

Powder Metallurgy
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Lecture – 40
Tape casting

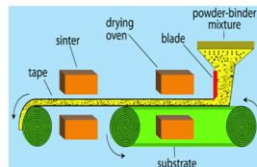
Hello everyone and welcome back. So, slurry techniques are the methods best suited for flat and long products. And, in the previous class we learned about the slip casting and the freeze casting process in this category. In this lecture we will learn about some more slurry techniques.

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Tape casting



- It is used to produce thin foils or tapes or for hard or protective coating using a powder-binder slurry.
- Typical binders are acrylic, waxes, PVA or polyvinyl butyral dissolved in appropriate solvent. A plasticizer like PEG is also added to provide stability.
- The slurry is fed into a moving paper or plastic sheet that passes through a controlled opening provided by a blade called "Doctor Blade".
- The blade spreads the slurry into a thin continuous sheet. The thickness is controlled by the controlling the gap between the blade and the sheet.
- The sheet can be either cut in required size or spooled.
- The sheet can be dried by moving it through a heater or drying oven before sintering.



The first one that we are going to discuss in this class goes by the name Tape casting, which is basically a process used to make thin flat products, such as foils and tapes; so, this process again uses a powder binder slurry. Typical binders are acrylic, waxes, PVA or polyvinyl butyral dissolved in an appropriate solvent.

And, apart from the binder a dispersant and plasticizer such as PGE is also added to the slurry. The dispersant controls the stability and the rheological behavior of the slurry. The binder and the plasticizer on the other hand imparts adequate strength and flexibility respectively to the green tape.

Each of these components of the slurry which is used for tape casting will have their own role to play; and, therefore, this is something that we need to discuss in little more detail, and I will come to the that in a little while. So, the way the process goes is the slurry is fed into a moving paper or plastic sheet.

The sheet passes through a controlled opening provided by a blade known as the “Doctor Blade”. So, the primary component of this tape casting machine is the doctor blade, because that is what controls how these slurries will be spread onto the paper or the plastic sheet which is used as a substrate. And it also controls the thickness of the tape which is going to be made.

From that perspective the blade is the most important component in a tape casting machine. So, the blade will spread the slurry into a thin continuous sheet, and the thickness is also controlled by the blade, and it can be done by controlling the gap between the blade and the sheet. The gap can be controlled with the help of a screw gauge or similar devices, in order to control the thickness of the tape which is being made.

Once the slurry is spread into a tape of a particular thickness. It can be either cut into required size or can be spooled for longer lengths. It can also be dried by moving it through a heater or a drying oven before sintering. So, there are lot of additives and other components present in the slurry.

So, all those components have to be removed before the tape is sintered for the final densification. Otherwise, they will leave behind porosity and the final tape which is obtained after sintering will not be fully dense. So, that is why the next step before the sintering is done is to heat this tape to a temperature, which is lower than the sintering temperature, in order to remove all the volatile components, present in the green tape.

This drying can be either done in line when the tape is being spooled by passing it through a heater or can be done afterwards also using an oven. Now, apparently the process looks very simple but there are things to be controlled in order to make a defect free tape. Specially, the slurry which is used here to make the tape for every given

powder the slurry composition in terms of this, components that you have has to be optimized to get a defect free tape of required quality.

So, the components are basically the binder, the solvent, the dispersant and the plasticizers. So, all these components have to be mixed in a particular proportion for a given powder to get the optimized slurry and then only you can expect to have a quality tape, which is free of defects. So, I am going to demonstrate that with the help of an example as to how this composition of the slurry will play a key role in deciding the quality of the tape.

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Tape casting

Slurry preparation



➤ Selection of solvent, binder and dispersant. Optimizing their proportions.

