Indian Institute of Technology
Kanpur

NP-TEL
National Programme
on
Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-02

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Well in the last class I have discussed about the efficacies of using advanced characterization techniques and I also discussed that why different techniques to be used in combinations in the subsequent lectures we are going to discuss our different microstructure related advanced characterization techniques microstructure is the basic theme of material science so that is why most of the research studies involve only not only seeing the microstructure using different kinds of machines but also to analyze them.

So in this lecture we are mostly going to concentrate on electron microscope especially the transmission electron microscope before I just begin I must tell you that why the transmission electron microscopy is very important and how this technique has been developed over the time period as you know electrons are discovered in the beginning of the last century or maybe end of the 19th century and then people started realizing that electrons can be used for many purposes but as far as microstructure is concerned if you want to discuss about microstructure.

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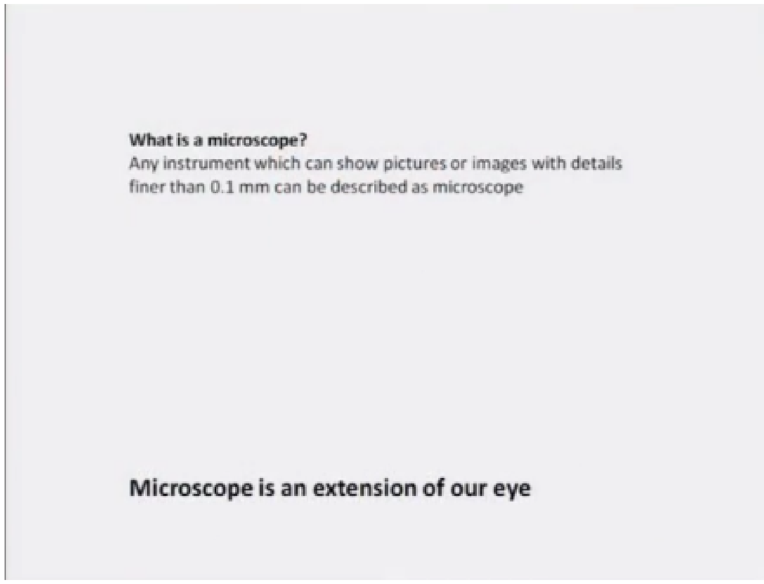

We should know about microscope so first let me discuss about the microscope what is, a microscope we know that our normal I can see the feature as small as 0.1 to 0.2 millimeters so I can see any future which is in this range.

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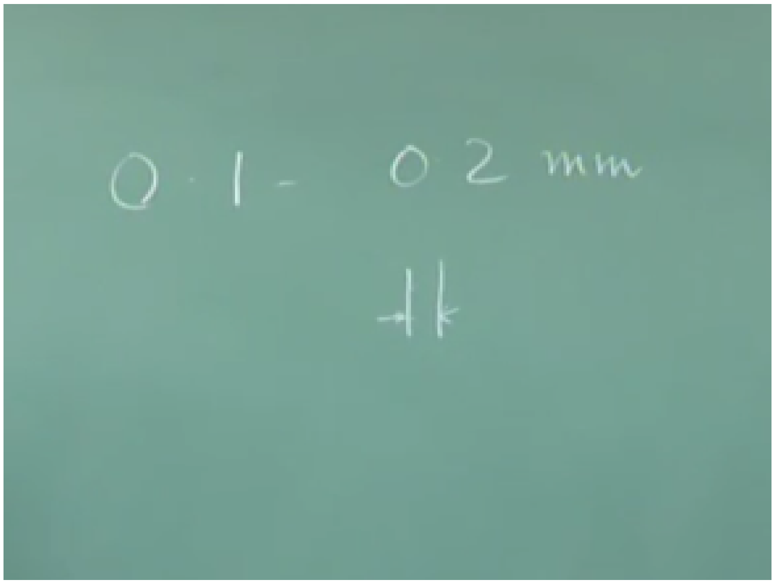

0.1, 0.2 millimeters that means if there are two points on the space somewhere normal I can resolve them very precisely the distance between these two points range from 0.1 to 0.2 millimeters say any machine which can dissolve two points closer than this numbers from point 1 to 0.2 millimeters can be defined as a microscope, so therefore this study of developing those kind of machine is began long back in fact in 17th century by Leeuwenhoek.

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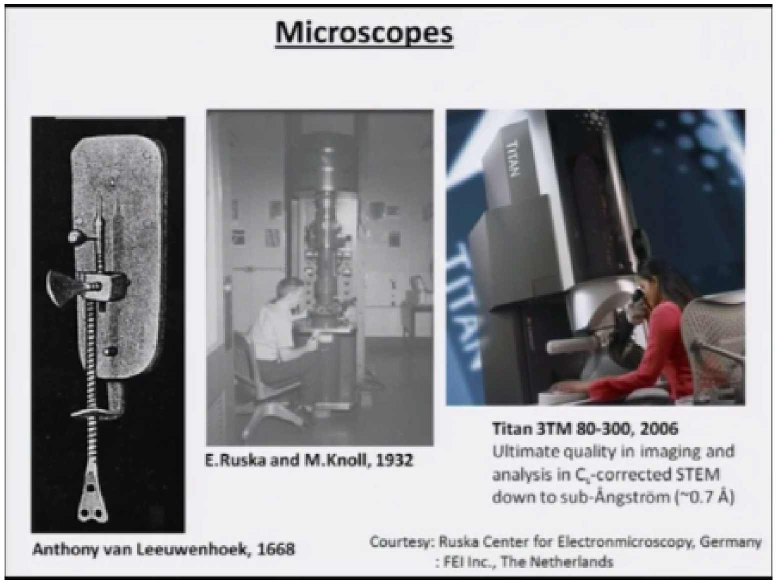

First time in the year of 19 1668.

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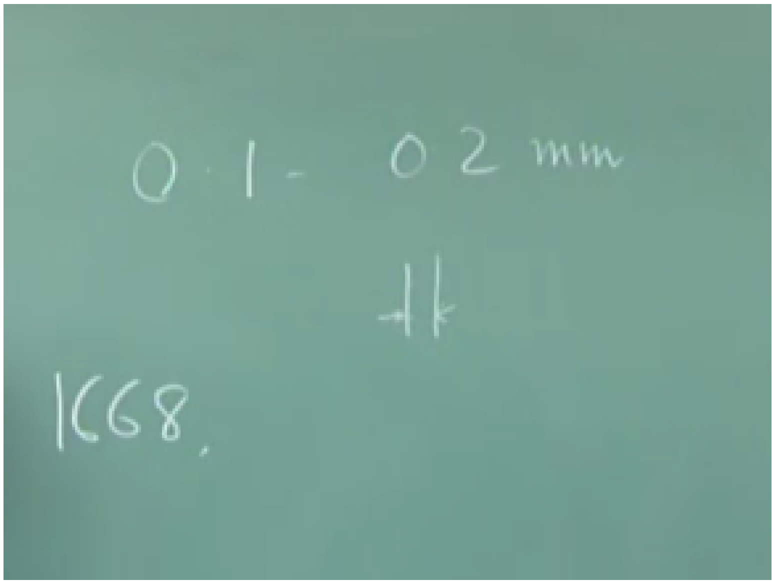

Leeuwenhoek discovered the optical microscope in which the normal light was used to image many important algae are even small features which are present for the small insects so that is the beginning of the microscope the using of instrument which can dissolve objects finer than or I can do but it went on for the next subsequent two centuries or three centuries the optical microscope was refined and used for many purposes but in the end of 19^{th} century and the beginning of the 20^{th} century people started realizing that electrons can also be used for electron microscopes are can be used to image things which can resolve objects much finer than what an optical microscope do.

