Fundamentals of Materials Processing (Part–1)
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Lecture Number 29
Powder Characterization Techniques

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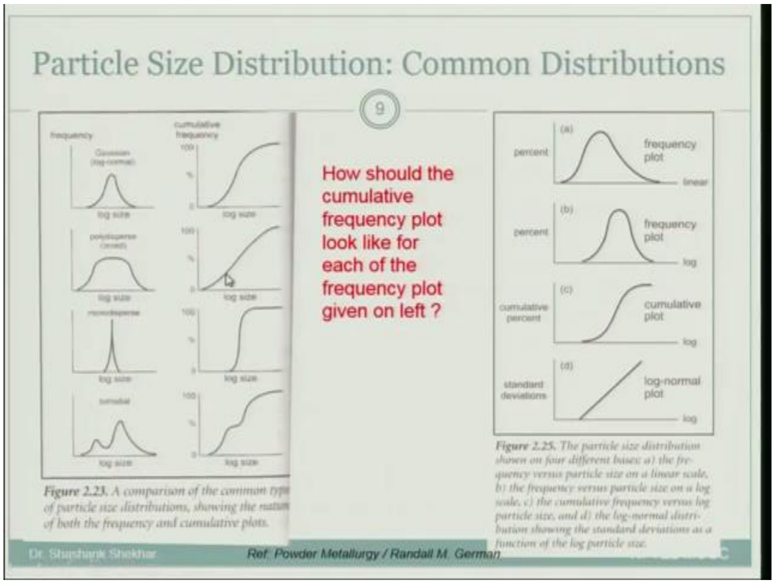

Particle Size Distribution: Common Distributions

Figure 2.23. A comparison of the common typ of particle size distributions, showing the natu of both the frequency and cumulative plots.

How should the cumulative frequency plot look like for each of the frequency plot given on left ?

Figure 2.25. The particle size distribution shown on four different bases: a) the frequency versus particle size on a linear scale, b) the frequency versus particle size on a log scale, c) the cumulative frequency versus log particle size, and d) the log-normal distribution showing the standard deviations as a function of the log particle size.

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Ref: Powder Metallurgy / Randall M. German

So we come back to this particular slide, I hope you have tried to draw the plots, the cumulative plots. So now let me reveal how there the cumulative pot look like. First one is very easy it is a single sigma model curve. Now, when you have point very broad (disperse) distribution as you see in this particular case. This line, this cumulative plot also becomes point very wide or the slop becomes very small. On the other hand, when your, when your distribution is very narrow like in this particular case which is also called mono dispersion, point mono disperse distribution then this becomes more like a step function.

Most of the data would lie in within this, it starts from zero and reaches 100% very soon. And this how the cumulative plots look like when you have bimodal distribution, so you have one mode here another mode here. So here in terms of cumulative plot what is he is a step saturation and then again it starts to increase beyond a certain point.

So these are different kinds of cumulative plot that you can obtained. However like I said point log normal is the most common point when you have particle size from one particular point method. Let us say you did, you mixed particles from two different methods. Or point obtain and the two different conditions. In that case you may as well get a bimodel distribution and so on. Now coming to the different kinds of plots. So this is your simple linear plot. So here this is how the distribution looks like as you can see it is right skewed.

This is how the log normal distribution looks like, this is how a cumulative plots looks like which we have been discussing so far. And the last one is a very interesting. Here what we are trying to define is on the y axis standard deviation and the x axis log normal distribution. And now you remember for each and every point diameter. point we have you can say there is a D50, D10, D90. So for each and every standard deviation, you can say point there is a corresponding diameter which represents how much fraction of data lies over there.

For example if you go to the zero standard deviation which will be somewhere over here. This will be D50. Now if you go to point +1 standard deviation and -1 standard deviation then over here it will be D50 + 32, so so on, it will be D82. And here it will be point D50-32 point like D18, so D18 to D82. So this is how you can always draw a corresponding diameter which represents how much fraction of data lies below this and draw it draw against it number of standard deviations.

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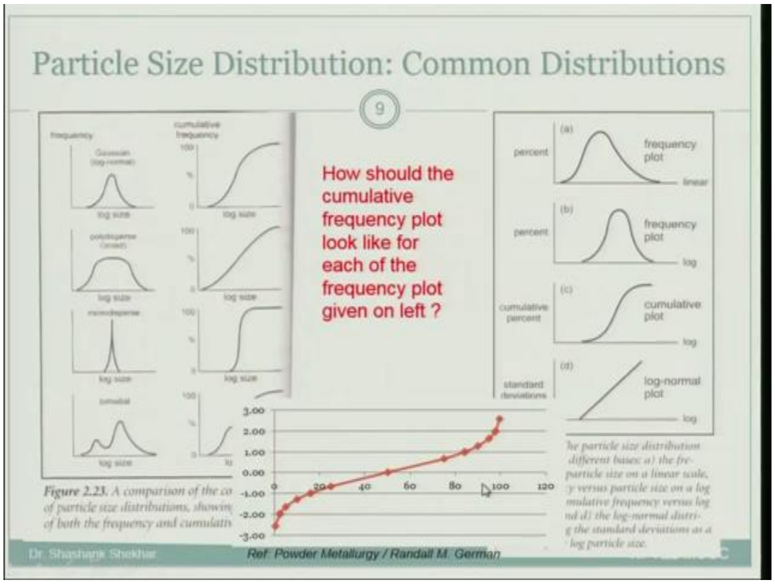

