Chemical Engineering Principle of C V D Processes
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Lecture No 10
C V D Film Property Measurements

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Good morning and welcome to this lecture in our course on Chemical Engineering principles of C V D processes. In the last few lectures we have been looking at various types of C V D processes and reactors, various conditions of pressure, various patterns of temperature distribution in the reactor, and various types of films that we deposit on substrates.

So today we are going to talk about methods of measuring properties of C V D films. Why do we need to measure the properties of C V D films? There was actually a saying by Lord Kelvin who invented the Kelvin temperature scale. He said, what you do not measure, you cannot control. It is an interesting statement.

What he is saying that unless you are able to measure something, you cannot guarantee either its quantity or quality. Typically to guarantee the quantity we have to do quantitative measurements and in order to guarantee quality we have to be able to do qualitative measurements. And so when we talk about C V D films both types of measurements to be applied.

There are certain properties of the film that we need to be able to characterize in a qualitative manner and certain properties that we need to quantify. Also there are certain properties that are functional. What the film is supposed to do for you that has to be measured.

Whereas there are some properties which are less direct and these indirect measurements however have a large role to play in terms of process control and optimization and ensuring that we get the functionality that what we are looking for.

Now when we do measurements in general, you can classify them in two ways. There are what are known as in situ measurements and ex situ measurements, also called online measurements and offline measurements. An in situ or online measurement has a huge benefit that the data are being measured in real time.

So as you process wafers and they are going through with a film being deposited on them, you take the measurement right there. So you have visibility into what is going on with the process virtually at any instant in time where as an ex situ or an offline measurement is, as the name suggests done remotely. So typically every shift in the manufacturing line, you would take it to a sample.

You would take it to the laboratory and subject it to various types of analysis. Now the downside of such measurements is there is a time lag. So by the time you take the samples, by the time you do the measurements, by the time the data comes back, you would have processed many more lots of wafers or other products.

So if you detect a problem in your offline test, everything that you have produced between the time you took the sample and the time the data came back would be compromised. And so you do not have the real time feedback, so that is the biggest downside. The upside though is that, the offline measurements can frequently be done using much more sophisticated instrumentation.

The readings can be much more precise, much more accurate and can yield much more detailed information about whatever you are trying to inspect. So they do have, you know pluses and minuses. What you really need then is a combination of the two. So typically in C

V D industry both types of tests are employed. There will be series of in situ tests which typically will provide quick data, mostly quantitative which can be used to essentially derive process control charts and put in, you know S P C methodologies, Six Sigma methodologies and all that to ensure that you maintain the quality of the output.

In addition you would be simultaneously doing offline testing on the same C V D films to look for more detailed characteristics, for example the structure of the film. It is not easy to do that online. Although now there are online X R Ds that can give you a brief snapshot of how crystalline the structure is but if you want a more detailed, for example sectional profile of the film to see how the composition is changing across the thickness of the film, you cannot do that online.

So a good combination of both types of measurement methodologies provides the most value, both for the producer as well as the customer. Another way to classify measurements in such instances is the direct versus indirect. When we say direct measurement, what we mean is the reason you are realizing the C V D film on the substrate is for a certain purpose.

We have looked at several of them. You know, it could be to provide conductivity variations. It could be have a conducting film, semi-conducting film, dielectric film, or it could be for completely different purposes, may be to provide thermal protection or moisture barrier capabilities or it could be corrosion protection, erosion protection, wear protection, galling, I mean there are many, many reasons why C V D films are put down on surfaces.

So the most direct test would obviously be one that tests the purpose for which the C V D film was put down. I mean if you are putting it down to achieve a certain conductivity at a location, obviously the most logical test to do is to measure the conductivity there and see if you have realized your objective.

And from the customer viewpoint that is really what they care about. They do not care about what it takes to produce that film. What they care about is, are you able to deliver to their specifications? And so the direct measurements are of most value to the customer who is using the product. Now this may not even be an external customer.

It would be an internal customer. For example you could be making silicon wafers and part of the process may be chemical vapor deposition of certain layer on the wafer and then it goes to the next step in the process. The next step becomes the customer for the previous step, right. So when I say customer, do not think about external customers. It can also be an internal customer, may be just your division next door.

But they would expect certain properties of this product that you are making. So you know, obviously conductivity measurements are easy to make. One thing that is different though, when you are talking about C V D films, I am sure you have done conductivity, resistivity measurements in high school, in college. The difference is of course, with a C V D film you are looking for planar properties. You are looking for sheet resistance properties, planar conduction.

So you have to be able to do essentially multi-probe type of measurements in order to be able to measure not only the conductivity or resistivity at a single point but rather across a continuous plane of materials. But apart from that, many of the techniques you have already learnt to measure conductivity and so on are quite applicable here also.

The other difference of course is that you are talking about measuring conductivity across a very thin film. So that requires certainly more precision particularly in the positioning of your probes. You want to be able to take your measurements only across the film and you know the probes should not, for example touch the substrate. So the positioning to nano or micrometer resolution becomes important as far as keeping the probes on the surface.

And also sometimes you are interested in what are known as breakdown voltages. This is particularly important when you have an oxide film on a surface. You want to be able to tell how much of the voltage applied can that film support before it breaks down and allows, for example conduction to happen. So this is called the breakdown voltage.

So essentially you would be testing conduction as a function of voltage and you keep increasing the voltage until you get to a point where there is a sudden sharp spike in the conductivity and that is where the oxide film basically breaks down. That measurement is important to a, it can actually be used as an indirect characterization of thickness of the film because you can actually pre-calibrate this breakdown voltage or voltage as a function of thickness and using that, you can use voltage measurements, sorry current measurements for a given voltage as a measure of thickness.

So that is one use. The other use is it is important to find out what is that breakdown voltage for a given thickness or a breakdown thickness for a given voltage so that you can ensure that the minimum thickness never falls below that value. Because you know that if that happens you are going to have a catastrophic situation.

The other type of measurement that is done uniquely for C V D films is called the, the, it is basically an electron mobility analyzer, Hall Effect analyzer. And this provides more detailed instruction not only on the conductivity; it also tells you the conduction paths that are present in your film. So if you have localized discontinuities in the film or defects in the film it can be used by making such measurements.

Of course when there is, the purpose of C V D film is to provide protection to the surface for example coatings then the protection properties can be tested. If your C V D film is being placed on the surface to provide corrosion protection, then you have to device an appropriate corrosion test.

So this could be something as simple as an environmental chamber where you put the substrate with the film into a temperature humidity chamber and cycle the temperature and the humidity through the specified values and look for signs of corrosion on the surface. It is an accelerated test for corrosion.

The other way that corrosion is frequently tested with even more acceleration is what is called the salt spray test. So you take a solution of salt and spray it on the substrate and see if corrosion is initiated. So there are very specific corrosion tests typically developed by certification agencies like A S T M that specify exactly how the test is to be conducted and you can certainly conduct the test as specified.

Similarly erosion testing, there are erosion testers and erosion test procedures, standards that have been defined and so you can take the substrate, mount it, and subject it to various types of particles impaction for example and see if erosion develops and what is the rate of erosion for a given particle size, given velocity, given angle of impact and so on.

So these are obviously functional properties that

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you are testing. And this data when it is applied to whoever is consuming the product is going to make them happy because they know that it is doing the job that it is supposed to.

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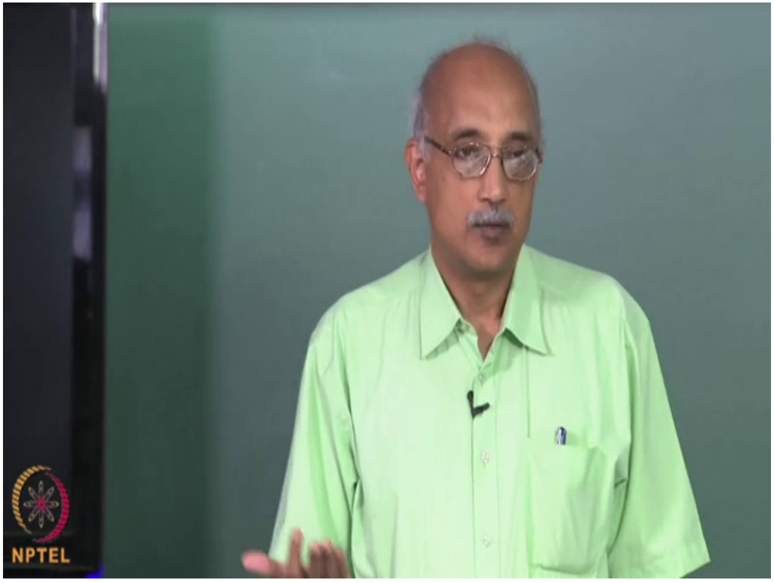

But as a C V D process engineer or equipment engineer, you know that you do not have direct control over that property.

What you have direct control over is the thickness of the film, the composition of the film, the uniformity of the film and so on which you can control by controlling the process parameters and the design of the equipment and the controls that you have on the process and so on.

So you have to identify the controllable parameters in your process and start measuring them so that you can establish the required controls over these controllable parameters and deliver the required product quality to the customer.

For example if I am trying to provide certain corrosion protection then I know that the way I am going to achieve that is by having a certain composition of film on the surface, by having a certain thickness, by having a certain density, by having a certain level of impurities, by having a certain structure and so on.

So I can list 7 or 8 process or product parameters which if achieved will enable me to achieve the functional specification on the product that I am making. So that is an exercise that you have to go through and identify the critical parameters in your system.

So when we look at a C V D film, you know things that obviously pop out, in terms of important parameters to measure, indirect parameters right, not the functional parameters but the indirect measurements.

The key ones would be I would say the number 1 property that

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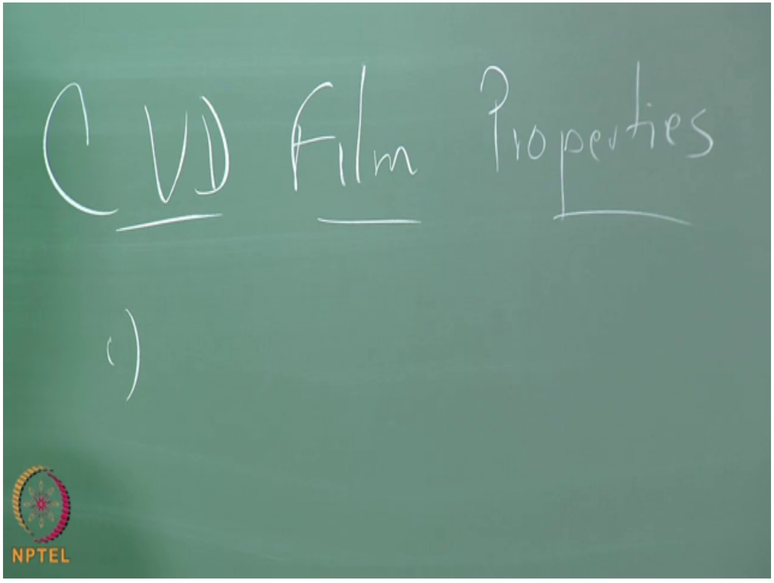

CVD Film Properties

i)

you would try to measure is thickness.

