Powder Metallurgy
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Lecture - 30
Interparticle friction

Hello and welcome back. We have been discussing about different Powder properties and so far, we have learned about different characterization tools to, characterize the powder to, measure these properties. In past few classes we have discussed about two very important properties of the powder, the particle size and the surface area. Now, there are few more properties which are as important as far as the powder metallurgy process is concerned.

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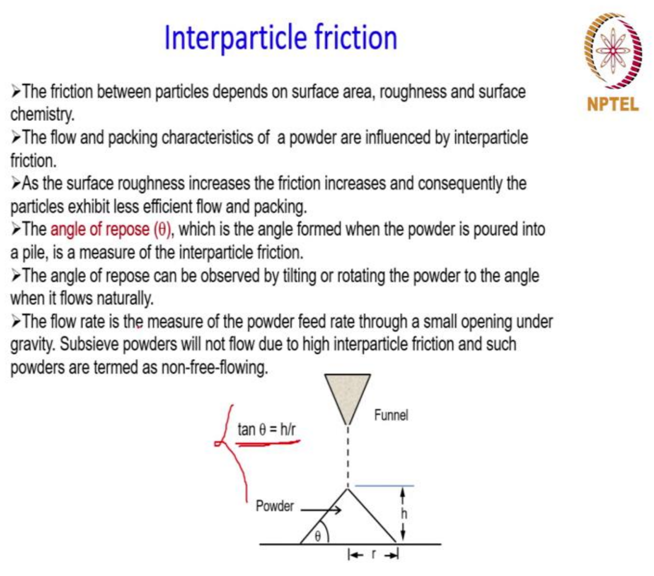

In this lecture we are going to discuss about those properties. Inter particle friction is one of them. Let us first try and understand the importance of inter particle friction and then we will talk about how it is measured? See in a powder metallurgy process what happens is, you start with a loose mass of powder and it is then consolidated to make a solid product out of it.

So, it is all about how the powder particles can be compacted to give it a particular shape to make that solid final product which you wanted. Therefore, the packing of powder particles becomes very important in this powder metallurgy process. And these packing characteristics of the powder are influenced by the inter particle friction. Because packing means the powder particles have to come together and pack together to close the pores in between them and consolidate the powder.

And how closely the powder particles can be packed? That; obviously, will depend on the friction between them and that is why inter particle friction becomes so, important in the powder metallurgy process. This friction between the particles will depend; obviously, on the surface characteristics, for example, the surface area, the surface roughness, and the surface chemistry. So, these are the surface characteristics which will affect the inter particle friction.

As I said the flow and packing characteristics of the powder are influenced by the inter particle friction. And, as the surface roughness increases, the friction increases which is obvious and consequently the particles exhibit less efficient flow and packing.

If you have a powder where the particles have high inter-particle friction, then it might become difficult to pack them and the compaction process may be difficult. So, the way it is measured from this parameter called angle of repose, which is nothing, but the angle which is formed in a heap of powder. So, a heap of powder you might have seen around you.

For example, if you have seen a heap of sand or even a heap of the common cooking flour in the kitchen, you would have seen how it forms a pyramid kind of thing? And the angle that it forms here, that is an indication of the inter particle friction. So, if we can measure the angle, we can get an idea about the inter particle friction of a powder. So, this angle of repose can be observed by tilting or rotating the powder to the angle when it flows naturally.

Let us say you take some amount of powder in some container and then keep tilting it unless the powder flows, naturally out of the container. That particular angle at which it is tilted when the powder starts flowing, that can be taken as the angle of repose. And the flow rate is the measure of the powder feed rate through a small opening under gravity.

If you want to know how the flow rate of the powder is because this is one property, which also depends on the inter particle friction. So, that can be measured by letting the powder flow through small opening under gravity. And, the rate at which it flows through that small opening can be taken as the measure of the flow rate.

