

Power Metallurgy
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Lecture - 20
Powder Characterization

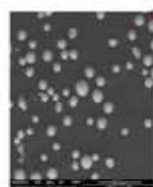
Hello and welcome back. So, in this course on Powder Metallurgy so far we have seen different techniques for fabrication of a metal powders and we have discussed almost every details of these techniques one by one in past several classes and we have also seen in for some of those methods like the atomization process, how the microstructure of the powder can be controlled.

Now, one thing that you would have realized by now is that a particular type of fabrication route will give rise to a particular type of particles especially in terms of their shape and size. For example, if you see in this particular image over here.

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Irregular
Mechanical



Spherical
Atomization



The particle shape is irregular, and if you remember these particles are generated by the mechanical fabrication process and on the other hand, if you see on this image the particles are spherical and this is something that we got in the atomization process right.

Like that every process can generate different types of particle shape, size distribution and so on and therefore, it is important for us to know as to how you can characterize the

powders and get to know about this safe size and other powder properties because such properties are going to affect the entire powder metallurgy process as such.

Because what happens as you would have seen before also in a powder metallurgy process, you start with the powder and then you follow certain processing steps and in each of those steps you know a particular process is involved which uses some kind of toolings and so on to first compact the powder to give it a particular shape and then finally consolidate and densify it to get the end product, ok.

So, this compaction and the consolidation part that is going to be affected by these powder characteristics what we talked about such as in this powder shape powder size and so on, right. So, therefore it is necessary for us to understand these parameters and also, we need to know how this can be analyzed and evaluated right.

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The diagram is a tetrahedron with four vertices. The top vertex is labeled 'Properties'. The bottom-left vertex is labeled 'Powder'. The bottom-right vertex is labeled 'Processing'. The right vertex is labeled 'Chemistry'. In the center of the tetrahedron is a red circle labeled 'Characterization'. A red checkmark is next to the 'Properties' label. To the right of the diagram is the NPTEL logo.

- Powder type and fabrication influence the ease of compaction and sintering.
- The consolidation method and sequence used affect the final compact properties.
- The powder processing and chemistry will decide the desired properties .



So, having said that I want you to now go back to this particular slide over here that we discussed in the beginning of this course if you remember and here we talked about the importance of this central part of this powder metallurgy tetrahedron that you see over here and at the center of this, you have this characterization right.

And as I said unless you characterize the powder, you will not know what kind of powder properties you have obtained in a particular powder which has been processed by a particular fabrication route, right. So, that is what we are going to talk about in today's

class and in next few classes as to how you characterize the powder in order to know the different powder properties.

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Powder Characterization



So, that is going to be the topic for today's class - Powder Characterization.

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Powder Characterization



- A powder is made of particles which is the smallest unit that cannot be divided further.
 - P/M generally deals with particles which are bigger than smoke (0.01 to 1 μm) but smaller than sand (0.1 to 3 mm).
 - Powder characterization involves the measurement of particle as well as the bulk powder properties.
- Particle size and distribution ✓
 - Particle shape and its variation with size ✓
 - Surface area ✓
 - Flow and packing ✓
 - Interparticle friction ✓
 - The internal structure of particles ✓ *Microstructure*
 - Composition, homogeneity and contamination ✓



Now, when you talk about powder characterization as such you are basically talking about the powder particles and their different characteristics. So, first let us define what a particle is and then we will see what those particle properties or the attributes that we are

looking for in order to know the characteristics of the powder. So, a particle can be defined as the smallest unit that cannot be divided further.

So, that is the basic definition of a particle and as far as the powder metallurgy process is concerned, it generally deals with particles which are bigger than smoke. That means, bigger than this size range 0.01 to 1 micron, but smaller than sand which is having a size range of 0.1 to 3 millimeter, right. So, that is the kind of a size that we are talking about in the order metallurgy process. And the characterization process for the powder will involve the measurement of a particles as well as some of the bulk powder properties as well.

Particle characteristics are such as particle size and size distribution, particle shape and its variation with size and the surface area of the particles. So, these are the attributes of for the particles as such and then some powder properties like the flow and packing characteristics of the powder as a whole has to be evaluated when you talk about characterizing the powder. Then you have properties like inter particle friction which is going to affect the process particularly the compaction process that is done for compacting the powder and get a shape out of it.

And of course, you also need to know the internal structure of the particles which is also known as the microstructure and if you remember this is something that we have already discussed in first several classes. And composition homogeneity and if there is any contamination, that also has to be evaluated because each of these characteristics will have their own influence on the powder metallurgy process, ok.

So, we are going to talk about each of these as to how do you evaluate these different parameters, how do you measure them and so on.