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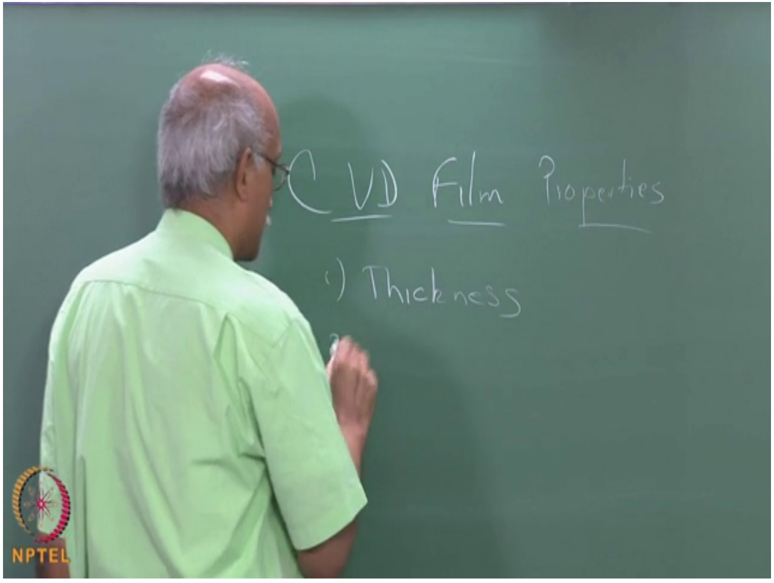

CVD Film Properties

i) Thickness

And structure.

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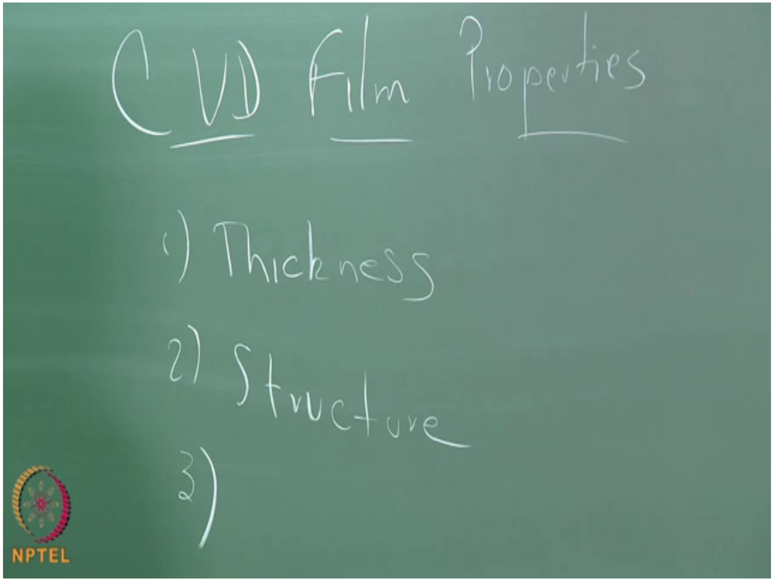

CVD Film Properties

1) Thickness

2) Structure

3)

The third one is what is known as line width, also critical dimensions.

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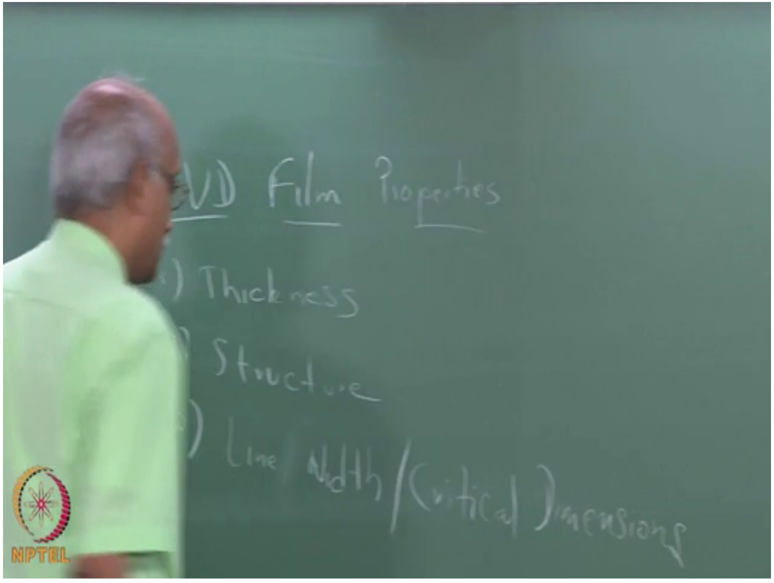

CVD Film Properties

1) Thickness

2) Structure

3) Line Width/Critical Dimensions

So what we mean by line width critical dimensions is if you have a substrate and you are trying to put down a film on top of it, there is usually a pattern to the film.

So you may have a certain

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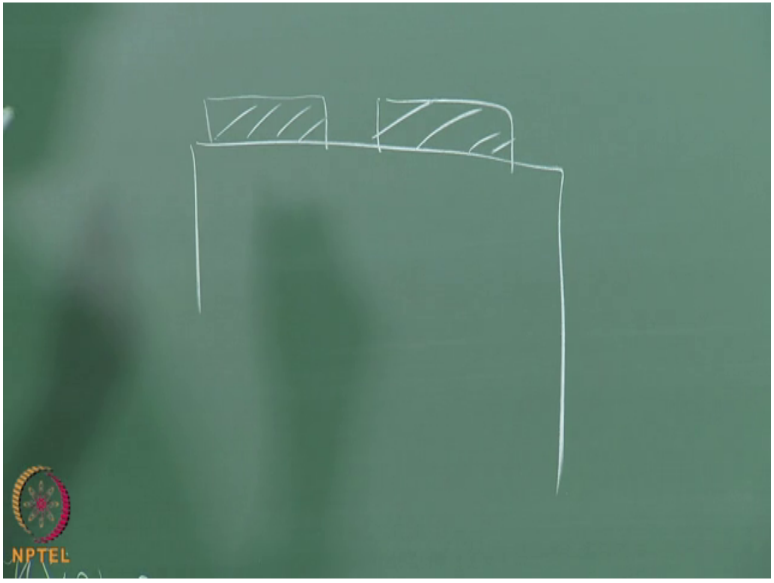

thickness up to here and then the thickness may drop up to zero here and then rise to a certain thickness later, right. So in this particular case, the line width as it is defined is the distance of separation

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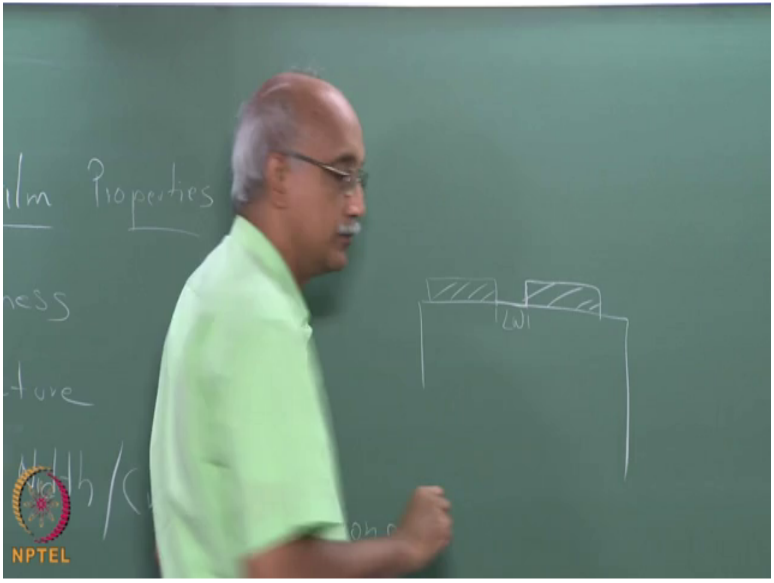

between the two C V D films.

The critical dimension is typically this and this, the length, the width and of course since this is three-dimensional, the height or the thickness of the C V D film. So these are the critical, geometrical dimensions that you try to measure on a surface. So thickness, even though that is listed separately is actually one of the

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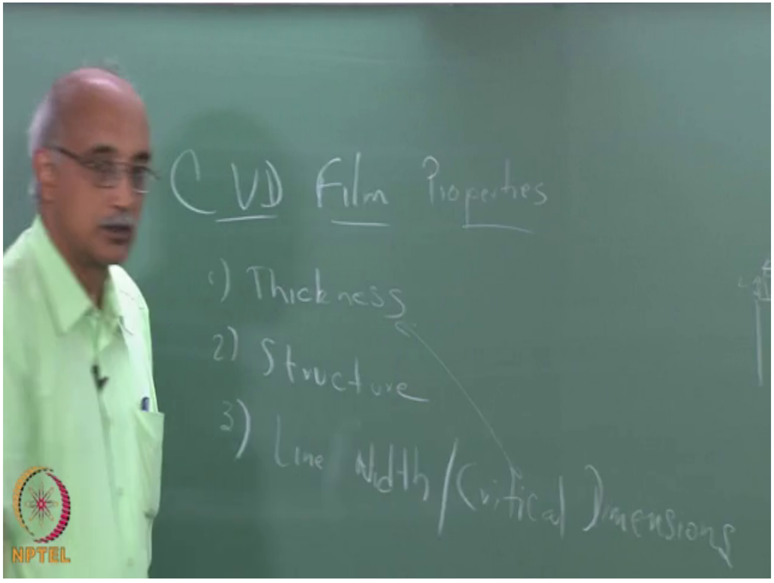

critical dimensions that you want to be able to measure on the substrate.

Composition is obviously very, very important to know. You are trying

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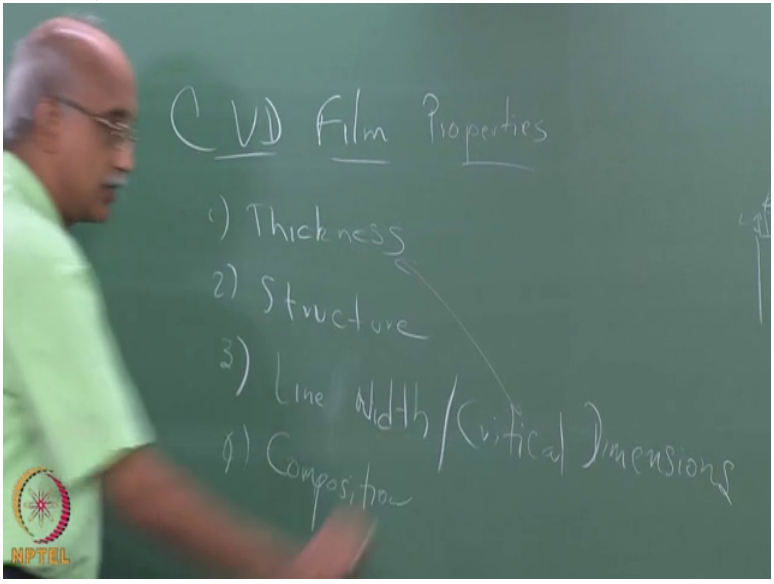

to deposit a certain C V D film; you have to be sure that is what you are depositing. And that is not some non-stoichometric film that you are depositing or something that is completely different from the chemistry that you are trying to achieve on the substrate. So the composition is important.