So this has led to a lot of discoveries in the arena of electron microscope which I am going to discuss first then finally I go to how the electrons interact with the material the most major discovery which has propelled discovery of electron microscope is by de Broglie in the year 1925.

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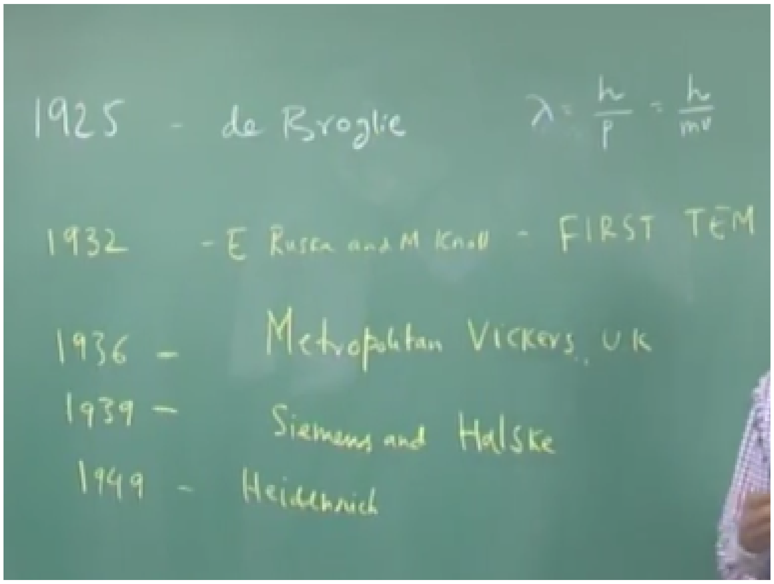

De Broglie in France he first time told electrons can be used as a wave and he said that electronic length can be defined by this formula where h λ is the wavelength of the electron h is a constant and P is the momentum of the electron so therefore we can write down h equal to this equal to h by mv where in the mass of electron and which the velocity of the electron it all started with the discovery by de Broglie in 1925 when he stated that electrons can be considered to be a wave with a wave length given by this formula $\lambda = h/P$ where h is the plants constant and P is the momentum of the electron.

So therefore we can write this is equal to h /mv where the m is the mass of the electron and v is the velocity so by knowing that electrons can be used as a wave or can be here like a wave one can use this particular feature of electron to make use in microscope within just next 7 years that is in 1932 to scientist E Ruska and M Knoll first time thy where stated that electrons can be used as a tool for microscope and first electron microscope a first rather Thomas electron microscope was demonstrated.

And this is a major discovery and for this discovery in fact Arms Ruska won Nobel prize in 1986 after almost 50 years so this said the tone are that electrons can be used as an imaging tool people know it because of the fact that electrons can a very small wavelength so let me just state how this history has developed first then get back into the efficacy of electrons in the microscope first electric commands electron microscope came up in the year of 1936 just after four years of first demo electron microscope so this was by a company named.

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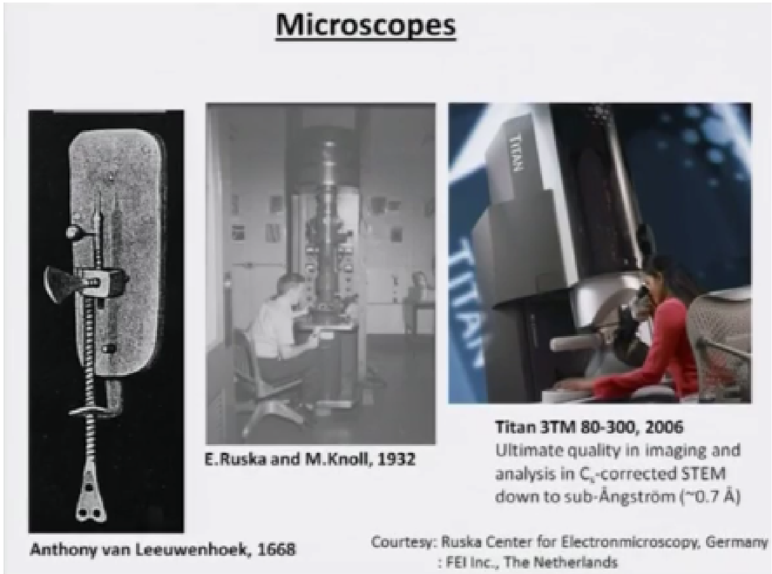

Metropolitan Vickers UK but this microscope was not successful so therefore the first successful commercial microscope came up in the years of 1939 just on the year of second world war by Siemens and Halske but this has basically made the electron microscope available even before the second world war but then after the first second world war they do not those days it was very difficult to prepare samples for electron my TM because sample needs to be very thin to observe under electron microscope.

Especially in T under TM so the tool for making those thin foils came up in later on by another German scientist in 1949 just after the Second World War known as heidenrich he discovered the technique to prepare very thin foil so that it can be observed by electron microscopy TM but subsequently many other companies not only Siemens and Halske but many other companies like Joule, Hitachi even Philips started manufacturing Thomas electron microscope.
And later on from 1950s to onwards TM was extensively used to image many samples which is either metallic or ceramic in nature so major groups which started working on this research was one in Cambridge University of Cambridge later on in this in US America so finally the theories of electron microscope was came up by few scientists in Cambridge.

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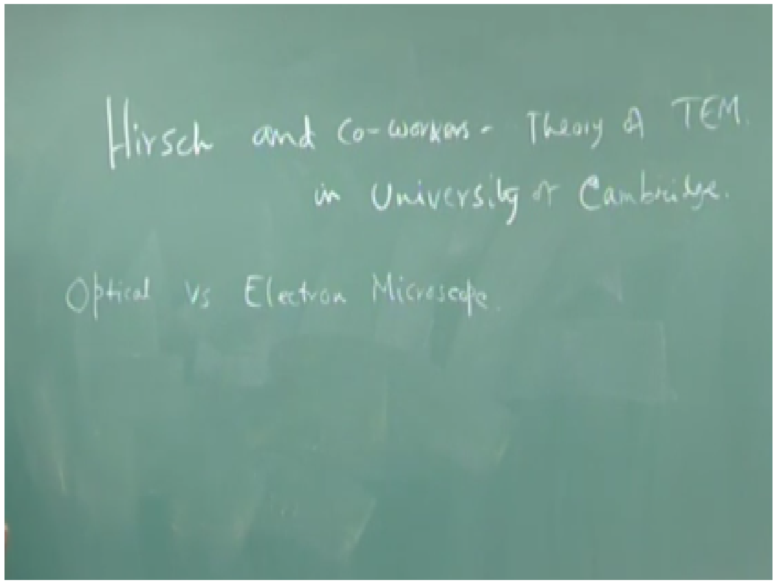

Especially from the Hirsch and his co-workers they develop a theory of TM in university of Cambridge and then onwards lot of work has been done on electron microscopes by different groups which I will not discuss so that is basically the brief the history of how as does this electron microscopes came up in the scientific community and later on used extensively to probe different kinds of phenomenon phenomena in material science and engineering.

