**Electron Diffraction and Imaging**
**Prof. Sundararaman M**
**Department of Metallurgical and Materials Engineering**
**Indian Institute of Technology, Madras**

**Lecture - 29**
**ELES and EDs**

Welcome you all to this course on Electron Diffraction and Imaging. In the last class we talked about one of the new techniques in which has come in transmission electron microscopy. In recent times namely scanning transmission electron microscopy, and see contrast microscopy these are all the two techniques which we have talked. In today's class we will talk about analytical electron microscopy; there are many microscopes are there where in addition to getting information about the microstructure of the sampled we can get information about the composition at different regions of that sampled with very high accuracy.

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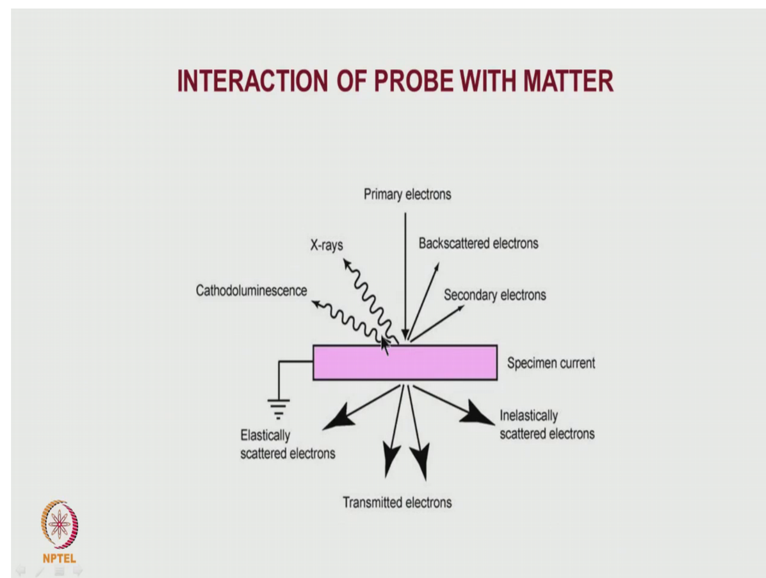


What all the advantages of it compared to that of other techniques where we can get composition like a s e m n that we will also discuss there, ok.

But mainly we will talk about a button I mentioned in the last class that when the incident probe is an electron and enters into a sample which is very thin, secondary electrons are produced in the material, backscattered electrons are produced x rays are produced these are all because of the interaction of the electron with that sampled. Then

the electrons are diffracted these are called transmitted electrons then elastically scattered electrons are there, this is what we use for c contrast microscopy in elastically scattered electrons which are the once which are used for energy loss spectroscopy.

What is essentially important which one should understand is that when we have a very thin file in which high energy electrons which are accelerated to maybe 100 k e v to maybe 300 k e v energy electrons are falling on that sample, and the thickness of the same is of the order of maybe less than 50 nanometer let us say that.

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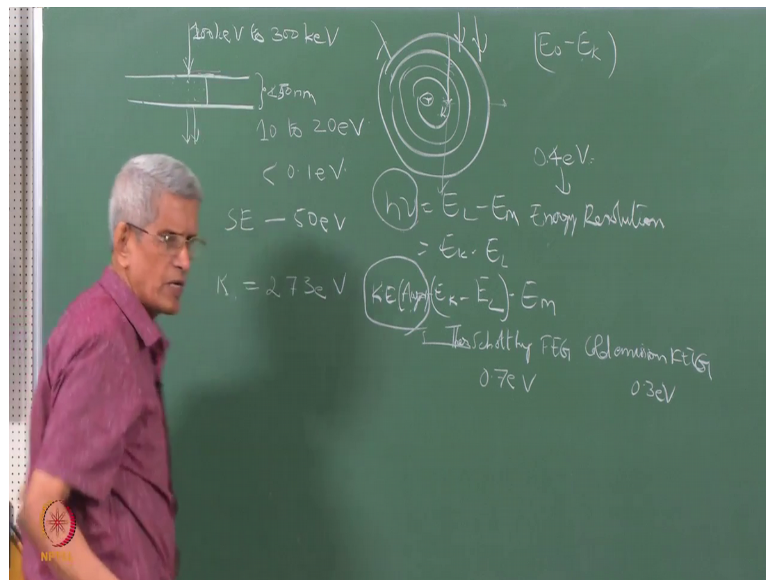


As the beam passes through the sample most of the electrons comes out without losing any energy or these electrons are essentially scat elastically scattered, but in addition to it a few electrons lose some energy by interacting with some of the atoms which are present on the sampled. This is a statistical process for each of this interaction there is some cross section which is associated on the basis of the cross section we can tell that if this is the incident beam current which we use it, or this is the total number of electrons which enters out of it may be only this much fraction we will interact with the atom and give rise to this scattered electrons.

The first thing is that when we said that electron interacts with that sample and various types of electrons are produced and what all the types of interactions which are possible one of the interactions which is called as a Plasmon interaction. What is a Plasmon? Especially that sample if you consider there. A crystal in or a non crystal in sample it

does not matter there are ion codes are there surrounded by electrons which are which are uniformly distributed in that sample. When the primary beam which this energy enters into their sample, its characters or import some energy to some of these electronics these electrons are diffracted from their original position and this gives rise to some charge imbalanced and the whole this electrons starts oscillating and d case and comes back to the road general position.

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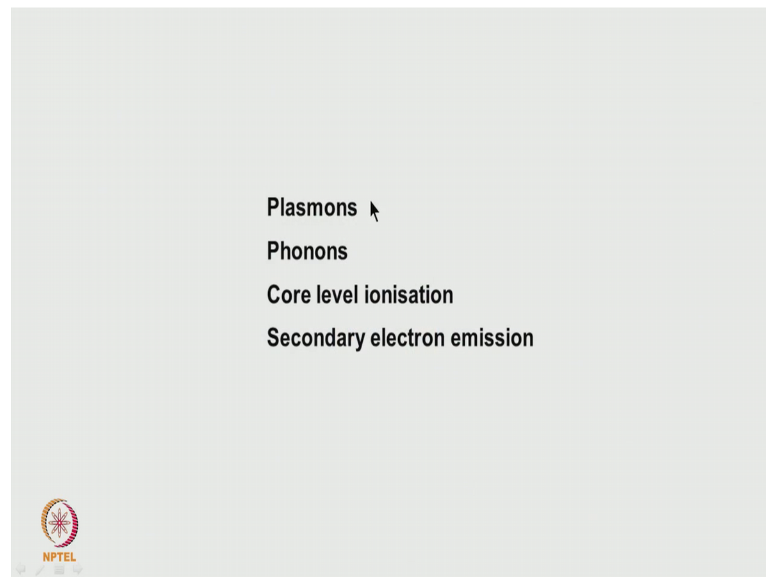


These sorts of oscillations are called Plasmon oscillations and the time scale of this operation is of the sort of femtoseconds. The energy which is being transferred to these electrons is generally of the order of 10 to 20electron volts these. So, this is one of the process by which electron loses some of it is the energy, the primary electron beam in a microscope loses some of its incident energy the other way in which the electron loses energy is pay phonon scattering. There is in parting some energy to atoms which are vibrating in the Latin, but the energy of this phonons is of the order of it is less than 0.1 electron volt. So, this energy is differences is normally a not very difficult to measure in microscope or the when we have a 100 or 200 k v electronics this sort of energy loss is not going to make a much of a difference.

Other than that what it happens is there the electron can; the primary beam when it has very high energy of the order of this energy it can knockout electrons for example, if it is a copper the sample is a copper base sampled, then it can electron from the k levels

which require around only about 8 to 9 k e v energy, and when electrons having such a electronics are knocked out from such levels which will be talking about a later. The atom will be in excited state it will come to the ground state by lot of radiative processes which are essentially a characteristic x ray emission or auger electron emission ok.

This especially the character these two processes could also be ocean the characteristic x ray could also be used for chemical identification of the elements, then secondary electrons are also emitted from the samples surface where the energy of the secondary electrons are less than 50 k e v a, 50 the electron volts 50 that is secondary electron energy is; for these are all the various processes by which primary electron beam loses energy as it passes through the sampled. What will the consequence of this sort of interaction immediately one can make out that when it loses energy when the electron comes out, we have some electrons which have not lost any energy there are some electrons which have lost some energy, because of that there is a variation in their wavelength of the energy of the electrons and the consequently there is a variation in the wavelength of the electrons.

