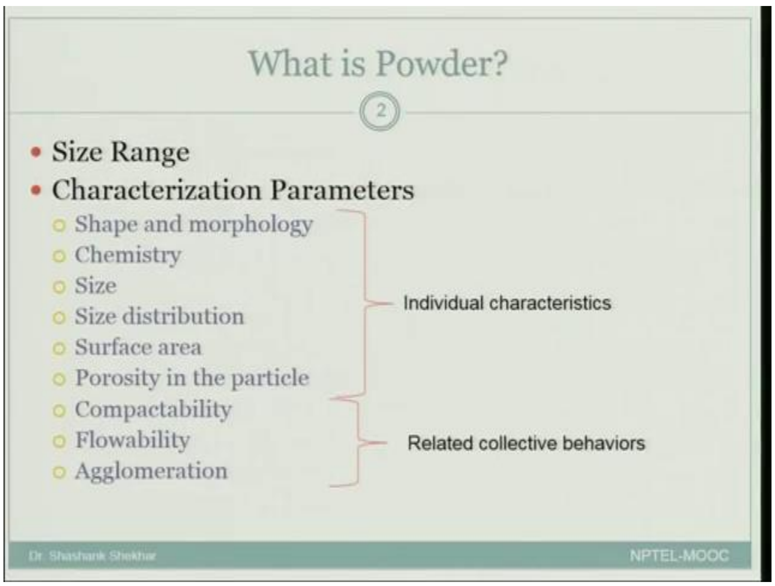
Fundamentals of Materials Processing (Part–1)
Professor Shashank Shekhar
Department of Materials Science and Engineering
Indian Institute of Technology, Kanpur
Lecture Number 28
Powder Characterization

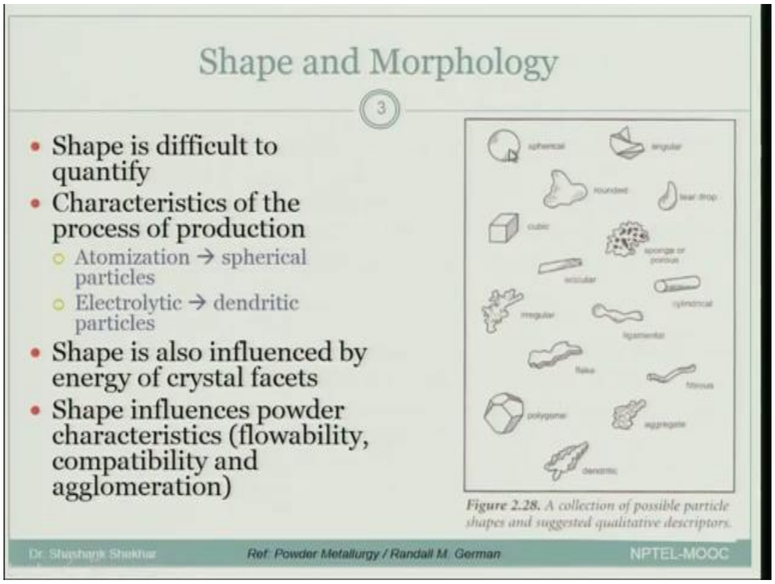
Ok so welcome back to the second module of the course which we have started in last to last lecture which was powder processing. So we are looking at a powder processing. And we have already introduced you to the various concepts and application of powder processing. And we also started with characterization of powder.

(Refer Slide Time: 00:40)



Now in the characterization of powder we look that the size range and you saw that what is the usual size range for powder is. Next we want to look at the characterization of powder. How do we characterize them?

(Refer Slide Time: 01:04)



And in that context we found that shape and morphology of the powder particles can be very wide and a very different it can be a simple as spherical, angular, rounded, tear drop, fluorosis sponge and unimetant you have a shape over there. So there are various kinds of shape that is the possible there. So it is difficult to quantify. Whether you’re talking about quantifying volume, surface area or a anything.

