Indian Institute of Technology
Kanpur

NP-TEL
National Programme
on
Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-23

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So we have discussed about Auger electron spectroscopy in the last class and this is the continuation of the that lecture and I have basically talked about the basic principles of audio spectroscopy the applications where it can be used, and i have given you some basic equations and diagrams to explain how the electrons are generated now in the subsequent lecture today we are going to see different applications of auger electroscope and finally i am going to show you the actual machine are actually configuration of a machine in the auger spectroscopy.

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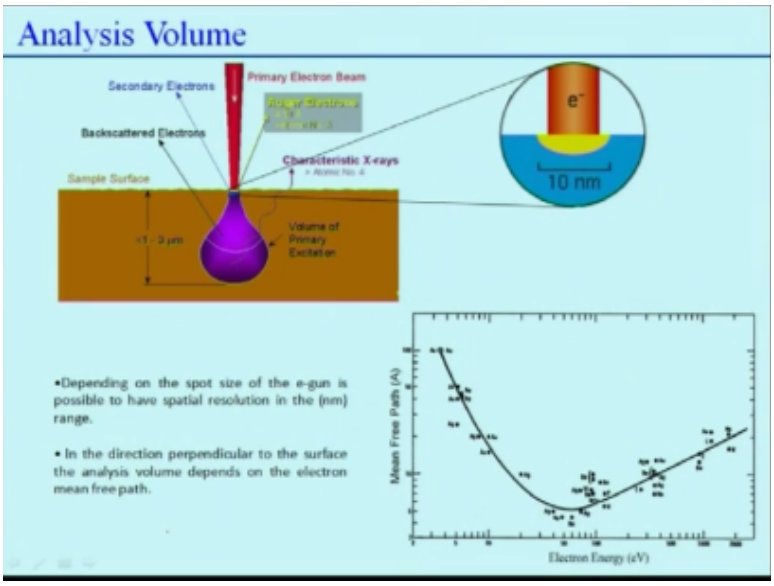

So before that let me just reiterate the following thing which is I should do it very carefully the reason og is very famous and very useful technique is because the signals which we get from the samples are basically from a very small thickness, very small thickness of the surface this is what is shown here, if you look at the sample surface and then there are different kinds of signals which is generated because of the inherent excitement are excited by the primary electrons as the primary electron falls on a samples.

It causes a lot of excitations and that leads to different kinds of signals can be back story later on signals can be secondary electrons, or it even a characteristic x-rays which are used in the scanning electron microscopes and also we will have auger electrons, but if we look at the interaction volumes from which the signals generate our get generated its valleys from a very large thickness to very small thickness depending on the type of signal, so for the characteristic x-rays the interaction volume actually is very large the depth from which the information can be can come is of the order of 3 < than 3 micron 1 2 3micron.

Actually on the other hand the back electron comes about see her back the electron comes about approximately about 2 micron secondary electrons comes from about a proximal about couple of hundreds of my am strong because under, so actually nanometers not am strong but auger which is very significant in no radiation comes from only for 250a depth and it is only possible to get auger excitation for atomic numbers higher than 3, so you can always get excitation from electrons a form elements which is having a to be number more than 3.

That is higher than the lithium, so that means the information which is coming for GS basically kind of a small depth on the sample surface and that is why is very significant tool to analyze those kind of small you know features on the sample surface auger, so this is what is shown here this is electron beam you can see here and then this is a depth oh the information comes in the auger depending on the spot size also a nominee spot size pot size electron gun the spacer resolution in this way resolution.

Special means this not the depth but the xy who depend on in the direction partner surface analysis volume difference in the electron mean free path that is what I am shown when you mean free path is high then electrons can you add more odds, here it is slow then mean free path is shown here as a function of electron voltage electron RG actually if you see her gold and this is silver they have a very small mean free path mean free path increases to you know like molybdenum, cerium beryllium okay.

So that means or even some other elements like phosphorus so that means the main FIFA actually depends on ethnic section and g is very high for the gold silver others were very low for the beryllium and other and the phosphorus and iron also so that that actually tells us that an apartment adept will also vary depending on the main pivot of the electron and different elements this also sets that what kind of elements you can analyze for a particular depth.

Well now let us move into the applications as I said we are going to discuss today that the analysis depth was just the information i wanted to convey clearly, we have already discussed about the chemical analysis.

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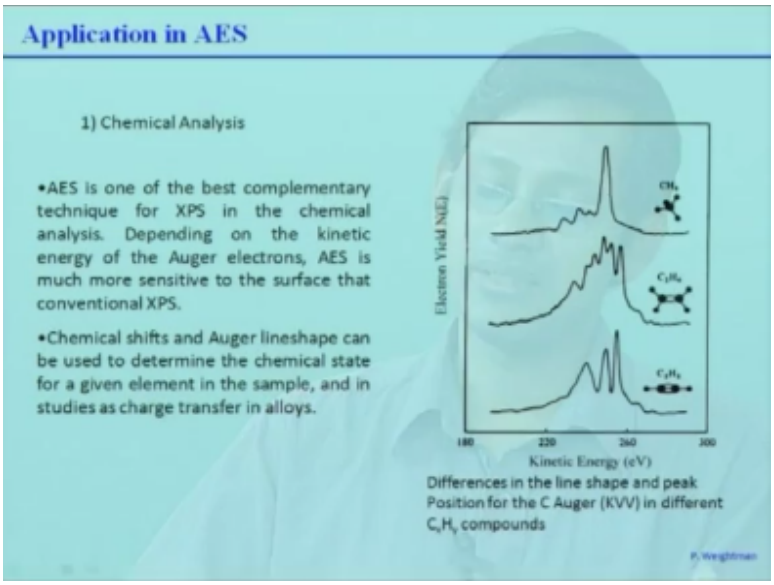

By using XPS or extras photo spectroscopy it is all the best complemented techniques for XPS in the chemical analysis depending on the kinetic energy of the auger electrons A is more sensitive to surface than the XPS it also gives us chemical shifts depending on the characteristics are they basically an extent of the element present auger landscapes can be used to determine the chemical state of a given element in a sample and then studies the charge transfer analyze this is very important one can actually study the start our install OS.

Let us now look at the differences in the line shape and the peak positions for the carbon auger spectra KVV transfer vacuum indifferent CX and hy compounds first, let us talk about if you know acetylene C_2 H_2 that in a triple bonds between two carbon atoms you can clearly see the three distinct speaks one prod and the sharp peaks coming at around for about 242 to 60 255 actually KV, but if you go to C_2 H_4 that is ethylene but there is a double bond you can clearly see this pic splits one two three four five six not only that the positions of the peaks are also a little bit has shifted to the left side.

There is lower kinetic energies now if I go to the element are they CH_4 that is the methane I can clearly see one big strong peak clearly visible and others very small picture visible, so that means the electron yield versus kinetic energy diagram can distinctly differentiate between carbon compounds from ethylene from acetylene to ethylene to meet in this is just basically to detect and a particular compound we can use these signatures, to detect whether they are present on a sample or not is the first thing you must know.

