Powder Metallurgy
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Lecture - 49
Sintering – 9

Hello and welcome back again to this lecture series on Powder Metallurgy; right now it is rather a lecture series on Sintering. So, we have been discussing about this process which is one of the most important steps of the powder metallurgy process in past few classes. And, so far we have already learned about the basic fundamental mechanism behind sintering, then we have also discussed about the different stages of sintering right.

So, you have seen that the sintering process goes through three stages; the initial stage, the intermediate stage and the final stage. So, all these stages we have discussed in detail and we have also learned about the different mechanisms of mass transfer that occurs during the sintering process leading to the densification.

So, we have seen that you know there are different well defined paths for the mass transport to occur. And, the neck which forms at the beginning of the sintering process that starts to grow when the mass flows to the neck. And, ultimately the pores which are left behind are all eliminated and the compact is fully densified.

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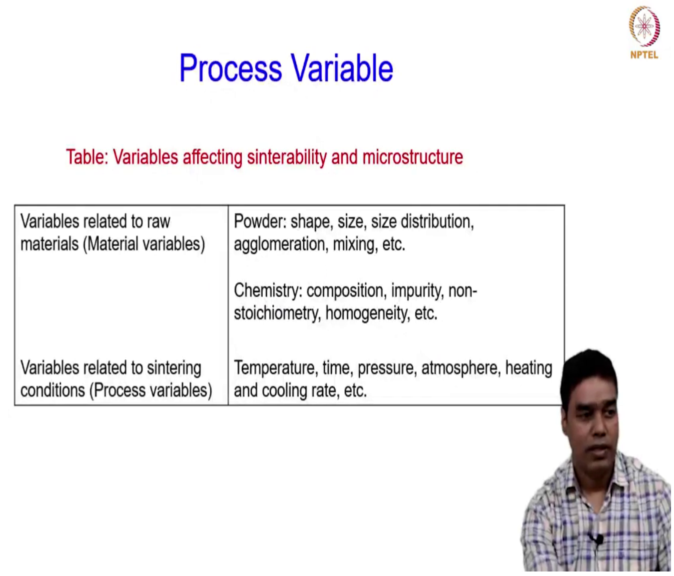

Then in the last class we also discussed about the process variables and here we have seen that there are two kinds of variables; one related to the raw materials like the powder shape, size and size distribution etcetera.

And other process variables of course, are related to the sintering process itself . So, now, apart from these process variables there are certain other things which can influence the sintering process and that is what we are going to discuss in this particular class.

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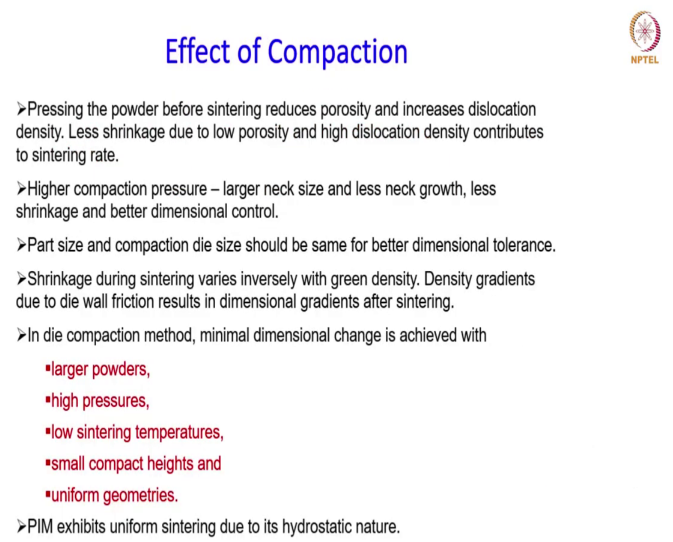

The compaction process has great bearing on the sintering process because the green compact which is being actually sintered comes out from the compaction process. And, we have seen that the pressing of the powder that is what is being done in the compaction process and this pressing operation before the sintering process actually reduces the porosity and increases the dislocation density .

