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NP-TEL
National Programme
On
Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-08

by…
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So we are going to discuss our EBSD today so look I discuss first the AVC principles and then I will tell you how the integration of the EBSD with computer has changed.

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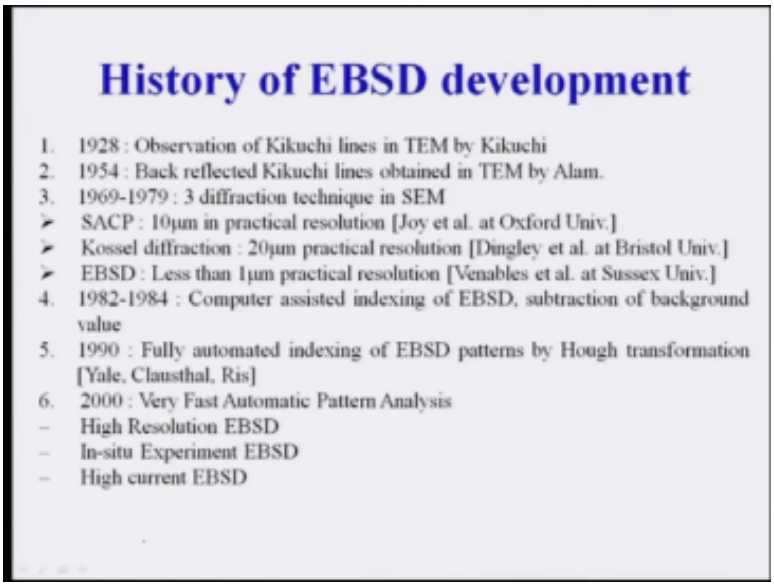

The analysis of the EBSD patterns and also related aspects during the study of the deformation behavior missed extra study of materials and finally I will show you how they EBSD is actually done in the real microscope EBSD has a long history as we look into the papers about 90 years back in 1928 kikuchi from Japan first time observed kikuchi patterns acuity lines and although it

has been reported that he did not observe he actually he postulated that there will be possibility of formation of kikuchi lines.

In the electron diffraction patterns and in fact kikuchi when turn later on when TEM was discovered and it was observed this lines to exist and they are basically due to the inelastic scattering of electrons, then after about another 26 years in 1954 black reflected kikuchi lines were observed in TEM by Alum then in 1969 to 1979 saw a huge change in the in the SEM techniques of EBSD three different types of diffraction patterns were detected one is the SACP that is selector area channeling pattern the one which I have discussed in the last class.

Where in fact 10 a micrometer resolution was obtained by Joy et al. at Oxford University and at Bristol university Dingley et. al or Dingley and others actually I found out the coastal diffraction patterns with a practical image resolution of 20 micron finally EBSD came with one micron practical solution by venables et. al at Sussex University again from England.

1980 to 1984 saw extensive uses of computer in indexing EBSD patterns and then from 1990 onwards we have fully automated EBSD systems with Hough transformation possible again major contribution came from Minnesota PL University of tousle and raise and from 200 onwards that is in the 21st century we have four very fast automatic pattern analysis systems. Obviously I have not listed the improvement in camera resolution and camera activities in this eve EBSD.

So now it is possible to have high resolution EBSD in fact people are doing in situ use the experiments which we will discuss and it is even possible to have high current EBSD, so I will not will discuss all of them I will discuss some of these dailies lecture.

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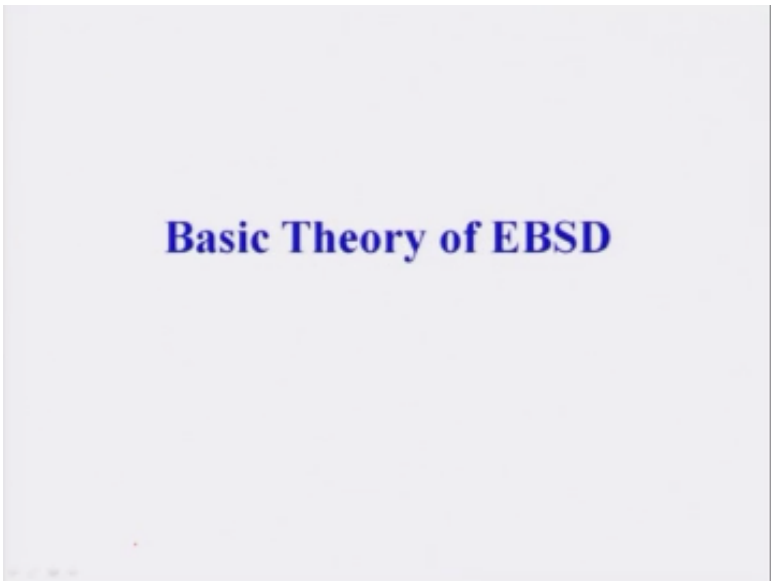

Let us first look at the basic theory of EBSD.

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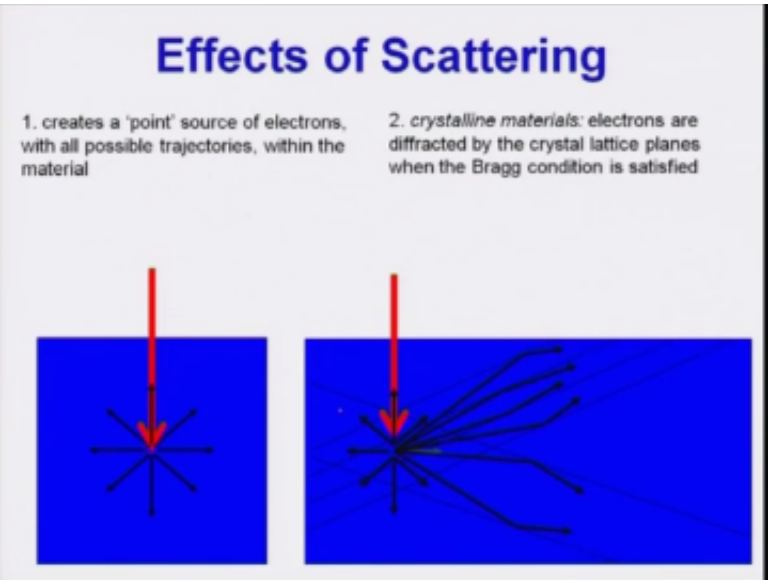

As we know the electrons from a source like tungsten filament or lab six filaments R FEG in ACM they accelerated at a very high voltage 20 30 40 maybe 60 kilo volts depends on the microscope, and allow falling on the sample surface. So therefore because of this interaction of the sample the electrons will undergo scattering one of the effect of this interaction is scattering and the scattering actually can create a point source of electron with all possible directories within the material.

Let me show you example suppose this is the material another electron and once the electron falls on the sample you can see it is going to create a point source here and from where the electrons surely it can move in different directions in the crystalline materials you have the atomic planes and these crystal planes can actually cause diffraction provided brag slow satisfied. So therefore increasingly material if you have number of crystal planes sitting on is on the inside akin the material and the electron falls and they can also actually get scatter.

But in some cases if the crystal planes are actually oriented properly with respect the electron beam so that bags lock and you satisfied we can have distractions, and as I said in the last class these if we change the beam orientation we can have the channeling of electron backs or electrons at the same time the scattering of Paxil electrons inside the crystals and this can give rise to black and white lines and those are actually basically keep you chill ions.

So this is basically the reason for origin of the EBSD the scattering are other Braggs law dominated determined clattering of the electrons in the material.

