Particle Characterization
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Module No. # 10
Lecture No. # 27
Chemical and Compositional Characterization: Analytical Methods

Welcome, to the 27th lecture in our particle characterization course. In the previous lecture, we reviewed a few chemical processes that particles play a key role - in particular burning of droplets and solid particles, dissolution, and sublimation are some of the processes we looked at. I mentioned at the time that the rate of such processes is governed by the physical properties of the particle; in particular, the size as well as the chemical nature of the particle.

So, in this lecture we are going to begin this by discussing various methods for chemical and compositional characterization of particles. Now, you really cannot do chemical analysis of particles without simultaneously doing physical analysis also; for example, any analysis at a single particle level requires that you first capture it in some viewing volume and then use certain appropriate technologies to characterize its chemical makeup.

But, as you do that you are also inspecting the image of the particle. So, the physical characterization goes hand in hand with chemical characterization; in fact, you can do chemical characteriza[tion]- or you can do physical characterization without doing chemical characterization, but not vice versa; you really cannot do chemical characterization without simultaneously also doing physical characterization.

When we look at chemical characterization of particles - just like - for example, when we are talking about shape analysis and size analysis there are really broadly two classifications of such techniques: one is bulk methods and the other is single particle methods.

In order to characterize the composition of, let us say, a powder of particles that you have you can choose to - either inspect the particles one at a time, or you can choose to characterize the entire population of particles using some method.

Now, such bulk methods for chemical characterization require that you dissolve these particles completely in some medium and then analyze the medium to obtain the elemental as well as the compound characteristics of the particles that were in the powder to begin with. So, you would typically take a strong acid and completely dissolve your particle population in that acid and then perform your analysis on this analyte - the liquid medium in which the dissolved particles are now suspended.

Once you have actually dissolved the particles into a liquid further analysis can be done using conventional means such as ion chromatography, i c p, atomic absorption spectroscopy and so on; but, the difficulty is in preparing the sample, because the really critical two requirements - the medium the solvent that you are using for dissolving the particles has to be very pure and it must be pre characterized before you start analyzing the solvent with particles dissolved in it, and the second thing is all particles must be fully soluble in the solvent.

Now, when you have a homogeneous chemical composition of the particle population then that is possible; even there, it may be difficult if you have a large range of particles sizes, because as we saw yesterday the dissolution characteristics are also size dependent. So, if you have a particle population with a very broad range of sizes the finest particles may dissolve completely, but the larger particles may not; so, it may require a lot of time and a lot of energy to achieve complete dissolution of all particles in a solvent even if they are all chemically similar.

If you have a particle population that is also diverse in terms of the chemical elements that are present then it becomes increasingly difficult to identify a solvent which will dissolve all the constituents equally well. So, the bulk methods of analysis are kind of crude methods that can give you an idea about the overall composition of an assembly of particles, but it is clearly not what you would want to use if you require more precision in your data.

If you really want to know, with a little more accuracy, regarding the composition of the particles that you have, you have to resort to single particle analysis. What are the difficulties in single particle analysis? The first thing is that a bulk powder could contain millions of particles; so, how many are you going to analyze? How do you set some guidelines for how many particles are sufficient number of particles?

If you have a particle powder that consists of, let us say, one million particles, is it enough if you analyze one particle and assume that it is a homogeneous distribution? Or do your statistics improves significantly if you analyze 10 particles, 100 particles or 1000 particles? I mean obviously the more particles you look at the greater confidence you will have in your data.

The confidence level in your analysis will keep increasing with more and more particles being sampled; but, the problem is the time that you have to put into it, the cost of analysis - everything starts to ramp up. Actually, nowadays, there are what are known as auto devices; essentially, you can take a sample, mount it overnight, and the system itself will do the scanning for you; by the time you come back in the morning you will have data on 10s of 1000 of particles. These types of auto S E M, auto T E M type of attachments are now available, but they also come at a cost. So, the biggest drawback to single particle analysis is the statistical unreliability of data if you are only sampling a few particles in your analysis; the more that you can sample the better.

The other problem with the single particle analysis is capturing it; this is something we talked about a little bit in one of the earlier lectures, that before you can analyze a particle for shape or size or composition you first have to immobilize it on some surface so that you can inspect it using various methods; so, in terms of sampling of liquids - the method that is most commonly used is filtration.

So, you take a filter and capture particles on it and then subject the filter medium to subsequent analysis; but, you have to be very careful with the type of filter that you use - there are essentially three types of filters; depth filters, which are essentially fibrous filters which filters particles primarily by the fact that there is a depth of bed that the particles have to make their way through and as they do the fibrous surface area - contact area - is sufficiently large that all particles will get captured in this filter; it is a wonderful method for very effective filtration, but the problem is if you are going to do subsequent analysis of the filter this type of an irreversible process is actually not very good for you, because you cannot see these particles any more they are hidden in the depths of the filter. By definition, a depth filter is not good if you are collecting particles for subsequent analysis.