That means chemical analysis means qualitative and quantitative analysis quality analysis means whether we are able to detect a particular compound or element or particular species on a sample surface or not and second thing, if I able to detect many chemical processes whether you can quantify the amount of the each of these elemental PCs are compounds present in the sample surface, this is very difficult of any spectroscopy but auger also gives us much you know extra information are then this only.

Well now let us talk about little bit about the I know elemental ships for the different no first transition metal cities candium titanium.

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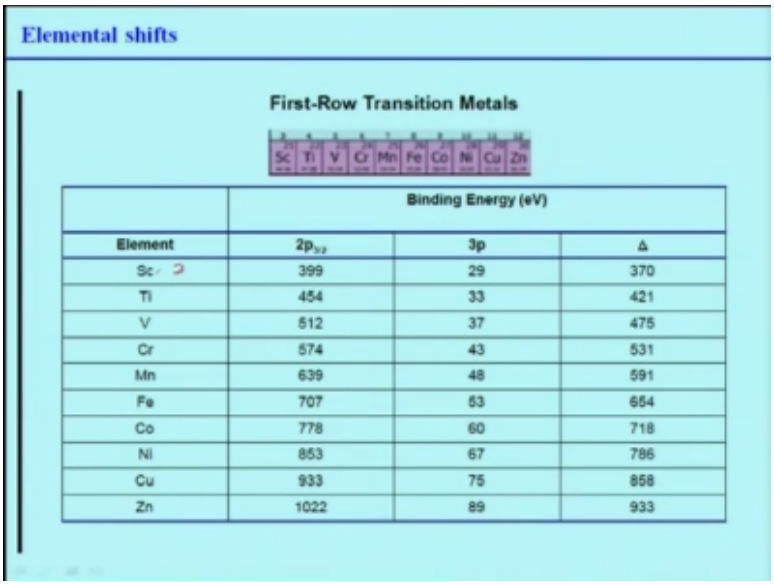

Can name 10 / 21 and then gene stands for actually 30, so if you go from 21 to 30 2p 3/ 2 Peaks actually shifts 399 electron volts 2022 electron volts 3p peak seeds from 29 to 89, now if you make alloys between them my school suppose if you make a lies between scandium and titanium they always shipped up 327 similarly between titanium and I receive top seed 421, so you can clearly see the ships because of the formation of allies among the different elements this allows means Sonny solution types.

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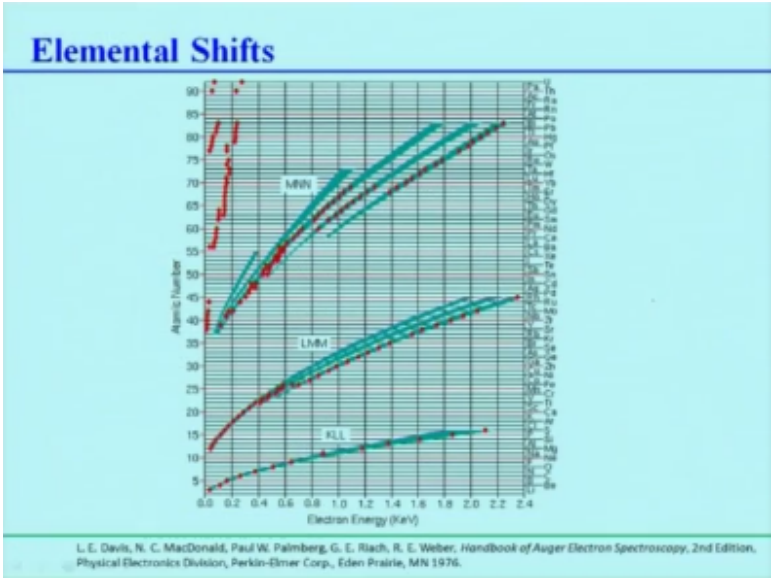

Well not only that one can actually look at the chemical shifts in terms of atomic number versus electron energy diagram this one I have always shown to you, now if you if you carefully look at this diagram it says different transitions like KLL transition LMM transition amen and transitions and higher depend on the atomic number, so obviously if the item number is less than about say 16 that is for sulfur, you will not have any Elam only KL conditions only when are to be numbers of the elements higher than 16 and lower than about 45 you have basically LMM transitions L.

And then there is a small overlap obviously between LMM and the KL conditions similar small overlap between LMM N and L mm transitions also but MNN technicians actually occurs mostly for the elements with a very hard to mean umber like more than 45 and less than about 84 bit three, so that means depending on this from this from this diagram actually one can actually understand that if I have alloys suppose, if I have a large Whitney aluminum and the niobium obviously there will be chemical shift of the pics from both element and IBM.

Because of these different tensions not only that that we also chemical shift if you considered only suppose element is a very close by suppose we consider boron and I know aluminum, so there will be some chemicals if possible even in KLL transition because of the atomic configuration electron configuration change a task of cash transfer issues in allies.

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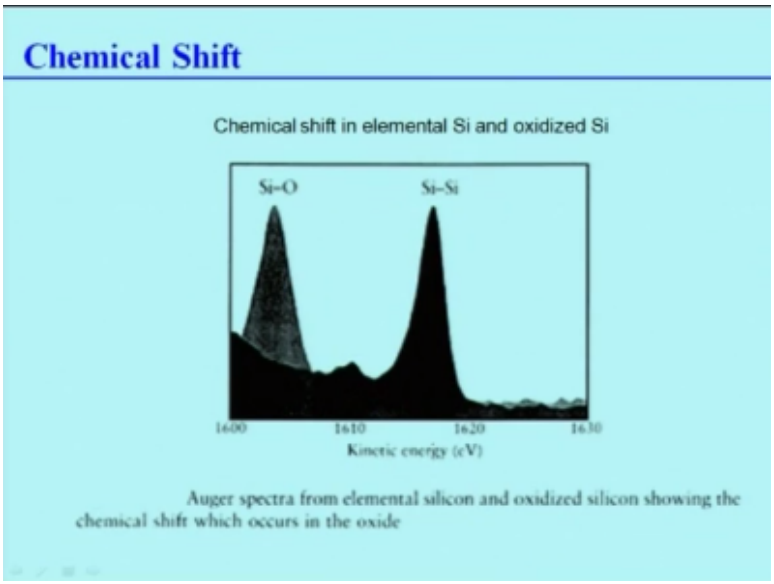

To give you much better perspectives how the chemical shift actually is observed in the auger I am not actually discussing the exact physics behind a chemical see because this is what i have done in a XPS the theory is same whether it is why spear auger only thing which I am describing here is that how this chemical shifts can be used to detect particular type of chemical bonding a particular type of way no electronic configuration, let us consider silicon an oxidized silicon in silicon yup silicon bonds.

And if I dick auger spectra a silicon bond gives us a very characteristic speaks at about1600 and 17 to 18 electron volt, but when the silicon is oxidized uses most of the cases happens on the surface of the silicon we have a silicon oxygen bond, so therefore because of the presence of silicon oxygen bond the peak from this one gets shifted to the lower value and it has been observed that peak comes about sixteen hundred and five electron volts, so auger spectrum a spectra from the elemental silicon which is very pure and the oxygen silicon which is having oxygen as one of the element present.

