Particle Characterization

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Module No. # 02
Lecture No. # 04
Morphological Characterization: Techniques of Shape Assessment

Good morning and welcome to lecture 4 of the course on Particle Characterization. In the last lecture, we started talking about shape assessment techniques, and we kind of went through the history of how particle shapes have been measured; we also discussed various categories of particle shape assessment, and we classified particle shape analysis methods into methods based on single particle analysis, methods based on analyzing properties of bulk powders, mathematical techniques, and verbal descriptors. These were the major classifications of particle shape assessment methods.

I also said that in each of these classes, you can further categorize the methods. For example, single particle analysis methods can be further classified into methods that are based on measuring the distances between tangents that are parallel to the contour of the particle, methods that are based on shape comparators, and methods that are based on measuring the lengths of specific types of intercepts. In this lecture, we will first begin by discussing these three methods in a little more detail.

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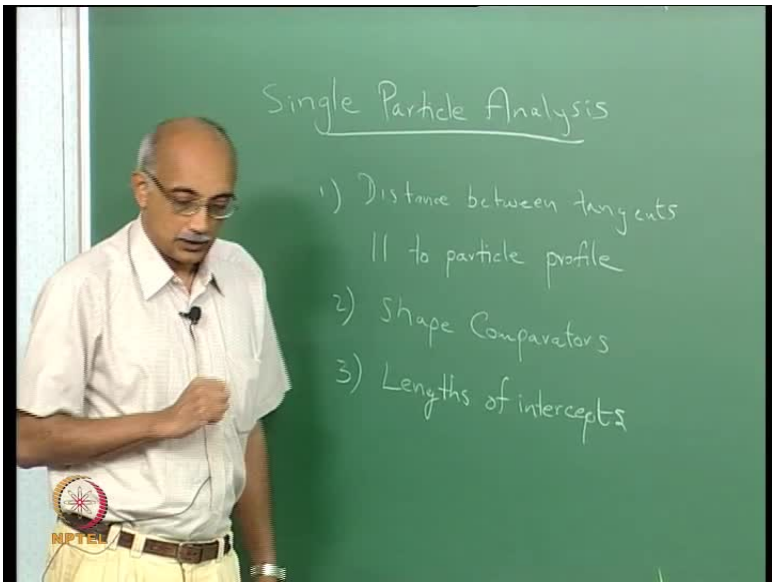

The first method is distance between tangents that are parallel to the particle profile. The second is shape comparators. The third method is lengths of intercepts. In a way, these were the earliest methods used for shape analysis. The reason for that is, because in the old days, the equipment that was available to us for doing this characterization was fairly crude; you could not achieve very high magnifications, or if you could, it was very expensive. So, people had to do with limited resources both in terms of analytical equipment as well as to some extent the expertise of the people who were using these equipments.

What they tried to do was minimize the number of data points that they took on each particle in order to do its shape characterization. Many of these techniques are essentially geometric techniques. So, they tried to reduce a 3-dimensional object-like a particle to a 2-dimensional profile, and then do the shape assessment based on this projected 2D profile of the 3D object. Certainly, limited in its scope limited in its effectiveness, but it was a right thing to do at that time. So, if you look at some of these methods, today, they may look very unsophisticated, but at the time when people started using these methods, they represented the state of the art so to speak.

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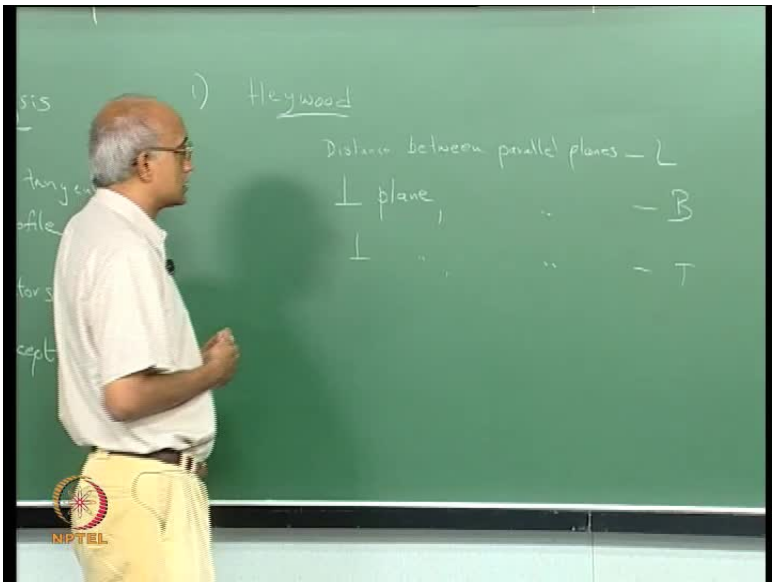

The first method – distance between tangents; the guy who really innovated this was a person by the name of Heywood, who took the profile of the particle; he measured the distance between parallel planes in one arbitrary direction and he called it L. So, the selection of this L dimension, was entirely arbitrary. Then, what he did was – took a plane that was perpendicular to this plane, again, measured the distance between tangents that were parallel to the counter in this perpendicular plane, and he called that B. Then, he took a third plane, which was perpendicular to both the first and second; again, he drew tangents to the profile of the particle in this plane and measured the distance between the tangents, and he called that T. So, this L, B and T; obviously, represent length, breadth and thickness.

However, Heywood really made no effort to classify them as such; he did not say for whatever reason that the longest dimension should be called L, the shortest dimension should be called T, and the intermediate dimension should be called B. The advantage of the Heywood methodology is the simplicity; it did not require a quantitative comparison between the three dimensions that he was measuring, but for every particle, it yielded three dimensions, which could be used as a shape representation.

