Powder Metallurgy
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Lecture – 39
Slurry Techniques

Hello everyone and welcome back. In the past few classes, we have been discussing about the powder shaping, and we have seen that the choice of the shaping technique depends on the shape complexity of the part. While uniaxial pressing can be used for simple shapes, powder injection molding is best suited for complex shapes.

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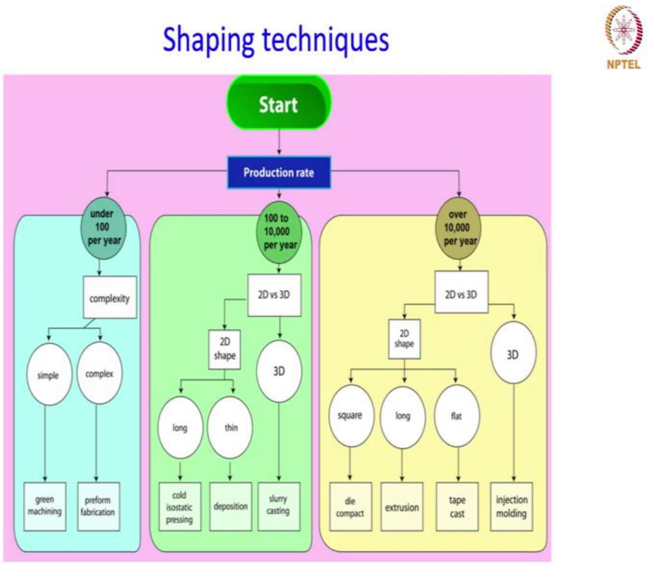

But as you can see from this shaping diagram, there are certain geometries. For example, a flat large area product or a long product which may require a different process altogether to get into that kind of shape. And in this class, we are going to learn about some of these techniques which deal with such shapes.

All these processes can be grouped into one category called slurry techniques because all these processes basically use a slurry to form the powder; so, let us see what kind of methods are used, and what kind of slurry is used in these techniques for shaping the powder into those kind of product shapes.

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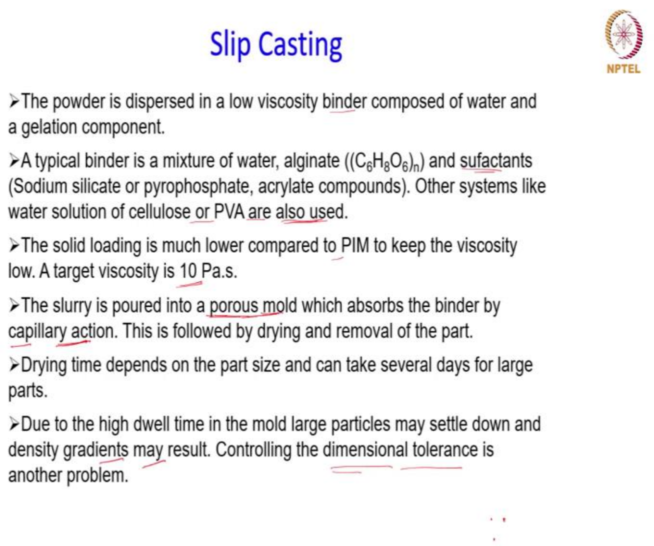

The first one that we are going to discuss is called slip casting. And in this process the powder is dispersed in a low viscosity binder which is composed of water and gelation component. A typical binder could be a mixture of waters, alginate, and surfactants such as sodium silicate or pyrophosphate, acrylic compounds etcetera. And other systems like water solution of cellulose or PVA are also used.

So, first the powder has to be made into a slurry, by mixing with such binders and other components like surfactants. And the solid loading in this case is much lower compared to what you have in powder injection molding; this is to keep the viscosity low. And a target viscosity in this case is about 10 Pascal second.

So, what is done in the process is after the slurry is made it is poured into a porous mold which absorbs the binder by capillary action. And this is followed by drying and removal of the part. So, the mold has to be having some kind of characteristics particularly with regard to this capillary action, in order to absorb the liquid that you have in this slurry.

