Particle Characterization
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Module No. # 02
Lecture No. # 03
Morphological Characterization:
Shape analysis methods

Good morning and welcome to the third lecture in the course on particle characterization. In the first two lectures, just to recall, what we have covered is the why and the what. Why do we need to study particle characterization and what characteristics of a particle are of particular relevance, both to industry practice as well as higher research and understanding of the behavior of particles?

In this lecture, we will begin our discussion of one of the primary characteristics of a particle. If we recall, in the last lecture, I said that we could actually classify the characteristics of particles into seven major categories: morphological, compositional, structural and so on. The first among morphological characteristics is shape.

Now, size is obviously also a very critical morphological characteristic of a particle, but the two are very closely tied to each other. We really cannot define size without defining shape and vice versa. When we represent a particle by a diameter, we are making the impressive assumption, that it is spherical in character and similarly when we describe a particle as being crystalline in its shape, we are also imparting dimensions to it. We are basically saying that dimension in one of the scales is much longer than the other.

So, shape and size are very intricately tied together. What is the difference between the two? Well, shape is the most visible characteristic of a particle. When you look at a particle, the first thing that you recognize is its shape, because it is a subjective or qualitative measure and a human brain is always better at doing subjective characterization rather than objective or quantitative.

In fact, if you look at particle characterization and you look at shape analysis in particular, the most sensitive instrument that we have for doing shape analysis is the human eye. We can look at a simple object and recognize its shape within micro seconds. If I give that same object to a machine and ask it to recognize the shape, it may take it a few seconds, whereas if I look at a complex object, I mean this, to us, is a very simple object - it is a bottle. So, instantaneously you are going to recognize what shape it is.

If you give the same shape to a computer and ask the computer to describe its shape, it will probably take minutes, because for a computer this is a fairly complex shape with a lot of non-standard features on it, which are going to confuse it and so even the best, most sophisticated shape analysis program may take several minutes to really understand what the shape is. So, clearly shape recognition is something that is intuitively easy for humans to do. But, it is very difficult to train any kind of objective, analytical equipment to do shape recognition, except in the simplest of cases.

Now, what is shape? I mean, shape is basically the external surface. It is the pattern, that the volume of that object, of that particle is fitted into. It is the profile of the particle. It is again, what is most visible to an observer. Shape is an intrinsic property, unlike size which is extrinsic. What do we mean by that? If you take two particles of two different sizes, you can say that, size one plus size two yields a size three, where size three is always greater than or equal to size one plus size two, because size is a quantitative measure, which is additive in nature. Can you say that about shape? Can you take two dissimilarly shaped objects and say that shape one plus shape two yields a shape three, where shape three is greater than shape two or shape one? No, it may be different. It may actually, even be the same. You can take two objects, that are essentially the same shape and when we join them together, you may retrieve the same shape or not.

So, shape is clearly very distinctively, different in that aspect compared to size. Shape, because it is the representation of the external contour of the particle, really reveals nothing about its interior structure. For example, a hollow sphere and a solid sphere are going to look like the same, strictly from a shape analysis view point. Extending that argument further, if you have a highly porous particle, verses one that is not porous, again you are not going to see the difference simply doing shape analysis. In fact, the porosity of the object will only affect your shape analysis, if the pores extend all the way to the surface. If all the pores in the particle are subsurface, you would not even see their effect. Even in the case, where the pores extend all the way to the surface, you are only going to see a limited impact of that. What you are only seeing is the effect of these pores on the external profile of the particle.

So, all these aspects of shape have to be kept in mind. So, how do you distinguish between a hollow sphere and a solid sphere? Not based on shape analysis, probably not based on size analysis, either. For that, you need structural analysis and that is the reason, why we have so many different categories of particle characterization and classification. A single characterization is never going to yield complete information about the particle. You have to do all the relevant types of characterization, in order to fully understand the functional impact of the particle in a process. So, going back to shape now, shape analysis is further complicated by the fact, that since it is a subjective measure, it is really the analyst choice, as to when the investigation stops.

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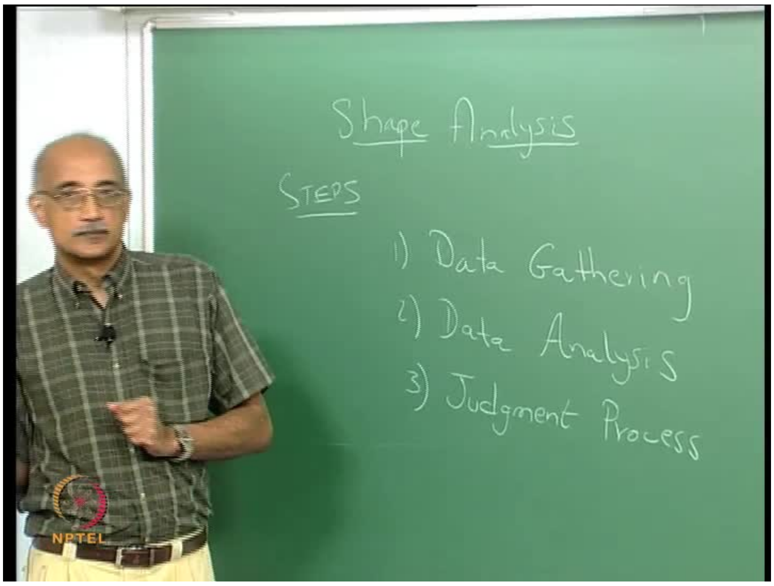