One and consequence of this wavelength of the electrons are the energy spread in the wavelength, gives rise to chroma increases the chromatic aberration this bring about the lot of loss of resolution in of the images, that is the resolution becomes poorer. Let us look at what is the cross section for this various reactions.

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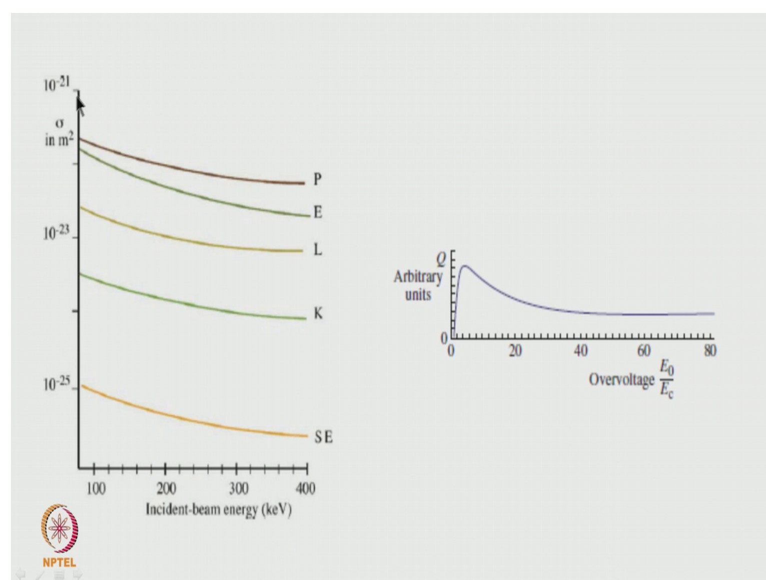

Here what is being done with this is you know there cross section is given in meter squared, we have discussed about cross section in some of the earlier classes when we talked about atomic scattering factor, this is for Plasmon loses the cross section is around 10 to power of minus between minus 21 and 22, this is for secondary electron emission the cross section is 10 to power of minus 25, and this is has the function of beam energy which we are plotting it and this is for K shell ionization this is for L shell ionization, this is for elastic scattering of the electron what is going to be the cross section.

So, if you look at it the Plasmon loss is the one which has got the maximum cross section, then comes the elastic scattering then is K and L shell ionization of the material secondary electron emission the cross section is extremely small.

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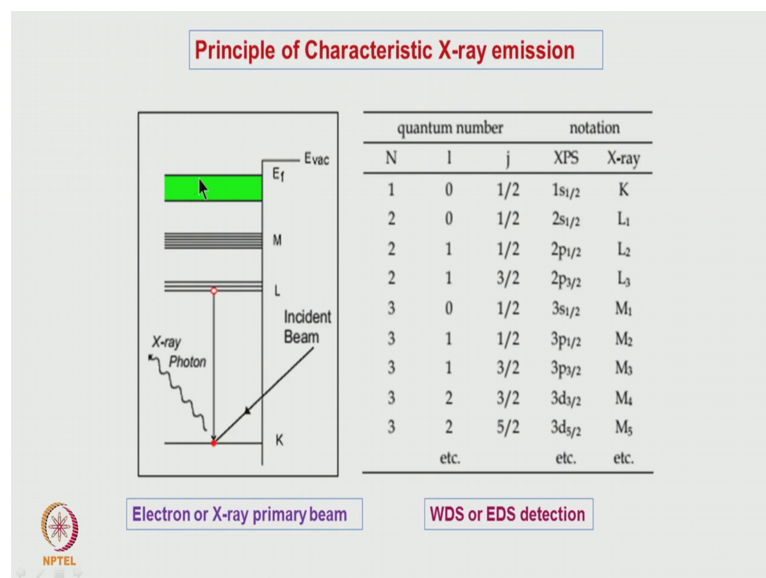


| quantum number | | | notation | |
|---|---|---|---|---|
| N | l | j | XPS | X-ray |
| 1 | 0 | 1/2 | $1s_{1/2}$ | K |
| 2 | 0 | 1/2 | $2s_{1/2}$ | $L_1$ |
| 2 | 1 | 1/2 | $2p_{1/2}$ | $L_2$ |
| 2 | 1 | 3/2 | $2p_{3/2}$ | $L_3$ |
| 3 | 0 | 1/2 | $3s_{1/2}$ | $M_1$ |
| 3 | 1 | 1/2 | $3p_{1/2}$ | $M_2$ |
| 3 | 1 | 3/2 | $3p_{3/2}$ | $M_3$ |
| 3 | 2 | 3/2 | $3d_{3/2}$ | $M_4$ |
| 3 | 2 | 5/2 | $3d_{5/2}$ | $M_5$ |
| | etc. | | etc. | etc. |

Now, let us look at what is the sort of interaction which takes place when an electron is entering in to the sample. So, when the primary beam enters into the sample since it has got a very high energy, you can penetrate that is there are electrons are this is the positive which are electrons are there in various levels, the primary beam could be entering here any where it is ok

Suppose its occurring very close to this electron which is a k electron that shall. We are in comes very closed to it the process when it comes from here to here itself it can lose some energy interacting with the outer most electron, even then the energy which is lost is very small it has sufficient the energy to give it to this electron which is there in the k

level. So, this electron if it has got sufficient energy it can be knock out from this level depending upon the energy which is required there is a minimum threshold energy which is required, that energy is the one which is required for the electron to be knocked out from the here and brought out to the outermost shell or the vacuum outermost level are the vacuum level or if the energies more than that it can be knocked out from the atom itself ok.

When an electron with this energy is knocked out, the atom is now in an exciter state to come to this ground state that is what essentially is being returned. This could be done by either x ray photon or it could happened that with that that incident beam itself could be an x ray photon or it could be an electron in this particular case we are considering the electron. When the vacancy has been created in this K level and the electron from this L level or from the M level or any of the above levels can come down to fill the vacancy in this level, and the electron in this level is the one which is going to see this difference; see the vacancy which has come in this which is able to fill it. So, this will come down and giving rise to an x ray photon.

This x ray photon if you see that energy of the x ray photon is essentially it has difference between these two levels EL minus that is h new equals EL minus E m or equals E k minus E L. This depends upon what is the sort of transition which is taking place. When an electron with this x ray with this energy comes out of the sample surface, this characteristic x ray we can measure it and this is counting the number of x rays with these energy is coming out we can find out what is the concentration of that element which is going to be there in that sample. This way we can find out the composition of the sample ok.

This technique is used in a c m this technique we use in a electron probe micro analysis, this technique is used in t e m as well especially in analytical electron micro scope it is being used. Other process by which and there are some rules which are governing this emission of this access the transition rules are there which has to be followed, and using either wavelength is passive detector or energy dispersive detector, this x rays could be generated which we will show later.

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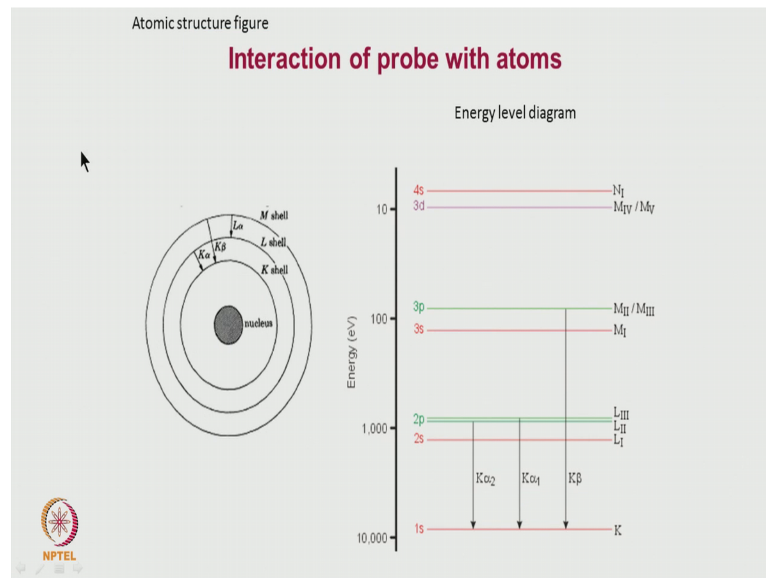


In addition to it when an the other alternative process by which this DK can occur from exited state to a ground state is there, when a vacancy has been created in this particular level we said that an electron from a higher level we will come down to this level for example, K alpha 1, this comes down to this level to fill the vacancy and x rays is generate. Assume that instead of it this energy is used that is this electronic comes down and instead and another electron is emitted from a higher level which requires very small energy; that means, that this energy which is available to used to knockout an electron from an another level, this transition is called as radiation less d k (Refer Time: 14:11) translation.

