Powder Metallurgy
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Lecture – 59
Full Density Processing – 4

Hello everyone and welcome back. So, right now we are in the final stage of the powder metallurgy process that is, sintering. We have been discussing about the different techniques which are used for sintering the powder compact.

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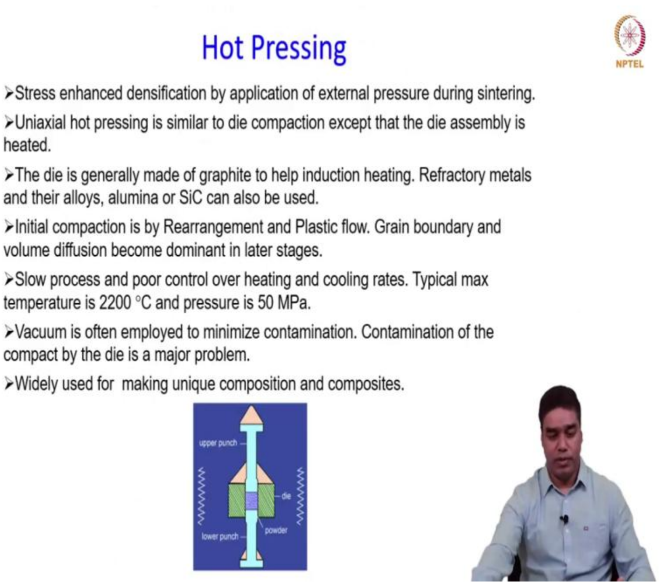

In the past couple of classes we have been discussing about the Full Density Processing and in the previous class we have talked about different techniques which are used for full density processing. The processes which are called hot consolidation processes, basically apply the pressure and the heat simultaneously.

These processes combine the compaction and sintering operation in one step unlike the conventional sintering process in which the powder is first compacted and then the compact is sintered separately. In the previous class we discussed about the hot pressing method.

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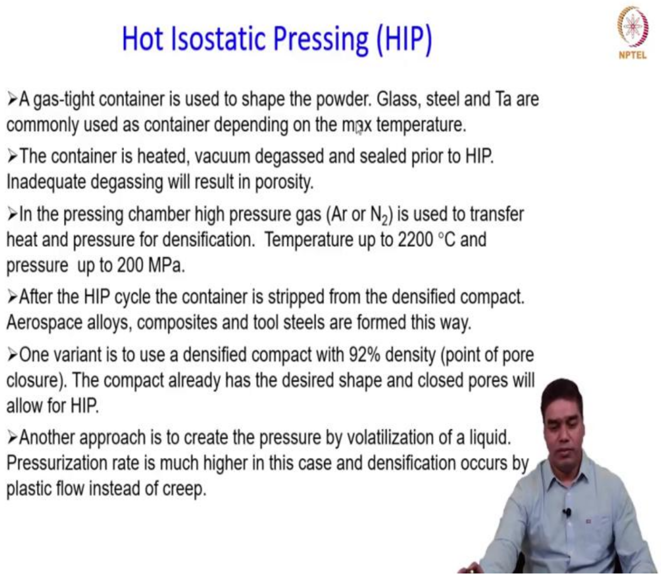

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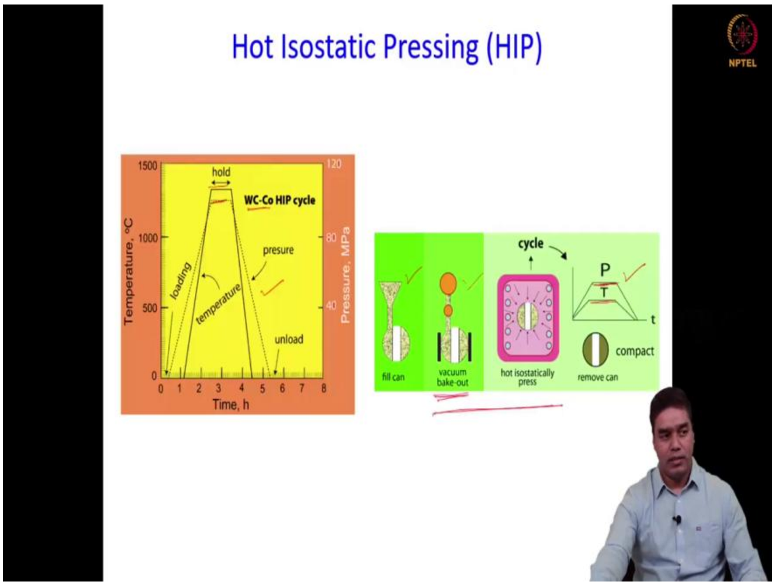