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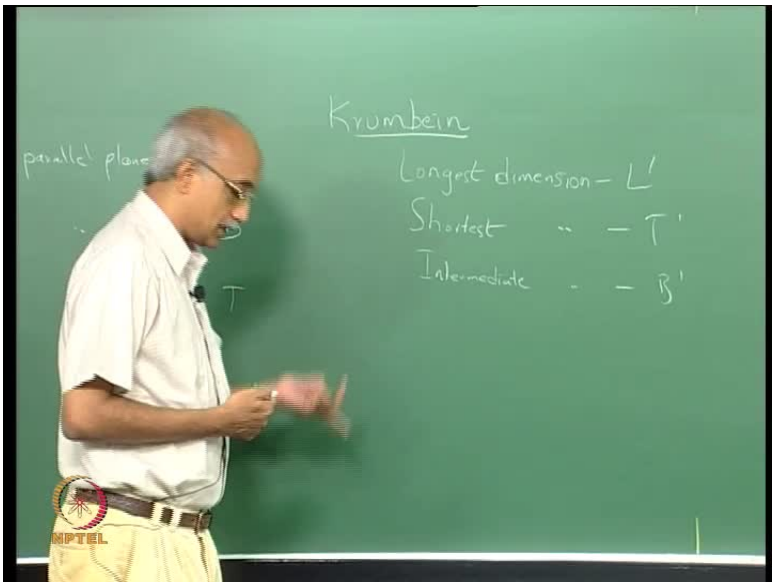

After this, there was a person by the name of Krumbein, who took this analysis one logical step further; he said the longest dimension that is measured in this fashion should be called length, L prime and he said the shortest dimension should be called thickness or T prime, and then the intermediate dimension will be called breadth. So, almost a trivial extension of what Heywood had done.

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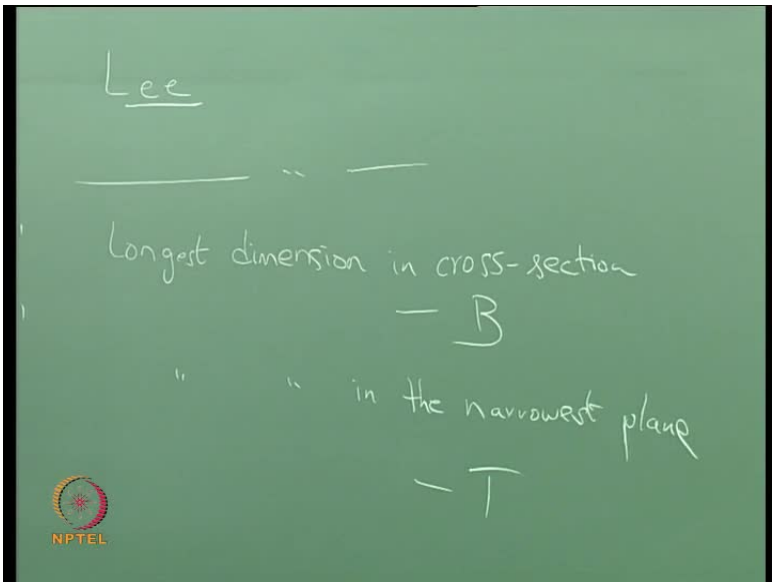

After this, the third person that really got involved in shape analysis was a scientist by the name of Lee, who made this approach more systematic and in a way, more

convoluted from an analysis viewpoint. He also said that we should start with a longest dimension and label it as L, but then, what he said was that we should take the longest dimension in the cross section of a particle and call that B. He said – take the longest dimension in the narrowest plane of the particle and call that T.

Now, the key difference though between Lee and Krumbein in particular was that Lee said – these dimensions do not have to be orthogonal to each other; they do not have to be perpendicular to each other. So, L, T and B could essentially be chosen as any three axes that represent the diameter of a particle in the various planes. As long as they represent the longest dimension requirements in these various planes, they could be taken as L, B and T. With this definition, it became easier for different groups of researchers to look at the same particle and characterize it by the same dimensions.

The problem with the Heywood approach is that even though it may have worked very well for his own research group, it was very difficult to share the results across labs and get convergence from various groups on what the actual dimensions of the particle were. So, this approach by Krumbein and later by Lee certainly made it a lot more systematic. This was really the first effort at shape characterization by identifying three critical dimensions of the object. This led to then, the second method of shape analysis, which is shape comparators.

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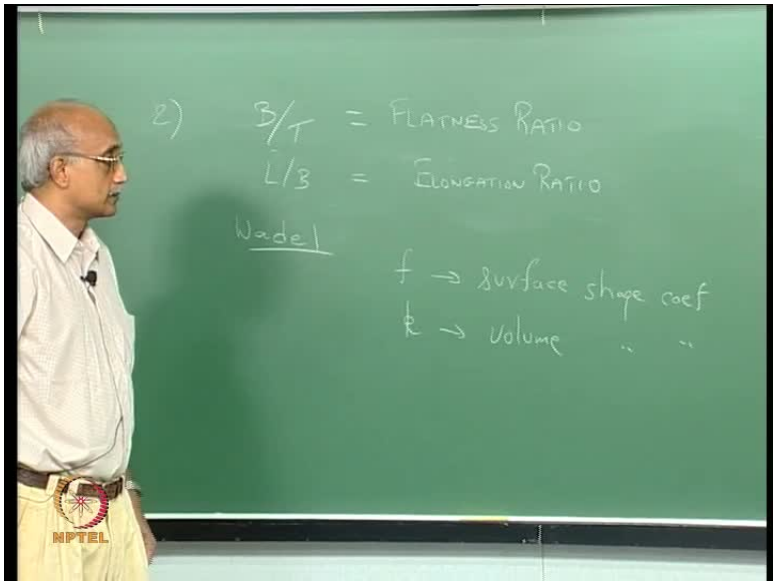

Now that you have measured L, B and T, you can always take for example, the ratio of B over T and you would call that? What would that represent? It was called flatness ratio. Similarly, L over B was called elongation ratio. What do these ratios represent? Why do we take ratios? It is the same reason why as chemical engineers, we try to non-dimensionalize everything. The reason is to be able to compare across systems. If you have two particles and you want to compare the flatness of these particles, then this ratio gives you a non-dimensional value that you can use to do this comparison. So, such ratios that are derived based up on your fundamental measurements are called shape comparators. These were actually defined by Heywood.