So, then the energy of these electron will turn out to be E k minus E L suppose the energy of the electron to this particular m double e c Em minus Em, this is what is going to be the kinetic energy of this electron. These electrons are called us auger electron and for this discovery for a auger in 1925 he was given the noble prize. So, essentially these are all the two process. So, this auger electron is all characteristic of the element. So, by measuring that energy of these auger electrons we can identify what is the type of element which is present on the sample surface and from looking at the integrated intensity of the peaks, we can find out what is the concentration of the element in the sample.

Since, we are using electrons as I had mentioned here as the electron enters into that sample it starts using energy before it interacts with that atom. What it essentially means is that either it can lose some energy and then come and knock out an electron from this level or you can so happened that it has not lost any and it has the full energy it has reached and part of the energy easiest to knock out the electron from this level to the way can level.

The now we know the energy which it has lost if the primary energy of the electron beam is E 0. E 0 minus E k is what is the energy with which the electron will be coming out of the sample surface; if an electron comes out with this energy this energy is also characteristic because a definite amount of energy which has be lost characteristic as of the element from which it has loss the energy, and we measure the kinetic energy of these electrons this could be used to identify the element which are present on the surface; that means, that the primary energy has lost some energy loss and this energy losses beam use find out the composition as well as do a quantitative analysis to find out the concentration of elements in the sample surface.

So, this is called as electron energy loss spectrometry. One of the important advantages of electron energy loss spectrometry is that even for either x ray emission or an auger emission vacancy has to be first created by knocking out the electrons from the various core levels has to be done, this process is always going to occur. Since this process has to all occur, we can use this technique is sensitive to all the elements which are present your respective of their atomic number.

So, even light elements could be detected using this particular technique we will come back to it in a minute. Another important factor which I mentioned is that there is a probability that has the electron enters into a sample it loses some energy, suppose it has loss sufficient energy it has not sufficient energy to knock out an electron from this, this we will contribute to the background level. That means that even if it has loss some energy to knock out an electron from this level some minimum amount of energy is required ok.

That energy is called as an over voltage which is required, generally if the incident energy is equal to three to five times the energy of the electron which has been knocked out from the particular level, then the probability for this process to occur is quite high

that is what essentially is being plotted here as the over voltage versus the yield which is going to take place. And here you can make out that as a function of overvoltage if you look at it at some particular energy that is the value of overvoltage it has reached the maximum value, then it comes down then it remains almost independent of the overvoltage. That means, that irrespective of whether we use a 100 k e v or 3000 k e v ok essentially the yield is going to remain that saying that is one particular one. Another important factor which we have to consider is that when a vacancy has been created in this level and the automation and excited state, it comes to ground state either by characteristic x ray emission or by auger emission. So, from this exciter level these are all the two processes by which an atom can come down to the ground state.

So, these two processes if you try to look at these are competitive process only one of them could occur.

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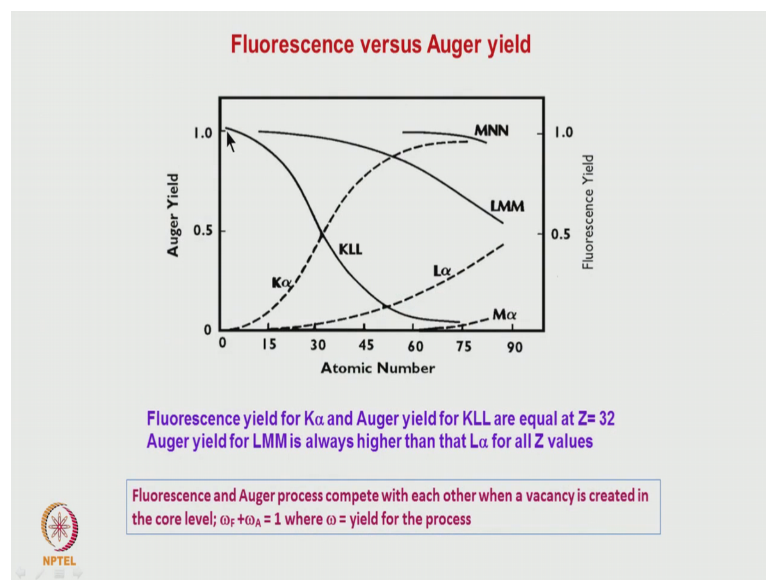


So, if you look at that yield ok for light elements, the auger process it is the atomic number with a auger yield when we plot it for the light element the auger yield is very high almost close to one because x ray yield is almost becoming 0 and whereas, as the atomic number increases we can make out that the fluorescence yield, fluorescence yield is nothing, but the x ray yield that is increasing and for k shell ionization close to around 30 or 31, this is our the atomic number around which both the auger yield and the x ray yield is becoming that same.

So, for atomic number smaller than this the K shell ionization auger yield is high. So, this is much more sensitive technique to find out the composition of the elements auger whereas, atomic elements higher than 30, essentially it is better to do energy dispersive analysis using the characteristic x rays to find the chemical composition because the sensitivity is very high.

So, for especially for light elements if you look at it for various reasons you know that the elements less than 10, it is going to a the detection is going to be very difficult for x rays. So, auger detection is going to be possible from this what it we can make out is that irrespective of whether it is a auger emission or an x ray emission which is taking place all these emission can take place only if a vacancy has been created in the core level. So, this creation of vacancy in the core level process is as we can make out is independent of the atomic number of the elements or that means that when the energy loss of the primary beam is taking place, this is not that sensitive to atomic number of the elements.

Because of that we can detect light elements as well as the heavy elements with very high sensitivity. So, this is one of the advantage that heals could be used to get concentration of light element like carbon, nitrogen, oxygen in the sample here the characteristic x ray.

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**Table 6.3** Characteristic X-ray Wavelengths and Energies of Selected Elements

| Atomic number | Element | $K\alpha_1$ | | $L\alpha_1$ | | $M\alpha_1$ | |
|---|---|---|---|---|---|---|---|
| | | λ(Å) | E(keV) | λ(Å) | E(keV) | λ(Å) | E(keV) |
| 4 | Be | 114 | 0.109 | – | – | – | – |
| 5 | B | 67.6 | 0.183 | – | – | – | – |
| 6 | C | 44.7 | 0.277 | – | – | – | – |
| 7 | N | 31.6 | 0.392 | – | – | – | – |
| 8 | O | 23.62 | 0.525 | – | – | – | – |
| 9 | F | 18.32 | 0.677 | – | – | – | – |
| 10 | Ne | 14.61 | 0.849 | – | – | – | – |
| 11 | Na | 11.91 | 1.041 | – | – | – | – |
| 12 | Mg | 9.89 | 1.254 | – | – | – | – |
| 13 | Al | 8.339 | 1.487 | – | – | – | – |
| 14 | Si | 7.125 | 1.740 | – | – | – | – |
| 15 | P | 6.157 | 2.014 | – | – | – | – |
| 16 | S | 5.372 | 2.308 | – | – | – | – |
| 17 | Cl | 4.728 | 2.622 | – | – | – | – |
| … | … | … | … | … | … | … | … |
| 19 | K | 3.741 | 3.314 | – | – | – | – |
| 20 | Ca | 3.358 | 3.692 | 36.33 | 0.341 | – | – |
| 21 | Sc | 3.031 | 4.091 | 31.35 | 0.395 | – | – |
| 22 | Ti | 2.749 | 4.511 | 27.42 | 0.452 | – | – |
| 23 | V | 2.504 | 4.952 | 24.25 | 0.511 | – | – |
| 24 | Cr | 2.290 | 5.415 | 21.64 | 0.573 | – | – |
| 25 | Mn | 2.102 | 5.899 | 19.45 | 0.637 | – | – |
| 26 | Fe | 1.936 | 6.404 | 17.59 | 0.705 | – | – |

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Element for various and the elements are given in this table.

