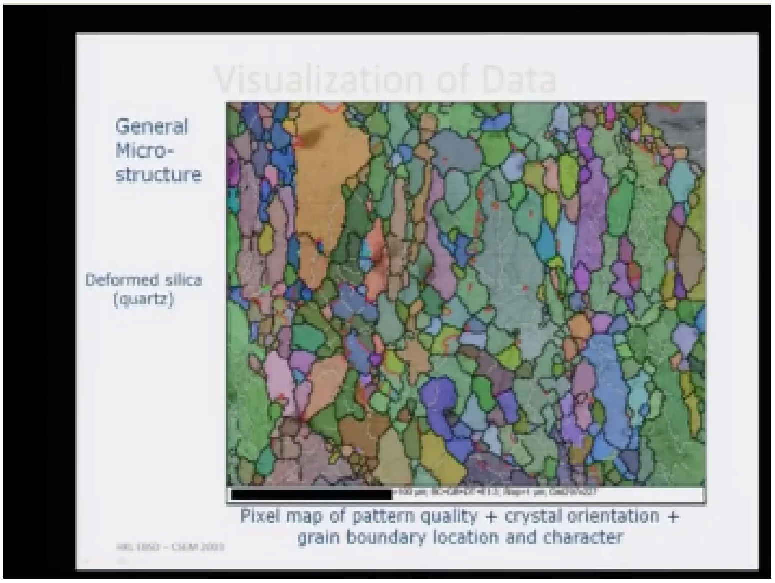

This is just one such picture what I am showing you this is as you see the different grains are mapped with different colors okay this is how the data is this is taken from one of the hk software manufacturing company in UK the oxford instruments we have the facility installed in our microscope is again oxford instruments.

This is just for their routine data so what you see is that the grains are different color depending in the orientation so if the once you obtain the orientation of the grains and depending on the orientation of the grain we can actually create a color map which is routinely done and this gives provides us lots of information gain boundary type of this expressions and many other things which we will discussed after we see the actually in the microscope next the subsequent lectures.

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