So let me just now get back into the comparison between optical microscope and the electron microscope why there is a need to use electron microscope when optical microscope was already existing what was the basic reason for that then we can understand many other features we know that any microscope the most important feature is resolution.

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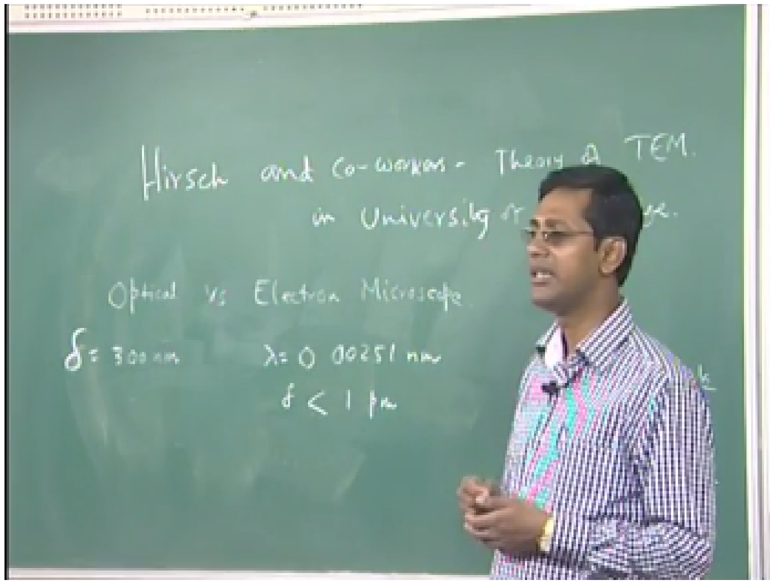

Resolution means ability to distinguish objects which are separated by small distance like the one I discussed few minutes back if I have two points separated by a small distance whether the machine can resolve this two point successfully or not and this was dealt extensively by rally and he discovered or he gave a formula to correlate the resolution with wavelength and other parameters.

So this formula is given by λ =0 .61 $\delta = 0.61\lambda / \mu \sin \beta$ where λ is the wavelength of radiation μ is refractive index of the medium and β is the ½ angle of the length of the collecting power of the lens so therefore if in a in extreme case if we consider $\mu \sin \beta$ to be = 1 then resolution become 0.61λ that is about 60% of the wavelength and if you consider the normal light which is used in the electron microscope, if the llama light has different wavelengths, if you consider the green light which is wavelength about 600 nanometers 550nanometers are not 60 nanometers, so the relieving resolution power of the light microscopes is approximately about 300 nanometers.

So therefore we can write down for optical microscope we can have resolution power of approximately 300nanometers.

So we cannot resolve any object whose side is more less than 300nanometers that is the basic need to develop electron microscope because we know that electrons has a wavelength much lower than the normal light, so if we consider the wavelength of electron we know that relative electron depends on the accelerating voltage and normally the wavelength of electron is given by a formula which can be written like this γ equal to okay, so a length of electron normally can be given by H / 2 m0 e $V^{½}$ or rather.

We can write down this is equal to γ 2 m0e constants and V to the power half, so therefore sorry h / 2 mge ^{½} so that means the wavelength of electron is a strong function of the voltage by which it is accelerated, so if we calculate that wavelength of electron at 200 clear bowls which is used to normal in Turkish electron microscope, so γ will be approximately 0.0251nm, so immediately you can see that if the γ is an electron microscope is given by this then Δ will be less than 1pm acting to this formula given by this.

So therefore one can clearly see that it is possible to reach a resolution theoretically from300 animators in optical microscopes to less than a picometer in a electron microscope but normally we do not achieve this resolution because of the problem in the lens, so if we consider the optical versus electron microscope the Trans electron microscope there is a huge benefit in terms of the resolution or resolving power of the microscope by knowing this one can in fact go on and develop different kinds of microscopes to get understanding of the phenomenon happens in the material science engineering let me just show you few of these features.

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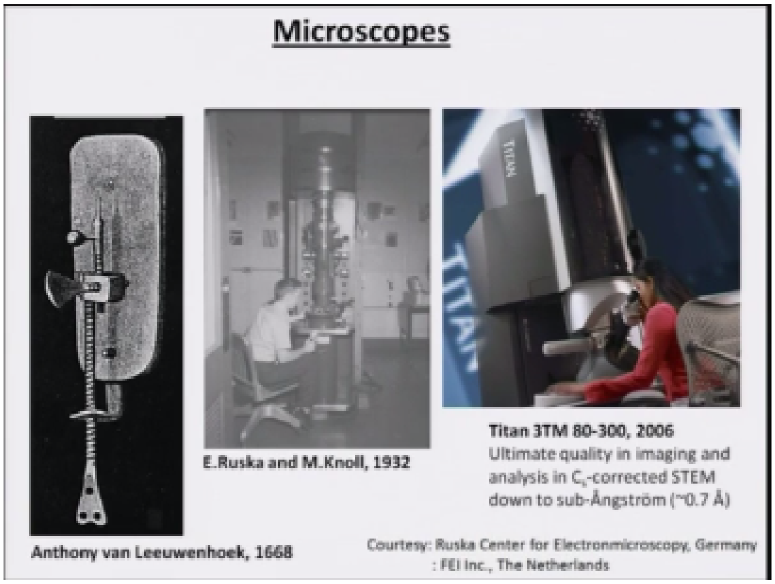

So this particular slide shows you how the microscope has developed from night 1668 by Leeuwenhoek to the 2006 by FEI a company which has manufactured the highest best possible resolution in a times elector microscopes obviously Ruska and Knoll contribution is extremely huge as far as the first microscope is concerned well from 1933 to 2006 in this about 80years approximately microscopes electron microscope seen a drastic development, so using the Titan microscope which is now sold across the whole world by the FEI one can reach a solution of order of less than 1Amstrong as i mentioned that although the theoretically possible resolution in the stereo microscopes.

One picometer but we can never achieve because the problem of lenses so by correcting different lens, it is possible to have a solution of approximately points 6 AM Stern which is the best possible solution till they asleep till today.