And then you know the surface properties, particularly roughness can be a very important parameter to measure.

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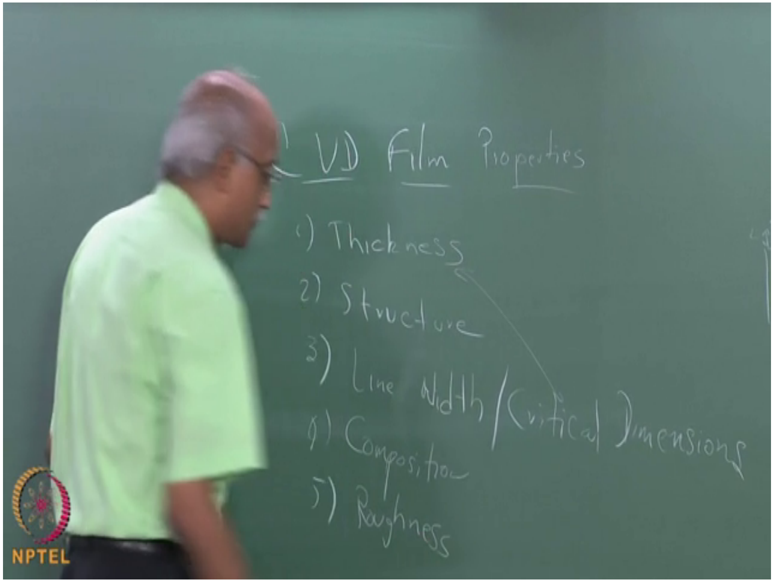

When you have a substrate it has certain predefined roughness. But when you deposit a C V D film on it, the roughness is going to change. That can be a very important parameter in many applications, particularly when you are trying to provide a certain, if the film is trying to provide certain protection.

Because rough surface for example, will erode very differently from a smooth surface, so the surface roughness becomes a very important parameter. And impurity levels are also important to measure.

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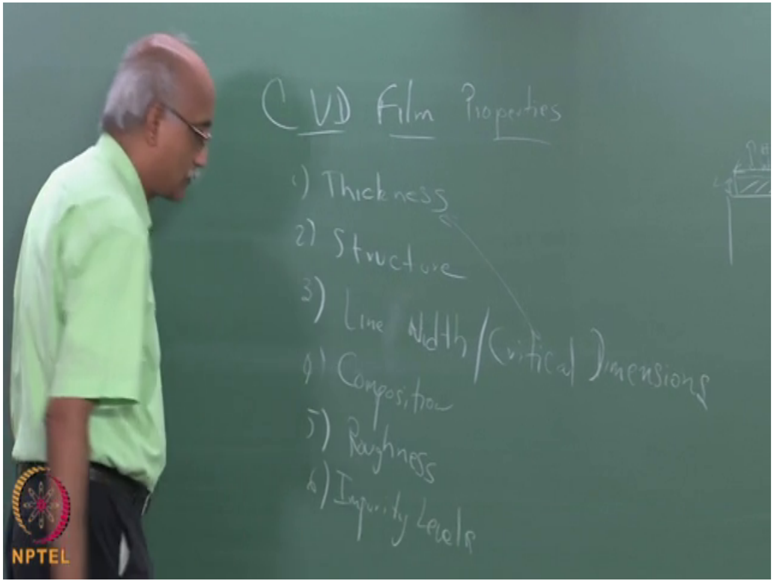

You want to know how integral the film is, both from a physical viewpoint as well as from a compositional viewpoint.

The other parameter that we have mentioned previously is grain size.

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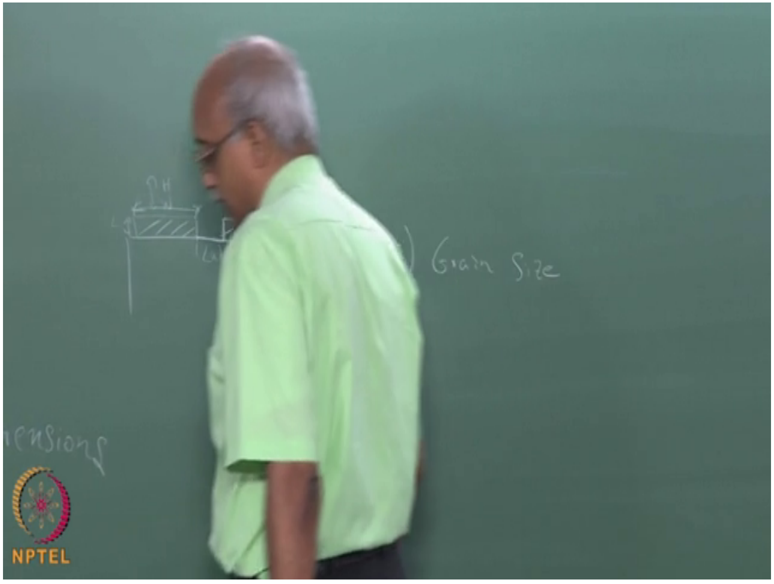

The grain size of the film to a large extent determines its strength, its durability, its permeability; many of the properties that you try to achieve with a C V D film are actually leveraged very effectively by using grain size as a controllable parameter.

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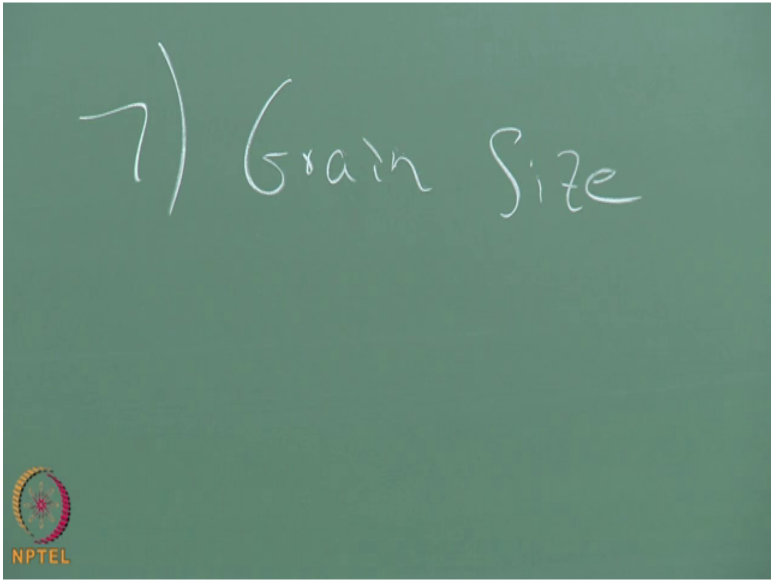

What else? I mean we certainly have to look at properties like hardness, density.

Another property that we have mentioned earlier is etch rate which is a good measure

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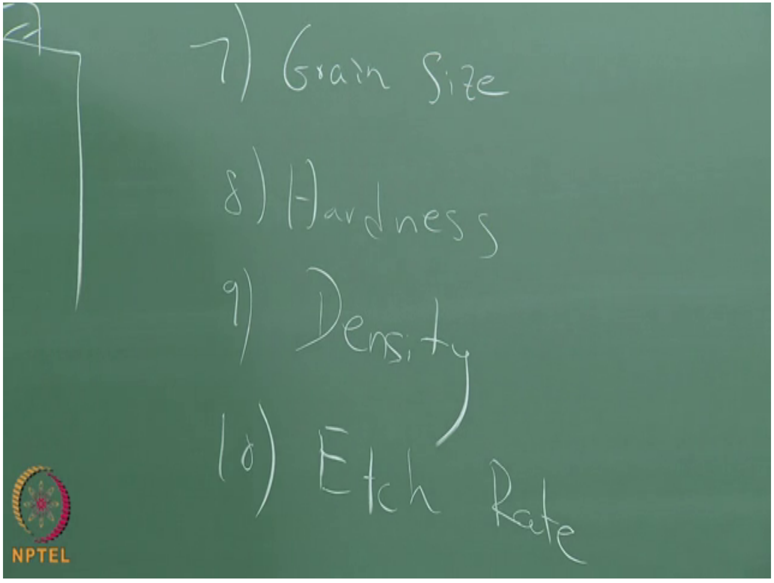

of how sustainable the C V D film is under aggressive environment. So I mean there is obviously fairly long list of things that you can measure about a C V D film and where you stop really depends on, you know again your process requirements and your customer requirements.

The more demanding your customer is and the more high end the product is that your C V D film is going to go into, typically the more measurements you have to do to ensure that the process stays under control.

Whereas if you are making the C V D film for the mass production, low cost type of device, then you may choose to do only couple of these and say that as long as these stay in control, I am going to assume that everything else stays in control. Again the primary way that you use these measurements, you know it is not enough to measure. It is not enough to simply react to the measurement.

A good manufacturing process will have a strategy in place where you look at not only whether a particular measurement is in spec or out of spec but you actually do some trend charts and make sure that the process is not showing signs of drifting out of control.
Typically the type of charts that you would do are x bar range charts, so you would measure this as a function of time, and there will be a

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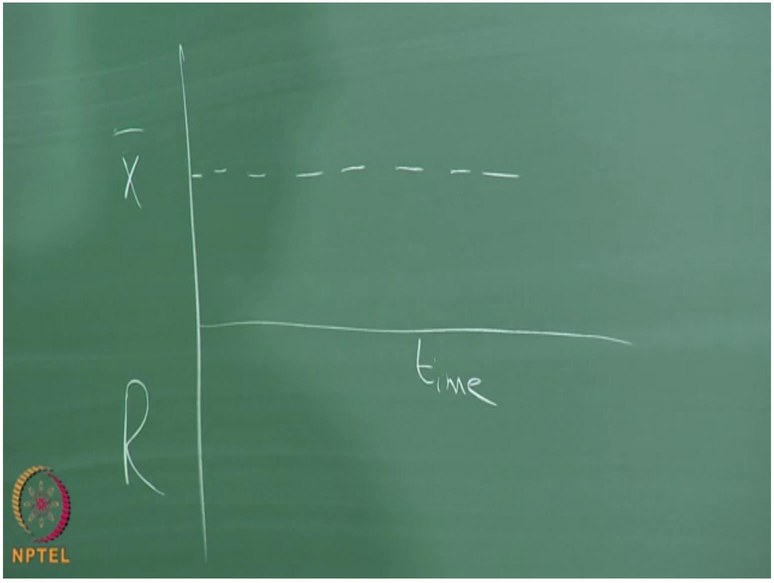

mean for the process. There will be a upper spec limit. There will be a lower spec limit. So for example these could be thicknesses of the C V D film and you would want to have a specification on it that is, say plus or minus 5 percent, right. So you would have these spec limits.