If, you have sub sieve powders that will not flow due to high inter particle friction and such powders are termed as non-free flowing. If the inter particle friction is high which is the case for this subsieve powders. Then it will prevent the powder to flow freely and that is why this kind of powders are known as non free flowing.

If you have a powder on the other hand which flows freely out of any container when it is being tilted or when it is being passed through a small opening, then that powder will be known as a free-flowing powder. That is the opposite scenario of this non free flowing which is a typical characteristic of powders like this kind of sub sieve powders or if you have other powder characteristics like the surface roughness or the irregular morphology, which increase the inter particle friction that may also give rise to a non free flowing kind of powder.

So, to measure this angle of repose, what you need to know is the height of this heap or this pyramid which the powder makes without any agitation and this distance *r*. So, from that you will get this $tan\theta$ and you will get an idea about the angle of repose for any given powder.

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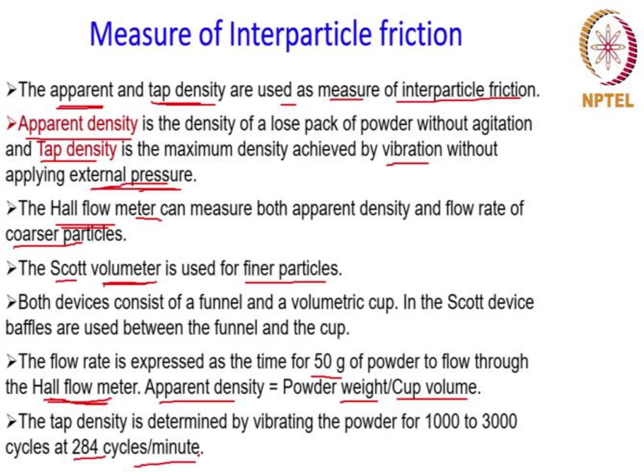

Now, let us see how experimentally this inter particle friction can be measured or what is the measure of inter particle friction in what way it is reflected? Let us see that. There are two parameters regarding the powder when it is filled in any container; one is the apparent density, and the other is the tap density.

These two parameters, are kind of the measure of interparticle friction. So, let us first understand what is apparent and tap density and then we will see how they can be measured? From that an idea about the inter particle friction can be obtained. Apparent density is the density of a loose pack of powder without any agitation. So; that means if you take any container and simply fill the container.

From that fill volume and the weight of the powder that is being filled, if you calculate the density without any movement or any agitation to the powder. That it does not settle down, that will give you the apparent density. On the other hand, tap density as the name itself suggests is the maximum density achieved by vibration without applying external pressure. So, here you tap the powder or provide some vibration, but without application of any external pressure.

So, although some vibration is given for the powder particles to settle, but there is no external pressure is applied. After that if you calculate the density from the volume and the mass, then that will give you the tap density of the powder. And the instrument which is used to measure this apparent density is known as the Hall flow meter, which can also measure the flow rate of coarser particles, and for finer particles one more device called Scott volumeter.

Both these devices consist of a funnel and volumetric cup, in the Scott device baffles are used between the funnel and the cup, And, the flow rate is expressed as the time for 50 gram of powder to flow through the Hall flow meter. So, that is how we can measure the flow rate of any given powder, you have to see how much time 50 gram of that particular powder takes to flow through the Hall flow meter.

And the apparent density of course is given by the weight of the powder and the volume of the cup. So, you fill the cup, and you can see the volume of the cup, because it is a volumetric cup. Once the cup is filled you can see you can read the volume of the powder. And, the weight of the powder is measured and from that the apparent density can be obtained. And, the tap density is determined by vibrating the powder first for about 1000 to 3000 cycles at 284 cycles per minute.

That is the kind of vibration rate, which is used to vibrate the powder and once it is vibrated it will try and settle down. So, the volume in this case will reduce compared to the original volume, when you had it without any agitation; that means, when you calculate the apparent density, that volume will be; obviously, higher than the tap volume or the volume after vibration.