The other type of filter that is widely use again because of its high filtration efficiency is a porous membrane filter; here, essentially you take a polymeric membrane and you have porosities in it and the pores, once again, have sufficient (( )) in them that particles will get captured with virtually hundred percent efficiency. There is usually a pore size that determines the cutoff and all particles that are smaller than that size will get captured within the pores and the larger particles will not make it through; so, it is kind of an absolute filter in that sense - it will give you a very clear separation by size; but again, the difficulty is, once the particles get into these membrane pores you cannot bring them back out for doing analysis.

The third type of filter, which is the one that is widely used for microscopic analysis, is a flat membrane filter with discrete holes on it; here, essentially there is only a single layer that the particles can adhere to - there is no depth; so, any particles that is captured on the filter is sitting on the surface so it is very easy to analyze it.

A good example of this is the nuclear pore filter, which is manufactured by a company called Millipore who also make water treatment systems and so on; but, they also have good filtration technologies which they use in their water purifiers; the nuclear pore filter is probably the one that is most widely used for capturing particles for characterization - whether for physical or for chemical characterization.

Now, the thing though is when you use a polymeric membrane if you are just doing optical microscopy, it is fine; but, if you are doing scanning electron microscopy or tunneling electron microscopy, as we will discuss later, they involve extreme environments - high vacuum, bombardment with electrons, and many of these polymers will just start burning when you expose them to that condition.

So, what you need to do is, essentially, coat these polymers with typically a conductive metal coating - usually gold, in order to protect the membrane itself from burning up. So, that adds complexity; also, if that coating material is close enough in its elemental characteristics to the material that you are analyzing it can actually interfere with your measurements.

So, coating is a necessary evil; especially, if you have light elements they frequently get occluded by the coating that is put on these membranes to prevent them from burning up; alternatively, you can use metal filters - there are aluminum filters that are available

which would not have this problem; they would not burn up if you put them in a scanning electron microscope. But, there the drawback is typically these metal filters cannot be procured in as flat and smooth a condition as a polymer filter; so, you kind of sacrifice resolution, because if you have a rough filter media then fine particles can hide in the roughness elements and you may not be able to detect them with any kind of accuracy or precision.

So, filtration is the most widely resorted to method to prepare particulate samples for analysis. In terms of gases, you can either use filters or you can use impactors. As we discussed in one of the earlier lectures, the way that impactors work is essentially by using the inertial characteristics of the particles to capture them - so, essentially you have a series of chambers in which particles are accelerated with progressively greater velocities and what happens is that you basically capture the largest particles in the first stage and then finer and finer particles downstream; now, that is used for - in the previous application we discussed in the context of size analysis, but it can also be used for compositional and structural analysis.

The advantage of this method is, as you accelerates these particles through these fine nozzles they are collected on a very small area - typically right at the center; so, you are actually concentrating your particle sample in a much smaller area compared to filtration. That is a huge advantage of inertial impaction over filtration, because in a filter the particles will be captured everywhere on the filter surface.

So, depending on the size of the filter you may have a fairly large area to go in and analyze later on; whereas, with an inertial impactor all the particles tend to collect around the center; in fact, what you can do is - for the collector plate - you can actually use the same coupons that are placed in the s c m chambers; they are called the stubs - actually called sticky stubs; they are very high adhesion coupons that are placed in the centers of these inertial chambers.

So, as the particles are accelerated they will hit these coupons get captured and then you just take this coupon and put it directly into the s c m; that really minimizes all the handling, coating and other issues that you deal with in terms of filtration - that is a huge advantage. The draw backs to this method - not very good for very fine particles; I mean, if you are talking about nano-dimensional particles this method of impaction would not

even work, so instead you have to go to the diffusion cells that we talked about earlier where you collect particles based on their diffusional characteristics, but other principles remain the same; instead of inertial impactors you use these diffusion batteries.

The other disadvantage is that there can be losses of these particles if they are not properly directed - you know they can be wall losses, there can be - nozzle itself can get occluded after while; so, if there is no good control or stability of the incoming sample that can affect the control and stability over the sample that you collect for analysis as well; but, regardless, it remains a popular method for collecting particles for analysis.

Let us say, that you have decided that you want to do analysis at single particle level and you have collected the particle either using filtration or impactions or diffusion or some other means - how do you now proceed for your analysis? Is there a logical sequence which will essentially minimize the number of analysis steps that you take and get you the maximum information the shortest possible time?