In the along the silicon can distinguish the type of bonding between these two very clearly and it is even much clearer in auger than in XPS that this kind of chemical shifts are actually observed and can be used to differentiate between the different kinds of warnings bonding in the sense of or this bond between similar atoms or dissimilar atoms oxidized nitrites all can be detected them to give you much even higher.

No much better perspective in the sense let us now talk about elemental germanium and germanium with a thin oxide layer same as silicon, but here the element of germanium is Devon singing crystals so you have germanium zero pick.

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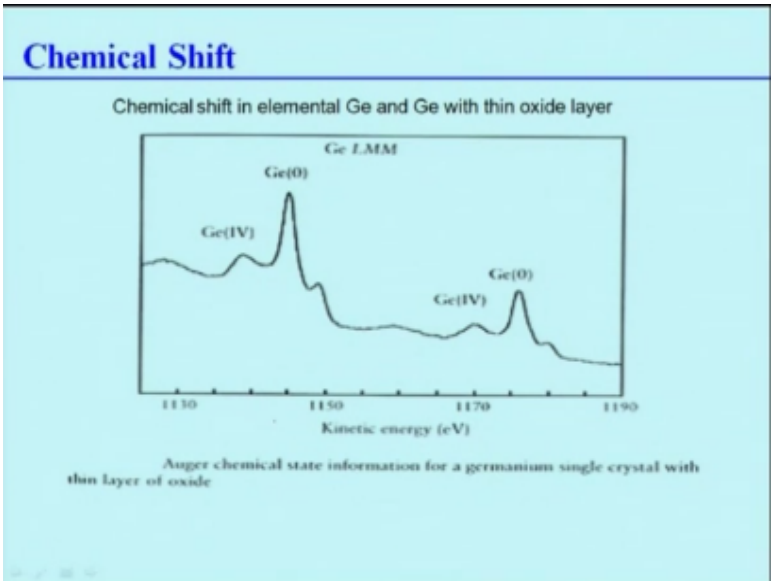

That mania for pick you can see this is that my name LM transitions, on the other hand if you look at if others oxide that saw they basically these two this is actually from the pure germanium this is from the oxidized pics, so this is oxide this is real germanium so you have oxide layer this is shipped from the pew germanium zero and germanium four peaks to the lower level say from 1175 1175 to 11 you know 45 there are many mg depicts chips.

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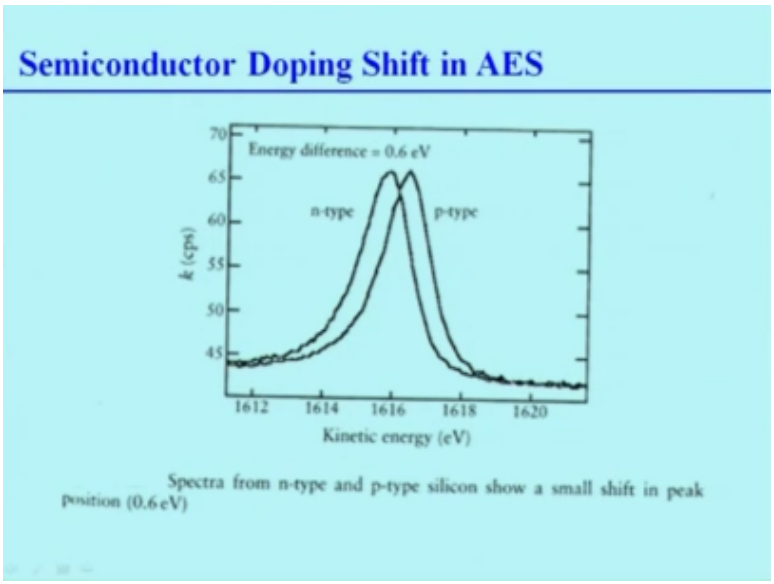

Not only that even the algae is very sensitive to presents a very small quantity element like Jimmy Carter doping let us talk about semiconductor up as silicon the difference between P and n type silicon, can also be distinguish seen in the audio spectrum that is what is shown in this slide, so I know you know that p and n type silicon can be created by doping different elements if you dope with you know p the pub five and a b like phosphorus silicon you get pit an-type and if you do pick boron you get p-type or other elements like.

So this is the yield in XPS and kind a can see plot you can see in type and p-type that is a distinct you know position of the pics and the another difference between these two peaks are about point 6 electron volts, so that means experience is so sensitive it can also detect this much of energy spread when you dope silicon to make it in or p-type the small shift is sufficient enough to tell us let us know that it is indeed a an n-type or p-type well one can actually detect even this pics save this picture is not very clear i have taken from a book but it is not clear but still Ito show you because so that you can get idea

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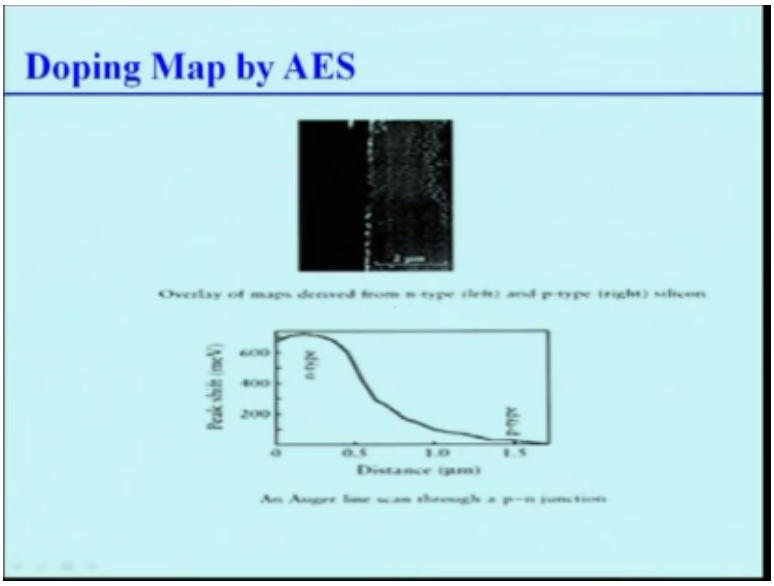

So this is basically difference between N and P type is n type this is p-type silicon in the image so we can actually take a silicon single crystal and dope elements of different types and make An pn junction a PN Junction basically here N and P this is the junction, so you can see that peak shift changes as a distance is basically changes continuously from n type 2 P type this is basically the quantified value of the peaks shift form n-type region to peak temperature, so one can actually get an idea of continuous you know change in the in the doping characteristics as a function of distance.

By doing taking XPS spectra at different points on the sample surface like this is possible there is methyl occasion.

