Powder Metallurgy Prof. Ranjit Bauri Department of Metallurgical and Materials Engineering Indian Institution of Technology Madras

Lecture - 22 Measurement of particle size and size distribution

Hi everyone. Welcome back to this lecture series on Powder Metallurgy. In the last class we were talking about the particle size measurement, with regard to this powder characterization that you have to do in order to evaluate the properties and characteristics of the powder particles.

(Refer Slide Time: 00:49)

Particle size measurement Techniques



Microscopy – Population based

It is a direct method to obtain both particle size and particle shape
Particles larger than 1 µm can be observed by Optical microscope.
For finer particles SEM needs to be used. SEM also shows surface topography and permits compositional analysis.
Image analyzer attached to the microscope can measure dimensions of the particles.

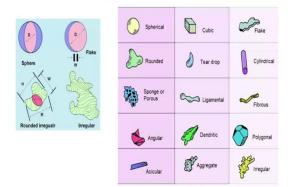
>This technique is not suitable to measure particle size distribution.

And with respect to that, one of the most important characteristics of the powder would be the particle size. And therefore, we need to discuss about the particle size measurement techniques, and see how it is done and what kinds of instruments are used and so on.



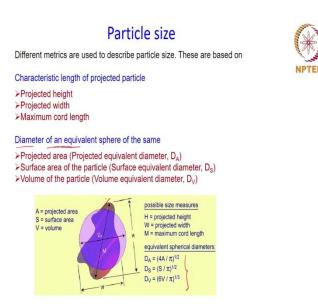
Most of the fabrication processes generate complex shapes.
Particle size and shape affect the flow, packing and compressibility of the powder.
Characteristics dimensions depend on the shape of the particle.

Particle size and shape



In the last class if you remember, we have defined this particle size and shape, especially for particles which are irregular or do not have any particular well defined shape.

(Refer Slide Time: 01:49)



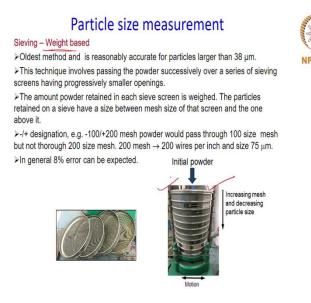
For those particles, we had seen in the last class, as to how you can define the size with the help of the equivalent diameter of a sphere having similar projected area, similar surface area or similar volume as the particle whose size is being measured.

Based upon that, we derived the three kinds of diameters which can be used to define the particle size for the irregular shaped particles. And then we were discussing about the

measurement techniques, and with regard to that I had also mentioned that a particular measurement technique uses a certain basis for measuring the particle size.

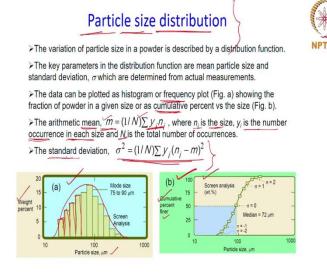
For example, the basis for the microscopy technique is population; that means you actually see the particles or the population of particles under a microscope. And then, physically measure the size of these particles using some software.

(Refer Slide Time: 03:30)



And then, we have also seen the sieving method which is weight based method. Here you use a series of sieves to let the particles pass through them depending on their size, and at the end of the sieving you see how many particles or how much particle is being retained in each of the sieves. And measure the weight of those particles. So, that is why this technique is weight based.

And we had discussed as to how it is actually done by using a set of sieves like this (slide above), where you have this series of sieves or screens in a progressively increasing order. And once you load the powder and do the sieving action, you can get the amount of powder retained on each of these sieves, and do the analysis in terms of the weight fraction and represent the data like this (slide below). The weight fraction can be plotted as a function of the particle size to get histograms as shown below and obtain the particle size distribution. (Refer Slide Time: 04:51)

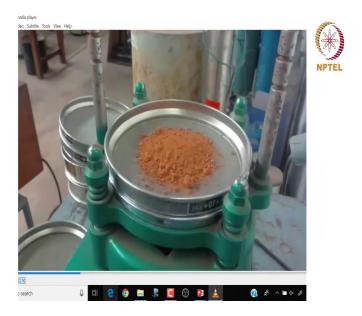


(Refer Slide Time: 06:32)



In the above figure, from left to right the size of sieves is decreasing.

