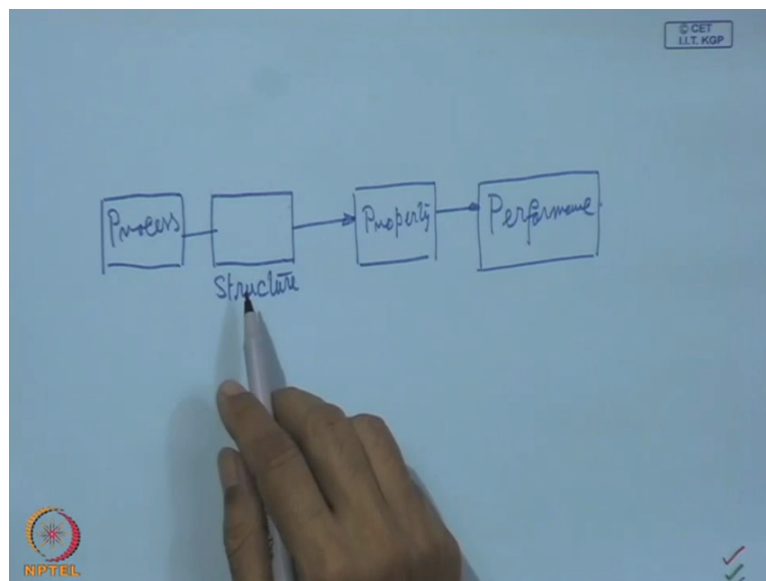


Technology of Surface Coating
Professor A K Chattopadhyay
Department of Mechanical Engineering
Indian Institute of Technology, Kharagpur
Lecture-34
Physical Characterization

Physical characterisation of a surface coating, now after this deposition of the coating it is finally the last stage of the total activity that means at this stage we have to evaluate and assess the properties, characteristics and performance of the 2 things, so this is the 1st step in that direction to know what about the quality of the coating, its property and possibly what would be the possible performance in the actual field. Now in this case what we have mentioned here as physical characterisation, now when we deposit a coating it is finally comes to this.

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It is process then structure that gives the property and finally the performance. So what is important here to know what I exactly what I mean by the structure of the coating, architecture of the coating and there are various issues we have to look and properly address if those conditions and qualities are not properly made or satisfied so that is why this physical characterisation of the coating that is so important before we go to the next step.

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Surface Coating

- Routine quality control in production of coating
- Improvement over the existing coating
- Totally a new development

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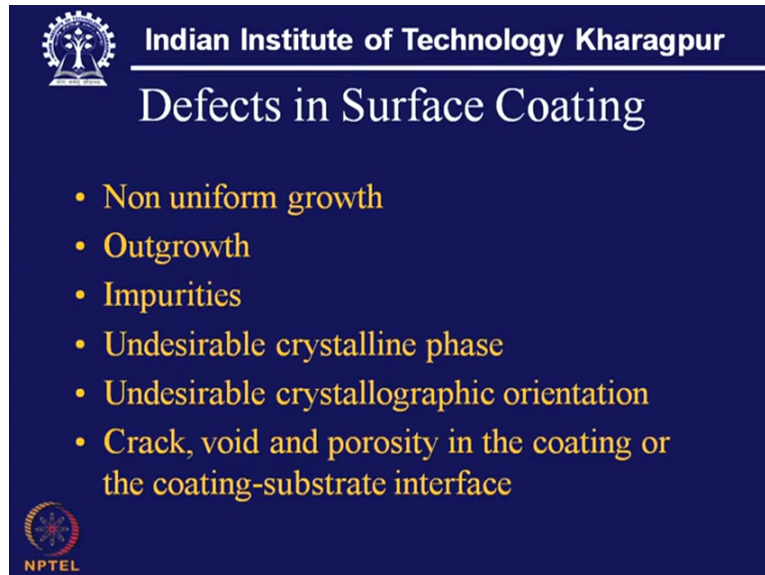
Now here what we can see that when it is a surface coating we can have 3 types of activities concerning the coating or around the coating. One is that a coating is deposited or produced in a mass production in a routinely matter, it is a routine activity and in this routine activity to know whether the process technology is consistent, reproducible and it can be handled without any difficulty and it is one of the most reliable technology time to time stock taking is important that means routine checking on the quality of the coating that has to be done and that is why we call it routing quality control and which is normally done in the industry the manufacturing unit.

Next come one of the activity, maybe we have one existing coating of a particular material but some efforts can be given to improve its basic property performance say hardness, density, wear resistance or corrosion resistance like that any of those functions properties without changing much of the basic material composition there can be some effort by adding little bit of material or changing the processing conditions, maybe there is a there may be a organised effort to improve the property and that is exactly what we call improvement over the existing one, there also we need this activity that means physical characterisation and other follow-up activities.

Now comes the third one it is totally new that means the coating is not known to us how it will be deposited or which geometry it will be deposited, what would be the material of the substrate, how many pieces have to be produced, what will be the operating condition for this coating, now things are not known totally a new activity. Even in this case even if we make

some trial run in the laboratory scale, but that is not to suggest that does not require any of those assessment of evaluation, it is equally important for a coating under development so in that case also physical characterisation that is also important. So we understand 3 types of coating, one routine, one just little bit of improvement and another is very totally new.

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Defects in Surface Coating

- Non uniform growth
- Outgrowth
- Impurities
- Undesirable crystalline phase
- Undesirable crystallographic orientation
- Crack, void and porosity in the coating or the coating-substrate interface

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Now comes the defects in surface coating surface, now what are those defects? Let us have a quick scan here, number 1 what we can see a non-uniform coating that means it is it has grown in very non-uniform manner over the entire substrate and depending upon some preferential sites we have large growth and in certain places it is very low growth. We have also what we call out growth in certain places and that looks like a mountain in those areas very easily identified. Unfortunately because of some reason whether it is in the gas phase or say the metal donor or the contamination comes from the reactor itself, it is an unfortunate eventuality and some impurities bought into this coating.

Then we have this is also very important to see that there are this is a coating but coating does not mean its composition stoichiometry and like that. There are certain crystallographic planes one like to have parallel to the substrate surfaces in order to enhance certain property and in order to avoid certain weakness so the coating should in a coating what we like to do, to minimise this weakness limitation and to increase its strength so there are situations where we may find that certain crystallographic plane of that coating material need to be aligned with the substrate surface but if it is not the case then the coating we cannot expect the coating to give its the very best and in that case the whole effort will be just counter-productive and the purpose will be totally defeated, so this is one important issue.