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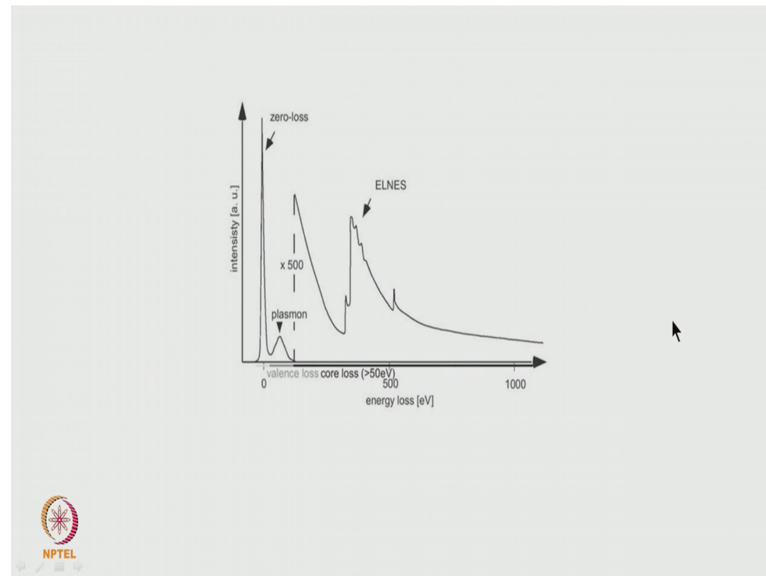


What is given here is that as I mentioned the electron with a particular energy enters into that sample we assume that it is a one particular type of it is a poor element like an aluminum or copper or a nickel, by various processes which are being mentioned the electron uses the energy. The electrons which are coming out of the sample surface if you look at it there mono energetic electrons; some electrons are coming out with the same energy there are some electrons which we have loss different amounts of energy by processes which we have mentioned, and if you try to find do a quantitative analysis of how much of the electrons which have lost energy, that is what essentially being plotted the energy loss versus the intensity.

Now, we can make out that this is the sort of a spectro which it comes, this is the electrons which I have not lost any energy their concentration is very high then with about 20 250 electron volt there say Plasmon loss which is going to be there, this what is shows the Plasmon luck speak and in fact, here from beyond this one the peak is going to be so small that we will not be able to see it. So, it has been magnified 500 times, now we can see that there are many types of peaks these are all corresponding to different absorption edges, because corresponding to K edge are Ledge are M edge; that is essentially what we are able to see. Then in addition to it there are some small peaks could be noticed beyond this absorption edges, after as well as before these also contain some information about what is the chemical state in which the atoms are existing that sample.

So, using this electron energy loss spectrometry we can get information about not only the elements which are present on the sample surface in sample surface, but also what is the chemical state in which these elements are present.

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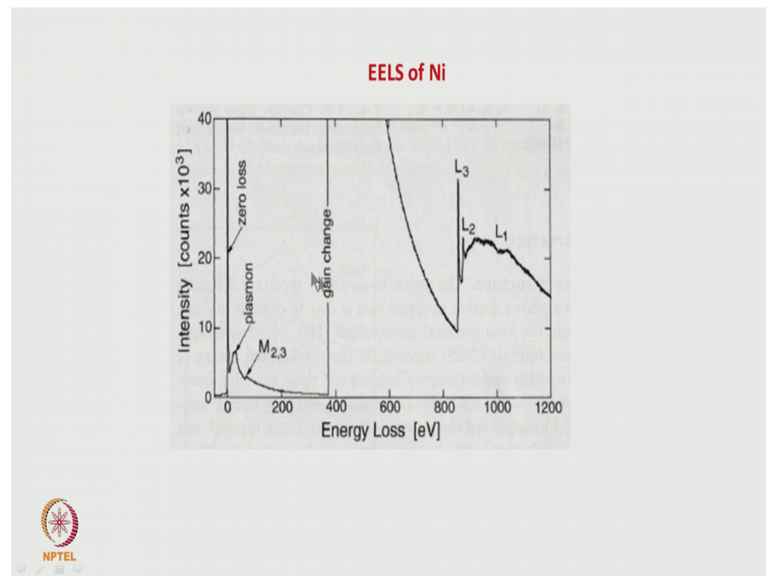


This is for a case of a nickel we are showing how this spectrum looks like the zero loss has got the maximum intensity, and the next comes the Plasmon loss than this is for nickelium 2 3 edge, then the earlier edge comes for which we can see that the gain has to be increase quite considerably to see these peaks; that means, that the probability of occurrence of this peaks or rather very small. What are the ways in which; that is what we mentioned so far is about what is the process which takes place ok.

How this process can be put to use in a microscope? There are various ways in which it could be done.

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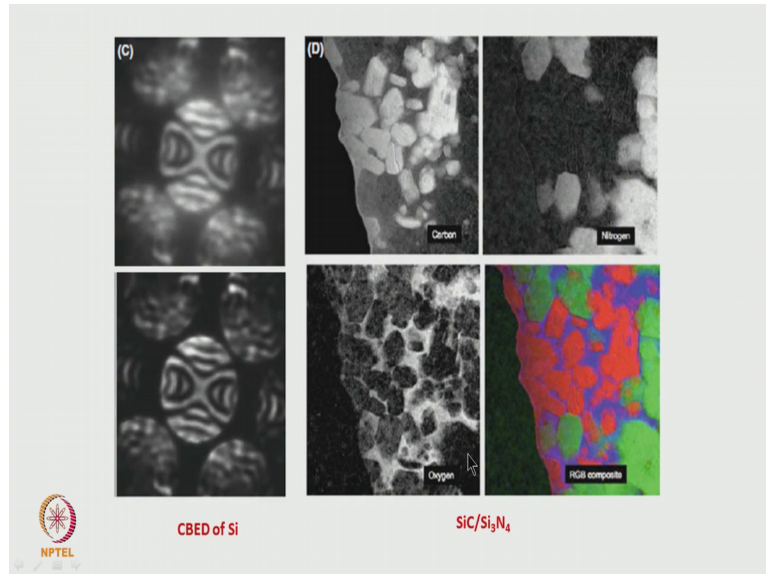


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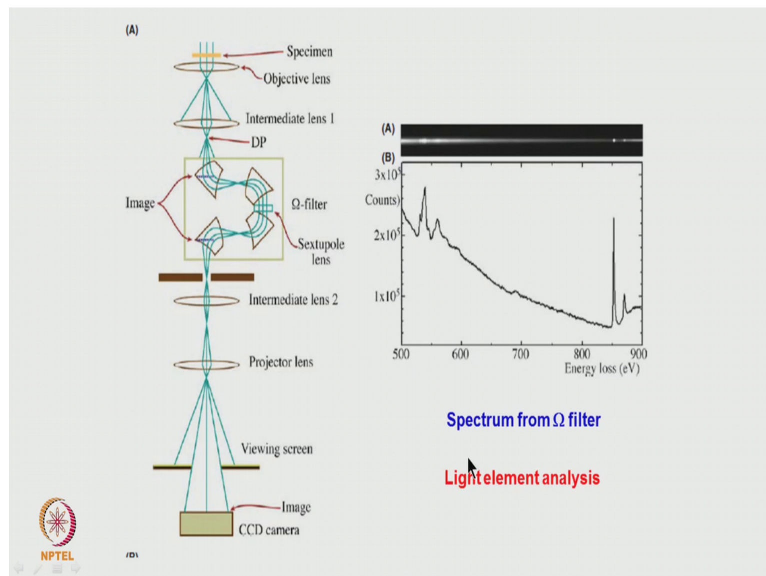