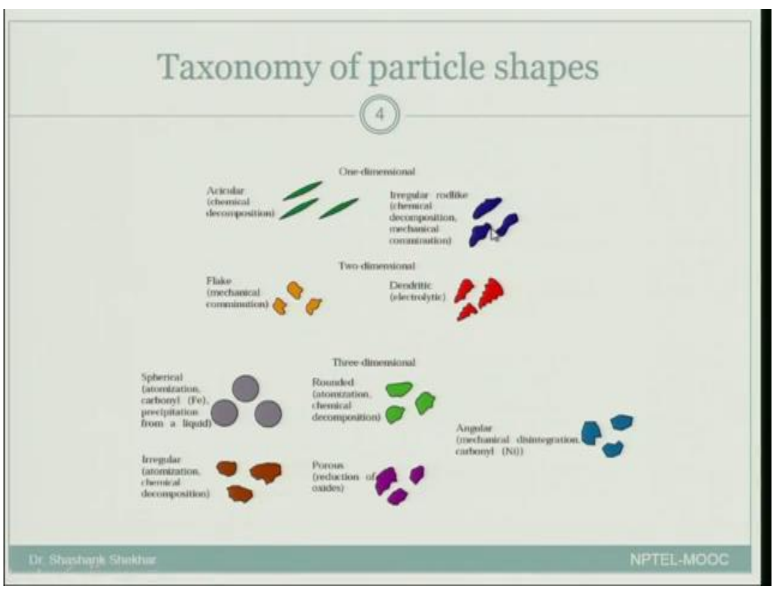
So individual particle bases you cannot quantify. And not only that there may be even variation in one set of powder. So for example even in on a general note what you will see is that all the particle may still be spherical because it will depend on what particular process your obtaining the powder, powder particles. But even in the spherical range you would see that diameter varies. There they may be some amount of ellipsometry.

It may not be a perfectly spherical obviously it can be perfectly spherical that would be art and not science. So that can be wide variation and therefore we need some way to characterize some of the important parameters like area, volume and so on. So will continue into that how do we do that. Now this a shape and a morphology of the powder . Depend mainly on what particular process of production has been used. For example atomization in on an average will always produce spherical particle.

On the other hand electrolytic process introduced something like a sponge or dendritic particles. So the powder shape and morphology is greatly determined by the process that you use. It is also influenced by the energy of the crystal facets. For example some of the crystal facets may be very slow growing and. Therefore they will be overcome by the other faster growing planes. And in that case you will see some faceted structure. So the crystal in faces of energy of the crystal facets. Also influence the shape and morphology.

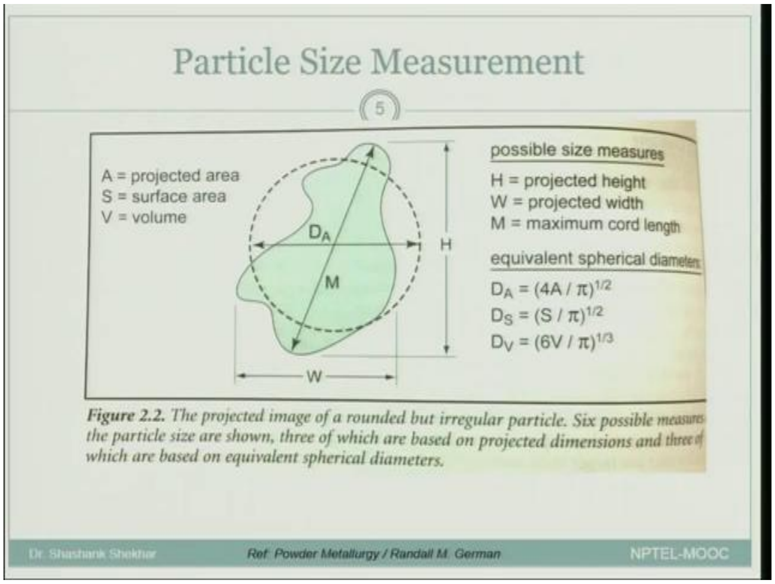
And this shape influences some of the powder characteristics. For example flowability, (compat) compatibility and agglomeration. So even though the average diameter to determine or to define the powders of a spherical size may be same as that for say a sponge or the dendritic kinds of powder. But there flowability, compatibility compactibility actually it is compactibility, how much you can (compat) and agglomeration characteristics can wide in a range. So you have to understand that there is always going into be some inherent variation in the shape and morphology.

(Refer Slide Time: 03:33)



Now we also saw that the, we can defined or we can basically classify them in different classes. For example you can say 1 dimensional, 2 dimensional and 3 dimensional. So for example acicular or rodlite or elongated, ligament kind, can be put as 1 dimensional then in 2 dimensional, it will be fleet type or dendritic type. Which doesn’t have very large dimension in the third dimension and then most of the other shapes will fall in the 3 dimensional categories.

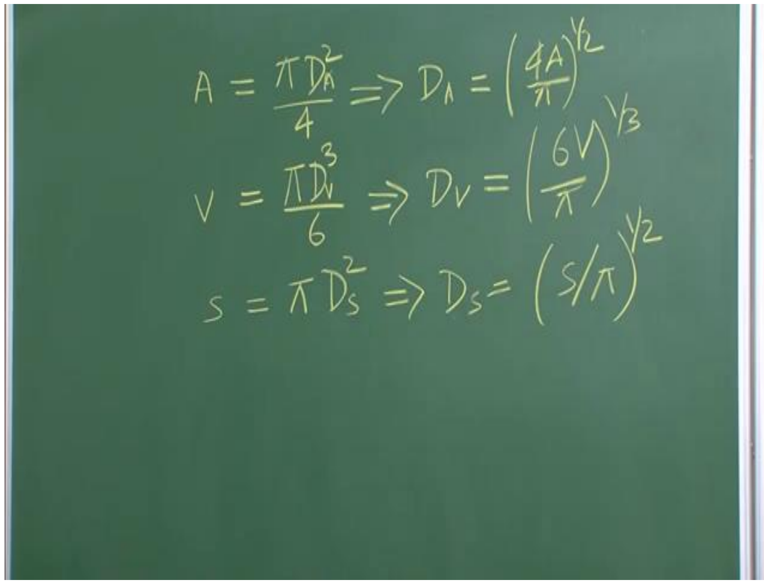
(Refer Slide Time: 04:06)