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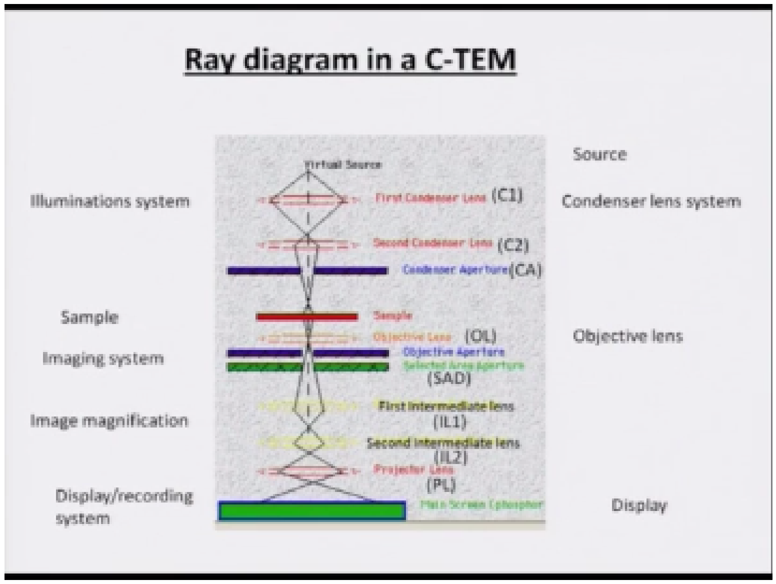

Now obviously one needs to know that in a normal time missing electron microscopes what are the different things are there as compared to it optical microscope let me just go back to the optical microscopes.

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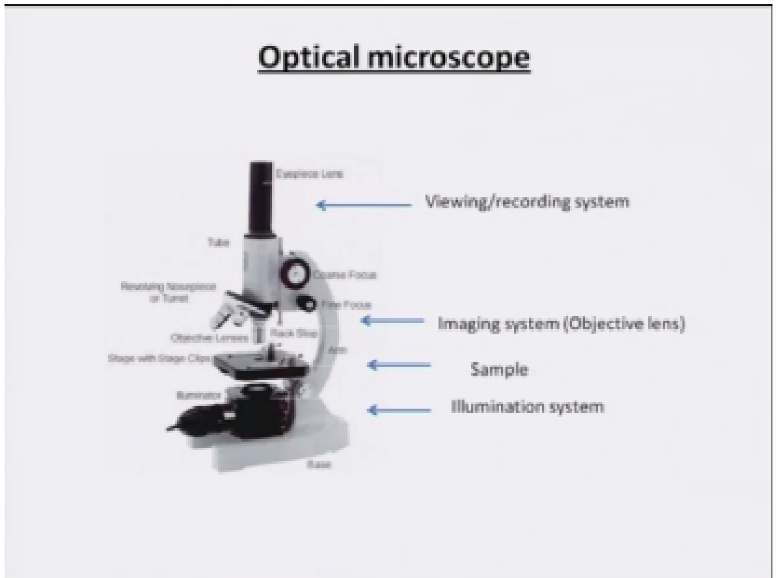

Optical microscopes consisting of basically three things one is illumination system that is a light in optical microscope you can either have an illumination system coming from down in a biological microscope or an illumination system came from top in a metallurgical microscopes so this provides a source of the light and then we have to have a sample obviously which is placed on a sample holder at the top of which we are basically imaging system which is nothing but an objective lens.

And from the objective lens the image forms and we can see this on eyepiece so that is basically a viewing or a recording system either you can view using a normal eye or you can put a camera so this is the basic construction of an optical microscope we will see that x electron microscope looks exactly similar except that a lot of complicated slain system inside and term x factor microscope and our source is obviously in electron beam not an optical light.

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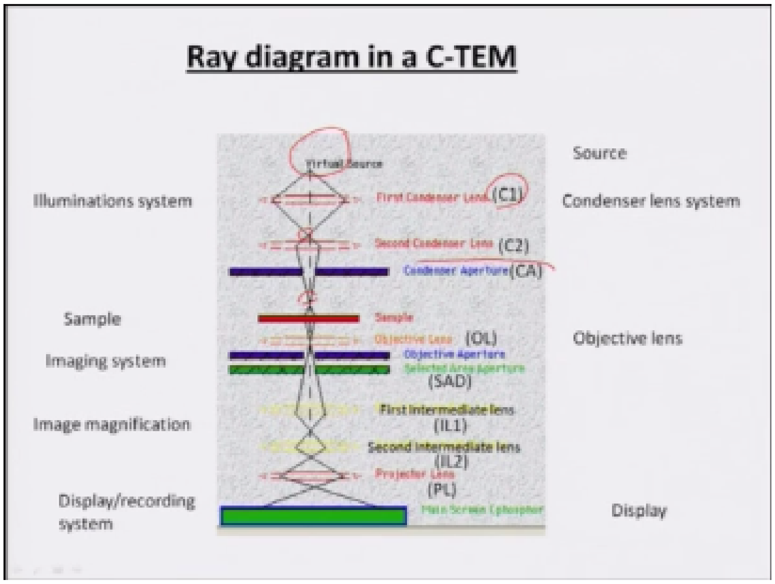

So if I describe this in terms of a simple ray diagram this one looked like this so in the slide so you are basically a source basically a source here sources can be electron will discuss how the different sources can develop an electron microscope also then from the source the electron beams are basically focused by first condenser lens to a very small focused beam and that focused beam can be again d focused initially buy a second car nestled in also can be focused later on in the on the sample normally in electron microscope.

We put lot of apertures to select a particular electron beam, so condenser aperture serves the one of such aperture choir which can control the beam size sample is kept or emerged rather within the objective lens I only toll in the few minutes back how the orbital ends is kept in optical microscope in electron microscope sample is just placed or immersed within the objective lens and then of these beams which falls on the electron beam which are falling on the sample they some of the beams are diffracted some of the built as meeting to the sample those beams can be used to form images by using objective lens.

Or they can be used to form diffraction patterns by the objective lens diffraction pattern normally forms of the back focal plane of the objective lens so these facts and patterns can be either a select area diffraction pattern or can a micro diffraction pattern or can be even converse of electron diffraction pattern depending on what kind of techniques you are using and this image is all the diffraction pattern can be then magnified using intermediate lenses or the projector lenses this is in a nutshell a structure of it conventional tem.

So therefore it is not as complicated as we think or as we observe a look at it in one of the lectures i am going to show you the actual x electron microscope and how is operates but for the sake of understanding today you can squarely see that, if we have a source electron beam source we can use it to form an image on a plate this is mainly because the electrons cannot be seen by I so therefore when the electron falls on a phosphor screen then all you can see the image or the diffraction pattern.

So that is kept at the bottom and that is why the person sits and looks at the screen when the microscope is running as a sample in the column the whole thing has to be under vacuum because electron cannot travel, so the whole thing is under vacuum you click on cannot travel in air so that is why we need to have very high vacuum system to have a very good microscope and the samples obviously are to be thin enough, so that electrons can pass through it candy fact it so as you can clearly see as the electron falls on sample there are a lot of interaction happening. And so now next thing which I am we discuss is that how this interaction can be you stop.