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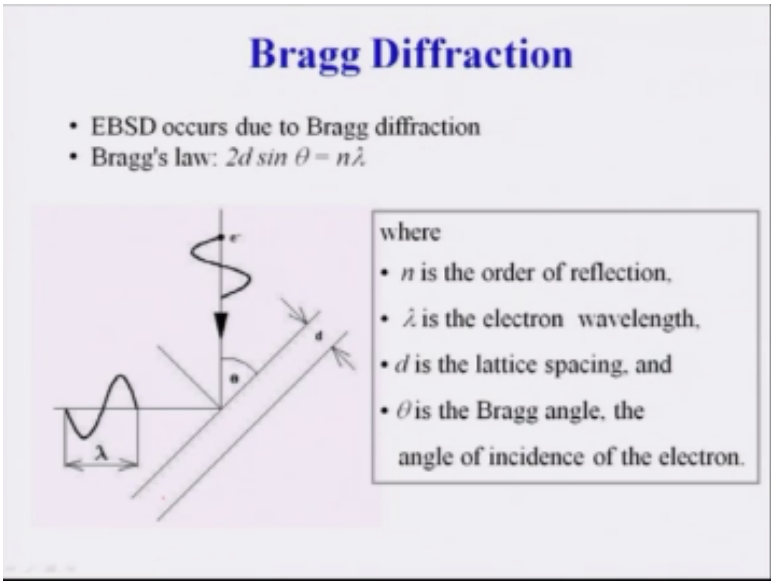

So therefore as you know all of you know because you have done the plenary course on they did the characterization the Braggs law EBSD occurs due to basically Braggs diffraction and we know Braggs Law is given by to design that equal to n γ everyone in this course will have idea about this particular equation which Bragg's Henry Bragg and William Bragg they discover long back.

And as you know that if the electron beams with certain length false or γ falls on a atomic plane of facing d it can get scattered by the Braggs law with an angle θ satisfying to design θ equal to n γ where n is basically order of reflection normally it is one in case of accelerant in case of electron diffraction can be more than one. So therefore whenever the electron beams are getting scattered by the lattice planes if they satisfy the Braggs law then there will be diffraction and then we will be able to detail the diffraction by using camera we will have the VSD patterns that see basically things.

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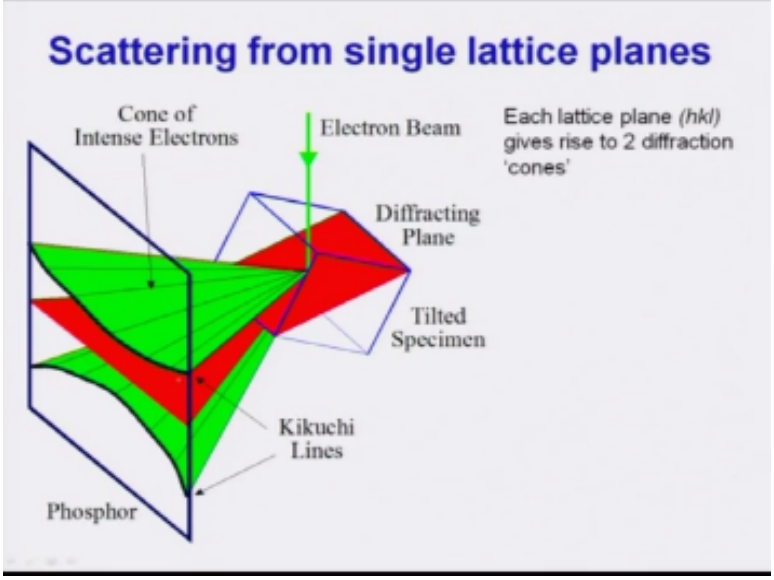

So if we want to go in detail of that scattering from a single lattice plane, so if you look at it that we know that each lattice plane what is can be indexed by hkl gives rise to 2 index of miller indices gives rise to two diffraction cones let us see that how it is operates, so suppose you have a crystal like this which basically cubic is shown like that and suppose this is the 110 plane in a cube.

And then if I have electron means falling on this plane and then they are getting refracted so this diffracted we will create a cone of intense electrons these are all can be easily borrowed from the exit diffraction anyone who study x-ray diffraction we know that even in x-ray diffraction camera also we have cones of reflections coming out because of the diffraction from a single crystals and then if we have a possible screen we can detect these lines with this lines actually called kikuchi lines.

So this is basically from a single lattice planes, now you have a multiple crystal billion in a crystal in a sample so therefore there will be different kinds of patterns are different would not a certain kikuchi bands will come different from the different planes of the crystals and then we can get actually a complex series department. So SEM electron wave lengths basically they are much larger than the TM the opening cone angles are found to be close to 180^0 this cone angle which is coming many times has been found to be 180^0 very large because wavelength is even larger here, so to design θ n but n γ is large then θ will be large because sine θ is proportional to γ.

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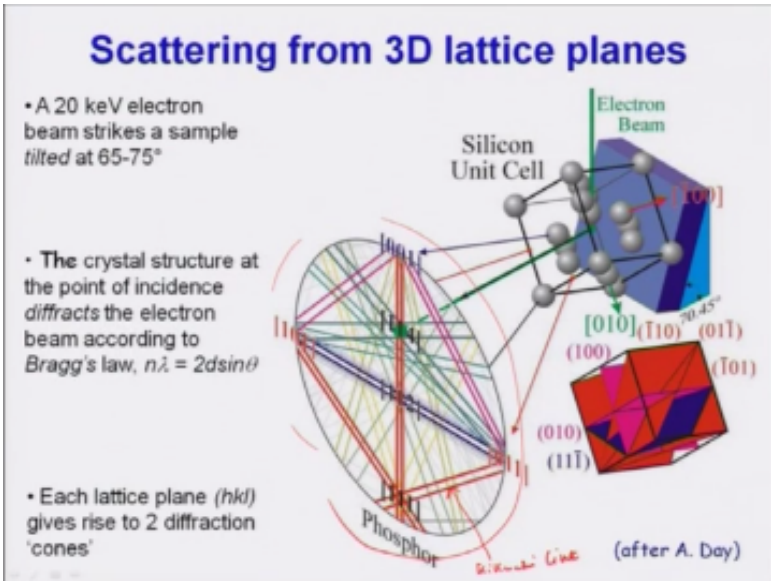

Now obviously one can look at the scattering from the 3d lattice planes and suppose you have a one who have just basically 20 kilo volts electron beam striking a sample which stilted to 65 to 75 ^{0} with respect to the beam and then we can think of like this suppose this is silicon unit cell again we have a basically diamond cubic structure electronic falling on this it can get scattered different planes will scatter and form these kikuchi lines which is shown in the picture here.

And then on the phosphor screen the crystal structure at the point of incidence of diffract the electron beam according to Braggs Law and each lattice planes basically you can look at it each lattice plane one on 0 or 1 0 type they are giving rise to two sets of basically cones one set is this other set is this. So whenever these two sets hit the phosphor screen they leads to two lines and this reliance actually call the kikuchi lines. So we can we are basically terming this as a kikuchi lines basically we are naming them by the scientists called kikuchi.

