Particle Characterization

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Module No. # 03

Lecture No. # 07

Morphological Characterization: Static methods of size analysis

Welcome to the seventh lecture, in this particle characterization course. In the last lecture, we started discussing particle size measurement techniques and I mentioned that essentially particle measurement strategies can be classified, as being in static mode and in dynamic mode.

So, we looked at a few examples, of size characterization in the dynamic mode, where essentially particle populations are certain motion and based up on their behavior under the flow field, we derived their particle size distribution characteristics.

And then we discussed, static methods and the first thing to realize is that in order to, analyze particles in a static mode, you first have to capture them and immobilize them. So, that they can be inspected and we discussed various techniques for doing this including impactors, impingers and filters and so on. So, in this class we are going to assume that we have employed one of the methods that we talked about in the last class.

To fix a particle in its place and now, we want to characterize its size the most obvious method for doing size analysis and static mode is visual inspection, if a particle is large enough, you can inspected once you have collected it on a slide or a filter or some medium with just your naked eyes, and do a reasonable size assessment. As the particle size gets smaller, you may need to resort to some magnification simplest thing being magnifying lens, which I am showing about used.

And then an optical microscope and finally, transmit through transitioning to a electron microscope as the particle size increases. Now, I mention the other day that the human eye can actually see particles that are as small as 50 microns. However, the resolution of the human eye is only about 200 microns; in other words it cannot distinguish between

two particles that are less than 200 microns apart. So, if you want to improve, your resolution to let us say half a micron then, obviously you need at least about a 400 x magnification and so optical microscopes, where essentially designed to provide magnification in the range from five x all the way up to 400 x originally.

Now, more recently technology has improved to an extend extent, even an optical microscope can give you close to 10000 x magnification. So, you are actually able to see particles even in the nano meter range using simple optical microscopy. The technique is never going to be as powerful as scanning electron microscopy primarily, because of the method of detection, which still involves essentially geometrical optics. Whereas electron microscopy is makes use of a complete different methodology involving, bombardment of the target with the electrons.

So, optical microscopy does have its advantages. For one thing, much cheaper, secondly, the fact that electron microscopy typically needs to be done under high vacuum. So, you really have to take the sample; put it inside a chamber and apply very high vacuum in order for you to do electron microscopy, whereas optical is something that you can do in an ambient environment.

There are several other pros and cons optical verses electron microscopy and we will discuss is in more detail, as we go long in this course but one of the first things, I wanted to point out is that there is a formula for calculating, the resolution of a microscope.

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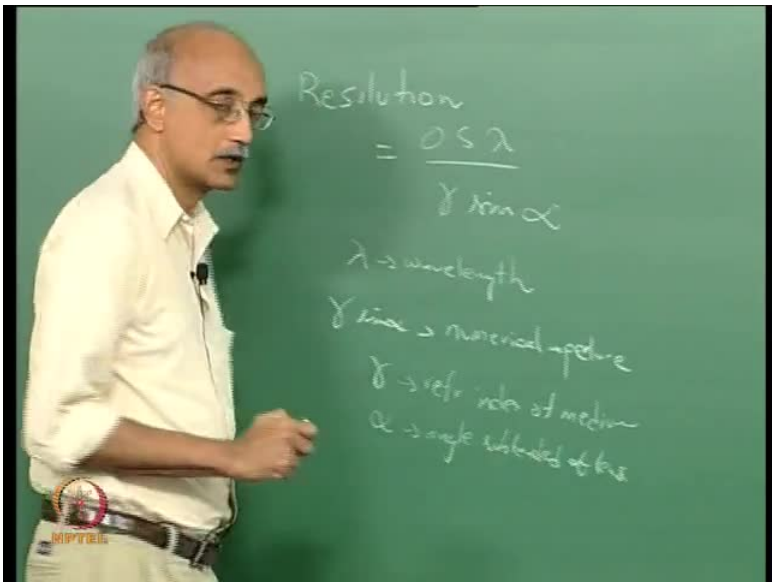

And probably familiar with that formula resolution is given as 0.5 lambda over gamma sign alpha, where lambda is the wave length of the optic light. The denominator gamma sign alpha is term. The numerical aperture and gamma is the refractive index of the medium and alpha is the angle suspended at a lens. So, in optical microscopy, if you want to improve the resolution, the parameters that you can really control or the wave length and the refractive index of the medium. Now, most of the time when you do optical microscopy, the particle is going to be essentially sitting on a surface; which is surrounded by air and so the refractive index of the medium also tends to be reasonably fixed.

So, the parameter that you do have some control over is the wave length. Now, you talking about the visible range, when you talk about optical microscopy, you are only concern about the vibgyor range, so to speak.

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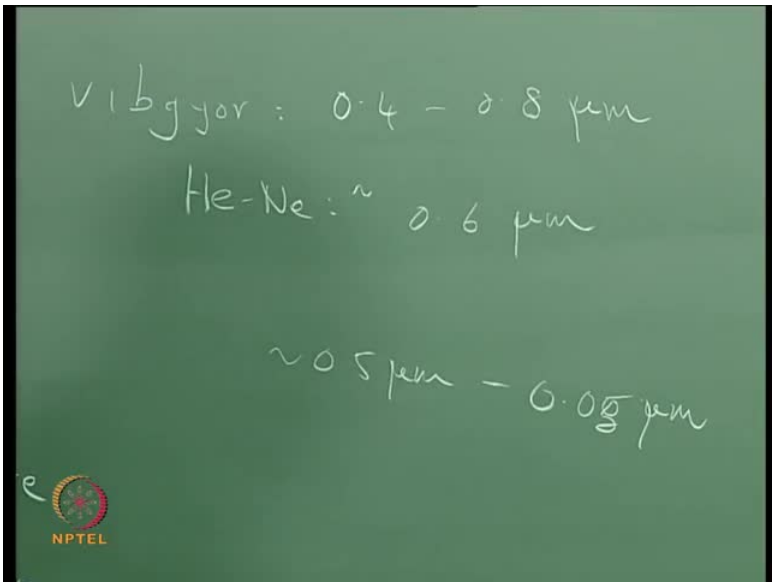

So, if you look at the range for normal visible light, it ranges from about 0.4 to 0.8 microns.

