

**Indian Institute of Technology
Kanpur**

**NP-TEL
National Programme
on
Technology Enhance Learning**

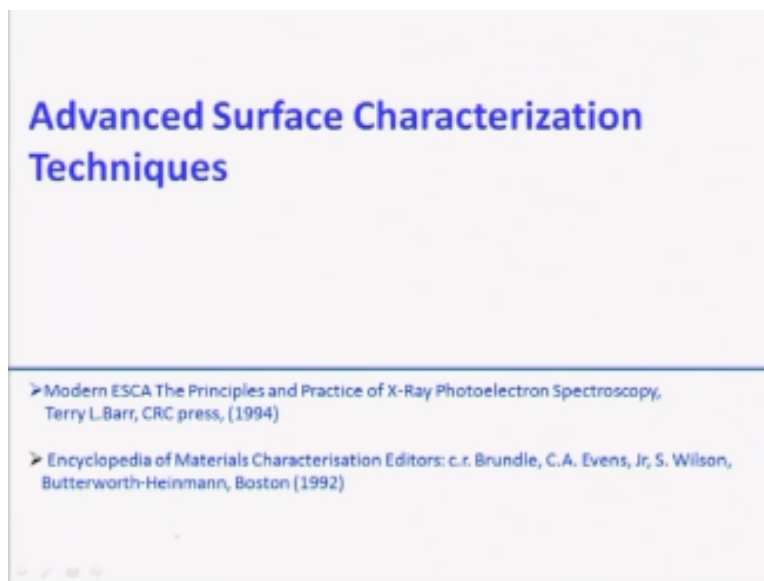
**Course Title
Advanced Characterization Techniques**

Lecture-19

**by...
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So welcome to this course again. So we are going to start new topics today. It is actually on surface characterization techniques.

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As we know there are many surfaced characterization techniques and in this course we are going to talk about some of the advanced surface characterization techniques. Simple surface characterization techniques involves measurement of composition of the surface of any material or even measurement of the oxidation oxygen concentration another concentration which are

present on any surface of the material. In case of materials science they are very important because we know most of the things start happening at the surface.

So therefore surface needs to be fully characterized not even that the best example of surface effect on the material world is in catalysis. As we know the catalytic reactions take place at the surface. So therefore the surface structure surface composition and the atomic electronic state of the atoms from the surface give play a tremendous role in catalysis. So therefore the surface is studied extensively and because of advent of new techniques surface characterization has become the important.

In this about nine lectures which I am going to take obviously we cannot discuss everything of surface characterization we have to limit ourselves because with time constant and I am going to discuss several important aspects, but before that let me just give you the reference books the first reference book is very important it is called ESCA modern ESCA principles and practice of x-ray photoelectron spectroscopy.

This is by Terry L. Barr, who is published by CRC press and this one the next one is encyclopedia materials characterization by editors by C.R. Brundle, C.A. Evans, Jr. S. Wilson, Butterworth Heinemann, Boston is also very important book where you can get all the information's. There many other books available in the literature which you can refer or even there is a lot of information is on internet. So we can also look at it.

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Outline

- Importance of surface characterization techniques
- Advanced Surface Characterization Techniques: XPS, AES and SIMS
- Comparison surface analysis techniques

So the way outline of this particular chapter of particular position the course is made like this first I am going to talk that is today I am going to talk about in importance of the surface characterization techniques why do we need surface characterization techniques. This is what I am going to talk today and then I will talk about other techniques which involved first one is the x-ray photoelectron spectroscopy. Second one is RGR electron spectroscopy and the last one is called secondary Ion mass spectroscopy.

So XPS is what is called x-ray photo electron spectroscopy AES what is called RGR electron spectroscopy and SIMS what is called secondary on mass spectroscopy. These three techniques will be detailed in this course. Finally I am going to compare the surface analysis technique so that you can get an idea which techniques have advantage and certain aspects over the others.

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Cu-Pb alloys

Used for bearing applications because of its anti-friction and self-lubricating properties, which demand retention of finer grains with uniform distribution of Pb.

•Essential requisites for bearing applications :

Must possess a duplex microstructure with homogenous distribution of interconnected softer phase.

-Hard matrix: resists the wear and softer matrix: uniform distribution of load and for lubrication purpose.

Casting leads to inhomogeneous distribution of Pb

Conventional pressure-less sintering: densification occurs at 500-900°C for around 6-10 hours, which leads to extensive grain growth and oxidation during sintering – leading to inferior properties/poor wear resistance.

T. OHASHI and Y. TANAKA, *Mater. Trans. JIM* 32 (1991) 587.
J. BRETT and L. SEIGEL, *Acta Metallurgica*, 11 (1963) 467

Well before I going to the detail of this techniques obviously it is very important to know why do you need to surface characterizations techniques that to advanced. You know when in our labs technique facilities available you start using it extensively, but that is not the way to do we need to understand that we need to regarding the scientific investigation of any kind of activity on material signs. That means whether a technique can be used to diverge scientific information's to understand certain phenomena.

That is why actually the importance and applicability of particular sign technique exponent technique comes into picture and one need to really understand it as a course goes on probably or getting and feeling that there are lot of techniques we are talking about it why which technique we need to use for a what kind of applications is not tell to do it. well that is what actually I am going to plan to do it to the end of the course I will go to give you some examples were a set of techniques are used and the scientific understanding of the phenomena going on in the materials processing and application will be talked about it.

This one I will talk about for copper lead Alice most importantly the copper, lead automata logy Alice which actually I would talked about this is first lecture of his course. You know that this allows a use for bearing application. So therefore there are some aspects from microstructure which we need to have in the applications. So most importantly we need to have a duplex micro structure with a homogeneous distribution of softer face in a hard face or hard matrix and therefore hard matrix will register were and softer matrix will act as a lubricant. In that is why

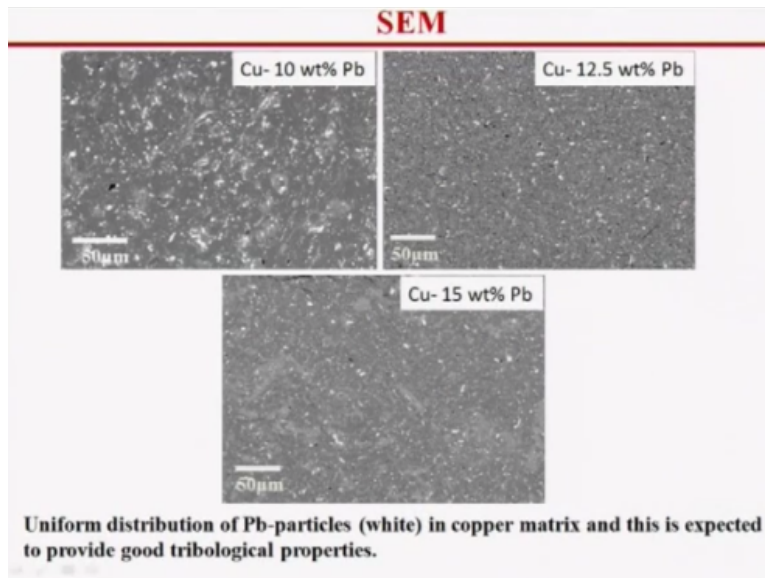
actually used in the bearing applications and most common technique which is used to produce copper, lead Alice costing root, but as you know lead is heavier than copper much heavier than copper.

