Fundamentals of Materials Processing (Part–1)
Professor Shashank Shekhar
Department of Materials Science and Engineering
Indian Institute of Technology, Kanpur
Lecture Number 32
Powder Manufacturing

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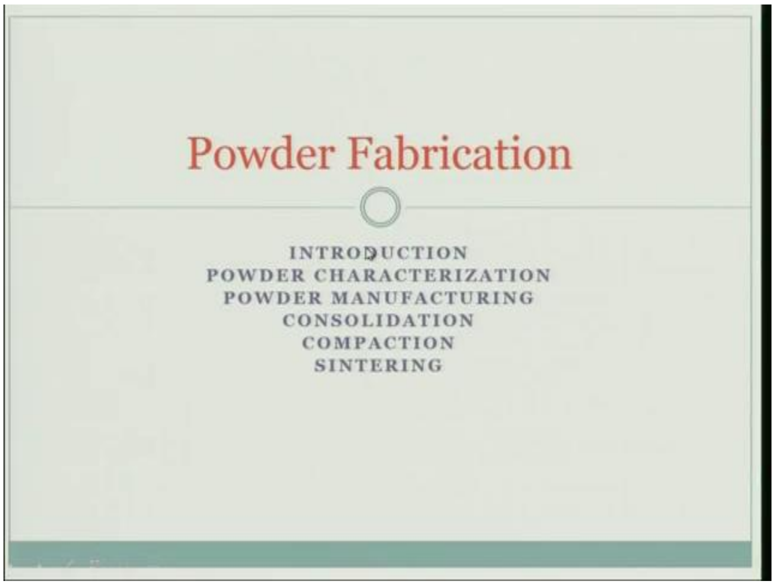

So we come back to powder processing and we have already finished the first two modules which is first is sub modules introduction and powder characterization. Now we move on to powder manufacturing, so now having introduced you to the importance of powder processing and introduced you to the ways of characterizing powder it let us look at how the powder are manufactured.

So basically we are looking at step one of the whole actual processing step that takes into place where you first have the powder manufacture powder and then you do consolidation, compassion and then do the centering. So first step is powder manufacturing how are the powders manufactured. There are various various techniques by which powder is manufactured and this can be roughly classified into some of these classes.

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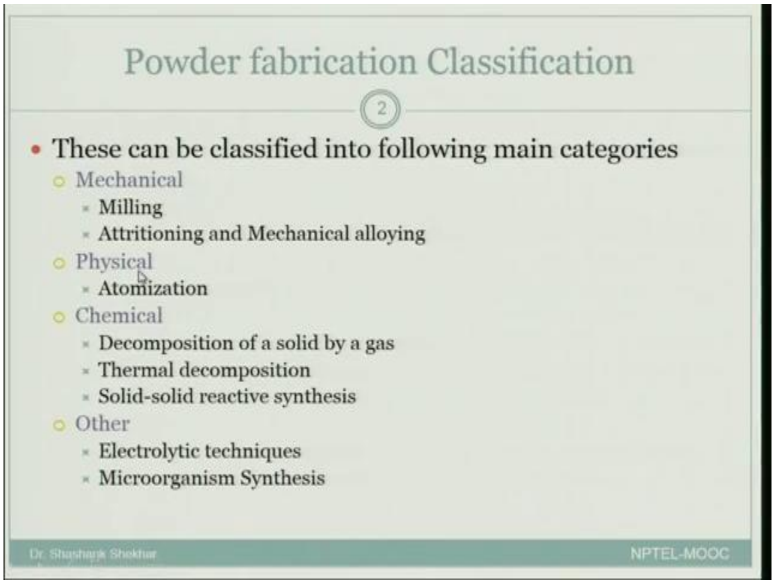

Like mechanical, physical, chemical and still still other classes. Mechanical processes includes something like milling or attrionally attritioning and which also involves mechanical alloying. Physical processes involves something like atomization, chemical process which is also a very large very important method and a lot of powders metal powders are produced by this.

It itself can be sub classified into techniques like decomposition, thermal decomposition, solid-solid reactive synthesis and so on. And even some of these which are not classified in the above there are examples of that for example electrolytic technique, microorganism synthesis and so on. So we will look at some of these processes some in mechanical particularly and some in chemical and then will towards the end we will look at, some atomization technique.