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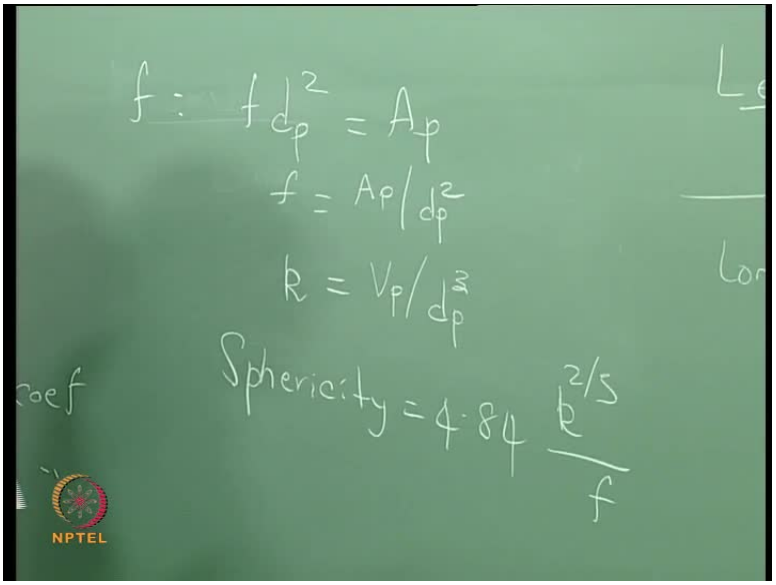

Now, there is another person by the name of Wadel, who defined his own shape comparators. He did that by first defining something called f, which is a surface shape coefficient and k is a volume shape coefficient; where, f is derived by essentially taking f times d squared equals p; where A p is the projected area of the particle and d p is the projected area diameter. So, the way you calculate f is simply by A p over d p square. Similarly, k was defined by Wadel as the volume of the particle divided by d p cubed. He then proposed shape comparators that were based upon these values of f and k. For example, he defined sphericity, which is obviously a measure of how spherical the particle is. His definition was sphericity equals 4.84 times k to the power 2 by 3 over f, which is a non-dimensional value by the way. Here again, what he is doing is similar to what Heywood did; he takes certain measurements of the particle whether its length,

area, or volume and then develops non-dimensional ratios that enable us to compare shapes of different objects.

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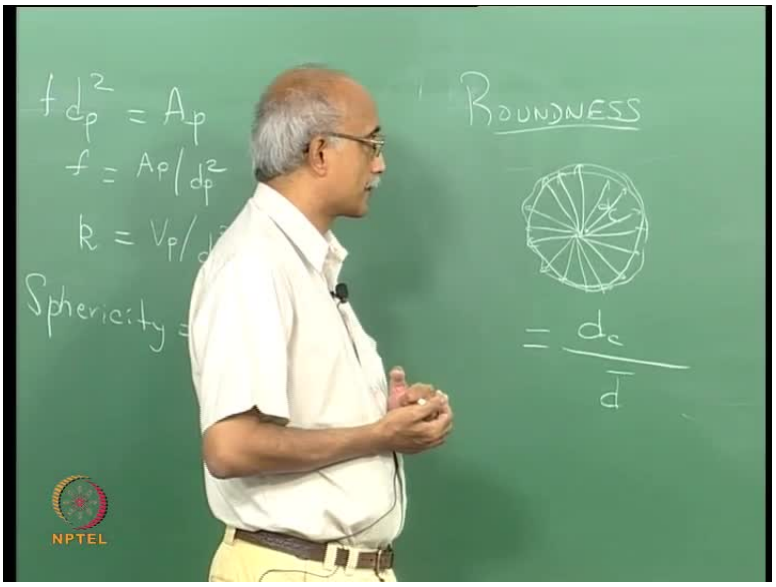

Another shape comparator that Wadel came up with was roundness. The way he defined roundness of an object of a particle was essentially by taking the profile of the object in a in a 2 dimensional plane, inscribe as a circle inside it, which gives the best fit. From here, you take the diameter of this inscribed circle, which gives the best fit to the profile. That actually goes in the numerator; let us call this d c. He then divided that by the mean diameter of all the curvatures that are present in the particle. So, essentially, he would draw radii towards all the peripheral points of the object, take the average of the actual radii or diameter that linked the profile of the object to its center; he would then average it and obtain a mean diameter that represents the actual profile of the object. So, the idea is that if you have a more rounded particle, this ratio would tend to 1, because d c will be equal to every d value; whereas, the farther the deviation from roundness of the object, the greater will be the deviation of this ratio from 1, because it could be greater than 1 or lesser than 1 depending on the shape of the object.

