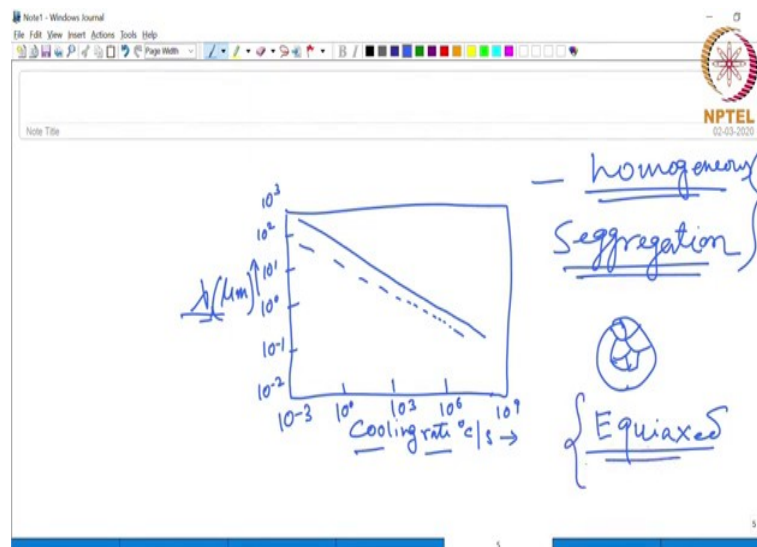


Power Metallurgy
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Lecture - 13
Microstructure control: Effect of process parameters

Hello and welcome back. So, in the previous class, we have seen that the cooling rate during the solidification which takes place in the atomization process.

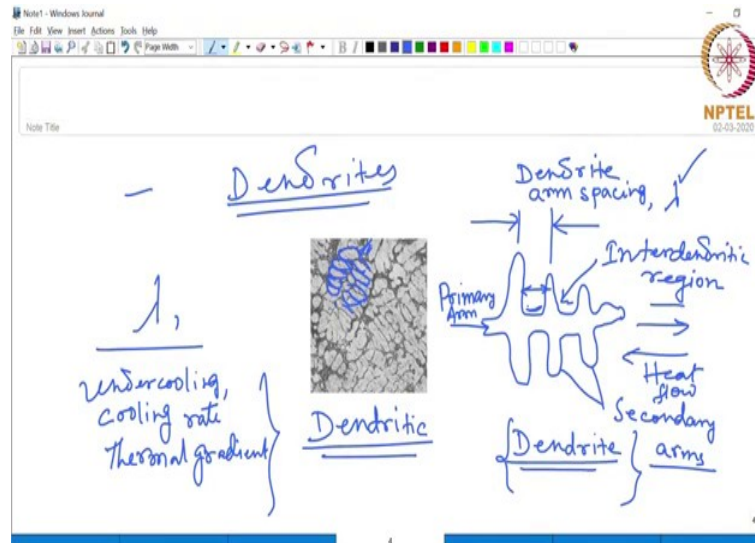
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We will have a great influence on the microstructure of the powder particles. As you increase the cooling rate (graph in the above image), the dendrite arm spacing λ decreases, and the microstructure becomes more and more finer. So, this is what we were discussing as to how these process parameters will play a role on controlling the microstructure of the powder particles which are generated in this atomization process.

Now we will continue on this, and then see how this microstructure can change to other type of microstructure, and how you exercise the microstructure control with the help of this process parameters.

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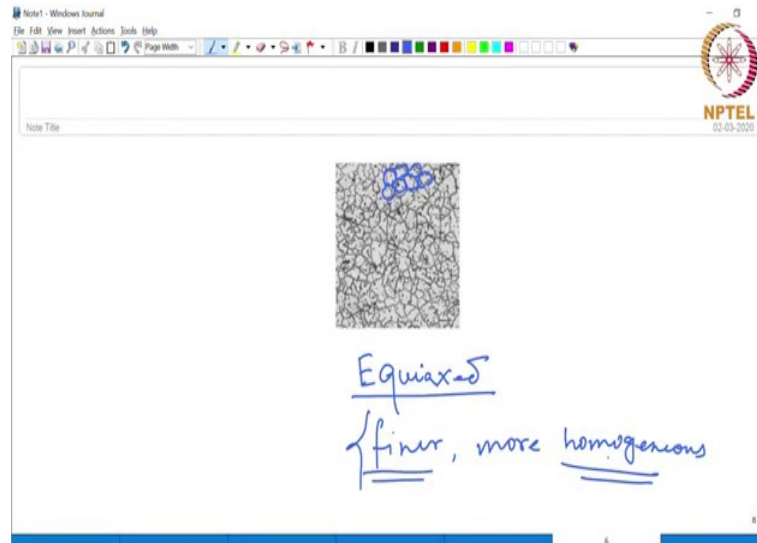
One thing that we have seen is the formation of this dendrites or these dendritic microstructures. And we have also discussed the conditions which leads to the formation of these dendrites. So, if you see it is kind of a directional growth, it is kind of growing in this particular direction because what happens the heat flow direction is on the opposite side.