Stabilized ZrO ₂ Powder (g)	Solvent (ml)	Dispersant (ml)	Plasticizer (ml)		Binder PVB (g)
			Dibutyl Phthalate (DBP)	PEG	
15	Xylene-10 Toulene-10	Tripinol - 0.5	1	1.3	3.5
15	Xylene-10 Toulene-10	Phosphate ester- 0.4	1	1.2	3.5

Solvents - Xylene, Toluene, Water
 An azeotropic mixture - MEK (ethanol)

This is an example of making zirconia tape stabilized zirconia, which is used in many applications. So, this is basically a ceramic powder. So, the first thing that one needs to do is to select a proper solvent. And, then the other components, that is the binder the dispersant and the plasticizers are to be mixed in a particular proportion.

There is a wide range of choice of the solvent, but most commonly used are the organic solvents although, other kind of solvents such as alcohol, or a mixture of methyl ethyl ketone and ethanol can also be used. And, in fact, people have also used water as a solvent.

So, for the solvents, organic solvents like, xylene, toluene etcetera can be used. And, apart from that an azeotropic mixture of methyl, ethyl, ketone or MEK and ethanol can also be used. Here the term azeotropic means it is a mixture, whose composition cannot be changed by simple vaporization or distillation.

Once the mixture is made into a given proportion, that composition cannot be changed if you heat and vaporize the mixture; because, when you vaporize the composition of the vapor and the composition of the liquid are the same. So, an azeotropic mixture or an azeotrope is nothing, but a mixture of liquids whose composition cannot be altered by vaporization or distillation.

So, the mixture of liquids which are used as solvent in case of tape casting are actually azeotropic mixtures like, mixture of MEK and ethanol. Similarly, here you can see a mixture of xylene and toluene in equal proportions; that is also an azeotropic mixture, which is used over here for zirconia powder.

Apart from these kind of organic solvents or these kind of mixture, water can also be used as the solvent, but that again will depend on what kind of powder you have. So, the choice of the solvent has to be made based upon the nature of the powder.

Now, the next thing is selecting a particular binder and a dispersant. Here again you have a wide range of materials to select from, but again for a particular type of material a particular type of binder and dispersant will work the best. And, therefore, there is a kind of optimization in selecting these two components as well. And apart from selecting them you will also have to optimize their content in the slurry.

First of all to get a good uniform slurry and secondly, to ensure that the slurry can be easily and uniformly spread onto the substrate to get a good quality tape. So, for this example again triphenol was used in a given proportion in a given amount as the dispersant.

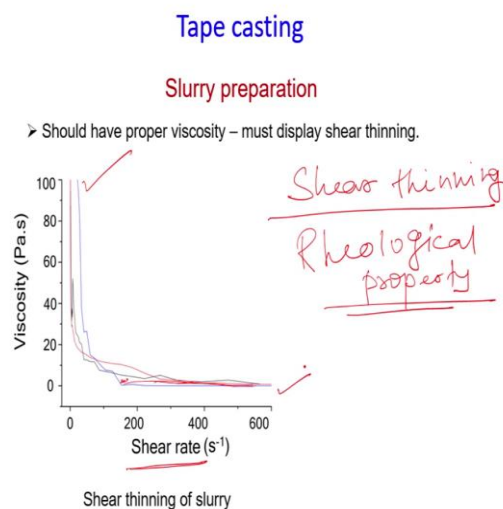
And, in another experiment phosphate ester was used as the dispersant; and, in both the cases dibutyl phthalate or polyethylene glycol was used as the plasticizer, again with a

given amount and the binder was polyvinyl butyral. So, all those were mixed uniformly, but there again you need to exercise caution, while mixing all these components together to make the slurry.

You have to do it one by one, first you need to take the solvent and add the powder. And first mix this together uniformly and only when this mixture becomes uniform, the other components have to be added one by one. So, the components have to be added in sequence one by one and every time you have to make sure that the next component is added only when the previous is completely mixed uniformly with the powder.

So, each of these steps will take their own time and you have to do step by step till all the components are uniformly mixed with the powder and the solvent. And only when that is done you will get a homogeneous slurry which is ready for the tape casting process.

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Now, in a tape casting process you need a viscous slurry first because it has to hold on, it should have enough stability and therefore, it should also have enough viscosity so, that when you feed it through this, feeding hopper over here. It will not flow like a liquid like a water and it should hold on with enough stability so, that the process can be carried out as it is suppose to happen.