So, once the volume is calculated after vibrating the powder, the weight is divided by that volume and the tap density is obtained. Now, let us see these two devices as to how they look like?

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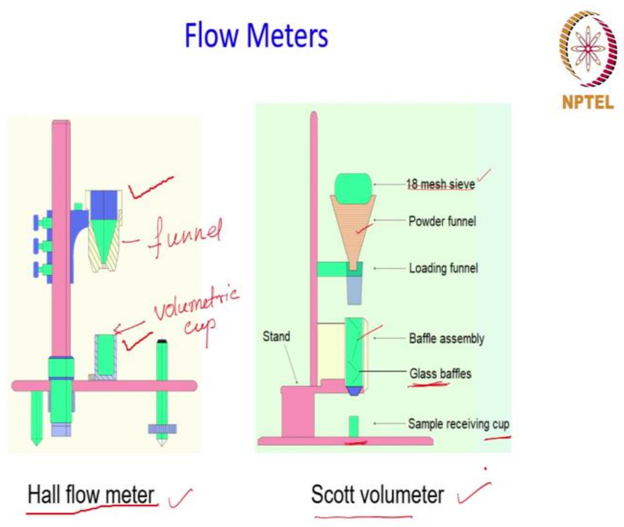

This is the Hall flow meter (left hand side image in the above slide). Here basically you have these two components one is this feeding cup to feed the powder and the other is this volumetric cup that you have over here. This is a cup which is having some graduated scale on this from which you can read the volume of the powder, which is being fed into the cup. Feeding funnel is on the top and this one is the volumetric cup (right hand side image in the above slide).

In Scott volumeter apart from the funnel you also have this baffle assembly. Before the powder feeds into the cup in between the cup and the funnel there are baffles this is to make sure that the powder flows and this kind of baffle assembly is particularly suitable for finer particles as we have talked about before.

So, the main parts of this the Scott volumeter are this. The funnel and before that a sieve is also provided, then there is a loading funnel here to guide the powder towards the baffle assembly. And, the baffle assembly contains these baffles which are made of glass.

The powder has to pass through these glass baffles before it comes down to the receiving cup at this end. Once the powder is received at the cup, the volume of the powder is noted and from the weight of the powder the apparent density can be obtained, and for the tap density it has to be vibrated for a particular number of cycles at a particular vibration rate and after that the volume is measured and from the weight the density is obtained.

So, this is how the tap density and apparent density can be obtained from these kinds of devices. And these two are the parameters which are kind of the measure of inter particle friction.

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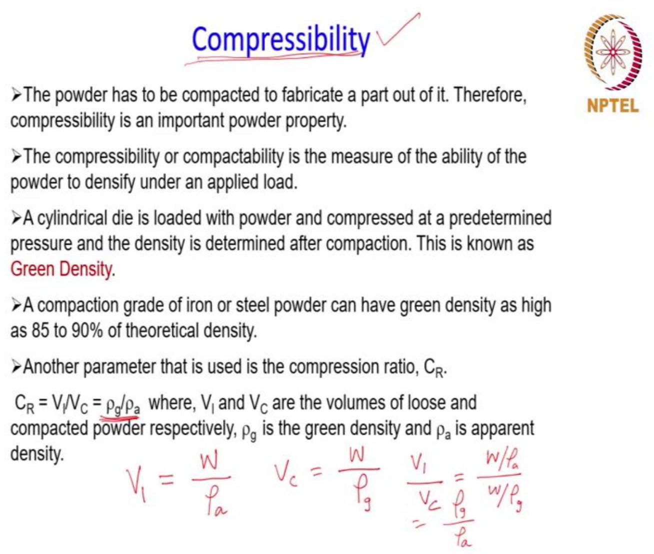

Now, the other property of the powder that you need to look at as far as the compaction process is concerned is the compressibility. Powder metallurgy process basically consists of compacting the powder into a particular shape and then consolidate and densify it to obtain the final product, which should be fully dense.