However whatever be the shape and size. We are always interested in calculating an effective diameter. Now that effective diameter that you calculate or you want to or that will define the size of a powder particle that you have obtained will depend on what is your method of measurement. For example if you, if you have the image, let us say you have obtained some (())(04:31) image or micrographs and from they are you're trying to calculate the diameter size then what your actually looking at is only the projected area. And therefore you convert that projected area into the into the equivalent spherical diameter spherical particle. And then calculate what is that diameter of that equivalent diameter.

Similarly if there is a mechanism or there is a technique why which your actually calculating the overall surface area. Then again what you say is that I will find a diameter of a spherical. Which have equivalent surface area and that is diameter that you will say. Similar and the other one which is the most simplest one if your some way able to calculate the volume. Then that will give you the usual pi d cube by c and that will your equivalent d. So you can see how this are obtained. And if you are not let me just quickly show it on the board on how exactly this will this equations come into picture. So for example let say you have a projected area.

(Refer Slide Time: 7:45)



So what you have, what your say, is that you know the area projected area form some technique most likely some imaging technique. And you say what is a equivalent diameter da which will have a sphere which will have a equivalent area. So that will be pi da square by 4, implies in this case if you knew the area then the da equivalent which determines or defines the size, would be given by 4a by pi square under root. So this is one definition of diameter.

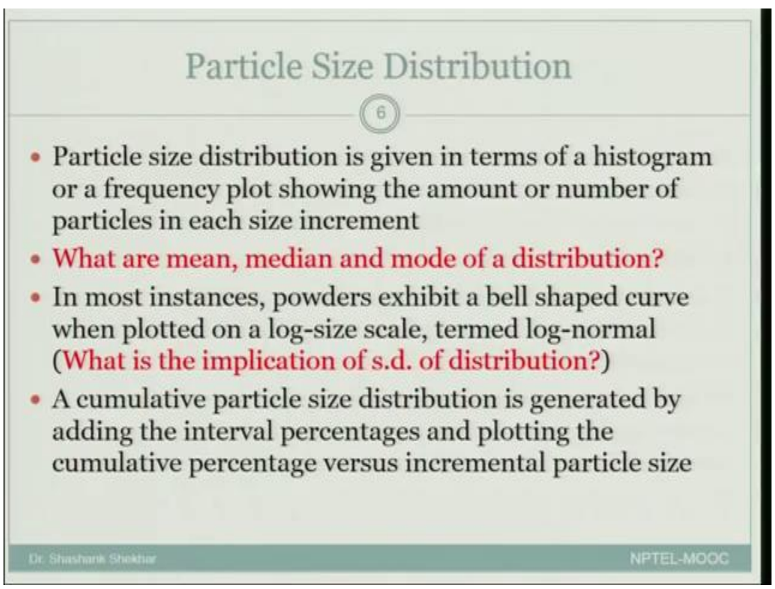
Similarly if you are able to calculate the volume which we uses the easiest one in term of theory but when it comes to technique or a actual characterization, this may be very difficult. So pi dv cube by c implies dv is equal to 6 v by pi cube root of this. And if you know the surface area there are of course techniques. In fact this is the most common technique where you calculate the surface area. So you calculate the surface area and then say what is diameter which has a equivalent diameter.

So spherical particle of diameter ds will have surface area equal to pi ds square. And you're saying pi ds square is equal to s. And therefore the equivalent diameter for this is under square under root s by pi. So these are different definitions. Now you can see that even though your particles may have similarly d value. But it, it need not always represent the same particle size or shape or even volume. Because it depend on how your calculating.

So for example let say you have calculated ds and da for two different sets of particles. One of them we have calculated based on projected area other you have calculated based on surface area. So all though these are, these two are same, but if you were to calculate the volume it may come out different and, and in most of the cases that is how it will be the case. So that is the particle size measurement so depending on the technique you will have different equivalent spherical diameter.

