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Lecture - 3 Nanocrystalline Materials Part – I

We are all aware, that this is one of the upcoming topics. Particularly, for the last, I would say the origin is basically, I would say from a paper in 1981 by a person by name Herbert Gleiter. So, Gleiter from Germany was can be considered as a pioneer in the nano materials. And they have made nano materials of various metals and ceramics starting from 1981. The initial work was on pure metals, by a technique called inert gas condensation technique.

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We will know more about it, as we go to various techniques; that are available for a production of nano materials. So, basically, inert gas condensation, as the name suggests, is basically a gas condensation technique. You make the metal in the form of a vapor and condense the vapor onto some kind of a cold substrate. And while condensation, this vapor come together and form the nano crystals or you can call them as clusters, depending on their sizes.

And you get these things deposited onto a cold substrate, which is usually referred to as cold finger in an inert gas condensation technique. And you basically, scrap this nano powders, from the cold finger and collect them and do whatever you would like do. If you want to use them as nano powders itself. You can use them as nano powders or compact them to whatever shape, you want to have it. And then use them as bulk materials.

So, this was the technique, which has come up primarily due to the work of Gleiter in 1981. And then it has picked up a lot of momentum and a number of people all over the world, including India, people have been working on this. Basically, if we look at nano materials, we have been talking about micro structure property correlation in the last class. So, particularly, the basic difference between a nano material and a micro material or a bulk material is basically that the grain sizes are much smaller then what you usually encounter in a typical material.

For example, if we are talking of a steel. The steel usually, when we talk about the micro structure, the ferrite grain size, they are of the order of around of 100 micron's or so. And we do some special treatments for these steels to make them finer. For example, you are aware of high strength to low alloy steels HSLA. In HSLA, we try to refine the grain size, by putting some pinning agents at the grain boundaries. For example, some carbides and nitrates, which can prevent the grain growth, during the grain growth stage, after the re crystallization.

When you take the steel, heat it to high temperature, we know that whenever you heat a cold work material, there are three stages. That are involved the recovery crystallization and grain growth. And that grain growth stage, which is the third stage, can be controlled by putting some hard particles at the grain boundaries, which act as pinning agents, for the grain boundary to move.

So, if you can somehow prevent the grain boundaries to move, you can control the grain size. This was the technique adopted in kind of in high strength to alloy steels. There also, many other techniques, which are available to reduce the grain sizes. For example, one technique is deformation itself. When, we deform a material, cold work a material, we know the grain get elongated and the sizes decrease and then followed by a proper heat treatment. To control the grain size, if you can stop it, at recrystallization stage itself. So, that, you can get a fine grain material.

And during the production of the material itself, the most important technique that we used to produce materials in metallurgy is basically melting and casting. So, in the melting and casting technique itself, there are a number of techniques available to control the grain sizes, which is for example, what is called a vibration. We must have heard of techniques, where the whole mould is vibrated, thixocasting, reocasting techniques.

Where, you break the dendrites, which are growing in the liquid into the form of a small fragments and these fragments, when they finally meet each other, you get each one of those fragment becomes one grain and you get fine grain structure. That is one of the technique and the other technique; we all know is the cooling rating itself. During solidification, if you can have a high cooling rate, we know that the cooling rate has a direct co-relation to the under cooling, that we can achieve.

And if you have high under cooling ultimately, so that you know, that the cooling rate. If you increase, the under cooling increases and if the under cooling increases, we clearly know, that the activation barrier for nucleation decreases. And the critical size of the nucleus also decreases. We know these relations from our metallurgical principals, that r star is nothing but minus 2, gamma by delta g v.

In our thermodynamics class, we have learnt this and in the kinetics class, also you come cross this. So, and delta g v is directly proportional to delta t. We all know delta g v is nothing but delta H f into delta t by T f. So, from that, we know that, delta g v is directly correlated to the delta T and r star is obviously inversely related to the delta T. So, as a result, if you can have high cooling rate, we can have high under cooling and that reduces the r star.

And at the same time, the delta G star, which is nothing but 16pi by 3 gamma cube by delta g v square. This also you are all aware off. So, here also, you can see the delta g v term is coming in the denominator in the square term and because, it is coming square term, obliviously there will be a delta t square coming into picture. So, as a result, delta G is star is inversely related to the square of the delta t. So, both r star and delta G star are inversely related to delta t.