So if want to do costing lead is segregate and create innovial distribution of lead in the microstructure and aoristic course distribution will happen because of the micro segregation and this is bad so therefore costing techniques cannot be used for preparation of test bearing Alice we need to use some other technique. So then people tread with pardametalogy root were you mixed copper and lead very nicely and then making in palate sintering by differ to seen to normal atmosphere a problem is oxidation first of all because in phase on a scenting those of you know are phase on a scenting you just put inside a furious for a long time on a high temperature 600 to 700 degree temperature.

To center heat to the optimum density about 90, 95 % theoretical density you start increase the copper, lead size and how to start creating the prolong oxidation. So comes a sintering has this problem of extremism can growth and oxidation and this leads to inferior properties. So what the solution not only that not the people of the world lot of things would nana crystal Alice they have excellent properties in for copper is one such material while nana crystalline grains of copper can give very hardness very high strength at the same time it can also you know impart lot of other advantages in bearing Alice.

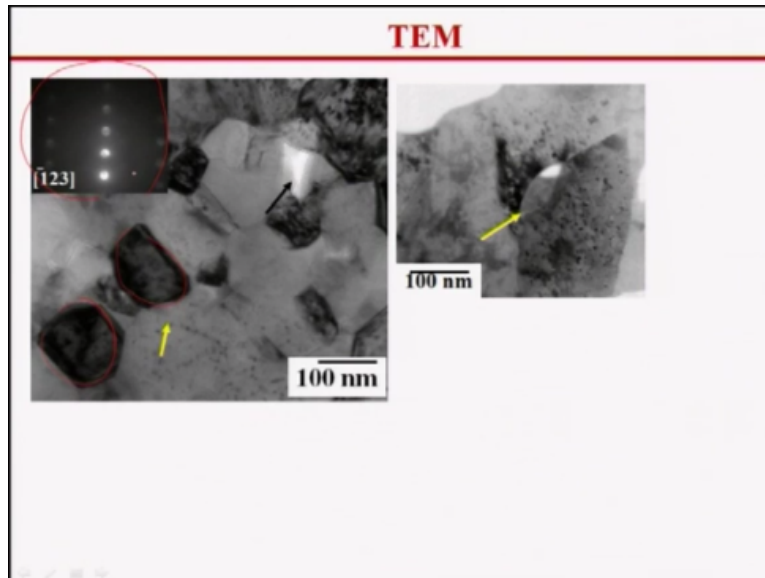
So that is why actually we started looking at nana crystalline copper, lead Alice in for it is our own business activities one of my own have done this work and we have used a phrasal sintering technique called plasma sintering which produce this can of micro structures.

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As you see here this is a copper 10%, 10 % lead, copper 12 % lead and copper 15 % lead. One can go on increase per then what will have happen of the sudden time the lead will be agglumalited distribution copper what is see here is that lead is informal distribution in the micro structure there is a few pores present obviously that is a problem of innig protometallurgy product.

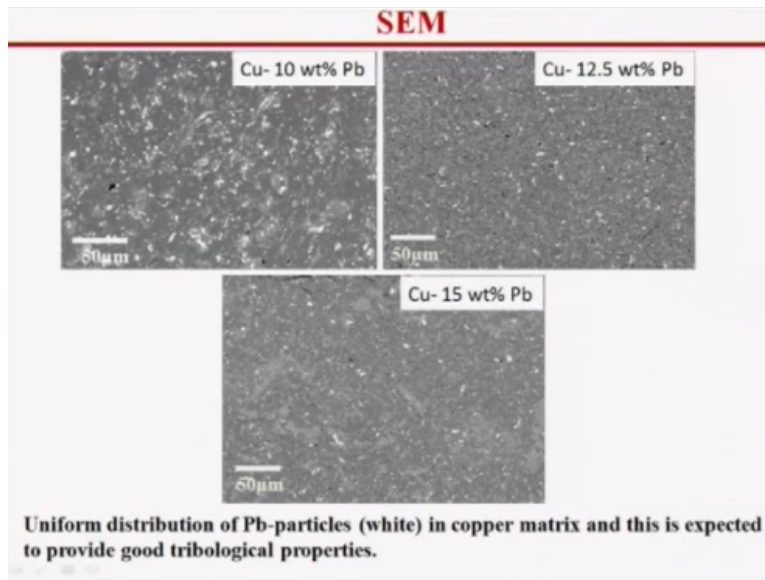
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But to understand whether we are able to produce nano crystalline grains are not written this is TEM picture you can see here this grains actually copper and they are of they are of 80 to 100 nano meters of may be little bit higher nano meters the diffraction pattern is taken from one search copper grain it is a micro diffraction pattern taken from one such copper grain so it is a copper not of side and you have late sitting like this and one as a ventricular lead between the two grains or at the grain junction points 50 lens of points is so at the lead seats.

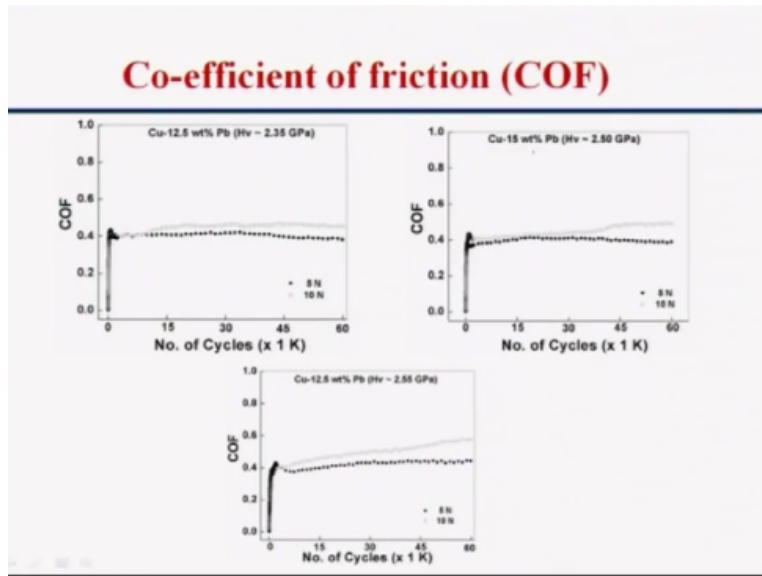
So therefore it is possible to gent retain the nano crystalline copper grains at the same time distribute the lead in a very nice manner and this can a micro structure which is shown here.

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Is a good candidate for study over?