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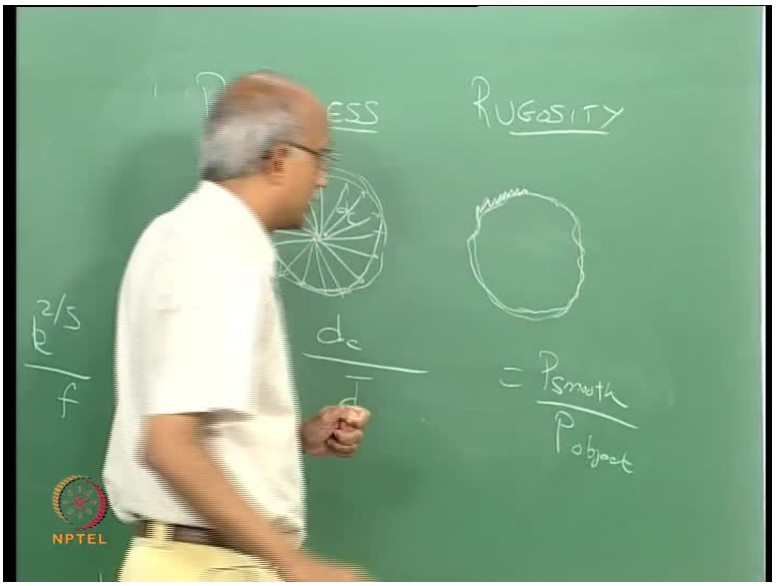

A similar shape definition that he came up with was called rugosity. Rugosity is a measure of surface roughness; how rough the surface is. The way that this definition works is in some ways similar. You take an object, which may be very rough, particularly in a 3 dimensional scale around the periphery; you fit it with a smooth profile; you take the perimeter of the smoothened profile of the object and divided it by the actual prevailing perimeter of the particle.

Now, when we say prevailing perimeter, you actually have to look at all the roughness elements that are present on the surface and you have to take the total length. So, you have to move along the roughness profile and measure the actual perimeter of the object using that technique. Again, this ratio gives you a quantitative indicator of how rough the surface is. The rougher the surface, the smaller will be this value, because the effective perimeter of a rough object can be many times the perimeter of a smooth object with the same radius, for example. So, all these definitions of flatness ratio, elongation ratios, sphericity, roundness, rugosity – are all examples of that second class of shape analysis methods, that is, shape comparators.

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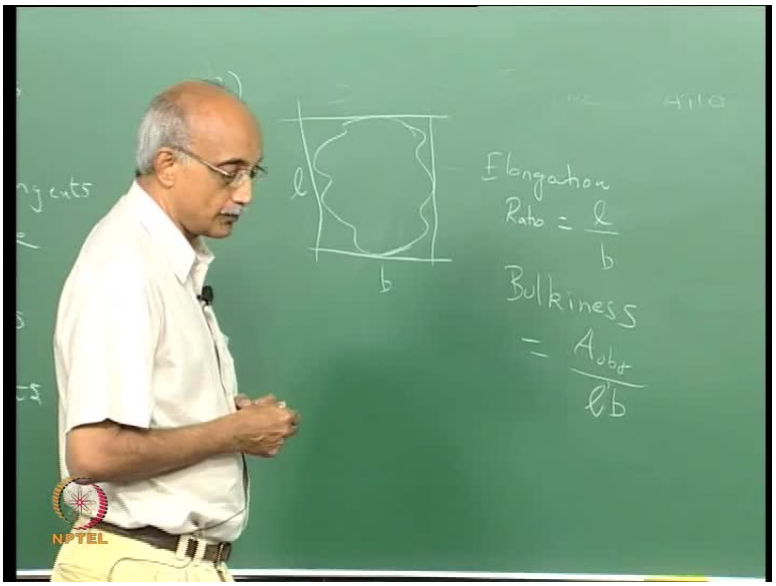

Let us look at a few more examples of how these shape comparators work. We go back to Heywood. He also defined certain shape values; the way he did it was again by taking the particle projection in 2 dimensions; he would then inscribe it in a rectangle, which completely encapsulated the object. So, it will be the rectangle with the smallest dimensions that could completely encompass the object. He would call the longer dimension here as l and the shorter dimension as b. He would then define an elongation ratio for the particle as simply l over b.

Now, the other thing that he also defined was a bulkiness index; how bulky is the object. This he did by essentially taking the area of the object in two dimensions, the projected area and dividing it by a times b. So, the larger this ratio, the bulkier the object.

A is what sir?

l times b. Again, these are examples of shape comparators, which can be very conveniently used by research groups working across several labs to come up with the same description of a particle.

Now, other people have actually taken some of these analyses and extended it to more classes of particles. In the early days, much of the work was obviously done with fairly large objects. So, particles were millimeters to microns in size. So, shape characterization was a very different challenge in those days compared to today when

many people are dealing with nano particles. The extension of some of these techniques to nano particles and sub-micron particles is something that particle scientists are still going through, because some of these methods do not really lend themselves to use as you start approaching the nano dimensions. As the particle dimensions shrink, it becomes more and more logical to use mathematical techniques, digitization and so on. However, these methods have their own attractiveness; they are very simple to use and very intuitive. A definition of something like this for bulkiness, you can intuitively understand.

If you ask a computer to come up with a measure of how bulky it is, it will probably take a long time to do it and then it will give you a number, which you probably would not even understand physically, what it means. So, there are certain inherent advantages the way that we used to do shape analysis. Even though they were very cumbersome, time consuming, and sometimes hard to reconcile between various researches, they did have the advantage of really capturing the essence of the shape of the object.

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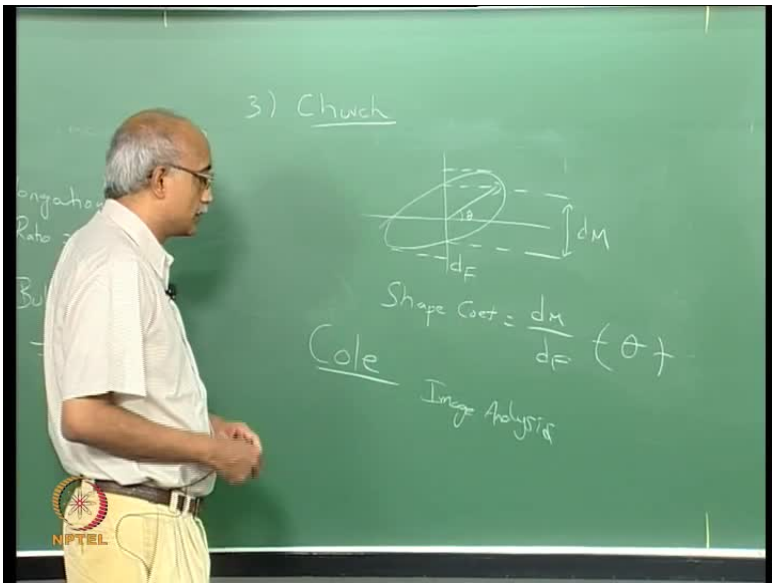