Therefore, it should have enough viscosity. On the other hand, you also want this viscous slurry to spread uniformly on a substrate. So, in one hand you want a high enough viscosity and on the other hand you want this viscous fluid to uniformly spread easily over a substrate. So, these two things cannot happen at the same time. What it means is if you have a high viscous slurry that is difficult to spread over a substrate uniformly.

That means, during the process this viscosity should come down and come to a level which is low enough for the slurry to be spread easily over the substrate. This reduction in viscosity when you apply shear force is known as the shear thinning, which is shown over here. The viscosity is plotted against the shear rate and as you could see as the shear rate increases the viscosity drops softly, but at some point, it becomes constant and holds on.

This is the point where the slurry will have a low viscosity and it will be easier to spread over the substrate uniformly in the form of a tape. So, if this does not happen and if it remains highly viscous, then it will be difficult to spread.

That is why this shear thinning behavior is needed and therefore, you also have to make sure that the rheological property of the slurry is optimized in order for you to get a shear thinning, behavior in the slurry when it is subjected to shear.

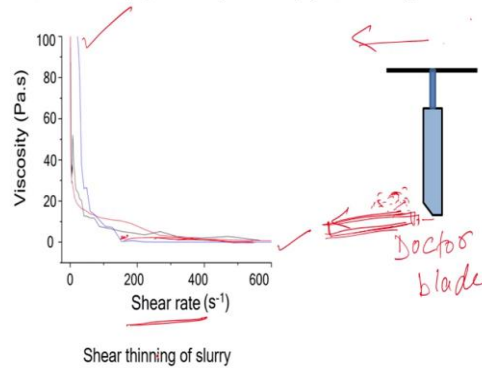
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Tape casting



Slurry preparation

➤ Should have proper viscosity – must display shear thinning.



The doctor blade provides the shear in this case. So, if you simply take a schematic of the doctor blade it will look something like this (above image). It has a sharper edge as you could see from here. So, this blade is moved into a particular direction linearly and here the slurry is already fed.

So, when the blade with its sharper edge moves over this slurry it actually provides this shear rate and as the blade moves over it the shear rate increases, and the viscosity drops drastically, and this shear thinning happens right. And, as a result of that it nicely spreads over the substrate which is below this slurry.

This shear thinning behavior of the slurry or the rheological property of the slurry also has to be taken into consideration, when you are making the slurry or slip for the tape casting process.

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Tape casting

Slurry preparation



► Deaeration of the slurry to remove trapped air bubbles.



Deaeration of slurry

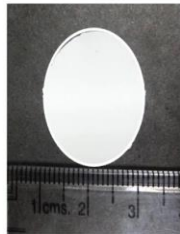
Final step before you take this slurry and you know feed it into the tape casting machine you need to make sure that there are no trapped air bubbles in the slurry; otherwise, it will lead to porosity and pinholes, which are not desirable for a good quality tape.

So, therefore, you have to make sure that if there is any trapped air bubble or any other gas into the slurry, it is removed. So, deaeration is the final step in making the slurry and it can be done by applying a vacuum as you could see here in this particular picture, this is something that we had done in our lab.

In fact, this example also that I had shown you was done in our lab for making these zirconia tapes, that we use for a particular application. So, here it is subjected to vacuum as we could see this is connected to a vacuum pump and the trapped air bubbles are removed by deaeration. So, that is the final step for making the slurry before it is used for the tape casting process.

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Tape casting



Sintered tape



Membrane electrode assembly (MEA)

Solid oxide fuel cell.
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Now, once the tape is made it can be spooled if you need longer lengths, or it can be cut into a required shape depending on the geometry of the product, which is needed out of it. This is an example where you can see that the tape is cut in a circular geometry and this was in fact, used for making a membrane electrode assembly or MEA for-fuel cell applications.