(Refer Slide Time: 07:41)



So, a certain amount of powder is taken and loaded on the upper sieves depending on the expected size of the powder, you can also choose a particular number of sieves instead of taking all of them. So, here the powder is first loaded on a particular screen size or a particular sieve, having a particular screen size. It has not gone through that sieve which is on the top. So, unless we shake it and give it a motion this is not going to go through. So, that is the purpose of having that shaking motion.

(Refer Slide Time: 09:04)



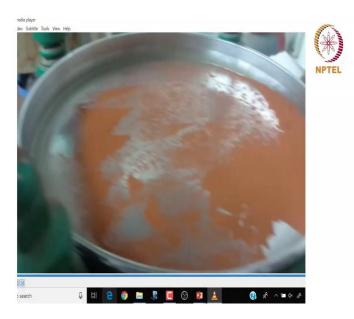
Once the shaking motion is given, the powders will go down through the screen depending on the size of the particles.

(Refer Slide Time: 09:42)



Here out of these two knobs one can be use to control the motion and the other can be use to control the time period.

(Refer Slide Time: 10:26)



So, after the shaking is over after a given period of time that is set in the machine, you can see that certain amount of powder has passed through the upper sieve and has come down to the lower one.

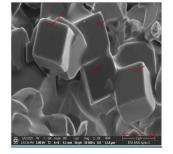
So, now we are going to collect this powder and measure the weight of it. The weight fraction for a particular size that is being retained here will be calculated. The weight of powder retained on the upper sieve is also calculated similarly. This is a simple method of measuring particle size.

In microscopy, you collect a set of images from the microscope and then you digitally analyze those images to get the particle size. So, there are standard softwares available which can do that, and today I am going to show you an example of how you can use one of those to calculate the particle size from the images which are captured under microscope.

(Refer Slide Time: 12:43)

Particle Size Analysis by Microscopy Technique



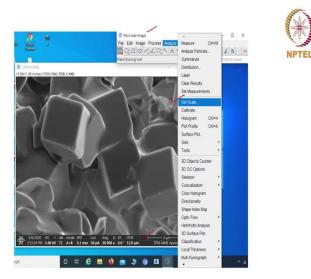


Here is an example of measuring particle size by the microscopy technique. Let us say we have this scanning electron micrograph, where you can see this kind of cuboidal particles, which were observed under the microscope and these images were captured. And here you can also see a scale.

So, that is our micron marker and with the help of the scale of this micron marker that we have over here, we can do the analysis and measure the size of each of these particles individually.

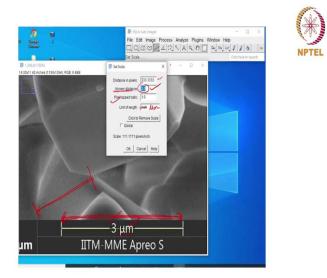
And then, once you collect all the size by using a number of images like this which are taken from the same sample, you can take an average of all those sizes and get the average particle size. So, let us see how this is done by using particle size analyzer software.

(Refer Slide Time: 14:02)



So, this is a software called imageJ. And this allows you to calculate the particle size using different kind of methods or techniques; we are going to take one of those as an example just to demonstrate.

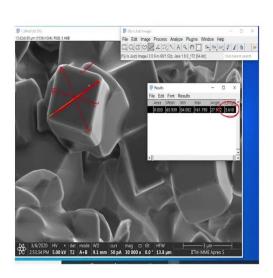
So, the first thing that you need to do to get the information on the particle size is to set the scale; that means, you have to define a known length. And on the basis of that known length whatever other lengths that you see can be measured. So, as I said the known length here is the micron marker that we have the scale which is already given on the image.



So, once you click that set scale, then it will give you this box, where in you can input the known distance. So, in this case you can see this is 3. And then, depending on whatever unit is mentioned (here it is micrometer) you need to change that. This is the calibration procedure.

So, in the next step you go ahead and take the length across the particles, and measure them with the help of this length scale which is already set over here.