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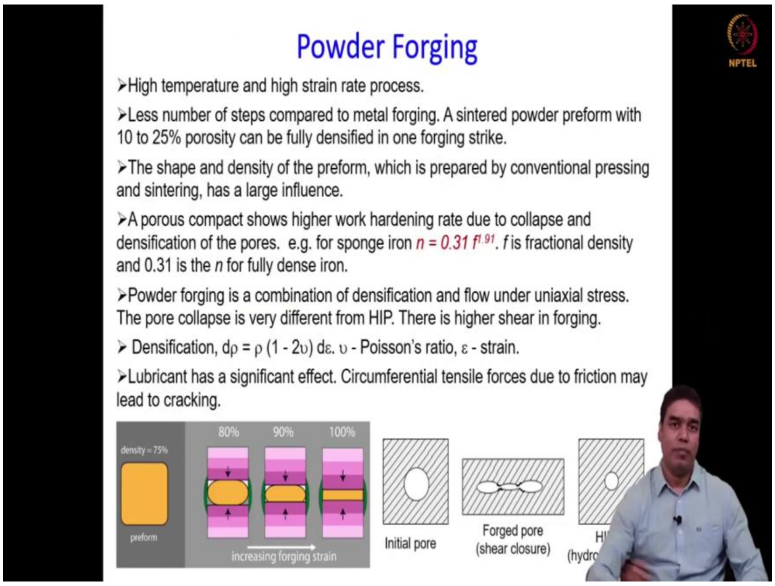

Then we talked about hot isostatic pressing and the powder forging operation. In this class we are going to continue and learn about some more methods of full density processing.

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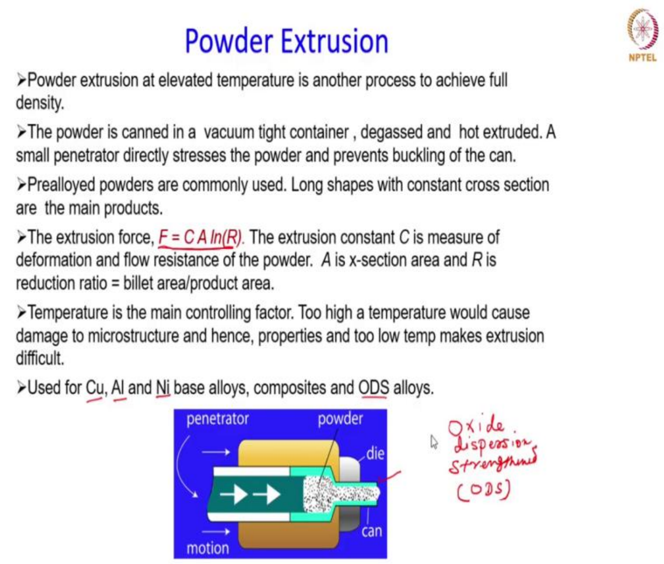

So, today let us start with one more process of hot consolidation, powder extrusion which is done at elevated temperature to achieve full density. If you remember we have talked about powder extrusion during the discussion about the shaping operation and powder extrusion is one of them, but it was done just to shape the powder using a slurry.

So, there was no question of any heat in that case, it was simply to compact the powder into a particular shape, but here we are talking about densification with simultaneous application of heat and pressure. So, the powder extrusion process that we are going to talk about right now is the one which is done at high temperatures.

So, here if you look at the process, the powder is first canned in a vacuum tight container and then it is degassed and finally, subjected to hot extrusion. So, this is what you see (slide above); the powder which is canned into a container and loaded into the extrusion die and then you have a small penetrator which directly pressurizes the powder.