So, how good is the compactability that will be given by this property known as compressibility. And, this compressibility or compactability is the measure of the ability of the powder to densify under an applied load. So, when you compact the powder, what is done is the powder is filled into a die and then a pressure is applied for the powder particles to pack together and give you what is known as a green compact?

This is the shape, which is finally, densified by heating it at a particular temperature, when all these pores between the particles will be closed and a fully dense compact will come out. So, that heating process is known as sintering which is another important component of the powder metallurgy process.

Once the powder is compacted inside a die by applying a pressure. A solid compact comes out of it at the end of the compaction process and the density of that particular compact is known as the Green Density.

This green density will kind of indicate how good is the compressibility or the compactability of the powder? If, the green density is high at the end of the compaction process, then we know that the powder is having good compactability. Similarly, on the other hand if the green density is low, then we will know that the powder does not have good compressibility or compactability.

For example, a compaction grade of iron or steel powder can achieve green density as high as 85 to 90% of the theoretical density. That is why these kinds of iron and steel powder also go by this name as the compaction grade, because their compactability is so, good that just after compaction the density is 85 or 90% of the theoretical density. That means, only 10 to 15 percent porosity is remaining after the compaction process.

And, another parameter which is used to get an idea about the compressibility is the compression ratio, which is written as C_R. It is given as the ratio of the volume of the loose powder and the volume of the compacted powder.

$$C_R = V_l / V_C$$

Where V_l is the volume of the loose powder and V_c is the volume of the compacted powder. And, this can be simply equated to ρ_g / ρ_a, where ρ_g is the green density, and ρ_a is the apparent density. Remember the weight of the powder remains the same before and after compaction.

Therefore, the volume before compaction that is the volume of the loose powder V_l will be given as the weight of the powder divided by the apparent density.

$$V_l = W/\rho_a$$

And, similarly the volume of the compacted powder that is V_c will be given as,

$$V_c = W / \rho_g$$

So, if you take the ratio of V_l and V_c that will come out as,

$$V_l / V_c = \rho_g / \rho_a$$

We are going to talk about more of this in our future lectures, because green density is an important characteristics of the compact which will affect the sintering process as well. So, we need to talk about this in little more detail and we do it as and when it comes up, when we discuss about the compaction process.

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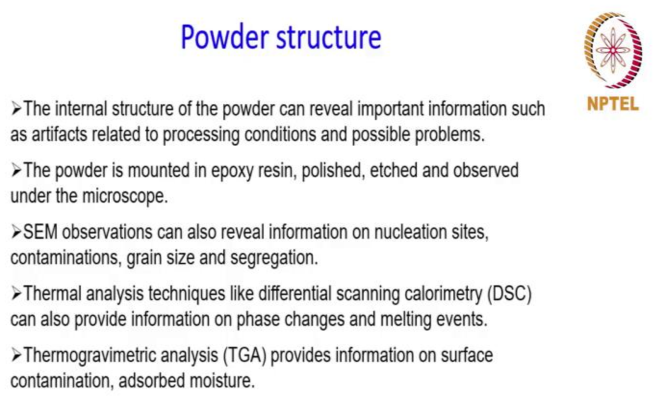

And apart from this external property of the powder that we have been talking about so far like the internal friction. The compressibility and so on, which primarily depends on the external condition of the powder particles, there are some internal properties also which have a bearing on the properties of the powder particles and also on the compaction and the sintering process.

And all these properties can be grouped into one category of this powder structure. Therefore, the internal structure of the powder is also important. Because it can reveal important information such as artifacts related to processing conditions and possible problems, if there are any in the powder which might lead to some problems during the powder metallurgy process.

