Power Metallurgy
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Lecture - 19
Effect of particle size on microstructure

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Hello everyone and welcome back, hope you all are doing good. So far we have been discussing about this microstructure control in the atomization process. And this will be the last class on this topic. So, what we have seen so far is that the process parameters can be tuned to tune the microstructure. You can control the process parameter to change the microstructure, for example, from a dendritic microstructure to an equiaxed microstructure and also to make it finer.

And we have also seen you know those parameters as to how they actually affect this process of solidification, and influence the final microstructure which is obtained in the solidified powder . And in the last couple of classes, we have been discussing about this amorphous versus crystalline structure as to what could be those conditions which can prevent formation of a crystalline solid and make it into a classy or amorphous solid right.

And we have seen that the cooling rate has a huge influence on that part. And the effect of the cooling rate we have understood with the help of this concept or this diagram known as Time Temperature Transformation diagram or T-T-T diagram. And we have understood that if we have a high enough cooling rate which can avoid the nose of the c curve of this T-T-T diagram, then that would lead to formation of an amorphous solid once the glass formation temperature is reached .

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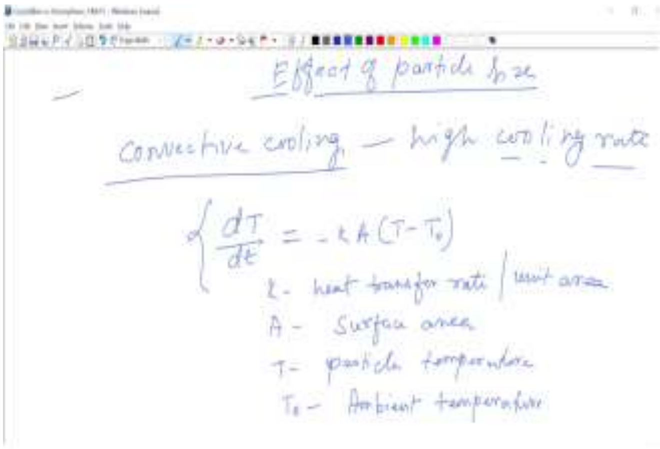

Effect of particle size

Convective cooling — high cooling rate

$$\frac{dT}{dt} = -kA(T-T_0)$$

k - heat transfer rate / unit area
A - Surface area
T - particle temperature
T_0 - Ambient temperature

Now, there is one more aspect to it as far as the cooling rate goes in this atomization process, and that is the effect of particle size. Because depending on the size of the particle, the cooling rate can vary.

Generally, a finer particle would lead to a higher cooling rate. And how is that? That is what we are going to discuss right now. And as far as the atomization process is concerned as such the nature of the process is such that it can also provide a higher cooling rate compared to many other conventional processes such as casting . Because in this case what happens there is what is known as convective cooling which generates high cooling rates.

Now, generally, if you talk about the cooling rate or the change of temperature with time dT by dt, where capital T is the temperature, and small t is the time that can be written in a general form like this. Where k is the heat transfer rate per unit area, A is the surface area; T is the temperature of the material. So, in this case, if you talk about the solidifying particles that is the temperature of the particle and the T_0 is the ambient temperature. So, this is generic form of the heat transfer rate or the cooling rate.

$$\mathbf{dT/dt = - kA (T-T_0)}$$

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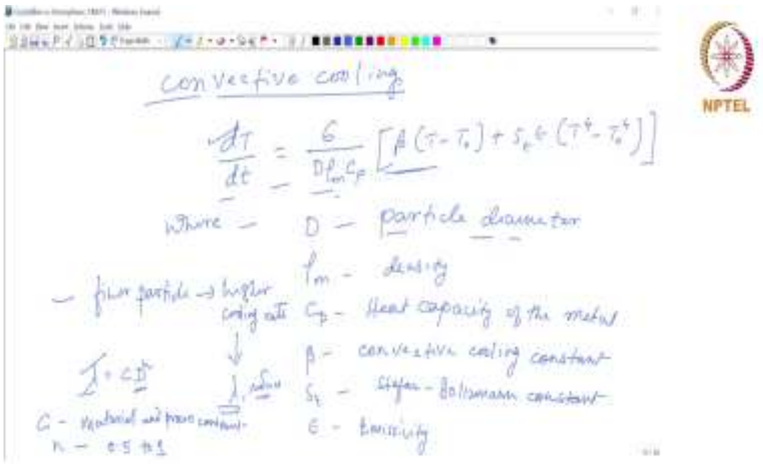