So, the mold therefore, is made porous to create the capillary action through these pores because you might know that any liquid when it comes in contact with the solid surface having some kind of opening like pores or other kind of openings on the surface. We will experience a capillary force due to the surface tension and this capillary action will actually suck the liquid into the pores. So, that is the concept which is used here in this particular process by using this kind of porous molds.

When it comes to drying, the drying time depends on the size of the part, and it can take several days for large parts, and due to the high dwell time in the mold large particles may settle down and density gradients may result. So, this is something that one needs to keep in mind when you have a particle size distribution in the powder having different sizes of particles both large and smaller.

So, when the powder is poured into the mold, because of the time that it takes to settle down or to feel the mold completely and take the shape of the mold, there could be chances of larger particles settling down. And the other problem with this kind of process is controlling the dimensional tolerances.

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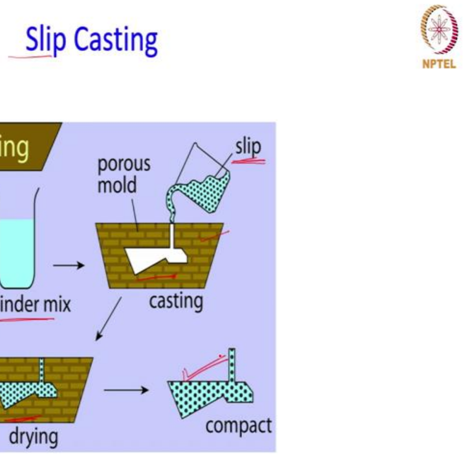

This is a schematic of the process as to how the slip casting process is carried out; a slurry or a suspension is first met, and then that suspension which is also known as a slip is poured into this kind of molds which are porous in nature. So, these pores absorb the liquid and the powder particles will take up the shape of the mold cavity.

And once it is formed into a particular shape, depending on the final product shape which is needed, it is left to dry, and at the end of the drying process you have the green compact which can be further sintered to get a fully dense product.

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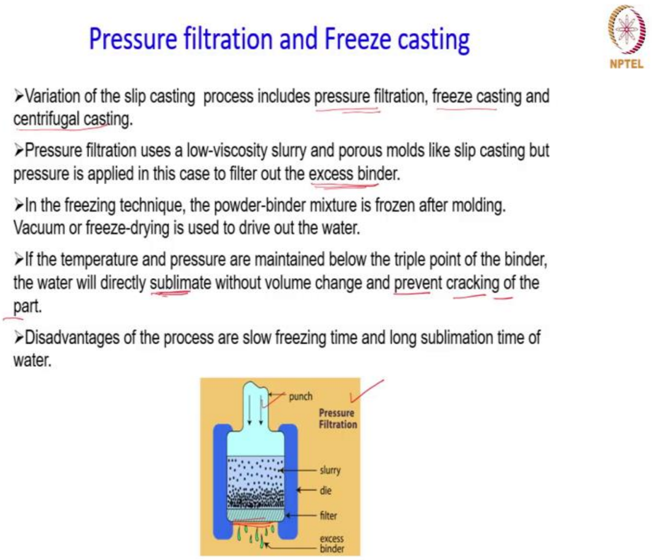

This was about slip casting. Now, there are some variations of the slip casting process and that includes pressure filtration, freeze casting, and centrifugal casting. Pressure filtration is a process which uses a low viscosity slurry and a porous mold just like the slip casting process. But in this case pressure is applied to filter out the excess binder. So, that is the difference between this process and the original slip casting process.

External pressure which has to be applied to filter out the excess binder; the process is depicted above in the image. Here, the slurry which is poured into the die cavity, and then with the help of this punch an external pressure is applied on the slurry, once it is fill into the die cavity to remove the excess binder through a filter on the other end.

So, as it is pressed from the upper side, the excess binder is squeezed out through the filter on the other side. The freeze casting is another process, another variation of the slip casting process in which the powder binder mixture is first frozen after molding.

And then, vacuum or freeze drying is used to drive out the water, which is present in the binder. So, once the water is driven out, the powder particles which were suspended into the water-based binder will now take up the shape of the mold cavity.

In this case, in order to ensure that this water coming out of the compact or coming out of the powder which is being filled into the die cavity, does not create any stresses and cracking of the part. The water is sublimated by maintaining the compact at a temperature and pressure which is below the triple point of the binder.