(Refer Slide Time: 16:58)

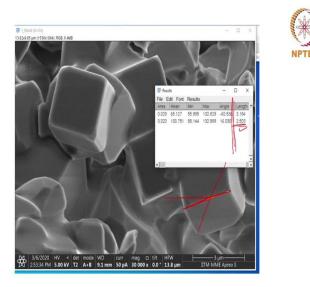




So, you simply take the option of drawing a line. And start drawing the line across the particles like this from corner to corner to measure the length of this particle. Since a particular length has already been defined before, any length of the line that you draw over here can be easily measured.

So, that is what you can see over here, this is the length of this line which is from one end of the particle to the other end. So, this gives you an idea about the size of the particle. And to be more accurate, you can do this measurement across different directions. For example you can measure the length across these two corners or across these diagonal also, then you can also measure like this and so on. And then, you can take an average of all this which will give you the average size of this particular particle that you see over here (slide above).

(Refer Slide Time: 18:35)



Similarly you can see the next particle is this one, again the same thing is being done. You measure the length across this particle in a particular direction like that. And now we can see this is around 2.5 for example, and as I said, for this particle also, you can measure from other angles or other directions like this.

So, for all the particles you have in a population in a given area of the image, you can calculate the size of each of them in this manner. And then, you collect this data and you can take an average of that and that will give you the average particle size.

So, this is also a simple technique, where you can get the particle size by simply measuring those lengths. Such techniques also allow you to measure the particle size by other approaches. Like in this case we had used the length of a given line segment, there are other approaches that can be used to measure the particle size.

For example you can capture the images and instead of taking a line segment, you can draw an outline around the particles. And then calculate the area of that. And from that area you can get the particle size as the radius of a circle having identical area as the particle. So, that is another kind of approach that can be adapted to derive the particle size from those images.

Then this is the kind of data that you obtain (slide below). And it can be plotted in two ways, when you are talking about the particle size distribution. Apart from particle size, the particle size distribution is also important because, in a given powder sample generally there will be more than one size. When the particle sizes remain same throughout the sample lot, the particle size distribution is said to be monodispersed.

So, that is what we call as the particle size distribution and it is also important to derive that information when you are doing the particle size analysis. So, this histogram is one way of doing it, where you can see the fraction of each size class in terms of a histogram, which actually shows you the frequency or number of occurrences for a particular size.

And the other way of representing the same data is known as a cumulative plot. So, in a cumulative plot, the previous size classes are all summed up and then, it is plotted as the cumulative percent of the smaller size.

From the particle size distribution, we can get some parameters like the average particle size. The mean or the average particle size is a very common way of reporting the particle size of a given powder sample. And that is the arithmetic mean of all the sizes that you have obtained in the powder sample.

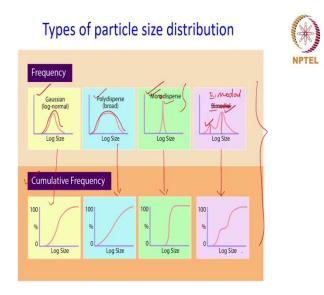
To obtain the mean particle size, you sum all those size classes and divide that by the total number of occurrences. So, here in this particular equation

$$m = \frac{1}{N} \sum y_i n_i$$

 n_i is a particular size; y_i is its number of occurrences and N is the total number of occurrence. So, that is how you will get the arithmetic mean m. And then you can also obtain the standard deviation from the equation

$$\sigma^2 = \frac{1}{N} \sum y_i (n_i - m)^2$$

(Refer Slide Time: 25:17)



And as far as the types of particle size distribution is concerned, there can be various types of distribution depending on the kind of powder you have or the fabrication method used to make that powder and so on. In a Gaussian or log-normal distribution you can see a kind of bell shaped curve.

And then, it can be a very broad distribution like this (slide above). And this kind of distribution is also known as poly-disperse where you can have a wide range of the particle size in the same powder.

The complete opposite of this is known as a mono-disperse wherein, you can see a single peak; that itself indicates that all the particles are of equal size. And that is why you have this one single peak and that is how the name mono.

Then, you can also have two peaks like this what you see over here (slide above). So, this indicates that there are two sizes which dominate and thus two peaks will be seen in the plot, instead of a single broad curve. Since there are two dominant sizes, this kind of

distribution is known as bimodal. And the same curves are being represented in terms of the cumulative frequency. So, each of these plots will correspond to the ones which is above (slide above). So, this is how the particle size and distribution data is represented.

And with that we come to the end of this class.

Thank you for your attention.