Essentially there are three steps in shape analysis - the first is data gathering, the second is data analysis, the third is the judgment process. So, what do we mean by this? Data gathering, as the name suggests, is the first step, where you take the particle whose shape you are trying to assess and collect data on it, in terms of its surface profile. Data analysis refers to how you use the data. Collecting the data is the sometimes, easy part. Analyzing the data and extracting useful information out of it can be a more difficult aspect of work. Finally the judgment process and here is where you have to make a call, on how much data is sufficient and what level or depth of analysis is sufficient for the purpose at hand.

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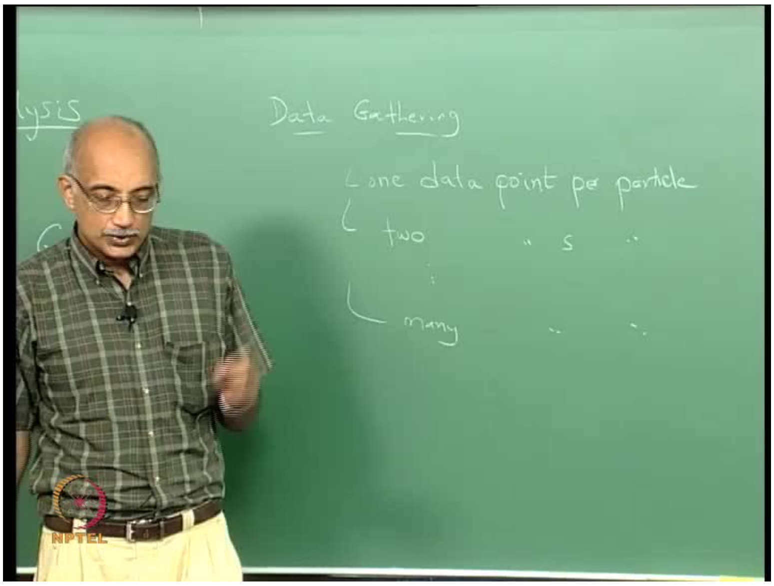

Now, if you look at the first step, data gathering, there are various modes of gathering data for shape analysis. So, if I just look at data gathering as the first step, it can be as simple as one data point per particle. In other words, you could say that I am just going to look at the largest dimension of the particle and I am going to represent the shape of the particle by its largest dimension. Or more commonly, it could be two data points per particle. For example, you could choose to find the largest dimension and then, another dimension that is perpendicular to it. Represent the largest dimension or the longest dimension as l and represent the transverse dimension as b and take the ratio of l over b as an elongation ratio, for example.

So, this approach of representing a particle as an ellipsoid, by taking at least two perpendicular dimensions is very common. And, you can extend this, all the way to many data points per particle. In other words, you could essentially choose to digitize the profile of the particle and you can take as many data points as you like, depending on how fine a detail that you want. So, for the same particle where you can take one or two, you could take a million data points from the same particle. Of course, the more data points that you take, the more data there is for you to analyze. But again, you have to exercise caution in terms of, not wasting your effort. You always have to think about, 'Is this enough?' You are not doing this just for the fun of it, you are doing it for a very specific purpose and as long as you have that purpose in mind, you can decide how much data is adequate.

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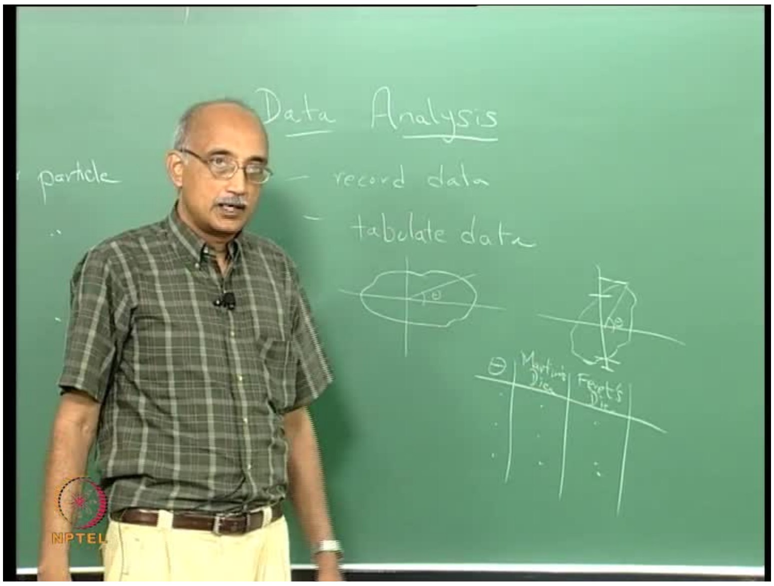

Now, the second part of it is the data analysis part. Again, there are many modes of data analysis, the first most common is just recording the data. If you only take one dimension on the particle, all you have to do is have a log book or an excel work sheet and simply record the data or you can tabulate the data.