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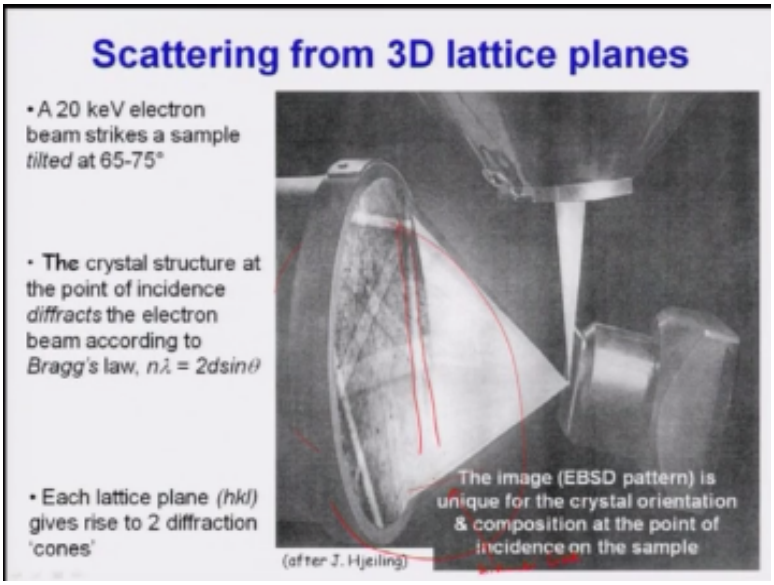

So in actual sense this has taken form J hatchling actual sense we can actually see this kikuchi lines to in experimentations if we have a camera put inside the SEM, so if you see this is a sample which is tilted with respect to the beam avatar out or this way the normal plane with at about 65 to 75^0 Celsius and then the electron name is falling like this is mythical schematic obviously one cannot email the electron beam by using normal camera electron is invisible, so and then it generates the scone the scone actually has two cones actually.

So they fall on this phosphor screen and screen produces two lines on the other screen so this is the origin of the conclusive patterns or diffraction patterns.

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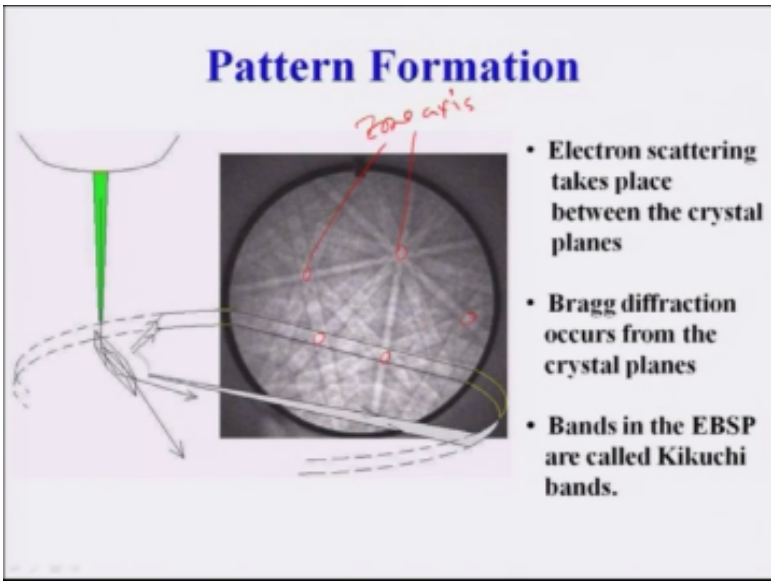

And in a two-dimensional plane as I have shown here this is a crystal this basically creates this lines and bands and these bands as you will see the exists or meeting points this meeting points are called zone an axis in the literature because they are like different roads coming from different places and meeting at some points we know that with it there are junctions, so these are actually called zone axis that's one here they are there for their many more one they are actually so one can actually index the zone axis using copper in a scheme again using Braggs Law.

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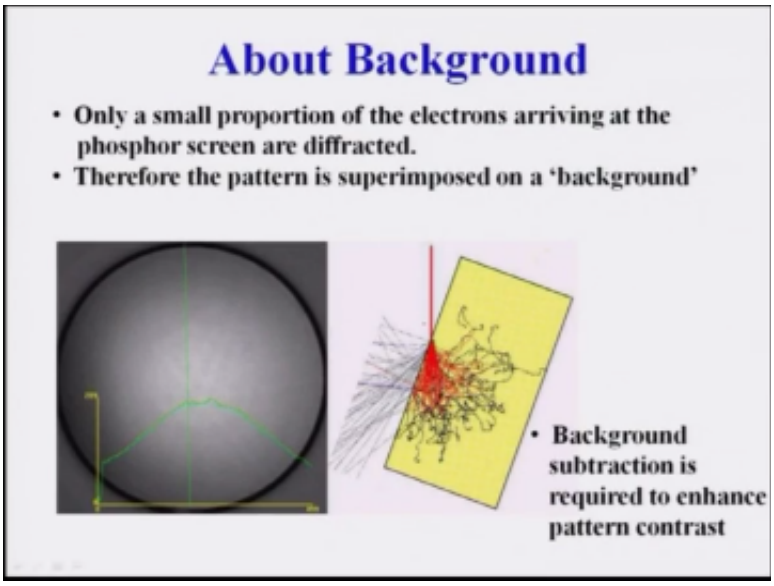

Well that is very what is called one, so that we can get a very nice affection done by the same time one must understand that all the background electrons which are falling on the sample they may not produce the scattering which will be detected by the scheme, so therefore there will be as only a small proportion of the electrons arriving at the phosphor schemes are diffracted. So bulk of these electron moves are not distracted they will undergo multiple diffraction and then getting absorb into the material I will be coming out but then they are not contribute to this kikuchi bands.

So therefore a pattern is always superimposed with the back on a background it is just like excited fracture pattern in x-ray diffraction button you have a background in density come from beam staling and are the peaks is occurring from the scattering do two backs okay. So therefore this background needs to be subtracted otherwise we do not get a very nice pattern these are all actually done in a computer nowadays.

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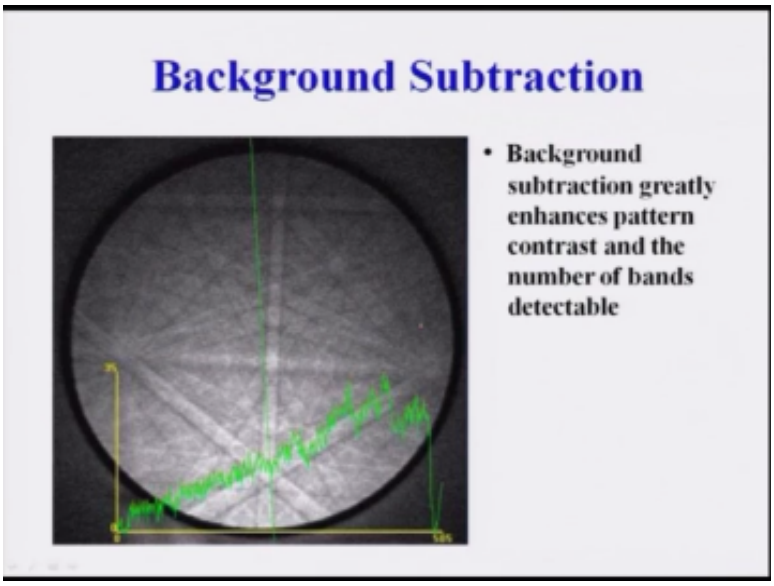

So one can look at in fact the backgrounds diffraction can be sub-second can be done same way like any radiations and this is done here you can see that background, so for like this with respect to this is very small intensity although, but still it has to be subtracted then to get octane a very nice EBSD pattern.