Let us talk about the third type of method, which is the length of intercepts. We briefly looked at an example of this in the last lecture. There was a person by the name of – I guess the first one was Church, who developed the technique that I outlined in the last lecture. You take an object or a particle and take a microscope, you orient the particle at some angle theta, and then you measure two intercepts: the shorter intercept with the y-

axis is called the Martin's diameter and the longer intercept with the same axis is called the Ferret's diameter, d F. Church, basically said that a shape coefficient could be defined as a ratio of d M over d F.

Here again, it is really a measure of elongation more than anything else. This method was most popular for the use of elongated particles; the closer this ratio is to 1, the more spherical the particle is; the more elongated the particle is, the greater will be the difference between d M and d F. So, the ratio will be much smaller. So, for a highly elongated particle, this ratio will start approaching 0. So, the way that Church did it, was to do this analysis for a few random values of theta, then average the data, and statistically analyze the data to come up with a metric for the shape of the particle.

A person by the name Cole took this idea and just made it more systematic. So, he said why do not we do a proper image analysis study using a computer and image capture visualization systems, and look at as many values of this ratio, which is a function of this value theta. So, let us increase the number of theta values to as larger number as possible until we have a converging value for this ratio.

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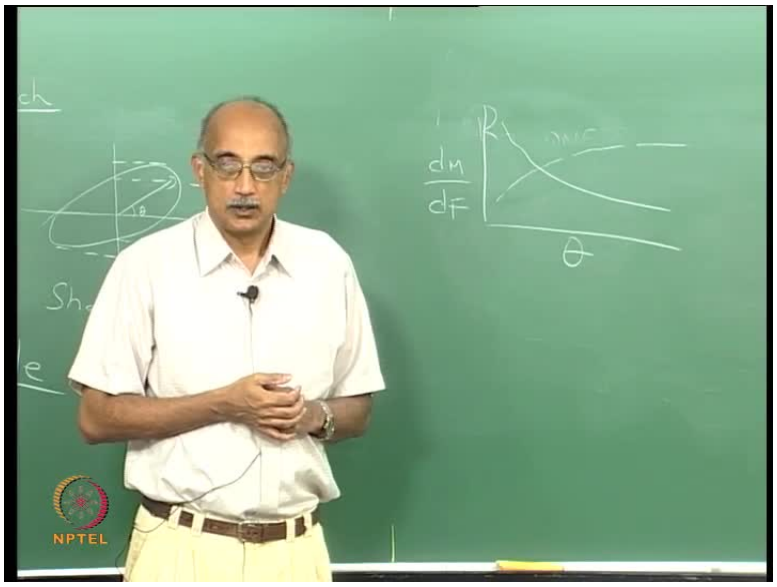

The Cole technique was to plot theta versus d M by d F and essentially, reach a converging value after a certain number of theta values. So, essentially the image analysis and the calculation of the Martin's and Ferret's diameter, will continue until you reach a limit, which could happen either this way or this way of course, but essentially

what you look for is a value of this ratio, which is invariant with change in theta, because at that stage, we have essentially converged to a definition of these diameters, which you know will not change just by taking more observations so to speak. So, these two methods are very similar, very closely related to each other, and they are based upon intersections of the 2 dimensional profile of the object with the vertical axis in your eye piece in a microscope, for example.

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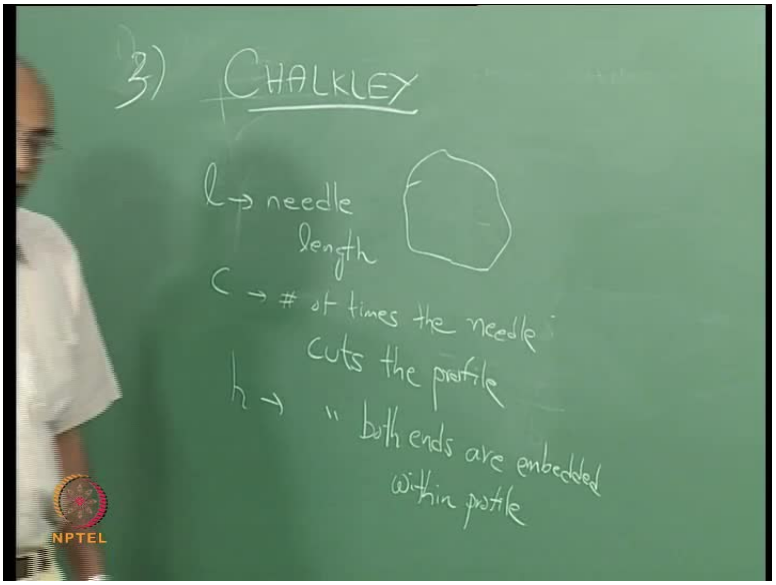

The third method under this category of lengths of intercepts was contributed by a person called Chalkley. He is the one who proposed the needle throwing method that I again briefly described in one of the earlier lectures. He said – if you have an object and I want to know essentially either the ratio of the diameter to the perimeter or area to volume of this particle, then a way I can do it is to take needles of some known length, l and throw it with a known force across this profile. Let us say that C represents the number of times the needle cuts the profile and h represents the number of times both ends of the needle are embedded within the profile.