Now, when we talk about tabulating data on the shape of a particle, what do we mean by that? One example could be, where you have a particle like this and you could take a coordinate axis. This could be your microscope IPs, for example and oriented at various angles, represented by some angle here, theta and for each angle theta, that you orient the particle at, you can take for example, let us say that, your particle is being held at this angle and let us say that this represents your theta value, you could actually take two different diameters. For example, these intersections, you could consider to be a representation of the particle and these intersections, we could also take as representing the size of the particle and you can actually tabulate these values. So, you could essentially have a tabular representation here, where you take theta and the smaller diameter. By the way, in this representation this is called a Martin's diameter and this larger intersection is called the Feret's diameter.

So, for various angles of values of theta, you can actually tabulate the values of Martin's diameter and Feret's diameter and you can use those as the representations of particle shape or actually you can take a ratio of the two and represent that as a shape index. Because, as you can imagine, if this is a perfectly spherical particle, this ratio will always be equal to one, whereas if it is a highly elongated particle, this ratio would tend to zero. So, just simply by taking the ratio of the Martin's diameter to the Feret's diameter, we can represent that as a shape coefficient for the particle. So, this is a second approach, which is definitely more rigorous in terms of capturing the data and analyzing the data.

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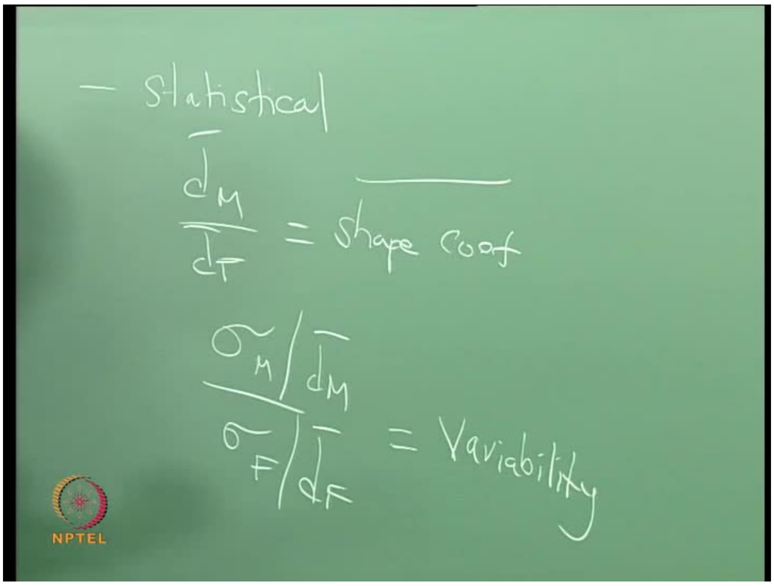

Now, extending this further, the more sophisticated data analysis could be statistical. For example, we can simply take this data and average all the Martin's diameters as the function of theta and average all the Feret's diameters and take the ratio of d Martin over d Feret ratio and as being an average shape coefficient, you know, where the bars essentially represents averages. So, this is a very simple statistical representation, where you are only taking the means of your measurements as a function of theta. However, if you want to do more sophisticated analysis, from a statistical view point, you can also look at the standard deviations, right?

So, the sigma values in the Martin's diameter and the sigma values in the Feret's diameter, which are the standard deviations, can also be taken into account and typically that is done, by dividing these by the representative mean diameters to give a variability coefficient. So, this gives you a representation of variability as measured in particle shape as a function of theta. So, in other words, the higher this parameter - variability parameter the indication would be that, it is a highly irregularly shaped object the depending on what angle of theta that you look at you can come to completely different conclusions about the shape of the object. So, this is a statistical way of analyzing particle size.

Now, extending this further, obviously what we have been talking about is shape analysis for a single particle. But, if you have a population containing a large number of particles, you can imagine doing this for a hundred particles chosen at random or a thousand particles or any number of particles that you want. Then, the statistical aspect of data analysis becomes even more critical, because you know shape is something that is absolutely unique. It is like a finger print. Just like no two human beings can have the same finger print, similarly no two particles can have the same shape. Every shape is completely different. Even what we call perfectly spherical, you know it all depends on your definition. We have a tendency to represent three dimensional shape in two dimensions, that is hardly ever true.

Even when you say shape, I mean a sphere, for example, it is a three-dimensional representation of an object. But even that is neglecting the small variation that you can have, simply because of the - for example – roughness, that is present on the surface. A surface is never two dimensional. As I think, I mentioned this briefly in one of the previous lectures, a surface is always three dimensional and therefore, essentially any object that you represent even in a three dimensional way from a shape view point, you are actually neglecting a fourth dimension, which is there because of the irregularity of the surface profile in a three dimensional context. So, statistical analysis of shape becomes very critical, particularly when you are characterizing shape distribution across an entire population rather than shape of a single particle.