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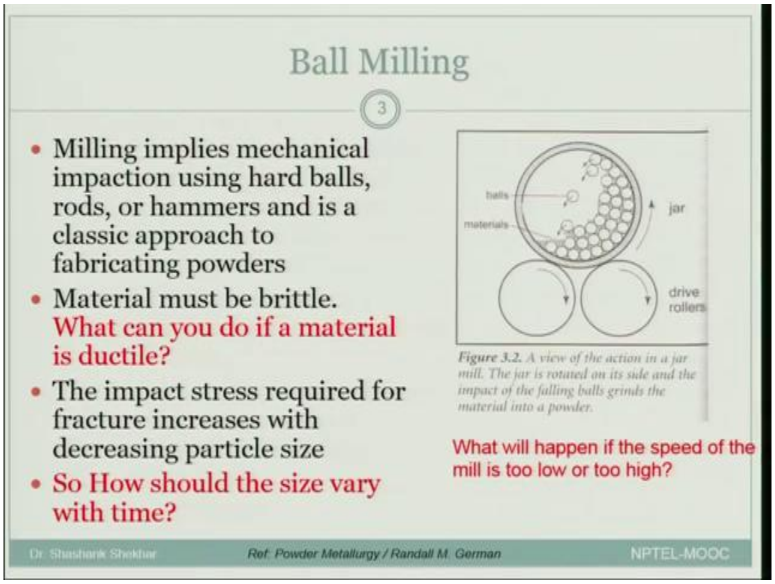

So let us gets started with mechanical method, now the mechanical method what you need is it to begin with you will have some bulk material. So you will have a large chunks of maybe cylinder or or a billet and then from there you need to produce powder. You cannot start to produce very small particles from the first stage itself. You have to do it in stages, so the first stage is to make it into smaller into little bit smaller particles or something like a particle. Initially it may be in a bulk bulk form the first step is to reduce it into some smaller sizes.

And one of the very common techniques is to use the scraps from machining. You see those chips that are formed during machining can also be used as scrap material and then it can be taken for further processing. Some of the other techniques for course milling are roll crusher so let us took let take a look at what is the usual layout of roll crusher.

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So in roll crusher what you do is let us say we have very-very large particles. So roll crusher will have design something like this, there will be two big rollers with huge power rotating and the gap between the two will set what is the largest size particle that will come out. And you will put some very large pieces chunks of material over here. And after crushing it will come out as smaller particles and so on. So this is a roll crusher and these are the rolls which are rotating and they are backed by very huge power.

Because you need to break down some of these particles so this is the way a roll crusher works. So this is another mechanism for course milling and one still another method which is called jaw crusher that works something like this.

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And you will have a see-saw movement of two. So this can be pivoted over here and it will move back and forth like this. And again here you can put some very large particles. And since you are moving it like this and this it will break those they are moving like this so the particles will break and on the bottom side you will get much finer smaller particles. So these are some of the techniques which are used for course milling so that you can reduce the particle size to somewhat manageable and you can get to the next step of refining it to the desired particle size.

So, so far we are just looking at you can say a rough estimate of particle size. We are just, we just want to break it down so that you can later do the processing and bring down the size. Now for fine prior processing or to bring down the particle size to within a desired range you use a much more sophisticated technique. And we are looking here at one of those mechanical techniques which is called ball milling.

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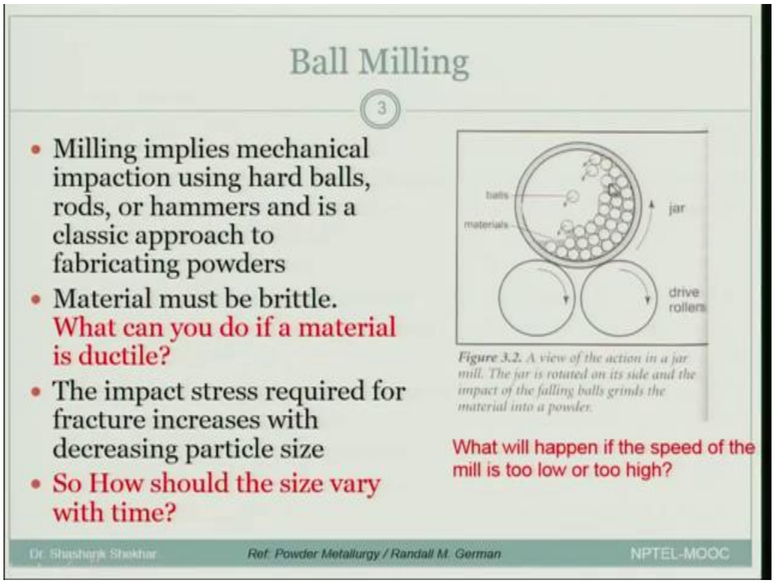