With the help of this penetrator, the pressure is applied on the powder as it is being heated up and this kind of application of pressure through a penetrator will also make sure that the buckling of the container is prevented. The other end of the extrusion die has an opening the size of which depends on the final diameter of the rod which is needed.

So, as the powder is pressed through the penetrator ram, it gets densified and then comes out from this opening as a solid rod. And pre alloyed powders are commonly used here for making the product and the geometry that you obtain at the end of this process is basically long shape with constant cross sectional area.

The main products which come out of powder extrusion process are of long geometries. The extrusion force is given by an equation $F = C\,A\,\ln(R)$, where C is a constant, it is a measure of the deformation and the flow resistance of the powder, A is the cross sectional area and R is the reduction ratio, which is given by the ratio of the billet area to the product area, that is the ratio of the area before the extrusion and after the extrusion.

Here the temperature is the main controlling factor. Too high a temperature would cause damage to the micro structure and hence the properties. On the other hand if the temperature is low, it makes the extrusion process difficult because high stresses are involved in the process. High temperature can also affect the tool life and shorten the tool life and therefore, in this process the temperature has to be properly controlled.

The application of this process is to make product out of different metals like copper, aluminum and also alloys like nickel base alloys and it can also be used to make composites, because a second phase can be mixed with the powder and can be consolidated along with the powder to give rise to a composite. Another type of alloy which are basically high temperature alloys known as Oxide Dispersion Strengthen alloys or ODS in short, which basically contains oxide particles as dispersion and these particles provide strength at higher temperatures. These are alloys which can be used at high temperature applications and powder extrusion is an attractive process for making such alloys. So, this was all about the powder extrusion process.

So, far the processes of full density that we have seen are all by hot consolidation where the heat and pressure are applied simultaneously. There are also some cold methods of full density processing in which there is no need to apply the heat simultaneously and a density close to 100 percent can be achieved even without that. Powder rolling is one such process.

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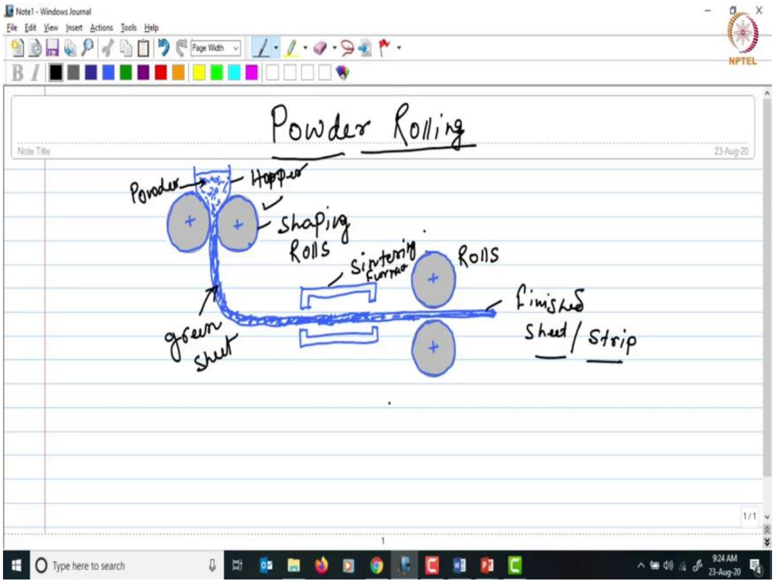

So, this is how (slide above) the setup of powder rolling looks like. Here a loose powder is gravity fed in the gap between two rolls which generate a strip or a sheet. The shaping rolls generate the green sheet or strip. The powder is fed through the hopper and once the green sheet is made, it is sintered in the furnace to further densify it.

Then it is further rolled to fully densify it. Since there is no application of heat in the beginning while the powder is being shaped, it has to be sintered in order to achieve a 100 percent density and it is also rerolled to make sure that the end product which comes out is 100 percent dense product.