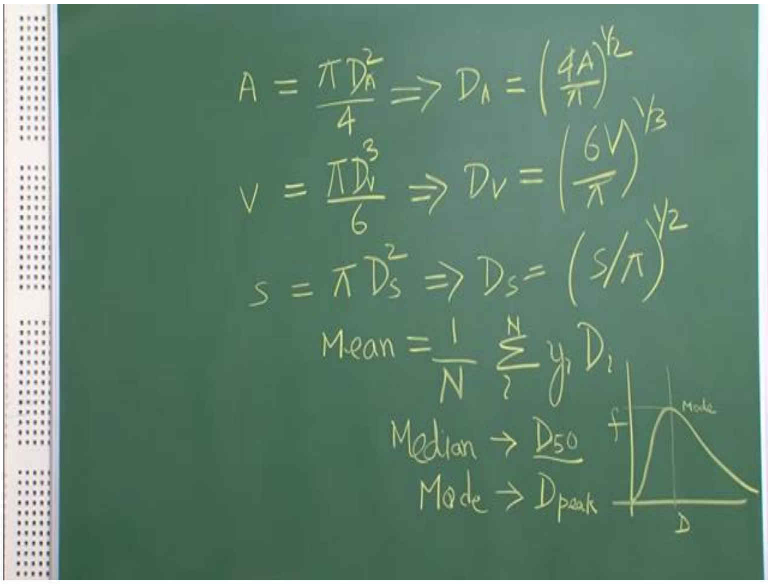
So now that is one particle. But in general you will not get just one particle. You will have you remember we had even the largest powder size which we take we have 10 to the power 9 particle per kg. So that is the kind of particle, number of particle that we are looking at. So you will and all of these cannot have exactly the same diameter. So there will some variations and that distribution is what we need to understand. So particle distribution size distribution is given in term of a histogram.

(Refer Slide Time: 8:00)



Or a frequency plot showing the amount and number of particle in each size increment. So on the axis you will have increment increasing size. So you will have different, different diameter in the increasing order. And corresponding to that for a small re-increment you will find what is the frequency or total number and then you will plot it. So this kind of frequency plot or the histogram is what will defined the particle size distribution. If it were a perfectly uniform size distribution then you would get a straight line.

(Refer Slide Time: 11:22)



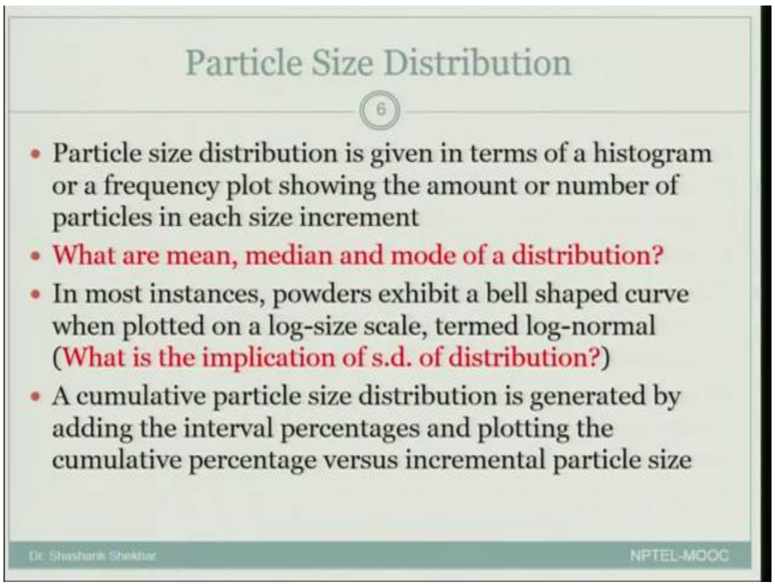
And then you can imagine that is obviously not ideal case. In most impact I should say 100% of the cases that is not what you will get. You will get a distribution and that distribution needs to be defined in terms of some parameters and the most usual parameters are mean, median and mode. So what are mean, median and mode, mean most . Most of you would be aware mean is given by if you have n number of particle then mean would be given by where yi is the frequency of the particle of this size.

So let say di is some diameter then the number of particles in that of that particular size. So this is the frequency and i equal to 1 to n. So these are the different number of diameter. And so you divide by 1 over n. And this is this will give you the mean. Median would be D 50 meaning the particle whose diameter is exactly at 58^{th} percentile. So if you were to calculate or put the size in ascending order then the particle and there are say let say 100. To make it even simpler let say there are 200 particles. Then the 100 or the 1001 depending it is odd number or even number particle. The diameter of the 100 or the 100 first particle would define D 50 and that will be the median.

And another one is mode. So mode is nothing but the diameter at the peak. So if you were to plot the distribution so let say this is frequency, frequency this is diameter. And this is how you getting so whichever is the diameter just at the peak. This is the mode. So these are the user statistical terms you must be a familiar with but just for sake for completion and putting it over here.