Well, it turns out that there is a logical path that analysts use when they obtain a sample for analysis; let us say, that they receive a filter with particles on it - what is the first thing they do? Naked eye inspection - now before you start using any fancy instruments just look at it; again, if you are an experienced analyst, that initial look will tell you a lot about what type of particle it is, what it is, even its chemical nature - it all comes with experience.

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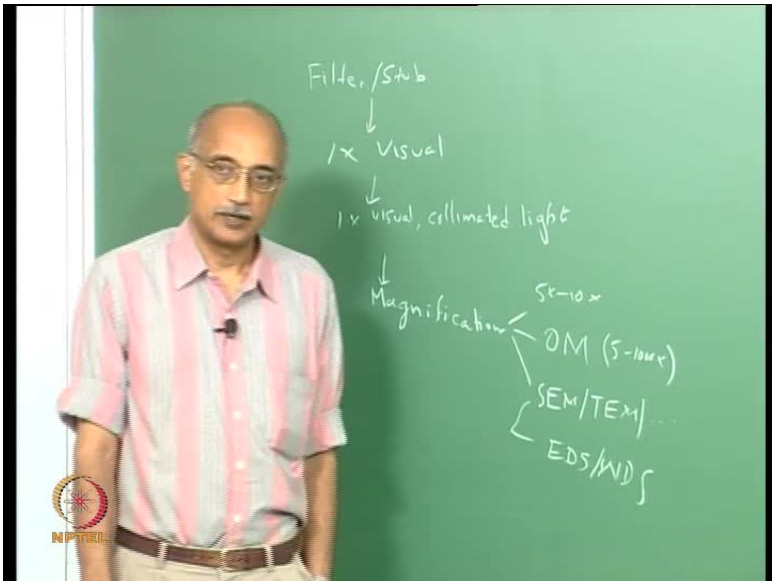

The sequence that you would normally follow with - look like this; so, you bring your filter or your stub or whatever that you have for analysis and you do a simple 1 x visual - somebody just sitting and looking at the filter to see what does the particle look like, what are its obvious characteristics, what are that conclusions we can draw just from looking at it; is it luminescent? Is it florescent? Does it look like a metal? Does it look like a nonmetal? Lot of these - your eye can actually do a very sensitive job of classifying.

The next step is again 1 x visual, but with collimated light. What we mean by that is, a high intensity light beam that is shone on the sample - particularly at a gracing angle; when you do that your sensitivity of detection increases several orders of magnitude.

Simply keeping your sample on a stage and then shining light on it at an angle; basically, that is what we mean by that and that can give you a lot of additional information about what the sample is.

The next step is inspection at magnification. The magnification again should be - it should start with the lowest magnification and slowly increase the level of magnification; the reason for that is, at low magnification you can see more of the sample as you increase the magnification, yes, you can see things in finer detail, but your field of vision is also narrowing; so, you typically start with something like a 5 x to 10 x magnification, which can be done with the simple magnifying lens and then you step it up in magnification using an optical microscope - an optical microscope can have magnifications ranging from five to as much as one thousand x and of course later on if necessary you can go for S E M, T E M etcetera.

But, at each stage you should stop and ask yourself - do I need to go to the next stage? For example, if at 5 x to ten x magnification you are already able to classify the particle in terms of its physical as well as chemical characteristics there is no point in going any further; if you are not able to make a judgment at that stage, then you go to the next stage and do optical microscopy where you are essentially increasing your magnification.

Now, the limitation of optical microscopy is that it does not give you any chemical information; an optical microscope will only blow up the image and show the details - the physical details - at much greater magnifications. So, you can really do a good job of characterizing size, shape, morphology characteristics, little bit of even the structure -

you know you can say whether it is an amorphous or a crystalline material and so on; what you cannot say is what is its chemical composition - you cannot say that this contains elements so and so or chemical compounds such and such.

That information is not available to you in optical microscopy; in order to get that type of information you have to go to something like a scanning electron microscope or a tunneling electron microscope, but even these instruments only give you even higher magnification imaging, they still only tell you morphological information.

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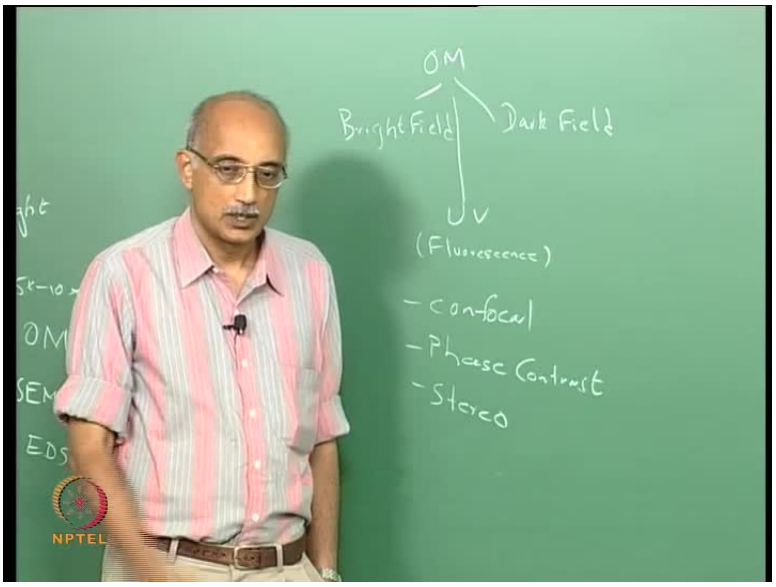