And we know that, the delta G star is the one, which decides the nucleation rate. So, if you take the nucleation rate I, nucleation rate is nothing but some pre exponential term multiplied by exponential delta G star by R T. This is, in a nutshell, but we have to have

the diffusivity term also to be added there. Let us not go into that at this moment, but basically the delta G star, which is the activation barrier for the nucleation controls the nucleation rate and if the smaller the activation barrier, the larger than nucleation rate.

So; that means, if you can have a high cooling rate, one can have a small nuclei large number of them. And that is again controls the grain size. That is, one of the technique by which people, use for example. Rapid solidification processing, which we are going to discuss a little later is one of those techniques, which has been used by people to refine the grain size.

Even, you know from our typical foundry techniques itself. That when you go from a sand casting to a metallic mould, from there to a copper cool water cool, copper mould. So, you can have higher and higher cooling rates and finer and finer grain size. So, this is known to every metallurgist. And we also have another technique, which has become very popular, from almost last four decades, also which is called inoculation.

That means, take the liquid metal, add some kind of nucleating sites into it, which is, what you call usually heterogeneous nucleation. So, if you can add a heterogeneous nucleating site. And if the site has some kind of a conformability, with the solid, that is going to nucleate. You can have large number of nuclei coming out, depending on the number of nucleating sites. That you have.

So, whatever is the number of active nucleating sites, that you have in the liquid metal, that is going to decide, ultimately, what is the grain size, that you going to get. So, if you have a numerous number of fine nucleating sites available. If they are all active, then you have large number of these grains forming and ultimately, the grain size will be finer.

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That is true. So, that, you can control it, if you can somehow control, if the whole liquid is having a large number of these nucleating sites. So, what happens is the grain gets nucleated on that and as it tries to grow the other grain, which is nucleated on the other grain acts as a hindrance. So, you have a physical barrier for growth of these grains. So, as a result, ultimately you see the two grains come and meet each other and finally, you get fine grain structure.

So, one way is to have a large number of such nucleating sites, which we ourselves have done it in our department. And we have seen, we have developed the number of these grain refiners, which are called. And these grain refiners basically depend on the type of metal. Because, it depends on the type of metal, what is the type of nucleating site that we can use for a particular metal? For example, if it is aluminum, aluminum structure is f c c.

So, you need have a nucleating site, whose, which has some of compatibility with the f c c structure. That means the surface on which the nucleation is taking place, should be compatible with the structure. That means, some atomic planes in the f c c structure, should be having similar atomic arrangement, with the surface of, with the atomic plane, which is parallel to the surface of these nucleating sites. So, that, you can have a nucleation possible there.

This is similar to epitaxy, we talk about, when we are talking of thin film deposition. But, the problem is, with all these techniques that we have talked so far, which are techniques for grain refinement. You can never achieve grain sizes below about 10 microns are so 10 microns, maybe 5 microns. But, if you really want to go into a few nanometer range, you need to really go into newer techniques.

And these techniques are basically; either gas condensation will come to them, a little later. And also, there are techniques, for example, high energy boll milling techniques are available or precipitation from the liquid state, we can do it. And also solidification at very high cooling rates, not as we usually observe, during our foundry techniques. But, at rates, which are much higher, like what we usually see in rapid solidification processing, such as melt spinning and so on.

So, such techniques, can lead to these nano materials. But, the basic starting point for this, could be, how do, we define this nano material. As everybody talks about, everybody says, can we say even a 10 micron material, call it as 10,000 nano meters and call it as a nano material. Just because, we are telling the dimension in the time in terms of nano meters or is there any limit, below which we can call it as a nano material.

The usual limit people take it is about 100 nano meters. But, this 100 nano meter is not a real sacrosanct number, basically because; it all depends on the type of material. So, in principal the definition of where you would call it as a nano material is that size, below which some property, significantly changes. If you talk in terms of a, let us say optical property.

There are some materials, such as barium titanate, which are usually opaque. But, the moment you take them below a certain crystallized size, they become transparent. There complete optical property changes from opaqueness to transference. And similarly, there are some materials, whose magnetic properties change. And in case of metallic materials, we also know, that there strength changes significantly, in some of these nano materials.