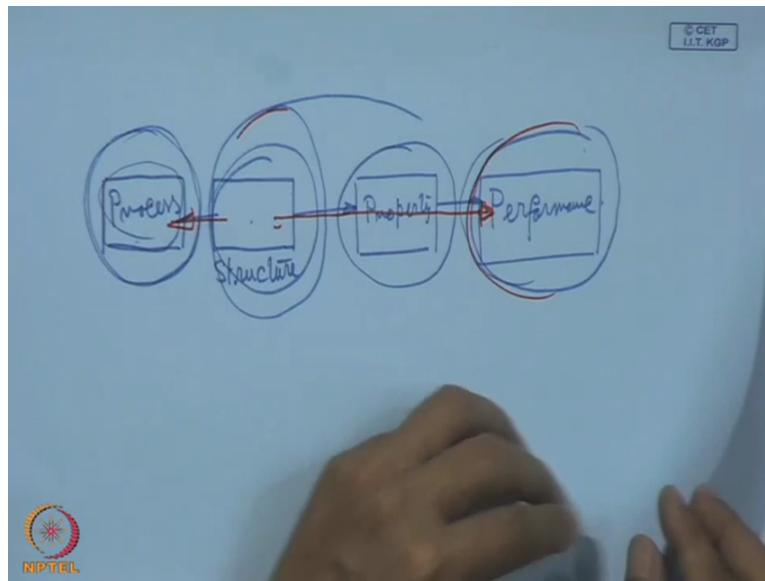
Then we have some kind of undesirable phasing the coating so this phase is not at all wanted and we have certain thing so it is not the question of that bulk percentage of the 2 or 3 components, it is actually in what form it is existing so that is also very important issue that is why we call it this undesirable phase and that should be detected properly. We have also finally this is totally a thing say for example now we can see crack in the coating, a void we can immediately detect in the coating, porosity in the coating or it can be even in the coating substrate interface.

So this coating substrate interface it is not visible once the substrate is fully covered but we must have some ways and means to examine in that level that would be sufficient to give clear picture well we have one of the strongest interface most I mean best produced interface and one of the strongest and that will give the expected or even better performance with that strongest possible coating and substrate interface addition. So these are the few things one has to look before we can go for any follow up step in the downstream side and that has to be taken up in proper manner. Now with this what we can find here that is what we call here this is need of physical characterisation of the coating.

So if we have understood that what are the needs for these defects to be proper importance and we cannot just ignore if we give due importance then definitely that necessity is immediately felt that need of physical characterisation of the coating when one looks into those possible defect which is very-very unfortunate but for certain reasons sometimes we just cannot avoid and proper action has to be taken to remove those and to minimise to a very-very insignificant level so that those defects we can for all practical purpose we can ignore them, so that is need of physical characterisation of the coating.

Now here comes we need some tools, some measuring device, some instrument, some equipment for characterisation and this is also it is just not qualitative assessment but it is good or better best but also all there must be some kind of quantification either in number or in percentage so from that we can have total assessment on the quality whether it is a routine production or whether it is a improvement in the existing coating or it is something new, something very new thing which is not at all known and that in that case also we need to have all this characterisation activities and the necessary tools and techniques for that.

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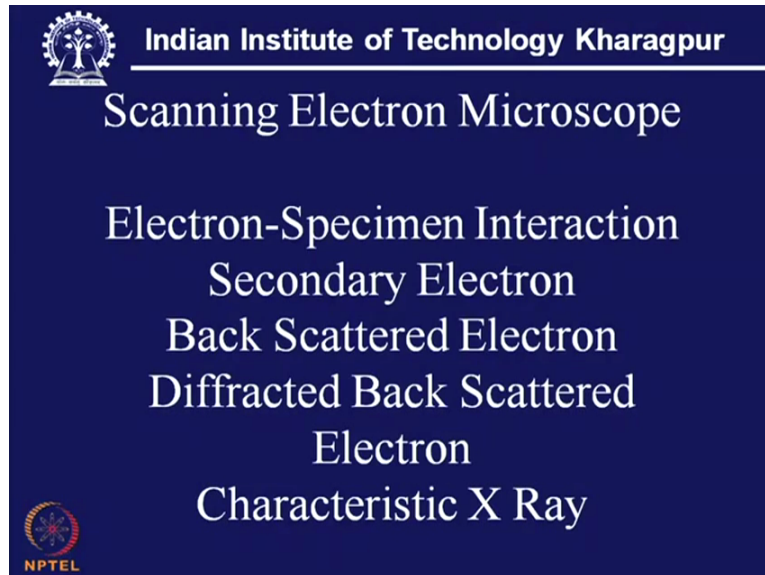
Now from the experience and from the general use in by various groups people of various disciplines, we understand there is a numerous equipment for characterisation those but few of them have their extensive use and we like to see how they do work and how they can be really used make use of this from application point of view so that one application engineer or or the coating operator, coating technician, technologists can understand that what is the what we have seen here this what we what goes wrong with the process and how this physical characterisation give us clear understanding what we are lacking in the property and how this property can be improved through the improvement in the structure and finally it is the process where a lot of connection has to be made.

So performance means it is the application engineer and here we have the coating technologist here and this is something this structure of the coating here we need an analyst so coating technologist, material analyst then property understanding of the property and finally it is the application engineer, so each has to understand what is going on in the upstream side and how we can really make use of this knowledge of this physical characterisation to get correction here on this side and also that will be reflected on this side. So this physical characterisation means actually analysing the entire coating structure, it may be from the factograph, from the cross-section, it can be from the top, it is just a micrograph, it can be a metallograph, it can be X-ray, it can be quantification.

So with that we have to have 1 wing on this the needed connection and with that we can straightaway push it on this side for expectedly a better performance. So coating process and

actual performance in between we have this very important activity and that is physical characterisation and here what we like to see that that there are certain tools, equipments for this physical characteristics. And here what we like to see that there are certain tools, equipment for this physical characterisation and how they are working, we can have a look.

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Now one of the most demanding instrument whether the ultimate application is materials corrosion or mechanical, it is really in great demand this scanning electron microscope. And this is one of the instruments the application engineer also must know how to read this instrument that means the finding of this instrument on the on the Sample that one should be able to read to record this message.

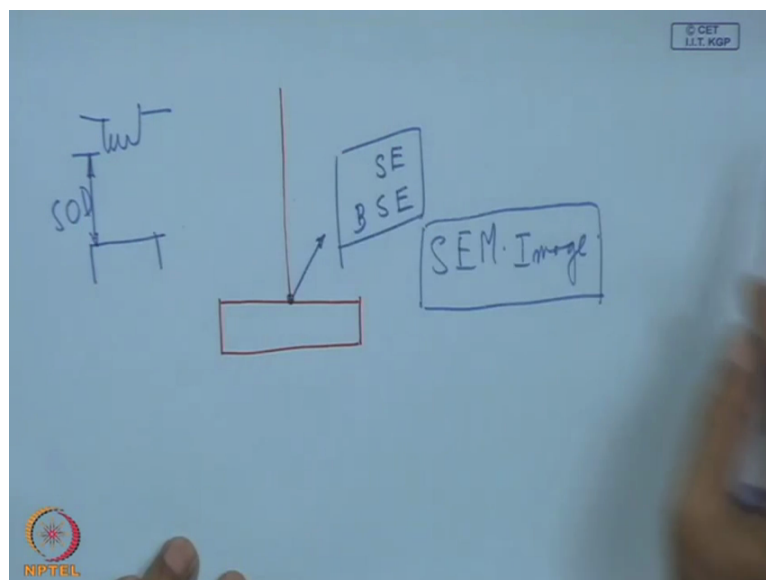
Now what is the message given by this instrument? So from that we expect so the analyst can generate a lot of information here and from that he can be able to speculate or the application engineer can also see those data or the results it can be a chart, it can be a image or it can be a quantified value and from that he should be able to have a very good speculation what would be the expected performance or the analyst and the processing engineer that means this coating engineer or the technologist, he should be able to understand how the process parameters including the substrate preparation that step should be properly tailored so that the desired structure can be obtained without any problem or hindrance.