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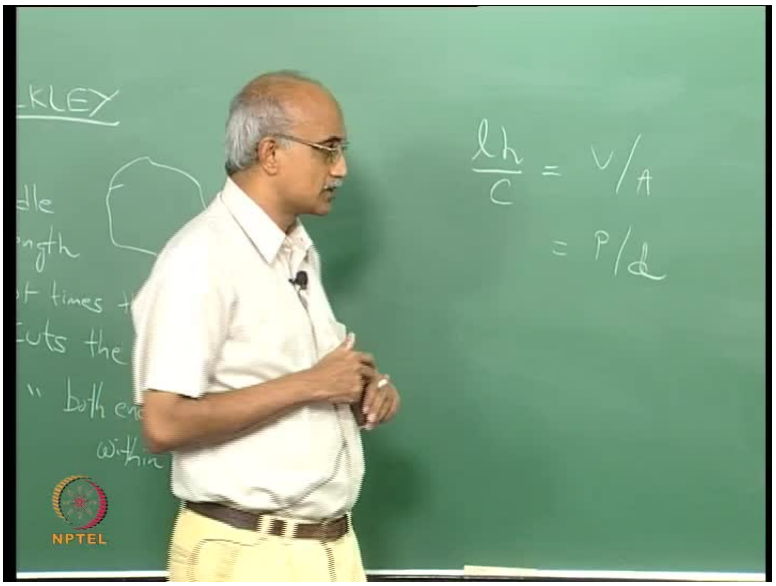

Then, he said – if you just take l multiplied by h and divide by C, this will give you essentially again the way that the quantity of material is packed within this particle, which is what we call shape. So, the idea is that if you take the length of the needle and you multiply it by the number of times they are both embedded within the profile, that tells you that the profile is very long essentially, because the more elongated the profile of the object, the more likely that the needle will become completely encapsulated. So, if you take l h by C, that should give you an idea of volume per area of the particle; the projected volume to the projected area of the particle. Basically, it would say that for a given area, if a needle is likely to get embedded in the profile, it is likely to have more volume, which is essentially an indicator of how bulky it is. So, l h by C; you can look at as an indicator of again how bulky the shape is.

Similarly, you can also look at a perimeter over diameter. Very roughly speaking, there should be good correlation between these two values with probably some constant multiplication factors. However, this ratio of l h by C should be a reasonably good representation of both the volume per unit surface area of the particle as well as the perimeter per diameter of the particle. So, these are some interesting attempts to characterize particle shape in a very intuitive way instead of resorting to highly sophisticated visualization or measurement or mathematical techniques. However, these were still designed to probe individual particles.

Now, the other classes of analysis methods that I had mentioned earlier were those based on bulk properties of powders, which simplifies the problem even further. So, we will discuss that briefly.

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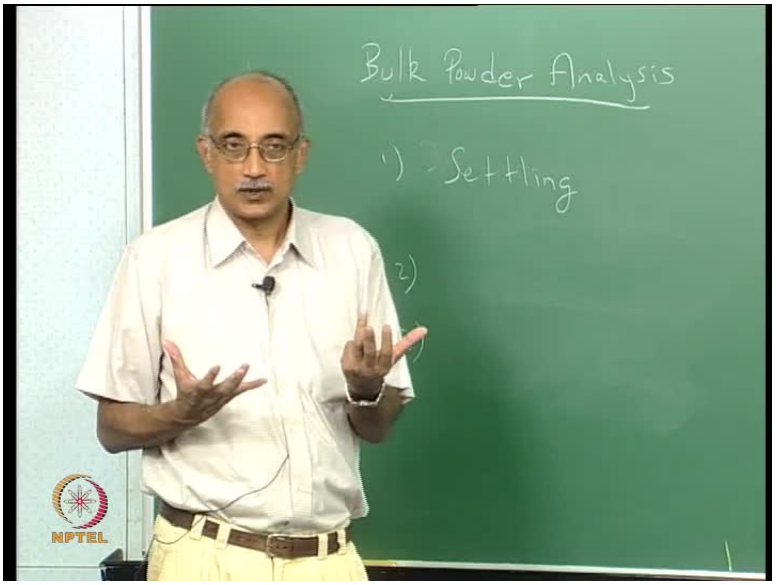

When we talk about analysis of bulk powders to extract shape, what we are trying to do is actually look at predominantly transport and storage characteristics of these powders, and from that, obtain information about shape distribution within these powders. So, one of the key parameters is the settling characteristics of a powder; settling not in the sense of particles settling in a fluid, but rather settling during storage, which is represented in the density. Essentially, if a powder is stacked in a very loose fashion, the bulk density would be very low, because there will be less mass occupying more volume. As you compact it further and further, the density will keep increasing.

Supposing you take a loosely packed powder and measure its bulk density. Then, you did something to this powder to make it settle and become more compact, and then you measure the density again. The ratio of the bulk density in the free powder form verses the density in its packed form is actually an indicator of shape distribution within the powder.

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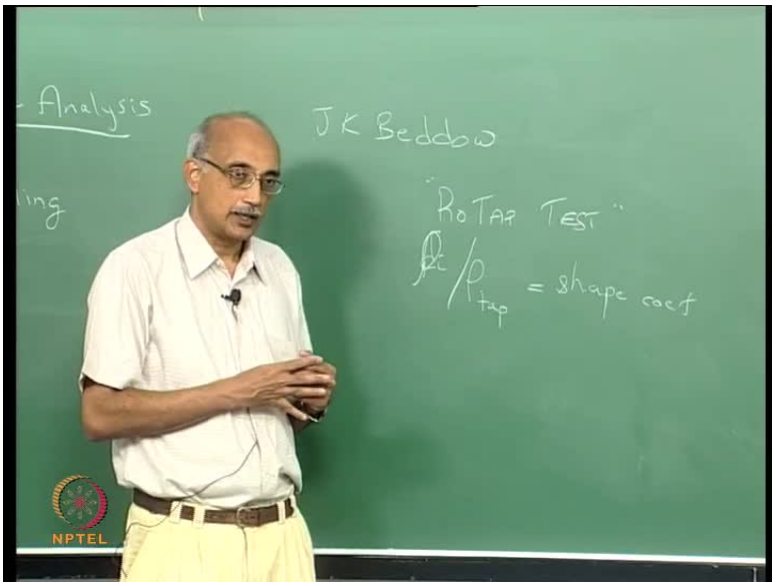