We also have come into regime, which we will talk a little later, where the size is so small. That probably, it will not lead to any increase in the strength, which is, we call it the reversal of the hall-petch. We will talk about it little later, where the grain is so small, that the hall-petch relation really cannot be used there. And then you can have, what is called a softening effect there.

So, we will also talk it, there some cases where people have proved it, but it is not still proved as a universal phenomenon. So, still we can say there is a debate going on, whether really a softening occurs or is it system depended and probably in some systems it happens and some systems does not happen. This is something has to be proved it future.

But, basically if you look at this picture, what you see is, that any material is made up of the grain and grain boundary. So, and if you take a material, which is basically made up of polycrystalline materials, of course, I am not talking of single crystals. So, if I am talking of polycrystalline materials, you have obviously, grains and grain boundaries.

And usually in most of the polycrystalline materials, which we commonly come across the grain boundary is very thin. And the volume fraction of the grain boundary, in terms of the total volume of the material is very small of the order of around one percent or so.

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Usually, if you look at the volume fraction of the grain boundary, the volume fraction of the grain boundary almost raises kind of exponentially.

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If you talk of volume fraction of grain boundary, other functions of crystallized size and below around 10 nano meters or so. The size almost reaches around 5th; the volume fraction almost reaches about 50 percent. You see here, we have plotted the grain boundary volume fraction, the triple lines volume fraction, quadruple notes volume fraction.

All of them give you an idea, that for example, if you look at the grain boundary, it reaches, almost about 50 percent. When, you reach below about 10 nano meters, around 5 nano meters or so. How do, we calculate this, simply assume that, you have a grain in the form of, I say, as there as a square or spherical particle. And then imagine a grain boundary thickness of the order of around, let us say, a delta, let us say the size of the grain as d.

One, can calculate, what the volume of this grain boundary is by simply finding out the total volume minus the volume of the grain. And this is, inversely proportional to the grain boundary volume fraction, grain boundary percentage. If you say, this becomes inversely proportional to, if you derived the whole expression; it becomes inversely proportional to the d, the grain size.

If you use about 10 nano meters, you reach for a grain boundary thickness of the order of around 1 nano meter. Usually, grain boundaries have a thickness of about 1 nano meter. In principal, one has to also consider, that as you decrease the grain size, the grain boundary thickness itself keeps on changing. And it keeps on increasing, when you decrease the grain size below around 10 nano meters or so. Beyond, about 50 nano meters, the grain boundary thickness does not change that much.

But, if you go to really very small grain sizes, the grain boundary thickness also will be larger. And as a result, this delta is not a fixed number, but for all practical applications, usually people assume the delta to be around 1 nano meter. And if you take that, with this kind of a rough formula of grain boundary percentage volume fraction is about 3 delta by d. It will give you an idea, that about 30 percent you get, when you have about 10 nano meters, 33 percent or so.

And if you come to around 5 nano meters, you will see that almost you get about, how much, if you come to about 5 nano meters, what would be the volume fraction from this.

Student: sixty percent

Sixty percent, so you can imagine that, you have now a material with about 50 percent of grain boundary. So, the whole property of the material is going to be decided to a large extent by the grain boundary and not the grain itself. So far we use to think off the properties of material, basically depend on the grain structure. And all for example, the

hall-pitch relation everything, we talk in terms of the grain boundaries, acting as the obstacles, for the dislocation moment.

And then so everything is dependent on the just a grain size, without considering much about, what is there in the boundary and how does this boundary affect the properties. And we usually know that, the grain boundary is basically a disordered region. When compared to the grain, as you can see here these black regions are all the grains, which are ordered arrangement of atoms. And the grain boundaries, which are basically disordered region.

In fact long back, when Gleiter has discovered these nano crystals, he even said, these grain boundaries are gas like structures. Though, people have proved little later, using high resolution microscopy. That there is no such a gas, like structure, really in the grain boundaries, but definitely, there is some kind of a disorder there.

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So, because of that, disordered the properties of such a boundary is going to be different from that of the grain and once, the volume fraction of such region becomes very large. So, one has to consider, how the properties are going to be effected with such a boundary. And usually, we take the property as just as a roll of mixtures. For example, if we know the property of the boundary, any property, for example takes it as x.