So here what is the basic principle, we can have a quick look to this. Before that we have actually optical microscope but with the optical microscope this is the wavelength of light and wavelength of electron that makes the whole difference and with this resolution

capability that means how many lines can be separated per unit length that is actually the resolution and with that definitely this scanning electron microscope has a clear advantage. Now this is scanning electron microscope which is conventional but over that also we have FESEM Field Emission Microscope gives still further resolution.

And for those nanoscale nanostructure coating, even it is better mechanically functional, if it is chemically factional but it can a nano structure so in that case this field emission scanning electron microscope that is one of the very-very powerful tool to judge the total architecture of the coating how it is build up from the interface to the topic atomic layer and in between we can see the intergrowth habit if there is any flaw then crystallographic patterns everything then crack in the coating, some voids in the coating so the analyst should be able to record all these things from his observation, okay.

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Now what happens it is actually the process, it is actually a sample is here and that has to be put inside a vacuum chamber so it is actually vacuum chamber and there we have one electron gun. Now electron gun means we have all sorts of peripherals along with the electron gun that means it is basically a filament and it is surrounded by one anode ring and then we have like 1 well-established electron gun, we have various magnetic and static lenses for focusing of this game sharply on substrate surface or it is the coated surface.

And also there must be some coil magnetic coil for swinging the beam for scanning the beam so that the entire surface area can be recorded and registered with that beam so image will be taken and this is going to be an electron image from this secondary emission or backscattered

emission and that gives us that resolution and that will be useful for analysing the entire coating architecture and morphology and finally the prediction for the performance of the coating that has to be given from that particular image and picture. So here what we see that this electron specimen so it is actually electron and specimen interaction.

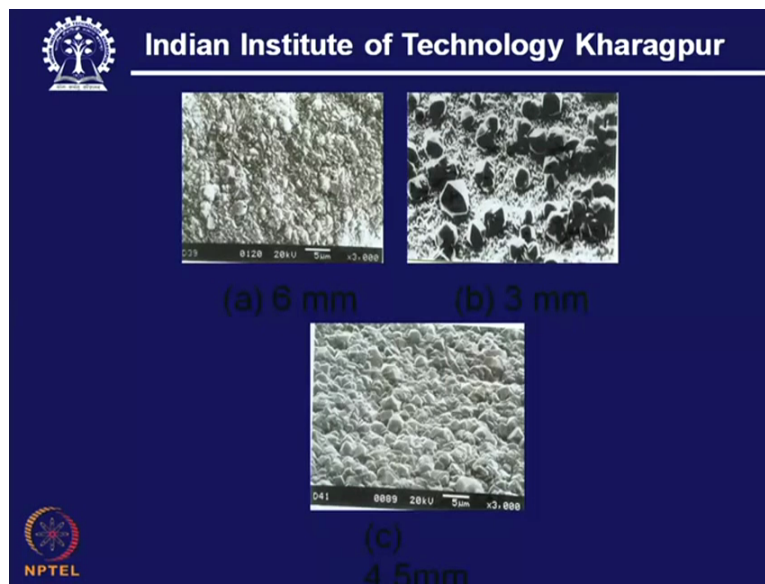
So here from this electron gun near this electron falls, now as a result of this what will happen from there, there will be some kind of thing will emerge out and these are actually one we call electron secondary electron, we have secondary electron that is used for we have also backscattered electron so this secondary and backscattered so this secondary electron and backscattered electron they are used to record the image SEM image which we or normally examine SEM image. So this is one thing we get then we have a diffracted backscattered electron and that is mostly used for studying the minerals and in that case that is used for analysing the structure of the minerals and orientation of the various crystallographic planes in that middle so that is one of the important issue and that is backscattered electron.

Further to this we also have characteristics X-ray, it is a characteristics of a particular element from this periodic table we have all this material, there is some gradual variation around the row and there is similarity along the column or the group, so this x-ray is characteristics of such variation in the element so this x-ray itself becomes a representative on one element if we can detect it during this bombardment of this sample by this electron and this characteristics x-ray can be captured and it can be processed to get some information about the material of construction of this coating

And here we can say one thing that it is actually the scanning electron microscope that can be also used for bulk material, it is can be organic, inorganic, biological but here we are mostly interested in metal, few of those non-metal, some ceramic, hard metal and their combination, so it is mostly in our case it is inorganic and effective mechanical sample, naturally it comes from within the metal group, alloy group, we can have hard metal type material that means some transitional carbide nitride with some of the metal as the binder.

Even we can have straightforward ceramic for example, aluminium oxide, silicon nitride, silicon carbide, but wherever this examination is necessary this instrument with all those outcome that means this secondary electron backscattered characteristic x-ray they are the characteristic features which can give us the ready hand information about that coating.

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Now here how we are going to look into this? Now what we see let us examine, this is actually a secondary electron microscopic image of a diamond coating. So what we see here that these are the diamond crystals, through SEM image one should be able to recognise these fine crystals which are part of this coating and this is deposited by this hot filament technique was so that is one thing. But what this SEM gives us actually if we see this one naturally we find a difference between this so that is the reading from SEM, so this image and this image there is a difference.

So here we see a well-developed crystal with a well-defined crystal habit which is expected from this coating activity, it is a low-pressure synthesis we do not have any high-pressure to facilitate diamond formation. However, here we do not have such favourable situation, this is very adverse situation the outcome is that not fully grown and it looks like that crystals are not at all grown so it is something like very towards the amorphous side some of those. So just looking at one can find out the difference between these 2, so here we have to train ourselves that how these 3 images can give us that message that yes this is one good coating, well-developed and this is a poorly developed morphology.

And if we know there are certain process parameters in diamond coating and one we know that it is actually filament from hot filament to the substrate distance and in that this SOD which is 6 millimetres, 3 millimetre and 4.5 millimetres and if we vary that one we have different results say for example, we can have just a quick look this is the filament and there is the substrate if we recall and there is SOD so this experiment give us clear indication that

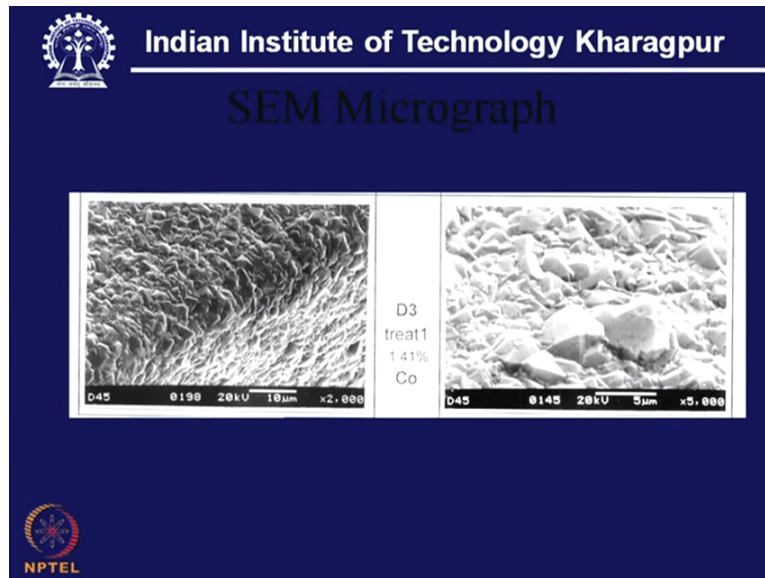
with this 3 millimetre it is so delicate and this delicacy can be understood just by the image produced by SEM and here we can see well-developed crystal, individual crystals are larger than this one.