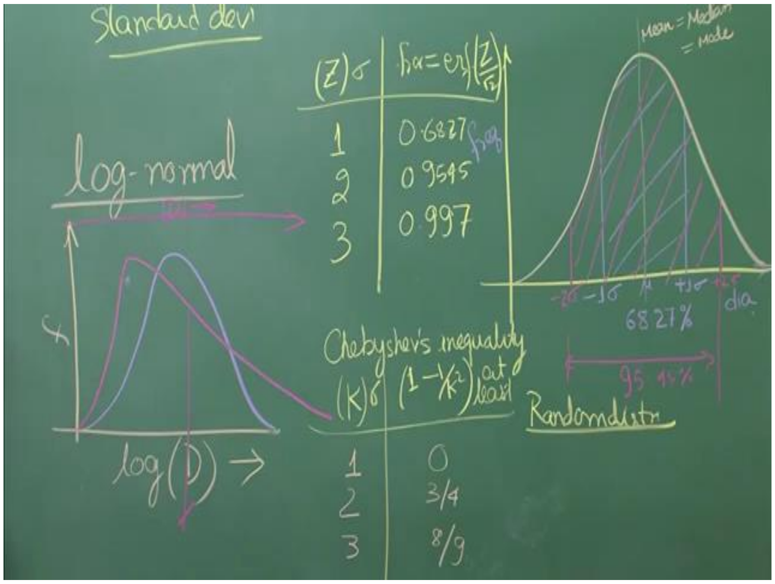
And that will define you're the different-different kinds of ways to define the distribution or the to represent that distribution. But this is just one number and it is, it is quite possible that two different distribution may have the same mean or median values. And still they may have different distribution. Therefore something else is needed to describe this distribution.

(Refer Slide Time: 11:44)



And if that is, that particular quantity which is use very common term to describe this is standard deviation. So for any kind of distribution you would like to have a standard deviation define. And it is the standard deviation which defines what is the variation or what is the range of distribution in the, in the particle size that you have. Now this standard deviation you may have seen that it is just given as one value. But what is the meaning of this standard deviation one value. Let say this is some the over here. We are given some diameter. Let say D is equal to 50 micron. And standard deviation is given as 5 microns. So what is the meaning of this 5 micron. Does it say that certain fraction of data should lie within this.

(Refer Slide Time: 21:37)



What is this standard deviation? It is important to know this. So the important of this standard deviation is particularly for distribution which have normal, which are called as normal distribution. So let say I have, let me use few more colours over here. To define this in a better way. This is called a normal distribution or a bulk of distribution. Because it look like a inverted bell. And there is mathematical equation which defines it. It's not just the schematic shape and you can call it anything as a bell curve. So it the form, it of this form and now this will be it is symmetric in nature with respect to it maxima. So this is where your maxima lies and over here if I say this is my mean so in this particular case mean will be equal to median because it is symmetric and it will be equal to mode.

Now what is standard deviation? So let say there is some value of plus minus 1 standard deviation. It is usually denoted by sigma. So I have writing like this plus 1 sigma and minus 1 sigma. So what standard deviation defines is that whenever you have a distribution like this then at least 6 not at least actually exactly 68.27% of your overall data should lie within this range +- , - sigma to + sigma. So this on the x axis what do have here, you don't forget that this is actually a diameter.

So corresponding to this diameter you have some frequency. So this is describing a distribution and this is some value. So this is some diameter for example in our previous, what we took as an example it was 50 microns. And we also said that 5 micron is the standard deviation. So this is -5 micron and this is +5 micron, from this mean value. Which is usually denoted by mu. So this for this particular case it will 50 micron, this will be 45 and this will 55.

So between 45 to 55 microns we should have 68.27% of your particular size. So now you see you can get a quantitative feel of how much powder lies on number. What is the number of powder particles that lie within a particle size range? Once we have this value standard deviation. So standard deviation does define the width of distribution. Similarly you can get +, -2 sigma to +2 sigma. So in this case it since sigma was 5 microns so this becomes -10, so 50-10 this is 40 and here will have 50+10 so 40 to 60.

Now within this range you should have 95.45% of data. So this is not just arbitrary number, these have been a calculated from the form of equation and you can predict how much so how much data will lie within that. So for example let say Z number of sigma we are talking about, then the fraction can be given by erf Z by root 2. And therefore if you are saying 1 then this is erf 1 over root 2 which is what comes out to be 0.6827 or 68.27 %. When you say erz equal to 2 then this comes out 0.9545 and so on so.

You can write keep on going like this by putting in the values appropriately over there. And so we as talking about standard deviation, so let me also quickly say that it is not necessary that you will always get a bell cure or a normal distribution. You may get some random distribution. So in there what will happen in those cases? This particular equitation is valid only, only and only for this kind of distribution. There may be other symmetric distribution. Then may be other, beautiful looking distribution.