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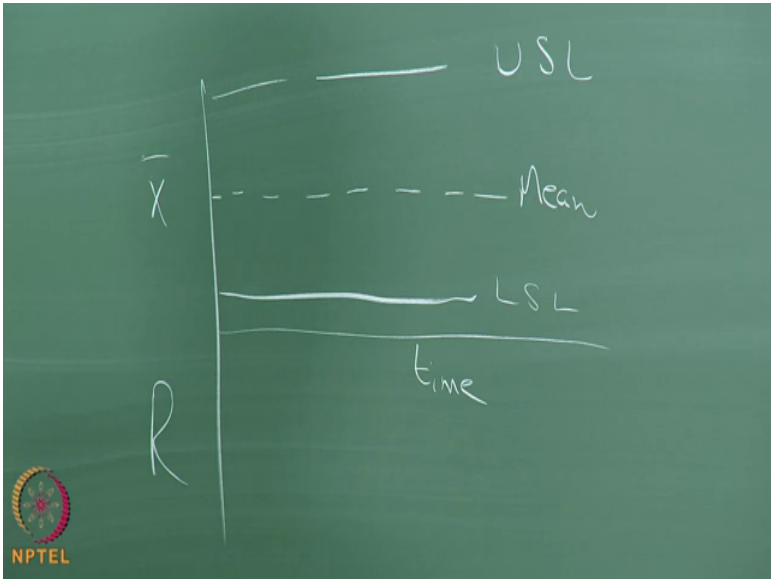

And so your mean would essentially show some trend like this.

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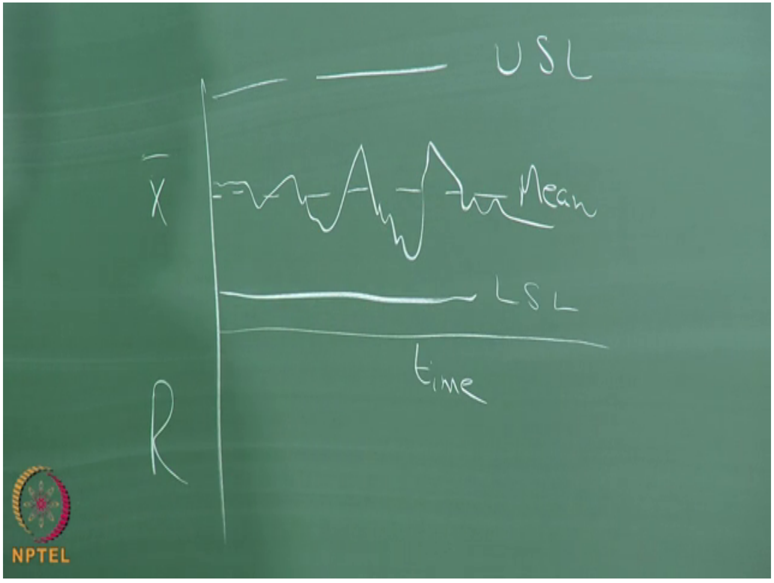

So the first thing you would ensure is it stays within specs. Because if it fails, exceeds the spec limits on either side you have to throw the product away. But more importantly you know, in an enlightened Six Sigma factory what they will do is they will be also measuring the range.

The range is the distance between max and min or sigma which is the standard deviation and that would also be monitored as a function of time. And in addition to spec limits, control limits will be established, U C L and L C L. The difference between a

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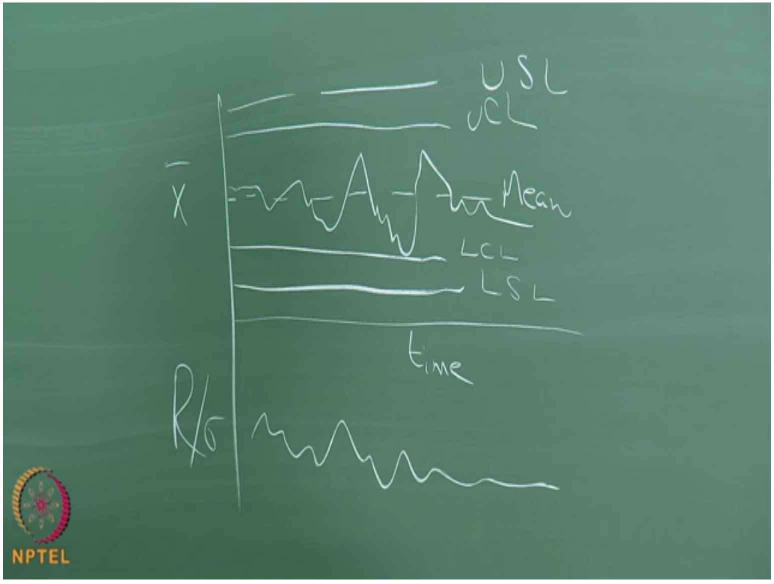

control limit and a spec limit is the specification limit is imposed by, typically the customer or the product requirements. The control limit is derived from the behavior of the process.

So typically the upper control limit would be set at x bar plus 3 sigma and the lower spec limit will be set at x bar minus 3 sigma and you would want to maintain your process within the control limits which should be tighter than the spec limits. So it provides you a safety band, you know.

If you can control your process within the control limits the chances that you will have occasional excursions beyond the specification limits are significantly lowered. So this S P C methodology is absolutely crucial to maintaining quality control over your product.

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So when people talk about Six Sigma quality, right, what they are talking about is this kind of methodology where you try to essentially maintain your process within plus or minus 3 sigma. But the incorrect assumption over here is sigma over x bar is much smaller than 1. 0:21:48.4 Mean this whole thing will,

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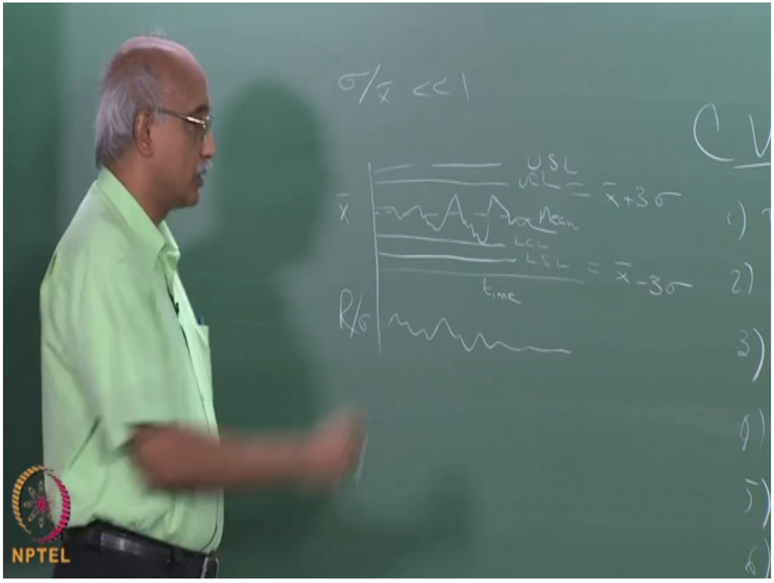

you know, break down if your standard deviation in the process is very high.

Because then your x bar plus 3 sigma can actually exceed your control limit and your x bar minus 3 sigma can exceed the lower control limit. And in fact typically in a well-behaved process the ratio

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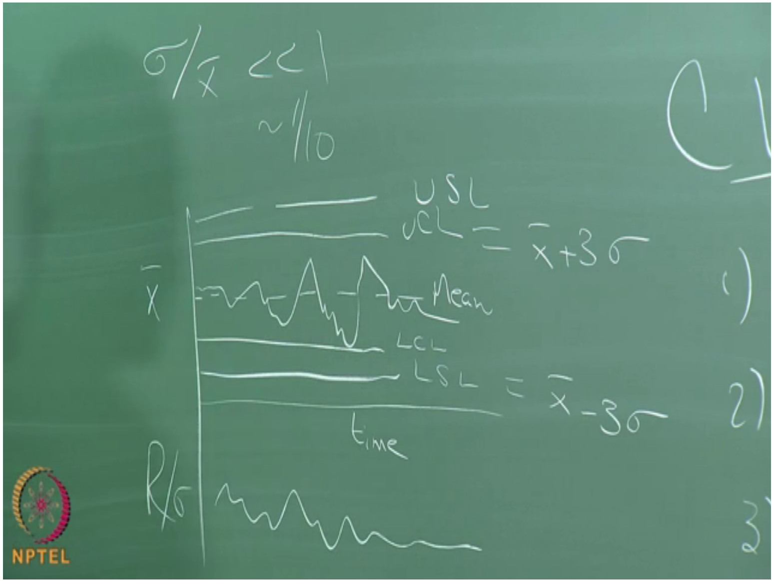

of standard deviation to mean should not exceed one tenth, Ok.

This parameter is called the variability coefficient and in a good process, robust process that is running under well-controlled conditions that ratio should never exceed point 1 or 10 percent. If you do that then obviously your U C L or L C L are going to be much tighter than the corresponding U S L and L S L and that is what you are aspiring for.

Now this is good but there is even a level of quality control that is tighter than this. And that says I do not care if my process is within the spec limits, I do not even care if my process is within the control limits, I do not want my process to drift from the mean.

As soon as I see even the smallest deviation from the mean where the process is supposed to run, I am going to take action. That is basically called trend chart analysis, so you do not wait for; you know for the readings to actually exceed one of these limits, you watch the trend.

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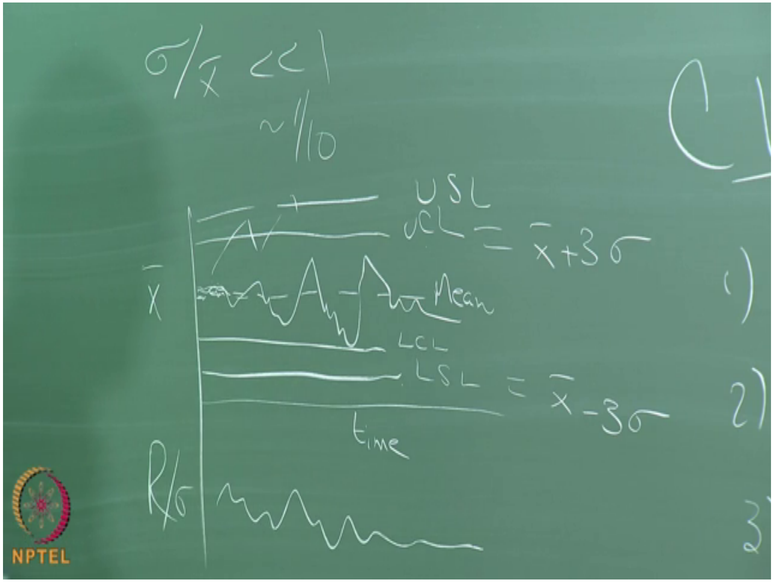

For example, if 6 consecutive measurements are on one side of the mean, you would probably think something is not right. You know, it is not random statistical variation, it is a systematic variation. And then if that happens you have to tweak that process to bring it back to the center line. So this is actually called the Taguchi approach to quality. It is called the Taguchi quality loss function.