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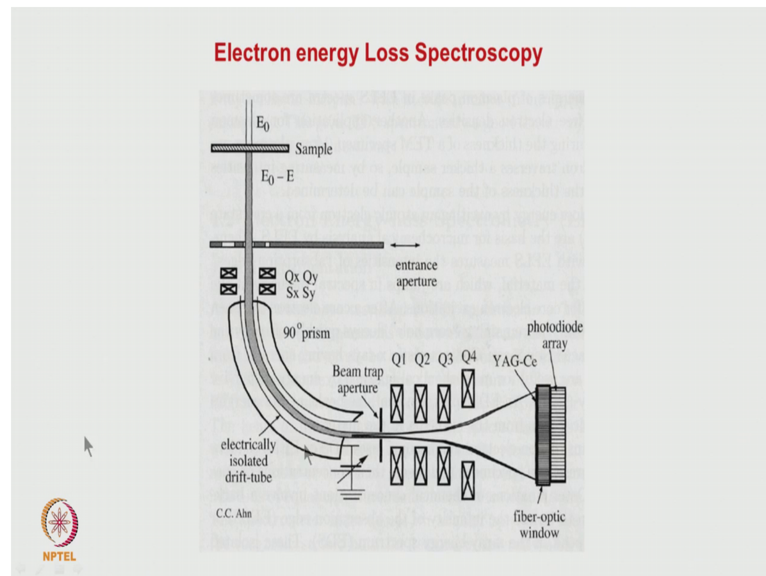


One way in which it could be done essentially is the electrons are falling on to the sample surface and that beam has been made into a very fine beam. In fact, if you want we can scan that beam on that sample surface also are we can make a stationary find beam on to the sample, the electrons which I have last various energies when they come out. Then at the back of that microscope we can put a magnetic sector analyzer so that the Lorentz forces will be acting on this electron. So, since electrons are got different energies they will be focused two different points, and this thing using various lenses their separation has been magnified and they will be following on a photodiode array and depending upon where they come, from this we can immediately find out what is going to be the energy of this electrons.

This way we can find out an energy analysis and find out what is going to be the intensity corresponds to a concentration and the position at which they occur that all about from which elements there are coming. There are is what essentially this is a sort of peaks which we a get it. So, this we can use to get information about it.

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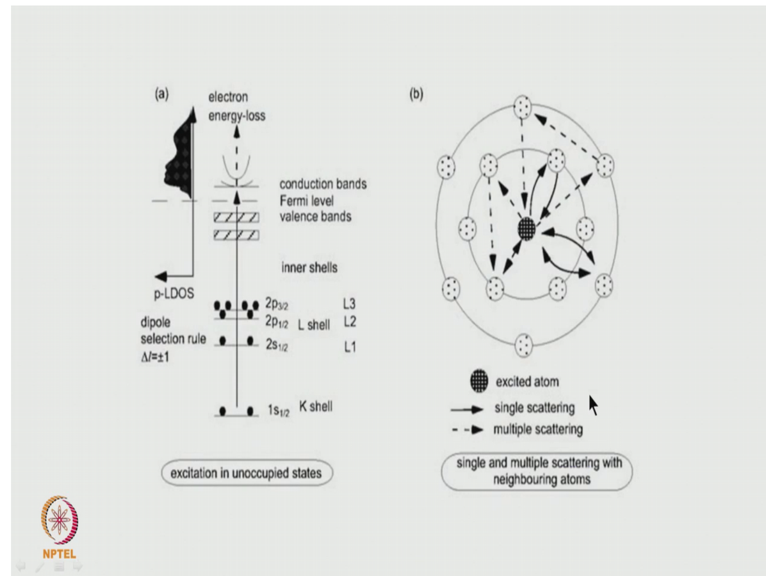


Another way which this has been used very effectively in a microscope is incorporation of a magnetic sector analyzer in the column of the microscopy itself. So, if what one can make out is that is the sample specimen in which the electron mono energetic electrons are entering into this sample, this electrons as it passes through the sampled various types of interaction takes place, each interaction gives rise to some specific loss of energy.

So, the electron which comes out is polo energetic electrons which are coming on the sample surface, they are being focused using an intermediate lens and they enter into a magnetic field sector, where they are dispersed and then using different magnetic sector with it is we can arrange the magnetic field in such a way the electron having a specific energy only will pass through the centre and come back to that optic axis; that means, that by choosing the strength of this magnetic field, we can choose the electrons having specific energy to come through this lens. And only those electrons are going to contribute to the formation of that image on the viewing screen. That means, that on the viewing screen the image which we form one thing which we can do it is that, we choose the electrons only if the electrons having the primary energy.

So, all the electrons which has lost their energy they are removed from the beam and the electrons which has been only elastically scattered recommend form and image on their screen. What is the advantage of it? Suppose the sample is a thick sampled in which it enters lot of this inelastic scattering processes we will be taking place, electronics I have

lost in energy in a conventional sample if you look at it this will give rise to a poor resolution the image quality becomes very poor, and if you remove all these electrons then only the elastically scattered electrons are taken and we get a much clear picture of that sample. The same thing can happen in the case of diffraction also because we know that when the simplistic kukuchi diffraction takes place, which gives rise to a uniform background on the sample surface ok.

This background could be removed very easily by choosing electrons which are only elastically scattered because that a normal diffraction and takes place only because of elastic scattering this we can see it has an example.

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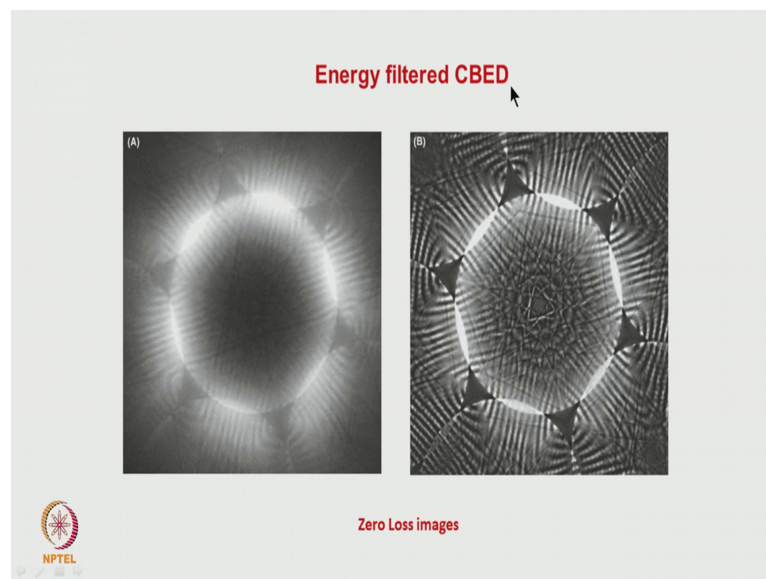


I have just taken with an energy filtered micron convergent beam diffraction pattern from a thick region of that sample; this is without any filtering when we will see this is how the convergent beam diffraction pattern looks like. When the zero loss images are taken; that means, that the electrons which have loss some energy or the other, they all been removed from the beam optical path then we are able to get a much clear picture of the diffraction pattern this is one.

And another is electrons which are loss some specific energy like suppose for different elements that sample contains two elements, they contain some carbon or they contain some nitrogen and some carbides of nitrates of form we want it to indentify whether it is a carbide or an nitrate then what we can do essentially is that the same technique itself

can be used and we know that if the primary beam interacts with that electron to remove in the case of a carbon, it is about I think some two 207 t r 273 I do not remember that value this is what the K alpha the energy which will be required to remove electron from a K is tell, this is the electron energy which we will be required or 277 of this order ok.

So, the electrons which we have lost this definite energy we can adjust the magnetic field in such a way that only those electrons you enters into that optical path and form and image then what is going to happen is that only regions which contain carbon that will be lighted and that is what essentially which is being shown here in the case of a sampled which contains, carbon, nitrogen, oxygen. We can immediately make out that these are all the regions which contain carbon I think it is nasty, and these are all the regions which are rich in nitrogen these are all the regions which are contains oxygen, this is the composite picture which shows giving different color code this essentially in a silicon carbides to silicon nitrate.

So, how the carbon oxygen nitrogen or distributed on the sample surface, that information we can get it. And another important factor which should remember in the case of a sample is there I mentioned that looking at some of these peaks which are occurring very close to the k edge we can find out the chemical state. The shift in chemical state is going to be extremely small of the order of a few electron volt because of this if the primary beam itself has got the high energy spread.

We will not be able to measure this shifts very accurately that is why in this case you see primary be beam having primary beam as monochromatic as possible is required and as we have talked about in an earlier class we have mentioned that for short key FEG the spread in energy is about 0.7 electron volt, and for cold emission FEG it is about 0.3 electron volt. So, the energy spread is very small or if you use other types of the sources like thermionic sources and LAB 6 we have to use some monochromator to bring down this energy spread so that if you use energies of this order then we can find out the chemical state of the elements very accurately.