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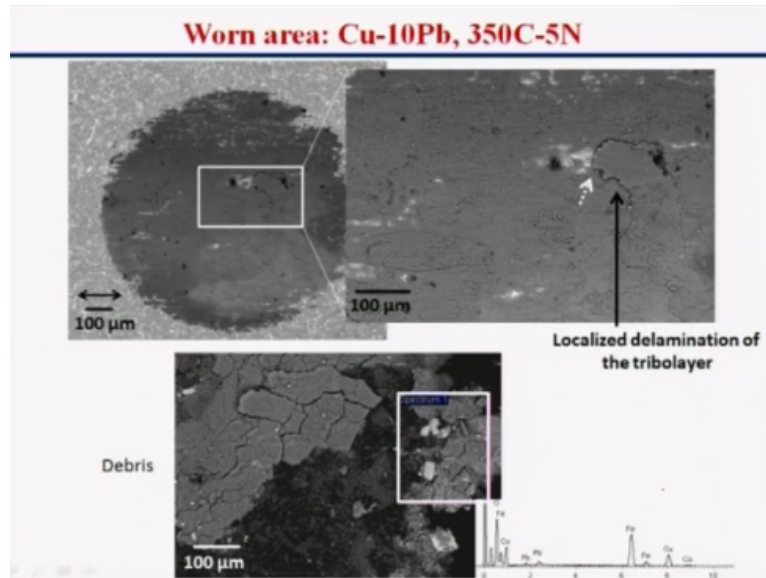


So we need to do that and that is what is done because bearing application means wear in tear so when is to we studied so once you do this to wear for different lead concentration 12.5, 15 this is also 12.5 as you see and know the higher this is 10 sorry this 10, 12.5 and 15. So this is 10 % lead, this is 15 % lead, this is 12.5 % lead. So if you look at the coefficient friction values this is done in fitting wear because fitting is more severe than sliding wear almost of bearing sexually undergo kind of fitting wear.

So that so you want to do it in a normal atmosphere. That is what actually they are applied and filled this you could see the coefficient friction for fitting cycles up to 1000 it is remains constant from most of the cases close of 0.4. This is again steel wall that this wear test has be done. So the fretting ball is steel as you know the co frictional friction between component and as to the sting as actually against this actually 0.78, 0.8 almost close to.

So therefore we are able to reduce the co frictional friction extensively. That is the biggest advantage of line lead, lead acts as a lubricant and as reduces this so, but what does actually happen in the oxidation or ware phenomenon has not cleared now. We will only see the friction of coefficient.

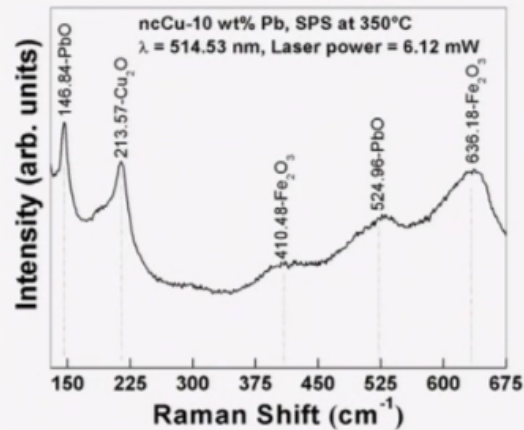
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Now if you look at wear surface then something things become clear. Here I am saying you these off to scanning electron microscope as you see here this is the own area you can see a lot of cracks, facials and you can even see the lamination permission of tabular and if you were take the were Debary you could see the oxygen present lattice present there you can see a white color and copper is present in a delaminated form. So that is what you have seen this is when the centre 350 and load was 55 Newton. Now it does not even tell you what is happen in the ware process.

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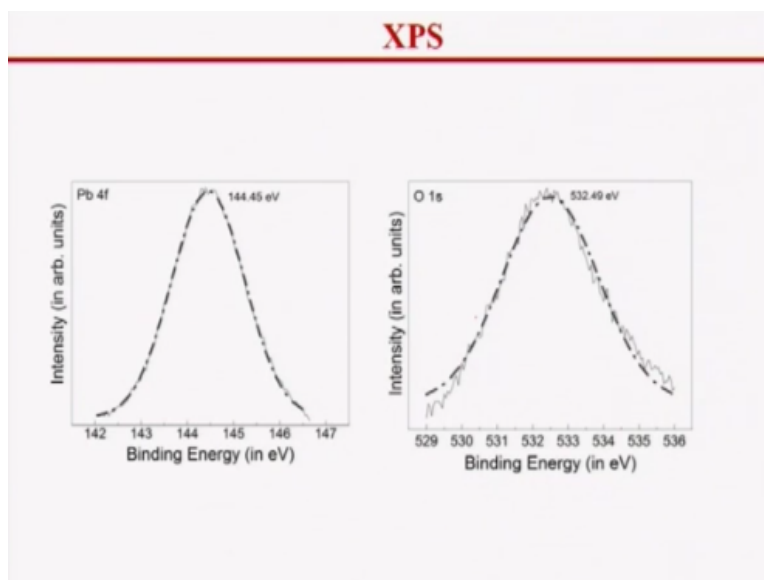
Raman Spectroscopy



If you do Raman spectroscopy that is why actually spectroscopic techniques comes in to picture I have shown you Raman spectroscopy discuss Raman spectroscopy if you Raman spectroscopy status saying lot of oxides presents and this scales with the actually the was called state of electron lectured atoms you have seen iron oxide, copper oxide, iron is getting from steel ball lead oxides.

Now lead has different kind of oxides pbo, pb3o4 were lead although it gives you very nice signals of very broad signals of Raman spectroscopy of the oxides were that may not be suppressing and of to really pin point what kind of lead oxide spread that is what is this electronic state of the lead and oxygen is not clear that can only be done if you use XPS that is x-ray photoelectron spectroscopy and that is done here.

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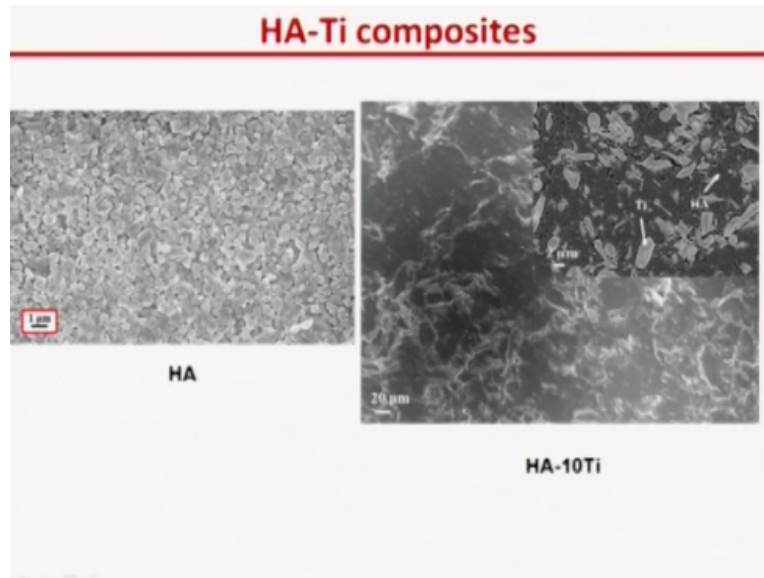
As you can see a very clearly this is the state of the lead of the onset particles on the own surface and this is the energy level binding energy the vesicle express is problem between the intensity and binding energy. This can be easily fitted with lead 4f state and in fact I am not going to talk it can be even done better so which I will discuss detail I will do XPS analysis, XPS discussion and oxygen is 1 instead.