In order to get chemical information from these instruments you have to combine them with E D S or W D S - that is, energy dispersive spectroscopy and wave length dispersive spectroscopy.

Now, at each stage you also have to think about - I mean the magnification is not your only tool, lighting is another major tool that you have. For example, when we talk about O M we can do the optical microscopy in essentially 2 modes as we saw earlier - that is, the bright field microscopy and dark field microscopy; and there is also U V microscopy, which is also known fluorescence microscopy; we have discussed these in a little bit of detail earlier on. In bright field microscopy, the surface is bright and particles on a surface appear dark - so if you are using this to look at a filter the filter itself will appear as a bright image and the particles that you have captured on the filter will appear as dark spots.

Whereas, in the dark field it is basically reversed - the surface is a dark and the particles that are sitting on the top or bright; the reason that you want to use one or the other - to some extent it depends on the nature of the particle and the nature of the surface - you have to choose a technique that gives you the best differentiation between loose particles on the surface versus surface features like dimples or grooves on the surface and so on.

The method of analysis has to be able to clearly distinguish between surface features and particles that are loosely adhered to the surface; the u v fluorescence method is used when you have essentially particles that can fluoresce; typically organic particles - many of them have fluorescence characteristics, so if you have essentially - you turn off all the lights - have a dark room or a dark box and you shine u v light these organics particles will really light up; these are variance of microscopy that can improve the resolution and sensitivity of your measurement. There is also something called the confocal microscopy - in a confocal microscopy, essentially, you try to minimize loss due to scattering; so, you shine the light through holes that are enough a fan that is rotating

The plane of the incident light is kept to a very small dimension at narrow plane so that you can minimize scattering of light from the surface when the light shines on it. There is another variant of microscopy called phase contrast microscopy, which is particularly useful in identifying subtle differences between various phases of the same material that is present in the sample. The stereo microscopy, which essentially uses two different eye pieces with different focal points to improve the resolution of your measurement.

If you look at any standard book on microscopy there are literally dozens of variance on the basic microscopic technique, but the underline principles remains the same - you are shining light visible light on an object and you are looking at it under an eye piece; it is a very physical method - I mean you are seeing what you are observing.

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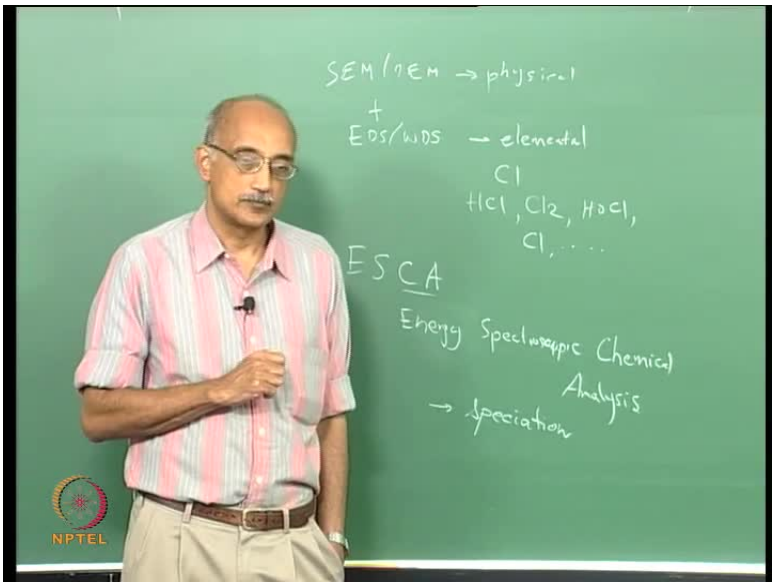

Now, when you go to methods like electron microscopy, the principle that it is based on is very different; whether you are talking about S E M or T E M, the primary principle here is you are actually bombarding your sample with a high energy material - in this case, electrons; I mean, you do that and you knock of electrons that are present on the sample to begin with; these are called the secondary electrons.

By capturing these secondary electrons with c c d or a photo multiplier, you can actually reproduce an image of the particle itself; so, when you use an S E M or T E M the intensity of the electron emission really decides how bright the sample is or how clear the image analysis is in terms of its physical structure.

