Powder Metallurgy
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Lecture – 58
Full Density Processing - 3

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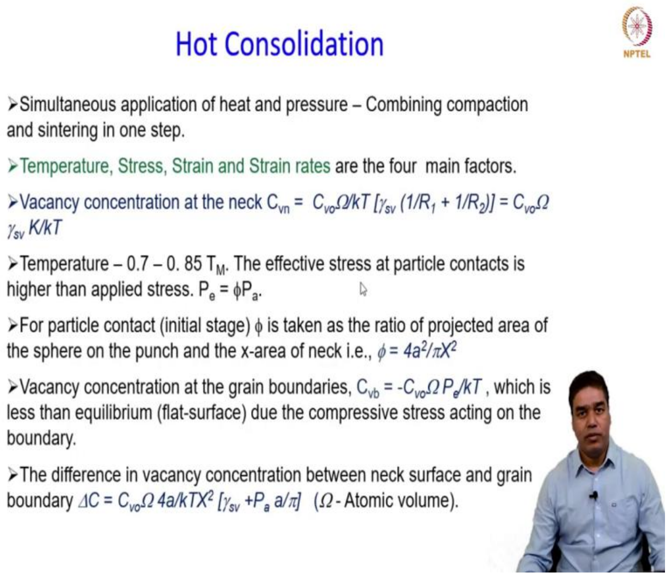

Hi everyone. So, currently we have been discussing about Hot Consolidation or Full Density Processing. In order to achieve densification simultaneous application of heat and pressure is the approach.

Unlike the conventional sintering process in which the green compact is sintered separately, in the full density processing, the compaction and the sintering operations are combined in a single step because here pressure and heat are applied simultaneously.

And in the previous class, we were discussing about the mechanisms of hot consolidation and that the mechanisms can be derived from the creep deformation mechanisms; because in creep also a similar kind of deformation takes place at high temperature when the compact is pressed at a particular temperature.

And if we can account for the porosity which is present in the green compact, the creep equations can be used for the mechanisms which are operative in the hot consolidation process.

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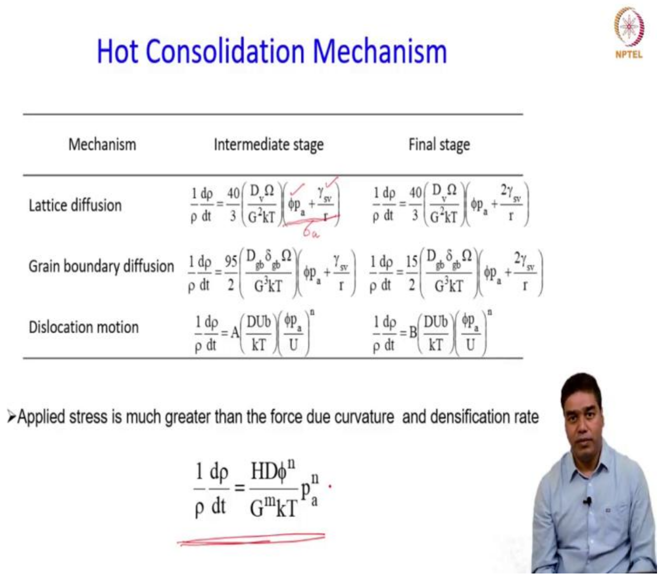

So, here in this table the equations and the corresponding mechanisms are listed. So, in case of simple creep where an external stress is applied at a particular temperature the term $(\Phi P_a + \frac{2\gamma_{sv}}{r})$ will be only the applied stress (σ_a).

We are considering a green compact which is being pressed at high temperature. Apart from the stress we also need to consider the porosity which is present and that is where this term ΦP_a has come into picture here where Φ is the stress intensification factor which comes in due to the presence of the pores and P_a is the applied pressure.

And apart from that the pores have a curvature and due to that there is a stress which is given by the term $\frac{\gamma_{sv}}{r}$. So, when you combine these two you have the effective driving force for the hot consolidation process.

So, that is how the porosity has been taken into consideration in this case when you replace the applied stress with the effective driving force which takes into account both the applied pressure as well as the presence of the porosity.

And then finally, we have seen a generalized form of the densification rate which is given by this particular equation

$$\frac{1}{\rho}\frac{d\rho}{dt} = \frac{HD\Phi^n}{G^m kT} P_a^n$$

and here the exponents m and n have different values depending on the operative mechanism.