So, what is ball milling? Milling you implies mechanical impaction using hard balls, rods and hammers. And this is a classical approach for fabricating powders, especially at a lab lab scale or at a research scale. So what you have is a big drum, so this is a big drum rotating and inside it these large spheres that you see over here are actually what is called as milling media.

So this is, these are very heavy and large balls which keep on rotating and while rotating they fall onto each other. And in between this are the material that you want to reduce to the desired powder size or desired range of size. And once what what happens is that when this milling media or the milling balls fall onto each other and there may be some particles in between them, so those particles will get crushed into smaller and so on this will keep on rotating until our desired size is achieved.

And what is the desired size achieve depends on so we will see what that depends on but before that you must realize that this will be much more useful only when the material is brittle. If the material is not brittle what will happen it will just get flattened out it will not break. What we want here is that the material should break while we are doing this milling. So this jar keep rotating because of this drive rollers.

So these are the rollers and the (ba) jar is rotating like this and the balls keep falling on to each other and in between this the particles come in and they break. But if it is a ductile material it will not break it will only get flattened out. However even if the material is ductile there are some ways to make it brittle. So this is what you can do if a material is ductile you can make it brittle.

One for example metals can be very easily met brittle by introducing them to hydrogen. So hydrogen is called hydrogen (embrit) embrittlement, so you can do this in the hydrogen atmosphere or the metals which have been exposed to hydrogen so that they become embrittled and then you can use this boo milling media millings method and get small sizes of powder.

But it is much more useful for let us say metal oxide or ceramics, because they are very brittle in nature. They impact stress for fracture increases with decreasing particle size. Now the this reduction in particle size is dependent on the crack size. And because we are looking at fractured or basically the material is getting fractured. So let us look at the sigma fracture the stress required for fracturing.

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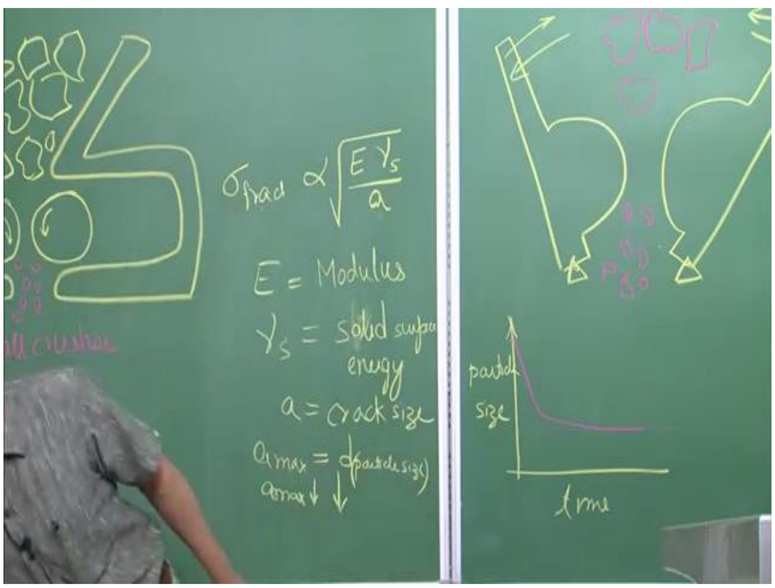

So this stress required for fracture can actually be written or is proportional E gamma s by a. what is E it is the modulus of the material that we are looking at, so higher the modulus higher the fracture stress required. Gamma s is the solid surface energy, now whenever you are breaking the breaking the particle you are also increasing the surface which means the surface area energy is increasing.