The advantages of S E M and T E M are high magnifications - you can go up to 50000 x plus high resolution, scanning electron microscope can resolve down to about 15 nano meters, a tunneling electron microscope can resolve down to 1 nano meter; the difference between the two is - in a scanning electron microscope the electrons hit the surface and bounce off, in a tunneling electron microscope you essentially send the electrons through the sample; but, T E M requires much greater sample preparation because of that - you have to really produce a very thin section of the sample and you have to allow the electrons to burrow through it. The sample has to be mounted on a metal filter, in fact with T E M the initial sample preparation is a difficult part; once you have prepared the sample then the analysis is not very different from S E M, but because now you are

taking into account only the scattering characteristics of the surface but also the transmission characteristics it improves the resolution of your technique down to nanometer levels.

Both S E M and T E M do require high vacuum, so that does drive up the complexity of the analysis - you need a trained operator to do S E M/ T E M analysis.

You also need to coat the sample, which has been discussed earlier; it can be an intrusive effect as well; but, S E M and T E M are really the work-horses of the high magnification microscopy industry. Any characterization that you are that you do, particularly in the submicron to nano size range, you always start with S E M /T E M.

Now, there are a couple of other techniques that are widely used for surface characterization, atomic force microscope and of course x r d x-ray diffraction, which is used to characterize crystal structure; now, these two are extremely important techniques so we will deal with them separately; the next lecture we will devote to just talking about a f m and x r d methods of particle characterization. When we look at S E M /T E M, as I was mentioning, this only gives you the physical image; when you combined this with E D S or wavelength dispersive spectroscopy this gives you the elemental composition; essentially, in this case, you look at the x-rays that are being emitted by the sample in response to the electrons that are impinging; the x-ray emissions are characteristic of the elements that are present and that is how you get the additional information regarding the elemental composition, but it is still only elemental.

Now, what do we mean by that? Suppose, you have a sample on which you detect, let us say, chlorine; but, you do not know if the chlorine was present on the sample in the form of H C L or C l 2 or H O C L or C L - there are so many forms in which chlorine element can be present and E D S or W D S really will not tell you which particular chemical form it was present in.

For metallurgical applications that may be sufficient information, but for a chemical engineer it may not be; you may want to know exactly if it is present as H C L or if it is present as C l 2 or if it is present as H O C L or present as C l; so, clearly this method is not going to give you that information.

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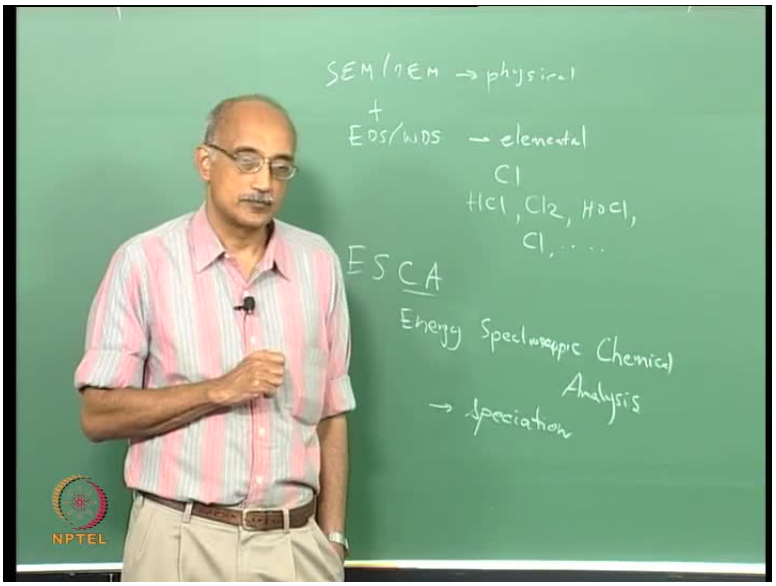

By the way, the energy dispersive spectroscopic analysis, the big advantage is that it is much quicker; in fact, most S E M (s) /T E M (s) that we use come with E D S as the attachments for doing the elemental analysis; however, W D S actually has much better resolutions, sensitivity - this can actually detect down to the beryllium element, whereas an E D S typically only detects down to fluorine.

Light element detection is much better with wave length dispersive spectroscopic analysis; although, now a days they do come with something called carbon window, which enables them to now measure elements down to carbon which is still the lightest element that you know people are able to get to with microscopic analysis.

How do you then analyze for chemical composition? If you want to know exactly what the species - is that doable? Is there an analytical technique that can enable us to do that?

When we talk about particles and understanding their composition, particularly inorganic particles, the only known method that works is called ESCA - ESCA stands for Energy Spectroscopic Chemical Analysis.