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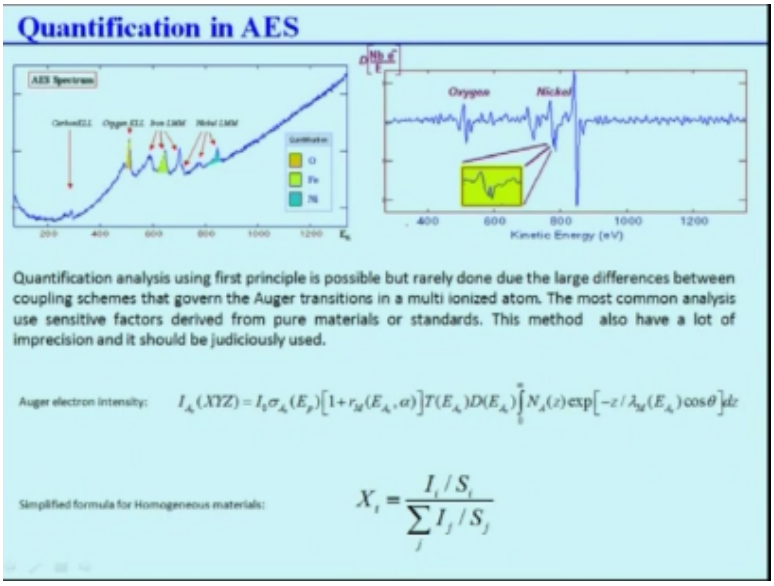

Next thing which I would like to discuss is the quantification in XA yes I think I have discussed a lot of a quantification XPS similarly auger specters can also be used to quantify different elements, so quantum analysis using first principle is possible but normally not done to rule our difference between these coupling schemes that govern the auger transitions in a multi and as Adam what is it means is that if there is a multi auger atom then there will be large differences between the couplings of different transitions.

And this makes the quantification difficult the most common analysis use sensitive factors derived from the pure materials or standards these materials also have lot of problem of position and should be used judiciously, but i like to know like you to know that he is allows us to quantify different elements present on the sample surface but physicians are much poorer the next PS, so what is the actual mathematical equation which uses since we factor actually all the electron density in a penny position xyz is given by this big equation.

This Is the instrument in CT this X cos section this is the energy level this is the diameter on which it is coming is also functional and as level and they is in beam angle and these are all this is the item number of items which has undergone transitions exponential transition wavelengths and cost they are scattering this is very complex formula and I do not want to discuss in detail with that requires a lot of theories to be first discussed simplified formula for homogeneous material is that Xi the amount of particular element.

I present is given by is x SI / ∑ πj x S_J a JS gave me some, so therefore I is basically the scattering factor and I the intensity, so if I know a scattering factors of the elements present and if I know the intensities of the each peaks I can actually indirectly calculate what is the amount of present in the particular specimen, so let us go back to the auger spectra this is auger spectra coming from oxygen iron and nickel and obviously Kelvin is in evidently present are omnipresent in any sample.

So a small carbon peak which is sometimes used to even calibrate save oxygen KLL transition here then iron a LMM transition here and nickel this is a small peak here and these two peaks basically LMM transitions, now as you see here the big pics are this one for oxygen this one for iron and this one for nickel correct, now I can actually calculate the area under these three peaks and get the intensity right, so and then if I know a scattering factor I can calculate I will quantify each of this element presence one a done can actually take is basically differentiate.

These it intensity respect to energy then γ can get much better plot which I have discussed already about the auger spectrum oxygen spectra becomes like this nickel comes like that so this way one can actually improve the quantification position much better a well.

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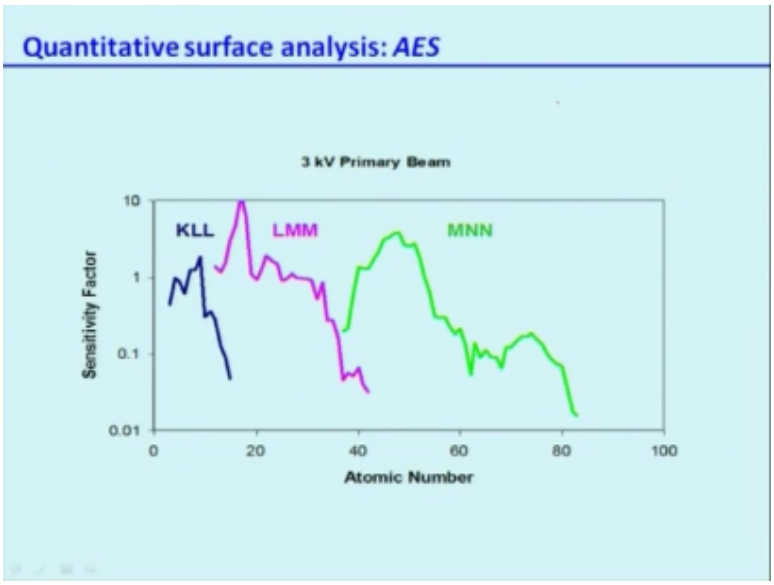

Let us now look in much detail this is the sensitive factor with buses atomic number since we factor is what I discussed this is a sensibility factor as you see from this part to this part it contains the tannins energy level the scattering cross sections incident beam energy level, so sensitive factors when very form 10^{-2} to 10 depending the Atomic number and depending on the transitions for the three key level of kilovolt primary electronic this is calculated for KLL transition sensitive factor will be very much lower.

And it basically close to one for most of the elements who studied this is very important because you know KLL transitions conditions is normally seen for all elements than not we number less than 16 or which are lower actually than sulfur and, so therefore this is what we get and the sensitivity factor is low that means the quantification done from this elements why he is even much less precise the highest stencil factor is obtained for LMM transitions an illumination actually takes place for a large number of you know elements.

Starting from atomic number 16 to add to my number about 45 a 65 let me just go back there and to show you what is that say this is yeah this is 45 this basically starts from 12 to 45, so there is a large range that is and that is the range we are having a very large value, so you can see here this is something like 12 this is what love something like 35 or 40 40 here so in this range from these two this actually the census factor is quite high, so therefore our measurements will be better mm in 10g amen in transition 1600 occurs at large at me numbers more than 40 and our 285 and this is also very high for a no atomic number ranges from 38 to 62.

So that means from the all the atomic elements with atomic number from about 16 to our 62 the synthesis factor is quite good and actually it is higher than point five that means we can actually use AZ is for this elements to get much better quantification analysis, well if I this also depends on the primary beam as you know if i use attend collect electron primary beam this is how the cops get shifted well not much shifting has happened except for a I know l we are getting an extra transition at LMM conditions.

But LMM transition sexually or we are getting still extended LMM transitions you can see they are, so little bit of change happens but not much.

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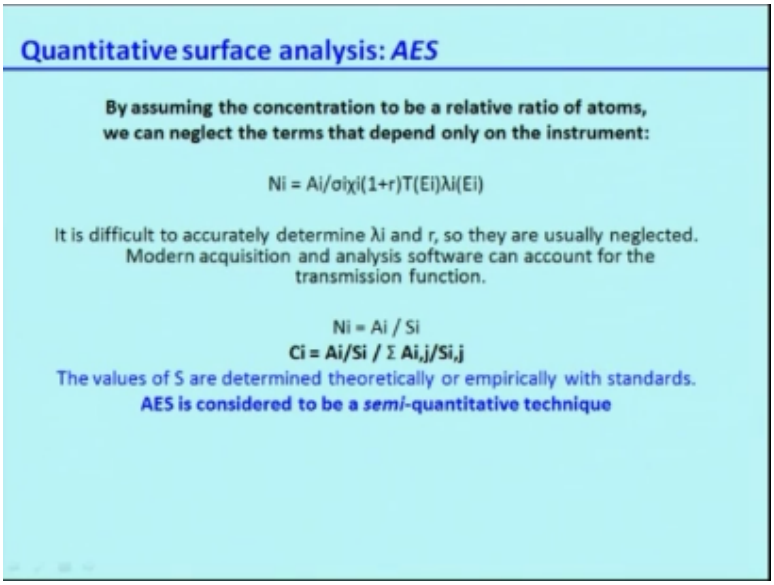

Now we can actually do it this is the sensitivity factor sorry so we can actually do it much better way that by assuming the constants into a relative ratio of atoms, we can neglect the term that depends on the instruments so we get this is AI σiI I think there is no need of discussing have already discussed about that, but this is different scheme the important thing is here to understand it is a semi-quantitative me it is not a quantitative technique / say like XPS.