So the stress required must be proportional to the surface energy. And ‘a’ is the crack size, so the smaller the crack size larger the stress required. Because the crack has to grow, if the crack if the crack size is very large then the material can much more easily break. So you want the crack size is to be large, but how large can the crack size is be. Obviously the ‘a’ max can only be equal to the particle size.

Let us say ‘d’ this is a particle size, so ‘d’ if you are trying to find what is the maximum stress that is required you would see what is the lowest crack size and the lowest crack size if we want to see what is the the maximum crack size we will have to look at the maximum what is the total particle size. And if the particle size reduces, let us say that the particle size reduces over here then it implies that ‘a’ max also reduces.

And when the ‘a’ max reduces it implies you need to require larger stress. So it gives you an idea of what is the largest stress you can create which means it will be related to the maximum ‘a’ that can be that can be cracked and which will be related to what will be the maximum or what is the minimum particle size that you can obtain. In term what is it, what I am trying to say is that for given maximum stress there is a smallest particle size that you can achieve.

You cannot go below that particle size. And therefore if you were to look at particle size versus time. There will be a limit or the lower limit of the particle size. Because that depends on the maximum stress that you will create. And what is that maximum stress dependent upon, that maximum stress is dependent upon the milling media material that you are using and from how large height it is falling.

So the size of the drum, the weight of the milling media this will determine what is the maximum stress that you can generate. And from that maximum stress generated you can find what is the lowest crack size that can be broken and that will depend on the smallest particle size. And therefore this describes our smallest particle size.

Now to begin with let us say that this is our beginning particle size on an average. So will this change with time. So in the in the beginning there will be enough number of large particles to break. Break them down to smaller pieces and therefore it will very soon come down to smaller size but with time the probability of the particles which are which have enough crack length that can broken reduces.

And therefore it will become slower and slower and so it will asymptotically reach this lower limit value. So this is the way the particle size will reduce with respect to time. Now let us say we change certain condition, for example earlier we were using a dry medium and let me show this with a drawing over here.

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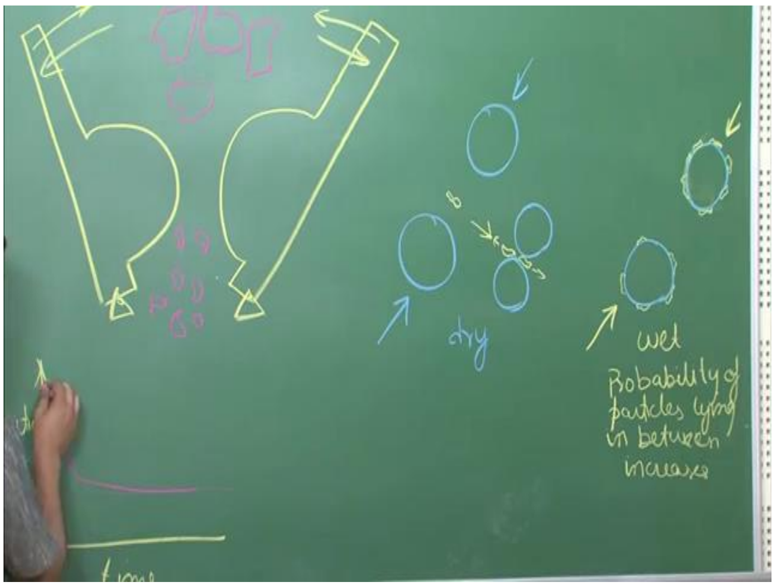

So in general let us say these are the two milling media and here we had some particle like this. And when this milling media balls come in together, this particle gets broken into smaller pieces and it moves out. And this is in dry condition. So now here we have a lot of things that must come together, two milling media balls must come together and at the point of contact you must have a particle which will get broken down into smaller pieces.

Now let us say we are using now wet condition, what will happen in the wet condition? Because of the surface tension of the liquid which is being used some of the particles will start to stick onto the surface of this milling media. So these are particles which are sticking because of the liquid medium that we are using and therefore now then when this balls come together there is a much larger probability for the particles to be in between the two balls that are striking.

And therefore here what will happen is that the probability of particles lying in between increases. So how does how should that change our plot, should it change the amount of time or should it change what is the maximum or what is the minimum size we can reduce. As you can see it is only the probability which has chased. And therefore the rate at which we are reducing the particle size will decrease.