Particle Size Distribution: Common Distributions

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Now this is a very useful plot because whenever you have a normal distribution or in this particular this log normal distribution, you can you will always get a plot like this. So here what is representing the fraction of data or equivalent or you can say equivalent to that what is the D, what is equivalent diameter. So for D for 100% data you will get a D100, for 50% data you get a D50, D50 is where you have the mean value. And for 20% data you get a D20.

And a just to bring back point in our memory that we are talking about log diameter. So this is not really D20 but log D20, this is log D40, Log D60 and so on. Now the interesting aspect about this curve is that it is very linear somewhere from 10 to 90. So 10% to 90% data when it is plotted against the standard deviation falls, it comes out as a standard it comes as a linear line, it is a straight line. Now if you know this fact and this will be true whenever you have a normal or in this particular case log normal distribution we can use a relation like this.

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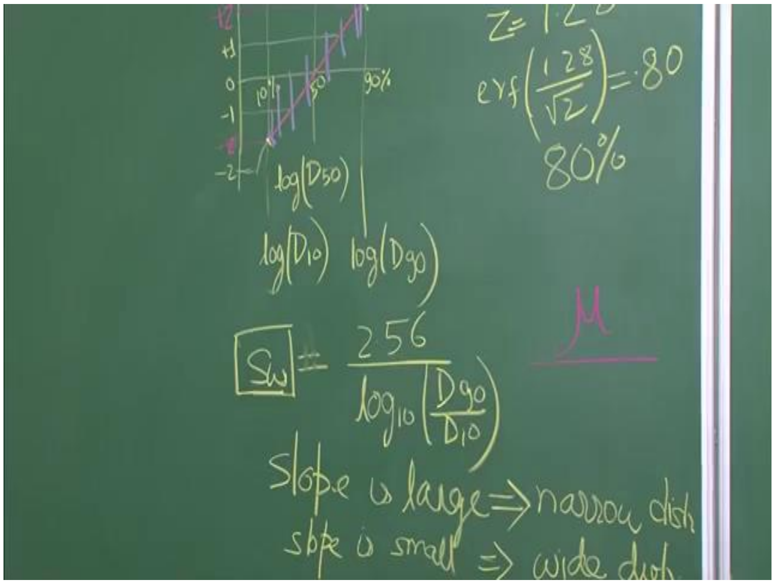

So now what we are saying is that this is the mean so this is D50. So this is corresponding to D50 actually log D50 to be precise. And like I said somewhere over here, you have a 90% data. So this was a 50% data for that matter. And therefore corresponding to that you have log D90. And somewhere over here you have 10% data therefore corresponding to that you have a diameter D10, log D10. And in between this so over here we have we can call it is standard deviation zero.

Somewhere over here you have -1. Somewhere here you have +1 so in between this standard deviation you have remember 60, 68.27% data. And somewhere over here you have -2 and +2. So although this is not linear but we still know that within this point +-2 sigma, you have 95.45% data. Now if we look at this value to this value and let me draw it with a different chalk, which is a straight line so for this particular line we can also find corresponding what is the sigma value. So there is a – let us say z and over here a +z.

And it is very easy to show that this z is equal to 1.28. So if you take erf 1.28 by root 2 what you get is 0.8, meaning 80% of data will lie within this from here to here. So let me again emphasize this, if you are looking about, so this will contain, this diameter to this diameter will contain 80% of the data. We know this from the corresponding standard deviation which comes out to be 1.28. And if we have to look at the slope of this line, the slope can be defined as this is +1.28 this is -1.28 so this minus this will be 2 times 1.28 which is 2.56. And on the x axis what have log D90-log D10. So we can write it as.

Now this is true for no matter what the distribution is because we are not we have not yet put any actual value. We have just put symbolic number. D90 represents some value where 90% of the data is. D10 represents symbolic, is a symbolic point character which represents the diameter, below which the 10% the data lie. So once you put this you will be able to get a slope of this line. Now when this slope is very large what does it imply? It will imply narrow distribution, meaning this line become very point vertical.

Then the narrow we have narrow distribution something like mono dispersion distribution. When slop is small meaning it goes like this. Remember it will still cross 10 and 90 at the similar values. So by getting this value of as Sw you will be able to find what is or what kind of distribution you have narrow distribution and wide distribution. And at the same time you have another point factor another number which is the mu, which is the average size average diameter.