So, both of this aspects are actually helpful for the sintering process because, if the porosity is low then you can also expect less shrinkage to happen. And, high dislocation density on the other hand contributes to the sintering rate by promoting plastic flow right, because in metals the plastic deformation happens due to the motion of dislocations.

So, if you have high dislocation density it will promote more plastic flow and the mass transfer which happens due to plastic flow will be enhanced. And, due to that it will actually be helpful towards the densification which happens during sintering. Now, if you talk about the compaction pressure it has a lot to do with the densification.

When you have high compaction pressure it will lead to larger neck size and less neck growth. And, it will also lead to less shrinkage and hence better dimensional control right. So, as we have discussed before during sintering apart from densification some dimensional changes will also occur because, densification is always associated with shrinkage as we have discussed.

And, due to that in the final sintered compact there will be some changes in the dimension because of this shrinkage. So, therefore, dimensional control is also a very important aspect that has to be considered while parts are being sintered. Now, particle size and compaction die size should be same for better dimensional tolerances.

So, here we are talking about let us say a neck shape or near neck shape kind of part. So, in that kind of product we expect that when the part comes out from the sintering process, it should have the dimensional which are actually needed in the final product right.

So, therefore, when the powder is being compacted you know the dimensional tolerance is have to be considered seriously and the die size has to be accordingly decided for the compaction process right. Now, this shrinkage that happens during sintering that actually varies inversely with the green density. So, that is why green density is such a important factor in the sintering process right.

And, we have also seen that the green density will actually depend on the pressure which is being applied during the compaction process and its uniformity. So, we have discussed before while talking about the compaction process that pressure gradients may occur during die compaction process. And, pressure gradients will lead to density gradients which is not at all desirable as far as the sintering process is concerned.

Because as I said the shrinkage depends on the green density; so, therefore, the dimensional tolerances will also depend on the green density. If the green density is high you can expect less shrinkage and vice versa and therefore, gradients in the green density will lead to different dimensions across the compact.

And, it will be difficult to maintain the dimensional tolerances in the sintered component. So, as far as the die compaction process is concerned these are the things which can be done to minimize the dimensional change.

And these are as follows, if you have large powders you can expect a better dimensional control because, with large powder the expected change in the dimension is minimum. High compaction pressure will again lead to better densification during the compaction process and the density gradients can be minimized. And therefore, this is also one factor which can lead to a better dimensional control during the sintering process.

And, we have also seen that the h by d ratio is very important factor during the die compaction process. And, a low h by d ratio is always better for uniform densification during compaction, low h by d ratio also gives you a lower density gradient or a more uniform green density. And therefore, small compact heights are good for dimensional control and of course, uniform geometries will always be better as far as the dimensional control is concerned.

But, some of this factors are also detrimental for the die compaction process itself particularly the tooling which are used for example, a high pressure would also lead to high tool wear right. So, maintaining all of this together during the die compaction process may be difficult.

And therefore, we have seen that other shaping processes are also used to have better control on the dimension particularly for parts which have complex geometries.

Powder injection moulding is one such technique which can handle complex geometries and can give rise to better dimensional control because; in this case the pressurization is hydrostatic in nature.

So, it is more uniform and therefore, the density in the compact is also uniform and therefore, a uniform dimension is also expected. And, when this kind of parts are sintered, the shrinkage will also be isotropic. And, hence a better dimensional control will be achieved for parts which are compacted by powder injection moulding.