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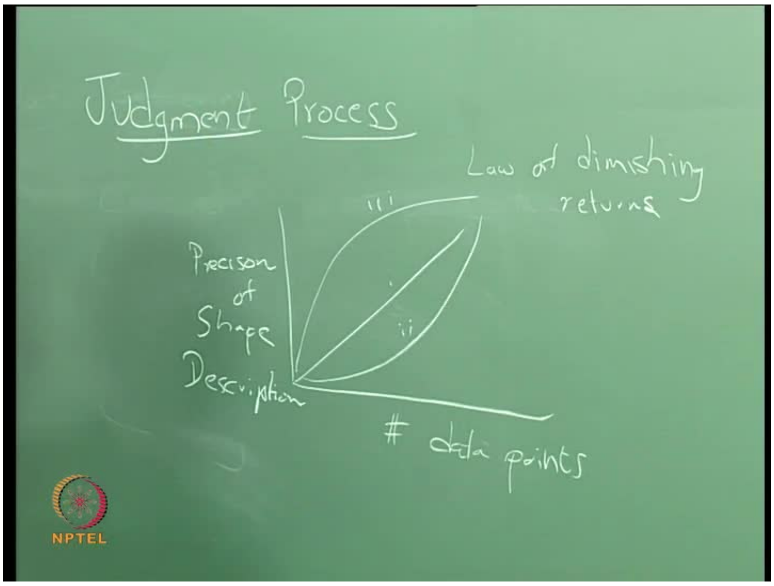

Let us talk about the third aspect now and that is the judgment process. Now, at some point, you again have to stop your data gathering and your data analysis and you are going to have to sit back and say – 'Now that I have done this, what is the shape of the particle that I am looking at?' I mean, that is a judgment process and that is where the human element gets involved, no matter how many machines you have, doing your data gathering and your data analysis, ultimately it is your call to say what shape does this profile represent.

Now, when you do that, obviously the more the number of data points, the more precise and accurate your description of the shape is going to be. But, it also involves more effort. Now, how many of you have heard of the law of diminishing returns? It basically says that, in most situations, the relative impact that additional effort or investment makes, always gets reduced as you expend more and more effort or energy to make it happen.

So, for example, if I were to look at number of data points versus, let us say, the precision of shape description, what kind of shape can you expect? Will it be linear? Would you expect that the more data points you take, the more information you keep getting or would you expect something like this, where initially, for a large number of data points, you do not get much information, but then once you reach a threshold, all of a sudden, something dawns on you and you start recognizing what this particle is or do you expect a shape like this, where initially for the amount of effort you put in, you are getting a lot of insight and knowledge and then slowly it levels off. So, suppose we call this 1, 2 and 3, of the three, which one do you think is most likely? 3, because intuitively it is obvious that, when you start with zero knowledge about the object, even looking at it will give you insight into what its shape is and even collecting one data point or two data points, actually will start propelling you towards a logical conclusion about the shape of the particle. But, then once you go beyond a certain amount, the more data points you collect, it is more for validating your judgment rather than forming your judgment.

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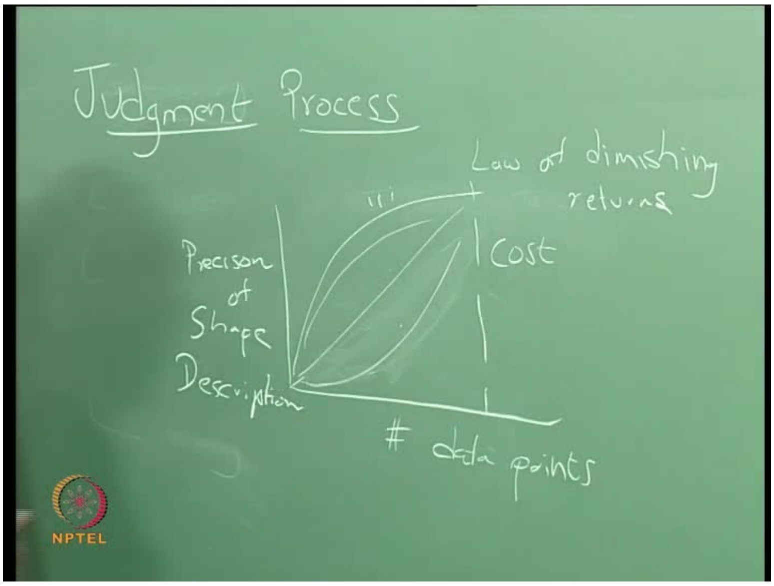

So, this is what we call the law of diminishing returns. So, what that forces you to do is to say, “At what point do I stop?” Now, this is what we have said is the expected shape of the number of data points versus precision of shape description. About cost of analysis, suppose I plot on this axis, cost, using a different color. Cost of analysis, what is that going to look like? Is it going to be essentially linear? Again, let us say that you have one choice like this, one choice that looks like this and another, that looks like that. Which do you think is more likely in this case; actually, this one.