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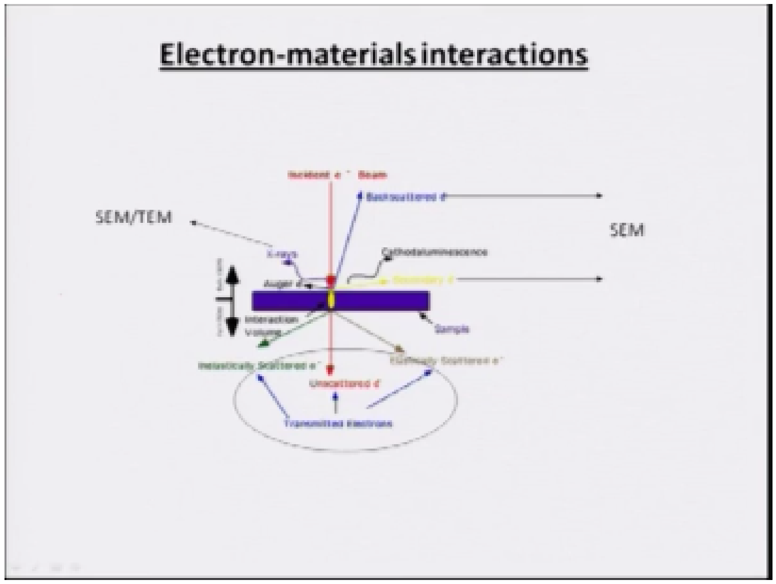

So let me just go back and tell you how this interaction can be used in the real for different purpose of the times electron microscopes.

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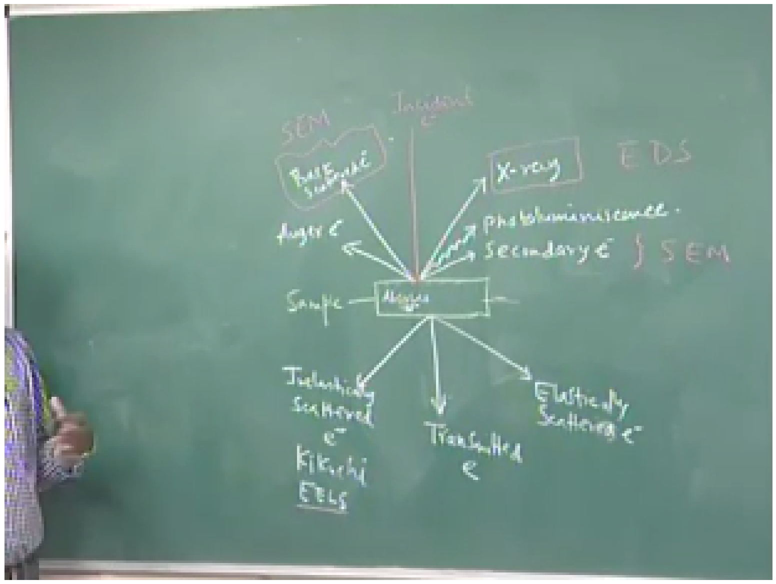

If I consider this to be my sample and let us assume that electron beam is falling on a sample this is my incident electron beam we are going to see how this electron is going to interact with the sample, because this interaction will give us an idea how they can be used for different kinds of analysis obviously for some of the electrons will get absorbed inside this material they can we call as absorb electrons, so the electrons will pass through they are known as attachment electrons sound the electron spiritual pass through.

They are actually passing this electrons which a transmitter passing through without undergoing any kind of deviation promise original path, so that means these electrons are not undergoing any scattering but some of the electrons which are passing through may undergo scattering or diffraction so these electrons can be of two types one which undergoes elects elastic scattering or we can call that elastically scattered electrons and other electrons can be elastically scattered electrons.

So elastically scattering electrons are the ones which undergoes the normal electron diffraction and from the electron diffraction pattern unless the scattering electrons can lead to different kinds of things obviously one who have seen the electron microscopes, we have seen the kikuchi lines activity bands they actually form because of the in less scattering electrons or we can use it for spectroscopic analysis like in energy loss spectroscopy which we will discuss later on now if let us look at what happens to the electrons which are reflected back or scattered in the direction of the another direction.

That is opposite to the direction radiation beam some of the electrons will come from the very small thickness of the top surface of the sample they are called secondary electrons some of the electrons this electrons actually generated because incident beam electron is having very high energy, so once this electrons fall on the sample they can eject something electrons from the outer cells of the sample and because of this electron is ejected from the outer surface samples will have low energy they will just come out from the surface and can be called as a secondary electrons some of the electrons which are falling daily from the on the sample may go and hit the Electra the nucleus of the atoms.

And they can be dead back scatter so they all call us a back scatter electrons okay not only that is how the incident electron Maeve and cause rejects ejection of the electrons from the outer cells of the inner cells not, so they are the cells so how the incident electrons can actually eject electrons from the inner cells of the atoms in the sample and once this ejection happens some of the electrons which is outer cell can jump in into the inner self which has become vacant, now and because of this transition from the outer shell to in a cell.

They energy can be released in terms of x- ray so therefore we can have x- rays coming out because of the interaction with the instant beam and the sample, so many cases it has been found that auger electrons can also come out from the top surface of the samples some of these samples like semiconductors they can even produce photoluminescence, so once you know that electrons during interaction with the sample can generate.

So many different types of signals we can use each of the signals for different kinds of purposes the signals which are passing through or other transmitting are getting either getting diffracted are not getting diffracted they can be used basically for the transmission electron microscopes okay x-rays generated by this kind of interaction of the sample can also be used by the time selector microscope which you will discuss when you discuss about the in this I elect energy spectroscopy.

The electrons which are described as secondary electron and bastard electron they are used in SCM for imaging rj electrons are used in audio spectroscopy for getting composition and the event to know the state of the electrons x-rays as i say it can be used for another dispersive spectroscopy or EDS analysis both in his he m and the TM and as I say photoluminescence can also be used for different kinds of semi-automatic analyzing the photoluminescence characteristics.

So by knowing that electron can interact the sample and lead to different kinds of signals I can we can now device the techniques in electron microscopes especially in terminal and scanning on microscopes to utilize each of the signals and provide the information from the samples which you are proving so next I am going to tell you how.