So therefore it is pbo or not pbo₃o₄. That does can be conforming so one is actually use this technique to make the understanding of the whole process very clear. That is what I has been done to understand they were mechanism of this nana crystalline copper, lead Alice we need to use this spectroscopic techniques both surface characterization, surface advanced characterization techniques and how so bulk to know what is happening they are.

So it is the oxidative were which the copper, lead Alice is due to expulsing when you use in normal Alice equation. Now detail of this work publish now one can look a leadership from all group, but obviously oxidation means a host of oxides lead oxide, copper oxides they are very hard. So that what is actual in effect of that of this oxidize oxides on you were way is very interesting thing.

So you understand that if I use these oxides and the on peroxides appearing a ideal loose, but they were hard so they will basically a bread the counter interface more easily.

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That is the one example, second example is again from my out of work one of my PHD student work is have titanium composites which are used for biological bio medical applications basically for orthopedic applications happens for your information's high is the constant of the bone the inorganic constants of the bone or therefore most of biomedical applications requires development of new materials one such of the material which is important for both dental and bone application is hide as appetite best.

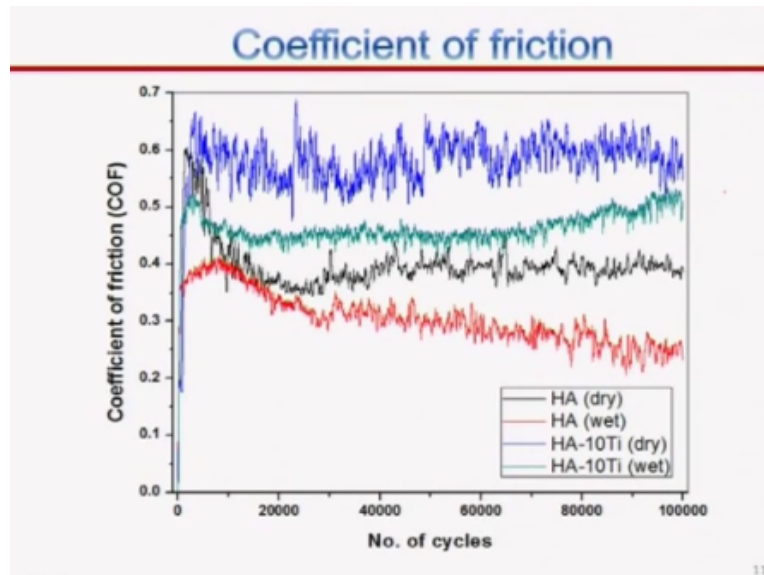
And hide is appetite is a normal material has since that it allows is basically bio compactable and bio active that means ones is goes in our body it allows the cells to grown on it and helps thus various process and many other things. So, but there is a problem high pitiable the structure point for K_{Ic} is basically $0.5 \text{ MPa} \sqrt{\text{meter}}$ is very low. Normal bone has a fracture term has between 2 to 12 cortical bones actually by $\text{MPa} \sqrt{\text{meter}}$. So it is very much lower than what is to be expected.

So there is always a research activity to improve this fracture term. So mechanical wave properties so that it can be used for load bearing application like bones or even teeth because in your teeth you are applying lot of forces to chew a food. So what to do you add a medal and you can add all medals so people had titanium. Titanium is a biocompatible, but by you not and it is having high cosine resistance as you know.

So that titanium can distributed like this you have shown these are the titanium and attributed grains so once you put this hydroxyapatite titanium composites inside a body it comes in contact

the body fluid and as you go on doing on the daily activities it undergoes volunteer. So one is to understand what happens to what happens to where they wear on the such conditions.

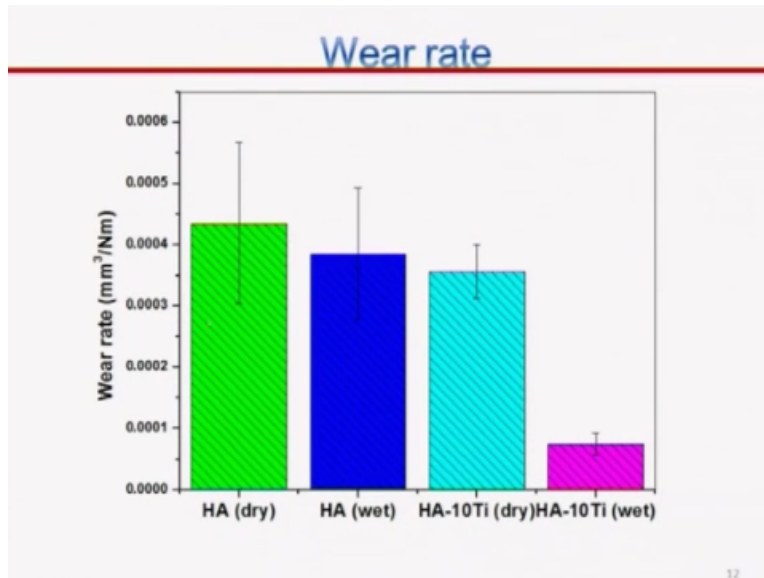
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So what people do normally is to study the wear way we are in simulated body fluids. Simulated body fluids have almost similar composition that of the body fluid and then we do this fitting were they are as then see here that under different conditions wet and dry pure adisepitide and adisepitide 10 TI the coefficient friction waves so given. This is again problems literature it is under problications so thus no problem so as you then see or if you use wet conditions you have lower value of coefficient frictions basically we add certain proteins also here albumin to you know create more like to a situation when in first physiological situations.