So using these two numbers you have complete information about the distribution of the particular size. So you see statistic come to our help here and we are able to deal with such a large number, something between that 10 to the power 9 to the 21 particles per kg. That kind of number can be dealt with with the help of statistics. And we have now reduced it the overall point distribution information to just two parameter mu and Sw. Of course you can also say mu and standard deviation. But then standard deviation in itself you will have to again manipulate a lot to get a better understanding much more suitable number is Sw and mu. So we are able to have a complete understating of this distribution using these two numbers.

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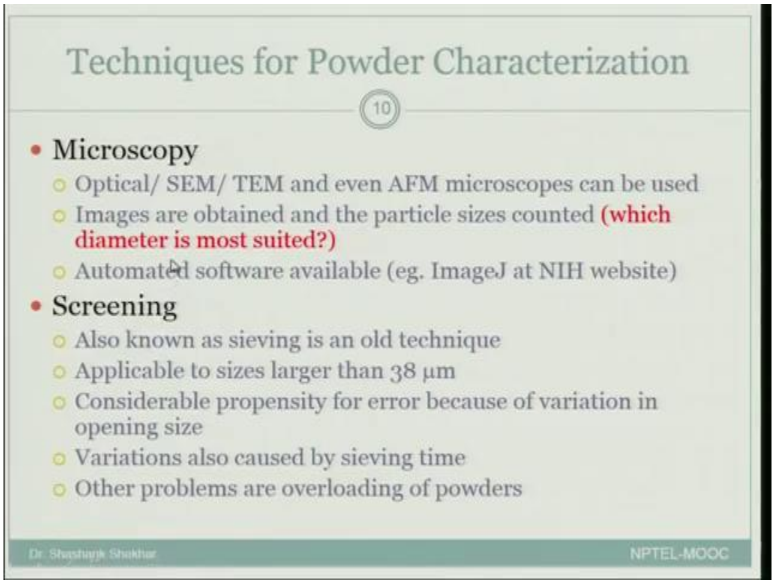

Techniques for Powder Characterization

- **Microscopy**
  - Optical/ SEM/ TEM and even AFM microscopes can be used
  - Images are obtained and the particle sizes counted **(which diameter is most suited?)**
  - Automated software available (eg. ImageJ at NIH website)
- **Screening**
  - Also known as sieving is an old technique
  - Applicable to sizes larger than 38 μm
  - Considerable propensity for error because of variation in opening size
  - Variations also caused by sieving time
  - Other problems are overloading of powders

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So now we have understood in great detail about the size distribution. It is time that we look at experimental technique so for it was theoretical. Now we have the theory to understand the distribution etcetera. It is time to look at some of the point actual experimental techniquesthat are used to characterize powder particular.

Two of the most commonly used techniques are listed here, Microscopy and Screening, So what do we do in Microscopy? We use optical/SEM/TEM or even AFM micrographs and within look at the picture and then try to obtain the area projected area and from there you will determine what is the D equivalent. You remember we, when we have a projected area from where we are able to calculate or we were we were able to obtain a equivalent diameter so we can obtain that.

So you can understand the projected area diameter will be the most suitable value when we are looking at images. And in fact now days you when have automated software available to recognize particles and then calculate the area and then it will automatically give you equivalent diameter. So these things have become very easy and one of the very common point image software that is available for free is this ImageJ. It is point at this website type for ImageJ at NIH and you will be able to get the link. It is a very dependable software to understand and to analyze the a particle size.

Second method is screening it is you can say the brute force method. You have sieves of different sizes where the largest particles are allowed to are made to stay on the top and the smaller particle are allowed to sieve or point permeate through the sieves. And then again you will have another layer of sieve which will allow even smaller particles. And the third layer and fourth layer and so on.

So you will have a different sieve carrying a range of a range of particle size. Range will, will now range will depend from the maximum point the minimum of that upper one to the maximum of the lower one. So that may you will have what we have been plotting as a frequency or histogram distribution. So each of this sieve will become one histogram. Now these are usual point usually used for particle size greater than 38 micrometer. You will not be able to use for very small particle size.

Considerable propensity of error now here when you are doing sieving there is a good chance of point errors seeping into your data. And that is because in here what you do is you take a sieve and you shake it. Keep shaking and until you have until you are convince that all the smaller particles have permeated through into the lower side. But then of course this may not be a 100% way there may be point lot of fraction of smaller particle still remaining on the on the of the that sieve.

And it will of course you can understand that it will also depend on the amount of the sieving time. If you do it find out very very long time you would get lot more smaller particular to point go down. If you do it for very small time a lot us small particular will remain on the top. And other problems are overloading of powder so for example. You cannot put too much powder on one sieve and so on. So these are the some of the problem associate with sieving technique or the screening technique.