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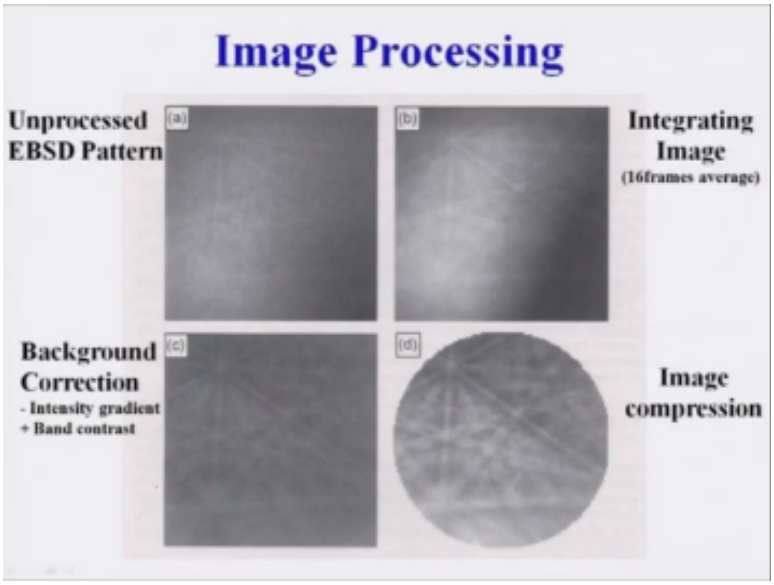

Not only that as I said in the beginning lecturer with the advent of computer only fast computers this technique has taken a big lead in the material science research activities so images which are obtained or patterns which are obtained from the HD camera they need to be processed and as you see the unprocessed EBSD pattern is in fact does not contain my information, so normally we take in several a VST patterns on a particular case, so if we select a point on the material and take several image pattern from that and then we just integrate them once you integrate them signal-to-noise ratio improves and then we correct the background intensity.

So that the brand contrast increases as many cases image accomplished to get even better clarity of the pants these are all routine down in the computer one is to it need not bother about it while doing the experimentations but you must know that these are required to obtain a good quality vs department.

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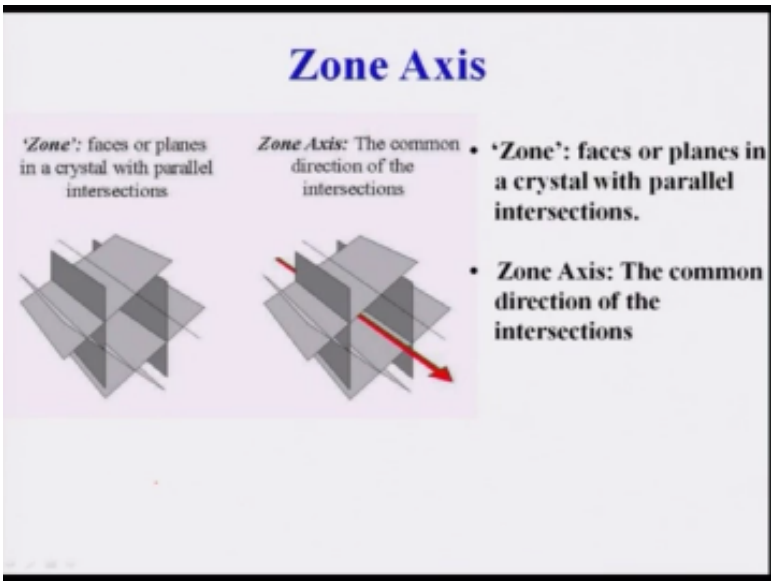

Now as I said zones or zone axis just server several two or three slides before zone is nothing but a phase our planes in a crystal with parallel intersections that is what is shown here you can see that there are phases which are actually we have a parallel intersections and zone axis is a common direction of the intersection obviously. So if I have seven planes there will be one direction which will can will be contained in all the planes that actually called zone axis.

So in wherever the EBSD patterns are a waist bands a cube and seller will meet in the EBSD pattern they actually can be defined as a zone axis and can be indexed using purpose scheme.

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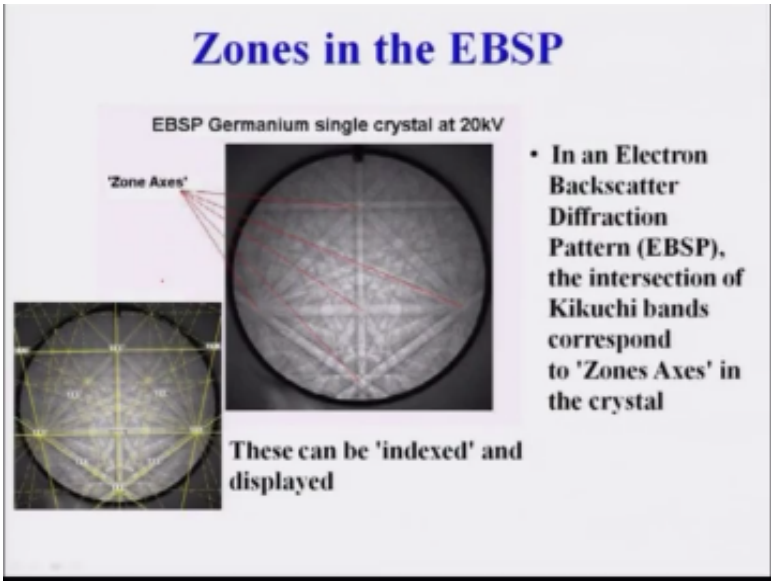

Just to show you that as I said these are actually join axis which is actually taken from germanium seen Krista like 20k level acceleration and you can see that these are the actually zone axis marked here by red and they can be indexed which I am going to discuss chapter some time. So these zone axis are actually carry information about the orientation of the crystals in the geometrical plane iron material, so our way into something grains.

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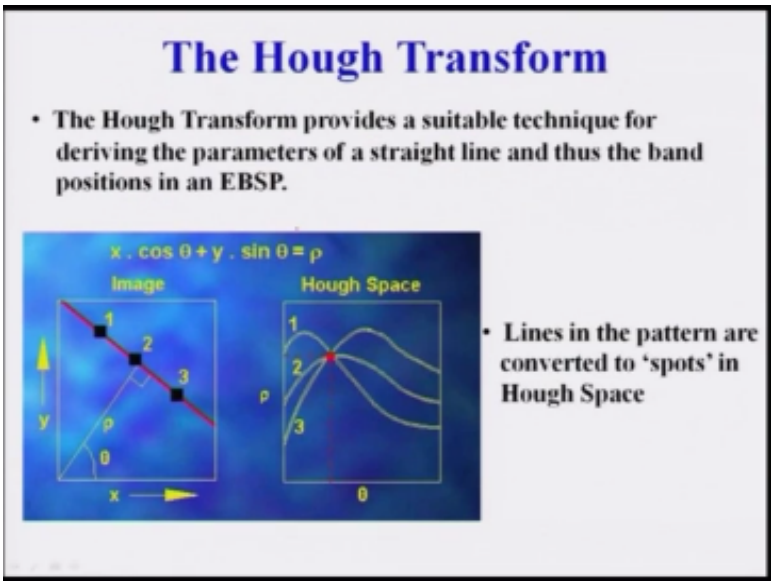

Well that is actually done using Hough transformations Hough transformation provides a suitable technique actually for deriving the parameters of a straight line and that is actually bands positions in the EBSD patterns let us see how it is done, basically you misty pattern is nothing but XY plot as we look at it well two-dimensional XY plot, so what you do is that it convert this XY into ρ θ plot okay.

So if suppose we have three zone axis one two three are three bands other on the EBSD pattern then the X is given by this one the horizontal variable and vertical one is basically y axis and then you can see that ρ is connected to x and y and θ which is given by the top of the that this picture x cos θ + y sine θ = ρ so we can convert this information from X Y space to the huge half space that is Rho θ space and we can get these bands because they are called huge bands Hough fans actually.

So lines in the patterns I can be converted into spots in the half surface and this is again done routinely by computer we do not need to do anything in the real material else's problem we go to the EBSD set up so the computational power is so much that these are done rooting in fact all online purchasing is possible also, so basically real information which is gathered can be at the same time processed in the same computer by transforming this information from these xy space to the half space.