So, if that is done, the water which is present in the binder will sublimate; that means, it will directly convert from solid ice into water vapor, and in the process, it will prevent cracking of the part. That is the idea behind sublimation of the water rather than you know removing it through vaporization. The disadvantage of the process is slow freezing time and long sublimation time of water.

Now, there is a very effective utilization of this process to create unique microstructures or unique internal structure in a part which is beneficial for many applications, such as pharmaceuticals, energy devices and many more such applications which require a particular type of aligned microstructure which will help improving the process that takes place in that particular device, or in that particular component.

So, let us see how these kind of unique structures are created with the help of this freeze casting technique.

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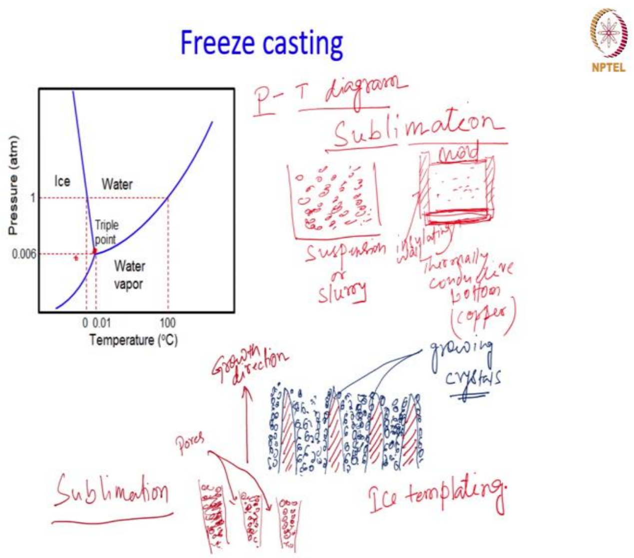

The process of removing the water has to be carried out below the triple point. The water phase diagram which is also known as the pressure temperature or P-T diagram (above graph in the image). Wherein, you can see the 3 states of water. The diagram depicts which particular state is stable under which conditions of temperature and pressure.

In triple point where all the 3 states of water coexist. This point for water is 0.006 atmosphere pressure and 0.01 degree Celsius. In order to sublimate the water, the conditions have to be lower than this point somewhere on the left of this point; if the conditions are maintained under those kind of conditions which is below the triple point, the water will directly get evaporated from the frozen ice, a process known as sublimation.

This is the process which is utilized in the freeze casting technique. And in order to create those aligned pores or pores having a particular type of structure or a particular type of orientation, the binder has to be first frozen in a directional manner.

If you have a water-based binder, it can be frozen if you cool it down below subzero temperatures. And if that cooling or the removal of the heat happens in a particular direction, then the ice crystals will also grow parallel to that particular direction.

Let us explain that with the help of a diagram. In the beginning the particles are suspended into this water-based binder. So, this slurry is now poured into a mold and this mold will have to have a certain characteristic in order to make sure that the freezing happens directionally.

The typical character that the mold should have is the walls should be thermally insulating and the bottom should be thermally conductive. So, insulating materials around these walls can be used insulating materials like polystyrene or Teflon can be used around the wall, and this thermally conductive bottom can be made of a highly conductive material such as copper.

Now, when the slurry is poured into this mold, this copper bottom can be brought in contact with a chilling agent such as liquid nitrogen and therefore, the heat will be removed along this axis of the mold, right. And as a result of that the ice crystals will grow from bottom towards up, like this.

These are the growing crystals which are being frozen and now the particles which are present in the slurry will now get pushed by the growing crystals and get concentrated around the crystals in the inter crystal regions or the inter dendritic regions. So, the particles will now be distributed around the growing crystals in this inter dendritic regions. So, this way crystals are grown in a particular direction.

If we remove these crystals by the process of sublimation, then these places occupied by these crystals will become the pores around the particles and the particles after this binder is removed will be densified. So, when the frozen liquid is removed by the sublimation process, so imagine that these ice crystals are not present anymore.

So, how that is going to look like? That will simply look like this. So, here are the particles which are now packed together. And these are the pores which are now created as the frozen liquid is evaporated from these places. And these pores, therefore, will be a replica of the crystals which were formed in these places.