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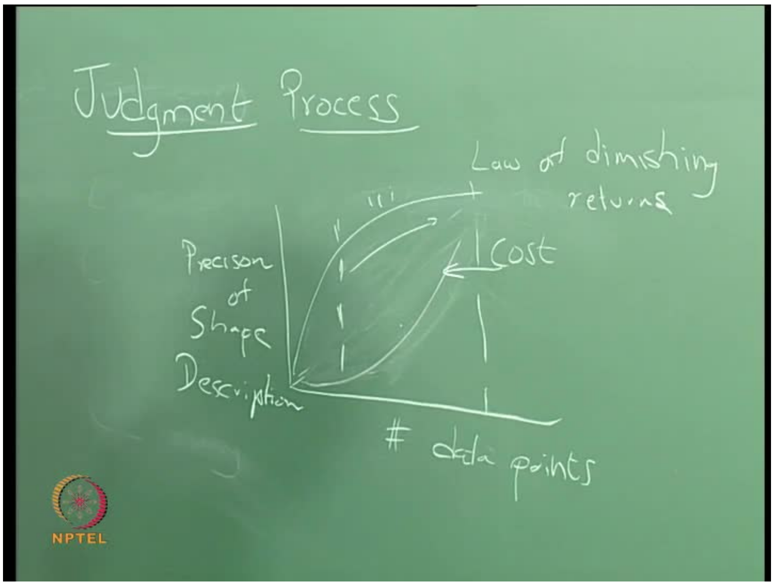

Cost of analysis quickly escalates as you try to obtain more data points. I mean, the simplest illustration of that is, you know if you are trying to gather data about the shape of an object, the least analysis you could do is just looking at it under some light without magnification, versus if you want to collect more data points, let us say you add some bright light and you add some magnification and say, you start using an optical microscope, so that adds cost. But then, after a while, if you still want to collect more data points, then optical microscope is not going to work for you, right? You are going to have to go to electron microscopy and that price rise is not linear; it is exponential. As soon as you decide to go to an SEM instead of optical microscope, there is a huge cost associated with it.

So, this is the point that cost escalates rapidly as you try to increase the number of data points that you have, whereas the precision actually flattens off. So, based on this, how do you make a judgment call as to when you stop? Now, there can be situations where cost is no object and you want the description to be as precise as possible.

One of the examples I was mentioning in earlier lectures is, now when you are looking at a brand new particle that you have just brought back from outer space. You want to know everything about it and the government has money to throw at the problem. You do not care, I mean you keep doing the analysis till you reach an absolutely asymptotic point and even beyond that. On the other hand, if you are working in the industry, I was mentioning that hard drives are susceptible to particle related failures. So, a hard drive fails in the field and the field engineer brings it back for failure analysis and the indications are, there is a particle that is stuck on the desk at a particular location.

So, you do your FA, you bring it into the lab and you carefully remove that particle from the disk using a tape or whatever, put it on a slide, put it under a microscope and you want to look at it to assess its shape, because the shape of the particle can tell you a lot about where it came from. The source can actually be indicated simply by, for example, if it looks like a shaving it probably came from a machining process. If it looks like a fiber, then it probably came from somebody's clothing or packaging or something like that. So, shape is certainly an important parameter or factor.

But how much effort do you put into it? I mean, as soon as you know that it is fibrous in nature, you will probably stop. You do not need a very precise estimation of what is its length to diameter ratio. All you want to know is, 'Is it roughly spherical?' 'Is it roughly crystalline?' 'Is it roughly fibrous?' .So you probably will stop the effort right at this point, as soon as there has been a sufficient ramp in your understanding of the shape of the particle. There is no point in extending it beyond, because you see, for a manufacturing industry, failure analysis is not a value add process. If you do your job right, in making the product in the first place, you would not even have failures. So, you do not even have to do failure analysis. So failure analysis is actually an acceptance, that something has gone wrong with your process and most industries do not want to spend; they will not even put their best people in it. It is something that they would like to see completely eliminated. That will be the ideal situation.

So, they are likely to truncate this fairly early in this process. So, it is important to understand this effect and in your judgment process, be very, very clear as to where you want to stop. So, this was something that, when shape recognition and shape analysis was emerging as a science in the early to mid [two thousands, sorry] twentieth century, the whole scientific field was struggling with it and finally there is a person by the name of Hausner who came out and postulated something, which now has become the guiding principle behind shape analysis.

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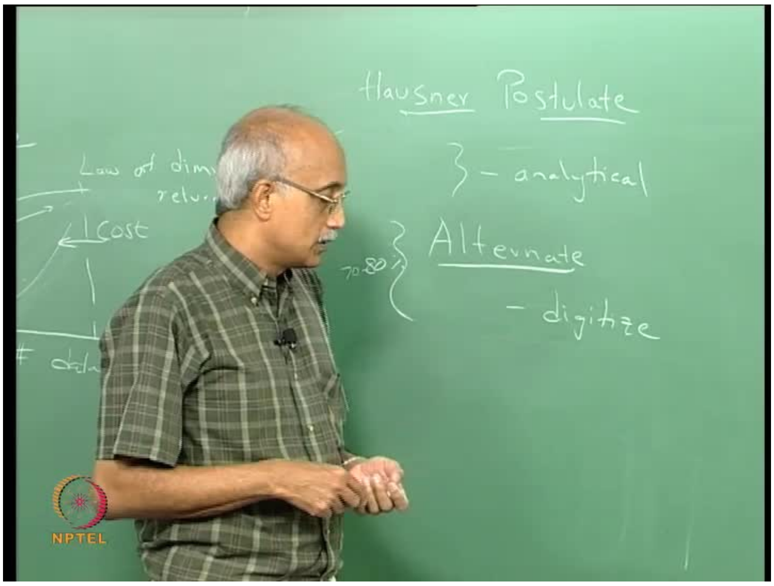

And it is called the Hausner Postulate and this postulate is actually, intuitively, very simple. He basically said that, once the data gathering and data analysis are complete, the observer or the investigator should be able to recreate the shape of the particle under analysis, to the extent that it satisfies the investigators requirements. So, he kind of, said that shape analysis is not an absolute science. You do not have to keep doing shape analysis till you are hundred percent sure about what the shape is. Instead, it should be very function dependent and so, it was simply a restatement of something that you just talked about in terms of the law of diminishing returns. Do not keep doing it till you have eliminated all uncertainty. The amount of data gathering and the amount of data analysis only have to be sufficient for the investigator to recreate the original shape of the particle to the extent that is relevant for the process that is being run.