Lasers, for example, helium, neon laser has a wave length that is sort of in the middle of this wave length range for visible light. So, again when you look at this it is not a lot of room there for manipulation. And so, there the way that optical microscopes are now improving the resolution is really by playing around with the optics more than anything else there is not a whole lock, you can gain by trying to play with these parameters.

Now, optical microscopy as I mentioned typically, today, is till about half a micron in most conventional microscopes, but there are what are known as high resolution or highres microscopes, which can take this down to roughly 0.05 microns.

As a resolution increases, there is a panel key that you pay and that is the fact that you are able to scan only smaller and smaller regions of the surface. The penalty that you always pay for higher magnification, higher resolution, higher sensitivity, is the fact that the sample under consideration shrinks in size.

So, for example, if I want to look at particles on the surface, if I were to use naked eye inspection, I can look at the whole surface within 10 seconds. If I use a magnifying glass you can imagine that for mentioned to go over this entire surface may take about an hour,

If I start using an optical microscope it is going to take me, I do not know may be a day to analyze this entire table, because start using an electron microscope for me to just analyze this surface can take up to a year.

So, that gives you some idea about, the scale of times that that we are talking about. Now, when we do again optical microscopy that are essentially, two ways in which microscopy can be done and that has to do with lighting. I mentioned that resolution is now being improved by looking at other factors. That influence the operation of the instrument and light is certainly one of them, the same surface; if I were to inspected under, let us say normal lighting verses suppose I had high intensity lighting, focus lighting, or even oblique angle lighting, would show particles very differently. And so one of the tricks that are used there in optical microscopy is that conventionally what is known as bright field microscopy?

You illuminate the entire surface with bright light and defects and particles on the surface; will then show up as dark spots. Now, that technique works well for the most part, if there is a clear delineation between a surface defect and a surface particle.

Now, in many applications, you may not care but sometimes you want to know whether something that you see on the surface is actually a feature of the surface or whether it is actually something sticking on the surface.Now, to be able to resolve between those two can actually be quite difficult and dark field microscopy, was actually introduced essentially to address this issue.

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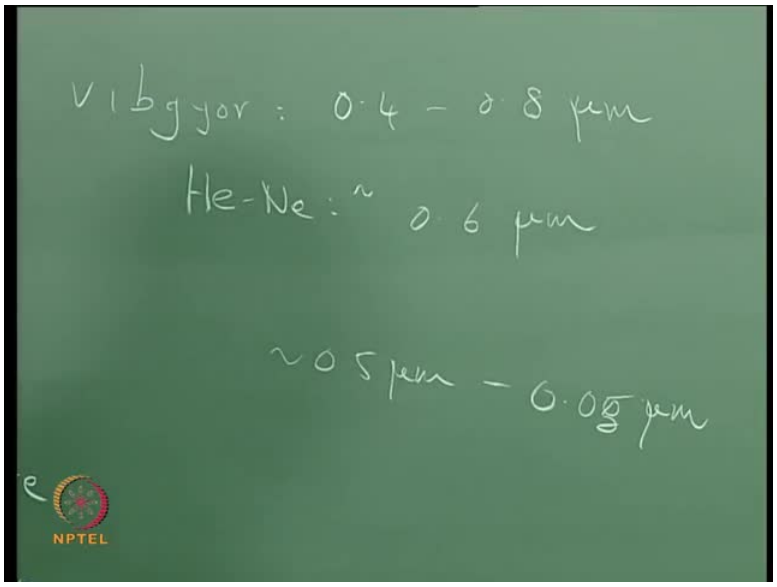

So, in dark field microscopy the surface that you are looking at will essentially be dark. And the surface features will then shine in contrast, when you do that you can actually fairly effectively distinguish between surface features or defects and surface are at the particles.

So, dark field microscopy provides you with the ability to resolve surface features with better clarity than bright field microscopy. Now with the way this is done is in bright field microscopy, you are essentially using the same aperture to capture both the reflected light as well as the scattered light.

Whereas, in dark field microscopy, you are essentially only capturing the reflected light and therefore, the contrast that you see between the surface and the particle is quite different in dark field microscopy and bright field microscopy.

There is another way, in which you can distinguish microscopy techniques, when you again, when you have a large flat surface and you are trying to do microscopic analysis of the surface, you can either initially capture an image of the entire surface and then, you software to do your scanning. So, for example, you would capture this image of this table top in a camera or some kind of a c c d or then use various algorithms to analyze the image.

And obtain the information that you are seeking. This is basic called the imaging mode of analysis; the other mode of analysis is what known as the scanning mode. In the scanning mode you will not try to do image capture.

Instead, it is basically real time analysis of the surface. So, you will essentially take your optics and traverse the entire surface with it and as you do your constantly getting a signal back to the instrument and you keep track of it.