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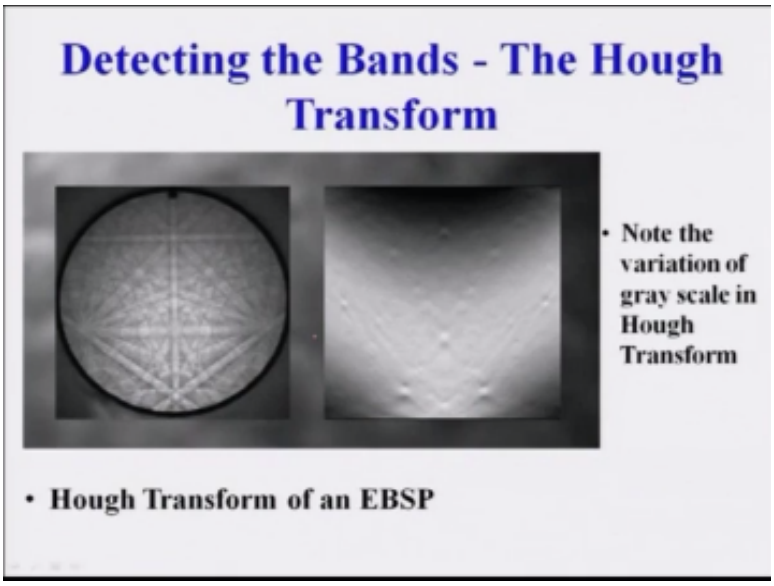

And then once you do this we have you have to detect the bands okay and these bands are detected by using indexing scheme oh that is what is called evasive pattern recognitions.

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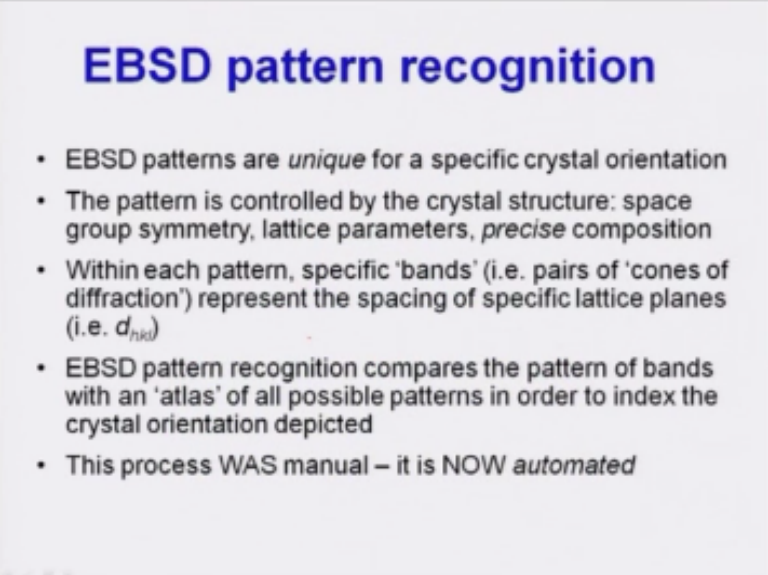

So EBSD patterns can be recognized or indexed using several schemes, so as you know obviously pattern obviously unique to our pacific crystal orientations even if I know the crystal structure of the material apparel it is basically depends on the how the crystal is oriented in space or the grain is related in the space. So this is a very unique thing about this, so dependent orientation just like in a TM diffraction pattern diffraction events actually depends on the original crystal in the plane or in the space same thing actually is true for here.

But resolution is poorer here at the same time you can actually have much largest as what is called area can be analyzed in it a CMS couple TM because thin area is very small TM sample but in SEM you have a large area which can be analyzed, pattern is controlled by the crystal structure obviously it is actual main space good symmetry lattice parameter and precise compositions if it is an alloy one is on the precise composition within each pattern the specific bands are this pair of cones of diffraction represent the spacing of the specific lattice planes as you have already told you when I discussing will be the backs law then evasive pattern recognition compare these patterns of the band with a atlas which are prepared for the so many years of experimentations to index the crystal orientation required.

So therefore this is again just like a diffraction pattern analysis, we have the exit diffraction taking from a sample and then he compared with the ad last which is available in the literature if there is a new crystal and we cannot index. So you have to go by again by the standard process our first doing the kiss effects of the material lattice parameter and other things to index it, and

then this information will go to the atlas just like the IC duty database in excel diffraction this also a database.

So that can be used to what is called index EBSD department this all databases all given to each and every software which are used in a EBSD analysis, so allow me the software that is are available this is just like a was manual it is fully automated now as I just now discussed.

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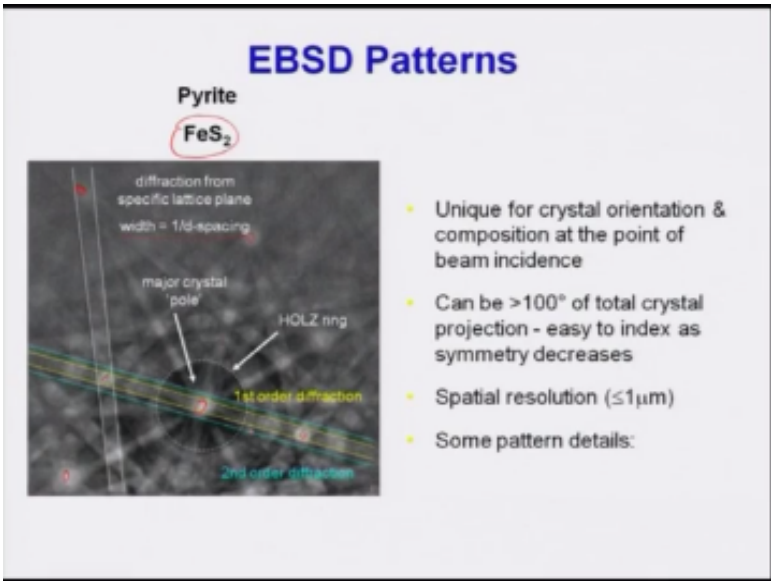

And can be used to index, so EBSD patterns let us see that this is taken from pirates pyrites is nothing but FeS_2 what can take metal also but I am show you as you see there is zone axis here they are in fact there one there one so maybe this zone axis is there this is zone axis there many okay, so basically this is the metal zone axis where the many lines are or menu in bands are meeting.

Now unique for the crystal orientation obviously and the composition for the pirates can be more than 100% and 100^0 for total crystal positions that is you can basically obtained for large angle of diffraction, and Priscilla's Lucien is nowadays can be often opt less than 1 micron possible and some pattern details can also be obtained let me get us, so you that a diffraction patterns on specific planes so that was this one is basically band, so we throw the band is basically one by D spacing of the of the crystal of the particular plane of the crystal.

This is the first word diffraction pattern okay as you see here this lines and this is basically second or diffraction the blue lines which is shown on the screen they actually second row diffractions the yellow ones actually first or diffractions just like in a tea and diffraction pattern here first or a second order third order a faster and higher order diffraction patterns.

So and this is a major poll axis as I just told because there is so many lines and meeting and their points the bands are meeting at this point and this is what is called Hough high order Louise Jones reams they are basically same like in seabed or convert them electron diffraction patterns in common emulate diffraction pattern you can get zero order lives on then fast or a second order of zero order and higher-order lines so this is a zero order and this is a first order.