convective cooling

$$\frac{dT}{dt} = \frac{6}{D\rho_m C_p}\left[\beta(T-T_0) + S_t\epsilon(T^4-T_0^4)\right]$$

where — D — particle diameter

ρ_m - density

C_p - Heat capacity of the metal

β - convective cooling constant

S_t - Stefan-Boltzmann constant

ϵ - emissivity

— finer particle → higher cooling rate ↓ λ reduce

$\lambda = C D^n$

C - material and process constant

n - 0.5 to 1

But in the atomization process, as I said there is convective cooling and the cooling rate for this convective cooling process is given as this. Where D is the particle size or particle diameter, ρ_m is density of the metal, Cp is the heat capacity of the metal, β is the convective cooling constant, S_t is Stefan-Boltzmann constant and ε is emissivity right.

$$\mathbf{dT/dt = \frac{6}{(D \rho_m Cp)} [\beta(T-T_0) + S_t t(T^4-T_0^4)}$$

So, this is about the convective cooling process which takes place in the atomization process. And here you can see the effect of the particle size as I was mentioning in the beginning, as the particle size reduces the cooling rate increases as you can see from this particular equation . So, that is what I said that finer the particle, higher will be the cooling rate. And higher cooling rate gives rise to a lower dendritic arm spacing.

If you remember, this is something that we have discussed before; λ is the dendrite arm spacing. And as the cooling rate increases λ will reduce giving rise to a finer microstructure . And this can also be seen from this particular relationship again which describes the relationship between λ and the particle size D . Here C is a material and process dependent constant. And the exponent n varies between 0.5 to 1 .

$$\lambda = C D^n$$

So, here also you can see that lower is the particle size or finer is the particle, lower will be the λ or the dendrite arm spacing. And as a result the finer will be the microstructure of the powder particles which are generated by the atomization process . And the relationship between cooling rate and the particle size which is described by this equation can also be shown by this graph which is a plot between the cooling rate and particle size.

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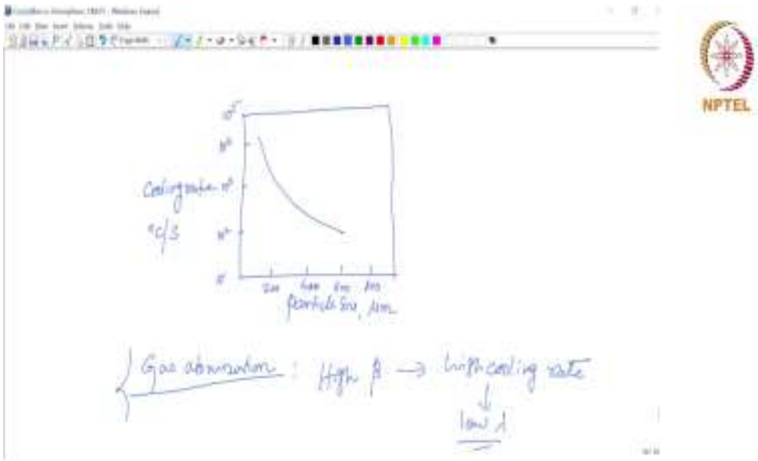

So, as we can see from that equation before as the particle size increases, the cooling rate decreases in a manner like this right. So, the correlation between the particle size and cooling rate can also be represented graphically right like this with the help of a plot like this.

And as far as the gas atomization process is concerned, the convective cooling rate that it offers will have a high beta which generates high cooling rate which we can see again from this equation.

And this will give rise to a low λ or a finer microstructure in the solidified particles which are obtained at the end of the gas atomization process . So, with that, we can conclude this particular topic and the microstructure control topic that we have been discussing in past several classes. But before we finish off today’s class like how we generally do, let us try and summarize this.