In order to reveal the internal structure of the powder which is also known as the microstructure? The powder has to be observed under a microscope. And, in order to do that the powder is first mounted in epoxy resin, polished, etched and then observed under the microscope. This polishing and etching is to make sure that the surface is ready to be observed under a microscope, because if the surface is not polished and not shiny enough, that is not suitable for observation under a microscope.

When scanning electron microscope or ACM is used for observations, it can also reveal information on nucleation sites, contaminations, grain size, and segregation. So, such kind of information will not be available if you are using an optical microscope, but scanning electron microscopes are quite capable of doing many other things apart from simply capturing the images.

Therefore, this kind of information if somebody wants can be obtained by observing in an ACM. Then if there are any chances of phase change, then that can be evaluated by techniques like differential scanning calorimetry, and it can also reveal melting events. That means, the powder will melt at what particular temperature that can be obtained by carrying out DSC experiments.

Both of these are important because the powder compact has to be finally, sintered at a particular temperature. That particular temperature has to be chosen from these kinds of analysis to ensure that during the heating process, there is no phase change or no melting.

Otherwise, there will be problem in terms of either change in the properties of the powder or if there is melting, then it is not only a change which is not desirable, it can also be dangerous to the system because the molten metal can come out and spill over and so on. Therefore, this kind of information is quite useful while selecting the sintering temperature for a particular powder.

Then if you want to get information like contamination, absorb, moisture etc, then a technique called thermo gravimetric analysis can be used to provide such information. This will tell you about the temperature at which the adsorbed moisture and other things which are physically or chemically adsorbed under the surface of the powder particles can be removed.

TGA basically is a technique which measures the weight loss as a function of temperature. As you heat the material all these moisture and other things which are adsorbed onto the surface will slowly start evaporating at particular temperatures. So, till the time this evaporation or this removal of the adsorbed species happens the weight will continue to reduce.

And, then there will be a temperature beyond which it will show no reduction in the weight of the sample. That is how you will get to know that, what is the temperature that the material has to be heated to in order to remove all the adsorbed species on the surface. And, that is also important because powder particles might have adsorbed moisture or even other species or contamination adsorbed on the surface and it is important to remove them, before heating the compact to the sintering temperature.

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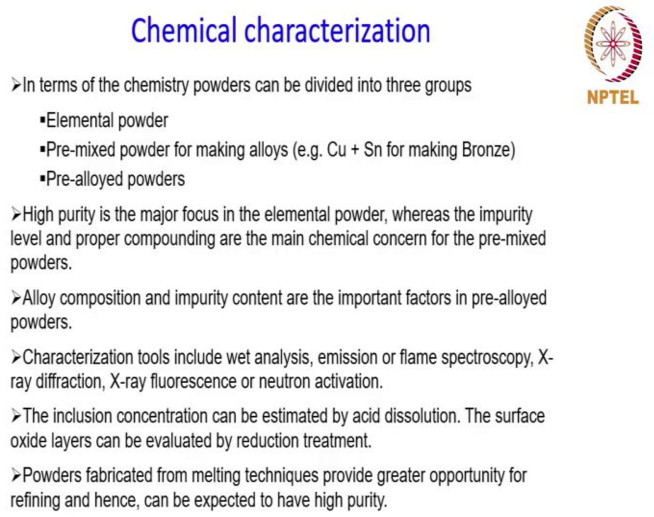

The chemistry of the powder will also have a lot of bearing on the properties of the material or the final product that you make out of the powder. In terms of the chemistry the powders can be divided into three groups.

Elemental powder that is the pure powder without the presence of any secondary or any other metal, then pre-mixed powders which are used for making alloys. For example, a mixture of copper and tin powders can be used for making bronze. And, then you will have pre alloyed powders where the powder is already alloyed before the powder metallurgy process.

High purity is the major focus in the elemental powder whereas; the impurity level and proper compounding are the main chemical concern for the premixed powders. Alloy composition and impurity content are the important factors in pre-alloyed powders, because here we are talking about the presence of a second metal or even the third metal, which are known as the alloying elements. And, they have to be added in a particular amount to make an alloy. Therefore, their content becomes important in such pre-alloyed powders.