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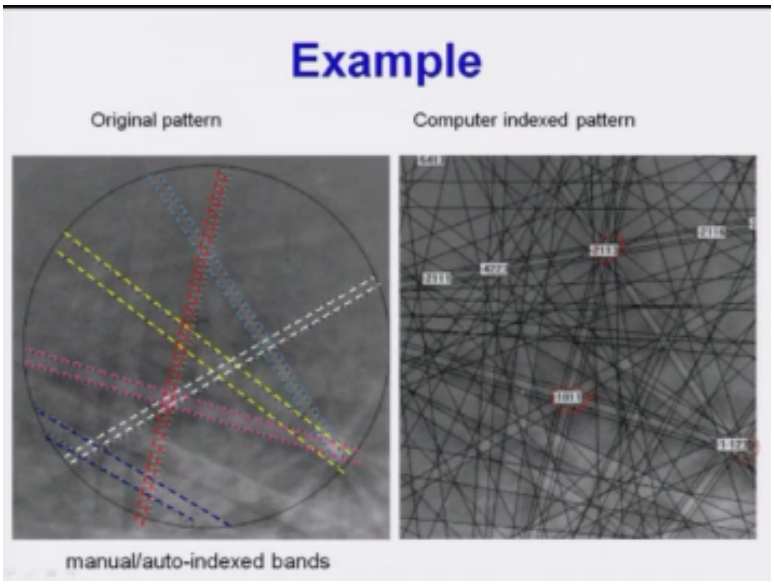

So that is basically way to read EBSD is the pattern, now to give you some other example so this is the original pattern of some crystal and now it can be either manually index or auto index, so auto indexing is done like this the way I shown you first the bands are marked by different lines and then they are indexed this is again computer done by computers you see this is a hexagonal crystal okay this is in fact bismuth so the different lines can be easily marked.

So in every cases you see one inside there are two lines outside there are two lines this is the first order this is the second order this fraction lines or cutie pants and this is a major john axis here this is another method zone axis there are some- on ax is also possible this element 106 to Nexus

here. So this can be easily compare soy gin pattern will be matching with this then we are sure that we have done indexing properly.

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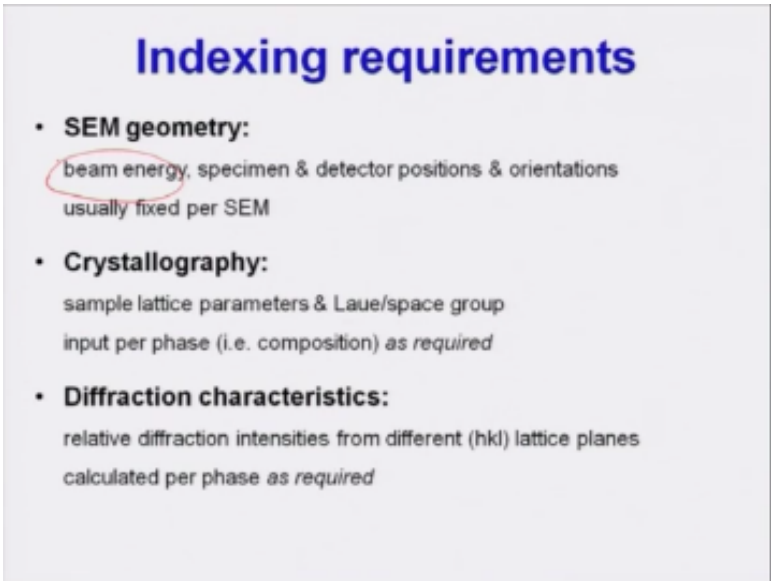

So to index we require many things not only the crystallographic information's you need the SEM geometry that is beam energy specimen detector position and also orientation of the detector, how the it is where introspect to the crystal usually which is these are all fixed by SEM beam energy some time can be changed, but specimen is also fixed with it by the user and detector position editor which is not fixed the only thing which you change which you can change actually bean energy.

And this will change the wavelength of the diffraction beam, crystallography as I said just now stood up is very important because you need to know the sample crystallographic ally sample that is the lattice parameter, space group all these things are required to be known and they are actually input or any kind of crystal and this fashion care text is to be also known that is relative to inspect intensities must be known this all obtained all given in the EBSD or SEM databases of the any crystals. S but I will show you some example how it is to be done and it can be calculated as I said.

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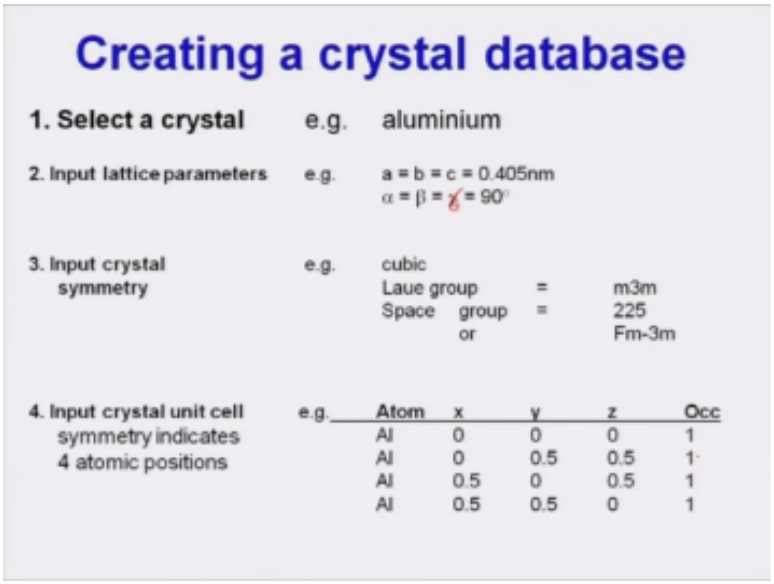

So let us see how we can create that we can create these directors so first let us suppose you have a sick crystal like aluminum which is a common metal you know is a safe see crystal structure and with the lattice parameters like ABC's 10.405 nanometers and α β λ this is λ is basically 90 ^{0} and the symmetry also to be can be obtained as a cubic the law group is same tree m space group is 225 or f 3 /m press enter m3 m, and unit cell symmetry in the sales there are for autumn equations we know that.

So let us see the adding positions there aluminum sheet set 00 that is occupied occupancy 1 and alumina again seats at the phase center point 5.51 also 0.550 sorry that is occupation c1 and you can have 0 . 505 of Kevin C 1 and point 5.50 who can see one, so you know everything about the crystal by knowing all this stuff's the lattice parameter symmetry and the atomic positions that is all we need to calculate x-ray diffraction pattern also.

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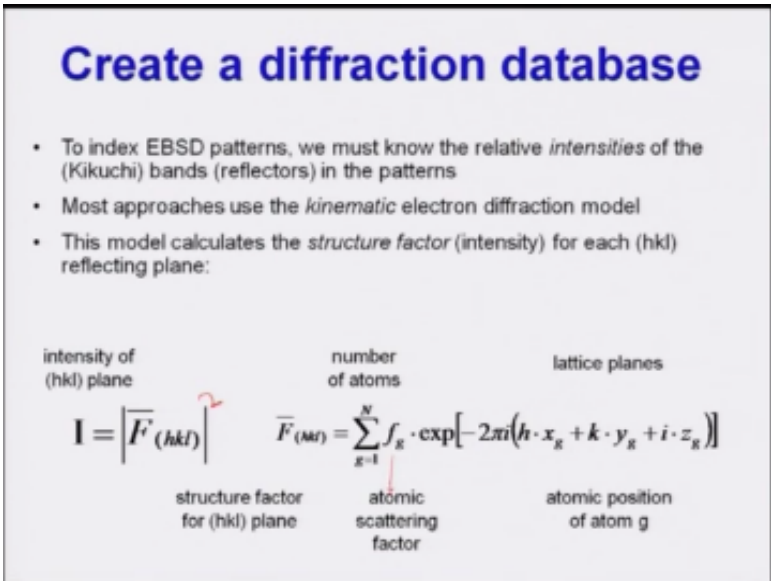

So how to index the EBSD patterning master relative intensities as I said that is of the bands or keeps the bands or reflections in the patterns just like x-ray diffraction pattern need to know the dative intensities of the diffraction peaks same thing down here. So and most approachable use is the kinematic diffraction theory which is very complex let me tell you in fact in TM probably it has been discussed to you but many cases it may not be discussed you in that case there is no choice other than going back to the books.