So, given a chance of growing under normal conditions, this will tend to grow in the opposite side of the heat flow direction. And as a result, you get this kind of structure growing in a particular direction.

If you eliminate the conditions which favor this kind of growth, then you can also eliminate the growth of this kind of dendrites, and you can expect these grains to be globular like how I said because in that case the growth will be random or uniform in all possible directions rather than a particular direction, and that is why these grains will take up this kind of globular morphology and not forming those kind of directional structure like what do you see in a dendrite.

This kind of microstructure is called equiaxed grains or equiaxed microstructure. Let me show you an example of an equiaxed microstructure.

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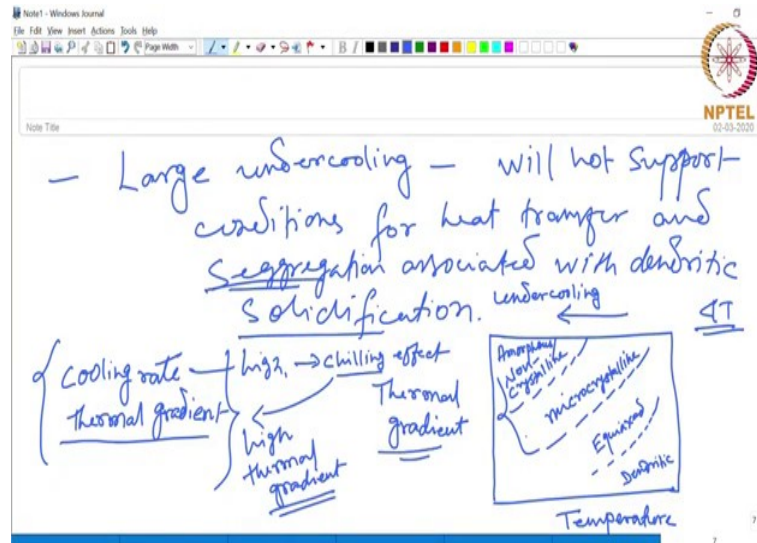
Right now you compare this with the dendrites microstructure. In this case as I said you can see this kind of globular kind of grains which are more uniform in all direction, and there is no preferential direction as such like what do you saw in case of the dendrites.

This kind of structure as I said is known as equiaxed structure. And here the grains are also finer and more homogeneous, because the segregation problem that you had in case of the dendritic structure can be minimized here, since those inter dendritic regions or those dendrite arms themselves are not present in this case.

If you could tune your processing conditions towards the formation of the equiaxed kind of microstructure and also create conditions for higher cooling rate or high thermal gradient or high under cooling that will give rise to a finer microstructure that will be more conducive for generating a homogeneous material as well.

So, our objective is to have this kind of uniform microstructure which will also give you microstructural homogeneity in terms of the chemistry as well.

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If you have conditions which can generate large under cooling, this will not support conditions for heat transfer and segregation associated with the dendritic growth. And as a result you would expect to get equiaxed microstructure and a material which is free of segregation or at least minimum level of segregation.

And this is again can be explained with the help of a graphical representation as well where on the y-axis you have thermal gradient; and on the x-axis, you have temperature. So, this is about the other two parameters that we talked about cooling rate and thermal gradient.

When you have high cooling rate, you have this chilling effect as I said before on the surface of the solidifying particles; and as a result of that, it will generate high thermal gradient. And you can expect to generate the microstructure which is equiaxed. So, if you are on the higher temperature side, then you are in the dendritic region, higher temperature and lower thermal gradient.

And as you increase the thermal gradient, then you can enter the equiaxed region (graph in the above image). So, this is a comparison between the dendritic and the equiaxed microstructure or how a dendritic microstructure can be transformed into equiaxed microstructure by controlling the process parameters like the cooling rate, thermal gradient and under cooling also will have an effect.

As you have higher thermal gradient the microstructure will be equiaxed and finer as well that you can see from here. And as you go towards lower temperature that also means that you have high under cooling. So, here you can also see the interplay among these three parameters – temperature, thermal gradient, and under cooling. As you reduce the temperature more below the equilibrium melting point, the under cooling ΔT is also going to increase which is quite obvious.

And of course, when you are on the lower temperature side, you have this chilling effect lower temperature on the surface, we will have this chilling effect, and that is also going to generate this large thermal gradient. So, this is a transformation between dendritic and equiaxed microstructure.

But when you talk about the material structure the internal structure, there is one more aspect which describes that the internal structure can also be described with that particular aspect of the material, which is whether it is going to be a crystalline material or not.