But that is not what this is meant for. It is only meant for this particular form of the distribution which is called the normal or the bell curve. If you have any other distribution and even random distribution. There is another way to define the fraction of data that will lie within that and that is called Chebyshev's inequality. What it says is that if you are within K sigma then at least 1-1 over k square at least, remember that is the important word here.

Here it was exactly; here it is at least 1-1 over k square data should lie within +-1 sigma. Now if you put k equal to 1 over here, what you get is zero now that is very surprising. If you go to k equal to 2, you get 3 by 4 or 0.75. If you go to K equal to 3 you get 8 by 9. Ok, other numbers look ok but what is a meaning of k equal to 1. It says only zero data lies inside it that does not make any sense, but then again if you go to what i said earlier at least. This it is saying at least this many data.

So it is saying that we can nothing can be predicted above +-1 sigma when distribution is a random distribution. It can have zero data. It can have lot more than zero data. But not much can shared, but only you go to K equal to 2 which is 2, +-2 sigma then at least 3 per 4 data must lie. So whatever be the form of distribution, no matter even if you draw random distribution on your own still when you have the mu value then you calculate the standard deviation from that then at least 0.75 or 75% of the data will lie within K – mu – sigma to mu + sigma.

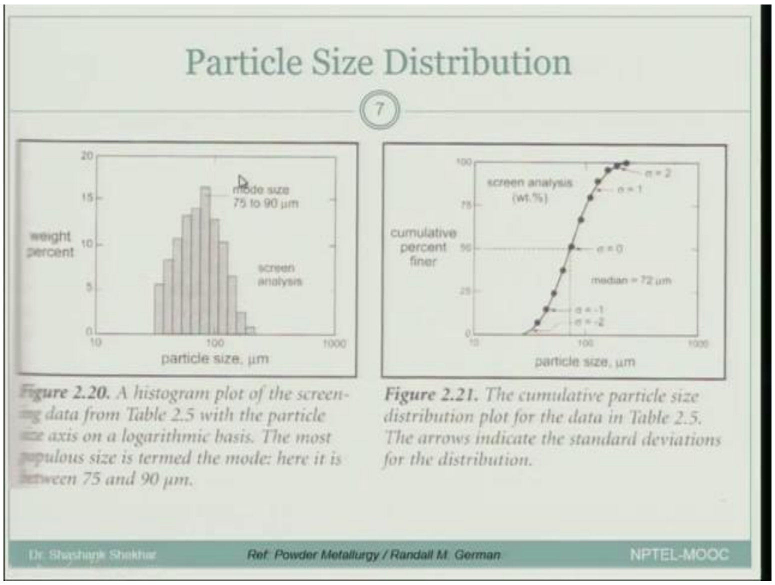
This is again a very powerful equation or powerful relation which predicts great deal about the distribution. However what you would find when you, when we go on is that . For the powder that we get from a particular method most of the time the distribution is what is called as log normal distribution. It's not normal it's not random its log normal distribution.

What is log normal distribution, it is similar to normal distribution except that you have to take diameter, log of the diameter. And once you plot frequency versus log of diameter then you would get the usual bell curve. So we have this log normal distribution how did it have look like. Instead of log D what have taken on D, it would have been right is skewed. So you see this not a normal distribution and here the axis is D. So let me draw this x axis over here. So this is D and not log D.

So when you have log D. This is what the distribution looks like. And when you have D this is how distribution looks like. So here you will not be able to apply the -+ sigma rules or basically, whatever the but to estimate what fraction of data should all within that. But if you use the log diameter values then you can use this value. You can use that relation to find how much fraction of data lies within a given set of given standard deviation.

So this is what the distribution is and that is the implication of standard deviation of distribution. So you can get a lot of information as you see when you have the mean and the standard deviation and particularly when you have a normal distribution. If it were not a normal distribution then you would see that you will not get be able to get not that more information that will discuss very soon. Only if because only because in most of the cases no matter what technique you use whether you are using atomization or electrolytic technique the particle size distribution that you get in most of the cases log normal distribution. And then we can apply some of the result that will see very soon.

(Refer Slide Time: 22:23)



