Fundamentals of Materials Processing (Part–1)
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Lecture Number 35
Powder Consolidation

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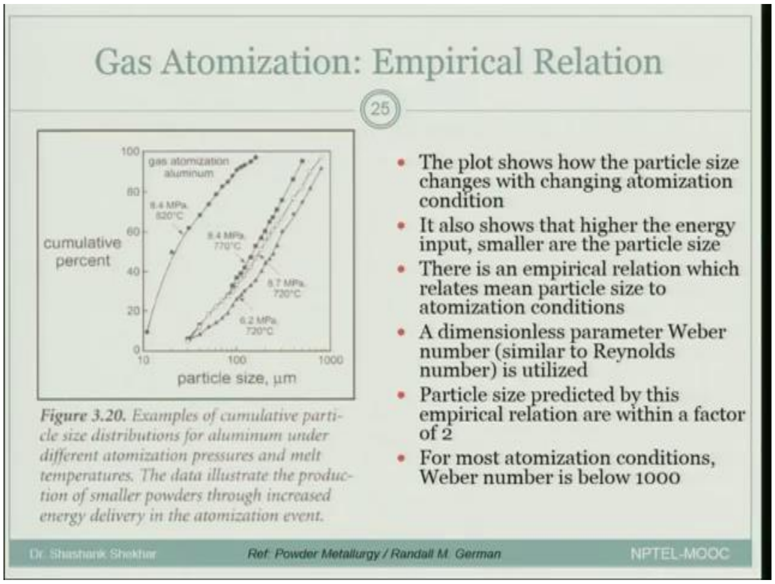

Gas Atomization: Empirical Relation

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Figure 3.20. *Examples of cumulative particle size distributions for aluminum under different atomization pressures and melt temperatures. The data illustrate the production of smaller powders through increased energy delivery in the atomization event.*

- The plot shows how the particle size changes with changing atomization condition
- It also shows that higher the energy input, smaller are the particle size
- There is an empirical relation which relates mean particle size to atomization conditions
- A dimensionless parameter Weber number (similar to Reynolds number) is utilized
- Particle size predicted by this empirical relation are within a factor of 2
- For most atomization conditions, Weber number is below 1000

Dr. Shashank Shekhar | *Ref: Powder Metallurgy / Randall M. German* | NPTEL-MOOC

So, we come back to this slide and the we are near completion of the (mod) sub topic of manufacturing of powder. So, we were looking at gas atomization and over here we are we would take a look at the empirical relation but before that let us take a look at this plot. Here what you see is the particle size and on the Y axis we have cumulative percent. So, the the plot is move to the left it would mean it has a smaller particle size and if it is much steeper then it would be it in very narrow in distribution. What we see is that slopes do not change much.

So, the overall size distribution it remains same no matter what is the pressure that is being applied or what is the melting temperature or what is the melt temperature. So, if you change the melt temperature it is not going to affect the overall slope however if we look on the shift of the plot when we see that pressure and the melt temperature both of them have affect on the distributions or the overall average distribution. So, when you point let us look at these two where we are applying the melt temperature 720 degree Celsius, so we have kept one parameter constant and we apply higher pressure.

So, here that gas pressure was 6.2 mega Pascal and over here the gas pressure is 8.7 mega Pascal, we see as you left shift of the distribution and that means that the powder size have becomes smaller and at the same time we also see that there is some effect of melt temperature for example if you compare this one and this one over here the temperature is sorry that the pressure gas pressure is kept constant which is 8.4 mega Pascal but the melt temperature has been increased.

So, if you increase the melt temperature again you are able to see a small drop in the particulate size. So, these are some of the experimental results and based on these empirical relations have been obtained to decide what is what would be the size of that particle. So, let me write down that empirical relation for the sake of completion.