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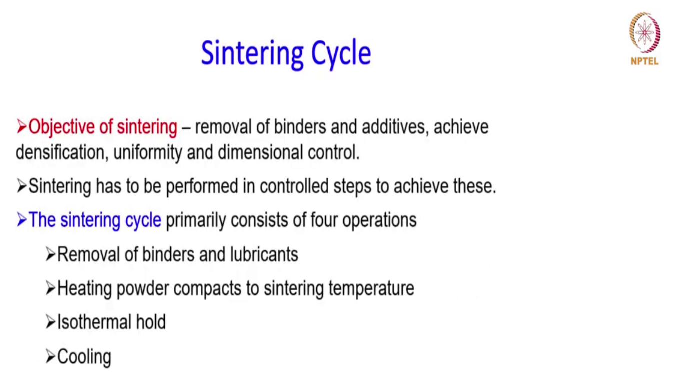

Now, sintering is always not about heating it to a particular temperature, but it is basically a sintering cycle kind of process wherein you heat the component at a particular heating rate, hold it at the sintering temperature for a certain period of time and then again cool it down in a controlled manner. So, it is a heating cycle which is to be considered as a whole when you talk about the sintering process as such .

So, the primary objective of the sintering is to remove the binders and additives and achieve densification and also maintain uniformity and dimensional control right. So, all this can be achieved only when the heating is done in a proper manner with controlled heating rate and things like that right. So, therefore, we also need to see how the sintering cycle is maintained.

And, you know what are the parameters and factors that one needs to take care in order to make sure that the heating process is uniform and you achieve all these objectives during the sintering process. So, as far as the sintering cycle is concerned, it primarily consists of four operations; first removal of binders and lubricants, then heating of the powder compact to the sintering temperature, isothermal hold at that temperature and finally, cooling right.

We have seen that the powder is often mixed with binder and lubricants to aid the compaction process. And, these are some kind of volatile matters and they have to be removed for you know better densification during the sintering process. So, if these things are not removed before you heat the compact to the sintering temperature, this will actually lead to porosity in the compact right.

Because, this are volatile matter when they evaporate out of the compact they will leave behind pores. So, therefore, before the compact is actually heating to the sintering temperature you have to first heat it to a lower temperature and hold it there for certain amount of time to remove the binder and the lubricants right. Then only it is heated to the actually sintering temperature right.

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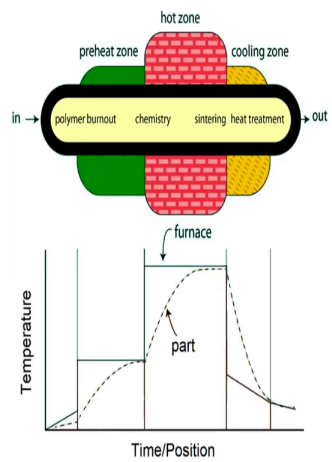
Sintering Cycle



So, this is what is shown in this diagram also. So, if you see the temperature time cycle of the sintering process, it is not a monotonous heating all the way to the sintering temperature. There are stages because; there are things which need to be removed before the sintering happens . So, you need to first heat it to a lower temperature which is you know the preheat zone where you burnout this binders and lubricants.

So, that is the polymer burnout stage of this heating process and then if you have chemistry to be maintained; that means, if you have pre alloyed powder or if you have mixed powder which you are expecting to you know dissolve into each other and form an alloy, then the powder again has to be heated to certain temperature and held there for this alloying to happen and for the chemistry to be maintained right.

So, once that happens then again it is heated to the sintering temperature and the compact is sintered. So, the powder chemistry or the powder character also has to be considered when you decide the sintering cycle in terms of the temperature and the time. Now, there could be also possibilities that post sintering heat treatment may be needed for some material.

So, in those cases before the compact is taken out from the sintering furnace it is again subjected to a lower temperature for a certain period of time for the heat treatment process to take place. And, only at the end of this heat treatment process, the compact is taken out right. So, this side is basically the cooling zone right, but as I said before cooling this can be again held at a lower temperature if there is a need .

And, this is the hot zone where the compact is actually being sintered. So, in this zone the temperature is higher compared to any other zone on either side of it. So, here the compact is kept at a higher temperature for the sintering to take place and this is again demonstrated with the help of a plot like this. So, you can see in the beginning it is heated slowly right. So, that you provide enough time for any moisture and things to simply evaporate out.