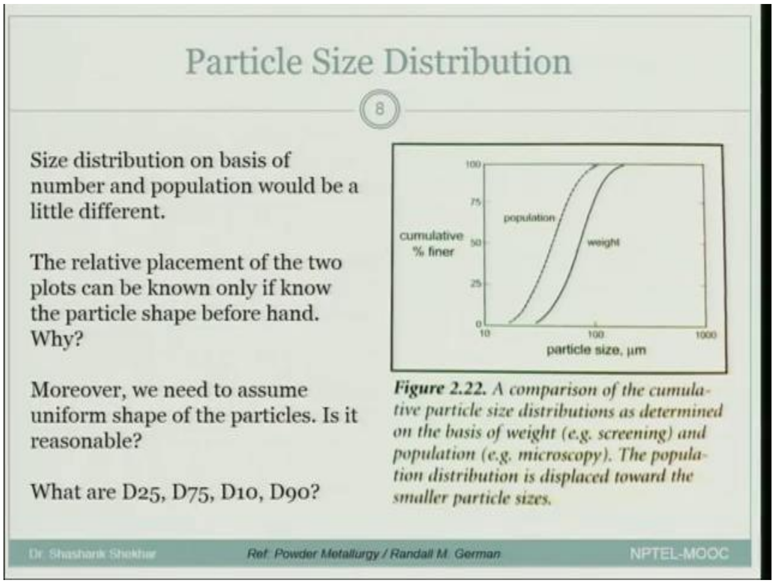
Now this is what a histogram would look like. So you see this is a particular size and you can have number over here or weight percent over here. So it can be frequency or the weight percent. So you can plot is as on the number bases or the weight percent or the weight bases. So there are different ways to plot it. It need not always be on the bases of the number. Other ways to plot in the bases of the weight or surface area and so on. And this the histogram and corresponding to that you can also draw what is called a cumulative distribution So what is cumulative distribution, it is basically adding all the incremental information in to the previous data.

So at this point whatever the data was you add the next from the histogram next set of information. Into this point and so on you can build a curve like this and this is called a cumulative percent data. So this is also a very useful way to see how grow the distribution is now here. You can even find what is where is a mean. There you can define as sigma equal to zero. And then you can define sigma equal to -1 sigma equal to +1 and corresponding to that you will have diameter.

And another thing is you see in both the both of this two plots x axis is in log diameter because that is when you get the normal, log normal distribution and you can see it is roughly log normal distributions. If we had a lot more statistic then it would have been much more smother curve.

But right now from the data it seem that the statistic is not too large. So you don’t see a very smooth curve over here. It could become very smooth if you have a much larger statistic. And this is the corresponding cumulative distribution.

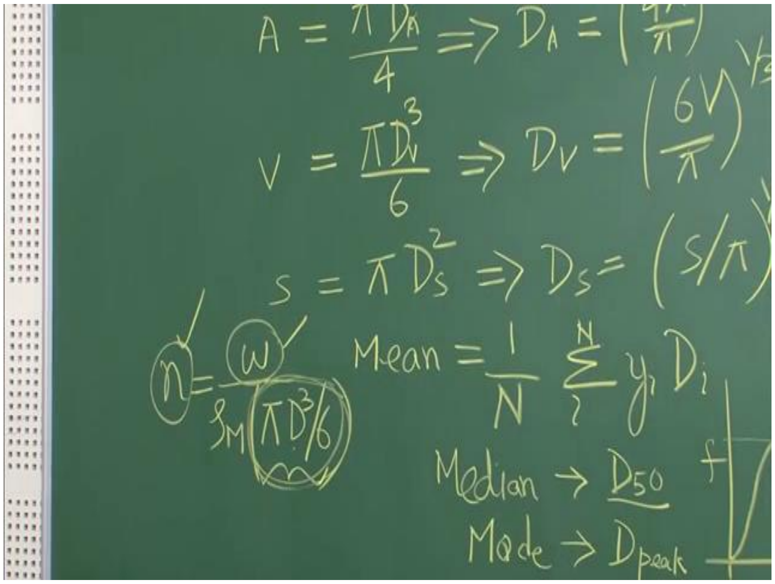
(Refer Slide Time: 24:18)



Now if we talking about cumulative distribution. We should also know that the size we have already mentioned that size distribution. That we have that we plot can be on the basis of number or on the basis of for example weight. So there are different basis on which you draw the plot. For example here this is the number basis, so basically population. So this is a cumulative plot. Similarly you can draw a weight basis plot.

So here the weight is increasing as we increase the total number of size. So the weight of all the particles up to this point is added at this point. At this point you can see all the weights have been added. So it is 100 % at this particle point 0 % of particle size lie and therefore zero it has 0% data. So that is what it is representing and there is, it is the, these two plots are for the same distribution, one on the basis of number and the other on the basis of weight, so that you have to keep in mind.