Generally the energy resolution which can be achieved in the sort of spectrometer the best resolution which has been achieved in this spectrometer is of the order of 0.4 electron volt is there energy resolution.

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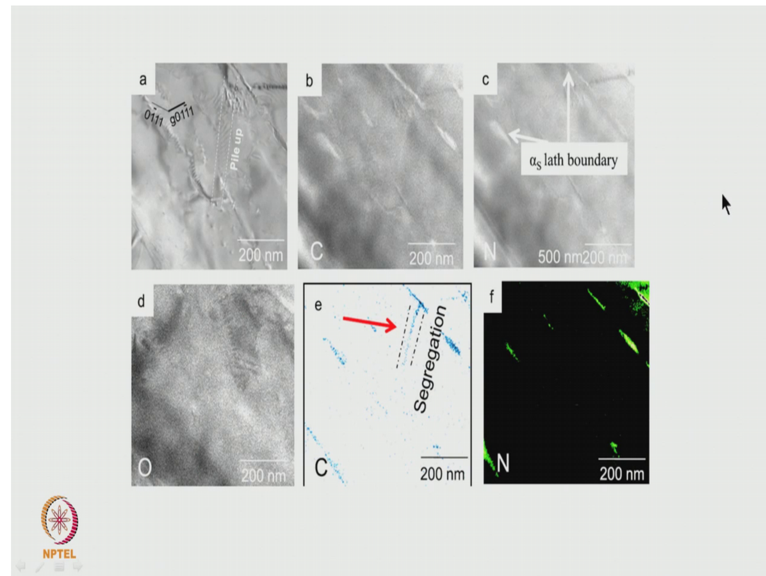


In fact, that we have use this microscope just one example which I am taking it is to find out how carbon oxygen and nitrogen all these elements are distributed in a sample which undergoes what we call it is DSA, there is (Refer Time: 35:36) dealing or that is what we called as dynamic strain aging behavior.

What is dynamic strain aging behavior, many samples which contain light elements at temperatures which are above the room temperature and many of this interstitial elements move very fast and they can form atmosphere around this locations, because depending upon whether this interstitial elements are whether they give rise to a stream which is positive or negative, they can segregate two this locations to the strain field after this location and form an atmosphere here.

One such an atmosphere has been formed it requires very heat stress for that this location to break this atmosphere and start moving it. This gives rise to some fluctuations in the stress strain plot. We wanted to find out because we know it is a titanium alloy which contains both carbon nitrogen as well as oxygen and we under wanted to know which element is responsible for this segregation, but if you look at what is the region over which this segregation is going to take place, this may be of the order of a one or two nanometers and that to with light elements with the other techniques it is going to be extremely difficult to identify them. Using this energy filtered microscope which is available at (Refer Time: 37:10) and now it is also available at NML Pune ok.

We could find out that there is segregation of carbon as well as nitrogen to these dislocations whereas; oxygen is uniformly distributed in the sampled ok. This I had mentioned about what is the effective of it in the quality of that image there are many applications which you can think of it is only left to your imagination to decide the what sort of experiment which you should do to get the best information using this microscope.

This is in a biological sample where we can enhance the contrast by remembering some of these Plasmon energy losses. So, so far I mentioned about electron energy loss spectrometer, what I had given is a very qualitative picture I have not gone into any of the mathematical this going to going to how to compute the intensity of the concentration, which is all given in most of the books and monographs which are available we can go through them and decide how to use this technique.

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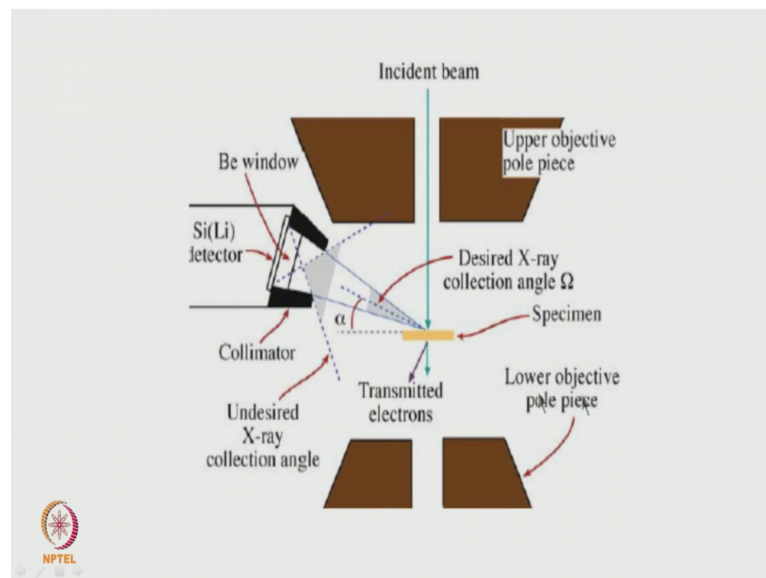


What is important is that I just wanted to give awareness about these techniques which are available in the microscope.

The other one is that as I mentioned there is when an k shell ionization has taken place, it can come down to a ground state by a meeting characteristic x rays. This characteristic x rays can be detected using sili detector this can be used to find out the elements which are present in the samples surface, in the modern day microscopes we have a special way in which this objective lenses are being designed, so that we can introduce so many

detectors right has close as possible to the sampled, and using this detectors we can get information about the x rays which are coming out of that sample surface.

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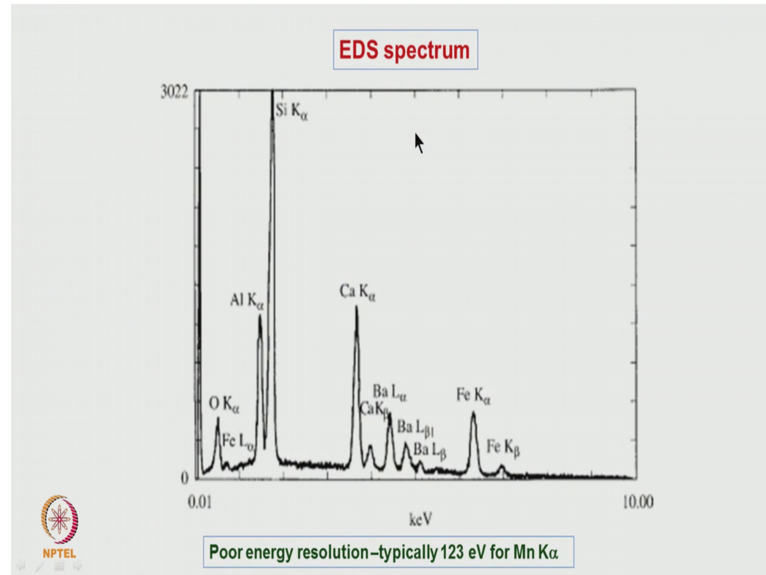


Using this detectors if we find out the intensity versus the energy of the x rays which are coming out we get some peaks, these peaks could be index because each peak corresponding specific energy value and from this we can find out what all elements which are present and using some calibration charts which are available, we can find out what is going to be the concentration of the various elements in that sampled.

There are two ways in which these concentrations could be determined, one type of a concentration which is called as a absolute concentration of the elements, and another is called as the relative concentration. Most of the commercial softwares which are available in the microscope they give only information about the relative concentration only they give they do not give in absolute concentration. If you wanted to find out an absolute concentration what we have to do is first we have to identify what all elements which are present then under the same identical conditions of beam current and the experimental condition we have to use standards for various samples and find out for that identical conditions, what is going to be the counts corresponding to that and that ratio has to be used between the counts which are obtained in the actual sampled to the counts which are available which are obtained from the standard sampled, and this could be used to find out absolute concentration of the elements present in the sample.