And, then it is gradually heated to a temperature where this polymer burn out can take place and the binder and the lubricants can be removed. And, only after this burnout period the compact is finally, heated to the sintering temperature and there again you can see ,a particular heating rate is used to heat the compact to the final temperature. And, this is also necessary as far as the dimensional control is concerned.

Because, a rapid heating can lead to stresses which might affect the dimensional control and might even also lead to cracking and things like that . So, therefore, the heating has to be controlled, it is not simply heating the compact to certain temperature and you know simply leave it there . So, what you can see over here with the help of these curves and their slopes that the heating process is happening in a controlled manner .

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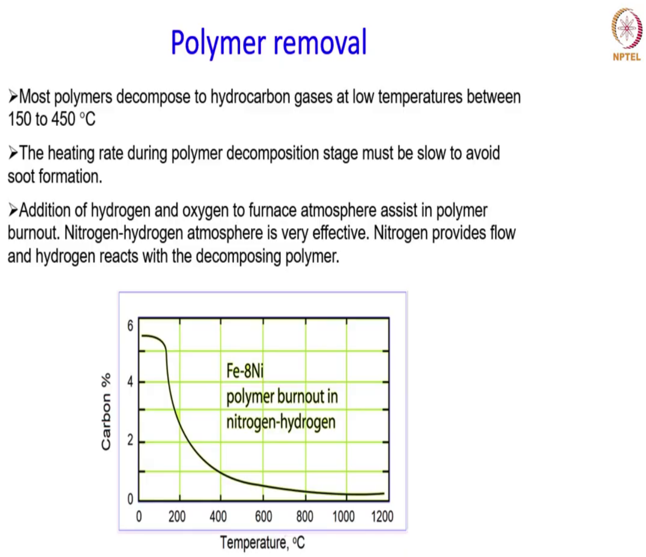

So, let us talk little more about the polymer removal which happens before the sintering. So, most polymers will decompose to hydrocarbon gases at low temperatures between 150 to 450 degree Celsius depending on you know what kind of polymeric materials you have in that binder. And, the heating rate during the polymer decomposition stage must be slow to avoid soot formation right.

So, this is another reason why the heating has to be in a controlled manner; here if it is not slow then soot will form. And, this is again not desirable at all as far as the compact is concerned. Addition of hydrogen and oxygen to the furnace atmosphere assists in polymer burnout. Nitrogen-hydrogen atmosphere is also very effective because nitrogen provides flow and hydrogen reacts with the decomposing polymer right.

So, some kind of atmospheres are also used inside the sintering furnace to make sure that the polymer burnout happens effectively. And, all the polymeric components which are present in the compact are completely removed before the compact is heated to the sintering temperature. So, you can see here is a plot which shows how the polymeric burnout happens in a nitrogen hydrogen atmosphere for a iron nickel alloy.

So, the polymer will have a particular carbon content and you see as the temperature is increased this carbon content is decreasing. And then finally, it kind of levels off which indicates that you know at this particular temperature where it is levelling off almost all of the polymer has been removed because, there is no more reduction in the carbon content. So, depending on the kind of polymers which are used the temperature of the burnout has to be decided.

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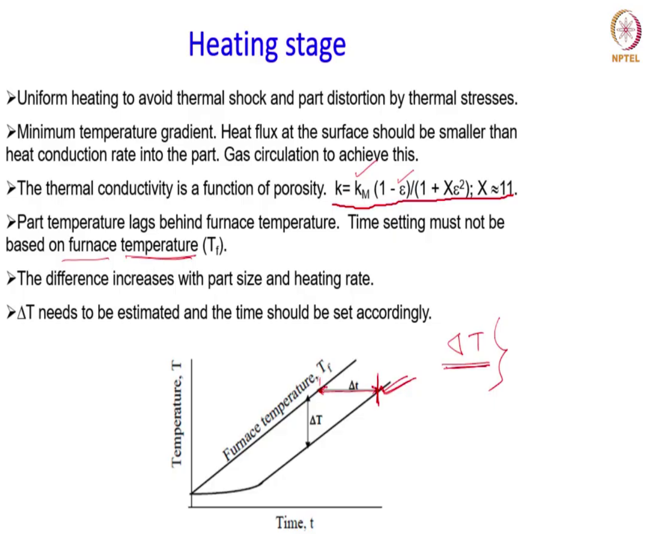

Now, if you talk about the heating stage and how it is actually being controlled, first of all as I said uniform heating is needed to avoid thermal shock and part distortion by thermal stresses. And, you also had to ensure that the temperature gradient is minimum. But, one thing that you might know is that the heat flux at the surface may be different from the heat flux in the core of the material at higher depth from the surface right.