The finished product which comes out after this process can be in the form of a sheet or a strip. The main challenges in this technique are the slipping and cracking of the feed material because here a loose mass of powder is fed through this rolls. So, although sometimes a binder is mixed, but still there are chances that this shaping will be a challenge because the powder can slip through the rolls or even if it is shaped into this kind of sheet or strip there are chances of cracking.

So, that is one of the major challenges in this process and the other limitation of course, is with regard to the product form. Here only a sheet or strip can be made, but nevertheless this is a process which can generate high densities even in cold conditions without the application of simultaneous heat during the shaping process.

Now, we are going to talk about a technique which is very effective in achieving full density in a wide range of powders starting from metal to ceramic to composites and whatnot because there the mechanism of densification is quite different in the sense the heating mechanism itself is very different and vacuum is also applied so, that the problem of trapped gas, which limits the densification is not there. The process goes by the name spark plasma sintering.

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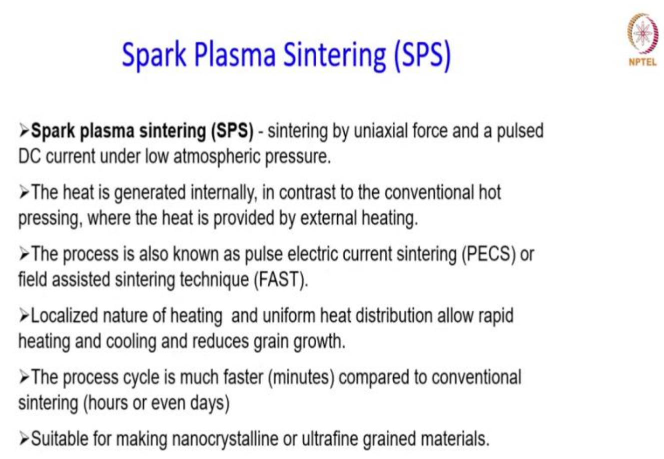

The heating mechanism is quite different from what we have seen. In all the processes of sintering that we have discussed so far the heating is done by keeping the powder compact inside the furnace and heating up the compact by conduction.

And this kind of heating sometime leads to problem in terms of achieving uniform temperature across the compact or it can also give rise to some differences that you need to first estimate and accordingly you will have to set the temperature.

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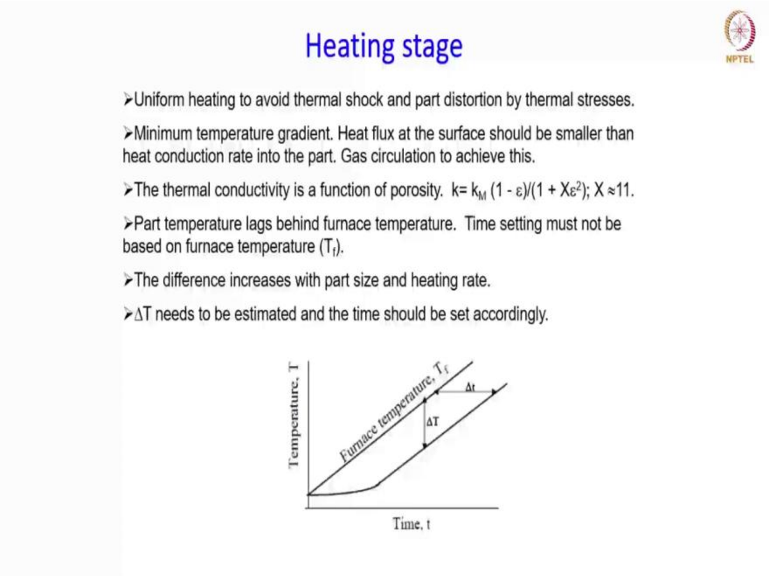

So, if you remember we had talked about this temperature setting for conventional sintering processes, where the heating is done by conduction. The powder compact is kept inside a furnace and the furnace is heated up and the heat is then conducted to the compact.

So, you can always expect a difference between the furnace temperature and the actual compact temperature and this difference ΔT has to be estimated if you want to achieve the temperature in the compact which is actually needed. Therefore, in these cases ΔT has to be estimated a priori and the temperature has to be set accordingly and the sintering time also has to be set based upon this temperature after it is calculated by considering the ΔT.