We have not change the important parameters which are related to this equation, which means the minimum particle size that we can achieve still remains the same and therefore if you were to compare a wet system and a dry system, this is how the plot will look like. So this is for wet system and this is for dry system. So this the overall understanding of how the say ball milling medium or the ball milling system works to reduce the particle size.

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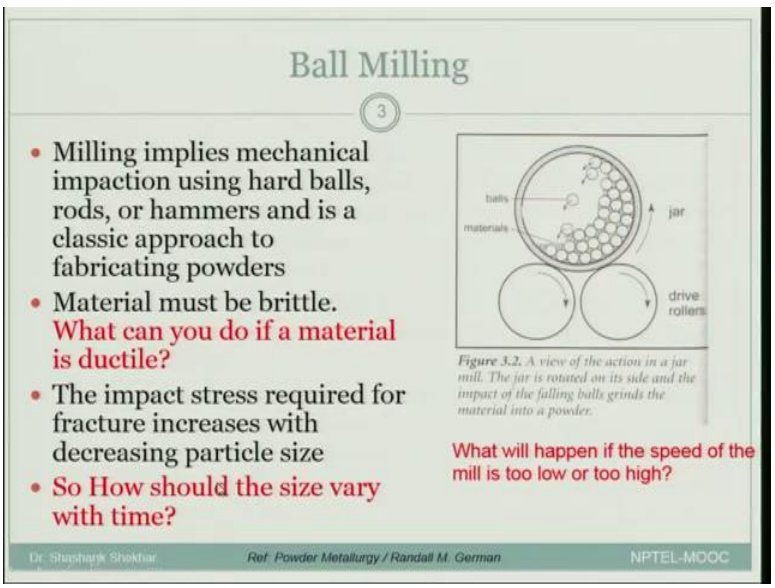

So let us get back to our slides and we have already answered the last question what we have over here which is how should the size vary with time. So this is the part we still need to answer what will happen if the speed of the mill is too low or too high. So, you see we are rotating this jar over here as the milling balls are falling on to each other to crush the balls.

Now there is another parameter which is how fast or how slow the jar should move. Let us say the jar is moving very slow, what will happen if the jar is moving very slow then the milling media will just roll onto each other. They will not impact or they will not fall over onto each other because the impact. So you do not want the milling jar to rotate at a very slow speed.

So there is a lower limit you should not set the speed of the jar. On the other hand if you keep increasing the jar speed so that it becomes very very large. Then what will happen is that because of the centrifugal force, the ball milling medium was the milling medium was will remain stuck on the surface on the periphery because of the centrifugal force. And it will not fall onto each other.

So again in this case there will be no crushing force which will reduce which will break the particles. Therefore there is also upper limit, so this in this particular system there has to be a optimum speed of the jar for at which you will be able to get very fast reduction in the particle size. If you keep the speed very very low it will not break if it will if you give the speed very fast again it will cub milling media will not fall it will be stuck onto the periphery because of centrifugal force and the particle size will not reduce.

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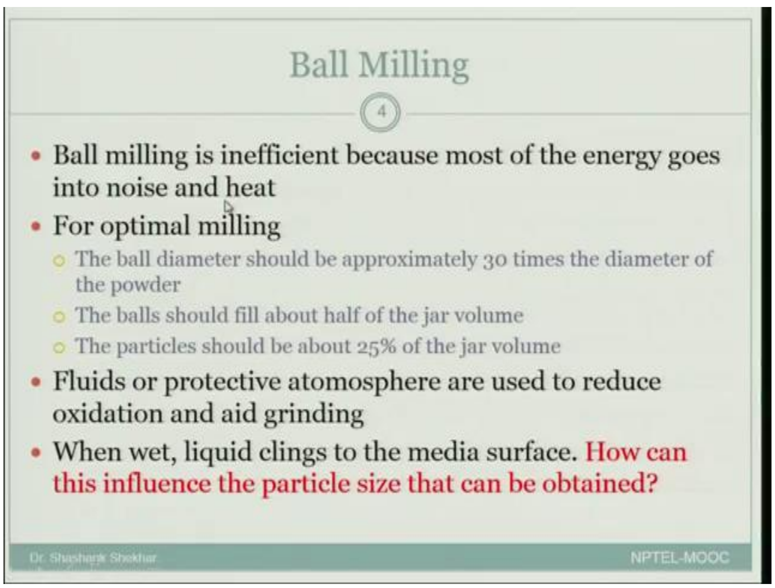

So moving on to understand a little bit more about ball milling, ball milling is inefficient because most of the energy goes into noise and heat. As you can see many a times when the balls the milling media falls onto each other there may not be a particle. And because of that all that energy that was that went into raising it all the way to the height of the cylinder jar and then allowing it to fall down that energy is lost.