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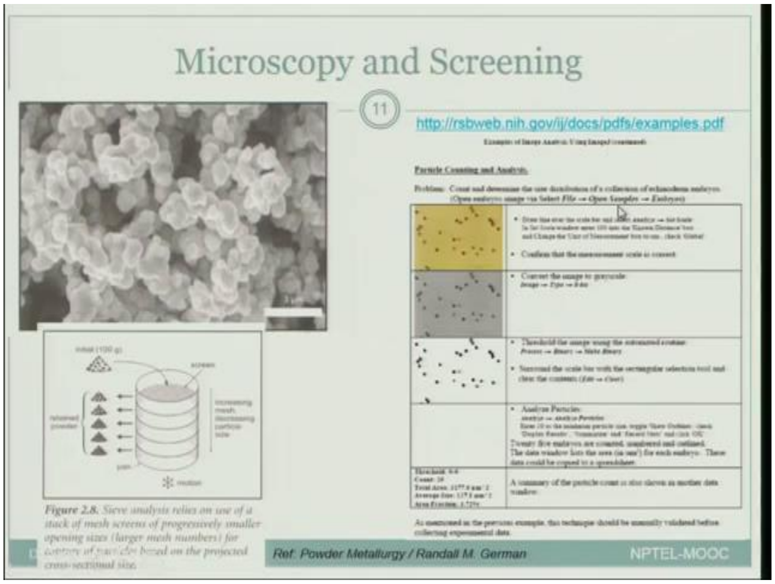

Microscopy and Screening

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http://rsbweb.nih.gov/ij/docs/pdfs/examples.pdf

Figure 2.8. Sieve analysis relies on use of a stack of mesh screens of progressively smaller opening sizes (larger mesh numbers) for sorting of particles based on the projected cross-sectional size.

Ref: Powder Metallurgy / Randall M. German

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Here is a much more you can say a figure to explain what we talked about the imaging technique. So here is a image with particles over here. Now this software as you can see which is by images software and here is also the link where you can download. So you can see that it can recognize different particles and then convert it to gray scale, and then it will be say area and hence the size of these particles. So this is by imaging technique and on the bottom left what is shown is the sieving technique.

So you see you put some say 100 gram of the powder over here. And then you have point sieves of different sizes, so that increasing mesh, mesh is the size of the, or the point density of the wires in that sieve. So the density of the wires is increasing which means smaller and smaller holes are available. And that particles point flow through to the smaller and smaller size and therefore this is the schematic distribution, you will have larger particle over here. Smaller, smaller and even smaller particles go into the bottom side.

Now that we are taking about the mesh. So for let me just give you a quick example when we say 200 mesh size what does it mean. We said that when you go when increase the mesh size there are smaller particles going down.

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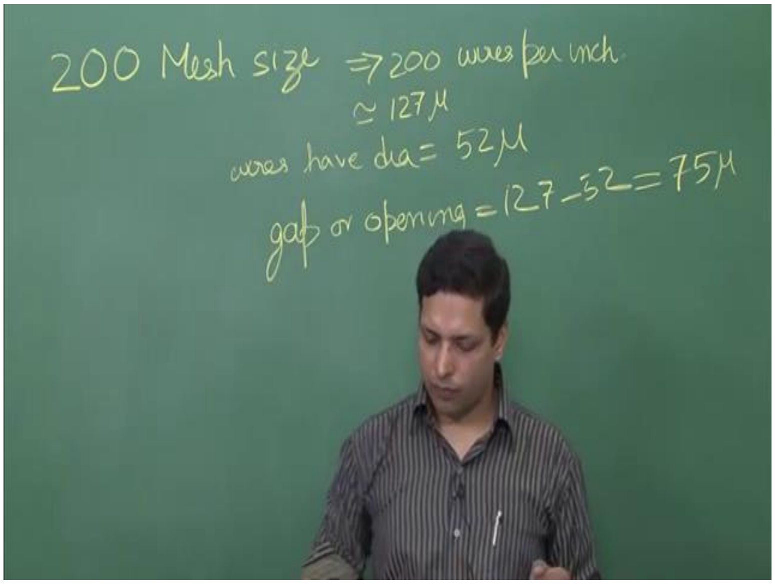

Now when you say 200 mesh size what it means is 200 wires per inch. So if you have higher the number higher number of wires available per unit inch. It means the spacing is as approximately 127 micrometer between each wire when we are using 200 wires 200 mesh size. But then wires itself diameter. And for example let say wires have dia equal to 52 microns. Therefore the gap or the opening is equal to 127-52 which will be equal to 75 micrometer. So when you are using a mesh of size 200 particles less than this size, which is 75 micrometer will go down, will go to the point bottom sieve and particles greater than 75 micrometers will remain onto this sieve.

And then you can increase the mesh size so over there gap would be smaller let say 50 micron. So in that particular sieve you will have a 75 to 50 micron particle size. And particle size less than 50 micron will go to the bottom sieves. So that is the way to characterize the powder, now you will have full histogram for each of this point sieve. So each will represents one histogram.