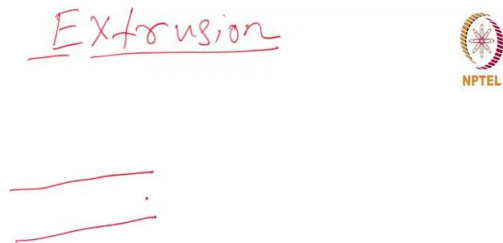
This was again done with the help of the same slurry. So, this is basically a stabilized zirconia tape, which is used as the electrolyte in a solid oxide fuel cell. And, now over this if you coat the electrodes on either side one side the anode which you can in fact, see over here and on the other side the cathode, it becomes a fuel cell, which can be used to generate electricity using hydrogen as the fuel.

So, this is a source of green energy, which does not pollute and does not produce any pollutants or obnoxious gases the only by product, that you get out of this kind of systems is water vapor. So, it is a completely non-polluting energy generation system, unlike the conventional energy generation systems, which generate lot of green house gases and are actually detrimental to the environment.

So, this is an example of some application area of the tape casting process, there could be

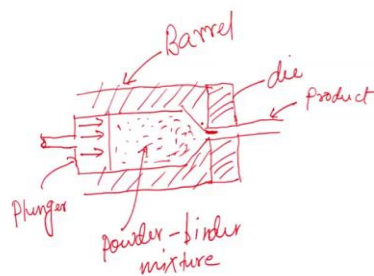
many other areas where in these kinds of thin membrane or other thin products are needed.

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The next slurry technique that we are going to discuss is the extrusion process in which the powder slurry is extruded through a die to form it into long products, such as tubes and rods. So, the equipment which is used for this process basically consists of a die through which the powder is extruded using a nozzle on the other end.

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Schematically if you see it looks like this. So, this is a schematic of the extrusion die which is used. So, the equipment basically consists of a barrel in which the powder is fed and pressurized. By a plunger as the plunger moves into the barrel it will pressurize the powder binder mixture and then there is a nozzle over here with a particular size.

And, as it is pressurized this will come out from this nozzle with a long product having a constant cross-sectional area. So, the product as we could see is in the form of whether this kind of rods and in order to create a tube a mandrel can be used here in order to create that hollow structure right. So, this is a process which shapes the powder into this kind of long or thin structures. Let us summarize this class before we wind up.

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- Summary
- Doctor blade
 - Slurry composition
shear thinning
 - Solvent, dispersant, Plasticizer
 - Extrusion



In this class we talked about two more slurry techniques tape casting and extrusion. In the tape casting process a slurry is fed through a tape casting machine, which basically consists of a doctor blade, which spreads the slurry on to a substrate in the form of a thin tape.

The main component of the tape casting process is the doctor blade and the other crucial factor in this case is the slurry composition, which has to be optimized for a defect free tape to form. And, apart from the composition the slurry should also display a shear thinning behavior. So, that when the blade encounters the slurry, the viscosity decreases due to the shear which is being applied by the blade.

And as a result of that the slurry spreads nicely on to the substrate and a thin and quality tape forms as the doctor blade moves over the slurry. And, then we discussed about what kind of solvent, dispersant and plasticizers are used for a particular type of powder.

We had taken an example of zirconia powder, zirconium oxide, and we had seen you know what kind of solvents and what kind of dispersant and plasticizers are used, and what are their proportion in order to get a good slurry which will give you a good defect free tape.

And, then we had seen that it is also important to degas the slurry before it is being fed into the tape casting machine, in order to make sure that there are no trapped gases including air bubbles. Otherwise, it will lead to pinhole kind of defects in the formed tape. Then, we discussed about the extrusion process in which the powder binder mixture is fed into a die which has a nozzle of a particular size.

And this extruder basically consists of a barrel in which the pressure is applied through a plunger. And, as the powder binder mixture is pressurized with the help of a plunger, it comes out through that nozzle as a long product like a tube or rod. So, extrusion is a process to make these kinds of products long and thin products with the help of these kind of extruder systems. And, with that we come to the end of this particular class.

Thank you for watching.