There is a person by the name of J K Beddow, who came up with a test – let us call the Ro Tap test, where he took this concept and made it in to a systematic procedure. The Beddow test essentially specifies how the powder is prepared for testing; it specifies how the bulk density is measured; it specifies how the powder is made to settle. In this case, it is basically done by using a hammer in a rotational setting to repeatedly tap on the container in which the powder is stored. So, as you tap on it more and more, it settles and essentially reaches an ultimate bulk density value for a highly densified powder. So, you take the ratio of your initial density rho i to the value of a tap density. He defined this as a shape coefficient for the entire powder. So, what is the relationship between the ratio of initial to tap density and the shape distribution in the powder?

When you think about it, if we have a powder containing very regularly shaped and very uniformly shaped particles verses a powder that contains highly irregular and highly non-uniform shaped particles, their settling characteristics will be very different. You would expect that if you have a powder that is formulated with all smooth spherical particles and you tap it, what is going to happen to it? It is going to settle into a highly compact, almost sintered type of a powder; it is going to have a very high density, because the surfaces are going to be very conformal. On the other hand, if you have a powder in which the particles of varying shapes ranging from highly spherical to highly elliptical and if you have a highly non-uniform distribution of shapes in the powder, the settling is not going to happen easily, because if we have two particle side by side and one is

spherical in nature and the other is needle-like, the surface area of contact between the particles is minimal. That alone as we will see later, prevents cohesive behavior. So, by simply doing this tap test, you can fairly, clearly identify whether a powder has predominantly spherical particles or predominantly non-spherical particles.

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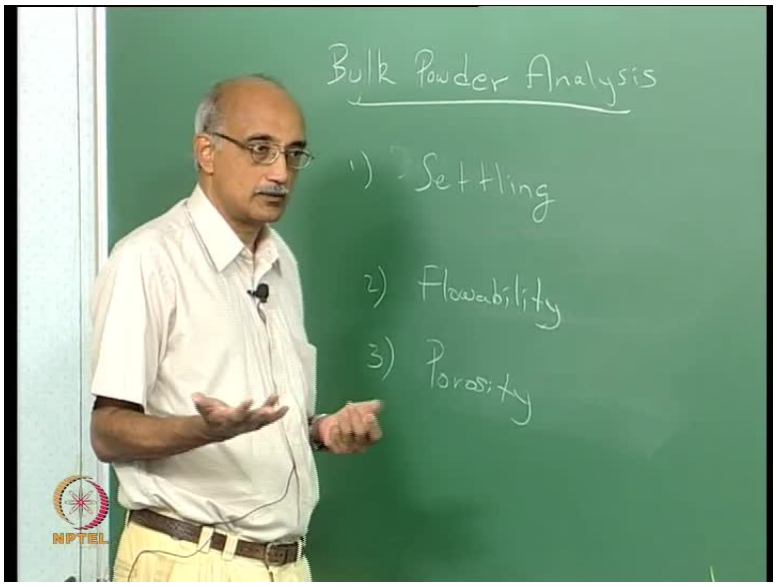

The other way that you can characterize shape distribution is by looking at flowability. Here again what you are taking the advantage of is the fact that a highly cohesive powder will flow like a slug or a solid; whereas, a powder that is not cohesive – that the particles remain as individual entities, will continue to flow like a Newtonian fluid. So, by looking at essentially the flow characteristics and characterizing how Newtonian or Non-Newtonian it is, you can actually get a good idea of how cohesive the powder is and from the cohesion characteristic, you can actually extract the shape distribution. Again, the basic thought process is – the more similar the particle are and the closer to sphericity they are, the more cohesive they will be during their flow process. So, this will give you kind of a macroscopic estimate of the shape distribution of particles within the powder.

The other characteristics that you can look at would be the porosity of the powder under different compaction stresses, which again is really a measure of how closely to the particles want to make to each other, how much interfacial space they are going to leave when you compact them. Here again, the primary consideration would be how much does porosity change as you compact the powder. If the powder porosity is greatly

reduced when you do compaction, again it is an indication of highly uniform and nearly spherical particles in the population; whereas, if the porosity does not really respond to compaction, that would be an indicator that you have particle that really or highly dissimilar and highly irregular. So, these are functional methods of testing shape. If you are using these particles in a powder form, in a process industry, and you primarily care about how they flow, then what is the use of doing the single particle analysis that we had described earlier? All you really care about is – when I make this powder flow, how is it going to flow, am I going to see Newtonian flow, or I am going to see Non-Newtonian flow?

Again, goes back to the point I made in my first lecture that particle characterization is very tricky business and you want to simplified as much as possible. If you can get away with just doing bulk property characterization of powders, fine, go ahead and do it, do not even bothered with characterizing at single particle level.

Any questions on these two techniques?

So far, we have talked about single particle analysis and bulk powder property analysis. The third class of shape analysis methods that we discussed was mathematical techniques. Now, if single particle analysis kind of it is in the middle, bulk property analysis represents one end of the spectrum that you are really minimizing the amount of information you collect on in individual particle basis. Mathematical methods represent the other end of the spectrum, where you are maximizing the amount of information that you extract about individual particle shapes. Now, the way that mathematical analysis works is the first step is always to.... – you have to capture the particle, you have to capture its image, and you have to digitize its profile; that is where it starts. So, essentially, capture the 3 dimensional image of the object and use software to discretize or digitize its outer profile. So, what you now have are a collection of x y z points in space.