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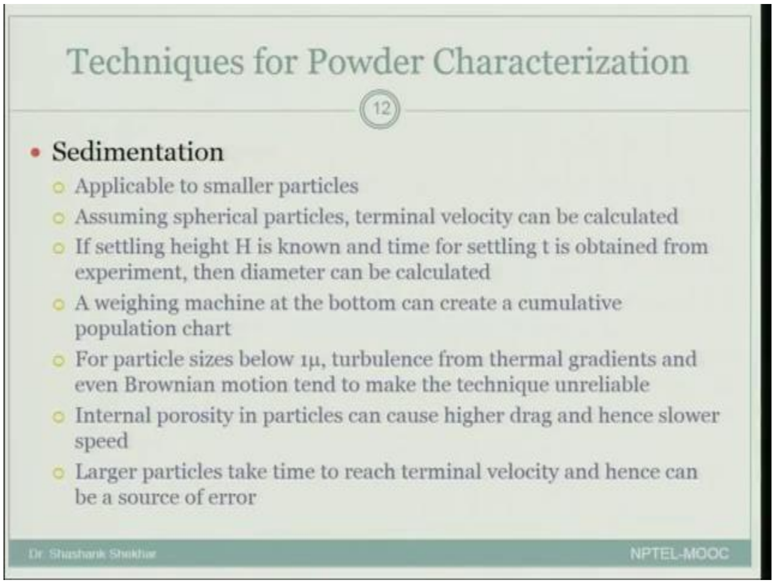

Techniques for Powder Characterization

- **Sedimentation**
  - Applicable to smaller particles
  - Assuming spherical particles, terminal velocity can be calculated
  - If settling height H is known and time for settling t is obtained from experiment, then diameter can be calculated
  - A weighing machine at the bottom can create a cumulative population chart
  - For particle sizes below 1μ, turbulence from thermal gradients and even Brownian motion tend to make the technique unreliable
  - Internal porosity in particles can cause higher drag and hence slower speed
  - Larger particles take time to reach terminal velocity and hence can be a source of error

So next point we look at even another technique for characterization of powder and this is a very well-known point technique which is widely used for characterizing powder particularly in the labs and this is called sedimentation technique. So what we do is put your powders through a liquid and allow the powders to move to it. Now this is based on what is called as Stokes law. Which say that for a in viscous liquid the particles moving through it will reach a terminal velocity. And why does it reach a terminal velocity, because there are different forces acting on to it.

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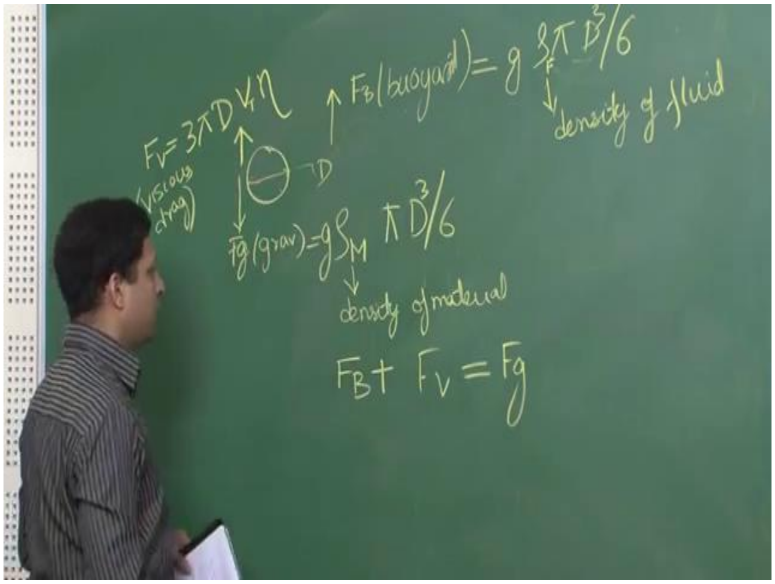


$F_V = 3\pi D V_t \eta$ (viscous drag)

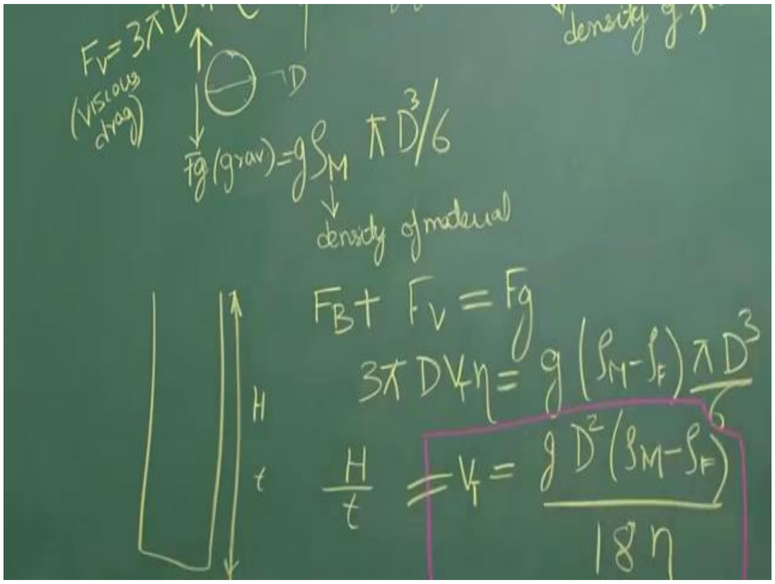
F_B (buoyant) $= g \rho_F \pi D^3/6$

density of fluid

F_g (grav) $= g \rho_M \pi D^3/6$

density of material

$$F_B + F_V = F_g$$



$F_V = 3\pi D$ (viscous drag)