And learn it kind of a diffraction theory is what is called nautili discussing the TM books and this model actually calculate the structure factor for each of these HKL planes how it is done when the intensity is basically spare the structure factor related to structure factor okay it is basically square of that and hfh scale is given by σ jiggle g goes to 12 n fgfg is basically atoms cutting factor in exponential minus twice πi h. XZ k + K .YG + I JD where hkl is the plane indices and XYZ the lattice the atomic positions this is the n is a number of atoms as you know and FG is basically scattering factor for each a pattern which can be obtained from the any database.

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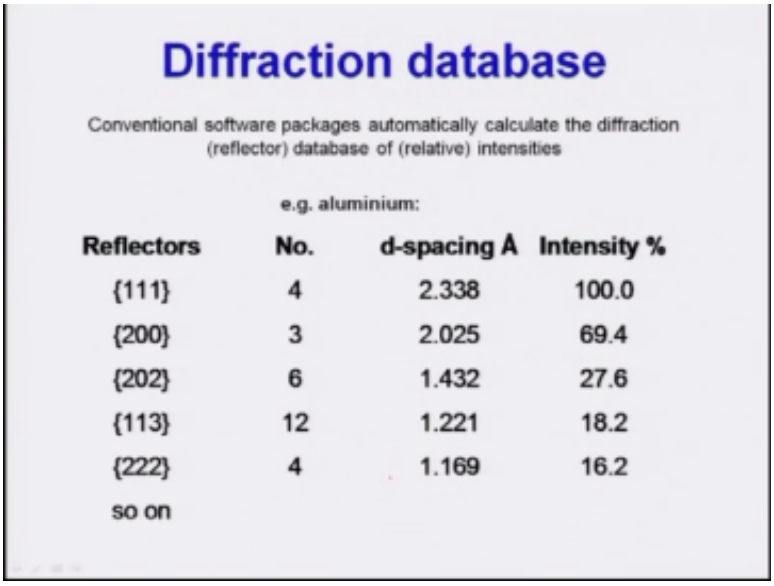

And lattice planes atomic positions already told you so how do I create it aluminum so let us do that we know that for whether a 1 1 1 therefore 1 1 1 planes, so this spacing is 2.338 with intensity coming to be 100% because 111 is the most dense atomically dense plane, so therefore it will have the largest intensity coming defecting coming from this plane followed by 200 again it has 3 such explains the sixty 69.43% density and so on. One can not actually go on calculating this for many large nor do planes and EVC we need to do that.

These are routinely done obviously for aluminum is very easy, for other things our crystals complex crystal is not an easy one needs to be used computers to do that. So manually doing is very difficult but aluminum manually can be all calculated.

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Well that is actually way this databases are created and then these are used to index the vsd the bands in the image departments and then obtain index those patterns to obtain the crystallographic orientations. So once you know the orientation of the each crystal what do I do with it that is what the basically important aspects one is to know is. Well as you know suppose in a crystal there are many grains I can draw these grains here like this in a material okay.

So if you have so many grains and I always knew that definition from the game tells us that each of these has separate orientations, so what you need to know is the movie HD is that once you put the electron beams on this crystal on this grain, we can obtain the condition of this crystal or the grain very easily by doing all these kinds of indexing these patterns from this grain. Similarly I go to the next crystal or next grain and do the same analysis and then this and then this and then these so on.

And once you do all this analysis we can store this information how the crystals are oriented with respect to the physical space X Y Z and then once you know that we can basically do this orientation contrast imaging. So we can plot this data on a micrograph to obtain the orientation information of the grains is grain, so that is actually call or indecent contrast imaging, so that means the contrast of this image will vary depending on the orientation of the each grain on the crystal.

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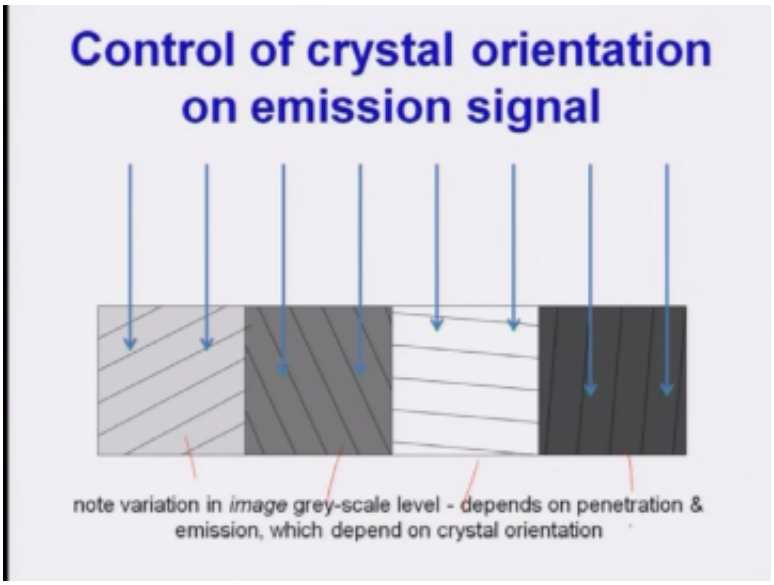

So in a poly crystal sample where suppose this is to talk about the emission signal or in how it is dependent on the crystal orientation, a poly crystal sample you have different grains one is this is another one another one this and the planes are oriented different differently and each of these grains. And now if we have electrons falling on each this suppose this particular grain and then we can obtain orientations just by doing this so we can basically give a grayscale image contrast on this okay based on whatever way we define a grayscale.

And then we can obtain from is we can make it darker depending the rendition of his crystal and this is again can be done this way this way, so that is how when next we obtained this grayscale level it depends on the penetration and the emission also emission means how much backscattered ready diffraction is coming out from this crystal and detected by the camera. So that's how actually this is this is very simple this is the simplest possible way of representing this in orientation images on the crystal on the inbound patterns are imaged from a large sample is done I am showing you.

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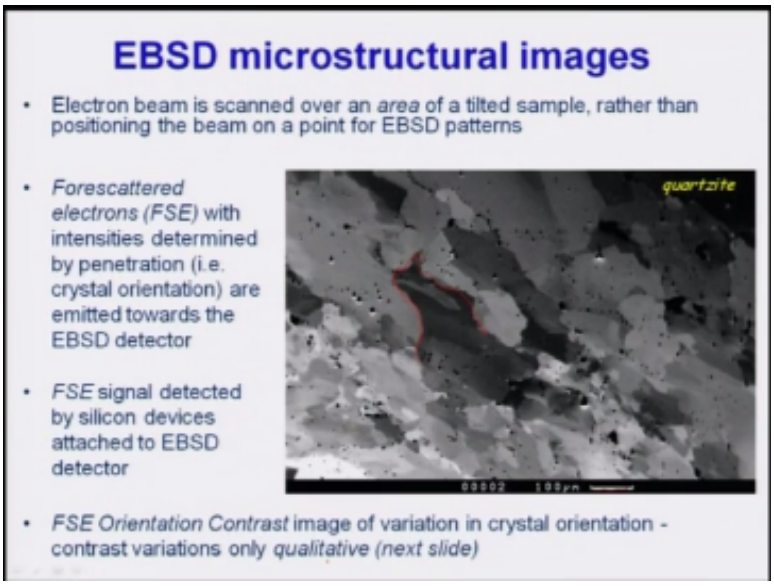

Let me show you some image electron beam is basically scan in a scanning electron microscope and sample is tilted, so rather than polishing the beam on a point on a EBSD because we are scanning the beam so he put the different position beam on the different person of sample and there is 4 electrons with intensity germinate penetrations and crystal orientation obviously and they are emitted towards the detector.