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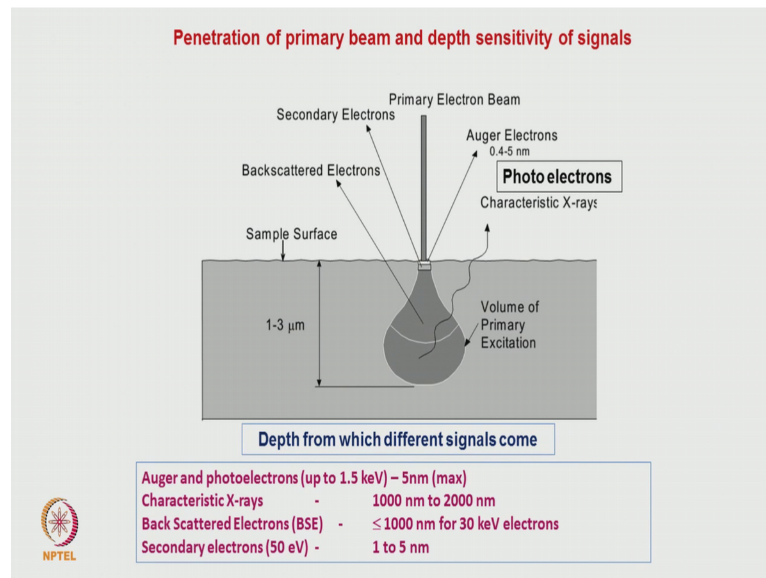


The same technique is being used in ACM also to find out the composition of the element. But what is the advantage of finding out the composition of elements in a electron microscope. For that we should understand that when the primary electron beam enters on to the sample for different process secondary electrons are coming out from some particular thickness, auger electrons from some particular thickness that is of the order of 1 to 20 a angstrom from the sample surface, backscattered electrons comes from maybe about half a micron and then the characteristic x rays are coming from the sampled about 1to 3 micron, not only that they electron be may sit enters their gets scattered in a bulk of a sample like the way we do it in NCM and they are getting scattered. And because of that even if the beam primary beam is a very focused verifying beam the x rays are coming out from a region which is very large, and this decides what is going to be the resolution of the detection of in the volume from which the composition is determined is essentially from this.

So, the special resolution for composition detection is rather very poor. What happens in the case of a microscope? In the case of a microscope essentially what is going to happen is that the sample is itself is about 10 nanometer to 50 nanometer. So, essentially the signal is going to come from this very small region. So, though the signal intensity has reduced, but the special resolution is enhance considerably, and since we are able to see the sample in the microscope, we know the region whether it is consist only of the precipitate or whether it consist precipitate and metrics all this information is available.

Where is in an s e m we look only at the surface of the sample, we know what is the depth to which the second phase is being present; because of which there is a lot of reliability issues regarding the composition of which we obtain from different region in an s e m that issue is reduced considerably in the case of a t e m.

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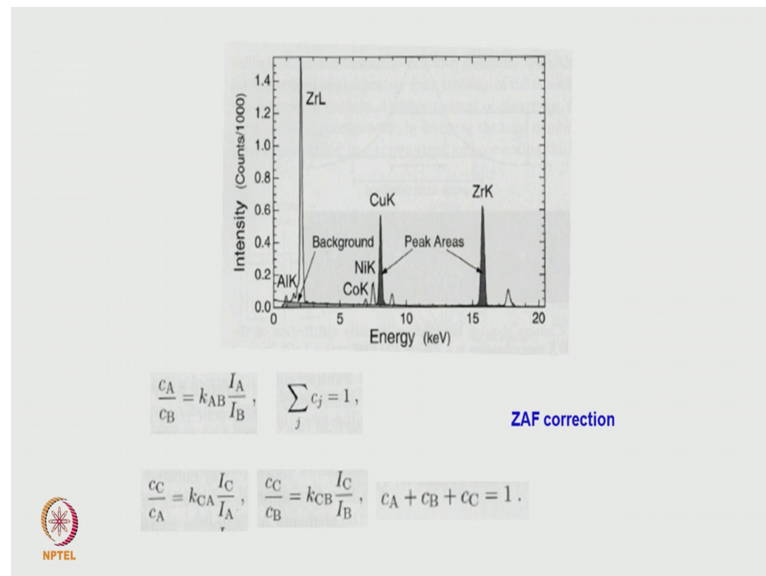


And another is that the composition as I mentioned we can find out the composition using that is if two elements have a composition CA and CB and the peak rate ratios are here copper and zirconium peak rate ratios are KA and KB ok.

When we look at the intensity of the peak rates that determines and various factors; one what is the probability of emission of l creation of a vacancy in the k level and what is the probability of an x ray emission and when this x ray is being emitted it comes out from the samples surface without using energy from some particular depth, then depending upon the incident energy there are many other corrections is said the f corrections that is for that absorption fluorescence all this factors which has to be taken into consideration, which you might have studied in the case of EDS analysis using in ACM.

Those things have to be taken into consideration all this factors are put together into a factor which is called as KAB this is called as a (Refer Time: 44:44) correction factor this has to be used and this is available in many of the softwares which are provided and these factors also can be experimentally determined how it is to be done, this is beyond

the scope of this lecture and I am not going into that, which you can look into the literature and we will be able to get this information ok.

Most of the time the concentration which we determine using the standard software are essentially the relative concentrations, and since the beam can be made into a very small beam the primary beam can be made to move on that samples surface. Suppose we have a p assume that there is a grain boundary is going to be there, when this beam is being scanned across it we will be able to find out how the composition varies across the grain boundary. This is one aspect which has been covered in this one. So, it is suspected that there is some segregation which has occurred.

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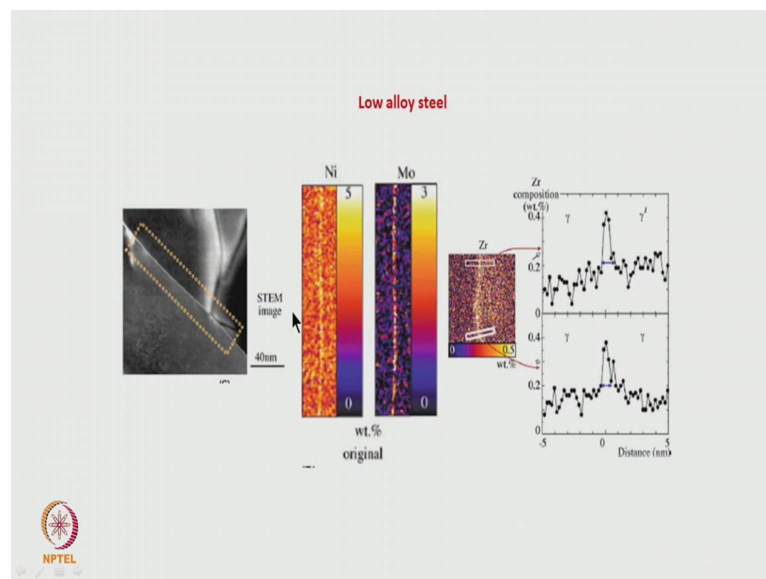


On to the grain boundary this is the scanning transmission electron microscope the image has been taken and then an analysis has been done on this sample which contains nickel and molybdenum both have segregator to this boundary, this is essentially a qualitative picture and then we can get some quantitative information also about how the elements are distributed.

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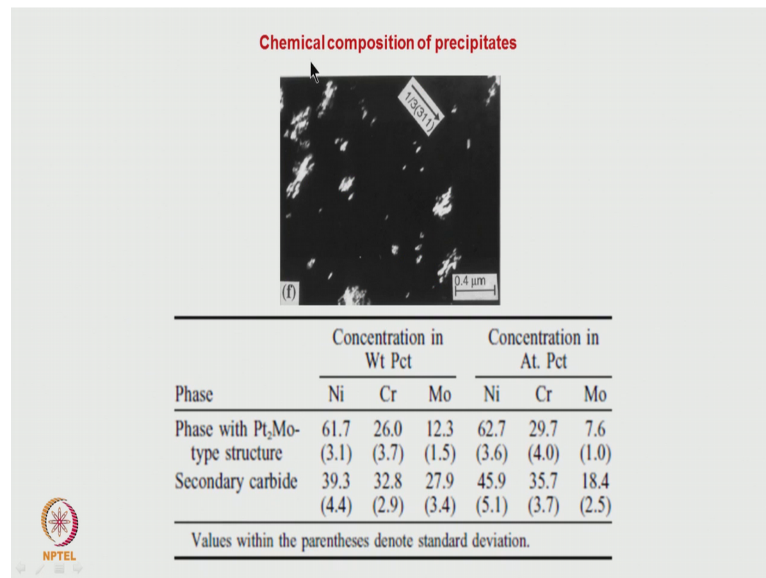


| | Concentration in Wt Pct | | | Concentration in At. Pct | | |
|---|---|---|---|---|---|---|
| Phase | Ni | Cr | Mo | Ni | Cr | Mo |
| Phase with $Pt_2Mo$-type structure | 61.7 (3.1) | 26.0 (3.7) | 12.3 (1.5) | 62.7 (3.6) | 29.7 (4.0) | 7.6 (1.0) |
| Secondary carbide | 39.3 (4.4) | 32.8 (2.9) | 27.9 (3.4) | 45.9 (5.1) | 35.7 (3.7) | 18.4 (2.5) |

Values within the parentheses denote standard deviation.