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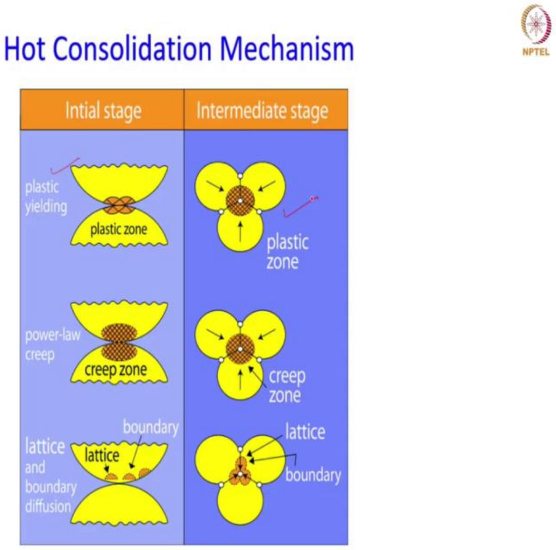

So, again coming back to the mechanisms of the hot consolidation process, this diagram also shows the mechanisms as a model considering the spherical particle in contact. So, here you can see that at high stresses when the stress exceeds the yield strength of the material, plastic deformation begins and the densification occurs instantly once the plastic deformation begins.

When the stresses are low, the diffusional flow can combine with the applied stress and the densification occurs by the creep process and the diffusion can occur either through the lattice or through the grain boundaries. When it occurs through the lattice the creep process is known as Nabarro-Herring creep and when it occurs through the grain boundaries it is known as coble creep.

So, depending on the applied stress it can either densify through the plastic deformation process itself or by a time dependent deformation process, creep. So, now, that we have understood the mechanisms let us go ahead and see the different methods which are used for full density processing. So, we start with the hot pressing process.

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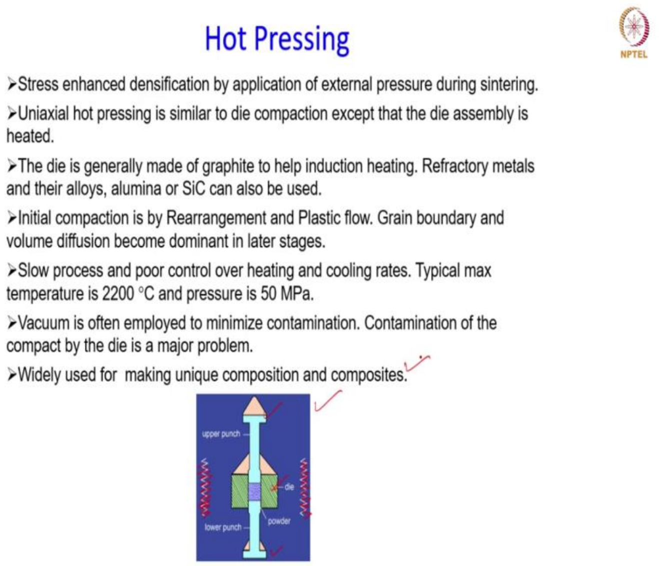

Here pressure is applied during the sintering process. So, it is basically stress enhanced densification by application of external pressure. The uniaxial hot pressing is similar to the die compaction except that the die assembly is heated up. So, that is the only difference otherwise this is very similar to the die compaction (refer slide above) where you have the die in which the powder is filled.

And then, you have an upper punch and there is a lower punch through which the pressure is applied onto the powder. So, here in order to heat the powder there is a furnace around the die. As the powder is heated up, it is also simultaneously pressed. So, the initial compaction in this case again happens by the rearrangement and the plastic flow as the powder is pressed and the temperature is raised.

And grain boundary and volume diffusion become dominant in the later stages when the stresses are high. When the compact reaches the sintering temperature and is at a higher temperature, the process is slow and the control over the heating and the cooling rates are not that great. Typical maximum temperature which is used in hot pressing is about 2200 degrees Celsius and the typical pressure is about 50 MPa.

And in many cases vacuum is used to minimize the contamination. Contamination of the compact by the die is a major problem, particularly at high temperature. And therefore, vacuum is used in most of the cases in order to eliminate the chances of contamination.

And this is a process which is widely used for making unique composition and composites; because here the powder can be mixed with some other elements to give rise to an alloy and this alloy composition can be varied depending upon the requirement in the final product.