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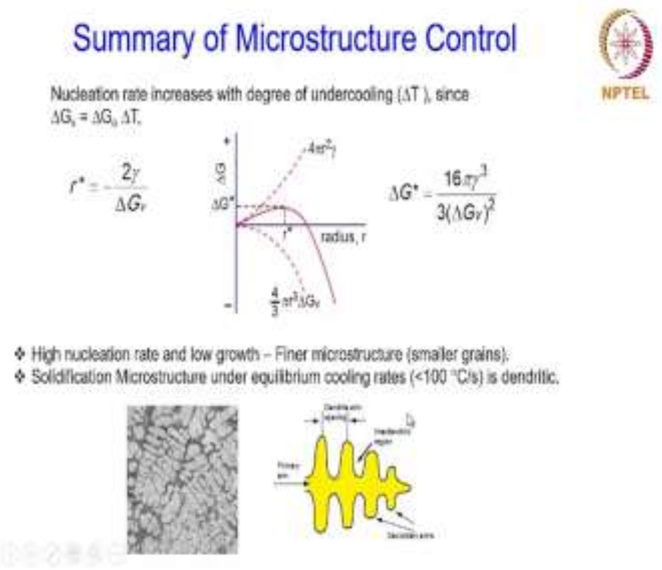

Summary of Microstructure Control

Nucleation rate increases with degree of undercooling (ΔT), since $\Delta G_v = \Delta G_s \Delta T$.

$$r^* = -\frac{2\gamma}{\Delta G_v}$$

$$\Delta G^* = \frac{16\pi\gamma^3}{3(\Delta G_v)^2}$$

- High nucleation rate and low growth – Finer microstructure (smaller grains).
- Solidification Microstructure under equilibrium cooling rates (<100 °C/s) is dendritic.

So, here is the summary of microstructure control. It all started with the nucleation and growth of solid from the liquid. And from this free energy diagram for that particular transformation between the liquid and the solid, this parameter called the critical radius of the nucleus was derived. And only when the nucleus which is the first particle of the solid that forms reaches this critical size it becomes stable and the solid will start to grow .

Now, once the solid has grown and the solidification process is completed, the final microstructure of the solid will depend on the process parameters. And in order to understand that we also need to look at the kinetics of the process as to what was the nucleation rate and what is the growth rate . If the nucleation rate is high and the growth rate is low, that will give rise to a finer microstructure.

For example, if the microstructure is characterized by the grain size, it will lead to smaller grains . The effect of process parameter on the nucleation rate can be understood from this particular equation over here which is a relationship between the volume free energy change **ΔGv** and the undercooling **ΔT** ok.

So, here you can see that the volume free energy change is directly proportional to the undercooling. And now if you come to this particular relationship over here for the critical size of the nucleus, you see that the critical size decreases as the **ΔGv** increases. Similarly, if you look at this critical free energy change for the nucleation to start, here also you can see that the critical free energy change **ΔG*** also decreases with increase in **ΔGv** . So, this means that the nucleation process becomes easier as **ΔGv** increases . And **ΔGv** would increase when **ΔT** increases.

So, this suggests that a high undercooling condition would lead to a finer microstructure . And we also know that a higher cooling rate promotes high under cooling. So, in other words, we can also say that a high cooling rate would lead to a high nucleation rate, and that would in turn lead to a finer microstructure .

Now, if you talk about the solidification microstructure which is formed under equilibrium cooling rates that is cooling rates in this range 100^0 Celsius per second or lower, then the microstructure is typically a dendritic kind of microstructure what you can see over here . And this is what we discussed in detail.

And we had also seen the different features of the dendrites including this dendrite arm spacing , λ which gives the size of the dendrites . So, this dendrite arm spacing , λ is a parameter which can be used to characterize the microstructure in terms of whether it is a coarse or a fine microstructure .

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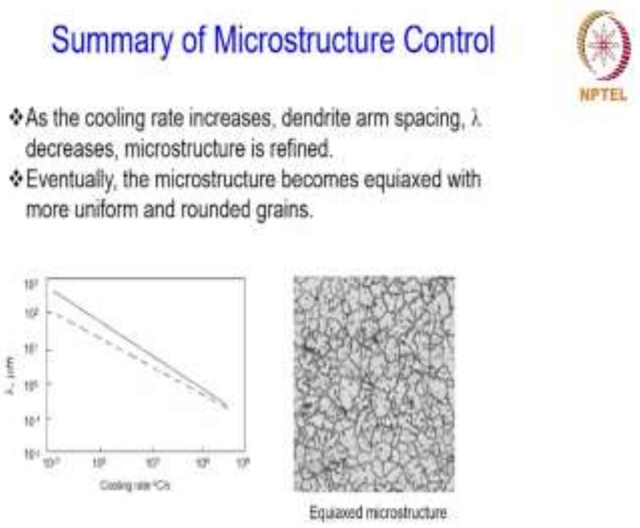

Summary of Microstructure Control

- As the cooling rate increases, dendrite arm spacing, λ decreases, microstructure is refined.
- Eventually, the microstructure becomes equiaxed with more uniform and rounded grains.