density of fluid

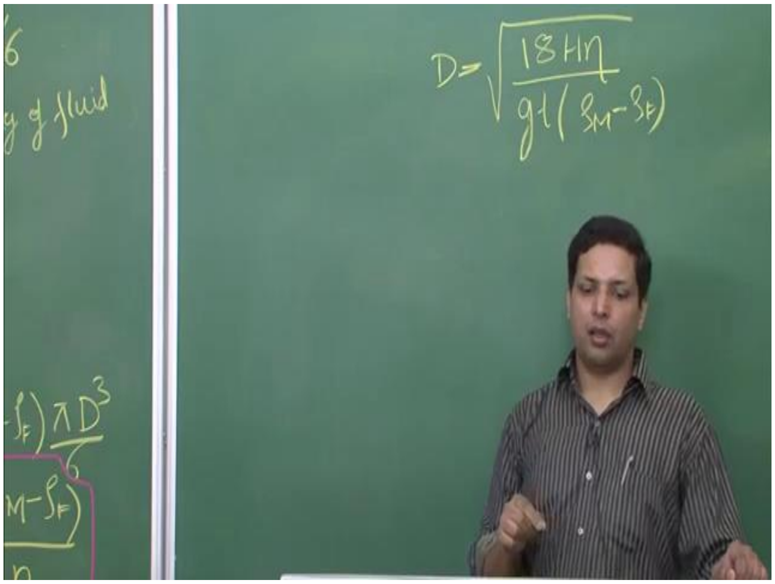
F_g (grav) $= g \rho_M \pi D^3/6$

density of material

$$F_B + F_V = F_g$$

$$3\pi D V_t \eta = g(\rho_M - \rho_F)\frac{\pi D^3}{6}$$

$$\frac{H}{t} = \boxed{V_t = \frac{g D^2(\rho_M - \rho_F)}{18\eta}}$$



So let say this is a particle of diameter D, since it is inside a fluid so there is a buoyant force acting on it which can be given by and corresponding to the weight or the mass there will be a gravitational force which will be similar except that here we had rho f ok let me I will define it in moment. But let me write down the equation first. Here you will have rho M, so you can see now what is this, this pi D cube by 6 is the volume, rho f is the density of the fluid so it is the amount of liquid that has been displaced.

So this is the total mass a total weight of this fluid which has been displaced and this represents a total mass or total weight of the material. So this is rho M is density of material that we are looking at. So it could have been metal, it could have been ceramicor anything but particles and this is density of fluid that we are looking at. Now at the according to Stokes law, these two when there is the vertical has been point allowed to settle in liquid, in a fluid then this particle will start to accelerate to the bottom of the, through the liquid it will start to accelerate.

And then the viscous drag will start to increase. And this viscous drag will cause the speed of this moving particle to reduce. And eventually a steady sate or a terminal velocity will be reached and at that particular terminal velocity the viscous drag would be given by. So this is the viscous drag. So this is also in a upward direction. Here D represents the diameter, v is the terminal velocity, eta is the viscosity.

So it is because of the viscosity of the material that this drag is getting generated. Now when a terminal velocity has reach therefore no net force is acting on the particle. And therefore we can write so the two upward forces are balancing the gravitation forces. And from here you can very clearly see or you will be able to obtain a relation for terminal velocity which will be given by this. So the particle is moving side the straight at a terminal velocity and a uniform velocity. And this velocity is now not changing.

Now if you know the velocity of a particle and if you have put this material or this particles in a glass jar of length H and if it takes time to reach then this Vt can also be written as V H over t, where H is the length that it is crossing and t is time it takes to reach the bottom. Therefore using this Stokes law we will be able to relate time it takes to the diameter of the particle size.

And from here let me write down the equivalent diameter. So you can see we have been able to relate D with that t, H is a constant, eta is a constant, g is constant rho M and rho f constant. So for if you now the time you will be able to calculate D. And there are very simple techniques, you don't need to calculate the time for each and every particle. In fact you can put a weighing machine and then, since you know the diameter for a particle time you will be able to calculate the weight, gain, or the cumulative weight as a function of time.

And then for there you can calculate the number fraction that is reaching that is reaching the weighing machine in that particular time. So there are simpler ways to get this. But the overall idea is that you can use Sedimentation as a technique to characterize powder and particularly powder size. So you can also characterize on the basis of size as well as weight.