If we know the property of the grain and the property of the boundary, one can take it as a roll of mixtures as the volume fractional of the boundary, multiplied by the property of it, plus the volume fraction of the grain multiplied by the property. But, before that, one need to have an idea of, what is the property of the boundary. So, for that, one need to do, how do you think, one can get an idea of, what is the property of such a boundary, is there anyway.

How do you know, the property of a, let us say a grain. If you want to know, individually, these two properties, any mechanical property, for example, metallography, gives you the properties, metallography, gives you only the structure.

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Correct.

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Correct. So, with the new techniques that is available, such as nano indentation techniques. One can get an idea of the boundary properties, without much problem. And one has now sophisticated techniques to visualize these boundaries; we have high resolution electron microscopy available to see the grain boundaries. We have also atom probe field ion microscopy available to see, how the atoms are arranged in such a grain boundary.

So, such sophisticated techniques are available to see the structure and also get to know, what is atomic distribution? What is a composition of such a grain boundary? Using techniques, such as atom probe field, ion microscopy APFIM, people call it are three dimensional atom probe, three DFAP people usually refer to as. And such techniques can be used, to find out the composition on structure. Structure can be obtained from high resolution electron microscopy as I mentioned. And one can get the properties, using techniques such as nano indentation.

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So, these are some of the techniques, which can give you an idea of the properties and this only go into the properties, we need to understand, what are the various types of such nano materials that are available. Usually, we broadly divide them as three types. In fact, I should say, even in the 4th type, because the clusters are usually called as zero dimensional nano crystals.

If you take a few atom clusters, which can be formed, because of the evaporated atoms coming together, forming few clusters of few atoms, such clusters are usually referred to as the zero dimensional, nano crystals. Then you can talk of one dimensional nano crystals, such as, if you have nano wires or nano tubes, people are talking about carbon nano tubes. So, you are all aware off.

So, such nano tubes can be really called as one dimensional nano structures and one can have two dimensional layered structures. You can have multi layers of nano crystals and one can also have three dimensional equiaxed nano grains. So, these are the four basic classifications of nano materials. But, now, one can have further classifications depending on, whether it is a single phase material or a multi phase material.

For example, as you can see here, that if the composition, chemical composition is the same, if you have two phase material, let us say you can still have layered structures with each layer having the same composition. But, they are deposited as layered structure. So, one can have layers of the same composition, one can have a rod shaped nano crystals,

which are of the same composition various rods brought together or various wires of nano wires brought together.

That is, one possibility and you can have the grains of nano crystals various grains each one having the same composition brought together. One can also have different compositions, again similar possibility of a layered structure or rod like structure or an equiaxed grain structure. Where, you can have different compositions, each grain has a different composition.

One can make various nano crystals of various grains and various compositions bring them together and compact them and you will have a heterogeneous property. And depending on the application, one can think of such of thing. And similarly, you can have the grain boundary composition, different from that of the grain. For example, if you have a layered structure, the composition of the layered region, can be a different.

So, one can have such materials, with the composition, at the boundary being different from that of the composition in the interior. And similarly, you can have nano crystals of a composition dispersed in a matrix. For example, nano composites, which people talk about nowadays, we can have a matrix. In fact, that is, what has made the definition of composite itself is very wage. Long back, we used to define a composite as something, which you physically introduce.

The reinforcement into a matrix, for example, was starting from a concrete or even a bone, for that matter. These are natural composites for example; wood and bone are natural composites. And we have the first man made, kind of composite probably is a kind of concrete. So, we are all aware of concrete, which we see day to day life. But, at the same time, these are all real macro composites.

But, we have come from that a long way into the nano composites now, where you can have a very fine particles of the nano material regime in deposited or dispersed into a matrix, even the matrix can be a nano material. So, you can have, such a region that materials having nano size, with different properties can be brought together. And compacted and you can have a composite is not only that you have a matrix, with a particles or you can have nano grains of two different materials, can be brought together.

And you can have a composite that way. So, the definition of a composite itself can be has to be looked in carefully. That any materials, with more than, one phase, in principle, people have started calling them as a composite. If that is the case, even steel for that matter, should be called as a composite. So, because, you have more than two phases, more than a single phase there and such a material can be called as a composite.