Equiaxed microstructure

So, as the cooling rate increases, this dendrite arm spacing λ will decrease. And as a result the microstructure is refined. And this is something that can be seen from this particular plot over here which shows the relationship between λ and the cooling rate. So, here also you can see that as the cooling rate increases, λ decreases right. So, a high cooling rate will give rise to a finer microstructure as I said before also. And eventually under the high cooling rate conditions, the microstructure would change from a dendritic microstructure to an equiaxed microstructure which is more uniform and the grains are more rounded .

So, this is what you see over here, this is an equiaxed microstructure wherein we can see these grains which are more rounded, and also the size of these grains is uniform across the entire microstructure . So, this is the effect of the process parameter especially the cooling rate on the microstructure changing from a dendritic to an equiaxed kind of microstructure .

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Summary of Microstructure Control

- Conditions for dendritic growth: Pure metals and Alloys.
- Thermal supercooling is the driving force for dendritic growth in pure metals.
- Whereas, it compositional or constitutional supercooling in alloys.

Crystalline vs Amorphous

- Very high cooling rates > 10^6 °C/s: Amorphous/Non-crystalline powder.
- At normal (equilibrium) cooling rates, time is sufficient for atoms to arrange in an order. At high cooling rate not enough time for the atomic sorting process to occur.
- Time-Temperature-Transformation (T-T-T) diagram.
- Rapid solidification processing (RSP): Making metallic glasses.
- Melt spinning: Dropping liquid metal on a rapidly rotating water-cooled copper wheel.

Then we discussed the conditions for the dendritic growth, for pure metals first and then for the alloys as well. In case of pure metals, the driving force for the dendritic growth is thermal super cooling, and this is something we have discussed in detail.

Whereas, in the case of alloys where you have another metal as an alloying element or as a solute, the driving force for the dendritic growth is due to compositional reasons and that is known as constitutional super cooling. So, the driving force for the dendritic growth in case of alloys is constitutional super cooling .

Now, apart from this transition or this shifting between dendrite to an equiaxed kind of microstructure, there could be also a shift between a crystalline and amorphous structure when you talk about the internal structure in terms of the crystal structure of the material.

A very high cooling rate in the range of 10^6 degree Celsius per second or higher would promote the formation of an amorphous or non-crystalline structure. On the other hand, if the cooling rates are low like what you have in equilibrium conditions, then it will lead a crystalline structure . And the reason behind that as to why that happens we have discussed it in great details.

So, what we have seen is that when the cooling rate is low, there is sufficient time available for the atoms to get arranged in an order when the solid is forming right. And as a result a crystalline solid will form under equilibrium cooling conditions . Amorphous solids on the other hand are solids which do not have any particular atomic arrangement in the structure.

So, an amorphous structure will form when the atoms do not arrange themselves in a particular order right. So, that will happen only when the cooling rates are high enough, so that the atoms do not get enough time for this atomic sorting process or the atomic arrangement process to occur ok. And therefore, under high cooling rate conditions, the solid which will form will be amorphous in nature ok.

And this particular transformation was discussed in the light of this diagram known as Time-Temperature-Transformation diagram or T-T-T diagram in short which describes the relationship between time and temperature for a particular transformation. So, in this case, the transformation that we are talking about is solidification.

And here we saw that if you have a very high cooling rate the nose of the T-T-T diagram can be avoided and it will lead to formation of a non-crystalline or amorphous solid ok. And that is also given rise to a technology or a process known as rapid solidification processing for making metallic materials which are amorphous in nature. And this kind of materials are known as metallic glasses.

And with regard to that we have also seen the process for making this kind of amorphous metallic materials. The process goes by the name melt spinning which involves dropping the liquid metal on a rapidly rotating water cold copper wheel which leads to immediate quenching and solidification of this liquid in the form of a thin ribbon which is thrown off the wheel due to the centrifugal force.

And this thin ribbon then later on can be crushed or broken down into a powder . So, that is how you can also generate amorphous powder with the help of this rapid solidification processing techniques ok. So, with that we can conclude this particular topic and also today’s class.

Thank you for your attention.