Most of the metals are crystalline meaning they have a particular crystal structure, that means, the atoms in metals are arranged in a particular order, so that is known as a crystalline structure where you have all the atoms arranged in a given order in a particular pattern.

So, this process conditions, and these parameters that we talked about that would also dictate whether this will be a crystalline material, you can also call it micro-crystalline, because these crystals are very small in size which can only be seen under a microscope, or whether it is going to be a non-crystalline material which is also known as amorphous materials.

The cooling rate or the thermal gradient that would also affect or that would also influence whether the material is going to be a crystalline material like what you generally get in the metals, or it is going to be an amorphous kind of structure like what you get in a glass.

The normal glasses that you see around you, those are all amorphous materials where the atoms do not have any particular pattern in terms of their arrangement. So, whether this

will be a crystalline material or a glassy material, that would also be dictated by these parameters.

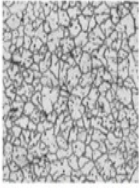

So, it is not only about the size of the grains or the morphology of the grains whether it is going to be dendritic or equiaxed, it is also about whether it is going to be crystalline at all or not. So, that part also comes under this microstructural control. Because going from a crystalline material to a completely amorphous or non-crystalline kind of structure can lead to a dramatic change in the properties of the material.

It is also important for us to understand as to how these parameters will affect this transformer and form a crystalline material to an amorphous material. So, apart from this transition between dendritic to equiaxed kind of microstructure, the process parameters can also have an effect on the transition between crystalline to amorphous.


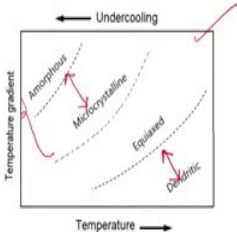
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Microstructure control

- Large undercooling does not support the conditions for heat transfer and segregation associated with dendritic solidification.
- Microstructure shifts to equiaxed.
- Very high cooling rates $> 10^6$ °C/s: Amorphous/Non-crystalline powder.
- Rapid heat extraction chills the surface giving rise to high temperature gradient (higher undercooling).
- Atomic diffusion is needed to arrange atoms in crystals. At normal (equilibrium) cooling rate time is sufficient for atoms to arrange in an order. At high cooling rate not enough time for the atomic sorting process.



Equiaxed microstructure



Which is depicted over here also (second graph on the above image). In this particular diagram that we have discussed before depending on this three parameters temperature, undercooling and thermal gradient, which are actually interrelated to each other. You can see how this transition happens first of all between dendritic and equiaxed microstructure and also between crystalline and amorphous structure.

This is something we will have to take separately to discuss in more detail as to what is meant by this kind of amorphous structure or this crystalline structure, and how do they

change from one to another what are those conditions, which can lead to this transition and so on.

But right now I can briefly tell you as to as far as these process parameters are concerned, what are those conditions which might either favor formation of a microcrystalline or a polycrystalline material, or formation of an amorphous or glassy material. So, this is based upon the cooling rate which will have an influence as to how this solidification will take place or how these atoms will go from the liquid to solid for the solid to grow.

This is something we have seen in the previous classes while discussing about nucleation and growth of the solid from the liquid phase. And as we have seen for the solid to grow, the atoms should get attached to the solid the atoms from the liquid should come to the solid and get attached to it.

There is a transportation of an atoms from the liquid to the solid phase. So, this is also known as atomic diffusion, the movement of these atoms and then building off of this solid as it grows from the liquid phase.

Now, depending on how much time is allowed during this attachment of the atoms to the solid, it might take up either a crystalline form or an amorphous form. For the crystalline form to solidify, a larger time needs to be allowed because a crystalline solid has a particular atomic arrangement.

And for the atoms to get arranged in that particular order or in that particular pattern, a certain time is required, so that will happen only when the liquid is cooled at low cooling rates like what you generally have in conventional casting processes or in conventional equilibrium cooling conditions as we call it.

So, when the cooling rate is low, you have more time for the atoms to get arranged in a particular pattern which will lead to the formation of a crystalline solid. On the other hand, if that time is not allowed for the atoms to get arranged in a particular order, then a the solid which will finally, form may not have that crystalline structure, because we have not allowed that time itself for the atoms to get arranged.

So, that also means that the cooling rate has to be very high for that to occur because when the cooling rate is high it is cooling very fast from the liquid to the solid phase. And as a result during that transformation from the liquid to the solid, the atoms do not get enough time to get arranged in a particular pattern.

If you have cooling rate in this range 10^6 °C/s or more, then it is a high enough cooling rate to prevent that the atomic arrangement. And the solid which will form under this kind of high cooling rate conditions will be amorphous or glassy in nature.