Taguchi was a famous engineer, statistician who said that any time the product measurement deviates from the mean or its ideal value,

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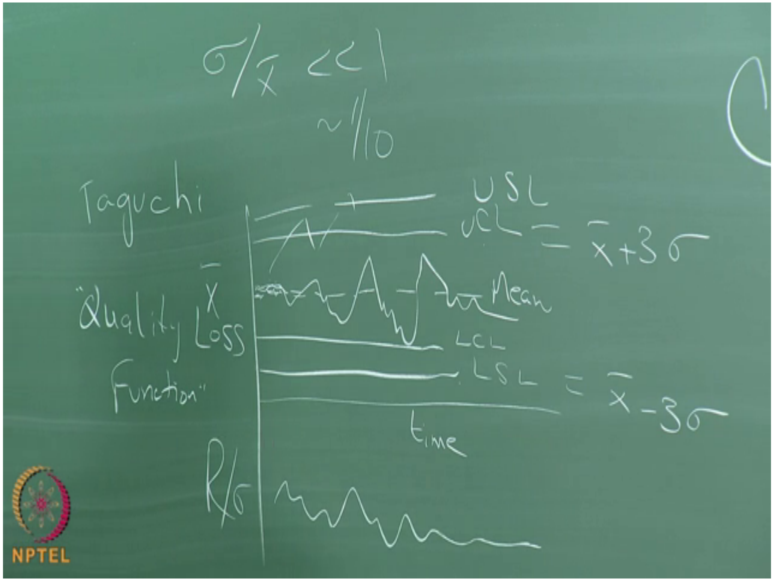

it is losing value; there is a loss associated with it, because the product is not as good as it can be. So he says any time you see any deviation from the mean value, treat it as the loss, add up the losses and try to minimize them.

So what he defines as quality loss function is the summation of all deviations from the process mean and he says in a good process, that should be zero or you should try to drive it to zero. In other words if you integrate and find the area between the trend line and the mean line

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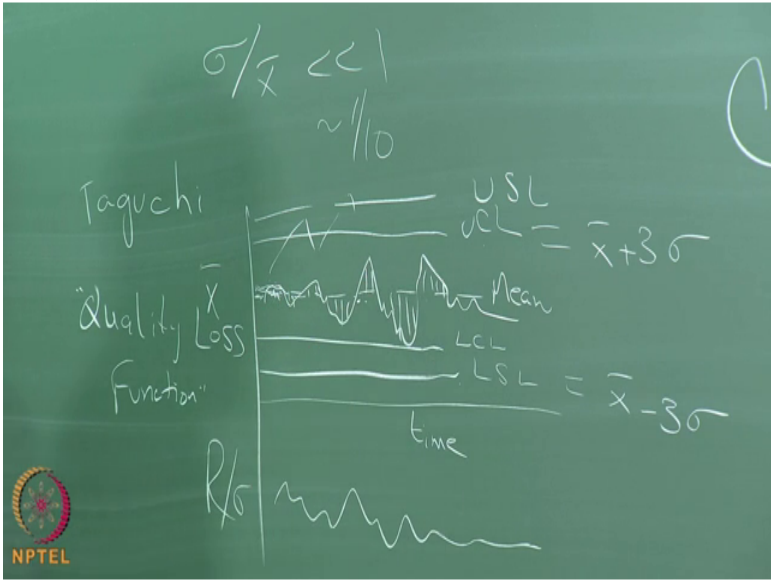

that integral should be driven to zero in your process.

That requires extremely tight process control. I mean somebody has to be sitting there, well not necessarily manually but at least you should have an ability to monitor how the process oscillates on essentially a real time basis and provide a real time reaction to continuously keep bringing the process back to the mean. So that is kind of the ultimate in quality control methodology.

Of course as you realize there is also cost associated with that. Because every time you tweak a process to bring it back to its ideal value somebody is taking some action, there is an intervention involved, there is a process disruption involved, all of that is cost, right?

So that is a cost, it is a cost of achieving quality and this is the cost of not having the quality and the two costs essentially must be added up to give the total cost to the manufacturer and you know, the most enlightened approach is not to say that I am going to minimize this, not to say that I am going to minimize the cost of tweaking, what you should try to minimize is the total cost of this plus the cost of tweaking to avoid this, right.

So that is the new evolution in quality control theory that is being worked on and some interesting findings are coming out because of this, you know, in terms of what kind of control methodologies are appropriate for what kind of processes, Ok.

Anyway the whole point is out of this list of 10 or more parameters, you have to identify which of the parameters is so critical that you apply this quality loss function methodology. Which ones are less critical and you can control them to control limits.

Which ones are even less critical and you can control them to specification limits and which ones are so non-critical, you do not even care, you know, you just take occasional offline measurements and as long as they show something good, you are happy.

So you have to go through the exercise of looking at all the measurable properties, identifying ones that are truly critical for what you are trying to achieve with that C V D film and applying appropriate measurement technologies and control methodologies for specifically those properties.

So that is all, you know discussion about how you use your measurements. But first you have to do the measurements, right? So how do you measure C V D films? The most common inspection technique is what, I mean, suppose you are asked to, you are given a C V D film and you are asked to measure it, what would be the first thing you do; any idea?

(Professor – student conversation starts)
Student: 0:27:37.5
Professor: No, that is way down the list. Look at it, look at it. I mean the human eye is one of the most sensitive detectors. It is amazing what eye can see that instruments cannot.
(Professor – student conversation ends)

Just like the human nose. You know human noses can detect part per trillion levels of things in the atmosphere. There is no sensor that is ever going to match the human nose in terms of its sensitivity. You can try and get close but even nano sensors or pico sensors or femto sensors that people are working on, you are never going to able to match the human nose.

Similarly you know human eye, particularly from a trained operator, you know, someone's job is to just sit and look at C V D films on substrate as they are passing by it is amazing how sensitive they can become. They can pick up very, very minute variations which are very difficult actually to measure quantitatively using any known tool. So visual inspection is the number 1 thing.

You have to continuously watch the product and make sure that, you know, it looks good. So the first thing is always a visual check.

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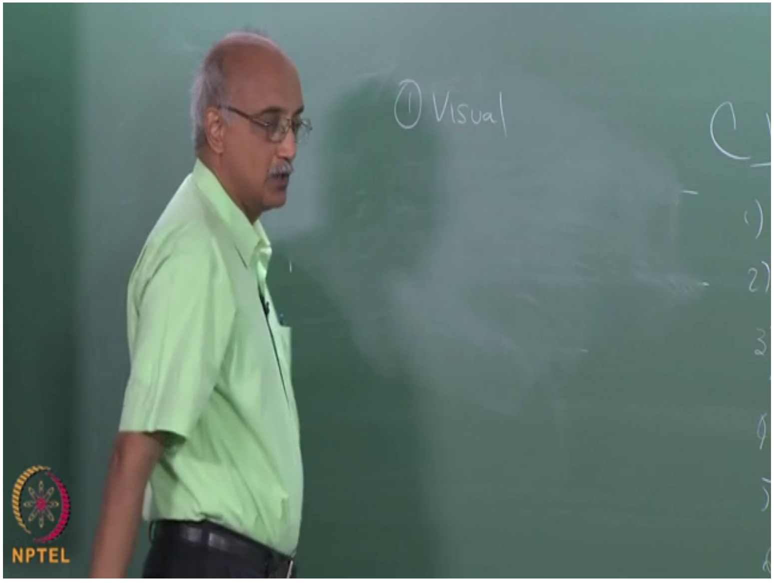

What is the second thing? Visually it looks Ok. What do you do next? You can 0:28:56.0 do one of two things. You can look it under different lights, right? So you can use bright lights, you can use U V light and see if something else shows up.

So lighting is an enhancement. So the lighting can be white light or U V light and so on. Typically white light shows up metallic materials very well, U V lights will show organics or fluorescent materials very well. So that is the next enhancement. Sometimes your eye cannot see things but with sufficient illumination it can see things.

And again the angle of illumination can play a role. You can have incident angles that vary. You can have light fixtures that kind of rotate around the specimen so that the illumination can be done in a 360 degree mode. What is the next thing you can do? Still looking good, you have looked at it, you have looked it under light, still looks Ok, what is the next thing you would think about doing?

Magnification, right? So that is where you start getting into microscope technologies. Because when you do

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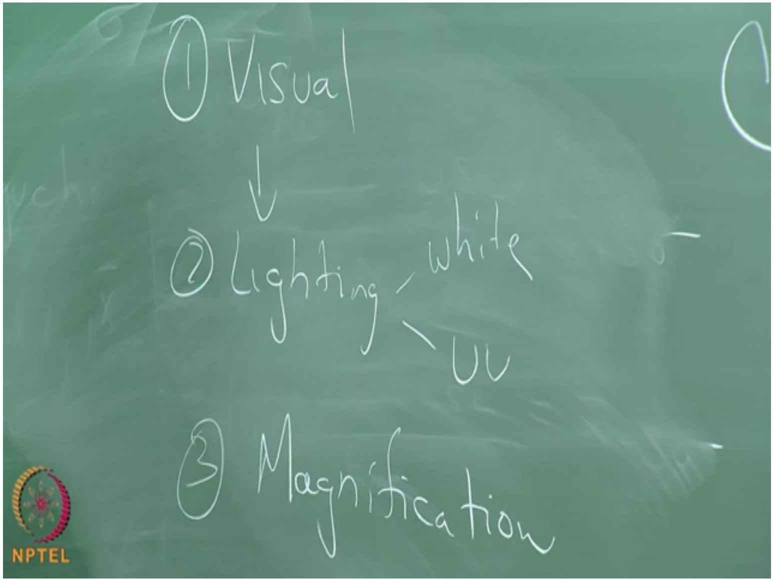

microscopy, even before you look at things like electron microscope, you first do optical microscopy.