Now, here you will use certain devices like photo multipliers; in order to, intensify the signal that you are getting from the surface, but you are looking at it real time. Now, there are obviously advantages and disadvantages to doing that verses doing the image capture methodology.

When you do direct scanning, the feedback is immediate and it is a highly sensitive instrument, because you are you can see real time, as you slowly move your scanner across a surface, you will see that the signal that you are seeing back is changing.

And you can quickly draw inferences and conclusions based on that however you can imagine that for example, if your surface is very rough then the interference that you get from the surface is going to be of the same order of magnitude, as the particle or defect that you are trying to analyze.

So, this type of, continuous scanning methodology works very well, when you have highly polished and smooth surfaces. For example, silicon vapours. The semi-conductor industry uses scanning microscopes extensively, because most of the time they are dealing with surfaces.

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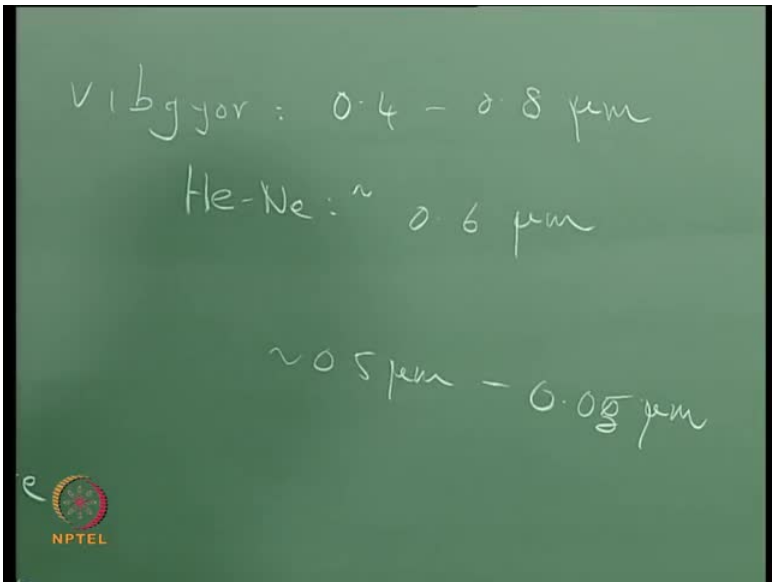

That do have a high degree of finish and the surface roughness is of a scale that is much smaller than the particles in another defects that you are trying to characterize on the surface. On the other hand, if I would really trying to characterize, particle distribution on the surface, I would not try to do it in a scanning mode, because I will just get too much noise compared to the signal, the noise being the reflection and scattering that you get from the surface itself.

Instead, I will try to grab, as refined a picture or image of the surface as possible and then depending, depend on my processing software, eliminate the noise and give me as clean a signal as possible.

Now, there are various companies that really specialize in making both dark field and bright filed field microscopes, imagining and scanning microscopes. One of them is k l a 10 core, you may have heard of them, I think they come in recruit in India quiet extensively, they are located in the one of the it parks on OMR. They are one of the major manufacturers of semiconductor characterizing equipment.

Another company that is the leader in this field is applied materials. Applied material primarily emphasizes analysis of process equipment rather than product wafers that are other companies that operate more in Nish markets. There is a company called Kendella instruments, who specialize in scanning of magnetic disk that are used in hard drives to look for contamination and defects.

There is another company called ADE, who primarily focus on the silicon wafer inspection market. And so there are there is another company called Inspects which is also a player in the silicon area.

So, there is some market segmentation of these various companies but they all do one of these four they either do image analysis or scanning analysis and they all do either, bright field or dark field analysis. Now, most of these techniques are useful for looking at surface particles.

Now, either, the particles may be on the surface inadvertently; for example, you are manufacturing a silicon wafer and we are trying to find out. What particle at contamination that is on the surface at the end of the process or these may be surfaces that have been used as, I was describing yesterday to collect particles for analysis and when you talking about analysis of surfaces that are intentionally designed to collect particles for analysis. They certainly or designed to have features that makes this analysis easy, for example there will be highly polished surfaces. So, that the surface interference is minimized however, when you are looking for analysis of particles, that have collected on surfaces inadvertently, then you do not have as much control over the finish or morphology of the surface itself and many of these analytical techniques that we have been talking about become a lot more complicated in that scenario.

Now, given the difficulties of doing microscopic analysis for particles, other techniques have they merged, as rational alternatives to microscopic analysis of surfaces, of the vary many techniques that are available; we will focus on one or two in this course and the one that is mostly used as an alternative to microscopy is techniques based on light scatter.

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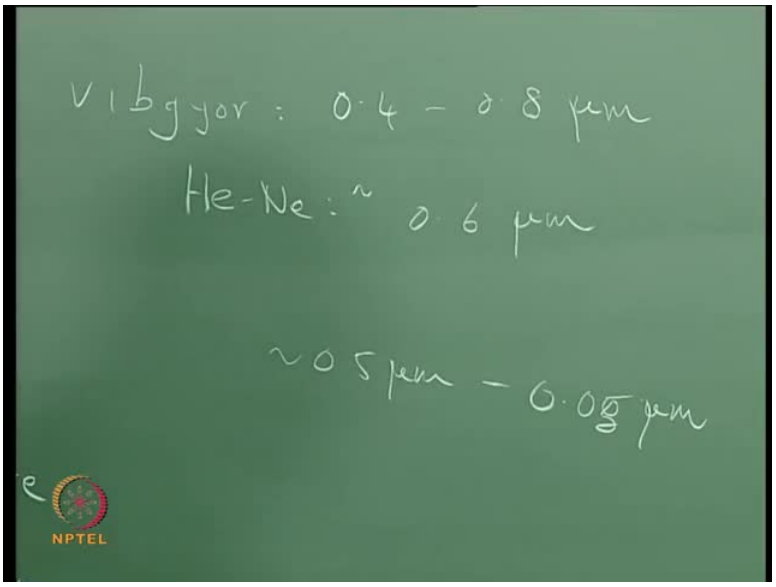

One of the very sensitive properties that particles have is that ability to interact with a light field. They interact with incident light by either absorbing the light or scattering the light or transmitting the light and so by looking at these three characteristics, you can actually get a pretty good picture of the size of the particle.