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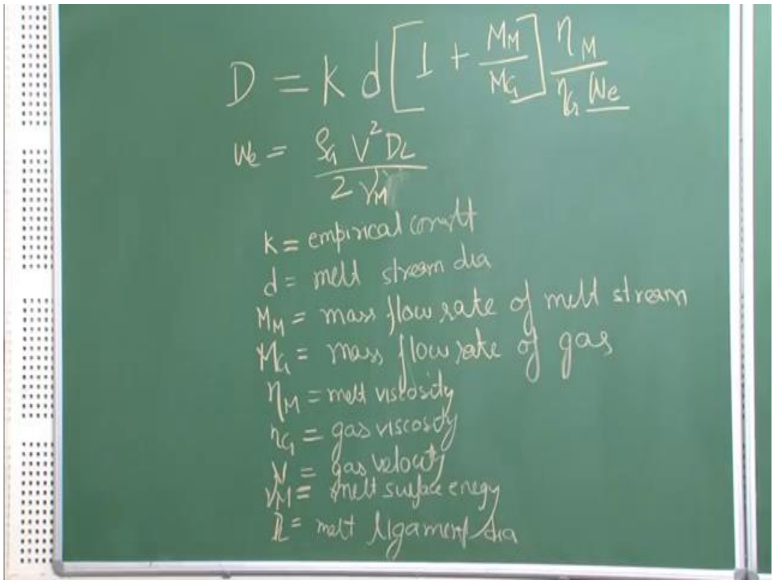

$$D = k\,d\left[1 + \frac{M_M}{M_G}\right]\frac{\eta_M}{\eta_G\,We}$$

$$We = \frac{\rho_G V^2 D_L}{2\gamma_M}$$

k = empirical const
d = melt stream dia
M_M = mass flow rate of melt stream
M_G = mass flow rate of gas
η_M = melt viscosity
η_G = gas viscosity
V = gas velocity
γ_M = melt surface energy
D_L = melt ligament dia

So, these the particle size and will explain the other parameters very soon and the this is there is also to take into account turbulence quantity called weber number is also utilized, will show what is the relation for that weber number. Will explain the other terms but this is the weber number that we were talking about and this is given by the relation, so this is (())(3:16).

So, this is the weber number which takes care or which point takes into account that turbulence and now let us look at what various quantities imply K is just is nothing but some empirical constant. So, you have this these relations and the functionality constant for these two diameter is by means given by this K. So, this is empirical constant, d is equal to melt stream dia and what you sees that the particulate size is directly proportional to d.

So, if you this you can also have expected could have expected from the relation that we showed for ligament size to sphere size from over there also we solve that if you want to decrease the diameter of the particle size we have to reduce the ligament size and the ligament size is again proportional to this d which is the stream diameter.

Mm is the mass flow rate of melt stream, Mg is the mass flow rate of gas, Eta are nothing but the viscosity, so Eta m is the melt viscosity that is a liquid melt, Eta g is the gas viscosity and V that we see over here is the velocity gas velocity, velocity of the gas and Gamma m is the melt surface energy and lastly we have this dl it is the melt ligament diameter.

So, although over here we see diameter is proportional to the small d which is the melt stream diameter but weber number is also proportional to this ligament diameter which comes in denominator however you when you put in the values you would see this would be the more dominant term, this one is or you can say this one ligament diameter is affecting that turbulence. So, the turbulence factor is being taking into account by weber number which is also indirectly contributing to the particle diameter. So, this is the relation for (dia) the empirical relation is defines the particle size for gas, gas atomization technique.

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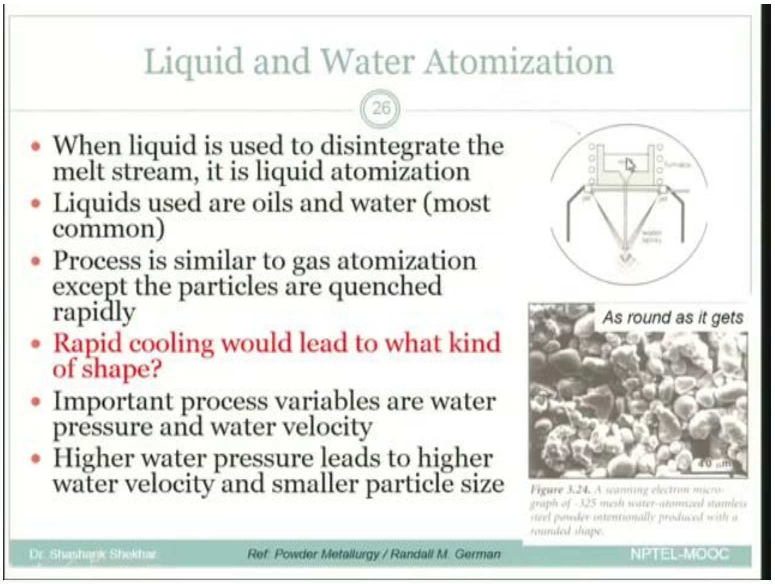

Liquid and Water Atomization

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- When liquid is used to disintegrate the melt stream, it is liquid atomization
- Liquids used are oils and water (most common)
- Process is similar to gas atomization except the particles are quenched rapidly
- Rapid cooling would lead to what kind of shape?
- Important process variables are water pressure and water velocity
- Higher water pressure leads to higher water velocity and smaller particle size

As round as it gets

Figure 3.24. A scanning electron micrograph of -325 mesh water-atomized stainless steel powder intentionally produced with a rounded shape.