Characterization tools to chemically characterize the powder include weight analysis, emission or flame spectroscopy, X-ray diffraction, X-ray fluorescence or neutron activation. There is a range of characterization tools which can be used to characterize the powder in terms of it is chemistry. Inclusions may be present in some powders in some materials and the concentration of such inclusions can be estimated by acid dissolution.

In that case what will happen is the metal will dissolve and the inclusion will remain back it will not be going to dissolve in the acid. Once you filter the acid solution this inclusion can be obtained and then you can measure their weight to get to know the concentration of these inclusions present in a particular powder. And, the surface oxide layers can be evaluated by reduction treatment.

So, when you reduce the oxide by exposing it to a reducing gas from that kind of treatment, it is possible to get some idea about the oxide layers, which may be present on the surface of powder particles. Powders fabricated from melting techniques provide greater opportunity for refining and hence can be expected to have high purity.

There are different fabrication routes that we have seen before and among those if there is melting involved. For example, the atomization technique that we talked about, those kinds of techniques are better equipped to provide a higher priority compared to other processes. So, before we wind up this lecture let us take a moment to summarize it.

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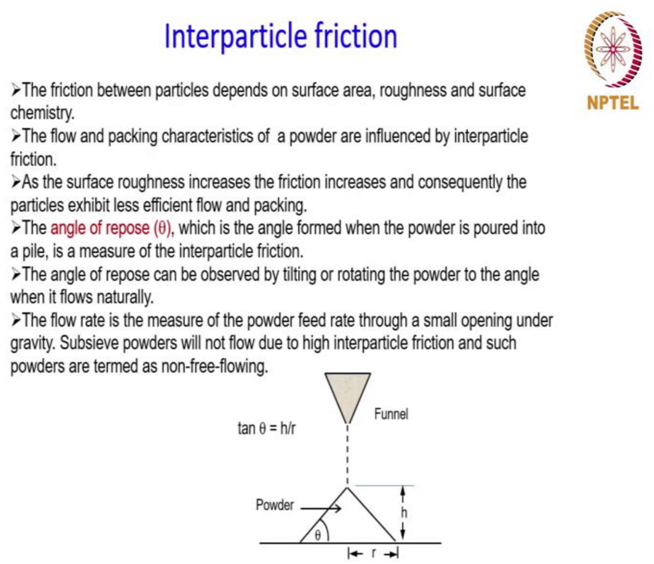

Today we talked about some other important properties of the powder, such as the inter particle friction which has it is own influence on the powder packing and the compaction process.

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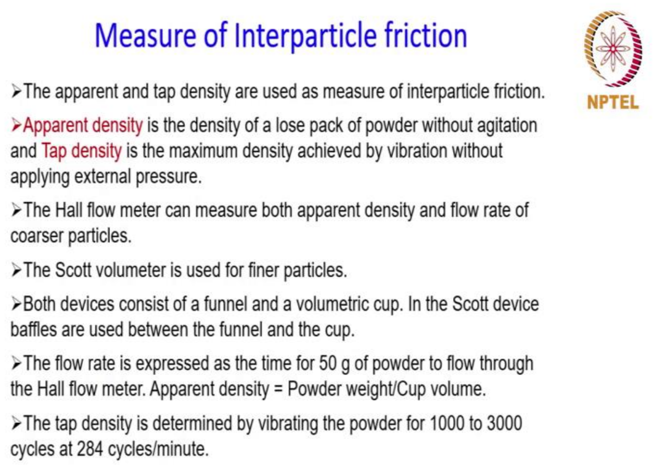

And, this is something that can be obtained from the apparent density and the tap density. Apparent density is nothing, but the density of a loose pack of powder, without any agitation or vibration, and tap density is the maximum density that can be achieved by vibration without applying any external pressure. These two are the parameters which are kind of a measure of the inter particle friction, and apparent density and tap density can be obtained by these two devices.