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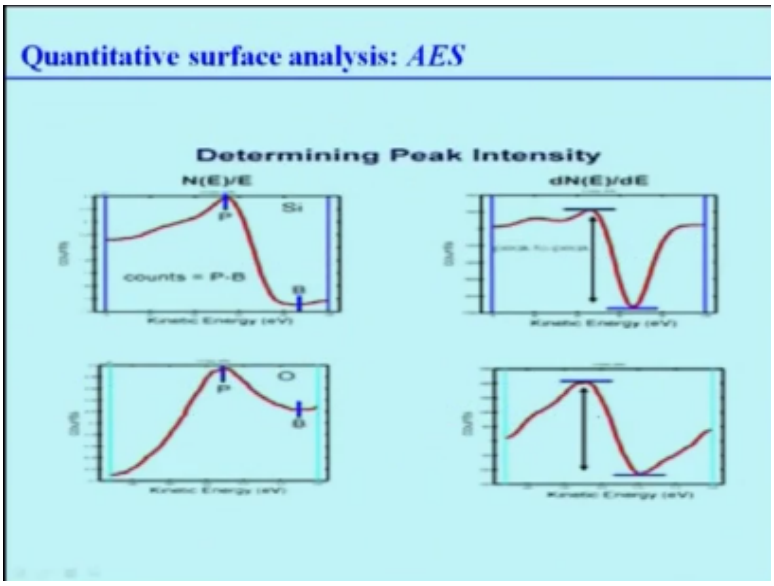

Well to determine the peak intensity I know that is what is very important because you want to calculate the area under the peak to know the in actually intensity, so one has to be very careful about to measure the peak intensity and show you some plots to make you understand how that you know judgment is very important this is the kind of quantitative is the CPS is you see this is the plot of silicon the p and b is basically the peak and the background, so you have to consider the peak area this way on the other hand if we take DNE /D but it is kind a you get a much better peak.

This is your peak area correct so that means it is better to use DNA / D than NE / E versus kinetic energy plot determine the area under the pics, so that quantization is much precise well.

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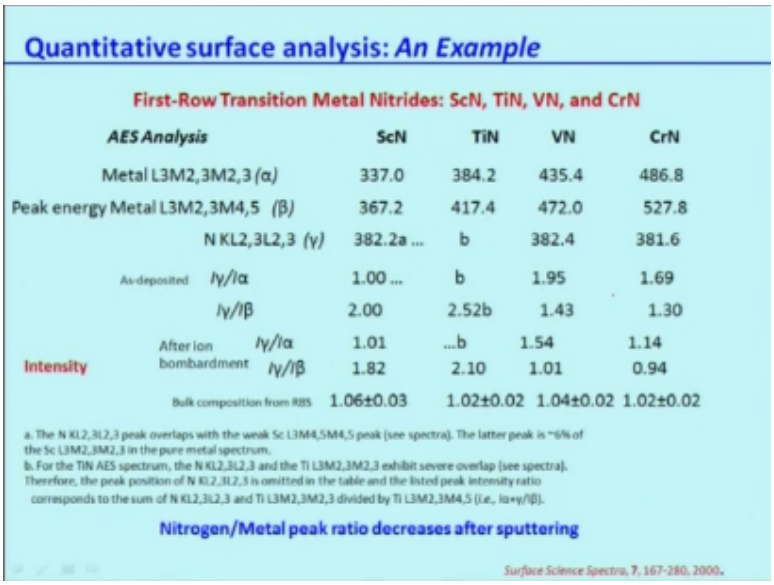

To give you analysis as compared to the you know others for compounds nitrogen compounds like this chromium Nitrides ferendum nitrate I I scandium nitrites the first two tiny metal nitrides you have basically l3m two or three m2 speaks for this you can see these are the peak positions the changes and for L3m to empty the peak positions actually are then this is what is important in as deposit the condition they saw the intensity ratios varies you know one p stands for you know for this once this is the sever will lab so you cannot detect be means we cannot detect this overlap these are the ratios.

After ion bombardment ratio gets modified right on the other hand if you do using RBS that is what we will discuss in the next the RBS means our for backscattering which is the seams tool the secondary ion mass spectroscopy this is the data we get so you can see this is very close for the panorama scandium nitrides, okay but this is not this is close to NM nitride this is a little bit lower the actual value so that means AES is not that get analysis as far as they seems r rays XP is concerned.

Second power observation from this is the if you do the bombardment after Bombardment before bombardment there is a change in the NAD into metal peg ratio this is the NAD into metal PE ratio Iγ/ Iα this big ratio actually decreases after sputtering that is what you see here from one from to it becomes1.8 2 2.5 to become two points 11.4 to become 1.0 1113 become point 94 that is clear that is because nitrogen actually gets removed in sputtering because there is a light element.

So many time sputtering is bad that is why it Is better to do this analysis in the vacuum without in his pattern next thing.

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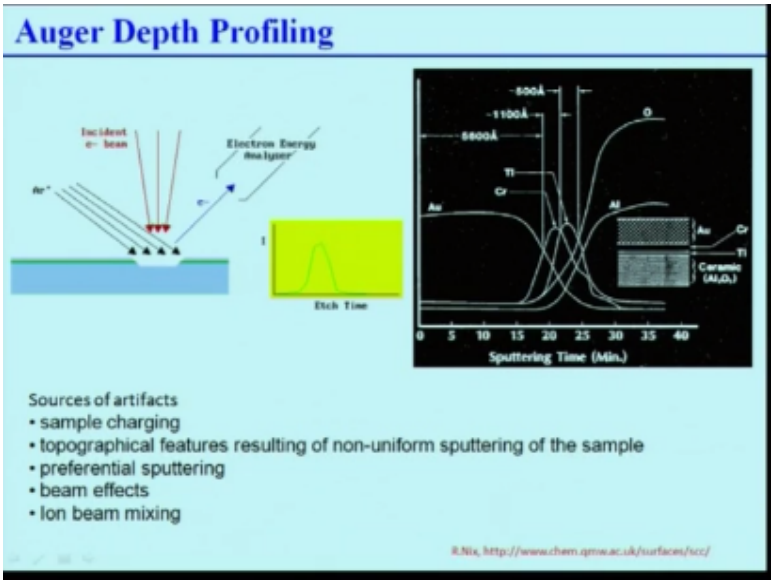

So after giving you an idea of chemical shift chemical detection of different elements are different pieces as well as quantification let us go to depth profiling audio definitely well what is auger profiling actually as you know you have this is a sample suppose and you have instant electron beam coming into picture, and then you generate auger electrons it goes to the electron energy analyzer and then you get the peaks many times actually you can routinely get this fixed now we can actually use argon ions to sputter the sample surface.