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Ref: Powder Metallurgy / Randall M. German

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And point like I said will I will spend just a little bit of time or liquid or (())(6:33) water atomization. So, most of the time the liquid that is used in this kind of atomization technique it is waterr. Over here we just as you can see over here also the melt is coming down just like in the previous case and through a jet you are forcing or you are causing the water to splash or splash against the melt and this again causes disintegration of the liquid, liquid melt.

So, the liquid that is coming through the jet with the high energy is disintegrating the liquid melt that is coming through over here. So, the overall principle as you can see is still the same. However there are some other parameters that start to effect and the empirical relation that we get is a little bit different from what we have seen over there, over here the angle at which the water sprays is splashing the melt. So, this theta this angle theta also comes into (())(7:30).

So, let say this is alpha because that is what we use in our relation, so this is an angle alpha then there is the point p the constant which incorporates both material and atomization technique and will see about that in couple of minutes and there is also the V which is the velocity. So, these are the parameter that will affect the water atomization technique and the and the particulate size that comes out of it. On an very important aspect or parameter which differentiates liquid atomization from gas atomization is the very very high cooling rate that you will get in over here.

So, the cooling rate here would be very high, so now what do you think would make a, what will or how will it make a difference to the overall particle size or particle shape. We remember over there we had particle shape going from flake to ligament to ellipsoid to finally the historical shape. Now since the cooling rate is very high what will happen is that the probably the particulate or the particles that are coming out will freeze even before the reach the equilibrium spherical shape and that would mean that what you would get out of it will be somewhat random sheet.

And now if you look at the particles that you get over here they are indeed not as a spherical as you would getting in gas atomization technique. So, this is because you are getting very high cooling rate, so the quench rate or the very high cooling rate is what is causing somewhat irregular shape, this is as round as it gets for liquid atomization. So, important process variable for the liquid atomization as you would see when we write down the equation or water pressure and water velocity, higher water pressure leads to higher water velocity and smaller particle size.

So, now that we have point talked about this will in a little bit of details, so let us look at the empirical relation which will define the particle diameter with respect to the several point parameters that has affected.

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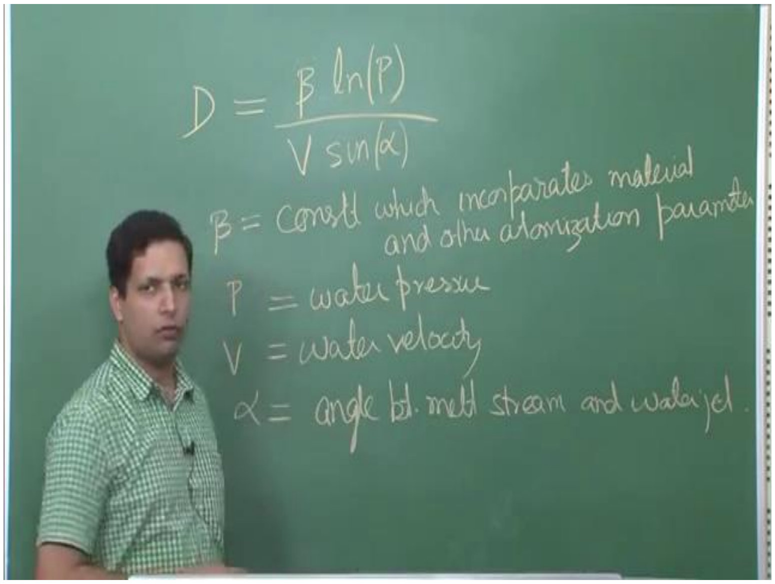

$$D = \frac{\beta \ln(P)}{V \sin(\alpha)}$$

β = Constt which incorparates material and other atomization paramete

P = water pressure

V = water velocity

α = angle bt. melt stream and water jet.