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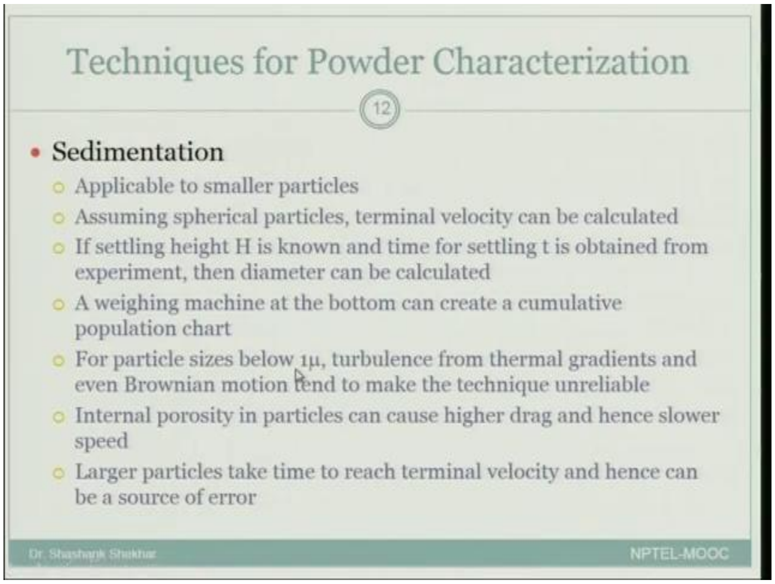

Techniques for Powder Characterization

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- **Sedimentation**
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  - Assuming spherical particles, terminal velocity can be calculated
  - If settling height H is known and time for settling t is obtained from experiment, then diameter can be calculated
  - A weighing machine at the bottom can create a cumulative population chart
  - For particle sizes below 1μ, turbulence from thermal gradients and even Brownian motion tend to make the technique unreliable
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Now for particle size below one micrometer, turbulence from thermal gradients and even Brownian motion tend to make the technique unreliable. So there are some limitation so for this technique and particularly it is because of the point turbulence and Brownian motion. And if fact this one micrometer size is still too small. You would not able to use it even for something like we have said earlier something like 10 or 15 micron. Because this is for the smaller size and the other side, we are the upper side upper end basically the larger size you will not be able to use it for very large particular size.

When the particular size becomes very largewhat happens is that the Renault number starts to influence the overall settling of the particles. Here so far we have been assuming that it is the viscosity which determines the settling and it is quick. But if the particle size is very large there will be two things happening, it will take longer time to reach the settling velocity which is the terminal velocity. And second there will be turbulence because of the increase in the Renault number. And therefore this particular relation will not be valid.

And if fact we will solve one example problem based on this to show that there is upper limit to the particle size. So there is both a lower limit and upper limit to the particle size that can be measured using this technique. Internal porosity may also exist in the particles and that may cause higher drag which we have not taking into account. So far when we took a diameter we assume that it was solid material inside it. But inside but it may be possible that inside the particle or the inside the powder there may be some porosity and because of which the density overall density is lesser than what we have been taking it to be.

And the last one the last limitation we have already mentioned that large particle takes time to reach the terminal velocity and hence can be source of error. So the idea is that there will be technique and there will be limitations and so far we have looked at imaging technique, we have looked that the sieving technique and this is the sedimentation.

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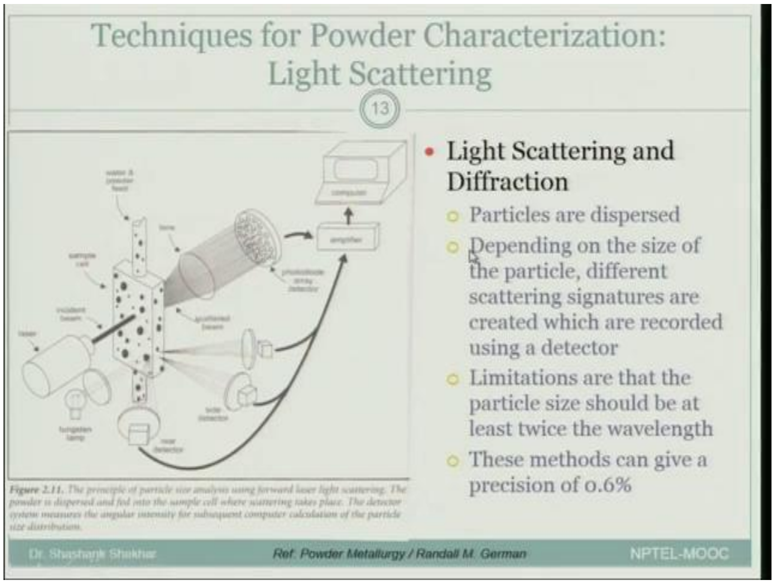

Techniques for Powder Characterization: Light Scattering

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- **Light Scattering and Diffraction**
  - Particles are dispersed
  - Depending on the size of the particle, different scattering signatures are created which are recorded using a detector
  - Limitations are that the particle size should be at least twice the wavelength
  - These methods can give a precision of 0.6%

Figure 2.11. *The principle of particle size analysis using forward laser light scattering. The powder is dispersed and fed into the sample cell where scattering takes place. The detector system measures the angular intensity for subsequent computer calculation of the particle size distribution.*

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Now similarly there are some more sophisticated techniques, one of them is what is described here. It is a light scattering and diffraction technique. So here is a cell into which you allow the particle to move or feed the particle along which some fluid. Now depending on the particle size and particle point morphology there will be different amount of scattering. And that scattering is captured by a photo diode array over here and some more side detectors that you see over here.