The Hall flow meter and the Scott volumeter, both of them basically consist of a funnel and a volumetric cup to measure the volume of the powder which is being fed and from that the density can be measured.

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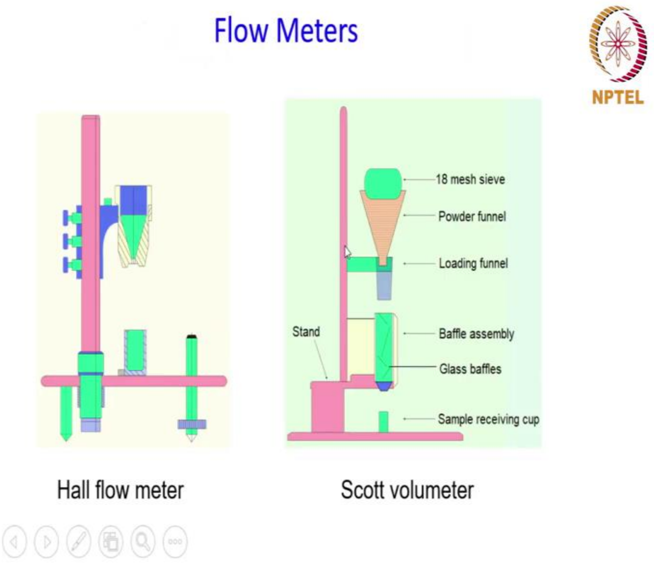

This is how these two devices look like, this is the Hall flow meter and this is the Scott volumeter. Then, we talked about the compressibility or the compactability which is a very important property of powder. Because, the powder has to be compacted in order to give it a shape. And, also how good the powder is in terms of it is packing characteristics that can also be evaluated by the compressibility of the powder, and green density is a very important parameter again as far as the compaction process is concerned.

Another parameter that can be used to measure compressibility is the compression ratio which is nothing, but the ratio of the volume of loose powder and the compacted powder, which is also equal to the ratio of green density to apparent density.

Then, we talked about the internal structure of the powder because the internal structure will also affect powder properties. And, this internal structure or the microstructure of the powder can be evaluated by observing it under a microscope. And, when scanning electron microscopes are used then it can also provide other information such as the nucleation sites, contaminations grain size, segregation and so on.

Thermal analysis techniques like, DSC and TGA will provide information such as phase changes and melting events or presence of contamination, or adsorbed moisture on the surface of the powder particles.

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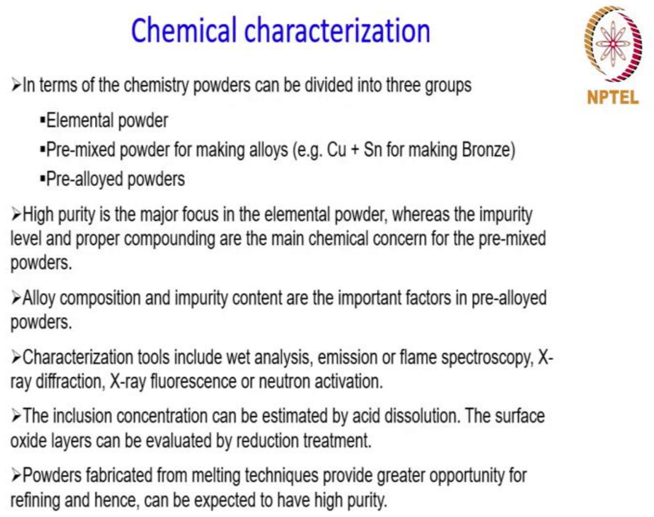

And, finally, we talked about the chemistry of the powder and in terms of the chemistry the powder can be divided into these three categories: elemental powder, pre-mixed powder, and pre-alloyed powders. With that we come to the end of this class.

Thank you for your attention.