And that we will also get from SEM but this is not covering fully so this is actually a carbide substrate so this is an exposed carbide substrate, it is fully covered but not a good morphology, this is one of the very good morphology and here we have discrete deposition that means what we can find here that this SEM image clearly give us the information nucleation centre or nucleation site so nucleation density is not quite high but in this case nucleation density was quite high that means so many points were available where the diamond could grow it and it can be inferred from the process technology if one really handle the process of this substrate preparation, one may have a very good reasonable speculation that it was also the good seeding.

If we have a good feeding with the diamond seed, fine diamond dust covering all the pits in the substrate then perhaps we can expect such thing and here this etching of cobalt and proper seeding with diamond that was not very good and as a result we have end up with such thing. Now the whole idea here to show this picture only to impress upon the fact that with this SEM picture we can get a lot of information about the quality of the coating and also with our experience we should be also able to make certain reasonable remark or observation it is with reason not without reason about the process itself in the upstream side and what are the preventive measure corrective measures have to be taken on that side that can be well understood from this SEM picture.

So this way lot of possibilities SEM can open up and this is just by one way of illustration that we can see the utility of SEM in examining the morphology of a diamond coating which can be grown by low-pressure and low-pressure and low-temperature synthesis using this methane hydrogen and filament and that is called HFC.

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Now here we can give another example that this is the diamond coating and again it is the ACM picture, now it is the cutting tool and if we know that this is going to be the cutting-edge, this is the top face of the tool and that is the flank of the tool, the surface is relatively wrap compared to any PVD coating this is also a fact. However, it is well covered that means from this we can also have a good inference that the process technology allows us to have a full coverage of the cutting-edge and it is well built cutting-edge, it is integral this coating is integral with the substrate so this picture this recording of this picture is so essential with much clarity that this is the face and flank of the tool that is well built with this coating and there is apparently no micro void of cracks.

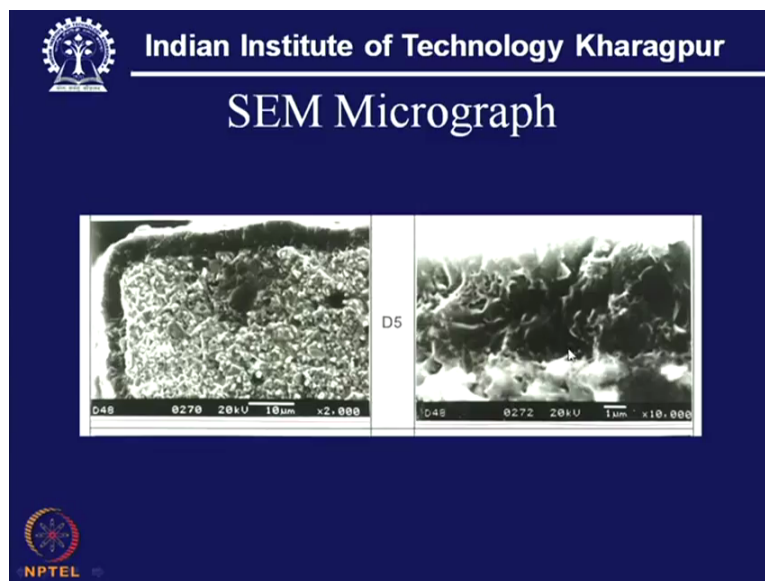
And to examine into deeper we can go for even higher magnification but if it is so needed and scanning electron microscope with this high-resolution power that can give us that leverage that with that we can make lot of analysis on the quality. Now here that is one weakness or fault in the coating that is also identified, this is a nice coating deposition which is recognised by SEM but this is at the same time is also a fault with the coating what we can see. The remaining part the coating is more or less uniform that means the crystal size is more or less uniform and that is one of the requirements we have mentioned that the very beginning of the lecture.

But here what we have seen that unfortunately we have 2 large crystals so it is like a mountain over this base and these are the odd elements in the whole matrix and this is going to make a fast contact the beginning they will make a contact or make some deeper scratch

during cutting or some processing, so this is not a very good sign in the coating and this is to be avoided so this SEM picture can reveal some anything apart from the crystal size, this uniformity, this void density, but it can clearly show this out growth.

If there are certain pinholes, it can also happen during this coating deposition, sometimes certain areas because of the very strong passivity is not covered at all by this coating that means this coating does not get attached to that and that is called the sticking coefficient so this lack of sticking coefficient can also make some pitting or void there but that can be also recorded by this SEM.

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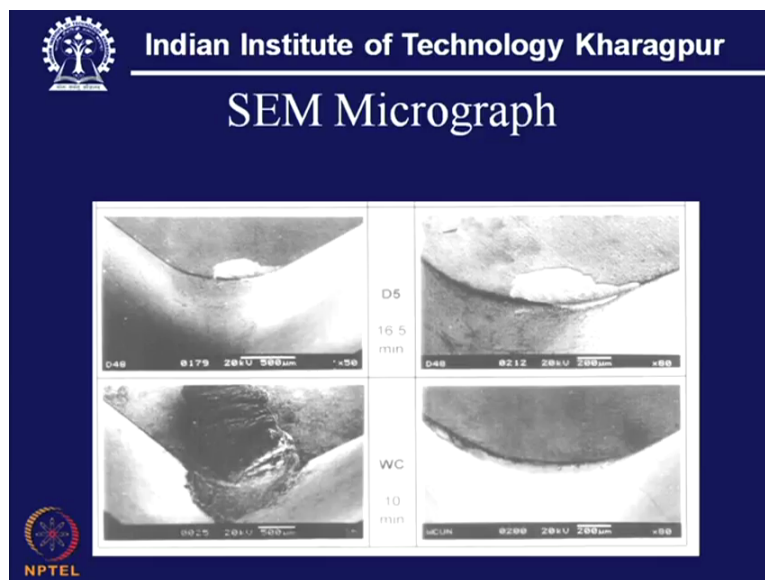
Now what we see, now we go to the cross-section that means it is a fractograph, so this is also the SEM which gives us that picture, so this is important in that this picture gives us clear idea about the coating process or the coating technology. That means the coating process itself is capable of just not coating a flat surface but it is just the 2 surfaces that means one face and flank that means 2 surfaces at right-angle and this is the edge. This is the edge that means if we go to the first slide this is the edge so if we make a cross-section here by fracturing then we get this picture.

This is the picture so it covers apparently there is no fault in the interface but still with higher magnification we can look into that what is the fault how this interface is built that can be also possible, then comes uniformity in the thickness of the coating so that is here we find the thickness is little more compared to the thickness on this side so that is also one thing one has to look in and what additionally we can see the substrate itself. The substrate is a hard metal

in this case of course so this is tungsten carbide and cobalt so the toughness of this substrate is extremely important and for that when we see that fractograph, on this fractograph we can see the average vane size which appear on this after fracturing.