Lights scattering from particles can be again assessed in two modes, individual particle mode and suspension mode, now when we talk about particles in suspensions.

And there ability to scatter light, there are some practical examples of how this affects, you for example, the fact that you have low visibility, when there is fog what is the reason for that?

The reason is that fog is essentially, water droplets that are suspended in air. So, your ability to see is affected, because optics are influenced by the presence of these droplets and they do not allow for efficient transmission of light, which is wide visibility is very poor in fog that is what causes of delays in flights. That is what makes driving very difficult under foggy conditions or misty conditions and same thing, when you have dust laid in air your visibility is very poor, the reason again is the same particle that are suspended in air have the ability or the propensity to affect transmission of light through that medium.

And similarly, when you have particles that are suspended in liquids there is a corresponding reduction in light transmission. In the metro, water departments they talk about turbidity of water, turbidity is a measure of the water to scatter light and therefore, it is indirectly a measure of the particular concentration and size distribution in the water.

So, in many corporations the quality of water with respect to suspended particles is specified in terms of turbidity. The turbidity of water that is used for, let us say domestic usage cannot exceed a certain value.

And the reason that it is specified in this fashion is turbidity is a measure that corresponds directly, with the concentration and size distribution of particles in water. So, this property of suspended particles to affect light transmission characteristics can have positive as well as negative consequences.

A negative consequences has be described or things like loss of visibility and so on, but it also has a positive influence in the sense that we can make use of the ability of particles, to scatter light and to absorb light.

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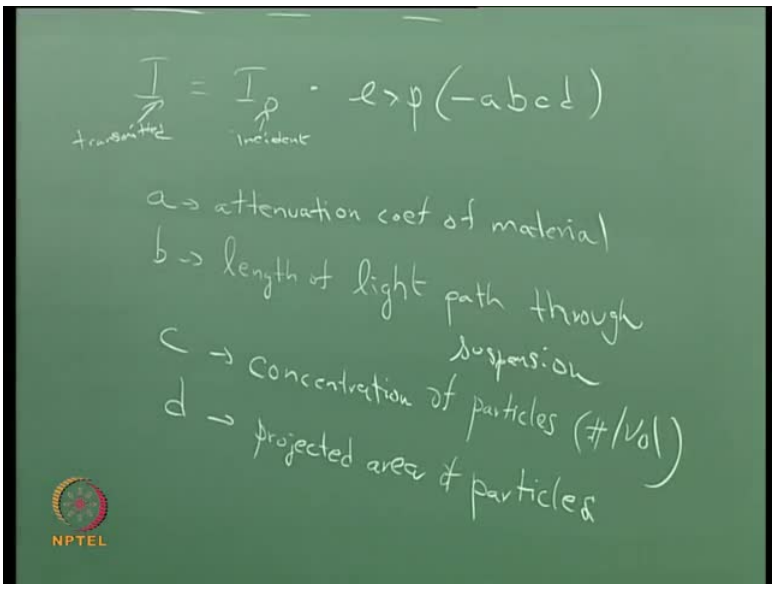

As a way, to characterize that size distribution in a suspension; that is actually a law associated with light transmission through a particular suspension. So, anybody aware of t name of the law?

It is called the Lambert Beer Law. The Lambert Beer Law states: that the intensity of light that is transmitted through a suspension is proportional to the incident light I 0. So, this is transmitted intensity of light, this is incident times an exponential factor, which is stated as exponential of minus a b c d as to make it easier to remember.

Where a, stand for the, attenuation coefficient of the material. So, the material type or particle type will show up in this a parameter. b stands for the, length of the light transmission path through the suspension. c stands for, a concentration of particles in the suspension, which is expressed in number per unit volume.

And d stands for, the projected area of particles on which the light is incident. So, clearly as the attenuation coefficient increases or as the length of the path that needs to be traversed by the light increases or as a concentration of the particle increases or as a projected area of a particle in suspension increases that is more attenuation of the light transmission through the suspension.

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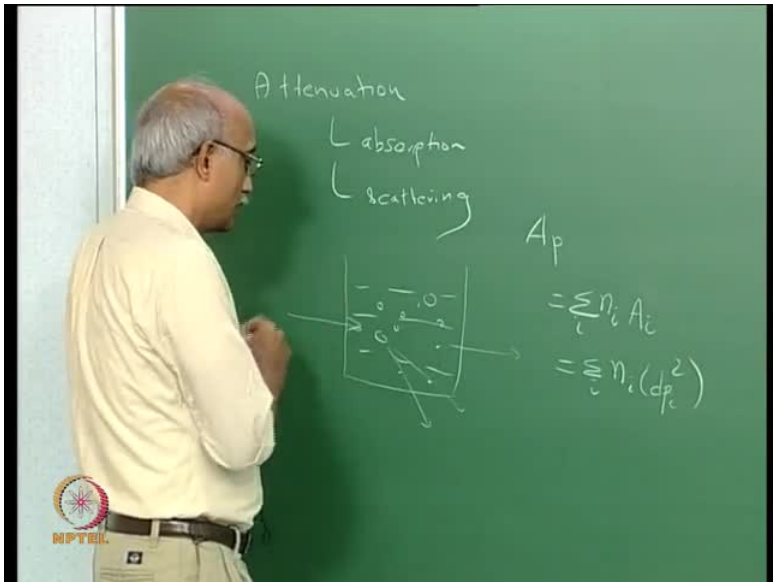