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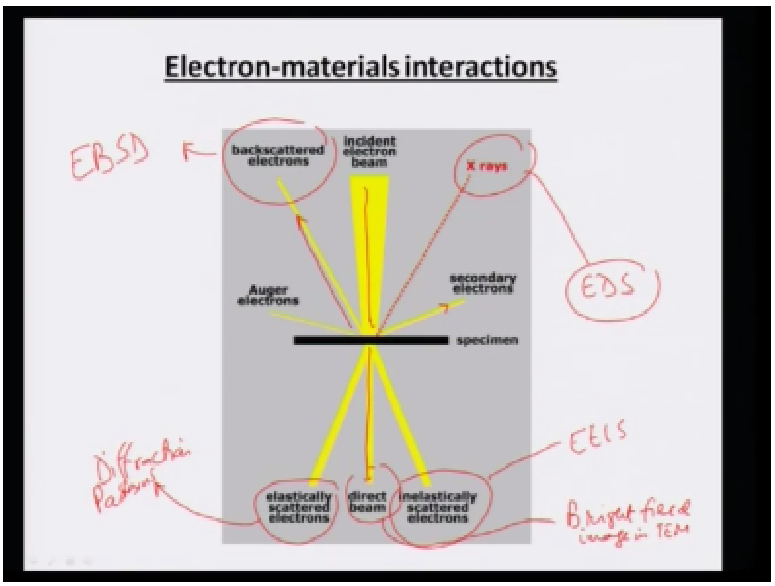

This can be used so this slide what has been shown is the basic features not all the features which I have shown in the board basic features so if you see the incident electron beams falls on a sample and then we have a direct beam the beam or the diameter William electron which I have written there which has not undergone any kind of scattering so that can be used to me to form what is known as bright field image in TM on the other hand less cutie cat or electrons which is shown even.

On the blackboard can also be used to from diffraction patterns English is scattering electrons are normally used in energy loss spectroscopy electron energy loss spectroscopy and for the electrons which like backscatter a second election as I said they can be used in the scanning electron microscope x-rays can be both used in the TM as well and ACM for energy dispersive spectroscopic analysis to get composition analysis in the sample so that is in a nutshell what is basically used what.

I have not talked about it is that this back scary electron can also be used to obtain crystallographic information in ACM there is a new technique which has been developed a plate from 1990s known as electrons backscatter diffraction or EBSD that is used extensively now to get diffraction information and as well as takes the studies in the material so therefore we shall discuss in our subsequent lectures the imaging in x electron microscope Whitefield and also the dark images isolate diffraction pattern and other contrast mechanisms we shall discuss about the EDS spectroscopic analysis.

In one of the lectures we say discussed about the EBSD in subsequent lectures also we shall discuss about something related to high logical microscopy.

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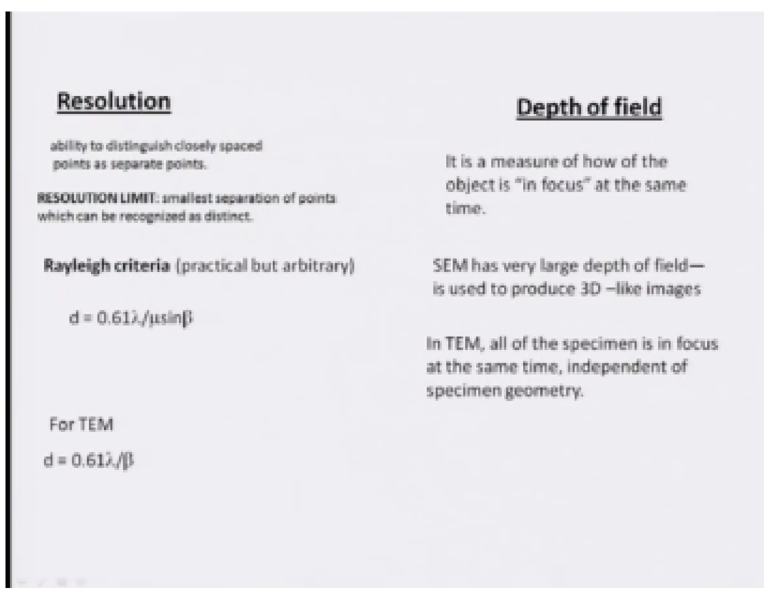

So, I will also like to tell you one more thing about the electron microscopes which is known as depth of field before we go into the details of the other features depth of field is basically measure of how object is in focus at the same time like if a very object which is very you know ups and downs are there are a lot of undue inner surface of the object whether can you bring the different portion of the sample or objects and focus or not as you know the optical microscope has a very low depth of field so therefore any sample.

Which is very rough cannot be bought in focus in optical micro so very easily on the head electron microscopes has a very large depth of field so that so they can be got in focus in electron microscope very easily that is why those of you already see in the electron microscope a TEM especially I have seen that many cases we record the images in a plate and the plates are kept at a distance much lower than the view skin so the depth of field is not high, so high we cannot even record this images are the same focusing conditions.

Because if the field is very high so therefore we can use both the TM and ACM to get good understanding of the surface features of the sample especially in ACM. So far we have discussed about the different types of signals which can be generated from the sample when electron beam interacts the sample let us now look at seriously each of this in time electron microscope in Tanis electron microscope electron beams accelerated by about 200 to 300 Claire volts. I allowed following the sample.

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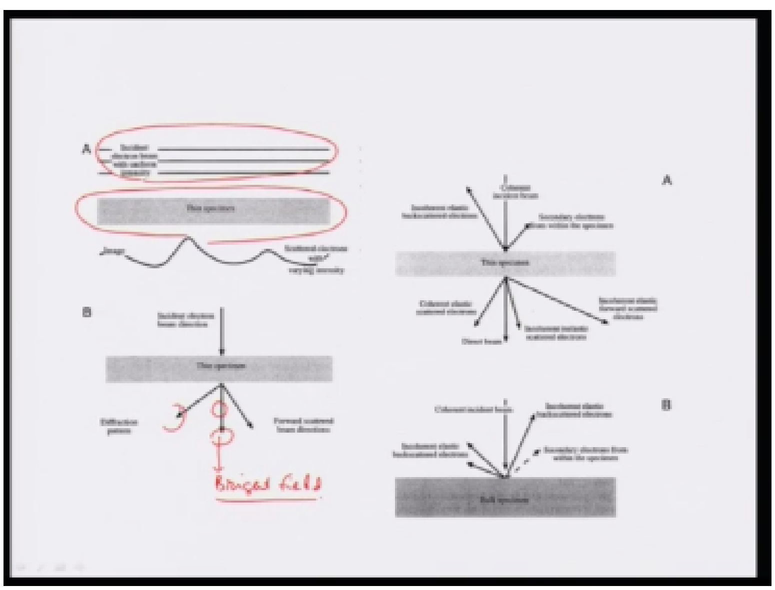

So therefore incident beam which is very high intense in false and thin sample and then interacts the sample as I said the during interaction it can either have it can either create a scatter electron beam which is elastic scatter or initially scatter or it can create it can generate the beam which is not undergone any kind of scattering so in a normal comes electron microscope we can use the forest scatter or the transmitted electron beam for rightful image this is done by putting a small aperture here so once you put an aperture apart nothing but a thin plate with a hole .

So we can block all the other radiations except a stagnant electron beam so therefore image form by this can be easily called as bright field image because this image will have bright trust or the image will be brighter contrast that is why it is called a bright field image to give an idea how this is done.