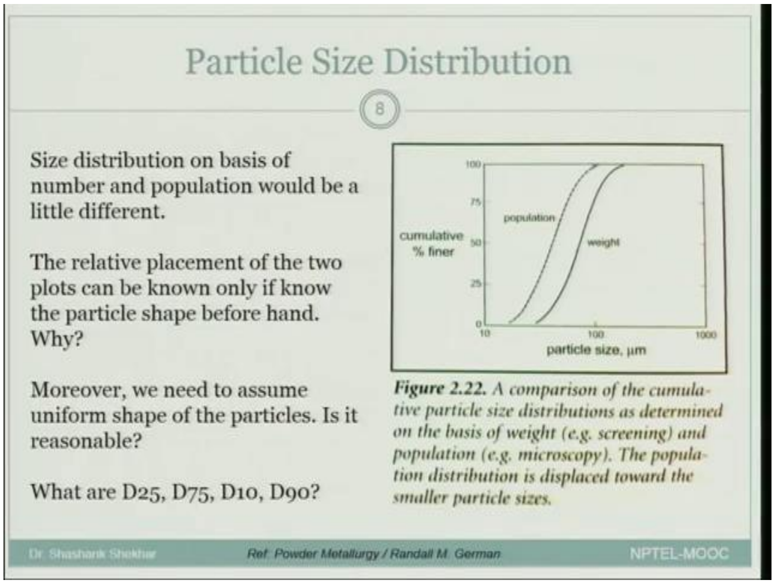
(Refer Slide Time: 26:36)



And this will be displaced. Now the amount of displacement will depend on what is the particle shape. And why will it depend on the number particle shape, because we know the relation, you remember, we talked about relation where we said if n is the number then n number per unit weight, sorry not the number per unit weight, but the total number and w is the weight or that particular mass then number of particles in that weight is given by pi depend cube by six.

Now you see we have assumed here. A spherical diameter, a spherical particle, if we had assumed some different size then this quantity will be different. So this is your weight basis plot. This is your number basis or population basis plot. How much it is shifted is determined by this value. And this value will depend on whether you’re taking spherical particle. If you had a (())(26:12) then this will be a very small quantity. It may have a constant thickness so it will become pi D square by 4 into h. So it will be proportional to 1 over D square instead of 1 over D cube that is over here. So the amount of shift will depend on what particle size you are assuming.

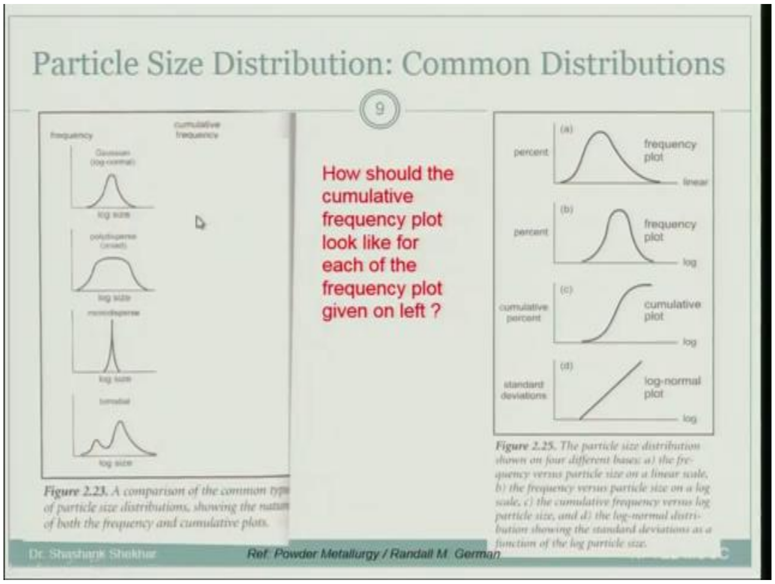
(Refer Slide Time: 26:36)



Now another thing is that more, we also need to assume uniform shape of the particle that we have already mentioned. And again ask you question is it a reasonable assumption. So we have already made that answer. And the answer was that it is a reasonable assumption as now as we are using in the same process to generate the particles. So the same, same process would give you on an average similar shape of the particle size. Similarly size of the particle.

Will it get what is D50? Now similarly you can also determine or define what are D25, D75, D10 and D90. D25 will be the diameter of the particle, weight 25 % lie below this. 75 will be diameter of the particle, Where 75% lie below in terms of may be number or in terms of may be weight. But whatever it is it depend depending on what particular basis you are taking, number basis of weight basis. You will, you can get D25, D75 similarly you can also get D10 D90 other values like this. Why are we interested like this, will become very obvious in the next slide.

(Refer Slide Time: 27:47)



Before that lets look at we talk about this cumulative plot, so how would the cumulative plot look like for D is different kind of variations. Now you feel this is the only normal log normal distribution other are not log normal distribution. So we would be able to use this particular values or the particular relation that we described our standard deviation, only for this one but not for other. However cumulative plot we can still find for each and very each one of this. So look at it try to estimate what should be the cumulative plot for each of this .

For Gaussian distribution for upwardly disperse yet the distribution is wide. Here the distribution is very narrow. Here you have bi model. Bimodal mean is at this point you have a mode; at this point you have a mode. So there are two modes. This is the global mode at the global maxima. Here you have a local mode or local maxima. So these kind of distributions what should be the cumulative frequency plot. You try to do at your own and we will reveal the answer in the next lecture. So see you in the next lecture, thanks.