So, in order to achieve a minimum temperature gradient, the heat flux at the surface should be smaller than the heat conduction rate into the part. And therefore, the heat has to be removed from the surface so that the conduction along the compact is better. And therefore, many a times gas circulation is used in the sintering furnace to achieve this. This will actually remove the heat flux from the surface and bring it down.

Therefore, the heat conduction rate into the part will be better and you can expect a better temperature distribution or minimum temperature gradient. Now, the thermal conductivity for this heat conduction to happen across the compact is a function of porosity right. And, that is given with an equation like this,

$$\mathbf{k = k_M\ (1 - \varepsilon)\ (1 + X\varepsilon^{2})}$$

here k_M is the thermal conductivity of the material, ε is the porosity and X is the parameter whose value is about 11.

So, that is something that one has to keep in mind because, here the heating happens through the thermal conduction. And, when you consider this conduction heating you also have to remember that the part temperature will lag behind the furnace temperature because, here the compact is kept inside the furnace and it is being heated by conduction right. So, therefore, it will always need some time for the temperature first of all to equilibrate with the atmosphere around it.

And, also the actual temperature of the part will be somewhat lower compared to the furnace temperature so, right. So, this is a very important aspect that one has to keep in mind while deciding the sintering temperature. If you simply look at the furnace temperature, that is not the actual sintering temperature of the compact because the actual temperature of the compact will be always somewhat lower compared to the furnace temperature.

And therefore, this ΔT that you have, this difference has to be noted and the actual temperature that you set in the furnace has to be accordingly decided . And, the time which is calculated it has to be basis that temperature after considering the delta T factor. So, what is done as we have seen you heat the compact to the sintering temperature and then you hold it there for a certain period of time.

So, this counting of the time which should start when the temperature comes to this actual temperature, that you need in the compact right and it should not be based upon the furnace temperature; because it is going to be different from the actual temperature of the compact. So, the basis for the time calculation should be the actual temperature once you provide this ΔT and once this temperature is reached, the time should be calculated from there on.

So, the first thing when you decide the temperature for the sintering you need to know what is the ΔT. So, actual measurements has to be done first before the sintering is actually carried out. The actual compact temperature and the corresponding furnace temperature has to be measured and this ΔT has to be calculated right. So, whenever you sinter the compact this ΔT has to be considered and the furnace temperature has to be set accordingly.

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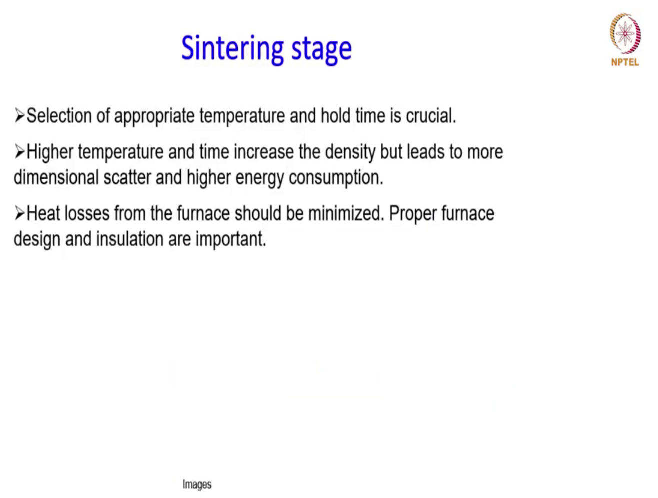

So, the two crucial things here for the sintering process are the selection of the appropriate temperature and the hold time . It is not about only the temperature, but the sintering time is also important as we had discussed before. Higher temperature and time increase the density, but it will also lead to more dimensional scatter and higher energy consumption . And, during the sintering process the heat losses from the furnace also should be minimized.