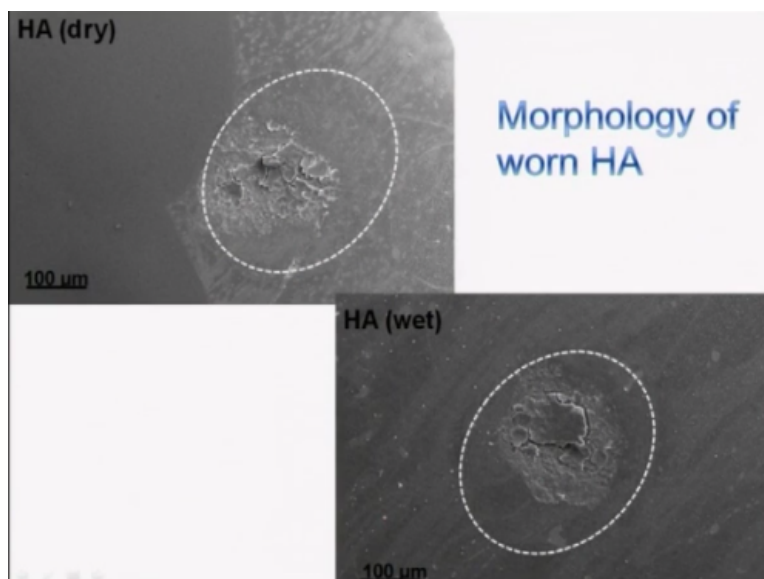
And information fixation values ware is from 0.3 to 0.6 depending on the material depending on the kind of to know way or dry conditions.

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And if you look at the wear rate it is also pretty low you can see here they wear it dry and wet condition is reduced substantially. So that means this materials so it will be able to do better job inside the body, but what actually the mechanism were that is what one is to understand.

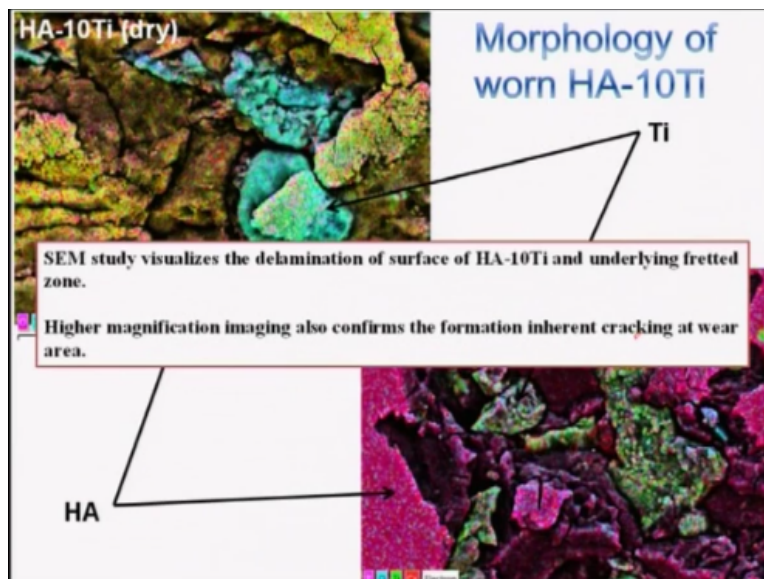
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So if you look at this again morphology bones surfaces for pure HA dry and pure is your wet and so you can see the laminated layers and breakage of that. Not only that if you look at morphology more closely you can see this. So that you can see that appetite crystal forming on the surface and they help in reducing the wear. If you look at 10 Ti you can see even the laminated layers, but you also see the small particles present or then titanium is that titanium big ones small particles presents their okay.

They are actually appetite crystals which forms when this material comes in contact with the simulated body full its fluids and they are good because their appetite they are good for her body they are good for many things. So they actually starts interacting with it with the, own in a counter body and this for actually were deep exchanges to understand a more.

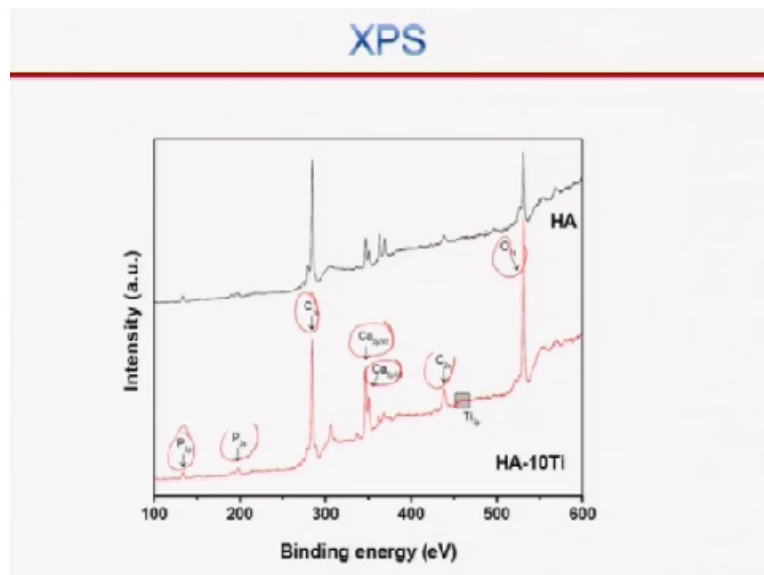
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We have done actually that is morphology of wear this is actually E - mapping I have already showed you imagine this spectroscopy mapping is possible if you map it these are the titanium's and these are the actually ideas sexpartite, but you start you can see this can the crystals which

are basically a norm appetite crystals which found because of the interaction with the simulated body fluids. So we can see the formation of you know appetite crystals also in underlined.

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Now once you take this can a material and do what do I say at the XPS after were so own surface who started seeing this calcium two states which we are a signature of two different compounds. So one is hydroxyapatite other one is appetite which is present. You were also see the carbon pix carbonyl present because your, were lot of solutions then phosphorous and 2s and phosphorous 2p pics oxygen is 1 appending presents.

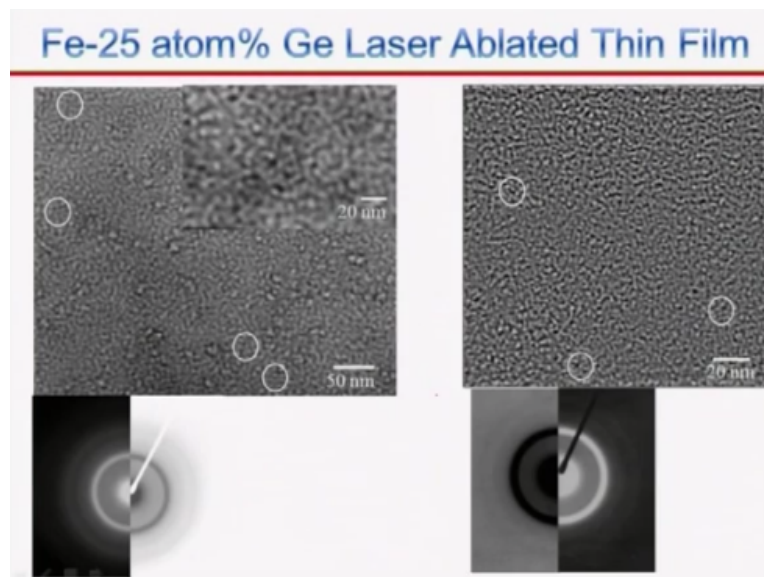
Okay you actually see a titanium pix so therefore the presents of appetite crystals are very clear by dying this XPS is I have been telling you this crystal actually formed in this process ware changes the mechanism much it because it is forming a layer on surface of the material and then

ion contract between the counter body on the surface is no longer they are new contactable of and that is of where we with changes.