So, when we talk about attenuation, there are two components to it. One is absorption and the other is scattering. So, if we have a suspension and you have all these particles that are suspended in this liquid, and you have incident light; only a part of it gets transmitted a part of it gets scattered at various angles and a part of it gets absorbed.

Now, when you talk about the projected area of particles, in the suspension how does that relate back to the diameter of the particle.

A simple way to look at a projected area of particles, in this case just to say that a p equals summation over i n i a i, where you essentially take that particles population and break it down to various size channels.

And you look, at the number of particles in each channel and scale it by the area of the particles in that particular size channel and basically submit all up. Now, this a i of a particular particle is roughly proportional to the particle size square.

Assuming, that the particle is spherical in nature or can be represented by a sphere of equivalent scattering characteristics and so particle size here is not directly measured. Now, instrument that is actually used that operates based on this principle is turbidity meter that is used to measure turbidity of liquids.

And what turbidimetry does is exactly this, the shine light on a surface and then they take the light that is scattered and measure it. The scattered light intensity is a measure of turbidity in the suspension. And the scattered light is what is proportional to the total scattering cross section of particles in suspension, which is related to this parameters summation over i n i d p i squared. So, from this how do you extract, let us say a mean size.

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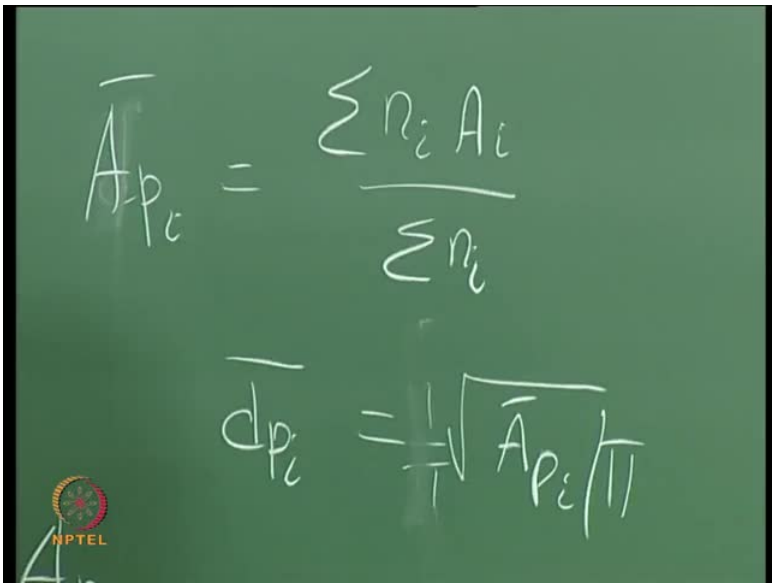

You can define a, d p i bar as equal to summation n i a i divided by summation n i, well actually you first define, an average area of the particle as summation n i a i over summation n i and from this, you can get an average particle diameter as a square root of the average particle size one over i again assuming, that you have taken the particle as being roughly spherical in shape.

Now, this pi should be inside. So, this is basically, how a turbidimeter is used to characterize mean particle size. The thing you have to realize is two fall one is that the assumption here is that the particle are roughly spherical in size in their shape.

Therefore, the diameter mean particle diameter that you extract from a turbidimeter, it is not based on single particle inspection, this is obviously looking at the entire powder and looking at its overall light transmission characteristics.

And estimating the mean size of the particles, using that technique, so, that is one limitation, the second is that the inherent sphericity assumption is another one that always have to keep in the back of our mind.

But this restriction of the assumption of a spherical particle, as we will see is applicable to virtually every technique, we talk about all the particularly, the light scattering type of the techniques, all make the implicit assumption that the particle is at least roughly spherical in shape.

There are very few theories of light scattering from highly non-spherical particles. The assumption always is that the sphericity parameter that we had discussed earlier is reasonably close to unity. So, let us say that you have calculated or estimated an average particle sized based on this technique. Now, there are many ways of calculating the mean size of a population of particles.

The size, if we calculate this way is called the area mean diameter, you can also calculate a number mean diameter, you can calculate a volume mean diameter and when we talk about individual particle size measurement, we will also see that there is something called a scattering mean diameter.

Now, these are all essentially various moments of the particle size distribution law. And in fact, the different movements will represent different mean diameters. So, we will discuss that in detail, when we talk about particle size distribution characteristics of a population of particles. But the key thing to remember, at this time is that when you measured the light transmission characteristics through a suspension of particles and you extract in a mean particle size using, this approach there are always questions about the size I mean one way to think about it is.

The total particles characteristics cross section in a suspension is summation n i a i. so, let us say that you have two solutions and one has twice the turbidity that the other one has. Now, what can you conclude from that can you conclude that this solution, has twice the concentration as this one or can you conclude that the particle scattering cross section is twice as much, in this as this you do not know, because it is really a combination of the two.