So, again d will be the particle diameter that you obtained using liquid atomization technique and this will be given by beta lnP and V sine alpha, you remember alpha (())(9:53) the angle between the water jet and the melt. Beta is again some constant this point incorporates or this takes into a account all the material and atomization technique parameters.

So, it incorporates material parameters and other atomization parameter P is we know water pressure, V is water velocity and alpha is the so, here what you would see is that the relation look somewhat similar but that is because these are the more important parameters, other not point so influential parameters are clubed into this beta. This is the empirical relation for liquid atomization and the previous one that we look that was the point empirical relation for the gas atomization technique.

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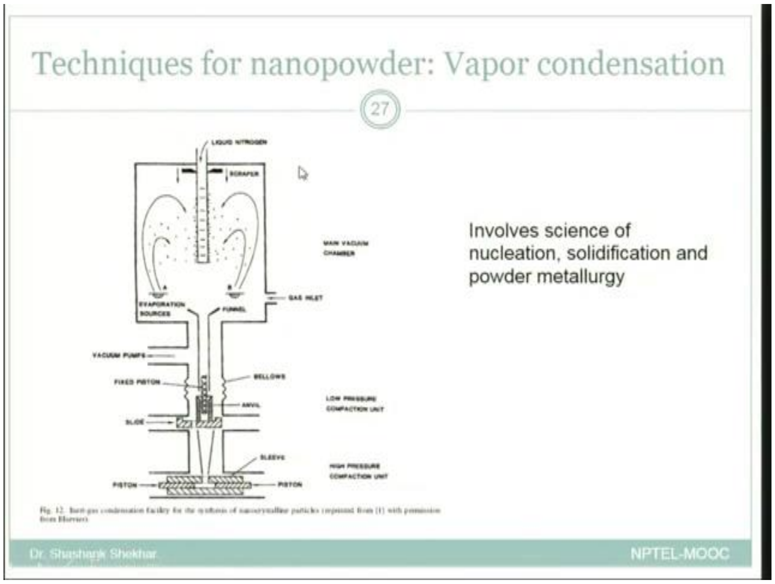

Techniques for nanopowder: Vapor condensation

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Involves science of nucleation, solidification and powder metallurgy

Fig. 12. Inert-gas condensation facility for the synthesis of nanocrystalline particles (reprinted from [1] with permission from Elsevier)

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So, now we come to the last slide on atomization technique, so this is a technique that I was talking about earlier. So, that you can use atomization also for creating Nano powders and over here there will be little bit of difference. One of the biggest difference is that this will be a very high vacuum chamber because you want you do not want the particulates to class or a strike against many other gas molecules because that may also calls cause condensation and even heterogeneous condensation and over here this is the liquid nitrogen, so you want a very very super cooled condition.

So, that you get very homogeneous nucleation. So, this is a liquid nitrogen column and this is the evaporation source over here you are heating whatever material you want to create and these point molecules or these are in point you can say point most molecular state and when they reach this liquid nitrogen which is very low temperature then over there they because of the super undercooling very large undercooling they will have, they will undergo homogeneous nucleation.

So, what you want is the over here the vapors to come into contact and then point very quickly solidify because of very large undercooling and under homogeneous nucleation condition and that is when you will get very fine powders. So, these fine powders come over here and there is a scrappled which will come down and all the powder particles will get collected over here. So, this is a schematic which tells you how you can get even Nano size is scale powders using atomization technique and if when you are talking about this then this will also involve science of nucleation, solidification and powder metallurgy.

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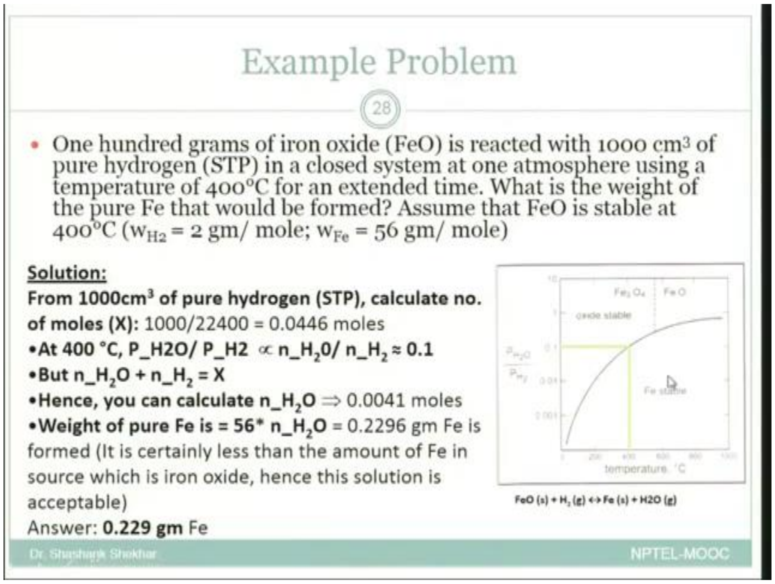