So using these kinds of techniques what can really understand the actual inside actual side actual scientific phenomena actually one can go into the details of the scientific phenomena which is there. These are normally that done routinely so, but is really want to do scientific understanding of any process that is what actually you need to understand the characteristic techniques you need to use it.

Many labs actually have an ID compression also were to get XPS very soon and is available routinely so one can do that.

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Well the third example is I am going to give is from my own PhD work that is on adding germanium 25 percent pattern percent germanium lesser thin films. You know thin films are actually routinely used many applications. We wanted to understand if I take alien 25 % germanium alloy and form a thin films by liver ablation which is very well known technique what kind of structure develop.

As you see here these are all very happy high reunion pictures. Thermo electron pictures, but this are actually I am digging all this things I only taught you high resolution electron microscopy, were as spectroscopic techniques and I want to bring them together and make you that this you

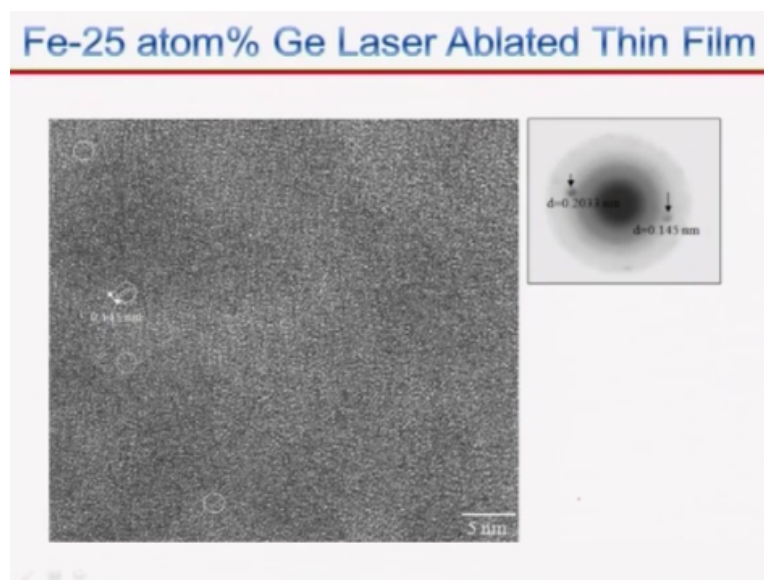
know why I we need to use surface characterization techniques to that if for the scientific insides of this processes, of this structures.

As you can clearly see we see clusters. This kind of clusters will over the clusters inside this thin films when they are as the prosaic conditions. Well detailed conditions all in the available in the literature you can search. But not the issue what you see as this one also they are atoms remember they are the cluster of some atoms. That is it is very important to note and if you do the diffraction analysis you see board rings signature of among first structure.

That is what happens if you take any kind of alloy started doing other oblations. Most of the cases you started forming in normal fast 10 films. Now this thin film which deposit on the surface then it is to be double and has the function temperature also as the function of in a compositions then all they can be used for applications because real application temperature increase so what the people need to study if we can actually the thermal response on this thin films.

And that is very important since that you know our fast structures are normally concerned to be non stable. So they are actually prone to get task form into crystalline forms. So to do that actually this is in a better high lesion picture.

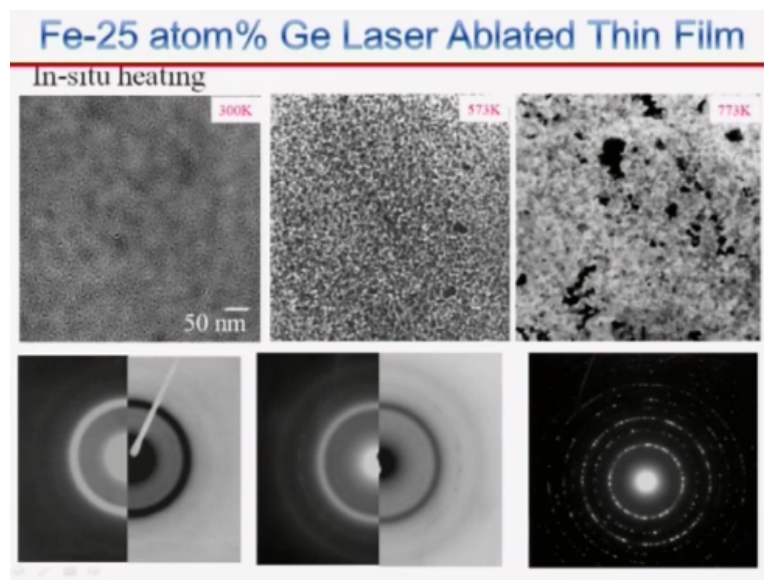
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And in fact one can actually do this using a modern electron microscope TMS and you can see a some kind of this kind of class test present they are I do know whether you can see you will be able to see them and this class test actually are you know you can take actually nano diffraction pattern that means we can take a nano beam and put it they are and granite diffraction pattern that is what is done here if you do that we started seeing diffraction spots here you can see and this diffraction part D values are written.

If you look at there are very close to RN. This is the RN. Okay 1145 and Armstrong is basically close to 200 line spacing of BCCRN and 2.003 is basically 110. So that means we can actually see this class test actually PURN. They are not actually PURN was some solution between BCC RN we germanium is they are. So things will be very clear once is started showing this pictures.

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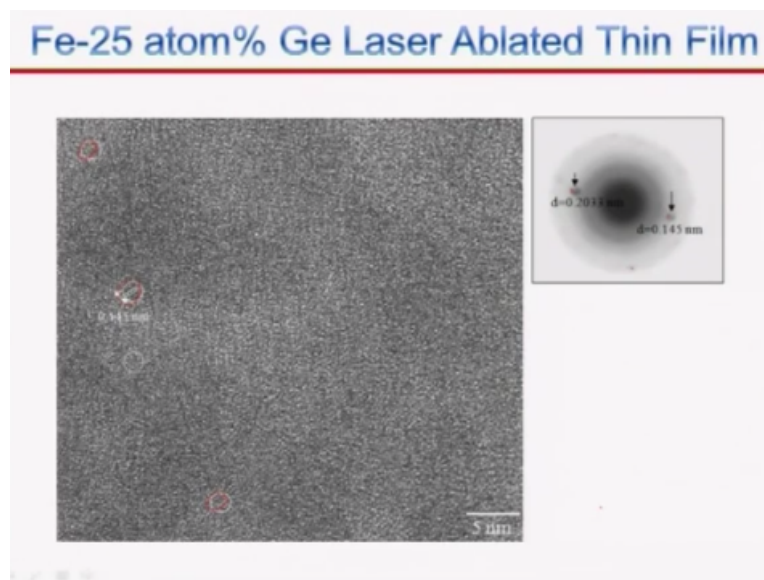
This actually in situ heating microscopic this thin films if I put inside a heating holder that has been electron microscope and then he did enter the microscope and observe it during the heating. That is what is actually done here so you take this 10 films and then heat it up once heat it up inside the microscope crystalline changes. So that is what you see here you see this is getting change into these this and finally from 300 to 770K you started forming crystalline things and you can see from the diffraction pattern itself.