In this method, what you are doing is instead of irradiating the sample with \ electrons you actually irradiate it with x-rays; again, the nature of interaction between the incident beam and the sample, when you use an x-ray the interaction becomes highly dependent on the molecular structure of the material; not just the chemical element that are present,

but actually how these elements are put together or link together to make a chemical compound.

When we look at the impingement using x-ray and then you analyze the emitted signal using spectroscopic methods that is known as ESCA and this is the only method that is capable of providing speciation - that is, resolution of what chemical species the various elements are present in. ESCA is a very special tool that is used primarily in chemical industries where the precise nature of the chemical compound - whether it is stoichiometry or not - you know all those issues become important, then this is the method to use.

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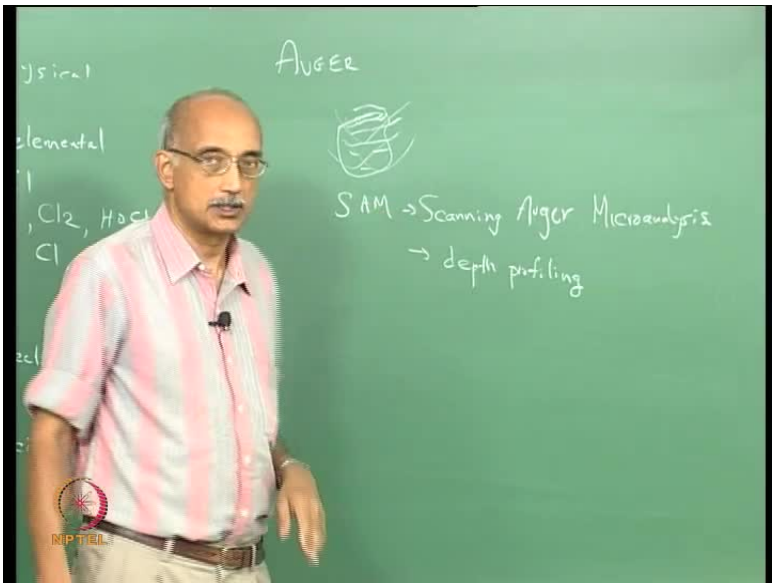

Going back to S E M and T E M though, as I said they are the work horses; now, S E M can be used in various modes - there is something called AUGER spectroscopy; an AUGER spectroscope is very similar to a scanning electron microscopic method except that you control your radiation, your incident energy transfer, such that you only knock off electrons from the first few layers of the particles - the first few nanometers of the particle - or in the case of a nano particle you can actually tune your AUGER to only knock off particles from the first few angstroms of the particles.

If you have a particle that looks like this, normally when you do S E M /T E M you are essentially knocking off electrons from all over the particle; in AUGER you control the

emissions such that the electrons only have sufficient energy to knock off electrons from the first few mono layers of the particle or surface that you are looking at.

You can actually characterize just this part of the particle instead of - as we had discussed earlier the surface and subsurface. AUGER spectroscopy enables you to characterize just the surface and the subsurface without penetrating into the bulk of the particle.

What is the advantage of the AUGER method? Now, you can actually think about sectioning the particle - you can get a sectional characterization of the particle; you can knock off electrons only form this layer and then tune it so that now you knock off electrons from this layer, this layer and so on.

You can actually start getting depth profiling; that is a very valuable attribute of AUGER micro analysis or AUGER spectroscopy; in fact, there is a technique called SAM - Scanning Auger Microanalysis, which essentially does that; it scans the particle at various energy levels so that the various sections of the particle get exposed and analyzed.

SAM is a method that widely used for depth profiling. In AUGER spectroscopy, all you are doing is knocking of electrons form the surface and you immediately visualize them, capture them and develop an image of the surface; or you can combine this with E D S and W D S to obtain chemical elemental information layer by layer, but actually there are some other interest in things that you can do with this technique.

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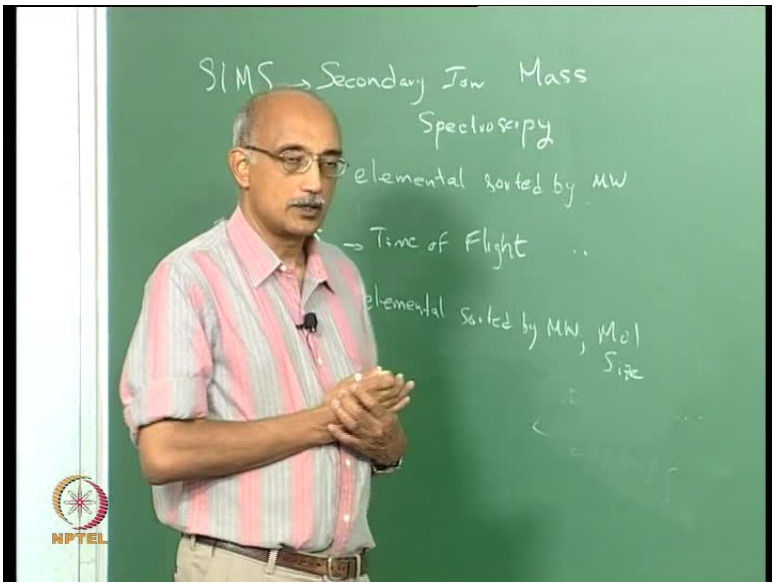