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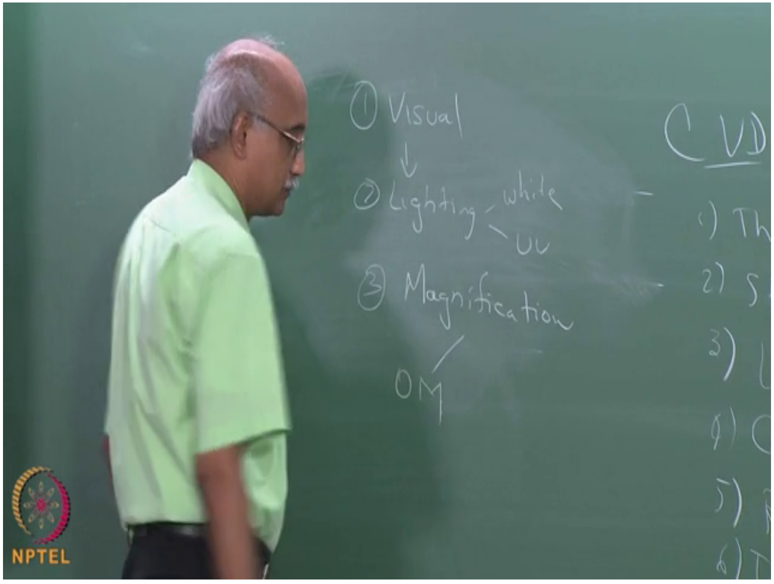

This provides some magnification, magnifying lens, magnifying glass typically 10 x but nowadays there are optical microscopes that can give you 10000 x magnification.

So that technology has certainly evolved to a point now where you are able to get fairly high magnifications but it is still going to be less than what you can get with an electron microscope. So what are the advantages, why would you want to use an optical microscope if electron microscope can give you much higher magnification?

The disadvantage of going to higher magnification is that you look at less and less area, right. To take a wafer and inspect it using you know one micrometer or even one nanometer resolution is going to take you a very, very long time whereas doing an optical magnification inspection at a lower magnification can be done much faster. So certainly speed is much better.

You can cover a much larger fraction of the total area. Typically electronic microscopic inspection can only be done on a small fraction of the surface and you assume that the rest of the surface looks similar. Whereas with an optical microscope, when lower magnification, potentially you can look at 100 percent of the surface. So that has that advantage.

The second advantage with optical microscopy, again it is a quick and dirty test. If there is some feature on the surface, it will actually show up quite well when you do optical microscopy. Of course you will be using again lighting as an aid, so you can do optical microscopy under bright light, you can do it under U V light and so on; it will show up different things.

It gives very good information on morphology. In other words by looking at a C V D film under optical microscope you quickly get a feel for what it feels like, what it looks like. It is not going to give you detailed information on the structure or on the dimensions but you will be able to tell, is it a crystalline C V D film, is it an amorphous film, is it well-adhered to the substrate? Is there any evidence of sliding between the layers, does it look like a pure surface or does it look like there are little impurities stuck in there?

So that kind of high level information is very easy to gather when you do optical microscopy. Of course complexity, cost all of these are issues that do not bother you when you are doing optical microscopy. When you are doing electron microscopy, first you need a trained operator, a person; an untrained person cannot do it. So you need to pay the person, right.

You need to pay the person for the training that he have undergone to be able to use an electron microscope. And secondly the equipment itself. In an S E M, scanning electron microscope, the samples have to be prepared in a special way and they have to be mounted into a vacuum chamber and you have to have a source of high energy electrons and you have to have a way of collecting the scattered electrons and tracking them and measuring them and so on. So it adds cost and complexity and it requires a trained operator to perform.

Where as in optical microscopy, someone who has, you know, who does not even have high school education can be trained to do it and typically operators on manufacturing lines who inspect products using optical microscopes do come from that type of background. But what they gain is experience. Once they have sat and looked at enough of the product they know what to look for.

And again their eyes get calibrated and they can actually pick up defects that even a sophisticated electron microscope cannot. So the optical microscope has several attractive features. The biggest disadvantage of course is the limitation in the magnification. Ok, so let us say that you have done that. Now what is the next step?

Do you immediately go to an electron microscope? No. Because at this stage you really have to think about what you are seeing. When you look at a C V D film under an optical microscope you have to, you have to grasp what you are looking at. Are you seeing things that look, let us say organic in nature or inorganic in nature?

Are you seeing things that look particulate in nature or fibrous in nature? Are you seeing things that look like defects on the surface or asperities on the surface or impurities or contaminants on the surface? So you have to be able to make these kinds of qualitative decisions by inspecting the surface using an optical microscope and then based on what you decide your further inspection paths will deviate.

For example if the optical microscope shows that you are mainly seeing inorganic materials and again the way you can tell that is, by using different lights and different illuminations,

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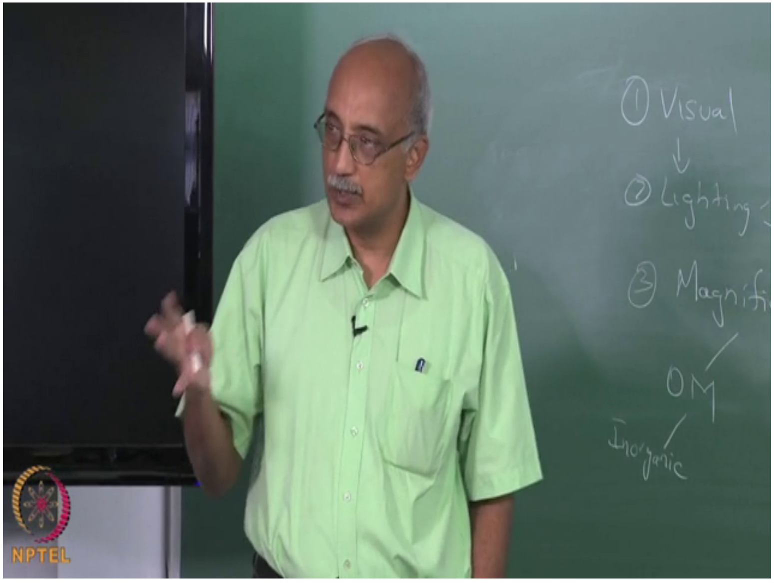

metal particles for example will show up very differently than non-metallic particles.

So using an optical microscope it is possible to make that assessment. So the other possibility is it could be organic and

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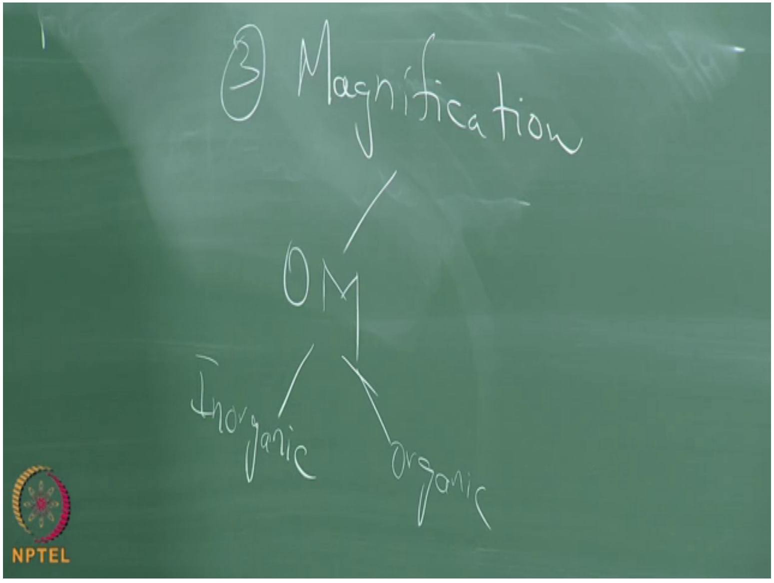

what is the other possibility, it could be fibrous. So depending on what you think you see under the optical microscope you will then go to the next set of instruments that you use. For example if you think that you are predominantly seeing inorganic materials then you would certainly think about using electron

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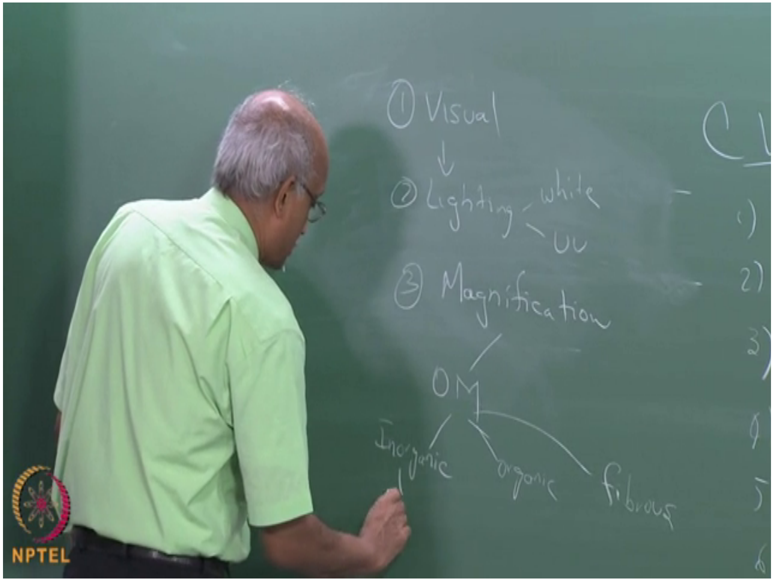

microscopy.

Whereas

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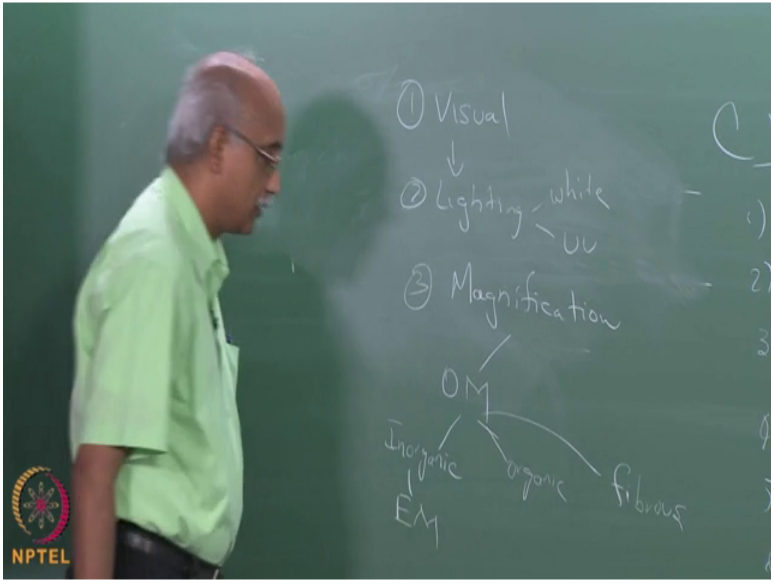

if you think it is mostly organic, what would you choose; any idea? How do you analyze organic materials on the substrate? Have you heard about infra-red spectroscopy? So typically I R techniques, I R

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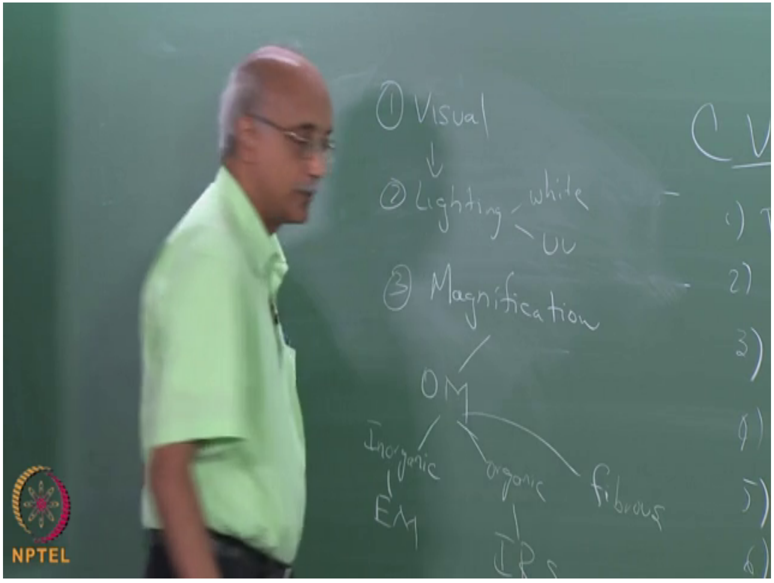

spectroscopy techniques will be used. If the material you are seeing has a certain shape to it, like for example it is fibrous, then what you do?