Example Problem

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- One hundred grams of iron oxide (FeO) is reacted with 1000 cm^3 of pure hydrogen (STP) in a closed system at one atmosphere using a temperature of 400°C for an extended time. What is the weight of the pure Fe that would be formed? Assume that FeO is stable at 400°C (w_{H2} = 2 gm/ mole; w_{Fe} = 56 gm/ mole)

Solution:

From 1000cm³ of pure hydrogen (STP), calculate no. of moles (X): 1000/22400 = 0.0446 moles

•**At 400 °C, P_H2O/ P_H2 ∝ n_H₂0/ n_H₂ ≈ 0.1**

•**But n_H₂O + n_H₂ = X**

•**Hence, you can calculate n_H₂O** ⇒ 0.0041 moles

•**Weight of pure Fe is = 56* n_H₂O** = 0.2296 gm Fe is formed (It is certainly less than the amount of Fe in source which is iron oxide, hence this solution is acceptable)

Answer: **0.229 gm** Fe

$FeO(s) + H_2(g) \leftrightarrow Fe(s) + H_2O(g)$

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So, this is the much more involved process so to say. To end this point the to end this topic sub topic on the point formation or the powder manufacturing technique will look at one example problem. So, let say we have 100 grams of iron oxides, so we are looking at the example related to oxide reduction. So, you let say we are the one hundred gram of iron oxide that is reacted with one thousand centimeter cube of pure hydrogen. So, this hydrogen that is being reacted is given in volume because it is in gaseous state and the iron oxide that is being reacted is given in weight because it is in solid state. In a closed system at one atmosphere, so you have given the pressure using at temperature of 400 degree Celsius for extended time what is the weight of pure Fe that would be formed.

So, given there is information you have to find out what is the weight of pure Fe that would be formed. So, we we what will need to do look at this equation, you see this is the equation that takes place or this is the reaction that takes place. So, FeO in solid state reacts with hydrogen in gaseous state, to form iron in the solid state and leaves out moisture in the gaseous state and the temperature pressure phase diagram for that different phases are given over here this is the region over which iron is stable and this is the region over which Fe3O4 is the stable and if you go to the higher temperature FeO become stable.

So, that overall this is your line of equilibrium, now here what we lead to do is first find out what is the number of moles of hydrogen and number of moles of H2O that will be formed and if you know what is the number of moles of hydrogen that has that exist and compare it with what it was earlier which is point based on the F 1000 it centimeter cube will know how many of them have reacted. So, how many moles have reacted those many moles of FeO with have reacted or that many moles of Fe would have form.

So, that is the overall thought process that we are following, so what will do is you know one, one thousand centimeter cube of pure hydrogen and it is already given STP will calculate the number of moles state a number of moles and it comes out to 0.0446 moles and then next will looked at what is the ratio of H2O and H2 molecules that we will be able to know because we know what is the pressure ratio of H2O and H2, PH2O by PH2.

So, here is your 400 point the this is that temperature and this is the pressure that we are looking at sorry this is that temperature and that this temperature will look at the equilibrium diagram where both both Fe and Fe oxide exist together and will assume that we have still in the both of these are FeO we are we are not going to complicate by assuming that this is Fe 3 O4 as you can see you have starting with FeO. So you will assume that this is still FeO.

So, we are t this reaction boundary and the pressure ratio that we see over here is 0.1 in the pressure the partial pressure of H2O to the partial pressure of H2 is known which is equal to 0.1 then this has to be proportional to the number of molecules of H2O to number of molecules of H2 and therefore we now know the number of molecule ratio of number of molecules of H2O to number of molecules of H2 and which is equal to 0.1. But you also know the total number of molecules of H2 and H2O because we started with 0.446 moles of overall hydrogen.