And this is a simple process where it is easy to control the composition and therefore, it also provides you an opportunity for making unique compositions and you can also mix a second phase which does not dissolve in the metal and that will give rise to a composite. And since the sintering process is pressure assisted, here the densification of a material like composite where you have insoluble particles can be done easily.

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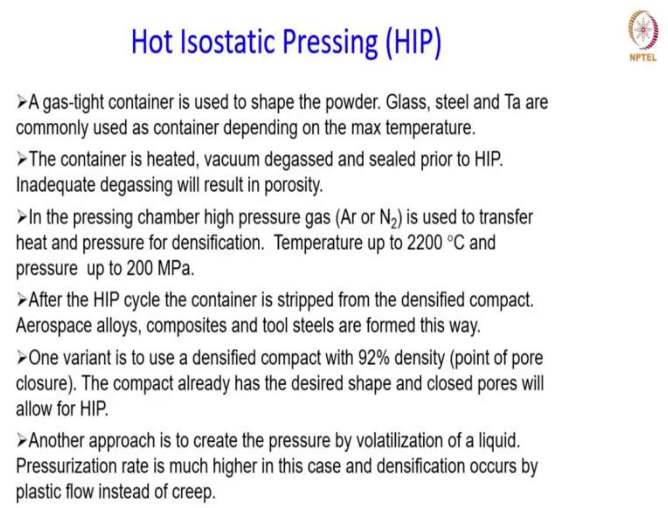

We have talked about cold isostatic pressing before the CIP process for compacting the powder by applying the pressure uniformly from all sides. The Hot Isostatic Pressing or HIP is the hot counterpart or the hot consolidation counterpart of the CIP process. And therefore, the compaction and sintering processes are combined into one step and at the end of the process you get a fully dense compact.

Here a gas-tight container is used to shape the powder and in order to do that glass, steel or tantalum are the commonly used materials for the container depending on the maximum temperature.

The container is heated, then it is vacuum degassed and finally, sealed before it is subjected to the heating process. If the degassing is not adequate, then it will lead to porosity. If there are trapped gases in the pores, it limits the final densification and it is difficult to achieve full densification.

And therefore, the vacuum degassing has to be done carefully so that there are no chances of trapped gases and you do not end up with porosity in the final sintered compact.

So, in order to pressurize the powder a high pressure gas, a high pressure inert gas like argon or nitrogen is used and temperatures up to 2200 degree Celsius and pressure up to 200 MPa can be used for hot isostatic pressing.

And after the HIP cycle, the container is stripped from the densified compact and this is a process which is quite attractive for making aerospace alloys, composites and tool steels. And one variant is to use a densified compact with certain amount of porosity, let us say, 8 to 10 percent of porosity. The compact in that case has been already shaped into the desired final shape and the remaining pores are closed by the hipping process. So, here it is just to close the remaining pores which are there in the compact which is already made into the final shape.

And another approach of the process is to create the pressure by volatilization of a liquid. And the pressurization rate in this case is much higher compared to gas pressurization and the densification occurs by plastic flow instead of creep. So, here we have seen that when the stress is high and if it exceeds the yield strength of the material then the plastic deformation will begin and the densification will occur by plastic flow.

The schematic in the slide below shows the setup used for the hot isostatic pressing. A can is used to fill the powder that is the first step of the hipping process and then the powder has to be degassed. So, a vacuum chamber is used to degas the powder. So, that is the vacuum bake out process to remove any trapped gases and finally, once it is degassed, it is transferred to the hipping chamber where it is heated up under isostatic pressing.

The pressure temperature cycle is shown in the slide below. When the pressure is applied, the temperature also is simultaneously applied and at the sintering temperature, where it is held for a particular period of time, the pressure is also held during that time for the densification to occur.