So this SEM picture covers entire information on this coating up to this surface and there we can see how this coating with to what extent this coating is an integral part of this substrate. Now here what we see, it is actually this interface somewhere the picture has been taken so this is actually an interface and from this point to this point that is the coating so we can assess what is the coating thickness and with proper clarity of the picture we can also see the morphology of the coating. So it started from the surface of that was the interface and then we have some seeds also, so with this we can also see after seeding if we take a picture even if this possible that in the pores or voids because of this cobalt etching if we have these pores and voids there we can find that if these diamond seeds are rearsely captured in those pores and voids because that from that the tree will grow and then that will spread its branches and it will cover the whole area and we have a deep encourage inside the cavity, so all those things can be given by this SEM picture.

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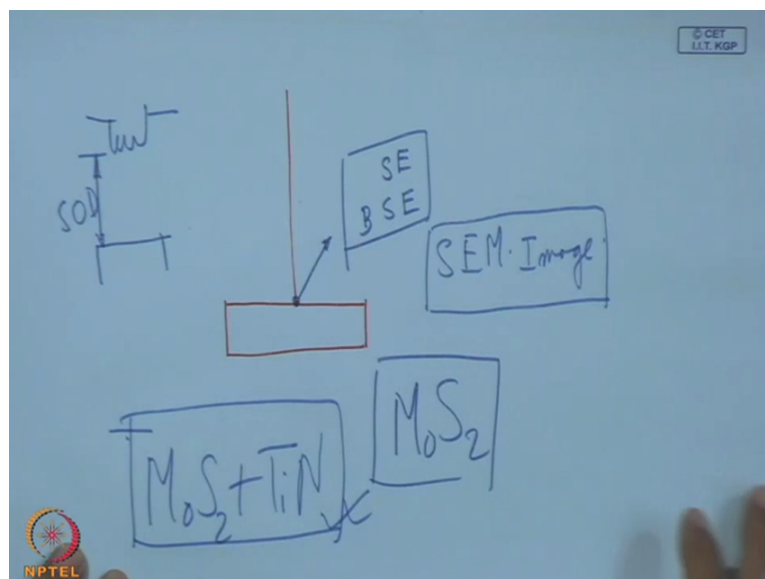
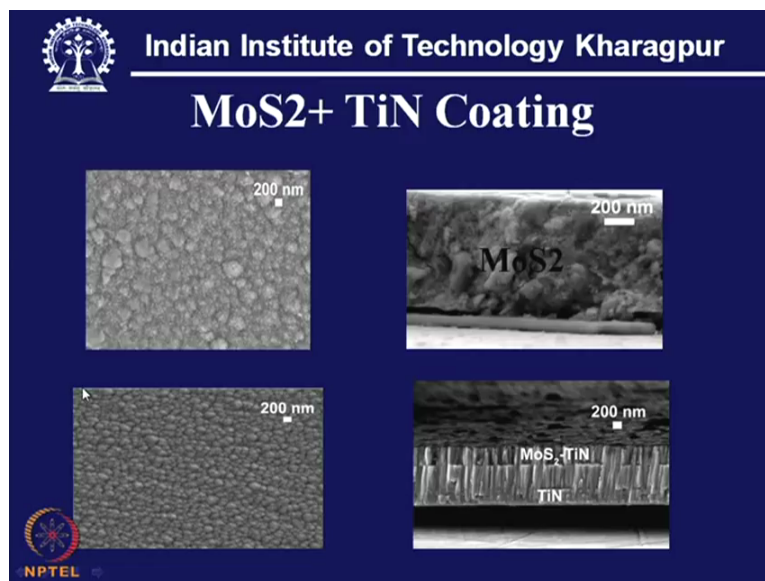


Now here also it is something very interesting that when the material is a tool top surface, if it is one non-compatible tool that chip gets stuck. However, when it is a diamond coating this chip does not stick but we can also examine how long this coating can survive, here we can see this is the substrate and that is the coating so it is also certain limitation on the coating that after doing some useful work after doing all the machining, damages not much on the flank but it is actually the chip is flowing on its side and because of some weakness at that

point of time the coating got flack in this area then exposing the substrate. But in this case what we see this is the principal flank side only here little bit of chipping, but this side this coating is still retain.

So what we see in this here that this side which will be actually follow-up zone, this site does the machining and this side it is a follower but that is the place where one has to see that this is the place where the work piece is in continues contact with the coating, so coating is not yet has gone so still it is retained and that is why we expect a very good finish that can be also recognised by SEM so we understand the role of SEM.

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Now here we can find very interesting picture, so this is also SEM study with SEM and how does it help the coating technologist to understand or the application engineer to understand

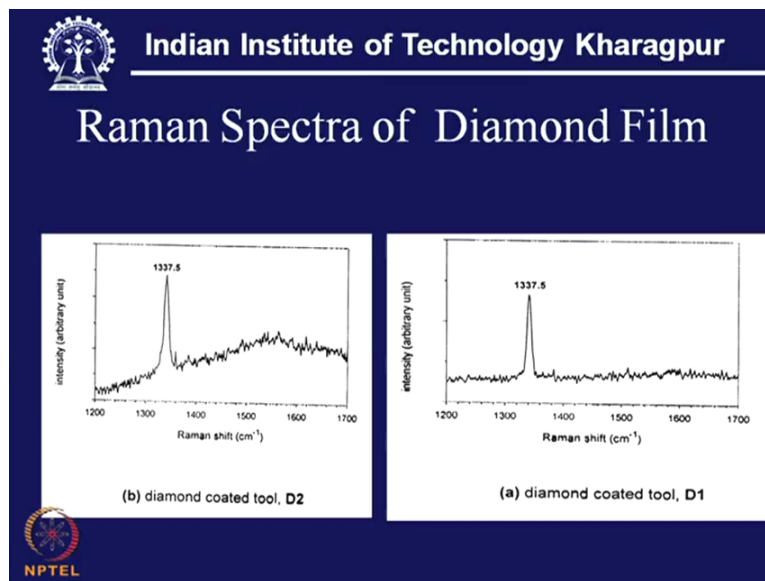
that when it is alone MoS₂ or when it is just not MoS₂ but it is MoS₂ molybdenum disulphide + titanium nitride so here physical characterisation is extremely important in that because that is a new development and we know MoS₂ in its individual entity, it is a lubricious coating and it has some limited applications, this type of structure or morphology this has a limited application in under vacuum environments with all those satellite mechanism and in those cases this is a good coating what we have seen.

But our purpose is to improve its performance so that it can be used in one of the most important tool that is cutting tool use of MoS₂. But we know that titanium nitride that is already well-known and hard coating, so on one side we have a soft coating and on the other side we have a hard coating but when we put them in the right proportion than what is the outcome? Now this outcome is immediately that message is sent by SEM picture, now here we see it is like a global, it is like a porous spongy structure, it is not so densified. However, when this titanium nitride and MoS₂ they are combined in right proportion what we see this grain refinement and densification of the coating.

And obviously one would expect that this coating will be better than this one from this cutting point of view and that what we see coming here that this physical characterisation here this architecture physical characterisation that gives us a clear message or a clear indication that this coating would be leave some promise and what we see here this SEM what we see here that this is the initial titanium coating followed by one MoS₂. Here also we have a titanium nitride coating followed by this MoS₂ titanium nitride coating and this finally that is also one titanium.