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Powder sampling

- Obtaining a representative sample is critical to ensure measurement of real particle characteristics.
- Particles in produced powders tend to separate during shipment and to agglomerate during storage.
- To obtain representative samples and minimize sampling errors
 - Samples must not be taken from the top or bottom of the powder container.
 - Samples should be taken from several locations and mixed together
 - Deagglomerate the particles by
 - Drying the powder to remove adsorbed moisture
 - Adding dispersants (organics with charged anionic or cationic terminal groups)
 - Mechanical stirring or ultrasonic agitation
 - Samples should be taken from a moving stream of particles
 - Use of rotary riffles divides the flowing powder into rotating containers and is a good and reliable practice for sampling



Now, when you are talking about characterizing the powder, you first need to collect the sample in order to you know use a particular instrument to measure a particular property, or to evaluate the powder you need to first collect a proper sample from a powder lot right.

Because as you have seen these powders are produced in large quantities and when you characterize the powder, you know you cannot take the entire sample or the entire lot and then you know you characterize the entire lot or all the particles in a lot that is fabricated by a particular process.

So, therefore, you need to first collect a small sample from a large amount of powder and you should collect the sample in such a manner that it is actually a true representative of the entire lot of the powder which is there. And that is very critical for the measurement of particle characteristics obtaining a representative sample. You know which will represent the entire lot as I said. And when you try and collect the sample, these are the things that you need to remember.

First of all, this powder is generated somewhere and then it could have been shipped or transported to some other place where it is actually characterized, right. So, it is possible that you know during this transportation and shipment, the powders will tend to separate and while they are being stored, they may also agglomerate right. So, this is something

that you need to consider when you try and obtain this representative sample in order to minimize these sampling errors.

So, in a powder sample it should be a random mix which is free of any agglomeration or any other type of suppressant depending on size or shape and so on, right. So, these are the good practices that one should follow in order to collect a good representative sample. First of all the samples must not be taken from the top or bottom of the powder container, then the samples should be taken from several locations and mixed together. This will ensure that it is a random mix and is a good representative of the entire lot of the powder.

As I said there are chances of agglomeration during shipment and storage, so it is always good to deagglomerate the particles by following processes such as drying the powder to remove the adsorbed moisture.

Because one of the major causes for the particle agglomeration is the moisture which is being absorbed because this moisture is going to act like a glue between the particles and make them stick together and as a result, you know they will tend to agglomerate right. So, when this moisture is removed by drying, the particles will also tend to separate from each other and then the powder sample can be deagglomerated.

And in order to avoid this agglomeration or sticking of these powder particles with each other, organics with charged anionic or cationic terminal groups can be used as dispersant right.

So, dispersant is a organic material you know the which will modify the surface of the particles and prevent their you know they are sticking together or they are agglomeration. Then mechanical stirring and ultrasonic agitation can also be utilized in order to you know randomly mix the powder and remove any agglomeration or separation that might have happened during transportation and storage.


And if this can be done you know the samples if it can be taken from a moving stream of particles rather than you know from a stationary, heap of a powder that is going to provide a powder sample which will be a very good representative of the entire lot of the powder because here you have a streaming feature where different types of particles are mixed. So, if you collect a sample from a particular location of that stream, it is going to

give you a good sample which will you know contain all kinds of powder that may be present in the in a particular lot, ok.

And certain devices like this rotary riffers can be used and this riffers divides the flowing powder into rotating containers and therefore, is a good and reliable practice for sampling. So, as the powder stream comes down, you know it can be sent through this kind of rotary riffers which will send the powder in different containers which are you know rotating around it and as a result, it will keep collecting powder sample as the powder comes down onto this device.

So, these are you know some of the measures that can be taken to ensure that you have a good representative sample which can be tested for evaluating the powder properties.

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Types of particles

Primary particles

- Smallest closed-pore particles
- Particle density is very close to true density of the material

Agglomerates

- Clusters of primary particles bounded by either surface forces or liquid bridges such as water.
- They can easily be broken up by shear stresses far below the material strength.
- The driving force for particle agglomeration is specific surface energy, $E = \gamma' A / V$

For spherical particles of diameter D


$$E = 6\gamma' D$$
$$A = 4\pi r^2 \quad V = \frac{4}{3}\pi r^3$$
$$\frac{A}{V} = \frac{4\pi r^2}{\frac{4}{3}\pi r^3} = \frac{3}{r} = \frac{6}{D}$$

Flocs

- Agglomerates that form in liquid suspensions
- The particles are held together by electrostatic forces.

Aggregates

- A mixture of two or more particulate material



Now, that we have talked about this particles and the sampling process, let us go ahead and see what are the kinds of or the types of particles that we are talking about here. These are the different kinds of particles that one might encounter in a powder.

Primary particles which are nothing but the smallest closed pore particles wherein the particle density is very close to the true density of the material. Then you have something called agglomerates and these are nothing but the clusters of primary particles bounded either by the surface forces or liquid bridges such as water.