And even when the particles are being broken a lot of that energy is going into noise and heat. So this is not really the most efficient method of repairing or creating ball creating powder size particles. However this is the you can say easiest and most controlled for at least for laboratory skill this is the most controlled technique and that is why it is still very widely used.

For optimal milling there I have to over the years people have done been dien doing ball milling for quite some time and for quite different types of variety of materials and therefore there are few understanding people have gained regarding ball milling to get optimal milling. One of it is that the ball diameter should be approximately 30 times the diameter of the powder.

So if you are looking at say 0.5 millimeter powder, then you should use balls 30 at least 30 times greater than that. So it it means around 15 millimeter diameter should be the ball size. The ball should fill fill about half of the jar volume. Again the number of balls that you can use is has to be optimal. If you less, use to less then there will not be enough number of balls falling onto each other to crush the particles.

If there are too many then there is not enough space for them to fall and the therefore the optimum amount is it should be half of the jar volume. The particle should be about 25 percent of the jar volume. So it should fill about 50 percent by the milling medium balls and 25 percent by the particles that you want to create or that you want to produce. Fluids or protective atmosphere are used to reduce oxidation and aid grinding.

Now the when you reduce the powder size then you are increasing the surface area. And even if it has even if a little bit of reactivity to oxygen it is very easy to get oxidized. And therefore you it will be advisable to use some protective atmosphere so that oxidation does not take place.

And another important aspect we already discussed is when wet liquid clings to the medium surface when there is medium is wet the liquid clings to the medium surface and also causes the particles to cling to the surface and therefore what you get is a plot like this where you get the particle size reduction at a much faster rate compare to when you are doing it at dry condition. So the wet condition is desired stage because that will lead to faster reduction in the particle size.

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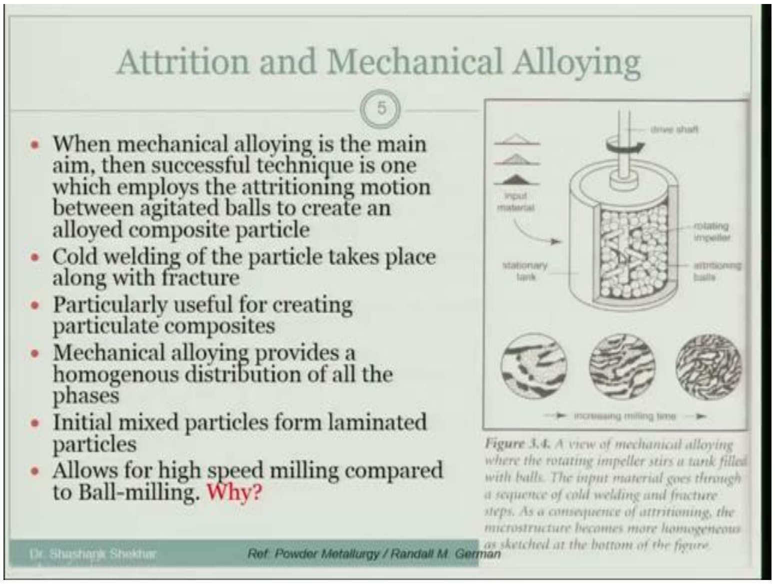

So that is about ball milling now let us look at a relatively or you can say related mechanical technique which is called attrition or mechanical alloying technique. Again here we are using milling medium just like the previous one, however instead of allowing the balls to fall onto each other. The center of access here is vertical and therefore these balls are not falling onto each other.

But there there are some rod over here as you can see in the picture which is the rotating impeller because of that the balls are made to you can say move against each other or share onto each other. And if there is some powder particle over there than those powder particle will also get shared. So this is a good way to share the particles. And when we are doing sharing it is it is very easy to get alloying.