So, when you have two samples in which both the concentration as well as size distribution are changing, it is very difficult to de-convolute, it to effects on total scattering intensity, if that one is fixed you can estimate the other through a relative measurement.

But when you have two random samples that are being supplied to you, let us say by two different cities and you are ask to assess, which one has higher concentration and which one has larger particles, you really cannot tell all, you can tell is this number which is really a sum of the two.

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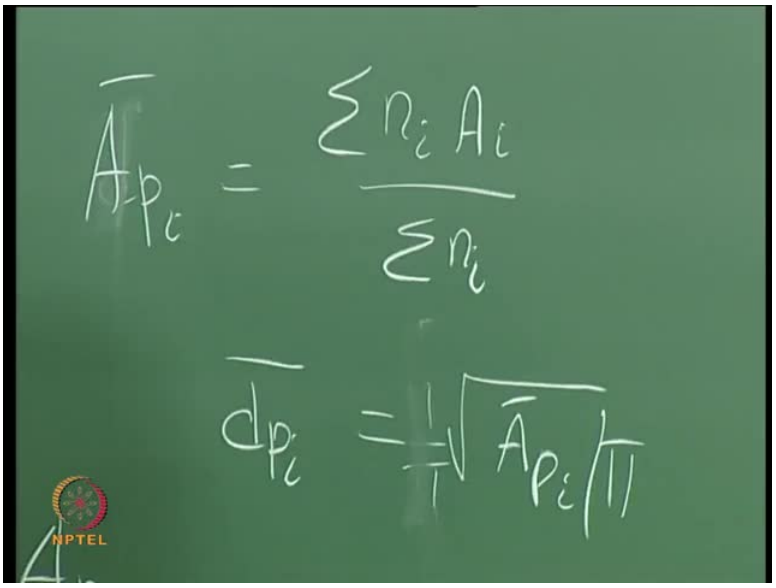

So, if you now want to go back and analyze to see, which of these two samples really has a particle size distribution that is on the higher end compared to the other you have to rely upon individual particle size analysis.

So, at that time you have to go beyond the particular suspension analicity you have done, to doing scattering measurements at individual particle level. Now, the difference is an instrument that is used to do this measurement, for example, a turbidimeter. Probably cause about 1 lakh, whereas an instrument that is used to do single particle size analysis using lights scattering even, if you are restricting your lower limit to about one micron still cause about 15 lakhs.

So, there is about a 15 x differential, now, as you go down to sub micron sizes, if you want a particle size analyzer that can measure down to let say 50 nanometers that causes closer to 25 lacks and if you want to go down to sub nanometer levels then the price just exponentially increases.

And so, you have to be again, watchful about the technique that you use for your size characterization measurement, if all you care is the is the mean size of a particle like population then first try dynamic techniques, secondly try static techniques that primarily involve visual inspection.

If neither one works as an option for you, for your specific application, then you use scattering techniques that are effective for an entire population of particles, if even now that if that information is not sufficient for you, then you start looking at scattering instruments that are designed for individual particles.

So, we will begin discussing particularly, the scattering characteristics of an isolated spherical particle just that really the fundamental basis, on which size measurement using lights scattering is built upon.

Now, in the case, of suspension of particles we saw that the transmitted light intensity is proportional to the incident light intensity and to a large extend there is true about single particles also. However, when we are trying to use optical methods for single particle size characterization.

We typically do not look at light transmission, what we do is look at scattered light and it turns out that the scattered light intensity is also proportional to the incident light intensity. So, if you look at a spherical particle.

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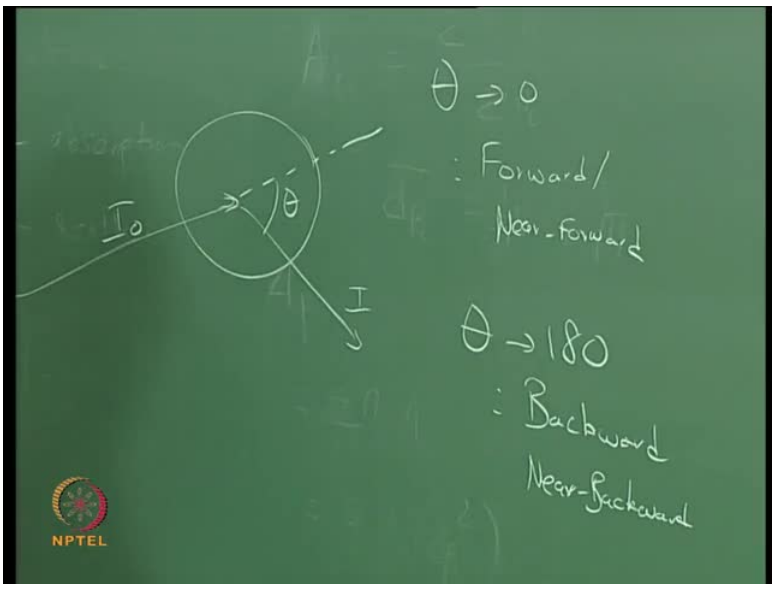

And you look at a light beam that is incident at some intensity I 0, it is going to get scattered at some intensity I. Now, the angle of scattering is measured in this fashion and the angle is a very important parameter in scattering intensity basically, the scattered light intensity will vary depending on the value theta. So, it is depend on value measure

it at what angle theta that you measure scattering intensity, you will get a different answer now when theta tends to zero. This is called forward or near forward scattering and as theta tends to 180. This is called backward or near backward scattering.

And so, you have to design your optics appropriately, if that your scattering characteristics or such that much of the light is going to get scattered in the forward region of the near forward region, then you designed your optics so that you primarily focus on collecting scattered light from that range of theta.