Now, what you do with that? The easiest thing is to essentially leave it as it is; leave the information as digitized information. Then, if you want to recreate the image of the particle, later on, simply feed in the same x y z points and the computer should to be able to recreate your object. Remember the Housner requirement I mentioned earlier that the objective of shape analysis is that the user should be able to reproduce the shape of the

object. So, with simple digitization of the profile, you can in principle, completely reproduce the external contours of the object. Of course, the most sophisticated your digitization algorithm, the more data points you take, the more precise is going to be your reproduction of the profile of the particle.

However, the other thing that you could do is instead of just tapping with the discretization, you can fit a polymer, and actually try to get a continuous representation of the surface instead of a discontinuous representation. So, polymer fitting and representing the surface of the particle in a polymeric form is certainly possible, but there are certain risks associated with that. In fact, there is a finding that the closer the fit of the polymer to the points that you have taken, the greater can be the deviation from the actual profile of the particle. In other words, if you are using certain mathematical algorithms, to get the best fit of a polymer to the digitized points that you have obtained, you can actually start deviating further and further from the actual profile by trying to do this curve fit or data fit. So, there is a risk associated with using the polymer fits – the more you try to improve the fit, the more error you may be introducing in terms of the deviation of the shape that is recreated from the shape of the object.

The third method is to use Fourier analysis. Now, Fourier spectra, essentially are unique to every particle shape just like Fourier spectra of organic materials are unique to every material. So, using a Fourier analysis, you should be able to capture the key features of the profile of the particle. At that stage, you can either use this once again to recreate the particle at a later time or you can store it in a database or in a library and compare it with shapes that have been generated – let us say by other researchers or by your own lab, where you have built up a database of known shapes and that associated Fourier shape spectra. Then, you can compare the Fourier spectra for your particle against the ones that are in the library. By doing this comparison, you can see where the best fit is and represent the shape of your particle by the corresponding best fit shape in your library. So, the Fourier analysis technique thus enable you to take your shape analysis one step further, and actually start building up reference spectra library that you can use in future and which other groups of researchers can use as well.

The mathematical techniques represent the most sophisticated method for shape analysis. It is very certainly very widely used now because of the computing power that is available, but you always have to be careful that in many cases, it may be an overkill. If

you are not a particle scientist working in the area of shape characterization, most likely it is too much. If you are a process engineer working in industry, the absolute recreation of the precise shape, 3 dimensional shape of a particle, may not really be required in most cases. So, do think and customize your method of shape analysis. You have three techniques to choose from so far – single particle analysis, bulk properties of powders, and then mathematical analysis of the profile. Again these represent the one end of the spectrum, the middle of the spectrum, and the other end of the spectrum. So, clearly, you have to make the choice has to where you want to be – do you want to be in the middle, or at one of the two extremes? That is where I guess your training as a particle scientist comes in, so that you get the minimum amount of information needed and simultaneously, the maximum amount of information needed as well; you do not want to exceed either limit.

The fourth class of shape analysis techniques that I mentioned was simple verbal descriptors. When we have defined these shape coefficients – whether it is flatness or whether it is roundness or sphericity or rugosity, whatever, instead of simply representing it by a number, if you can associate a word with it, again it becomes conceptually and intuitively easier for people to understand. For example, if you have a particle that has a very high elongation ratio – so, l by b is a very large value, instead of saying l by b is a large value, if you can say that the particle is needle-like or fiber-like, it conveys so much more as a visual characteristic. So, this fourth group of shape classification methods simply associates various verbal descriptions to the shape coefficients that we have derived in the other methods.

For example, you could call a particle rod-like if it essentially has cylindrical features; you could call it fiber-like; you can call it needle-like; you can call it spherical, certainly; you can call it elliptical; or, you can also call it something that is a little more descriptive – particularly if the particle shape itself has certain non-uniform features, you could say that it is a combination of rod-like and sphere-like features. So, the ability to express shape in terms of words certainly brings a lot more clarity to what you are trying to convey. In fact, there is something called a particle atlas that has been published; I think a copy is actually available in our library. If we look it up, it will actually tell you the various verbal descriptors that people have come up with over the years. There are

literally hundreds of them that people have essentially done shape analysis and represented the shape that we are seeing by words.

Now, the reason that verbal descriptors are important is because they do give again an ability of various labs to collaborate. However, it also increasingly highlights the importance of having convergence in descriptions between these labs. Two labs cannot look at the same particle: one says it is a needle-like particle and the other says it is a rod-like particle. So, this particle atlas actually gives fairly precise definitions of what each of these verbal descriptors mean, but typically, it is not done in a quantitative fashion. Instead, the particle atlas just like the name suggest, instead of providing maps of continents and nations, it actually provides images of particles. So, for a rod-like particle, it will give you about 10 images of what a rod-like particle is, so that you have an intuitive grasp of what people mean when they say that it is a rod-like particle. So, essentially, it gives you a spectrum of figures, diagrams, photos that you can look at. Then, compare your particle against each of these and say that this looks most like this; so, I am going to call it rod-like particle or a needle-like particle or whatever.

The verbal method is actually used as a supplement to the first three methods we discussed; whether you are doing single particle analysis or bulk powder analysis or mathematical analysis, ultimately, you have to communicate to other people what you think the shape of the particle is. They are not going to be happy just getting some numbers from you. That is why, even if you are trained in the other three methods, you still have to know how to express your results in a way that makes sense to other people. That is where the verbal descriptors come into play.

We will stop at this point. In the next class, we will start talking about some statistical methods for pattern recognition, which is very important in shape assessment. Any questions?

See you at the next lecture.