So let us say you use three different powder mat materials like this like shown in the figure then if you put them together because of this attritioning or because of this sharing these will get you can say cold welded onto each other. And therefore you will start to get something like this. So there are two layers which are shown here at least two layer in fact there are three layer.

This is the black white layer and this grey layer and after attritioning these layers get cold welded onto each other and at the same time they are get reduced in particle size. So the cold welded part gets reduced and it is getting cold welded and therefore over time you get very very uniform microstructure. So initially it may be something sorry let me again draw into schematically explain what is happening here so again let us say.

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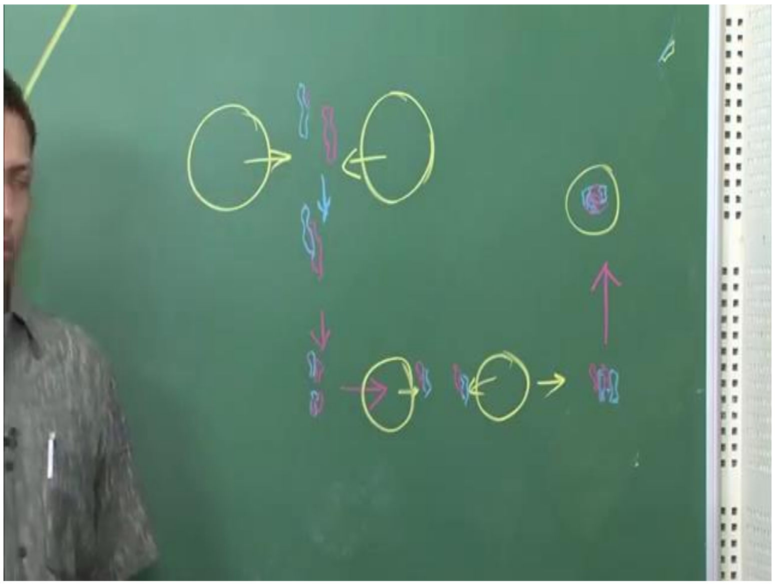

This is these are two ball milling mediums and you have two different layers. And because of the attritioning motion let us say these two balls come together and sphere the two particles together. So this will become something like this. So this is one step, but at the same time this particle can be further reduced in size so you will have and if you keep going and then these two are the particles which will get sphered onto each other. So you can say you will have let us say something like this and there are milling medium over here.

So it becomes, now you can see there are alternating layers it need not be exactly alternately but the idea is that different materials are getting cold welded onto each other. The and they or at the same time a breaking down. And the breaks the broken down particles will come together and form some alternating or lamellar structure and over time you may get a very uniform mix of different particles. Sorry the different materials that you have put together. So this is one particle that may be that you may obtain which is very homogenous in nature.

So over time you will get a very homogenous mix of materials. So this is a method which is very widely used to get the alloying of the material and getting homogenous distribution of the materials that you have used. Mechanical alloying provides a homogenous distribution like a set initial mixed particles form laminated structure and again it is also shown over here and we have shown it on the board also how the process is going.

Allows for high speed milling compared to ball milling. Now here we are allowed to go to a much higher speed which means you will be able to do it at a much higher rate or you will be able to mill the powders at a much higher rate and that is because here there is no problem of centrifugal forces there are powders mm milling media are already on the bottom and if you keep rotating it they will still be enough to fill that lower levels and your particles will also be on the lower side and therefore this attritioning motion will continue to take place and you will get reduction and alloying of the material.

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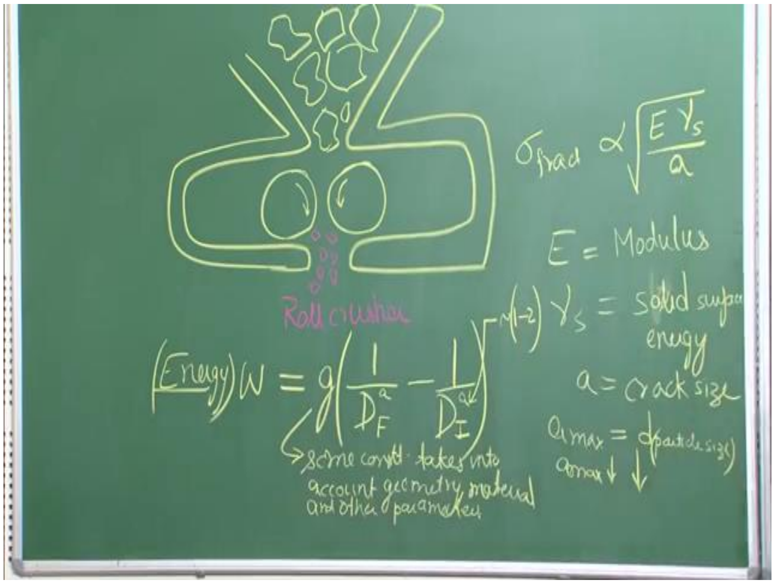