There is a method known as SIMS, which stands for Secondary Ion Mass Spectroscopy; in Secondary Ion Mass Spectroscopy you use something very similar to AUGER - you essentially control the depth of interaction between the incident electrons and the scattered electrons and ions, but now you actually take the scattered ions and you accelerate them to a mass spectroscopy column just like a gas chromatography (( )) which is commonly used for organic material analysis.

Essentially, by taking the knocked off ions through a column and by looking at the rate at which various atoms are progressing through the column - while looking at their transport rates - you can actually calculate what their molecular weight would have been. As with any mass spectroscopy techniques you are essentially classifying the particles based on their molecular weight as the primary characteristics; this gives you elemental data sorted by molecular weight.

A variant of this is a method called TOF-SIMS, which stands for Time of Flight Secondary Ion Mass Spectroscopy; the difference between TOF-SIMS and SIMS is that even before the knocked off ions enter the spectroscopic column you look at the time that it takes for these ions to reach the column, then you also look at the rate at which they are transported through the spectroscopic column.

The time of flight gives you additional information about not only the molecular weight but also the molecular dimensions of the various elements; so, here you can get

elemental data sorted by molecular weight, as well as, molecular size; this additional bit of information can enable us to again more accurately and precisely resolve what are the various elements that are present in the sample.

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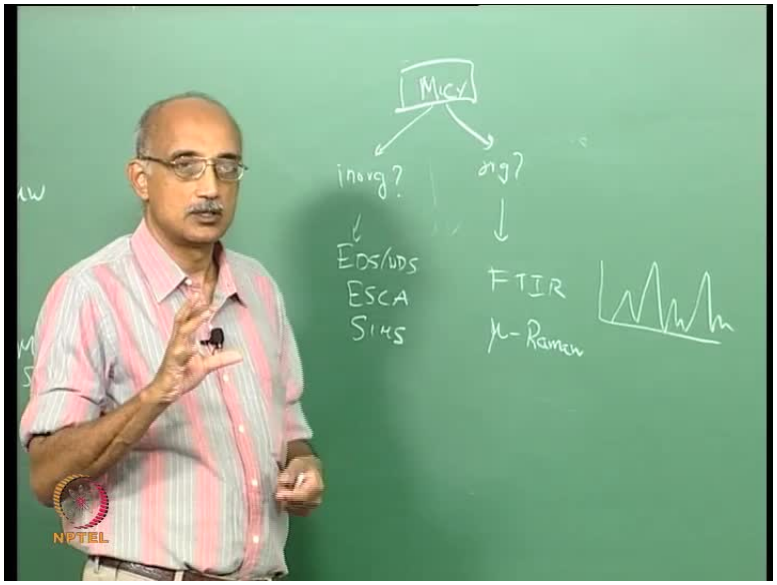

The techniques that we have been talking about so far for chemical or compositional analysis are primarily used for inorganic particles. Now, it is not always the case that you are measuring only inorganic particles, in fact when we do the microscopic analysis whether it is O M or S E M, whatever microscopic analysis we do, one of the first judgment calls that we should make is - are we looking predominantly at inorganic particles? Or are we looking predominantly organic particles on the filter or the impaction stub?

Because, the methods of compositional analysis are very different for inorganic particles and organic particles; all the methods that we have talked about so far - you know, starting with E D S, W D S and so on ESCA, SIMS all of these are really primarily for inorganic particles an inorganic elements.

The reason is - for example, E D S, W D S I said that the lightest element they can detect is fluorine, but you know that the when you talk about organics they are predominantly hydrocarbons - so hydrogen would not even be detected in these methods; so, if we have a C H 4 or a C 2 H 6 you have to know these methods would not even detect them.

They may detect the carbon but they are not even going to detect the hydrogen - so just tell you there is carbon element present, would not tell you any additional information beyond that. In order to do organic characterization, you have to use a different set of techniques - are you aware of techniques that are principally used for organic materials? F T I R is probably the most widely used method for characterizing organic materials and there is also micro Raman, which actually can be used either for organic or inorganic particles but it is predominantly used for organic particles.

What is the different between the two? In Fourier Transform Infrared Spectroscopy, you basically radiate the sample with infrared radiation and what you try to observe is the absorption spectrum by the molecule that is present; depending on the molecular structure the species that is present will absorb the AUGER light at a particular frequency; by looking at the spectrum of frequencies that are being absorbed on the sample you can essentially generate an interferogram and then by using fourier transform you can convert it into an absorption spectrum.