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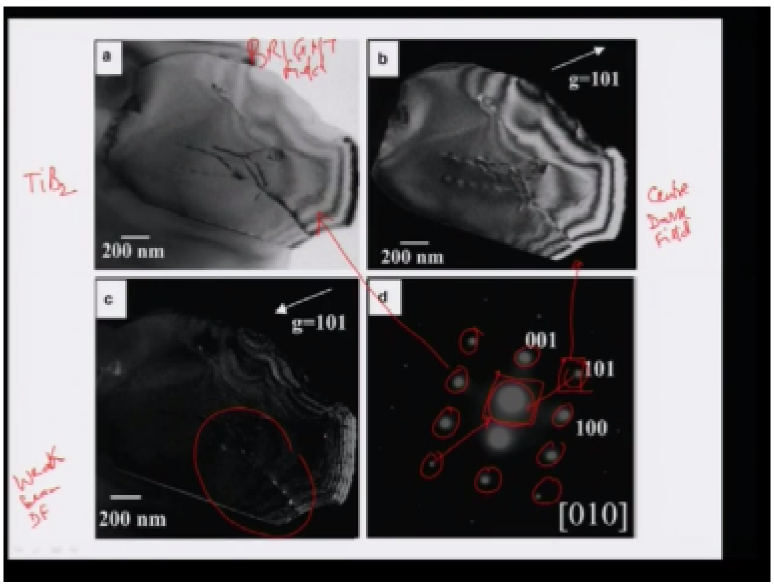

Let us see the hair this is taken form a sample which is Tasman TI b 2 or titanium diorite thinned by to the electron transparency so if I see the diffraction pattern obtained from this grain which is shown here the diffraction pattern will look like this with a thermos electron beam here and that means that this one is formed because of the forward scatter or the electron which is slightly passed through without undergoing any kind of diffraction on the other hand the electrons which have undergone scattering a diffraction can be seen like this are less equally scattered diffracts electrons.

Which has led to the diffracted spots now if I put my aperture only on the transmitted beam that is what I said you I generate this image at the top left which is known as bright field image another and if I consider one of these electron diffracted beams like this one here 101 and put my aperture around this and then I from image what, I get is known as dark full image or the central dark field image Center dark freely image this is obtained by bringing this weaker reflection on the opposite side which is same as this one to the center and put the aperture if we instead of bringing this week a reflection of center.

If you bring these one the strong reflection 101 to the center and we can form an image is known as the week beam dark field image as you can see in the bite fully mature a lot of dislocation inside a sample and these dislocations can be very nicely seen in a week beam dark field image or can be easily captured now agreed actual image the reason is this in a center dark field image the whole grain is illuminated strongly because it is diffracting softly, so therefore they are the defect structure cannot be seen so nicely on the hand in a week beam dark field image you can easily reduce the intensities.

Because you are bringing the strong parts in the central question dependence it will be reduced so therefore we can create a much better image so this is in a nutshell in a conventional microscope is done routinely that means you can go to a sample go to a region of the sample which is thin enough then oriented a particular crystallographic axis and get the diffraction pattern using the diffraction pattern you can either take a bite fill image or a sintered actual image or a weak beam image.

This is a very routine thing normally people do but in advance characterization goes like this we are going to learn how we can see even much better either you can see when atoms or column atoms in electron microscope or no in fact with the use of the advent of the new genesis electron microscope we can see very nicely the atoms a column some atom in a sample that's what I will tell you now okay.

Let me just get into that this again is basically to describe you how electron microscope can be judiciously used to obtain lot of information’s particularly the spectroscopic and the real space information as I said a diffraction pattern can be used to generate Whitefield Luck field week beam without eccentric filamentous the diffraction pattern is basically tells you the reciprocal space information as we have already learned from the normal the course on characterization techniques so the spots actually related to the reciprocal space.

So therefore to obtain they actually the atomic structure how the atoms looks like in a sample one need to New gate real space information this is where the atoms are sitting in a sample that is what is called the Elspeth information that can be obtained by something known as the phase contrast so that is what the contrast which I discussed here is basically coming from diffraction and they are known as diffraction contrast so they can be changed depending on the diffraction conditions on the other hand one can use something known as phase contrast.

Which you will discuss in detail in the next class phase-contrast what is that let us now look at first this sample this is xing cobalt gallium oxide the nothing but a kobold oak jink gallium oxide zinc cobalt and gallium to jink oxide and if you look at the diffraction pattern which is shown here it shows a particular symmetry and zinc oxide as you know is hexagonal crystal structure therefore this diffraction button is taken along 00 to one of the zinc cobalt gallium oxide and the crystals are the by taking the white fill image by selecting aperture from the central be mother are the horrid cactus beam.

We can see this the crystals of these zinc cobalt gallium oxide are of the size of about approximately 82 or 60, 200 nanometers and they are elongated so morphology shapes and everything can be seen some of the crystals are blacks on these tools are a little less black so the sum of the dark Cantus also light contrast. So this dark on does like wanted mainly because of the diffraction the one switches undergoing strong diffraction will show the dark contrast the ones which are not under strong diffraction they will be showing the light contrast now once I if I if I want to take if I want to take.

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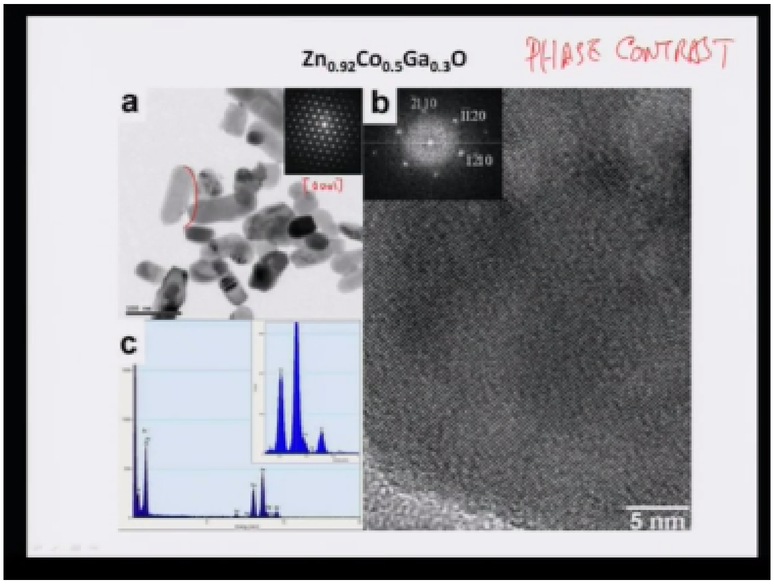

Basically the highest contrast image so what you need to do is that you can select a set of spots okay like first six and it along with the transmitted beam you can select that spots and put a big aperture like that and then you can form an image which is shown on the right hand side so as you know that the transmitted one or the beam which has passed through the sample has not been undergone any kind of diffraction it is considered so that is why it contains a certain phase of the electrons other than the other hand the diffracted beams have been undergone diffraction.

So therefore they contain a phase information faith means the electron phase so if i take select all the beams and allow to interfere them in a normal times retina microscope . I get something known as interference pattern an interference pattern will me the columns of the atoms which is shown here very clearly you can see the different columns of the atom sitting on the surface not only that in fact one can actually take this real space atomic element image and do Fourier transformation.