And this information are fed into the computer and then you would be able to point depending on the angle of the scattering, you would be able to estimate the particle size that is coming through. And with time you will be able to generate data for large statistic, for large number of particles. And therefore you will have good statistic for it. So depending on the size of the particle different scattering signatures are created which are recorded using a detector.

Limitations are that particle size should be at least twice the wavelength. Now here we are talking about scattering. So this is scattering is taking place because of the point light that is falling on to the particles. Now if the particle size are much smaller than the twice the wavelength then what will happen is that there may be some diffraction phenomenon that may start to take place. So what you want is that the particle size should be at least twice the wavelength.

So although the wavelength will be very small quantity or something of the order of the nanometer. So we are able to get or we are able to characterize particle to a much smaller length than we were able to characterize other techniques that we saw so far, like the sieving technique or the Sedimentation technique. But even here there is lower limit and that lower limit is determined by what is the wavelength of the particles. This method can give a precision of 0.6%.

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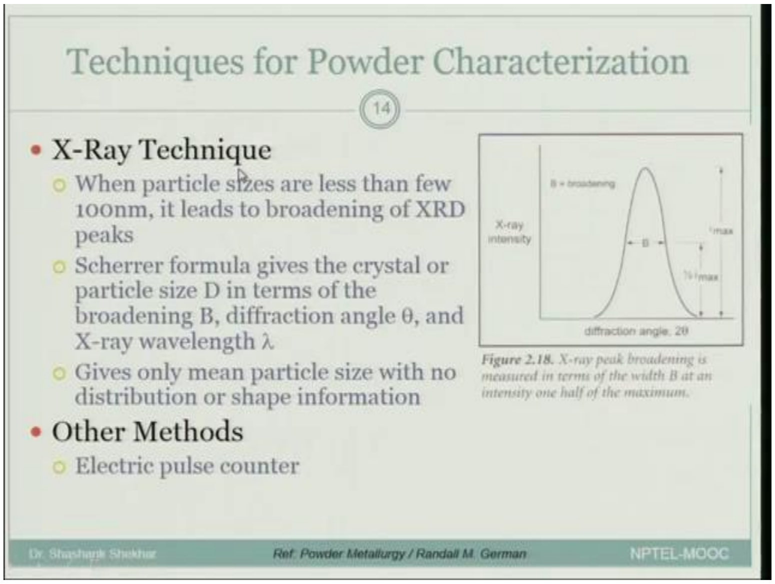

Techniques for Powder Characterization

- **X-Ray Technique**
  - When particle sizes are less than few 100nm, it leads to broadening of XRD peaks
  - Scherrer formula gives the crystal or particle size D in terms of the broadening B, diffraction angle θ, and X-ray wavelength λ
  - Gives only mean particle size with no distribution or shape information
- **Other Methods**
  - Electric pulse counter

Figure 2.18. X-ray peak broadening is measured in terms of the width B at an intensity one half of the maximum.

Ref: Powder Metallurgy / Randall M. German

So now let’s look at a quickly some one more techniques which is commonly used for determining the particle size and this one is x ray technique. Now when x ray, it is well known that point when you are trying to do diffraction. When you are, when you bombard the particles or the material with x ray, then the materials diffracts and you get somewhere a peak maxima because of constructive interference and at that particular point you have the relation and lambda equal to 2D sin theta.

But at other places what is happening is that there is destructive interference taking place. And when you have a bulk which is much larger in size then the destructive interference at the other points is much is complete. So for example let say this is the theta angle were diffraction should take place, so the, if your bulk size is very large then the diffraction will take place only at this place and at other places complete destructive interference will takes place and you will see a very sharp line over here.

However when the particle size or the grain size we are taking about are very small then the complete destructive interference is not possible. And some amount of remnant, remnant peaks or remnant interference, remnant constructive interference takes place. So and that is what leads to these tails over here. So ideally you would have, you would have seen a peak only at that particular theta, this particular theta

But because the particle size is limitedyou see some amount of peak or some amount of intensity at angle less than this theta and also an angle higher than this theta and overall you get what is called as broadening. And this broadening can be related to the particle size. And there are very simple relation, one of which is called a Scherrer relation to find the crystal or the particle size and you can estimate very accurately the average particle size. Although you will not be able to get a distribution out of it But you can get a average particle size that is possible.

So we will end at this particular slide. But there are some more simple methods one of which is listed below and will go through some of this methods before we move ahead point to discuss what are the technique to characterize powder, so see you in the next lecture.