So this SEM image here also gives us this it is such an effective tool that here we can separate out this is Ti Ti layer just like it is magnified and this up from this point to this it is titanium nitride and this to this it is a mixture that means it is core deposition of MoS₂ + TiN, so these are the picture for the field emission SEM which gives a better quality than the ordinary SEM.

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Now come very important tool and it is actually this Raman spectra. Now here this is very very useful if one is engaged in deposition of diamond coating, there are numerous use and application of this Raman spectroscopy but when it is a hard coating or super hard coating one can just go for use of this Raman spectroscopy with a very clear objective.

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Indian Institute of Technology Kharagpur

Raman Spectroscopy

- Spectral distribution of inelastically scattered light
- Molecular species in all phases of matter are finger printed by their vibrational spectra

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The slide features a dark blue background with white text. At the top left is the IIT Kharagpur logo. The title 'Raman Spectroscopy' is centered in a large, serif font. Below the title is a bulleted list with two items. At the bottom left is the NPTEL logo.

Now we see how this work, it is actually we can describe based work on base its the principle is based on spectral distribution of in-elastically scattered light so this is actually and then what we see here the molecular spaces in all phases of matter are fingerprinted so this is actually the wavelength or say frequency of that particular signal or from that spectrum and

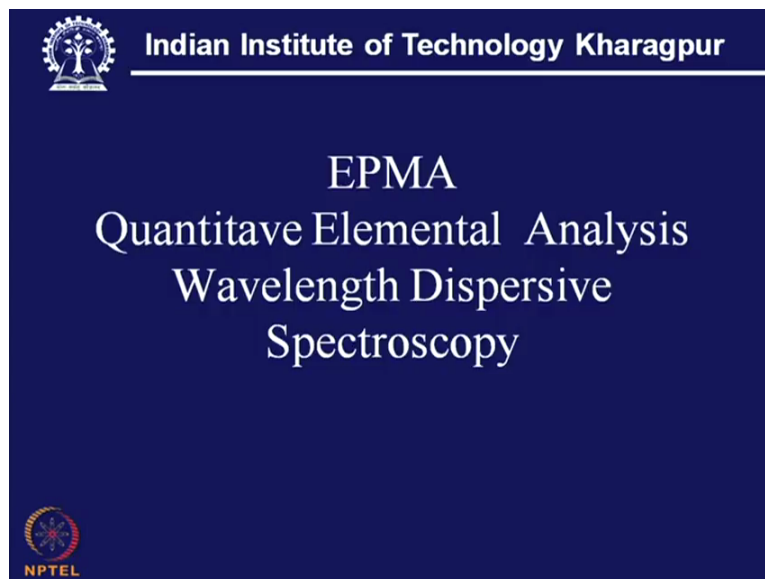
that is characteristic of a particular material. Now how it is becoming so important when we deal with hard coating particularly, it will be seen here.

It will be seen here that we want a diamond coating and without any non-diamond face. Now there are certain frequencies which will be given by say 1331 to 1337 per centimetre that is the wavelength and with that we expect a sharp peak which corresponds to diamond according to this Raman spectrum. However, if we have some non-diamond face which is amorphous then we also expect certain face around 1581 per centimetre that is the Raman shift and this way we can get non-diamond face, so we have another coating deposited in another condition so if we keep this picture side by side then we see that this is a coating free of that non-diamond face which will be weakness of diamond and here we have this kind of thing.

So accordingly this is a very delicate process dealing with CH_4/H_2 that flow ratio, the process pressure, the substrate temperature, but finally the outcome is here and this is actually this Raman spectroscopy from that we get to know what is the quality of the coating, it is just not SEM or x-ray diffraction diagram but it is actually this Raman shift from this Raman spectrum we ought to know we get to know what is the quality of the coating, so all the efforts should be made to have a spectrum like this even better with no hum formation and that will give us a clear idea about quality of the coating.

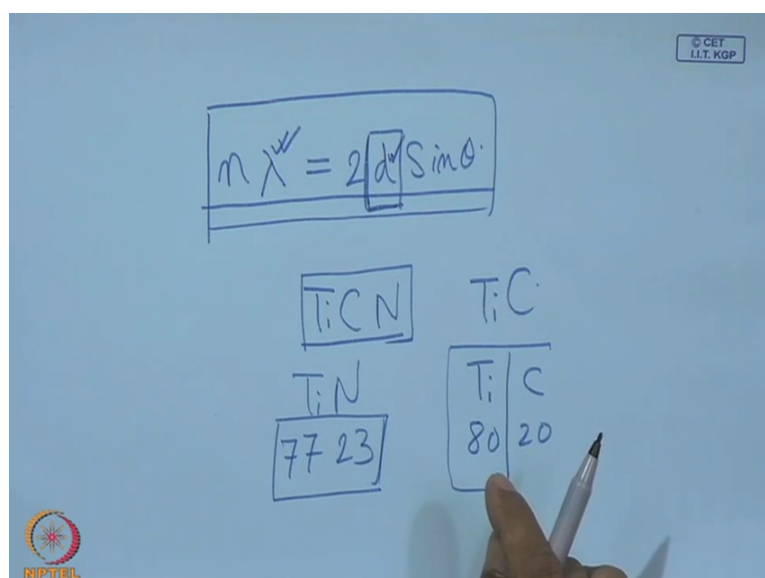
So this way we can find under various conditions with different substrate, different seeding we can have a high-growth coating process, we can have grain fineness so there can be a lot of measures to improve or to improve the yield of the process or to improve addition or the basic property of the coating. But finally before we go to any further step this diagram will definitely point at that particular direction that whether we are with good diamond or poorly grown diamond.

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Now we have what we call electron microprobe analyser Electron Probe Micro-Analyser, now this can be used along with SEM in the same chamber or we can have a different equipment for that exclusively for metal analyses and it is a quantified value, it is exclusively used for recognising the purity of the metal for any construction work from metallurgical point of view, from material science point of view and also from application point of view. So it is quantitative element analysis and it is called Wavelength Dispersive Spectroscopy.

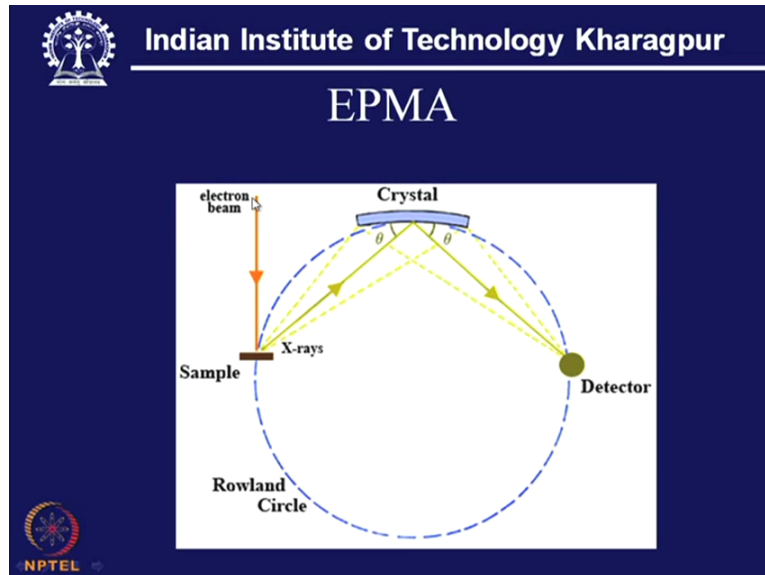
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So we have actually 2 types of spectroscopy; one is called energy dispersive, another is called wavelength dispersive spectroscopy, now let us see how does it work? Actually we can see we know that this is one equation what we call $2 d \sin \theta$ Bragg sign law. Now here it is

the wavelength of the x-ray that is the actually the distance between the 2 planes that is the space lattice and this is actually the angle along which we can get this thing. So that means this value of lambda and this Sin Theta they are related with this 2 d or say for this value of lambda if this d changes then also this value of Sin theta changes.