You do not, if it is that clear, I mean if you know that it is fibrous, then you do not even need to do additional analysis. You go, look for sources of the fibers, right? If the material can be identified quite clearly with an optical microscope, there is no need to do additional inspection.

Again another thing you have to remember, you know I said you cannot control what you do not measure, another truism or saying is that inspection is not a value-add activity. You have to minimize it. So any evaluations you do; measurements you do, always keep in mind that the customer is not paying for this. It is coming out of your own revenue, right?

If you can do zero measurements and still deliver high quality product that is ideal, not realistic. So you do have to invest in doing certain measurements to ensure that your process is in control. But again it should always be driving towards minimizing it. It is absolutely extraneous cost.

Your measurements are not what you ship to the customer. The product is what you ship. So all of your costs should go into making of good product. And as little of it should go into how you measure it, right? And so it is important to identify exactly what tests need to be done and do no more and no less.

So let us say that it is inorganic and you have

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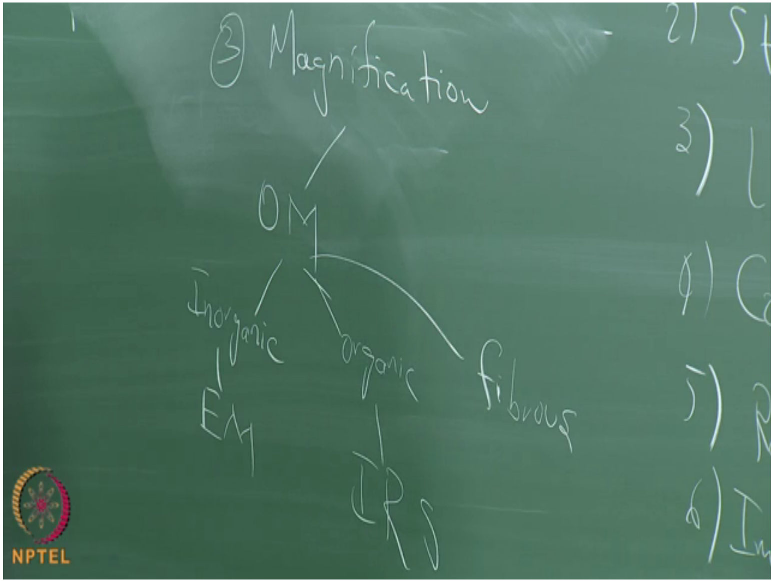

decided you need to do electron microscopy, then again there is bunch of choices you have to make. There is scanning electron microscopy, there is tunneling electron microscopy, there is Auger electron spectroscopy, there is secondary ion mass spectrometry, there is Time of Flight SIMS, there is, I know, there is a whole list of things that you could do.

Or you could do simple solvent extraction and do chromatography. For example, the inorganics are primarily ionic in nature, you take deionized water, measure its conductivity and then extract the ions from the surface and measure the change in conductivity of the deionized water. And that will tell you how much total ionic material there is on the surface.

And similarly if it is organic material, you have some decisions to make, to use F T I R, to use Raman microprobe, to use G C mass spec, gas chromatography mass spectrometry. So again there is battery of tests that you have to choose and decide which ones you want to run.

And also you have to decide whether, in terms of composition, elemental composition is sufficient or you need to know the exact species. For example if you have a silicon oxide film, if you simply do S E M E D X, you know it will tell you that you have silicon and you have oxygen. But it won't tell you, you have S i O 2.

For that you need to do additional tests to identify the actual composition of the species that it is S i O 2 and not some S i x O y, right? So it is like peeling an onion. You start with the film and you slowly peel it and you stop when you have done enough peeling, right. That is kind of the general philosophy that, that you have to adopt.

So when we talk about electron microscopy

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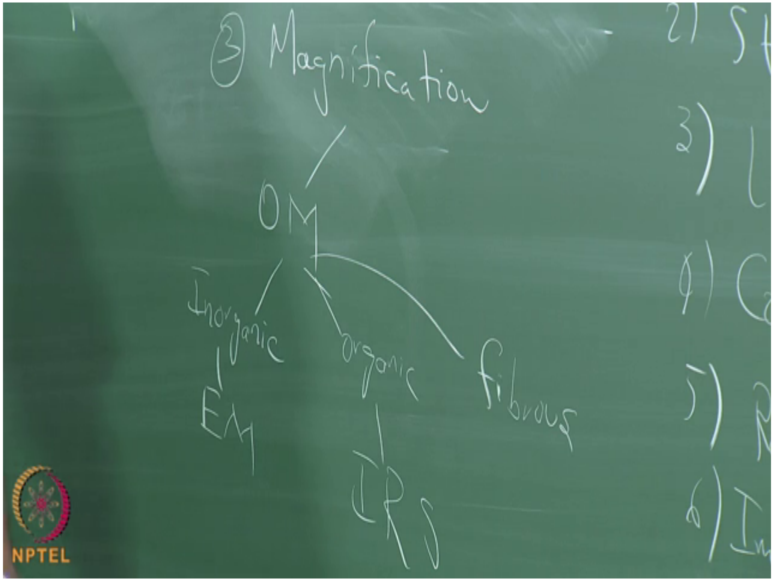

what are some of the considerations? How does electron microscope work? You basically bombard the surface with high energy electrons and they get scattered and by monitoring the scattered electrons you can get a picture of what the surface looks like.

So it is basically the simplest use of S E M is surface morphology characterization at high magnification and high resolution. Again the difficulty with the scanning electron microscope is that it is a time-consuming and a laborious process. You can only irradiate very, very small portions of the substrate at one time.

So to do a simple, if you take a C V D film, that is even a few Angstroms thick it can take you a day to do analysis on it. So it is not really a good use of a person's time to sit and do this analysis. Nowadays they have these automated S E Ms where you can essentially mount the sample and go home and it will do the analysis overnight and by the time you come back in the morning, there is an automated stage where the sample will move incrementally every few minutes and the entire sample will be scanned and characterized by the time you come back.

So these automated S E Ms are now making it a little easier to use S E M analysis to characterize the entire surface. S E M typically has a resolution of about 50 nanometers or so, 50 to 100 nanometers. If you want to improve it further, then you use tunneling electron microscopy where the electrons instead of simply being scattered from the surface, actually tunnel their way through the sample and they are detected at the other side.

This improves your resolution to about 10 nanometers or so. So it is much better than using an S E M. There are also high resolution S E Ms and T E Ms which, a high resolution T E M can give you a nanometer resolution in sample analysis. Because the drawback of T E M is that it requires some special sample preparation. To prepare the sample for doing the T E M analysis takes much longer than the actual T E M analysis itself. You also have to mount the sample on the metal substrate.

On the other hand, in a scanning electron microscope, because you are using high vacuum, you cannot stick organic materials into an S E M. So if you want to have an S E M analysis of an organic film done, you have to coat with metal, usually gold is used. Gold is buttered on to the material and then you can stick it inside an S E M chamber.

So the combination of S E M and T E M can certainly provide you with much better resolution but in order to get compositional information, S E M and T E M are not going to do it. So you attach it to an E D S or W D S setup which is energy dispersive spectroscopic analysis or wavelength dispersive spectroscopic analysis which essentially use X Ray spectrometry of the signal to identify the actual elements that are present at that location.

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They have differences in their sensitivity. The wavelength dispersive is slower but has better resolution than E D S or energy dispersive

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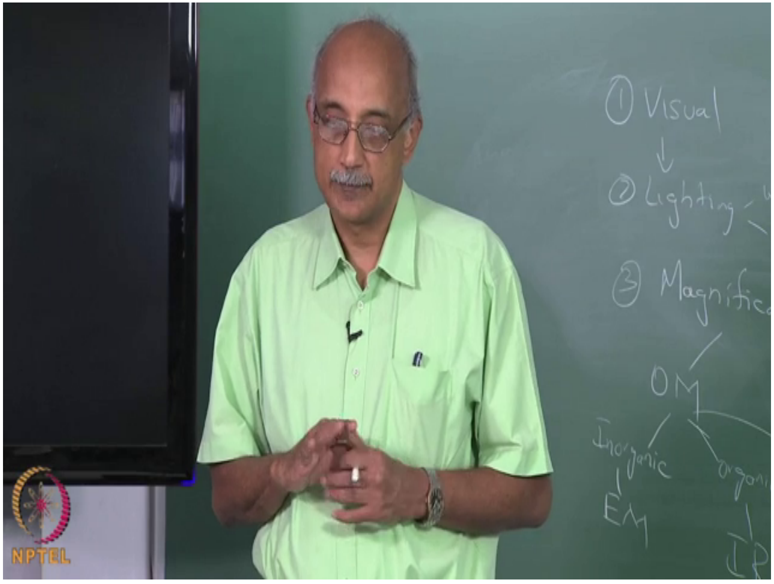

spectroscopy. There are also differences in the minimum atomic number that they can detect. With the W D S, you can detect up to beryllium where as with an E D S you can detect up to fluorine F, yeah so you can detect all elements heavier than fluorine or heavier than beryllium.

Of course nowadays both E D S as well as W D S apparatus are coming with, what they call a carbon window which enables it to detect all the way down to carbon. So the range of atomic numbers that we can detect using S E M and E D S slash W D S is constantly being improved and up to carbon is now detectable. So S E M, T E M are certainly possibilities for analyzing samples.

Under electron microscopy, S E M and the T E M are the most widely used. There is also a technique called Auger electron spectroscopy.