So, cooling rate in that sense is a very important parameter when you talk about this transition between crystalline to amorphous solid during this solidification process which occurs in the atomization process. That is something will be taking up later on and discuss in detail as to how are those conditions are what are those conditions which can either lead to a crystalline material or an amorphous material.

But for today this is all I have. So, before we close today's class let us take a quick summary of what we have learned today.

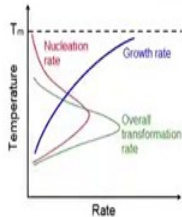
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Nucleation and Growth Kinetics


- Once the embryo exceeds the critical size r^* , the growth of the nucleus starts. Nucleation continues simultaneously.
- Nucleation and growth rates are function of temp. Nucleation rate increases with cooling rate and degree of undercooling ($\Delta T = T_m - T$).
- High nucleation rate and low growth – Finer grain size.
- The over all transformation rate is the product of nucleation and growth rates.



$$\Delta G_v = \Delta G_o \Delta T$$




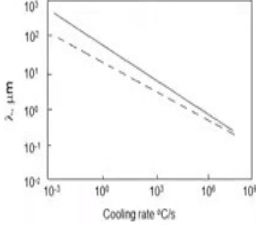
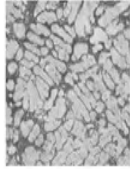
$\Delta T \propto$ Nucleation rate




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Microstructure control

- High cooling rate and undercooling: Fine powder particles.
- Cooling rate: Conventional casting < 100 °C/s. Atomized powder (high surface area/volume ratio) $10^3 - 10^4$ °C/s.
- Castings cooled at slow cooling rate (equilibrium) – Coarse dendrites with interdendritic segregation.
- Higher cooling rates – finer (lower arm spacing, λ) and more uniform structure, improved homogeneity (less segregation).





Today we talked about this important aspect microstructural control in case of the atomization process. And what we have seen is that the kinetics of the process or the kinetics of this nucleation and growth that is a very important aspect which is going to affect the microstructure. And here we saw that a high nucleation rate will give rise to a finer a more refined microstructure.

So, any process condition that leads to a high nucleation rate will give rise to a fine microstructure, that is one way of looking at it as to how to control the process parameters to tune the microstructure.

Then we had seen what kind of the microstructures are obtained depending on the cooling rate and the under cooling. If you have high cooling rate and high under cooling, you can expect to get fine powder particles and also refined microstructure finer or finer microstructure.

Cooling rate if you consider the conventional casting process is quite low, 100 °C or less. On the other hand, these atomized powders due to their high surface area to volume ratio can generate high cooling rates. So, this is a conducive condition, this is good for generating powder particles having refined microstructure.

And as far as the microstructural features are concerned, we have seen that you get this kind of dendritic structure or these dendrites when the cooling rate is slow the cooling

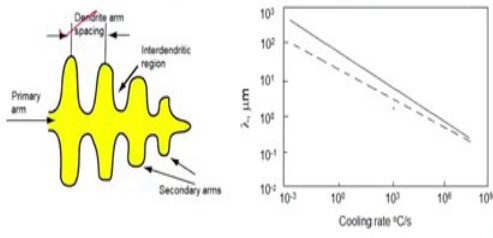
rate is low like what you get in equilibrium conditions which generally prevail in conventional casting processes.

So, that we will generate this kind of coarse dendrites and it will also have this interdendritic segregation inside. If you increase the cooling rate and the undercooling, you can see the microstructure is refined.



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Microstructure control

- High cooling rate and undercooling: Fine powder particles.
- Cooling rate: Conventional casting <100 °C/s. Atomized powder (high surface area/volume ratio) $10^3 - 10^4$ °C/s.
- Castings cooled at slow cooling rate (equilibrium) – Coarse dendrites with interdendritic segregation.
- Higher cooling rates – finer (lower arm spacing, λ) and more uniform structure, improved homogeneity (less segregation).



The diagram on the left shows a yellow dendrite with a primary arm, secondary arms, and interdendritic regions. The log-log plot on the right shows the relationship between arm spacing (λ , in μm) and cooling rate (in °C/s). The y-axis ranges from 10^{-2} to 10^2 and the x-axis from 10^{-2} to 10^4 . Two lines are shown: a solid line for conventional casting and a dashed line for atomized powder, both showing a decrease in arm spacing as the cooling rate increases.



And you can expect to generate this kind of equiaxed microstructure where the grains are more uniform or globular in terms of their morphology rather than like those dendrites which grow in a particular direction. And then we saw how the control over this parameter under cooling temperature gradient and the cooling rate how that can be represented the graphically with the help of this kind of plots.

And this again talks about the interplay between these parameters in controlling the microstructure of the powder which is generated in this process of atomization. So, with that I will conclude today's class. I will see you again in the next class.

Thank you for your attention.