Actually started forming this thin it is black region the crystals signifying this ring weak ring in the diffraction pattern and is fully getting this from at a 7 time think here. If you do it careful

diffraction analysis okay if we had a good electron microscope if it can do that and you have to also have good electron microscopes then you can see these grains which are they are actually ordered they are due to the ordered structure that is not getting into that and they are B2 domains and also due to 34 movements. This is B2 this is due to 3 domains presents.

So that means they have a high temperature at solid state and therefore you can see this to ordered domains. Now first of all that means we are started forming when you heat treat we crystallize BCC Fe. BCC as a solution of germanium in Fe and that is case ordered later on to form B2 and further ordered Fe to due to structure. So the nucleating phase is basically BCC solid solution of higher why does it happen. Thus the thin film has these clusters presents which I am indicating you will diffraction analysis do suggest.

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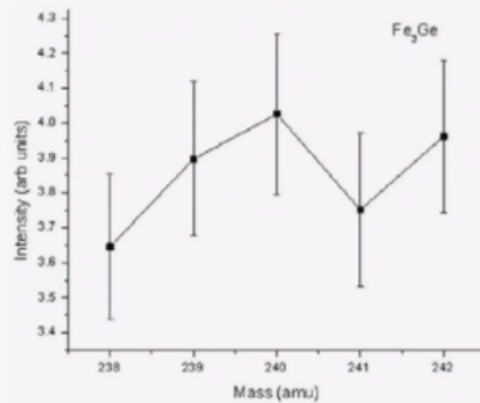


So this kind of clusters presents, but no conclusion is evidence it is all in a kind of one spot coming and we are saying that. So what you can do we can actually do a mass spectroscopy.

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Fe-25 atom% Ge Laser Ablated Thin Film

Mass spectroscopy

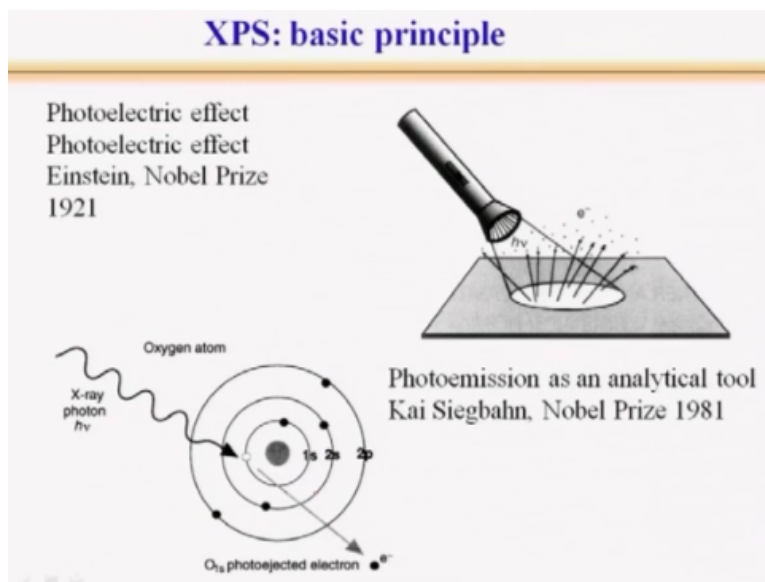


Of this in a clusters during the deposition with in film that is called inflat mass spectroscopy and this inflat mass spectroscopy will tell us what kind as mass thus present. This is that in, but intensity vs. mass in at in mass unit. Okay you would see large error parts that is okay for if you this see integral the most intense peak is 240 thus correspond so that FeGe and of clusters which have present they are. So this conforms actually our thought process are experimental by log thought whatever happening the experiments whatever you are thinking whatever contemplating it proves and that self publishing this paper also.

So I am just train you that by using this surface characterization techniques are the techniques which you allow to study the surfaces is basically giving us scientific inside into the problems. There are host of example one can talk actually I can keep on talking about it that is not the way because the time limitations of that also so I am just going to show you few examples and these are the things which you will do in your research activities were need to understand the scientific process happening.

So can you can actually bring up out you know process control later on and that is why these are used.

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Now let me just get into sound these things in the today's class. You know this basically the most important techniques surface characteristic is called XPS. X-ray photo electron spectroscopy. It transforms and Einstein discovery in 1921. You got Nobel Prize for that a photo electric effect. What is it? Well this is what you showed in this picture here if the x-ray photons have. Okay x-ray photon has energy $h\nu$ ν is the frequency h this plank constant. It falls allow to fall on oxygen add on.

What is going to do it? Otherwise high energy is going to knock out and electron from in a cell one is cell. One you sell a two electrons is given a knock out one electron and this electron comes out. Now this electron has a specific energy obviously it has been knocked out from the cell because the x-rays which has follow to it has high energy higher than the binding on to the electron.

Others it cannot knock out. So it has been knocked out and also it has been given certain character energy. So $h\nu$ must be equal to the binding energy of the electron plus the kinetic energy. Right that is what is actually the energy balance so now if I know the kinetic energy obviously I know $h\nu$ because I know what the wave length of the x radiation is. So I can calculate the binding energy and as you know the binding energy of an electron in any atom is very fixed.

It will vary from one element to other element. If it is a nitrogen binding energy of 1 is electron we different from the oxygen. So by using the by measuring the binding energy I can exactly say

what kind of element dispersion what is state electronic state of electron whether this is coming from 1 S cell 2 S cell or 2 P cell I can tell that also. Not only have that where evened the spine of the electrons which present. That does why it is $2P_{1/3}$, $2P_{1/2}$, and $2P_{3/2}$ one can tell what kind of spin it has.

So this is what actually is a major discovery done by carries seg 1 in 1982 one he got Nobel Prize for this. You must not forget. So the concept of Einstein for photo electrons has been used by sig 1 for a basic principle and which I have to go to discuss in detail about that.