Whereas, if your sample is primarily a backward scattering sample, then you design it appropriately and one of the key parameters that decides, whether this scattering is forward scattering or backward scattering is the light the size of the particle.

As you can imagine, the larger the size of the particle the more will be the probability that the particle will redirect the light to an extensive amount, whereas the finer the particle the greater the probability that light can actually be transmitted through without even being affected by the particle.

For example, if the particle happens to be much smaller than the wave length of the light then the scattering intensity is also going to be very small. In fac,t as we will see later on the scattered light intensity varies as the particle diameter to the power 6.

And that is a huge challenge because it means that even though light scattering is a very sensitive technique for particle size measurement, as the size shrinks if you start approaching sub-micron and nano dimensions.

The fact that I goes as d p to the power 6 is a huge limitation, a 10 extra in particle size let say from one micron to 0.1 microns is going to cause, what 10 to the power 6 times draw roughly in the scattered light intensity.

So, particle counts as that are very effective and very accurate up to a micron start to lose their measuring ability, as you go below the micron. That is, micron size it is kind of a magical cut off size most particle counters that are available commercially.

To a very good job, in the size range from roughly 25 microns to 1 micron, because if go above 25 microns use light scattering instruments is an overkill you can just as well do it

with microscopy, just optical microscopy would work fine or even sieving analysis or whatever you want to do.

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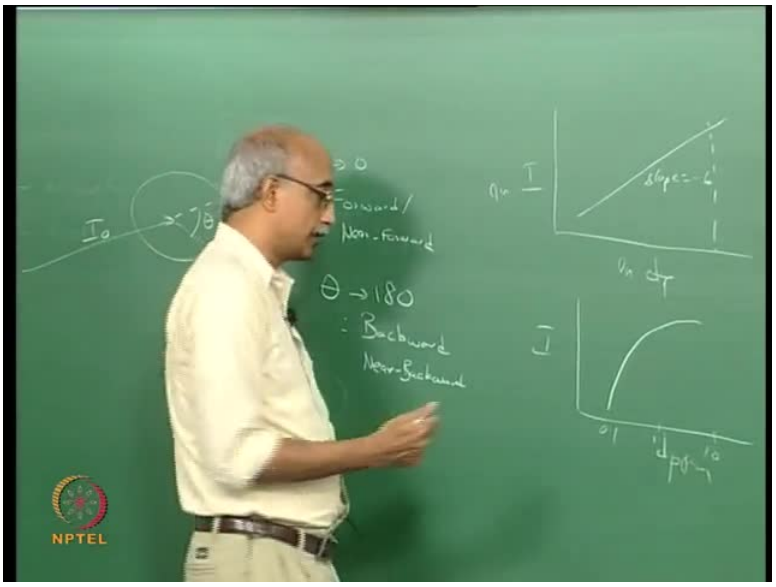

So, particle counters that are based up on light scattering have a fairly limited range in which they are truly useful and that is roughly from about 25 microns to 1 micron.

Because what happens is as soon as you drop below a micron, again if you plot this is quite dramatic so if you plot d p verses I and you will do it on long log scale then you essentially get a straight line, with a slope that is minus 6. So, the drop is quite significant, I mean it is actually probably more visually striking if you just do it as a linear plot is this the kind of trend that you will see that is basically one over that is a d p to the power 6 graph. So, let us say that this is your 10 microns, 1 micron, 0.1 micron your light optics that are perfectively fine up to a micron all of a sudden are not going to help you. As you approach the submicron range, so that is a huge challenge that we will discuss in more detail in future lectures. Suppose, you do want to analyze the size of nano particles or submicron particles, how do you do it.

Because, the theory tells you that there is scattering intensity drops exponentially virtually, how do you address that problem do you give up on light scattering and move to something else; the problem is there is no other technique that works even as well as light scattering, I mean what is your option you can if you want use something like a scanning electron microscope or a tunneling electron microscope atomic force

microscope, but to do particle size analysis using, such tools it is not an optimum procedure because these instruments are really intended as qualitative characterization tools.

You, want to use them when you are trying to characterize a surface of a particle or the morphology of a particle not simply to quantitatively determine the size it is an overkill and a statistically not very reliable, because of the time involved in analyzing a single particle the fact, that you have to really analyze multiple particles and get some statistics means that even if have a 100 particles in a sample.

And you do size analysis using microscopic techniques, it is when you take it forever and it is not a justifiable investment of your time or cost and so, you would prefer to use other techniques that will the most straight forward, faster more efficient and more reliable way directly yield particle size information. So, it is a challenge that you should think about let us say that you are stuck with light scattering techniques and you want to measure particles that are in this size range.