In order to avoid these kinds of issues with regard to the heating of the compact, the spark plasma sintering process that we are going to talk about now applies a different kind of heating. Here the heat is generated internally in the compact instead of conduction through the furnace and this process is also known as Pulse Electric Current Sintering PECS or Field Assisted Sintering Technique or FAST. So, these are the other names of the process and all these names arise from the fact that the heating method is through the application of an electric current.

So, that is how it is heated up internally through the joule heating mechanism where you apply an electric current and that gives rise to heating.

So, here the heating is localized; that means, it happens particle to particle inside the compact as the electric field is applied and therefore, the distribution of the heat is uniform and this also allows rapid heating and cooling.

And therefore, if you want to make materials where a fine grain structure is needed, that means, if you want to avoid grain growth, this would be the process of choice. Because, here the heating and cooling takes place very fast so there is no chance for the grains to grow during the heating or the cooling operation. Even the sintering time which is needed in this case is much lower compared to conventional sintering simply because of the fact that the heating process here is much effective as it is done internally in the compact.

So, that also ensures that the grain growth is limited and therefore, a fine grain structure will be generated in the final sintered product. The process cycle is much faster in minutes compared to hours or days in case of a conventional sintering process. So, therefore, it is suitable for making nanocrystalline or ultra fine grained materials, where you need to preserve the small grains. Let us see some details of the process.

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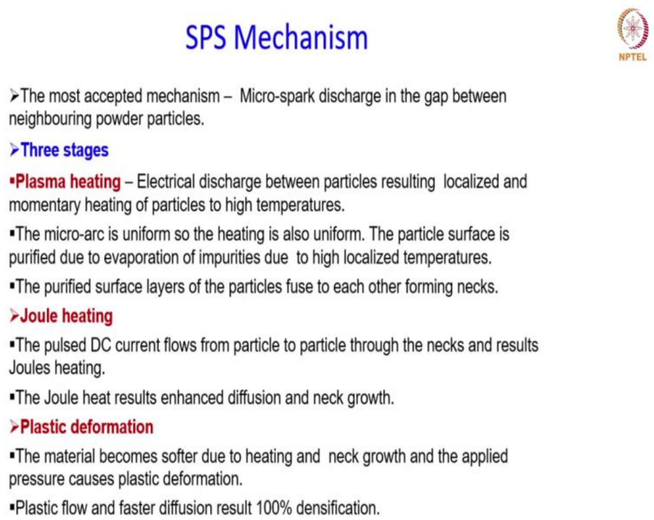

There are various mechanisms proposed for this SPS process, but the most accepted one is as follows. A micro spark discharge is generated in the gap between the powder particles when an electric field is applied and that leads to the heating.

And as far as the sintering process is concerned, there are three stages, but the phenomenon which happens during each of these stages is quite different from conventional sintering. In the first stage, plasma heating takes place when the electrical discharge between the particles resulting in localized and momentary heating of the particles to high temperatures.

So, instead of conduction heating through the compact which happens slowly and depends on the conductivity of material, here the heat is generated by the electrical discharge from particles to particle contacts. On a localized scale the heat is quite intense because, it is only through the contacts between the particles and therefore, the sintering temperature can be achieved very fast in a very short period of time.

The micro arc is uniform. So, the heating is also uniform because it is set up between the particles to particles and since in the compact there are particles all around so, you can expect it will happen uniformly.

The particle surface is purified due to the evaporation of the impurities due to the high localized temperatures. This is another advantage of the process. As the spark is set up between the particles, it generates very high localized temperatures and due to that whatever impurities are adsorbed on the particle surface will simply evaporate and you will have a fresh particle surface for the bonding to occur during the sintering process. The purified surface layers of the particles will fuse to each other forming the necks and the densification will start as the necks grow. So, this is all about heating through the joule heating effect.

In SPS, a DC pulse is applied which flows from particle to particle through the necks and that results in the joules heating, which is uniform across the compact because as I said it happens from particle to particle and this heating also results in enhanced diffusion and neck growth and therefore, the densification process is quite fast compared to the conventional sintering process.