So, what is important is, that you have two phases; each one has a different composition or different structure, basically different properties and brings them together. So, that you get a property, which is better than both of them. So, that is what is going to be the composite.

And how do we make them, we can make them by various techniques. Basically, from three sources, one is you can have it from a vapour source. Second, you can have it from a liquid source. Third you can have it from a solid source.

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So, if we take nano crystals can be synthesized basically from a vapour or a liquid or a solid. When, we talk of a vapour, basically when you take a liquid and evaporate it, what you get is, either atoms in the vapour form or molecules, which are dispersed in the space. And if you can bring them together by a condensation technique, you can get those clusters.

You can see, the single atoms can be condensed to form clusters and those clusters, if you compact them, you can get nano material, three dimensional nano materials. So, that is one technique, which is, what we call cluster assembly. And such a technique is, what is the IGC technique, which I have told you inert gas condensation technique, which is regularly, used by many physicist particularly. But, the only restriction of such a technique is, you cannot produce all kinds of materials.

First of all, you should be able to evaporate something and then form in the form of vapours. Particularly, high melting metals, it is very difficult to make, such nano materials, using such a technique. And what also is important is, first of all, before you evaporate, you have to evacuate the whole chamber. So, for evacuation of the whole chamber, so if you want to produce large quantities of materials, you need to have large chambers and these large chambers have to be evacuated.

So, as a result, so there are limitations, in terms of the capital investment that is required for such a technique. And the total quantities of materials that you can produce by such techniques are not really large. So, in India at the moment, there is only one place, where you have such a IGC technique. That is in university of Madras in the nucleus physicist division one, professor S Rama Swami, he has been working on this and he has developed an IGC instrument. Fabricated it here itself and he has been making a number of pure metals.

And also some of said, this is another advantage, that you can evaporate a metal and have some kind of an oxygen atmosphere. So, that the metal vapours react with the oxygen and you can have a nano particles of oxides deposited. And any other, maybe you can have a nano particles of nitrates deposited, so by controlling the atmosphere. So, one can have such reactive synthesis possible in the vapour stage, where the gasses atmosphere reacts with the nano particles. So, this is basically a vapour condensation technique.

And once, they are condensed onto basically, you have a chamber, with a cold finger, which is basically nothing but a tube with a liquid nitrogen, which is circulated through this tube and the tube is rotating. And you have a kind of a scraper, which scraps off, all those nano particles or the tube rotates. So, as the vapours get condensed, under the tube, which is at a much lower temperature and the scraper scraps up all those nano particles which drop into a collection chamber.

And you can have a compaction facility. So, that, before exposing these nano particles to atmosphere, you can even compact it, there itself. So, there are facilities, such as compaction within that chamber itself. So, that everything can be computed, either in organ atmosphere or in vacuum. So, that the particles are not exposed, because you know that nano particles have a very large surface area to volume ratio.

And because of which, once they are exposed, they may even become pyroferric. That they may even burn or they may get oxidized and their properties might change. So, as a result, one has to take special care in handling these nano materials. Another possibility is that, you can have these nano particles, which are formed dispersed in some kind of a fluid. Have you heard of Ferro fluids?

So, magnetic Ferro fluids, where the magnetic particles are dispersed in a fluid, which is some, kind of a surface active agent. So, that it does not bring in the nano particles together for agglomeration. It prevents the particles from agglomerating and so that, these nano particles are uniformly distributed in a liquid suspension. And these Ferro fluids are being used nowadays, for the memory, for enhancing the memory in the computers.

So, there is a lot of work going on such Ferro fluids. So, the whole liquid behaves likes a magnetic material now. Because, you have so many nano particles of these magnetic nano particles dispersed in a liquid like that. So, this can be done by such a technique. Second route is from the liquid, when we say from the liquid, there are two possibilities again. One is, you can take a liquid of a particular alloy, rapidly solidify it. So, that, you provide a large under cooling. So, that, you get a very fine grain structure.

There are number of alloys, where people have been able to make nano crystals by such a technique or the other possibility is that, rapidly solidify a liquid, to get a glass and then heat this glass. Because, we know that glass is always a meta stable material, meta stable structure. So, you can reheat this glass in a controlled fashion. So, that, some nano particles can precipitate out of this glass, because crystallization of the glass.