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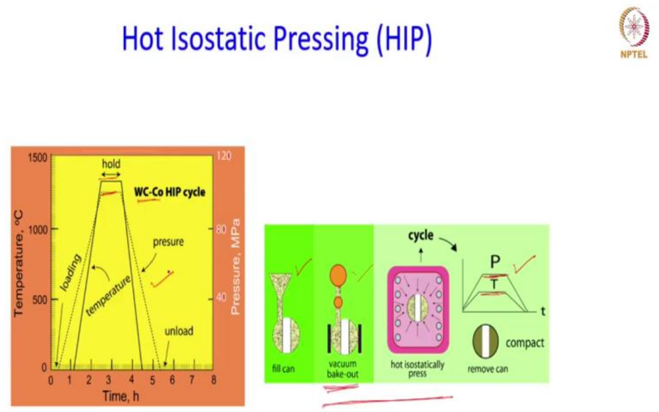

The slide shows the hipping cycle for tungsten carbide, which is a material difficult to process by conventional sintering, because this is a very hard material. The hot isostatic pressing is quite attractive for these kind of materials which are difficult to process by the conventional routes. So, here we can see the loading starts and simultaneously the heating is also done. So, as the compact reaches the sintering temperature and held the pressure is also held at a particular value depending on the type of material and once the sintering is over and the compact is cooled down the pressure is also released.

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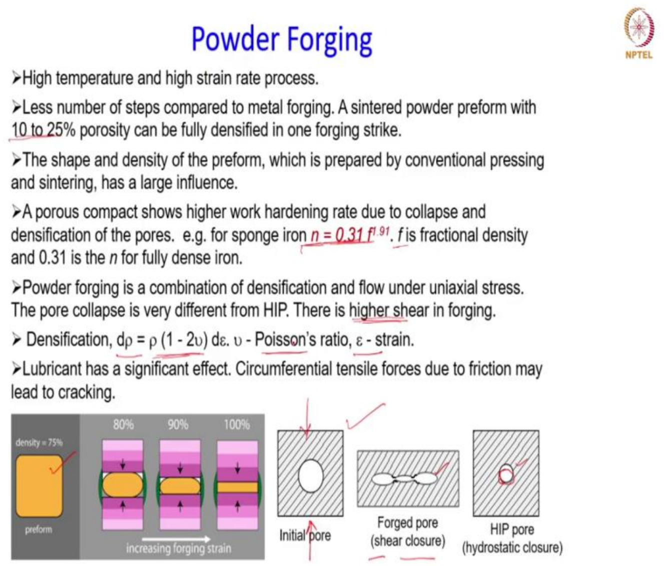

Let us talk about one more processing technique, powder forging. It is a high temperature and high strain rate process and it has less number of steps compared to metal forging.

In forging a metal is heated up to a particular temperature and then it is hammered or deformed to give it a particular shape and it can also modify the microstructure of the material. So, in this process of powder forging a sintered powder preform with about 10 to 25 percent porosity can be fully densified in one forging strike.

So, in case of metal forgings the heated metal is struck several times by hammer like blow. A typical example of the metal forging process can be seen in a blacksmith shop where a heated metal is subjected to repeated blows of a hammer to give it a particular shape, but in powder forging the full densification can be achieved in just one forging strike.

The shape and density of the preform, which is prepared by conventional pressing and sintering, has a huge influence on the densification process. In the first step, preform is formed. This is done by the conventional compaction processes where the powder is pressed in a die and given a particular shape and then it is sintered to reduce the porosity level. When it comes for the powder forging process, the porosity is only about 10 to 25 percent.

So, now, if this compact having certain amount of porosity is subjected to forging operation in the forging die, the densification can be achieved in just one forging strike. So, here is a schematic of the process (slide above).

The preform is subjected to the forging operation by loading the compact into a forging die. As the pressure is applied in the forging die and the compact takes up the shape of the die cavity, the densification also occurs and finally, at the end of the forging operation a hundred percent or full density is achieved.

A porous compact shows a higher work hardening rate due to the collapsing of the pores and the associated densification which occurs. For example, the strain hardening exponent (n) for sponge iron can be written as a function of the fractional density f, $n=0.31f^{1.91}$. So, as the densification is achieved due to the collapsing of the pores, the strain hardening also increases which you can see from this equation, where 0.31 is the value of the strain hardening exponent for fully dense iron.

Powder forging is a combination of both densification and flow under a uniaxial stress. As the stress is applied the pores will collapse. And the pore collapsing is very different from what you have in the hipping process because in case of the forging operation the compact is subjected to higher amount of shear and because of that the collapsing of the pore as to how the pores are closed is very different from the hipping operation.

And this is a schematic (bottom right image in above slide) which shows the collapsing of the pores as to how the pores get closed as the compact is being forged. So, let us say this is the initial pore having this kind of spherical morphology and as the compact is forged and it is pressed along this axis. Then the pores will become elongated which is known as the shear closure because of the higher shear that the forging operation provides and as a result of that, they will be closed in this manner compared to the hipping process where the pore closure or the shrinking of the pore happens in a uniform manner.