Now in general we can write a simple equation which describes how much energy is required for fiff energy is given by this W, a rough estimate of this energy that is required to reduce the particle size from D initial to D final is given by this equation. DF is the final particle size, DI is the initial particle size and g is some constant it is not gravity. It is some constant which takes into account geometry material and other parameters. Other parameters like what is the milling medium.

And ‘a’ is a exponential factor which varies between 1 and 2. Again it depends on various processing parameters. So the value of ‘a’ varies between 1 and 2. And this gives you the overall energy that is required for milling, there the DI is the initial particle size and DF is the final particle size. So you have reduced from this particle size to this particle size. And this will give you the total energy that is required.

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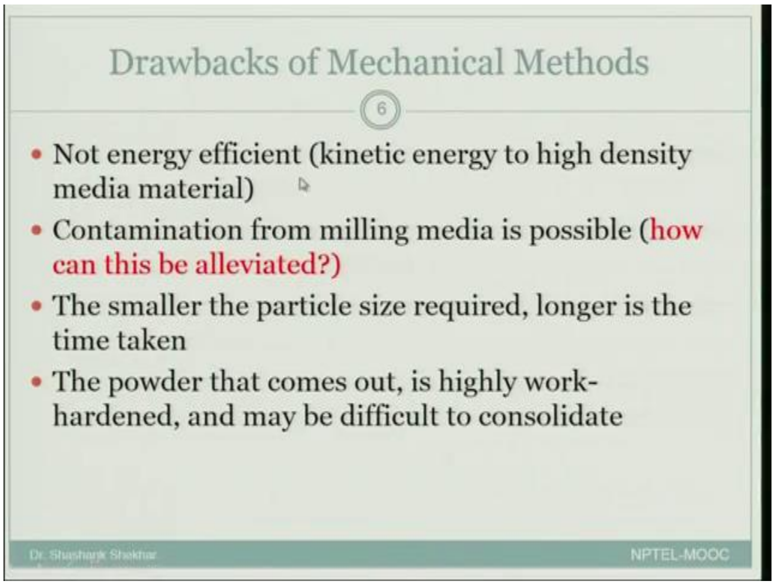

So let us now, leh take a look at what are the drawbacks of this method. We have looked at couple of mmm mth mechanical methods which are used for reducing powder size. So what are the drawbacks, there we have already discussed some of them and here is a summary of it.

The most important being that it is not energy efficient kinetic energy is provided to high density material and not all of that energy is getting translated or getting used for reducing particle size. Infact a very small fraction of it is being used for reducing particle size. Most of it is being used in just creating sound and heat. Contamination from milling media is possible let us say you are reducing powders of alumina al2o3 but you are using a milling media of iron iron balls.

So the iro it is quite possible that some particulates of iron ball may fall into the overall powder mixture and your alumina may get contaminated by these iron balls, or these iron particulates. The smaller the particle size required longer is the time taken. So although there is a saturation limit below which you cannot reduce powder size but even to reach that saturation time or to reach that saturation powder size it will take you very very long time.

So usually people achieve try to achieve only 70 or 80 percent of the particle size and take out the material. But if you want to go to your final particle size then it will take a much longer time.

The powder that comes out is highly work hardened as you can realize that the powder is very highly work hardened because you are doing so much work onto this.

A lot of deformation has taken place and therefore at many a times this is this powder may be difficult to consolidate. Because it has so much work hardening it may be difficult to consolidate. So the these are the some of the important drawbacks of the mechanical method. In the next lecture we will be discussing some other manufacturing techniques for powder. In the meantime take a look at or try to understand the two mechanical methods that we discussed today, thanks.