So, if you can cool the liquid rapidly, such that, the cooling rate is higher than the critical cooling rate. That is necessary for preventing any crystallization during solidification. So, you can get a glass as long as you come below the t g. Once, you come the below the t g, you have a glass, take this glass, heat it again. So, that, you can have a uniform distribution of fine nano particles.

That is another technique, but these are all basically used for mostly for metallic systems. But, there are also other systems, whereas for example, you want some oxide nano particles, from a liquid route. There are a number of techniques that are available, for example, sol gel techniques. So, solution, gelation, people call it. So, where, you have basically a liquid and you bring the conditions to such a state.

That some precipitation occurs, either by heating or by adding some other reagent, which can breaks this solution. To give out the ions and which get accumulated and form nano particles and get precipitated out. This is simply just precipitation from liquids. There are many such precipitation techniques available, most of the chemist, use such precipitation techniques to produce nano materials.

Here, one advantage is, you can really do on large quantities. If you can take few kg's or even tones probably one can think of only thing is, one have to control the conditions. But, the only limitation of this is, most of the chemical reagents people use are really toxic. So, because of which, so you have a problem of environmental pollution, so how to take care of these affluence that come out of it, the byproducts that comes of it.

But, hopefully, people will come out of some solutions for that. So, you have such chemical precipitation techniques or electro deposition techniques. We know electro deposition, we use for plating. So, same electro deposition technique can be used by controlling the current conditions and the voltage conditions and the concentration of the medium. Such that and the temperature of course, such that, you can have nano particles deposited on a substrate.

Particularly, when you want to have high ware resistant surfaces on any substrate, you can have such electro deposition techniques. You can also talk of lasers, where again, you take a bulk material, melt the only the surface layer and the cooling rates being so high. That you achieve rapid solidification conditions, and finally nano particles are deposited on that.

And when you melt it also you can introduce some other material, for example, laser surface modification or laser surface alloying people talk about. So, once, you melt the top layer and then introduce some other material. For example, simply, if you are melting a titanium by laser in a atmosphere containing nitrogen. Immediately, this liquid, titanium, which forms, can react with the nitrogen, which is there in the atmosphere and can form a titanium nitrate coating.

Thin coating of titanium nitrate, probably if the conditions are fine enough, you may have them in the nanocrystalline form. So, that, you can have a hard nanocrystalline layer on the surface of a otherwise a ductile material or otherwise a soft material. So, one can have a soft bulk material with a hard coating of nano materials on it. That is from the liquids. So, these are all the techniques from the liquid.

Then, the third possibility is from the solids. How do we do those from the solids, break down a macro material into a nano material. So, take a macro material, such as powder. So, let us say, atomized powders of the order around 100 nano microns are about 300 200 microns particles and such particles take them, put them in a ball mill or any another technique, where you can really break the particles into finer and finer size, this is similar to just communition, which we do in a mineral processing. Only thing is there, the energy levels are so small to really take these particles to the nano size. But, there is a number of high energy milling techniques available, where one can really make nano particles out of it. The only difference between this technique and the vapour deposition is, here it is what is called a top down approach.

Top down approach means, you are starting from a macro material and going to a nano material. In a vapour deposition it is like a bottom up approach, you are starting from atoms, and then building the atoms together and then forming a nano crystal. So, both are two different routes. And liquid is, somewhere you know it falls somewhere in between, basically liquid has a homogeneous mass, which on cooling you are developing either on cooling or on deposition, you are developing nano crystals.

And both this routes depending on the route, people have found that the properties could be slightly different. The same nanocrystalline size, if you make it, from a gas condensation technique or by a ball milling. The properties could be slightly different, why do you expect the properties to be different? Sizes are the same. I have made a nano crystal of about 10 nanometers or 20 nanometers of a pure iron.

Let us say made by ball milling and the same pure iron made by gas condensation compacted both of them study their mechanical property. And I find that there could be some differences.

Student: ((Refer Time: 41:41))

The defects, inside the material would be different and because of the grain boundary structure could be slightly different. So, definitely, just a size itself is not the answer. So, size controls the properties to a large extent. But, the grain boundary structure also has a lot of role to play. So, one has to consider that also into picture. We will stop now stop.