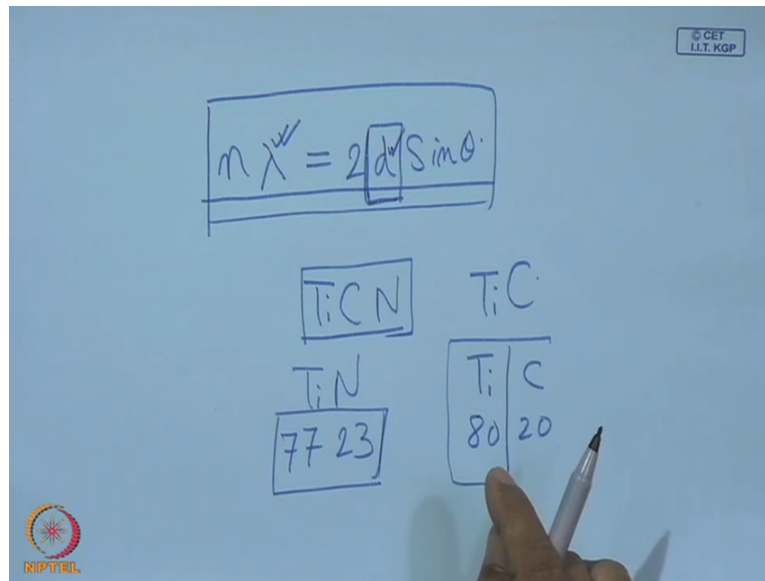
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So this way what we see in this case, electron beam it falls from this electron gun and this is the Sample. Now when we have mentioned about SEM we have shown that we have this secondary emission of electrons, backscattered emission of electron, those are used for imaging and we have this characteristic x-ray and this characteristic x-ray that is used for detection of this element. And this characteristic x-ray that is a typical of a particular element and that is also given by this one, so here we have this wavelength and this is also connected with this $d \sin \theta$. Now let us have a quick look how this principle how does it work?

This electron beam comes from the electron gun, it falls from on this and we have emerging x-ray which is emitting from this surface and there we have a detector crystal. And with this detector crystal what we see that this will be there and that will be a reflection here on this side and that will be detected and captured here. Now it is interesting to know that all these things are placed on the circle and that is called Rowland circle so what we can see these are on the diameter so whatever may be the angle theta, then ultimately this ray is going to reach this detector. Now what we can see depending upon the value of Theta this for the lambda this value of Theta will change.

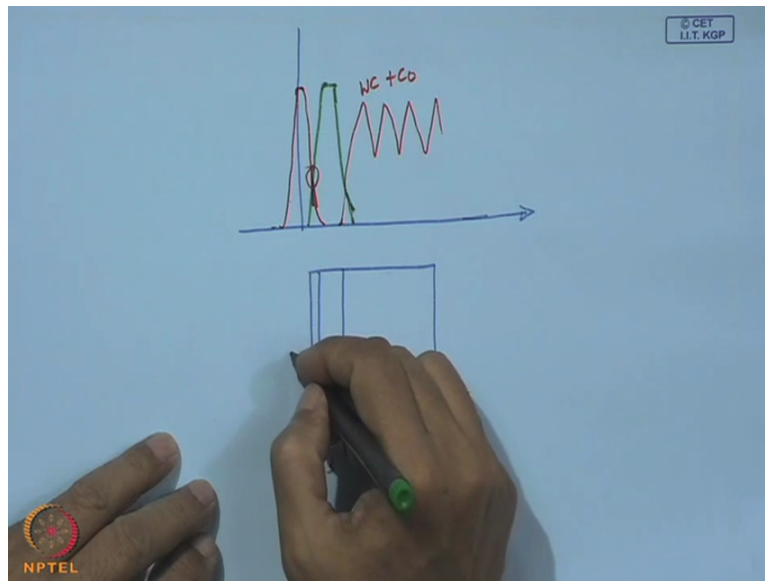
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That means if this lambda changes because of the particular material this lambda will change and that will be detected by this Sin theta that means if we change this orientation of this crystal then a particular wavelength will be captured and that will follow this particular equation, so that will correspond to a particular d value and this d value will be a characteristics feature of one particular element and this way we can have detection of different elements which are except few of those very light elements but most of the elements it can be detected and this beam this detector that can also probe over the surface.

So with this what we can find not only the compositional composition, quantification of the composition say it is TiCN, so value of for example value of titanium CN or say for example, titanium carbide. So it should be say it is 80 percent and Carbon 20 percent by weight, or if it is titanium nitride for example it will be 70 percent and 23, so this is way lot of analytical activity can be done, but this thing we can use to our advantage just by analysing the coating surface from the top or it can be done this way that say for example we have the coating something like this.

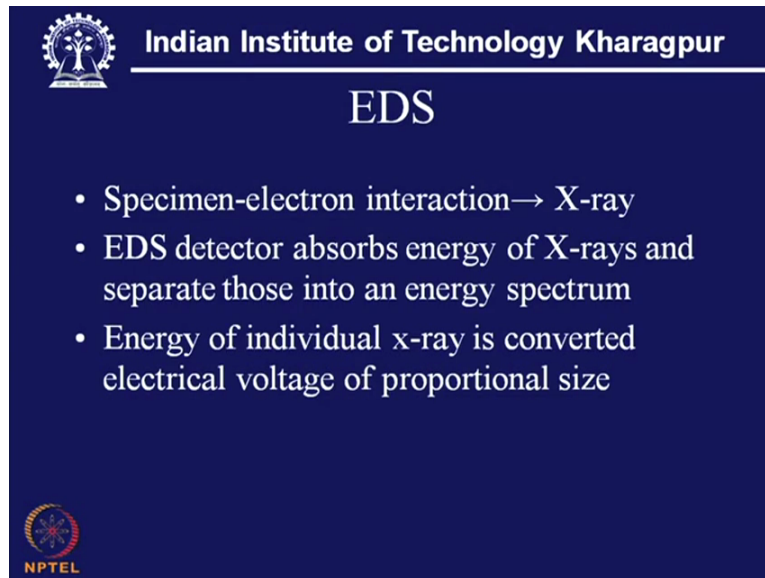
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There we have aluminium oxide coating and then we have titanium carbide coating and then we have this substrate here, so this is aluminium oxide followed by TiC and followed by the substrate and the beam can scan over this so if we look into this, this is the distance it travels and over this if we have the coordinate which shows the concentration then what we can find that at this point we can have this increase of this aluminium oxide aluminium and then it will fall immediately because it is a thin coating and then comes the concentration of TiC that can go like this and that will fall and then what we have, here we have the substrate and that the percentage of so this percentage will go like this because tungsten carbide + cobalt.