The densification in the powder forging process relates to the strain ε through the equation, $d\rho = \rho(1 - 2\upsilon)d\varepsilon$, where υ is the poissons ratio and ρ is the density. The density and the poissons ratio are both functions of the radial strain. Lubrication has a significant effect in the powder forging process, because without the lubricant, the forged powder shows low density because of the frictional drag that the punch and the die wall provide on the compact when it is being pressed and that in turn limits the densification.

So, therefore, if it is not lubricated a lower density is obtained in the forged compact. And the friction can also cause circumferential tensile forces and that may even lead to cracking up the compact. It can be visualized from a diagram in the slide below.

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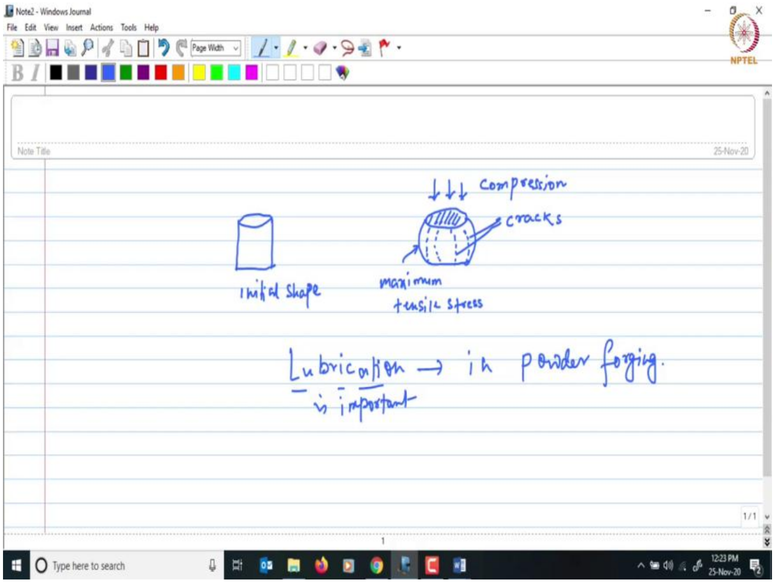

So, let us consider an initial compact having a cylindrical geometry like this and this is being forged for consolidation to densify it. So, when it is forged under compression inside a die, then along the circumference, the tensile stress will be maximum and this may lead to cracking if there is drag due to friction. Therefore, lubrication plays an important part in powder forging. So, these were some of the processes used for full density processing.

So, before we wind up this class let us take a moment to summarize it. So, to summarize today's class, we have started by looking at the hot consolidation mechanisms again and we have seen when the applied stress is high, the densification occurs by the plastic flow when the applied stress exceeds the yield strength of the material.

Otherwise, when the stresses are on the lower side, it is a combination of the diffusional flow and the applied stress, which leads to a creep type of densification process and the diffusion which occurs can be through either the lattice or through the grain boundary. So, depending on that a particular mechanism of creep will be operative during the densification process.

So, in this class we primarily talked about the different methods of hot consolidation for achieving full density. We started with hot pressing. And we have talked about uniaxial hot pressing operation which is very similar to the uniaxial die pressing that we have talked about before, but in this case heat is also applied simultaneously for better densification when the powder is being pressed in the die.

Then, we talked about hot isostatic pressing also which is the hot counterpart of cold isostatic pressing. So, here the only difference with the cold isostatic pressing is the application of heat and in order to accommodate that heating a container which is made of a material which can sustain high temperature is used.

And the pressurization can be done by a gas pressure using argon or nitrogen. And in another approach the pressure can also be applied by volatilization of liquid which leads to a higher pressurization rate and the densification in that case occurs by plastic flow instead of creep.

Then we talked about the hipping cycle also as to how the pressure and the temperature are applied during the process. And finally, we talked about powder forging process which is to subject a powder compact which is already preformed to the forging operation and here you know the densification can be achieved in a single strike of the forging die.

And we have seen that the pore closure which happens in this case is quite different from the hipping process, in the hipping process it is basically a uniform closure of the pore because the pressure is applied uniformly and here you can see the pressure is applied along a particular axis.

And therefore, the pore is subjected to more shear and the pore closure therefore, is quite different from the hipping process which exhibits uniform shrinkage as you could see from this picture over here. And with that we come to the end of this class.

Thank you for your attention.