In a shop using as particle different kind of softer switch available in the market and then one can easily get back this diffraction pattern which we have been obtained in an normal electron microscopes so therefore this can be used to prove that whatever image is formed using a phase-contrast mechanism is basically reflects the real space information's on the left bottom corner of this slide we have shown the ED acts or energy disperses their spectroscopic information from this one can see the peaks correspond to cobalt , sink ,gallium.

And never to this one corresponds to oxygen so by knowing this you can see here also oxygen zinc gallium cobalt pics very easily so by knowing all this information a normal white field the spectroscopic information as well as these the phase contrast image you can get lots of information from the terms electron microscopes.

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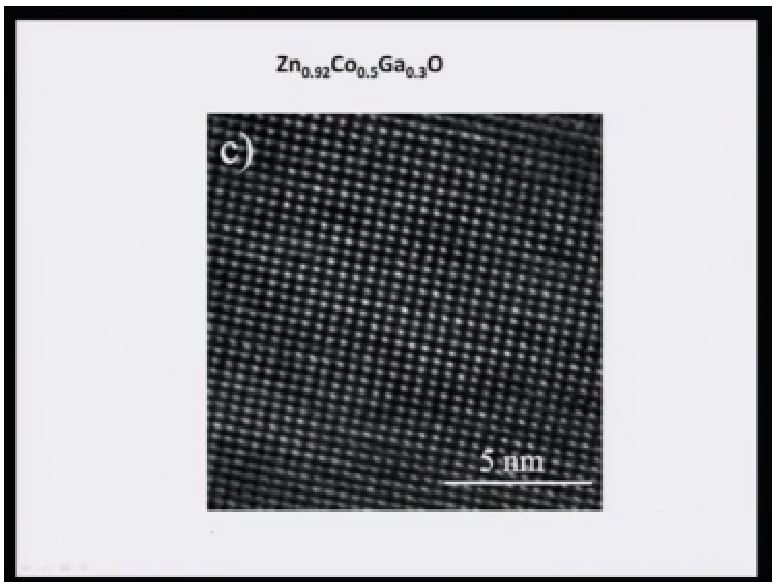

This is zoom view one can really see of this Lucien Michael image one can really see each column of atoms very specifically in a high Lucien image this can be done routinely now in a many microscope because microscopes capabilities has been increased as I said many microscopes have been produced obtained in the market now with a resolution of less than one am strong so that is why this are no longer a big problem nowadays in fact even a routine user can get this kind of images.

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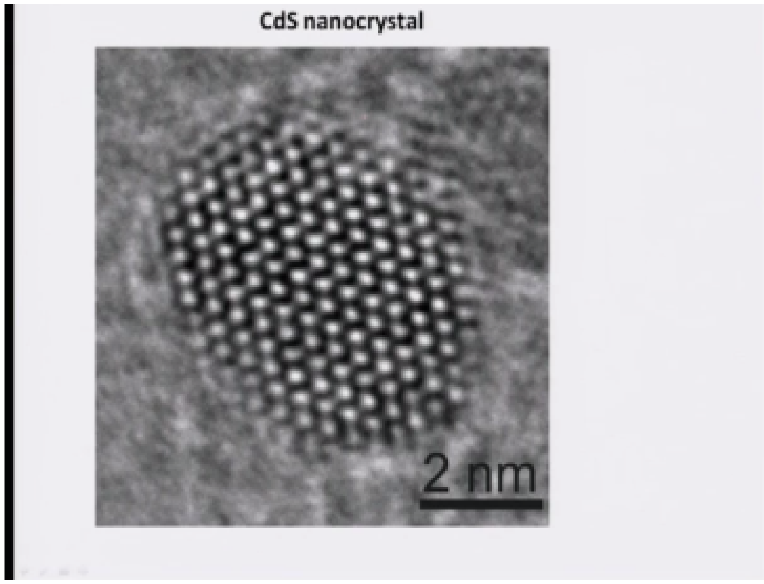

This another one whereas cadmium sulphide nano crystal has been imaged one can even see here the hexagonal as meant of the atom very nicely cadmium sulphide is basically having hexagonal crystal structure so this is very small crystal approximately four nanometers even you can see the atoms at the surface very clearly here at the edges of the sample is very nicely faceted crystal.

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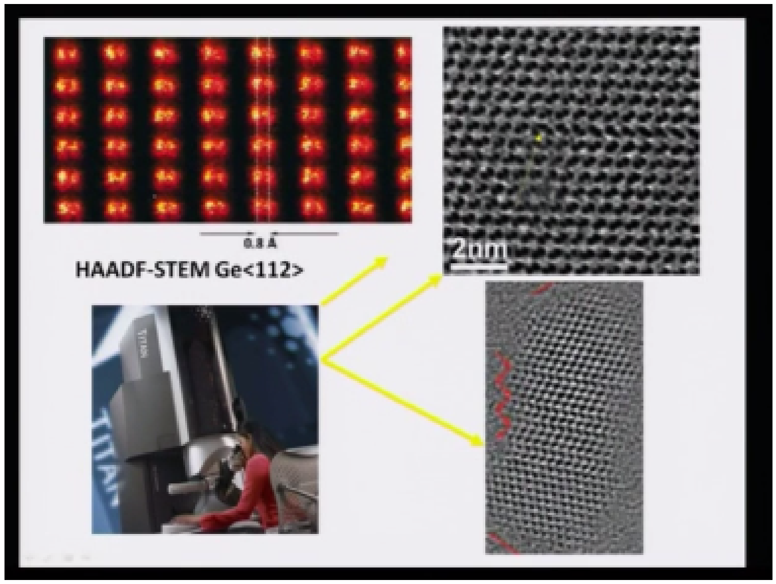

The advance has happened so much that in the microscopes called Titan which has the best possible resolution in the world can even tell you the type of item present on the sample if I go back here. I cannot see from this I cannot tell from this pair Lucien image whether this is a cadmium tama this is sulphide Adam it is very difficult to say the type of item cadmium of sulfur very difficult to do a normal extra microscopy but with the advent of the titan with new kinds of contrast mechanism one can basically tell what kind of atoms are present on.

A surface by something known as harden stem or high angle and low dark field image scanning transmission electron microscopy this image is taken from germanium crystal Lucia oriented along 112 directions you can even see clearly Germany and humble structures on the surface taken from this title microscope this is again taken from its zinc sulphide crystals we are getting us up at crystals where you can see the twin structure in a summary. I can say that I have discussed today you the interaction of the electron beams with the material.

And how this interaction can be easily used to generate different kind of images either diffraction contest images or a phase contrast images in Adams electron microscopes in addition at the beginning. I have discussed you how the text electron microscope has come up from nineteen twenty five to 2005 in this about 80,90 years of time period and also I discussed the resolution the depth of field along with different mechanisms of the image formation at times electron microscope.

In the next class first thing, I will go is I will take you to a times electron microscope and show you the real terms electron microscopes and demonstrating you how this different techniques can be used for analyzing the samples in a microscope thank you.

Acknowledgement
Ministry of Human Resources & Development

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