So this way we can find out the diffusion between interfaces or the thickness of the coating. So we can apply this principle of detection and that can be very well used for characterisation of this coating. In that it can be one of the instruments for quantification in a point all we can also have a line scan for analysis of this coating along the interface along the depth.

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EDS

- Specimen-electron interaction → X-ray
- EDS detector absorbs energy of X-rays and separate those into an energy spectrum
- Energy of individual x-ray is converted electrical voltage of proportional size

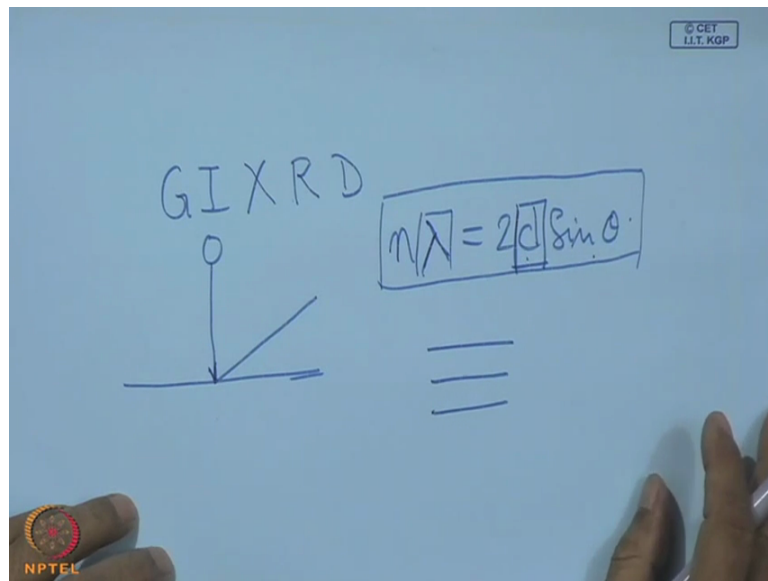
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Now we have EDS that is energy dispersive spectroscopy, so in this case what happens it is also the X-ray but it is no more the wavelength but it is the energy. Actually the EDS detector that absorbs the energy of x-ray and that and it separate those into an energy spectrum that means energy which is released as x-ray that will be absorbed and there will be spectrum and this spectrum is actually typical of all the elements which are present there and that will be then that will be a spectrum separating those.

So this is very important it should be able to separate and finally what happens this energy of individual x-ray that is converted into electrical voltage of proportional size. So this electrical voltage this x-ray that is typical characteristic of one individual element, so from that it is possible to find out the element which is present, what is the percentage and also the combination that means it is the percentage of element and also it is the atomic ratio in what atomic ratio it is present in in that volume. Now in this case what is important concerning the activities in the coating, so there are certain things what should be looked into say for example its application W S₂ coating of molybdenum disulphide coating.

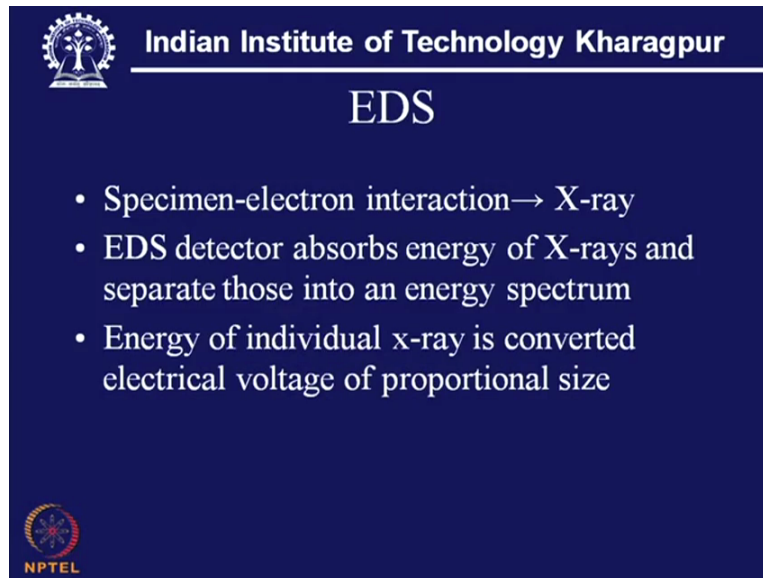
Now these are of immediate interest but also we see also we have seen that in this case incorporation or oxygen gets into that and that makes this coating inferior and this quality falls drastically. So in this case whatever may be the issue, only detection of oxygen is extremely important, now it is the typical application of these EDS or microprobe for this particular coating it is the detection of coating just like Raman spectroscopy we have illustrated that it is extremely useful for diamond coating but this is also extremely useful.

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Now we can also have say XRD now XRD means X-ray diffraction diagram and it can be also GIXRD that means it is for the film. Now what we see in this case it is also we can we know that this is $n \lambda = 2 d \sin \theta$ that is the Bragg sign law so depending upon the x-ray which falls on the surface this x-ray from the x-ray source it is falling and there will be a diffraction so this x-ray there will be a diffraction and the detector will detector is going to capture this diffracted ray depending upon the value of θ . Now depending upon the element or the face the value of λ will change and since it is actually the $2 d$ that means the spacing, now depending upon the crystallographic spacing this λ will be connected with $2 d \sin \theta$ that means if this d changes this spacing then a particular λ particular value of d that is connected with this $\sin \theta$.

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EDS

- Specimen-electron interaction → X-ray
- EDS detector absorbs energy of X-rays and separate those into an energy spectrum
- Energy of individual x-ray is converted electrical voltage of proportional size

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And accordingly in the XRD diagram where we put 2θ and we can get typical peaks which shows the very existence of a particular face or it can be this 2θ that can be measured here or it can be in another place. So this is also typical of a particular coating say for example WS₂, in this case what we are interested is in 002 bezel plane and this XRD diagram that will give us clear indication of this 002 plane depending upon the value of 2θ .

So for a particular value of 2θ if this peak is indicated we can immediately recognise whether it is 002 plane that is parallel to the substrate surface. If it is not the case is so that it comes at the lower angle, there are other plane which are h plane or 101 which appear at a higher angle, if those things appear then that is also a clear signal that this coating cannot be a good candidate for this lubrication or lubricious property it cannot give and it cannot have the satisfactory performance in that particular activity. So these are the few illustrations and examples, so this is actually how this XRD diagram can be used?

It can be used with a one particular coating having different crystallographic planes, so in one case may be 111 plane prominent but it can be also 100, so whether it is strongest textured coating or it is a coating with what we call equiax that means that type of coating that can be also detected by this XRD. So we have seen that this instrument of physical characterisation which are scanning electron microscope then XRD with this GXR that means this grazing incident XRD for analysing the film, then we have Raman spectroscopy, we have this energy dispersive analysis of X-ray.

These are the few tools of physical characterisation, these are commonly used for physical characterisation of the coating in particular for those mechanically functional coating and which are used for all sort of ware part, friction reduction tools of manufacturing, for those this physical characterisation is extremely important. And if we get a favourable response from this physical characterisation then only we go for all sort of mechanical characterisation and followed by this performance test. So this physical characterisation is one of the very important steps between the processing of the coating and finally evaluating the performance of the coating.