That is how the aligned pores are created by this freeze casting process. And this is particularly useful in applications like photo catalysis, pharmaceuticals, some of the biomaterials, and many other applications including energy devices for making the support and the electrodes for these devices in order to enhance the performance.

Creation of those kind of pores will have their own advantage for each of these applications depending on the kind of processes or the kind of phenomena which takes place in a particular component or a device, those phenomena or processes will be kind of improved when this kind of aligned pore structures are present. And since these pores are created by using ice as the template, the process is also known as ice templating.

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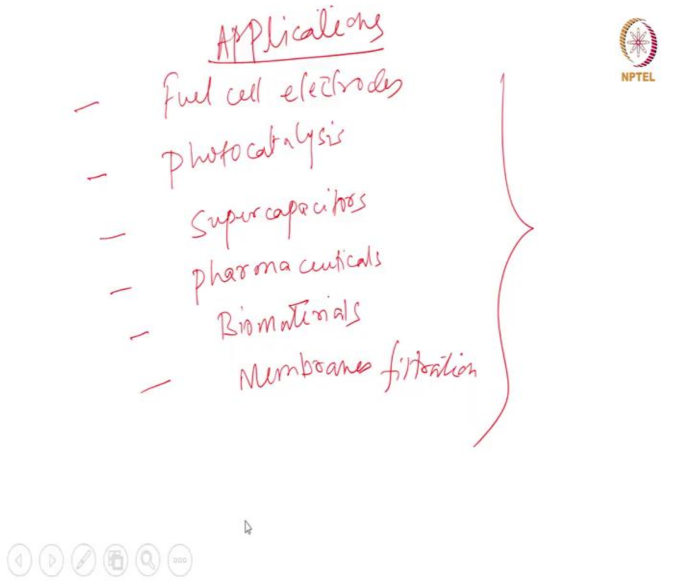

Some of the potential applications of this freeze casting process which creates this kind of unique pore structures are as follows. Fuel cell electrodes, where the electrodes will have that kind of structure having aligned pores and that will actually help in percolation of the gas through the electrode.

Then photocatalysis, substrates for super capacitors, pharmaceuticals, in biomaterials, membranes for filtration, ok. There are many such applications in which that align pore kind of structure will be helpful in improving the performance of such devices or components which are used in these applications.

Before we finish this lecture, let us take a moment to summarize it. Today, we talked about some of these slurry techniques starting with slip casting; it is a process of forming the powder using a slurry which is basically a low viscosity mixture of the powder and a binder. So, the process is done by pouring the slurry into a porous mold which absorbs the binder by capillary action.

And once the binder is absorbed and the powder takes up a shape of the die cavity it is followed by drying, and after the compact is dried, it is removed from the mold for further processing to densify it. And this is a schematic of the process as to how it goes starting from the binder mixer slurry to the final compact.

Then, we talked about some variations of this process which include pressure filtration, freeze casting, and centrifugal casting. We talked about these two processes, pressure filtration and freeze casting. Pressure filtration is like slip casting, except that here an external pressure is applied to squeeze out the extra binder as you could see from this diagram.

And then, we talked about this freeze casting process which involves a powder binder mixture that is frozen in the mold followed by drying under vacuum or freeze drying to drive out the water by sublimation process. And, we have seen this process can also be used to create unique structures inside several kinds of components which are used in different applications. And the process, how it happens, and what is the mechanism behind that we have discussed.

It basically involves freezing the liquid in a directional manner by using a particular type of mold which has thermally insulating walls and a thermally conductive bottom; with the help of that thermally conductive bottom, the heat is extracted in a particular direction. And, as a result the ice crystals grow directionally.

And when the crystals grow the suspended particles concentrate around them in the inter-dendritic regions like how you can see over here. And, when the ice crystals are removed by the sublimation process these places where the crystals grew are left behind as aligned pores as you can see, right.

So, since these pores are created by using ice as the template, the process also goes by the name ice templating. And the potential applications of the process could be in these areas such as fuel cells, photocatalysis, supercapacitors, pharmaceuticals, biomaterials, membranes, and so on.

Wherein, that kind of aligned pore structure is helpful in performing the operations involved in each of these cases. So, with that we come to the end of this lecture.

Thank you for your attention.