So, as I said before in the beginning there are chances that moisture can be absorbed by the particles and in that case you know this moisture or water will act as a liquid bridge between the particles and they will stick to each other forming the clusters right, but these are all you know loosely bonded particles and therefore, these agglomerates can be easily broken by shear stresses which are far below the material strength, right.

So, if you apply a small amount of force, a small amount of stress, these agglomerates can be easily broken down and the driving force for the particles to stick to each other or to form this agglomerate is the specific surface energy E which is given by this

$$E = \gamma * A / V$$

where in A is the surface area and V is the volume and γ is the surface energy. So, if you take a spherical particle of diameter D , then this specific surface energy is going to be

$$E = 6 \gamma / D$$

because for a spherical particle $A = 4 \pi r^2$, where r is the radius and $V = 4/3 \pi r^3$ and therefore $A / V = 3/r = 6/D$.

So, that is how you get E equals to 6γ by D for a spherical particle. Then there are two more types that you can get in a powder. One is known as Flux which are basically agglomerates that form in liquid suspensions and here the particles are held together by the electrostatic forces, right and the other type is aggregate which is nothing but a mixture of two or more particulate material . So, these are the different types of particles that you can expect to encounter while you are characterizing the powder.

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Particle size and shape

- 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.

Spherical	Cubic	Flake
Rounded	Tear drop	Cylindrical
Sponge or Porous	Lignin-like	Fibrous
Angular	Dendritic	Polygonal
Acicular	Hierarchical	Irregular

So, when you are talking about evaluating the particle characteristics, size and shape are the two main characteristics that we are looking for here because that is going to have a great bearing on the compaction process which we are going to see later on. So, what we have seen is that most of the fabrication processes generate complex shapes and the particle size and shape. We will affect the flow packing and compressibility of the powder.

So, this is something as I said is very important as far as the compression process is concerned wherein the powder has to be filled in a die, it has to be packed and then it has to be compressed, right. So, that is why flow packing and the compressibility of the powder are very important characteristics. And as far as the size is concerned, since there are different kinds of shapes that you can generate in powders, you need to choose a characteristic dimension to define the size or shape of the particle right.

For example, if it is a spherical particle, it is far easier to define that particular dimension because in this case it will be just the diameter of the sphere, but as the shape becomes more and more complex, you need more than one such parameter to define the shape and size of the particle. For example, if it is a flake like what you have over here, then you need at least two parameters here. One is the diameter D and other is the thickness of the flake. So, these are the two things that you need to define the size of the particles.

On the other hand as it becomes little more complex like this rounded irregular particles, then you can see it also becomes more difficult to define a particular dimension or a particular geometric parameter to define the size of the particles. So, for example, in this case of a rounded irregular particle you have several of these parameters like the maximum length or the width of the particle or the projected height of the particle or the diameter at the center.

So, these are more than one parameter that one has to define in order to define the size of this kind of particles and when it becomes completely irregular like this, then it becomes really very difficult to come up with a particular characteristics dimension that will give the size of the particles.

So, therefore, you know we need to have certain measures or certain techniques in place which will help us in characterizing the particles in terms of their size and shape, right. And the first thing that is done towards that is to you know give some kind of qualitative description to the shape of the particles especially for the ones which are not very regular right like we can see in this particular image over here.

Spherical particles are well defined as we discussed just now, but as it becomes more and more irregular, then it has to be defined by you know some description which is more of a qualitative kind of description to just to come up with certain terms right which will define the particle shape. So, these are those different terminologies which are used to define the particle shapes and as I said this is more of a qualitative description of the particles.

So, what we discussed today is if I want to summarize now is that it is very important to characterize the powder in order to know the characteristics of the powder and there are different properties which can be evaluated.

And some of these patterns to the particle as such and some of them correspond to the bulk powder and these are those characteristics that we are looking for as far as the powder and the powder particles are concerned, starting from you know particle size and going all the way to the internal structure and the chemical composition of the powder particles.

And when you try and evaluate the powder particles, it is very important and in fact its very critical that we have a sample which is a true representative of the entire lot of the powder and in order to make sure that we have a good representative sample, there are certain do's and don'ts that one needs to follow. So, these are the recommended things that one can follow in order to ensure that a good representative sample is collected like you know taking the sample from different parts rather than from only top or from only bottom.

And then you know deagglomerating the powder by different methods, different processes like drying or you know using mechanical stirrer or ultrasonic agitation and so on. And also at times it is good to collect powder samples from a moving stream which will give you a true mix of different types of powder particles that can be present in a powder lot and therefore, it will be a good representative sample when it is collected from a stream of particles.

And then we talked about this you know different types of particles like the primary particles, agglomerates, flocks and aggregates and then finally, we defined the particle shape and the size.

The size can be defined in terms of a characteristics dimension which depends on the type of particles that we have and the shape can be qualitatively defined by a term like this for different types of particle morphology as we can see in this particular image. So, with that we come to the end of this class.

Thank you for your attention.