So, that the uniformity of the temperature can be maintained all along the entire sintering period and therefore, a proper furnace design and insulation are also important. So, this is how the sintering cycle has to be carried out considering all this factors. And, it should not be just heating the compact to a particular temperature and holding it over there. And, only when all these steps are followed in a proper manner, a fully dense compact with proper dimensional tolerances will be obtained at the end of the sintering process.

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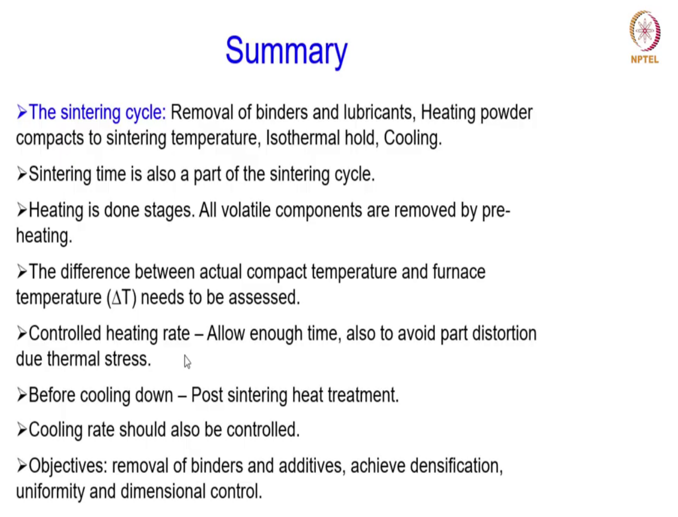

So, before wind up let us take a moment to summarize this lecture. So, today in this lecture we have primarily talked about the sintering cycle that is the heating and the cooling process involved in sintering. So, the sintering cycle involves these basic steps that is removal of binders and lubricants, heating the powder compact to the sintering temperature, isothermal holding at that particular temperature.

And, then when the sintering is done; cooling at the end of the process . But, apart from the temperature, the sintering time is also a part of the sintering cycle because as we have seen before , the sintering time also plays a role in the densification process which occurs during sintering. And, now as far as the heating is concerned it is done in stages because, there are things which are to be removed before the compact is actually sintered.

So, all the volatile component which are present in the compact had to be removed by preheating at the lower temperature. And, only when all these are removed it is taken to the sintering temperature for the actual densification to occur. And, when you talk about setting the temperature and the sintering time, the difference between the actual compact temperature and the furnace temperature that is delta T has to be assessed. And, the temperature and time had to be set after taking due consideration of this difference .

And, the heating is not done arbitrarily in this case; it has to be done in controlled manner. This will ensure that enough time is allowed for all the volatile matter to go out of the compact and it will also avoid distortion of the part due to thermal stresses. And, before cooling if there is a need for any heat treatment for a particular purpose for example, alloying or things like that , then a post sintering heat treatment can also be provided before the compact is cooled down after sintering .

And, during cooling also it has to be taken care that the cooling rate is controlled and cooling should not happen in a random fashion without any control on the cooling rate . So, apart from the heating rate, the cooling rate also should be controlled and then only you can expect a part which will be free of distortions. Because, controlled heating and cooling rates will ensure that, thermal stresses are not generated in the compact.

So, only when all these things are maintained in a sintering cycle, the objectives of sintering can be fulfilled. And, these are the primary objectives of sintering: removal of binders and additives, achieve densification, uniformity and dimensional control. So, all this objectives will be achieved only when the sintering cycle is maintained properly by taking care of all this aspects that we talked about in this particular lecture. So, with that we come to the end of this lecture.

Thank you for your attention.