Now, that is an analytical approach to shape analysis. So, this postulate essentially created an analytical or subjective frame work for shape analysis. The other extreme view point, an alternative view point to shape analysis simply says ‘Digitize the profile’. So, you reduce the problem to one of pure mathematics. You take the shape that you are trying to understand, describe it as discrete points and then take this matrix map and compare it with similar maps, that you have in your library or your database and whichever of these digitized profiles - standardized profiles is closest to the one for your object or your particle can be taken to be the representation of that particle.

So, in other words it is a purely mathematical procedure, which kind of takes a particle scientist out of the equation and brings it in to the realm of mathematicians. So, you take the particle that you have, digitize it, capture the data points in some kind of a discrete fashion and then compare it to similar maps that you have generated from millions of particles in the past and find the closest match. So, this digitization procedure versus the analytical procedure are kind of, two extremes of particle shape analysis; in the one case in digitization, you are saying that you want your mathematical description of the particle to be as precise as possible, so that you can, kind of take the human element out of it ,whereas the Hausner approach is to say that particle shape analysis is still predominantly a human endeavor and the tools that you use, you want to really minimize as far as possible and only do to the extent that is absolutely necessary. So, we will look at particle shape analysis from both view points and see, which one seems to make more sense.

Now, Hausner postulate was obviously fifty to sixty years ago, when our analytical capabilities were very limited. We did not have the super computers that we have today and so, doing this was really not a realistic option. Even what we call digitization back in those days, was done manually. You will essentially have a microscope operator who will sit and actually look at the hundred or thousand points on the profile of the particle, map it in the x y z scale and essentially, do the digitization in that kind of brute force manner.

So, obviously there was a lot of resistance to going this way. But, if you look at what is happening today, this is probably the easiest approach. You can just throw the particle on a stage and have automated equipment at whatever magnification you choose, do this digitization and recreate the image of the particle as precisely as you want.

So, now I would say that, if you look at particle shape analysis, probably seventy to eighty percent of it is being done in this fashion and only a few, I guess old fashioned people there are still doing what I would call the analytical methods of shape analysis. But it is still important to understand them, because it really gives you a sense of the history of shape analysis; where did we start and where are we today.

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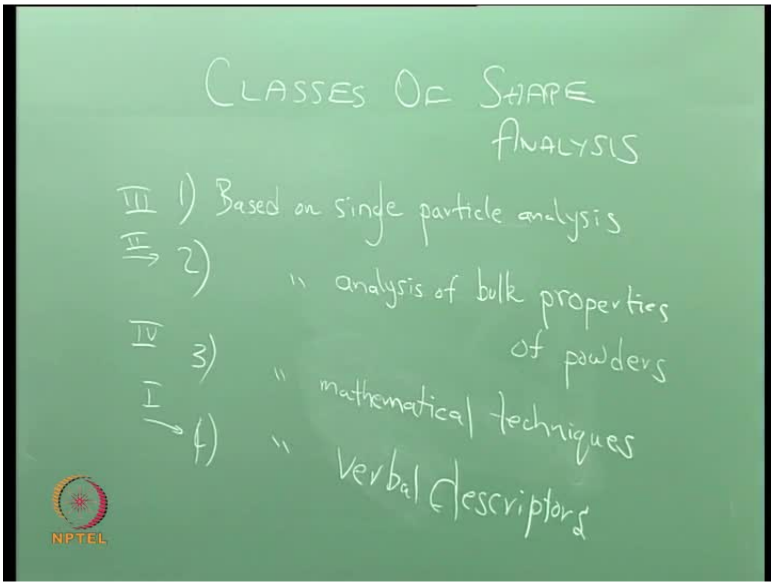

So, what we will do next is talk about the various classes of shape analysis methods. The first one is based on single particle analysis. The second classification is based on analysis of bulk properties of powders. The third method is based on mathematical techniques and the fourth is based on verbal descriptors. So, this is a very broad classification of how particle shape analysis is done and I think the terms are fairly self-evident.

You can do shape analysis by focusing on individual particles in a population. So, do the analysis at a single particle level or you can do it essentially by looking at how entire populations of particles behave - bulk analysis. The third is, the mathematical techniques that we just briefly discussed and finally, somewhat at the other extreme, verbal descriptors. So, essentially a syntactic method of describing particle shape by using rules of grammar essentially, just using english words and terminology to represent various classes of particle shapes. Now, when you look at these broad classifications of particle shape analysis methods, obviously the easiest is this, the next would be this. It is always more convenient to estimate shape by looking at how an entire powder behaves. I mean, a simple way to do this may be to take the powder, mix it in some fluid, let it flow and look at the flowability of the powder, which can actually tell you a lot about the shape distribution in the powder. Based on single particle analysis, in terms of complexity, it is the third and finally, mathematical techniques require the most complex analysis by the investigator. We can further subcategorize these techniques and we will see how we do that.