This is another example where we have very fine precipitates are seen. This precipitates are suspected be this is an inconel 625 alloy, where it contains nickel chromium and molybdenum. So, we wanted to find out what the composition of this precipitates using energy dispersive analysis, then we could find out what that composition is. Here the composition has been determined absolute concentration has been determined for which some standards have to be prepared, these things as you plan an experiment we will come to know of what all precautions which have to do and how you have to go about that part of it is not is beyond the scope of this lecture.

So, I do not go in to that detail using, which we can find out the composition of this phases and using diffraction we could find out what is going to be the crystal structure this information could be used to find out what the sociometry of this compound this essentially Ni 2 with C r M o.

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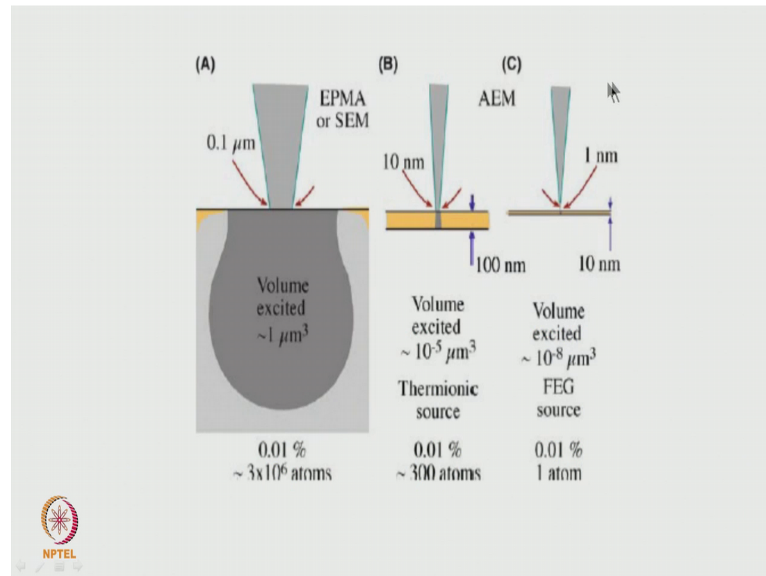


And this is something which is very important, what is essentially is being shown is that in the EPMA or in SEM, ok.

The exciter volume is very large size, though this is the beam size this is the size from which the signal is coming. Suppose we want it to detect some elements with concentration duration limit is 0.01 percentage, if that is what it is going to be the number of atoms which are going to be there of that element is going to be 3 into 10 to the power of 6 atoms. This is in an atomic there is analytical electron microscope where the beam size is 10 nanometer and the sample thickness is 100 nanometer. If you look at the exciter volume because you can make out that though here the excite the resolution is poor here the spread is rather very small because it is only a very small thickness which is because though the beam can come and enter one micron volume it can excite, the actual sampled thickness is only 100 nanometers, here the excited volume is 10 to the power of minus 3 here is the thermionic source, you about 300 atoms are going to be present which will be go which will give raise to 0.01 percentage the (Refer Time: 48:26) limit.

If we do with a probe character that is where spherical aberration has been corrected with a field emission gun, we can make the beam size one nanometer the sample size is 10 nanometer, then excited volume is very small and this percentage corresponds to

essentially detecting a single atom. In fact, using heals single atom identification is possible in the microscopes.

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$$\mathrm{MMF} \propto \frac{1}{\sqrt{P(P/B)n\tau}}$$

Statistical criterion for mass fraction

$$C_B(\mathrm{MMF}) = \frac{3(2I_B^b)^{1/2} C_A}{k_{AB}(I_A - I_A^b)}$$

$$I_A > 3\sqrt{I_A^b}$$ Statistically significant

$$\text{Relative Error} = \frac{3N^{\frac{1}{2}}}{N} 100\%$$

NPTEL

But what is essentially important is when we look at the peaks, especially with respect to the energy dispersive analysis when we are looking for the characteristic x ray p what is very important is that when should you decide that the peak a significant. That is if the intensity of the integrated that peak is 3 times, the square root of the intensity of the background peak then only we can see that it is statistically it is significant and that peak has to be considered in the analysis.

Sometimes to get this we have to do either a lot of experiments repeat the experiment or do a counting for a very long time. And the error which is going to be associated in that sample also which I think in standard statistics everything is given, you can find out that with the 99 percentage confidence limit if you wanted to talk about, it is essentially if n is the total number of counts then it will be 3 by root n into 100 percentage.

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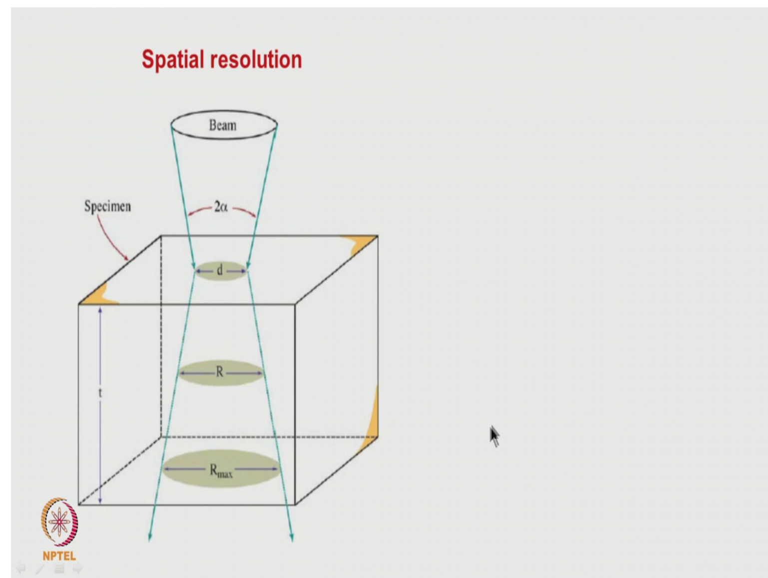


So here I am just showing that how the spatial resolution essentially is getting reduced because of the beam scanning when the beam size, as the sample thickness becomes smaller and smaller it has got a better resolution.

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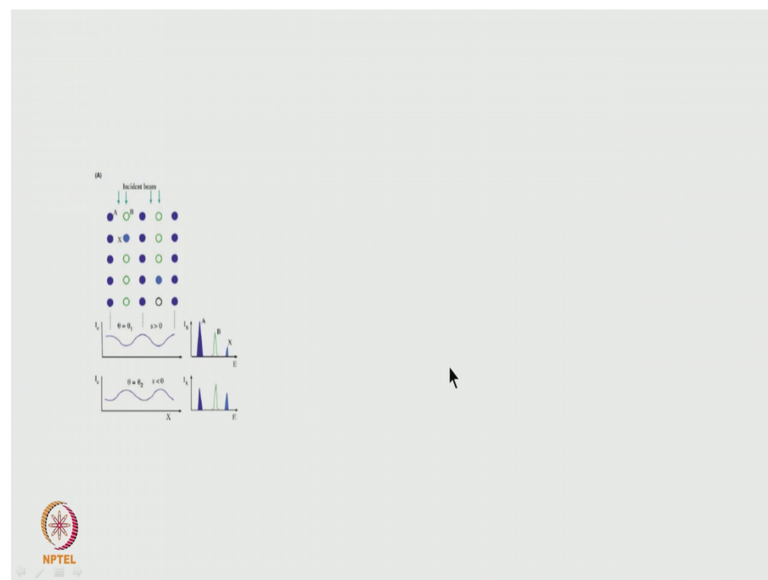


Another information which we can get it is using this technique, we can get information about the alchemy that is atom site location in that sample which atom occupies which location in the material, that information also could be used it can be obtained using energy dispersive analysis.

We will stop here, now essentially what I have done is said that we have try to give a brief idea of the energy loss spectroscopy and EDS energy dispersive spectroscopy is in characteristic x rays which are available in atomic not atomic analytical electron microscope.

Thank you.