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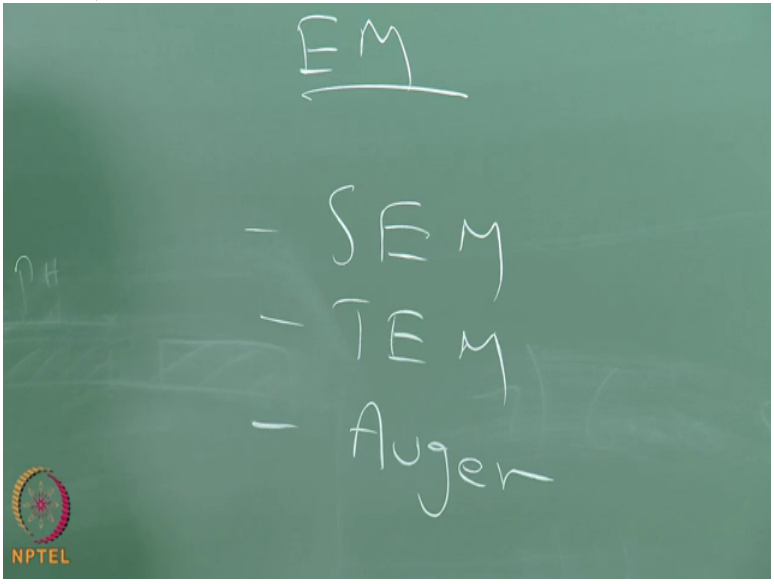

Auger electrons are the electrons that are knocked off from the first layer when you irradiate a sample with an electron beam. Auger analysis therefore can be used for sectional analysis of a C V D film.

So you would essentially continuously knock off electrons from various depths of the C V D film and do the S E M analysis and E D S analysis and so on. So it gives you depth profiling capability. So that is a big advantage of Auger spectroscopy. It is spelt A u g e r, not O J.

The other method that is frequently used is S I M S which stands for

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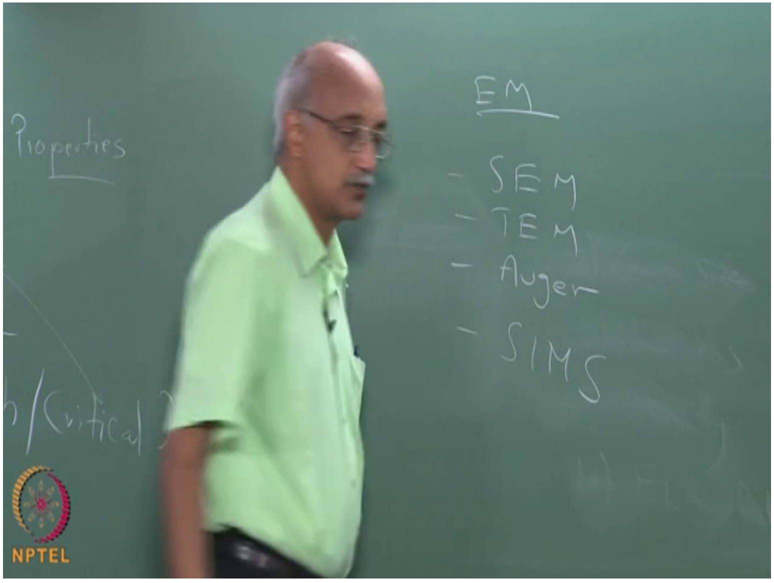

secondary ion mass spectroscopy. Here essentially you are using ions to knock off secondary ions from the surface but the secondary ions are taken through a mass spectroscopy column where they are spectrally resolved to obtain the atomic masses of the individual elements.

So this technique and another related technique called Time of Flight SIMS, TOF SIMS, they are kind of like Auger because they can give you this

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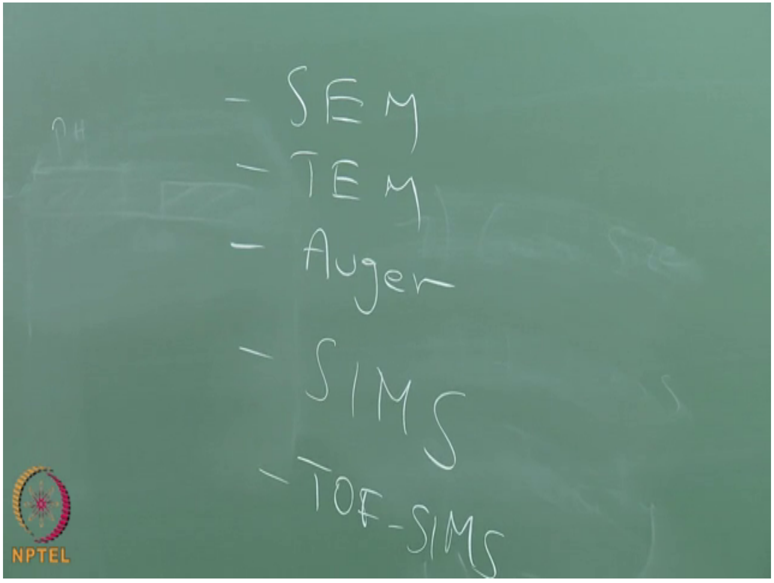

depth profiling but SIMS and TOF SIMS have better resolution than Auger spectroscopy. So if you want for example, Angstrom level resolution of a C V D film in terms of its elemental composition you use techniques like Auger, SIMS and TOF SIMS to be able to do that.

Another, so these are all methods that, by the way there is another one called LAMMA which stands for

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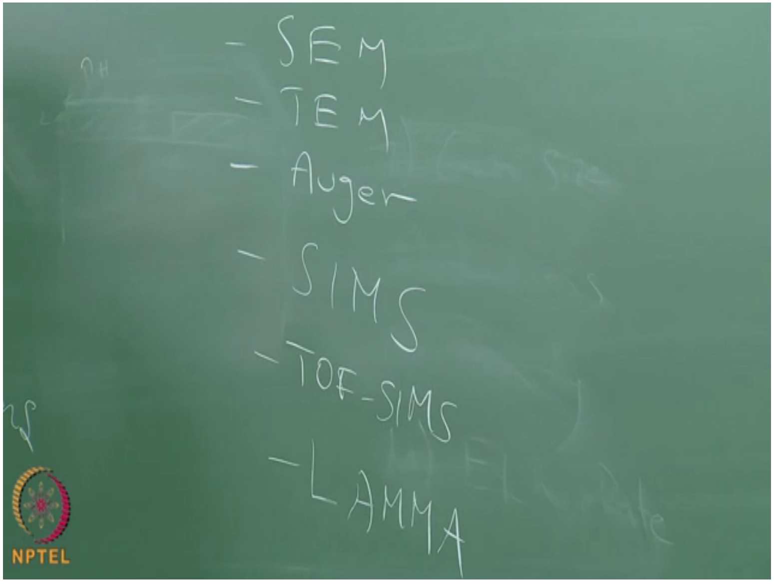

Laser Material Mass Analysis. This technique you essentially take the sample and you hit it with the laser and vaporize the entire sample. So you are not just doing knocking off of electrons, you are essentially vaporizing the sample and the vapors are then taken through a mass spectroscopic analysis and you detect the elemental composition.

So all these techniques are good for looking at which elements are present. But if you want to look at compositions in terms of products or species you have to use a different technique. Anybody know what is used to look for chemical species composition?

(Professor – student conversation starts)

Student: N M R

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Professor: N M R does not work for inorganics, it is, but for inorganics ... so far we have been talking about inorganic materials,

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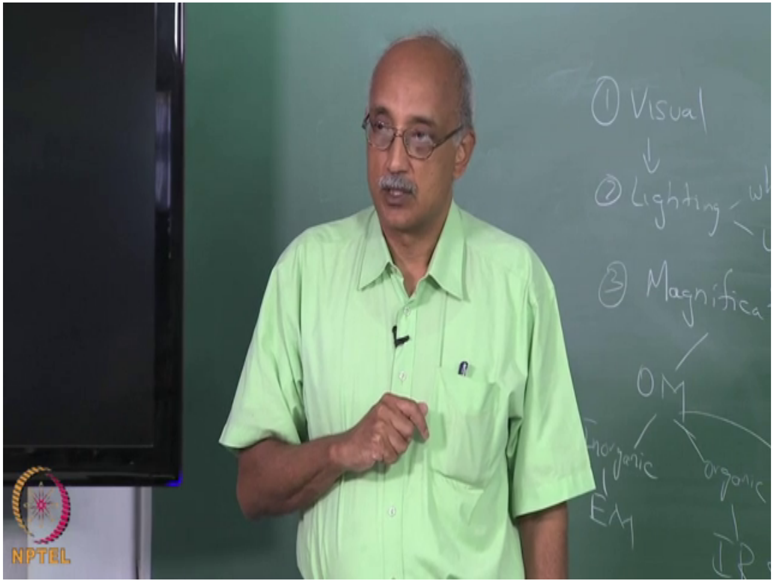

what is a good technique to use.

(Professor – student conversation ends)

ESCA, anybody heard;

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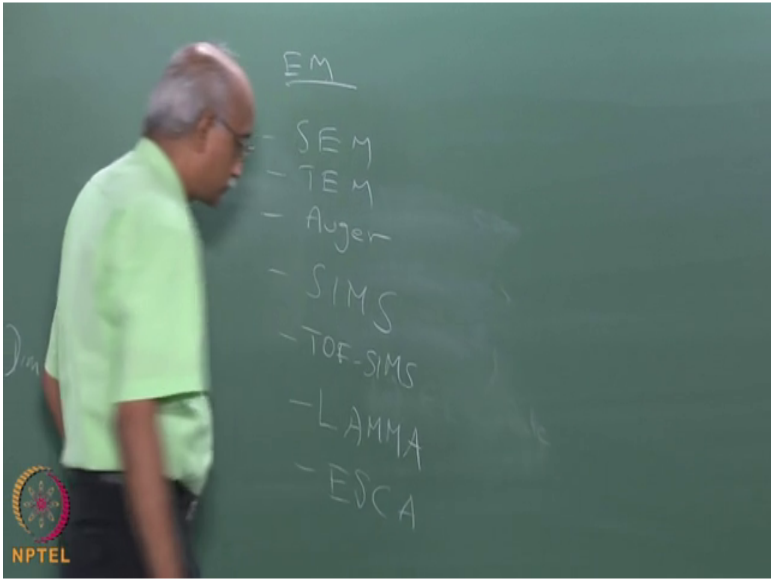

which stands for electron spectroscopy for chemical analysis, which basically impinges the material with X Rays, because X Rays scattering is a function of the molecular vibration levels and the molecular structure of the species that is present.

So by using ESCA you can actually get species differentiation. So you can tell whether silicon is present as S i itself or S i O 2 or S i 3 N 4, right, so instead of just knowing that S i and N are present, you know that they are present in the form of S i 3 N 4. Obviously when you are making C V D films that is very important.

You do not just need to know whether the elements are present, you need to know whether they are present in a particular stoichometry. And so ESCA is a workhorse particularly in the C V D industry. And in fact as far as chemical speciation information is concerned this is probably the only technique that works very well although certain types of materials can be analyzed by another means.

Ok so let us stop at this point. The next lecture we will start with methods for analyzing organics on surfaces and then we will also talk about methods to measure thickness and so on, any questions? Ok, see you at the next lecture.