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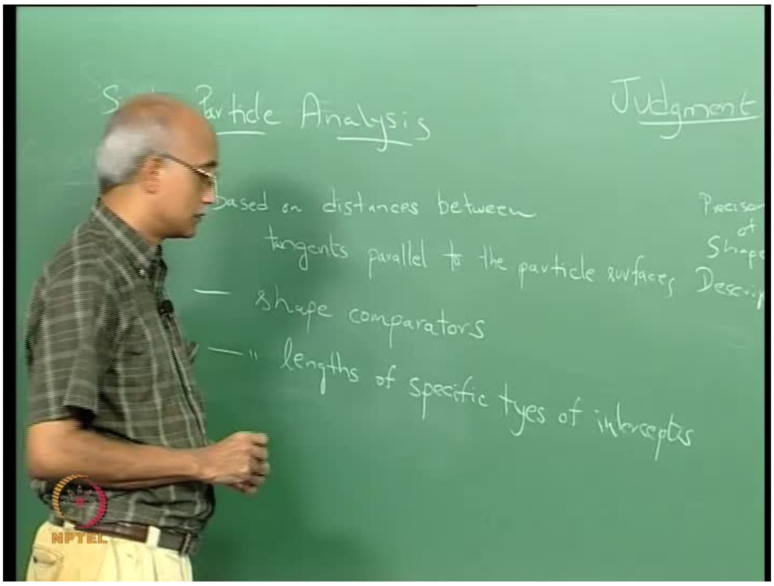

So, when you take that first technique, single particle analysis for shape determination, this can again be subcategorized into three categories; the first is based on distances between tangents, parallel to the particle surfaces, the second category is based on shape comparators and the third is based on lengths of specific types of intercepts.

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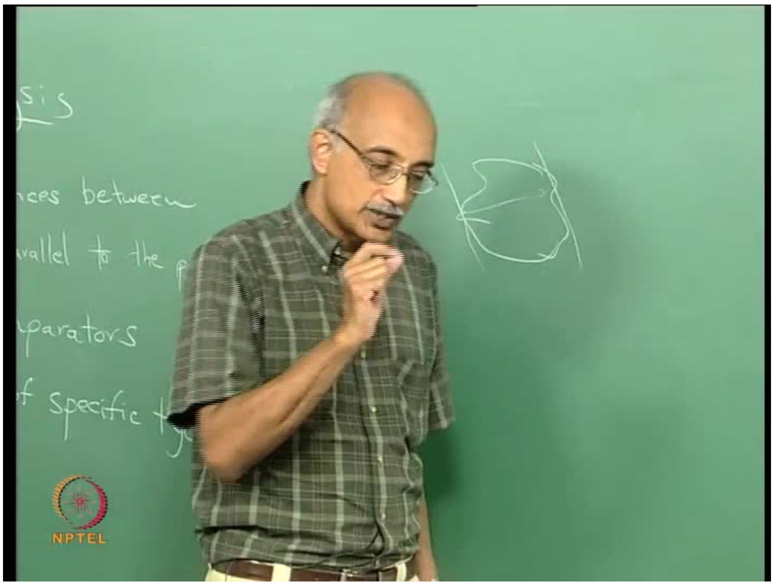

So, we will look at these in more detail in the next class, but I will just outline what we mean by these. When you are looking at a particle and again, trying to assess its shape, intuitively you can see that one way to do that would be to draw tangents and look at the difference between parallel or the distance between parallel tangents. You can use that essentially, as a representation of shape and if you do it in three dimensions, and let us say, you take a longest dimension in either, in each of the planes; that can give you an idea about the shape of the particle. What do we mean by types of intercepts? Well, here you have to visualize a three dimensional object, you know, let us say a balloon; well, probably a balloon is not a good example, but let us say a ball.

How do you assess its shape? One way to think about it is, supposing you take a needle, very sharp needle and throw it at the ball. How far will it penetrate before it stops? For a given length of needle, you just keep throwing it at random, from different angles. How many of the needles will become embedded within the ball and stop, and how many will actually penetrate on both sides?

What does that indicate, from a shape view point; the bulkiness of the object. The bulkier the object, the more likely that the needle will not cut both profiles and that its end will stop somewhere in the mid-section of the object. So, that is one illustration of an intercept, because what we are talking about now, is essentially an intercept that a thrown object makes, to this object or this particle that you are trying to analyze. Finally, what do we mean by shape comparators; again shape is not an absolute measure. You can take two particles and say that one is bulkier than the other. In order to do that, you have to develop some kind of a quantitative index for the bulkiness of the particle or you can say that one particle is flatter than the other. Again, you have to have a quantitative metric for flatness of the particle.