Sequencing that means if I have a sample initiating like this is taken from this website anyway then I take information at the beginning from this much area get the auger spectra then using argon ion I remove small thickness very small thickness sputter out of the order of tens of 10to 20 micro as am strong and then again collect the auger spectrum that is how I keep on doing it so that means slowly I increase the depth or socially increase the depth of sputtering and collect the auger spectrum.

And if you do that than one can actually do the profiling that means one can actually determine the elements present at each depth and also quantified different elements presence, so this is one plot which is showing here this is parting time versus you know intensity as you see here aluminum concentration gold constant decrease alumina say increases oxygen increases titanium

so you have chromium titanium gold aluminum oxygen, okay this is basically a gold layer on the top of aluminum oxide of the bottom and then have a inter layer between the coal and these aluminum oxide.

The inter layer consists of chromium and titanium and that is what you see in the Intel this concentration of the curve titanium is very high in the intermediate zone gold consolation was high at the beginning that is from 0 to about 20minutes you have a large coal concentration then it decreases and finally once you reach the aluminum layer alumina oxide layer of aluminum and oxygen, so what does it mean this is a very important aspect which you must know this is actually required in material science that means that.

This is a this very sensitive technique this can be used even do such a fine scale depth profiling by using auger sputtering well, it looks very simple and very you know genuine and very interesting method, but it has lot of problems it can lead to different artifacts what it can lead to sample charging because of the in or sponsoring it can leave topographical features from non inverting the sample it can lead to even preferences sputtering depends on the element present some elements is parsley sample power less easily.

Then there are beam effects like auger ion beams and you can also lead to most notably I and be mixing because you are putting our and auger ions that can lead to mixing up to different elements or two different pieces and that can modify the results, so one must remember these are the problems in auger spectrum filing otherwise this is a very nice technique.

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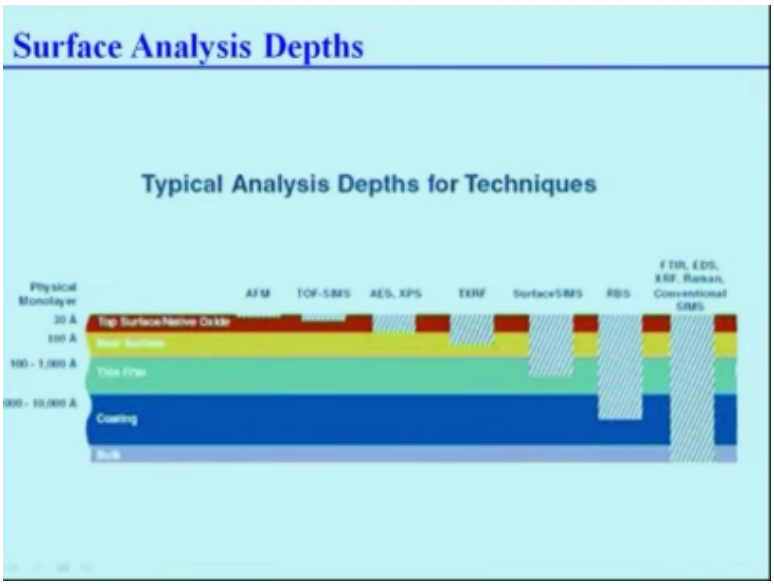

Well this is not clear to you visible, but just to show you that FM actually leads to very small thickness on the top surface auger is approximately 100 micron so auger this is or auger AS it is over 100 micron depth analysis can be done, others like experience can go up to very high RBS can go up to even mere couple of thousands of micron Armstrong and if TR things can go up to even micron level which I will discuss in detail when I compare these different techniques.

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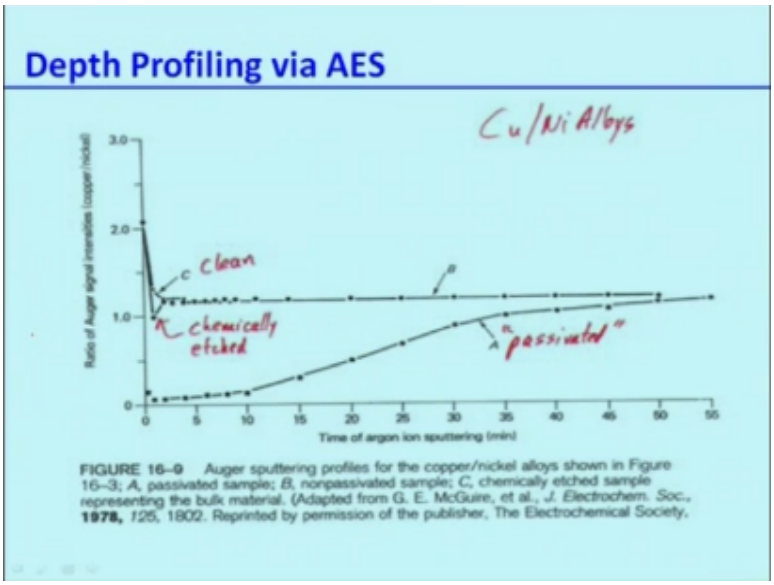

FIGURE 16–9 Auger sputtering profiles for the copper/nickel alloys shown in Figure 16–3; *A*, passivated sample; *B*, nonpassivated sample; *C*, chemically etched sample representing the bulk material. (Adapted from G. E. McGuire, et al., *J. Electrochem. Soc.*, **1978,** *125*, 1802. Reprinted by permission of the publisher, The Electrochemical Society.

So to give you much better idea this is another example sputtering and non sputtering copper and nickel aluminum alloys peasants and as you see here copper and nickel alloy actually not aluminum copper nickel alloys as you see here this is the tip of filing of a passive fitted layer this is the clean sample the clean sample is giving much better results signals then the passivity means it might have got oxidized surface layer, so you sputtering profiles for the copper nickel alloys is basically taken from Mac at all it is tells us that it is always better to clean the sample surface by sputtering and before taking even the profiles.

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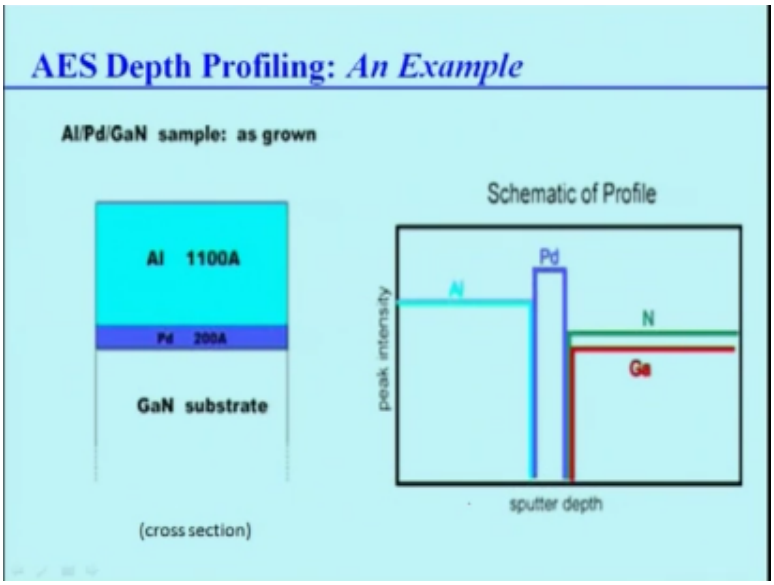

Another example this is a gallium substrate with a PD interlayer and there is a aluminum layer this is a 200 micron 200m PD layer and 11 100 Mike am strong aluminum layer this is basically a multi-layer as grown, no one can actually do this in a schematic profile or profile and that control this is the aluminum death PG depth and gallium nickel you can see the quality of data one can get very good call data one can get using this is another example i think this is pd germanium oxygen aluminum many things that presents this is part time versus atomic concentration.