So, some of them have reacted one one each mole of H2 that reacts it will form one mole of H2O. So, the number of moles are not lost, for each mole of H2 there will be one mole of H2O. rTherefore whatever is this 0.446 that will also represent the total number of moles of hydrogen and moisture. So, NH2O plus NH2 is equal to X which is equal to 0.446. So, if you take these two relations that is NH2O plus NH2 equal to 0.446 and H2O by NH2 equal to 0.1 you can calculate that number of molecules of H2O is equal to 0.0041.

Now this is the number of molecules that have reacted, so what we need to know by what you so we have already obtained how many molecules have reacted, So those many molecules of iron must have formed, so we will multiply this by the weight molecular weight of iron it is 56. So, 56 times this NH2O it is 0.0041 and therefore 0.2296 gram of iron should be formed. So, we started with one hundred grams of iron oxide and if we maintain these conditions then we see that 0.2296 gram of Fe is only formed.

So, this is a very small number and how do we increase it within it discuss with when we are talking about oxide reduction, you can very easily increase this by taking away by H2O molecules. So, now it is not we are not saying it is a closed system, this system is open, we are we will continuously take away H2O molecules. If H2O molecules taking a way more H2O will react and it will lead to further 0.2296 gram of Fe2 formed, then again we take away and saying these these steps that in reality it will be taking place in continuous manner.

So, again we take way that H2O molecules and 0.2296 gram of another 0.2296 gram of Fe will be formed and so on the process will keep continuing untill all of FeO has been converted to Fe. So, this is the example problem on oxide reduction method of manufacturing powder.

And now next will move on to our next topic as we remembered we have now covered introduction to powder processing, we looked at various important powder characterization parameters and techniques then we looked at so many different ways to manufacture powder and now we come to consolidation powder consolidation.

So, let say you have powder and we put them together, now you want to know if I just put the powder together even before compaction what is the relative density that I should get, what are the number of point contacts between each other particles because it is that those particular points where the powder particles get in contact where the eventual centering will start. So, these are the some of the questions that you were we will be able to answer then we look at powder consolidation.

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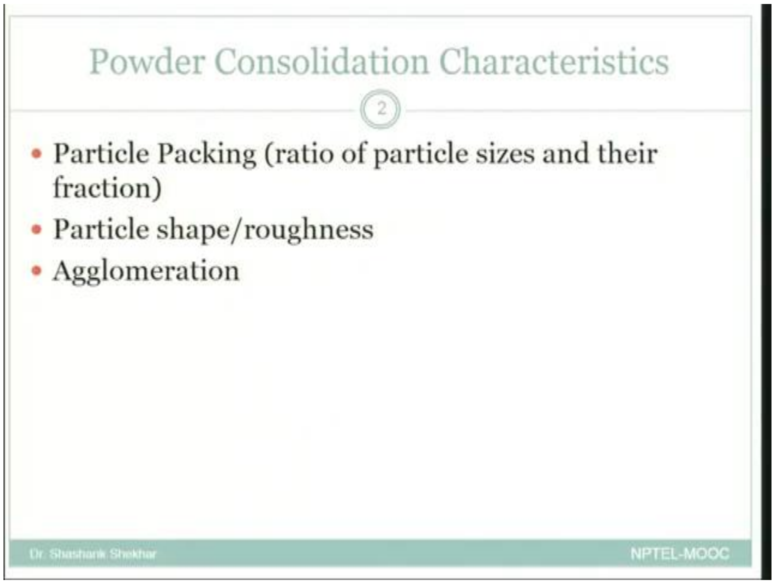

Powder Consolidation Characteristics

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- Particle Packing (ratio of particle sizes and their fraction)
- Particle shape/roughness
- Agglomeration

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So, the first thing that we will look at in terms of powder consolidation is particle packing. So, there are several ways we already are awere of some of those of about particle packing from our crystals structure knowledge we know about FCC, BCC etc. So, we can also take then as a model of how the powder particle have been fitted and find out what should be the packing fraction much will be equal to the relative density. So, let us look at some other well known point packing and the relative density that you would get over there.