So, in order to do shape comparisons, you need to define these shape comparators and here again, several investigators have done a lot of work to come up with very quantitative indices for many of these normal shape comparisons, that we do. Sphericity is probably the most common example. Now, when we talk about sphericity, it is a good comparative measure between two particles; is one particle more spherical than the other? But, you can only do that if you can define sphericity in a very quantitative fashion.

The second class analysis of bulk properties of powders; well again, what we mean is, what we try to do in this method is, you take a powder that contains many particles of probably varying shapes, but you do not really care about the actual shapes of individual particles. The only thing you are interested in is, how does the shape distribution affect my ability to use this powder in my process?

So, a very common method for looking at this, I mentioned flow ability earlier. Another may be that you look at the density of the powder and you look at the bulk density of the powder as it is, and then you essentially take it in a container and tap it and as you tap, the powder is going to settle. The density is going to change, so the comparison of the untapped and tapped density of a powder will actually give you a very good measure of the shape distribution that is present in this powder, because powders containing particles of identical shape will settle in a very different manner, compared to powders that contain particles of highly varying shapes.

Another example of a bulk property method may be looking at porosity in the powder as a function of compaction. As you compact the powder, the porosity will decrease; but how it decreases with compaction will again depend on the shape distribution. So, these are methods that you can use to assess shape distribution in a powder, by looking at the functional behavior of the powder, rather than trying to image-analyze individual particles.

The third classification which is mathematical techniques; I mentioned one of these techniques earlier, which is digitization. Simply take the profile and reduce it to a number of points, discrete points which you then analyze later, by comparing it to a standard library and so on. But, you can also imagine fitting a polynomial. You can take this digitized profile and get the best fit polynomial and you can use that as the representation of the surface. So, polymer fit is another method that comes under this classification of mathematical techniques. However, we will see later on, that it carries several risks when you try to do shape analysis by taking digitized points and doing a best fit polynomial, the error can actually increase as the fit increases.

Another technique that comes under the mathematical techniques category is Fourier transform analysis; you try to capture the most frequently recurring characteristics of the particle and again you compare it to a spectrum, a reference spectrum of shapes that are available to you, and it is just like you do fourier transform analysis for identifying the composition of organic substances. For example, every shape will have a distinct spectrum and so, if you have a sufficiently good library of reference spectra to compare to, you can simply compare the Fourier spectra for the shape of your particle versus the librarian and draw a conclusion. So, there are several subcategories under the mathematical techniques class of shape analysis methods.

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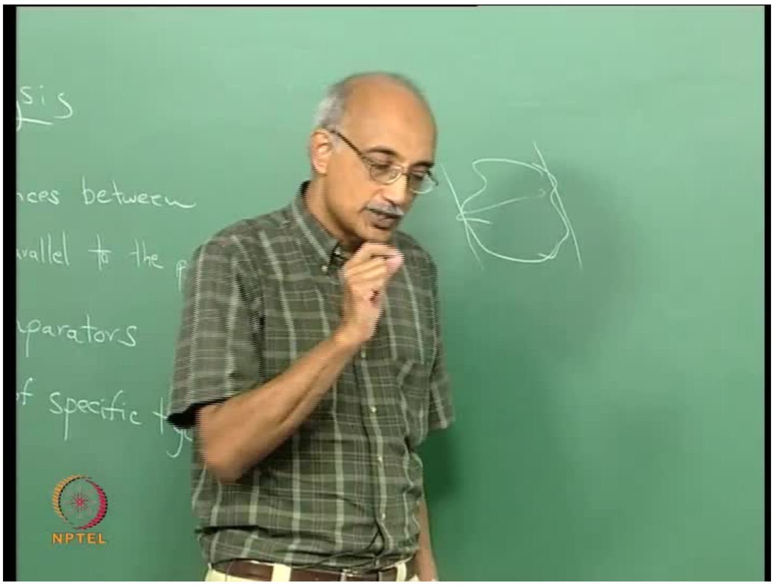

And finally, verbal descriptors; here again, that the whole point in shape analysis is to be able to communicate your findings and your conclusions to another laboratory and have them agree with your description, and that is actually the most difficult part to achieve in verbal descriptors, because, if you have a set of scientists in your lab and they look at a particle and they say it is, kind of, let us say, they are looking at it and they will say that it looks like a cylindrical rod and then you pass the same object to another lab where there are particle scientists, then they may look at it and say I do not know it looks to me more like a tube or something like that.

So, when you talk about verbal descriptors, there is always this element of subjectivity and so, this requires actually a very dedicated exercise across labs and essentially the way this was done was, collecting thousands of particles, selecting about twenty standard laboratories across the world, to do the verbal characterization of the shape and then doing a massive comparison exercise across all these labs to see if it could all converge to a common set of verbal descriptors.

So, even though intuitively, this is the easiest way to do shape analysis, in practice this took longer than any of these, because of the inherent disagreements in how different people viewed the same object. Shape, just like beauty is in the eyes of the beholder. Something that you call something, another person might call something else. So, that exercise actually turned out to be fairly convoluted. All right, so what we will do in the next class is, we will talk about these classes of shape analysis in more detail and after that, we will start talking about some systematic methods for doing shape analysis, where the precision of your shape assessment can be increased substantially, without necessarily increasing your time or cost of analysis. Any questions on what we have talked about today?

I will see you at the next lecture.