And these signals are detected by silicon devices or the camera which is basically Pentair cool camera and attached to the VST detector and this is how it is done. So once you have all this information we can plot it you can see this is a very large area this is only 100 micron, so that means this is approximately 10 times of that so 1000 micron is this length and this is approximately about 6,600 micron a very large area scan and we can see different grayscale contrast on the image.

So each of these actually showing the condition of the quasi grains on this and the sample, so this is you can see that this grain is darker, so that means this is oriented very prepare lee with respect to the and this gain is biter and there are lots of dots they actually that mean they are actually not contribute any information to the EBSD pattern. So that means the first grader electrons orientation contrast image of variation in crystal orientations the counter variation is only qualitatively.

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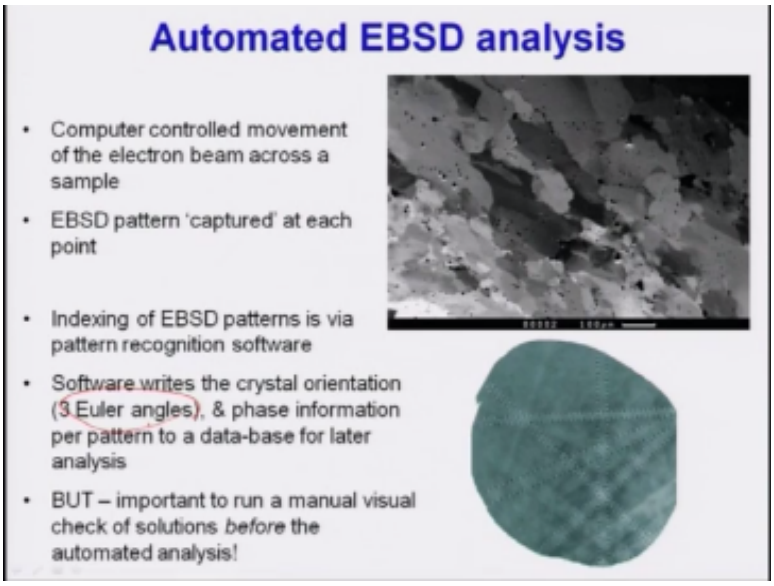

So you can do that so let me just tell you that how this is done in automated or computerized system computer, control movement of the electron beam across the sample can be done even see cat pattern can be captured from each point and that is what is shown. So you can basically go to each point and capture the EBSD pattern and this is the pattern from one indices pattern by using a pattern recognizing software and then software is I the crystal orientations in three overlay angles Phi 1 Phi 2 Phi 3.

And the phase information / pattern to a database for analysis so I am not going to discuss about how this Euler angles are determine this is again not possible in the short span of time but one is to know also. The important to run this manual visual check for solution before when we do this analysis because computer is a basically black box, whatever goes in is comes out. So if you put garbage in it will garbage will come out so one must actually run one must actually see visually these patterns and be sure about it what you are saying is what you are obtaining and then do this indexing in a computer.

This is again taken from one our sample this is the grain orientation image okay and you can see this is one pole figure okay and you can see there is predominant texture, that is why these things are looking black.

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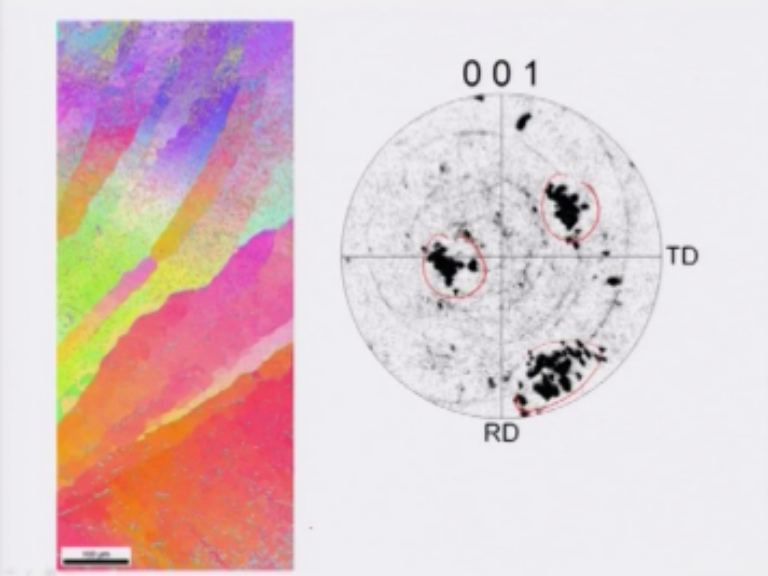

Those okay you have little bit knowledge about texturing you can understand and these grains can be shown to be oriented along different zone axis actually, this raid is actually close to 0 0 1 and blue is actually close to 111 and the magenta is close to 0 1 1. So therefore one can actually see the different guarantee shin and obtain this inverse bowl figure and then find out the wire this majority orientation coming into picture fancy largest cluster is coming here which is at angle almost like 90^0 from the pole.

And some of these are coming at along 111 okay this is close to 1 1 1, so if you know a little bit of this will understand that so we can obtain the majority crystal orientation from these images which is very nice a Nimbus poll figure of this will be this image. So you must have a little bit about how to generate these poll figures and how to study this poll figures.

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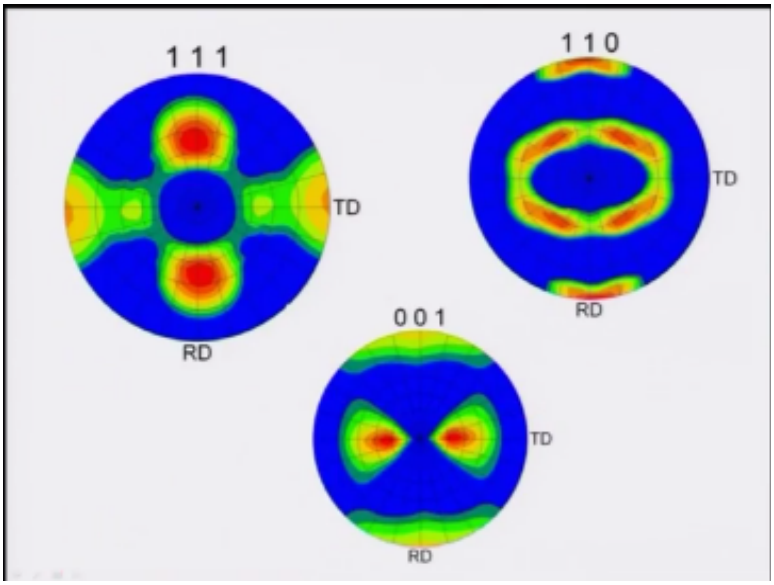

And then one can actually obviously obtained this kind of maps as you say this is 1 1 1 this is 1 1 0 0 0 1 and this is actually called rolling direction, transport direction this is a very standard in a texture I am not going to discuss deeply intake or texture in today is lecture. So that can be done so using EBSD routinely people nowadays study the structure of the material, texture means how in a multiple grain material crystals are oriented, if there is the orientations of the crystals are haploid then there is no texture is orientation with crystals are happening in a particular direction.

We dominantly then we call this a texture material, so that can be obtained and depending on people can actually go on doing research on different kinds of materials because nowadays we know that the wind crystals play so much important roles in dictating the material behavior that is why texture material is coming into picture.

So with this actually I close and I am going to show you some example in the next lecture of texture analysis how they can be used really to obtain information of the crystal orientations and also to obtain, the how the processing parameters can change these crystal orientations and this can be used for the day-to-day applications also very advanced applications, so we are going to do in the next class you.

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