This is aluminum is oxygen this is pd this is gallium this is nitrogen one can carefully do that same sample actually and then if an elite it gets changed I can see that the oxygen actually propel is remain same palladium profile has got changed where is palimony has moved into our inside the aluminum and on the nickel gallium remains same, so one can actually do this kind of a lice is also in the actual sense.

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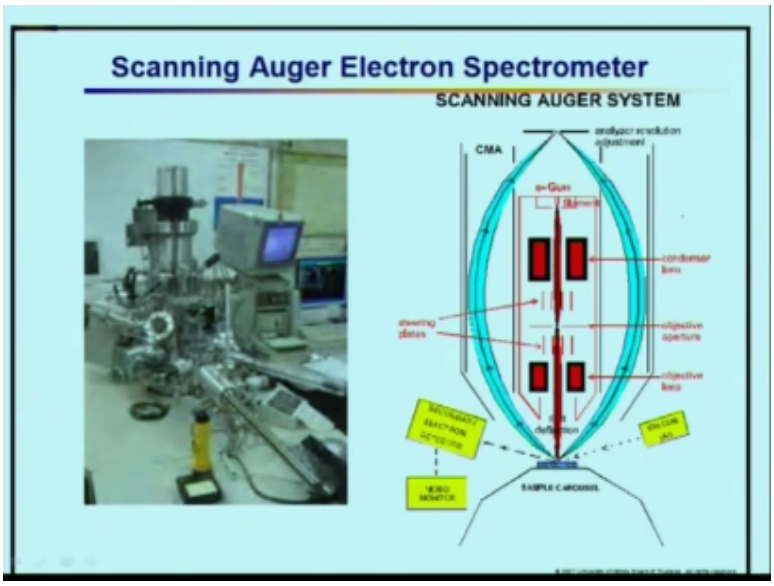

The last technique which is useful which I am going to discuss is the scanning auger electron microscopic still are in your spectrum you can actually scan the surface and do that what do you do is basically you can use the same technology as use in ACM and your beam which is coming and electron which is coming and following the sample surface can be allowed to raster on the sample surface or can be allowed to basically scan the sample surface, so as you scan the sample surface you can actually gather information from each point on the sample surface due to interaction of the electron beams with a sample surface.

And then generate an image instead of depth profiling you cannot actually an image so details is soon here what do you see here this is scanning coil this is electronic filaments this is objective lens, so this is the conditional lens so you were scanning coil it will scan the beam cannons call see nothing but basically same as an electron scan auger microscope then you have a second electron detector here this ion detector net here which is part the sample scare-ousel, so and the actual pike which is shown here this is taken from inversity noise they have a facility like this.

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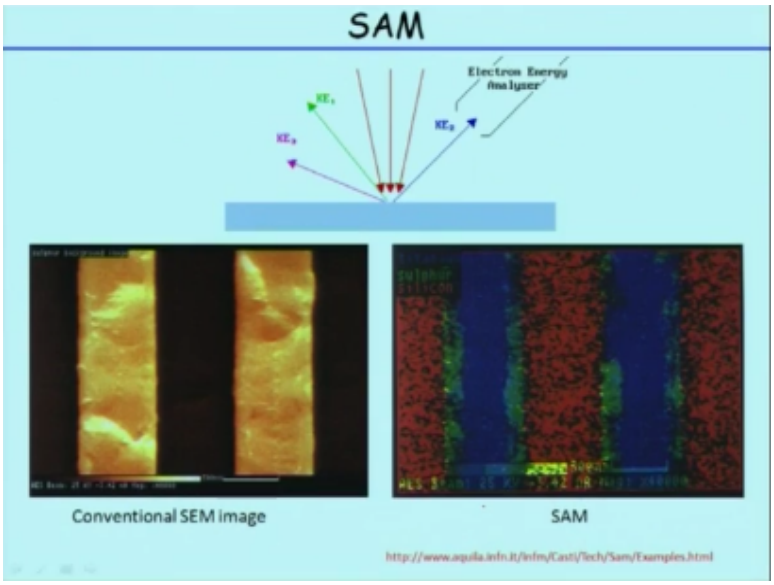

So what is done here let me describe the schematic diagram this is the electron beam falling on the sample surface, so you have basically generating KE1 KE2 KE3 two different kinds of you know transitions object energies electronic different energies this can be detected because the presence of different elements actually when you scan and then you can image this female actually is I do not know this is the blue is basically titanium this is taken from this website not from my work.

So they are pictures the sulfur is basically the Green is basically for sulfur and red is basically for silicon as you can see this is the cross-sectional conventionally same image which does not give much data it is only, so there are different areas sample surface red and the black this has been basically made color I think this is sulfur backs and Aga sulfa back scrimmage, so this is basically sulfur this yells so as you can see a sulfa is green and in the AESS am of the scanning RGS microscopy you can see these red regions are basically coming from silicon.

These are all silicon okay this division silicon and then you have sulfur and the titanium sitting so the blue here this much smaller is blue and this is this is this again sorry blue has basically titanium green is basically for sulfur, so can electron microscope shows a large area of sulfur auger microsphere source there is a thin region of sulfur between silicon and the titanium that is the difference actually one can clearly see when you use scanning or a microscopy which is much more sensitive than the EDS analysis electron you know energies perspective scandalous is in the scanning electron microscope.

Well last thing which I am going to discuss is the auger setup or how the auger letter on my garage is electron spectroscopy is done.

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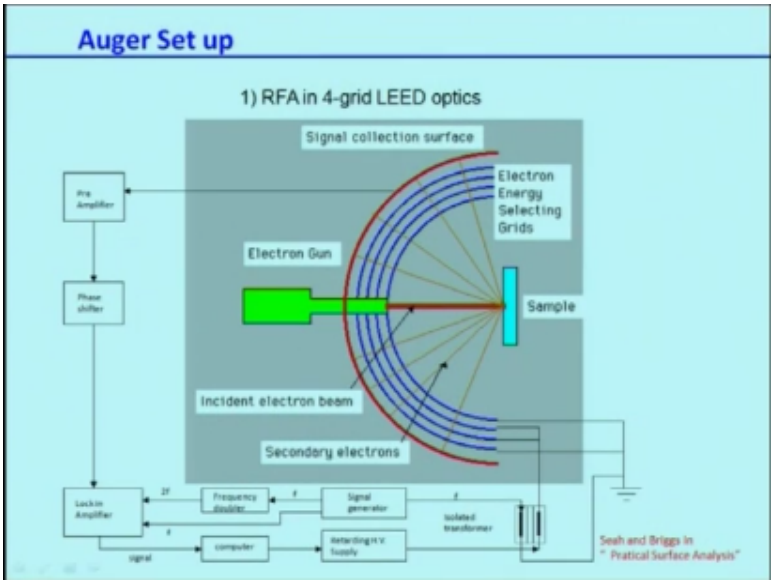

Yes so as you see here the schematic diagram here which is quite complex this is basically forged LED optics setup which is used to detect data this is sample and this is the electron gun select on crumbs from the gun and for the sample and then you have basically secondary electrons on at 80gr electrons generated, so one needs to detect them and that is why forget LED optics is you this signal this one is fed to P amplifier then there is a phase shifter then there is a lock amplifier and then it goes through different kinds of things finally you just detect the signal in a computer.