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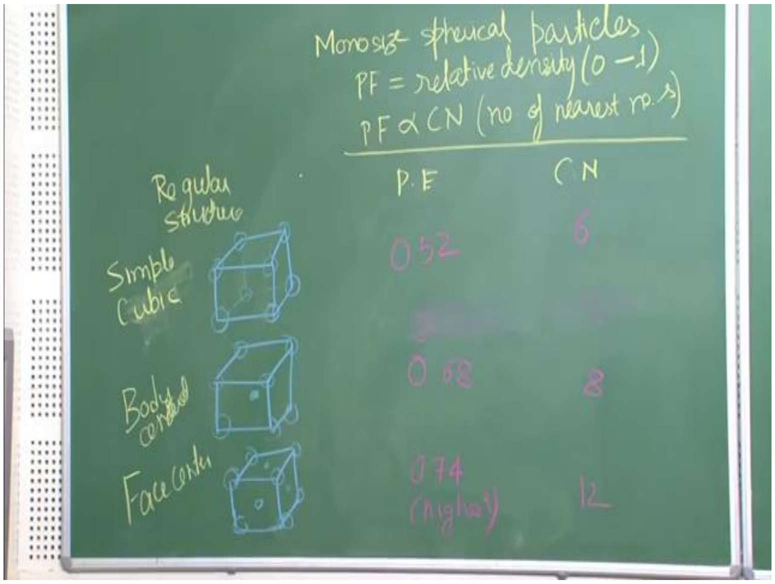

Monosize spherical particles

PF = relative density (0 – 1)

PF ∝ CN (no of nearest no.s)

Regular structure	P.F	C.N
Simple Cubic	0.52	6
Body centered	0.68	8
Face center	0.74 (highest)	12

So, let us take for example to begin with will keep it simple. So, when you take mono size spherical particle, okay. So, this is a hypothetical scenario because in reality you will never have mono sized but this is just to give us an idea how to relate some other important parameter to each other and when we say packing fraction this will be equal to relative density which will vary between 0 to 1 and we also know that packing fraction is proportional to coordination number that is number of nearest number.

So, with this let us start with point some of the simple structure, so will look at some regular structure and will look at what is there packing fraction, see what is their coordination number. So, let us first look at simple cubic okay we are not remember we are not talking about the crystal system we are look at we are using that term similar to what is used in crystal structure but we have at the corners or where ever there atom used to set we will be having only powder particles.

So, all spherical and that two mono size spherical partials, so this is a regular simple crystal BCC sorry a simple cubic use simple cubic. So, your atoms are sitting only on the corners and you would know what is the packing fraction for this, the packing fraction for this kind of system is 0.52 coordination number is 6. Let us get to a different system what is (())(23:33)again we are not talking about crystal systems at the at the edges or wherever the atom should have set we have powder particles.

But the packing fraction that we obtained for crystal system is also valid for this okay as far as we have mono spherical single particle mono spherical spherical particles. So at the other in the corner you also have at the body center and for this we know that packing fraction or the relative density is 0.68 and coordination number is 8 and then we also have phase center putting them at one place mix it point easier to compare and point comprehend.

So, other than the corners also have atoms at the faces at the center of the phase and then also on the other back phases we are not drawing the back phase and with that is not the phase center is supposed to have the best okay this is not coming the same line let me see this is other in same line. So this is the highest packing fraction that you see for in system and it is far it from highest and what is coordination number that is also highest which is equal to twelve.

So, these are the some of the while most well known system we are borrowing our understanding from atomic system or a crystal system and we are using it over here and now we will let it two sums and will relate now some of the other parameters that we know from again from our understanding of crystals systems.

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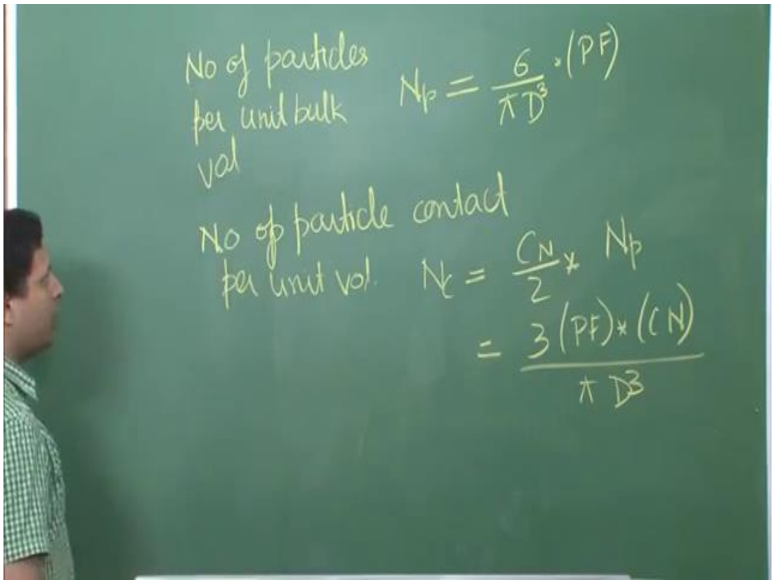