And during this heating there is simultaneous application of pressure because we are talking about the hot consolidation processes right now. So, this is also one of those. So, apart from the heating here you have simultaneous application of pressure as well.

That leads to plastic deformation and that is the third stage once the heating is achieved and the compact is heated up uniformly through joules heating. The plastic deformation will begin as the pressure is applied. Due to this heating, the material becomes softer and the applied pressure causes plastic deformation and this plastic flow and the faster diffusion will result in 100 percent densification. So, for most of the materials you can achieve this 100 percent densification in a much shorter period compared to the conventional sintering process.

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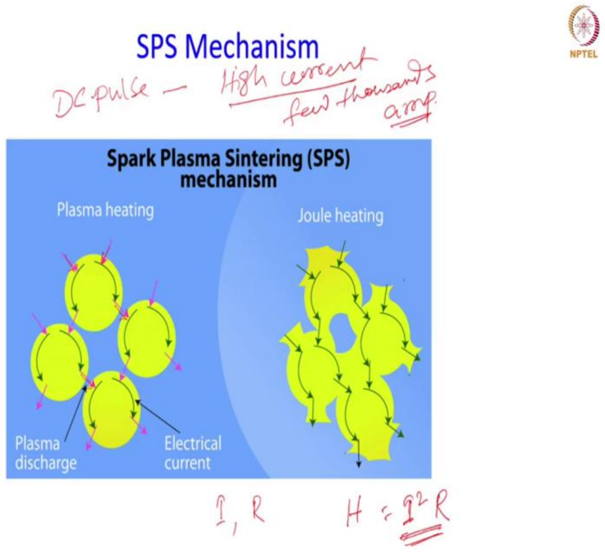

So, here is a schematic (above slide) which shows the heating mechanism of the SPS process. So, here an electrical current is used to create that joule heating effect and as the current is applied this plasma discharge is set up in the gap between the particles as you can see from these arrows over here the red arrows and that leads to the joule heating, which uniformly takes place across all the particles.

So, joule heating is the heating effect of current which occurs due to the resistance that is offered by the material to the electric current. So, if I is the current flowing and R is the resistance then the heat $H = I^2R$. So, in order to achieve a high temperature here within a short period of time a high amount of current is needed. So, therefore, the electric field in this case is applied through a DC pulse having a very high current to the tune of few thousands of amperes.

So, as this amount of current flows into the material there is intense heating due to the joule heating effect and that leads to very high localized heating of the particles and one effect of that (which is primarily responsible for a faster densification which takes place in this case) is the fact that this leads to surface cleaning because the particles have to bond through the contacts at the surface as the neck forms.

So, therefore, when you have a clean fresh surface the bonding between the particles will be enhanced and therefore, the densification will also be improved and a faster densification will take place.

So, that is what this joule heating effect does in terms of achieving a better particle to particle bonding and therefore, an enhanced densification. So, that was about the basics of this SPS process.

So, for today before we wind up let us take a moment to summarize this class. Today we talked about some more of full density processing; one was the powder extrusion in which the powder is extruded at high temperature. So, here the powder is now loaded in a die and it is being pressed with the help of a RAM and as it is pressed at high temperature the powder is densified and from the other end which is having the opening, it comes out as a rod.

So, the main products of this powder extrusion process are basically the long products having constant cross sectional area and here we have seen that the temperature is the main controlling factor, which will control the process and different kinds of materials including metal alloys and some specialty materials like these ODS alloys can be processed by this powder extrusion method.

Then we talked about very unique technique called spark plasma sintering which is quite different from all the other sintering methods that we have talked about so far. Because in this case the heating which is taking place to heat the compact and densify it is very different from the conventional heating processes that we have for the other methods that we talked about.

So, here a high amount of current in the form of a DC pulse is applied to generate the heating in the compact through a joules heating kind of effect, and that is what we have discussed in this class today as to how it happens.

So, a very high current to the tune of few thousands of ampere is applied in the form of DC pulses, which leads to intense localized heating and this intense heating actually leads to cleaning and purification of the particle surface resulting in better bonding between the particles and as a result of that the densification process is enhanced in this particular technique. And with that we come to the end of this particular class.