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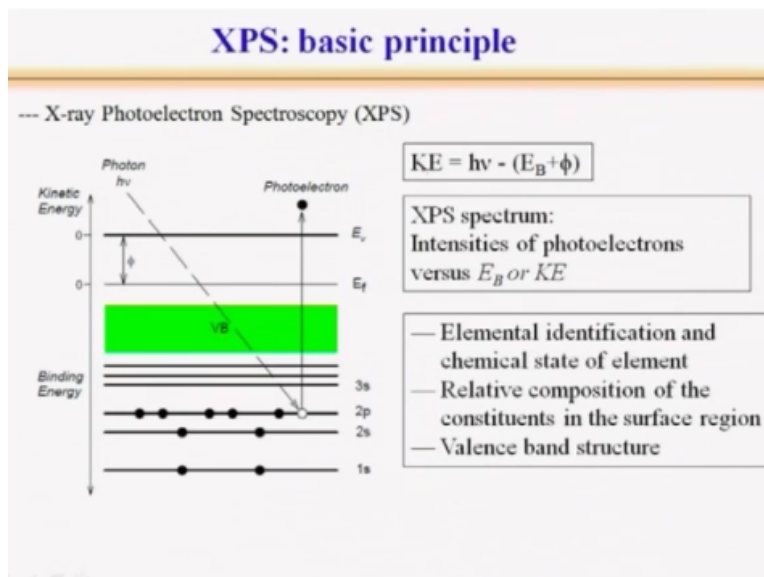
XPS, also known as ESCA, is the most widely used surface analysis technique because of its relative simplicity in use and data interpretation.

| | |
|-------------|---|
| XPS | X-ray Photoelectron Spectroscopy |
| ESCA | Electron Spectroscopy for Chemical Analysis |
| UPS | Ultraviolet Photoelectron Spectroscopy |
| PES | Photoemission Spectroscopy |

So XPS what you known as ESCA is now the most wildly use surface analysis technique because of his relieve simplicity and use in a data interpretation I only discuss what it there are various versions of SPS. First one is XPS. Second one is ESCA electron spectroscopy for chemical analysis. The principles are same. Third one is ultra violet photo electron spectroscopy instead of x-ray you can use ultraviolet ray and last one is called PES photo emission spectroscopy. You can use a light to emit electron.

That is what photo emission. So there would a light is nothing, but lesser beam helium lesser can be used to or any other laser actually which wide length comes in the light visible range can be used. So there are host of techniques the most important ones are these two which are going to discuss in detail.

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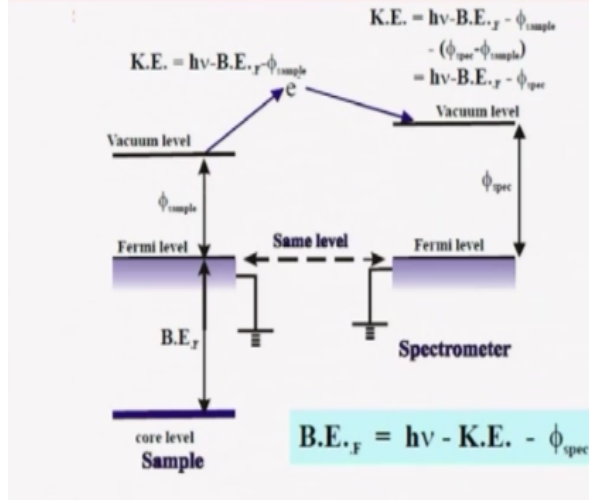
Well again to tell you details of that okay because was starting off as I discuss this is what going to have kinetic of the electron is to be $h\nu - E_B + \phi$ ϕ is the work function well to modify so therefore this is the suppose the binding energy this is 1s, 2s, 2p, 3s electrons and support a photon grams in terms of x-ray or it norms of ultraviolet all kinds of photo light photon and then it emits it ejects and electron from this 2 PCL and this will become a photoelectron.

So it has to go above the formidable and then it has more energy have the energy than the work function it will go even about the vacuum. So that is why actually is written KE kinetic energy of that electron is $h\nu -$ this you know the binding energy of the electron + 5. So if you know this KE we calculate what the binding energy is. XPS spectrum actually basically nothing, but intensity of photo electron verses E_B or KE.

You can either plot E_B or KB KE verses this it belong the, this is E_B or KE verses intensity. This is plotted this, what I have shown you. So it allows at the element into which a identification chemicals said the element related composition of the constitutions also balance band structure. All these things are possible.

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XPS: basic principle

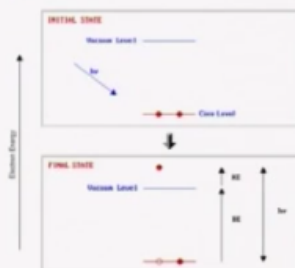


Well I am not going to detail about that, but you can see that this is what I have been shown this cold electron binding energy going to face formal level from formal level if you apply more energy that is basically called you know the ϕ and then goes to vacuum level and this is the theory or if its same level spectrometer actually that is the all the works then the electron comes it comes the peripheral level they have to spectrometer level a spectrometer defect. So therefore binding energy is given by basically.

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Auger Spectroscopy : Basics

- Photoelectron spectroscopy is based upon a **single photon in/electron out process** and from many viewpoints this underlying process is a much simpler phenomenon than the Auger process.
- Photoelectron spectroscopy uses monochromatic sources of radiation (i.e. photons of fixed energy).
- In XPS the photon $h\nu$ is absorbed by an atom in a molecule or solid, leading to ionization and the emission of a **core (inner shell) electron**. By contrast, in UPS the photon interacts with valence levels of the molecule or solid, leading to ionization by removal of one of these valence electrons.
- The kinetic energy distribution of the emitted photoelectrons (i.e. the number of emitted photoelectrons as a function of their kinetic energy) can be measured using any appropriate electron energy analyser and a photoelectron spectrum can thus be recorded.



KE: photoelectron's kinetic energy
BE: the binding energy of the electron

$$KE = h\nu - BE$$

NOTE - the binding energies (BE) of energy levels in solids are conventionally measured with respect to the Fermi-level of the solid, rather than the vacuum level. This involves a small correction to the equation given above in order to account for the work function (ϕ) of the solid, but for the purposes of the discussion below this correction will be neglected.

Second technique which you will discuss in detail is RGR. RGR is little bit different which I will discuss now then the, but RGR as the electrons comes from much lower surface depth and this XPS. Photo electron spectroscopies best of the single photon any electron out poses and for many, week point it is a very important. In XPS that is what I have showed you okay you have a co electron emitted and comes in RGR actually in RGR what happens you emit you are a photo electrons emit photon coming emit and core electrons.

And that electrons, actually hit and the electron in the outer cell and the electron from the out of cell get ejected. So therefore the electron which is coming out because of this process as much lower kinetic energy and therefore it can all comes from the surface of the material. It cannot come much lower larger depth. So therefore exact surface proving is possible by using RGR.

Okay and this is and one can actually calculate the kinetic energy of binding energy of the electron and then use it for many purposes and that is what is done in RGR and in same mass electron secondary I and mass spectroscopy is vesicle as is was in the mass spectroscopy in seems actually what is used is basically a helium ion source and this helium ion source then eject some ions inside the sample and we detect the mass of those ions.

And that is was goes secondary and mass spectroscopy and then it is used to basically major what kind of mass of this ions which are coming out and found that we can tell exactly the type of you know PCS present in the material that is of a disuse any so we are going to discuss in detail of these of this technique in a subsequent lectures.

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