No of particles per unit bulk vol $N_p = \frac{6}{\pi D^3} \cdot (PF)$

No of particle contact per unit vol. $N_c = \frac{C_N}{2} * N_p$

$= \frac{3(PF)*(CN)}{\pi D^3}$

So, we know that number of particles Np is equal to number of particles per unit bulk volume. So, what we need is packing fraction divided by the volume. So, this is saying that there is 0.74 of point the cube it is same, so what we are saying in 0.74 divided by the divided by the total volume of each particle is the number of particles per unit bulk volume. And then other parameter that we will define is number of particle contacts and these are all our understanding borrowed from point crystallography. So, this is given let us say bulk Nc you can write it by coordination number by two times Np.

So, number of particles per net volume and which are them have coordination number, some coordination number, so that is the total number of coordination number that it will be shared by another particle. So, will count only half that coordination number. So, this gives you the number of contacts per unit volume and for if we take Np equal to six by pie d cube times pf, then this becomes 3 into PF and you would see that we are using this terms PF, and CN because then we will be able to generalize it you will for the condition were crystallography does not apply for our non regular structure.

So, now we have written in a more general term the relation of number for the number of particles per unit bulk volume number of particle contact per unit volume and this is how it will look like and this these are very straight forward when we are looking at regular structures which again likely said for the systems that we borrowed from crystallography but then I have also said that we do not always get a regular system, in fact these are ideal case scenario in point in real life what you will get is not really point simple cubic or a body centered or a phase centered.

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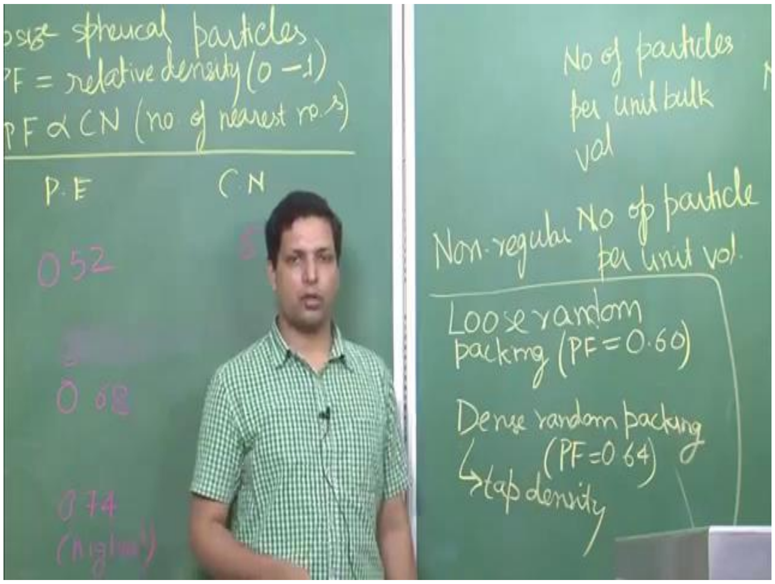

What we will get point we would like what is called as loose random packing. So, let me so let me say this is non regular those they are regular structure they had long range orders but in general what you will get is not our something which has long range order. So, this will be either loose random packing or dense random packing. So, in this case if you take PF it will come out to approximately 0.6, if you take the packing fraction in this case it will come out to approximately 0.64 and these are approximate numbers based on our (expri) experiments of different kinds of powder and these are that two most point observe two most observer.

So, you will see loose random packing that is when you have just put in the powder and not tapped it dense random packing when you put in the powder in a jar or point in cylinder and then it tapped it you must have seen that when you tap for example where you take grains you put it in a vessel and then you tap it then overall volume that was occupied bit reduces. So, those this is will you related to tap also whatever it call tap density.

So, after tapping how much you have looking in n this will before tapping. So, will get back to this and will continue our understanding on powder consolidation in a next lecture. So, will end over here and meet and see you next time. Thanks!