The absorption spectrum will give you information on how the AUGER light is being absorbed by the sample that you are trying to analyze. So, you will get some pattern - it will also have some characteristic peaks of absorption as a function of frequency; now, what you do is you compare this to a library of reference spectra and based on the match between the observed spectrum and your reference spectra you draw conclusions regarding what is this composition most likely to be - is it closest to C H 4 or is it closest to C 2 H 6 or C 6 H 6 or whatever, I mean, there are again literally a continuum of organic compounds that are available. This organic composition library itself is pretty massive, but again this is fully automated now - the system can quickly do the matching and tell you that this spectrum that you are collecting for your sample is most likely to be match with this sample or with this material so you can quickly home in on the particular organic compound that you are looking at.

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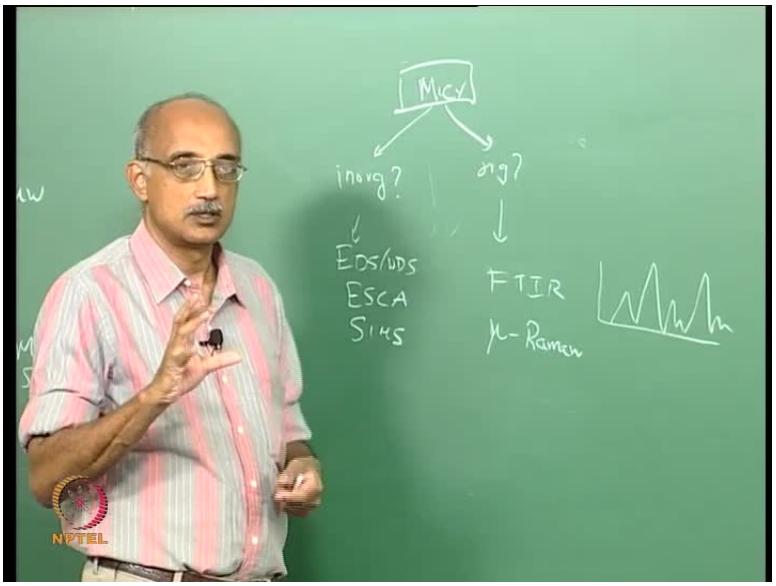

The limitation of the F T I R methodology is that it only works for fairly large samples - the particle has to be at least about 10 microns for you to be able to use F T I R for doing compositional analysis.

If you have finer samples, then you use micro Raman; now, the micro Raman spectroscopic method essentially consists of using not AUGER light but a monochromatic visible light to irradiate the sample and you look for frequency shifts in the light - so you essentially look at the light that is incident and the light that is scattered from the sample.

The frequency shift is indicative of the molecular structure of the compound, as well as - it can provide you a lot more details compared to F T I R; it can tell you about the number of bonds that are present, the types of bonds that are present, so it provides a lot more granularity in the data that you can obtain about the organic compound.

The micro Raman spectroscopic method is extremely powerful compared to F T I R, which is more of a gross method for doing characterization of fairly large samples; if you start talking about nano-dimensional particles and if you are ever required to do chemical compositional analysis of an organic nano particle then the Raman micro probe technique is probably the only one that that you can gainfully use.

So, these are techniques that are very powerful in terms of being able to precisely identify the chemical nature of the particle that you are looking at. Now, there are limitations with all these methods - there is really two: one is that they all essentially look at the surface in a two-dimensional way - they do not really provide you three-dimensional morphology information.

Now, from a chemical composition view point that may not be a big drawback; I mean, the chemical composition probably would not change much whether you are looking at something in two-dimensions or three-dimensions; but, in terms of surface morphology characterization the three dimensional imaging is a very powerful asset that you want to have, which S E M/ T E M, for example, do not have.

The other drawback with the techniques we have talked about so far - they do not tell you much about the crystal structure of the particle that you are looking at. So, in some applications particularly, for example, in semiconductor manufacturing where you need certain crystalinity to make certain functionality happen there is a necessity to characterize the crystalinity of the material very well; so, these are two requirements that have recently emerged as very critical requirements.

The ability to do three-dimensional mapping at nano scale and the ability to do crystalinity characterization again at nano scale; there are 2 techniques that have emerged over the years that really home in on these needs - the first is atomic force microscopy and the second is x-ray diffractional analysis.

As I mentioned earlier, these are very powerful techniques that are widely used by material scientist and engineers to do a more sensitive characterization of surfaces, as well as particles; so, in the next we will spend some time talking in a little more detail about these two methods in particular. So, we will stop at this point - any questions? I will see you at the next lecture then.