These are basically electronics used to modify not to modify to actually improve the signal to noise issue and this is the zoom it that means it basically is a kind of a hemispherical geometry is used to detect all the electrons present to him while, we cannot actually throw out fully the secondary electrons and that remains in the auger spectroscopy could be always, so silly clinical mirror analyzer is basically use the same thing with the electron gun.

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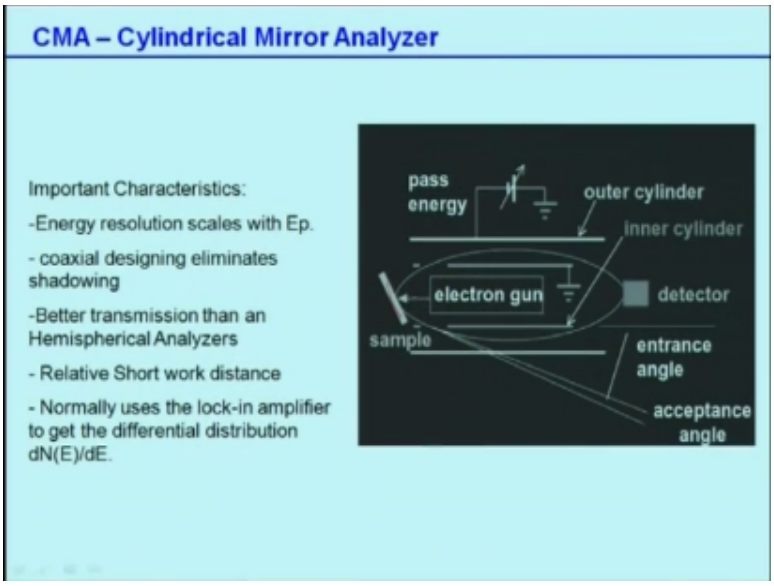

So it falls on a sample the sample isolate hang up for samples and they say you apply pass energy and then you can actually remove the secondary electrons to that so energy solution scale up to EP and then you have you have a coaxial design quick statement like this you can actually limits adding effect you can have better transmission relative short distance nor my lock in inter plus gate differential distribution very deeply let is a DN / D that is what I you plot DNE / DE is plotted nausea, so this is why you need to use lock-in amplifiers.

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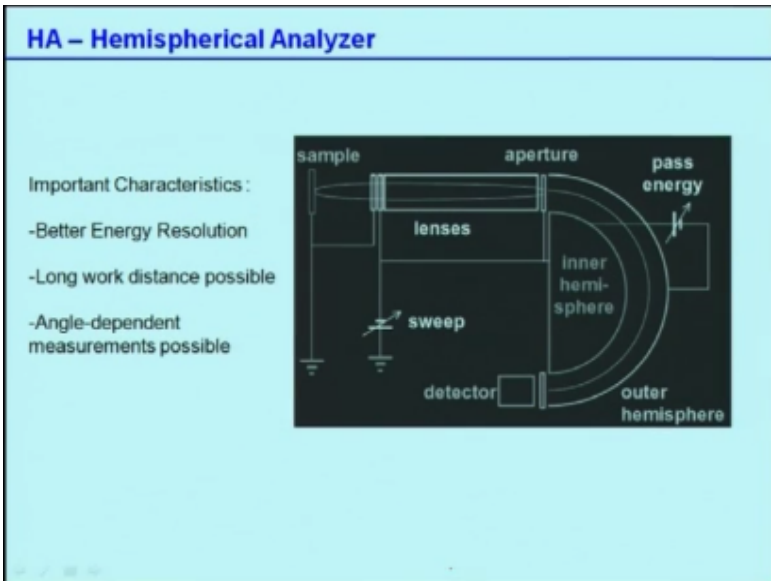

You can always use hemispherical analyzers what I have discussed the beginning this is the inner historical and energy and is aperture the sample electronic falls and then it is false goes to the semi-spherical detector and detected.

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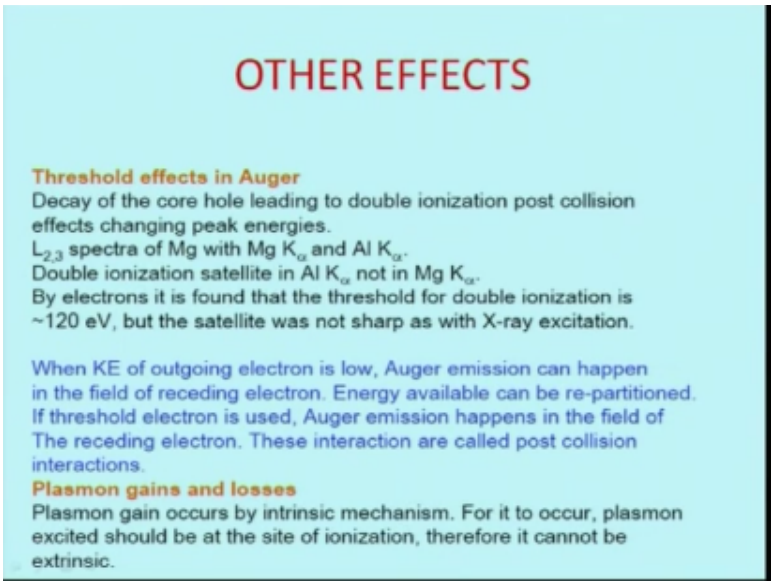

Well I think that is all resolve things you cannot this will be if i get time will discuss otherwise next to lecture I am going to start the secondary ion mass spectroscopy which is the last surface spectroscopic advance a prospectus with technique in this course and then i am going to compare the these three techniques and wind up this particular portion of the syllabus.

Acknowledgement
Ministry of Human Resources & Development

Prof. Phaguni Gupta
Co-ordinator, NPTEL IIT Kanpur

Prof. Satyaki Roy
Co Co-ordinator, NPTEL IIT Kanpur

Camera
Ram Chandra
Dilip Tripathi
Padam Shukla
Manoj Shrivastava
Sanjay Mishra

Editing
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Badal Pradhan
Tapobrata Das
Shubham Rawat
Shikha Gupta
Pradeep Kumar

K.K Mishra
Jai Singh
Sweety Kanaujia
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Sweta
Preeti Sachan
Ashutosh Gairola
Dilip Katiyar
Ashutosh Kumar

Light & Sound
Sharwan
Hari Ram

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Priyanka Singh

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an IIT Kanpur Production