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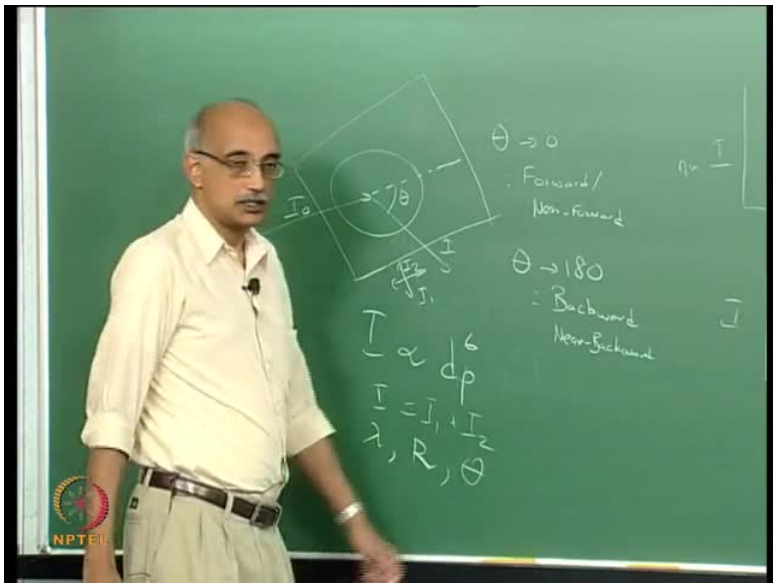

How would you do it, now just something that you can think over and then we will come back and talk about it latter on. So, going back to this chart that shows, how light is scattered by a single spherical particle, we defined something called a scattering plane which looks like this. yes

Minus 6

No the scattering intensity,

The forward the scattering and the forward forward or the

The scattering intensity talk about is the total integrated scattering that is occurring because of the presence of the particle.

It is always a good light scattering instrument will measure scattering as an angle, it will essentially look at scattering at various theta values and then summarize them. So, this use the correct dependence, I mean the intensity goes as d p to the power 6. As the particle size drops the scattered light intensity drops, as the sixth power much faster than the decrease in the particle size itself.

True, so as light is polarized. So, if you have a polarized light, it will have two components and when you have scattering going on and you defined a scattering plane. This scattered light can also be essentially, be convoluted into two components there is one component that is parallel to the scattering plane that is called I 2. And there is a component that is perpendicular to the scattering plane that is called I 1.

Scattering does not affect the polarization of the light. In other words, the polarization of let us say, if I 0 has polarized light in this two components I 1 and I 2. The scattered light will also have the same components that are polarized in an identical fashion.

And so on, the scattered light I is the sum of I 1 and I 2, which are the two polarized components of the light. So, when you look at light scattering from an spherical particle there are obviously two parameters that affect the scattering intensity; one is the size shape is incidental to this; you are assuming again that this shape is spherical, you can imagine that for the same surface area, if you have a highly elongated particle, its scattering tendencies will be completely different.

So, the assumption here is that shape is spherical and given that then the scattering light intensity is propositional or dependent on particle size, it is dependent on the degree of polarization of the light and what else will affect it. Certainly, the wavelength of the light lambda will play a role. The other one, that will always be an important parameter is the distance between the observer and the object that is being analyzed.

Now, in this case, that the observer may not be a person, it is actually a system instruments that are actually detecting the signal. So, the farther, the particle is from where the measurement or the detection is there is going to be attenuation in the signal.

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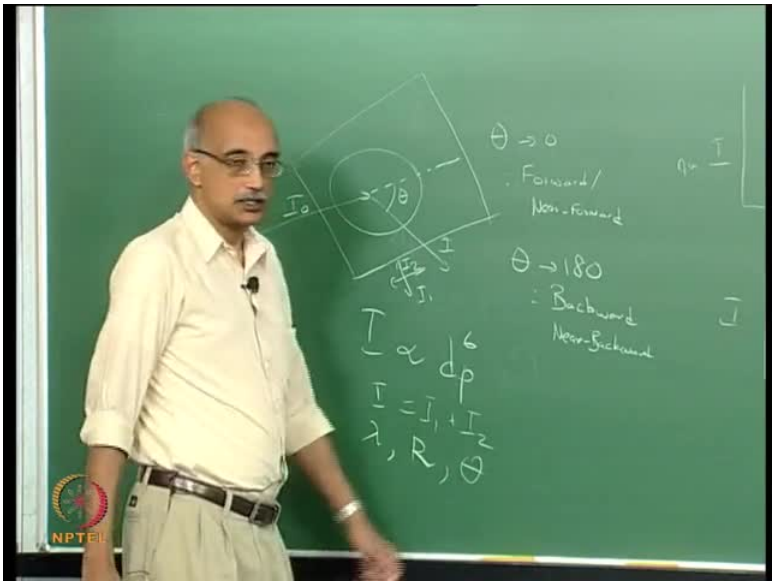

So, the distance between the detection point and the location where the particle is found is also an important parameter. And as we mentioned earlier, this angle theta also has an influence on scattering intensity.

So, when you look at the light scattering characteristics from a single particle and we will do this quantitatively in the next lecture. The key point is that there are many variables that affect scattered light intensity from a single particle, which makes the use of a particle counter that based on light scattering, somewhat questionable, in terms of validity, because two particles that are entirely different in size can give the same scattered intensity.

If some are these, other parameters are suitably adjusted. So, what that means is if you want to use a particle counter, that is based upon light scattering to do size analysis of a particle then, this particle counter must be carefully calibrated, with the same wavelength of light that you are going to use for your analysis. The same distance between the optics and the particle must be maintained. The scattered light intensity must be collected over the same range of theta as in your actual analysis. And also you have to keep in mind that the calibration must be done using particles that are as close to the physical particle, as

you can manage, for example, if you are trying to measure the size distribution of dirt in the environment, then the best calibration standard that you would use would be a sample of dust from the same location.

On the other hand, if you are trying to primarily measure, let us say the size distribution of plastic particles. Plastics manufacturing plant, then the calibration sample should also be a plastic material; again the reason for this is to minimize the number of variables that can affect light scattering characteristics of a sample that you are trying to analyze.

Let us stop with this point. And we will continue our discussion of light scattering from spherical particles in the next lecture. are there Any questions, see you at the next lecture then.