Nanotechnology Science and Applications
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Lecture - 09
Experimentally Investigating the Hall-Petch relationship

Hello. In this class, we will continue our discussion on mechanical properties associated with the nanoscale. In particular, we will look at the Hall - Petch relationship which is what we are investigating in greater detail and we are interested in knowing what is the impact of the nanoscale on the Hall - Petch relationship.

So, today what we will look at in this class, what we will look at is some experimental aspects associated with this investigation. So, for you to understand what you are accomplishing by going to the nanoscale, we have to do some experiments and so that experimental aspects of it and how you handle it is something that we will look at. And it also talks a little bit today, we will look at the material synthesis aspects of it and as I have mentioned in the context of each of these studies that we will discuss through this course, we will look at those relevant material synthesis processes rather than look at them independently.

Because then it gives you some sense of context and some sense of why it is being done in a certain way, in a certain place and so on. It also tells you that if you were to make that kind of a sample for some other purpose, you may encounter these nanoscale effects. So, that is the idea that we would like to explore.

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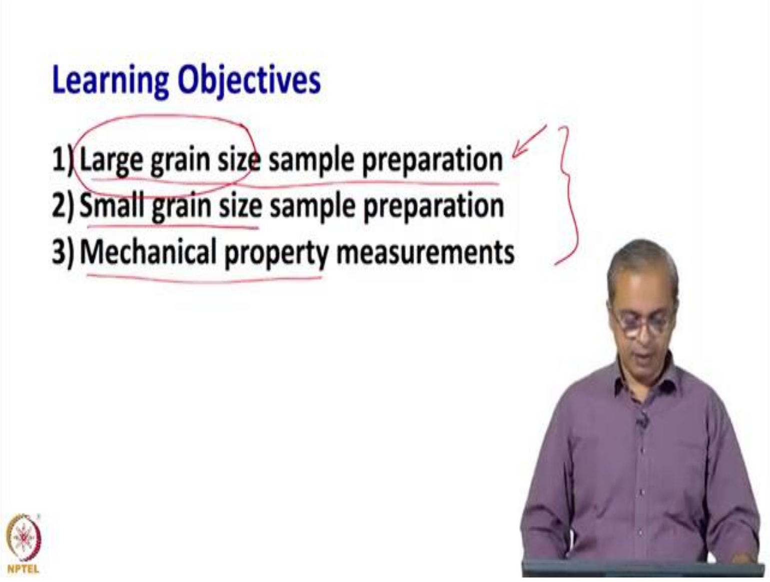

Learning Objectives

1) Large grain size sample preparation
2) Small grain size sample preparation
3) Mechanical property measurements

So, our learning objectives for this class are to look at how you would go about getting some kind of consistent large grain size samples that we would need for this kind of activity and this is particularly necessary because you need to have some frame of reference. So, when you say that you are seeing some nanoscale effect, you to convince yourself and therefore, to convince anybody else who is looking at that work that you are seeing a nanoscale effect, you also have to make similar composition samples in the non-nanoscale which is basically in the macro scale or micro scale so to speak.

So, in that context relatively large grain size is what we are looking at; relatively large grain size samples and how would you go about preparing them. Then, once you have that and if you have like a series of them, then you can understand with different grain sizes, then you can understand what is the impact of the grain size on the strength of that sample. Similarly, we also need to have a series of small grain-sized samples again it is a relative sense, but in this case the small and the large essentially are referring to the nanoscale in the small grain size range and a distinctly non-nanoscale in the large grain size range.

So, we also need to look at techniques which will get us this nanoscale grain-sized samples so that we can look at the trend in their mechanical properties as a result of the crystal size. And of course, importantly if we are looking at mechanical property in the context of this study, this kind of a work we also need to know how would you go about measuring that mechanical property? What is a good way to go about measuring this mechanical property? And which you can then, consistently use across these samples which are nanoscale in some case, non-nanoscale micro or macro scale in some case.

And then, get all the data together so that you can see a more unified picture and then, therefore, eliminate any experimental issues associated with the process and then, see if there is a trend and then make some understand try to get some understanding of what the trend is and why you are seeing a certain trend.

So, this is the idea here and since in this class, we are looking at the experimental aspect of it. In all of these cases, we are essentially going to focus on the experimental part of how you would get the large grain size; how you will get the small grain size and how you would make this mechanical property measurement.

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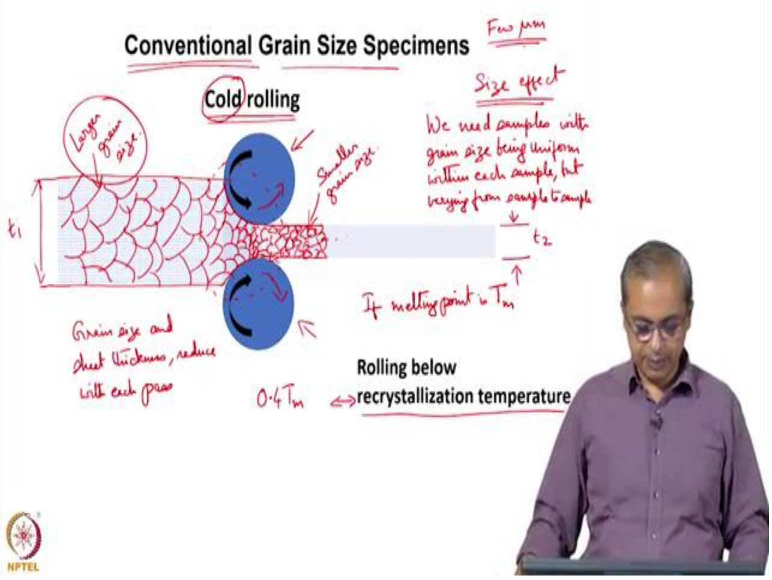

Conventional Grain Size Specimens

Cold rolling

Rolling below recrystallization temperature

So, if you see the larger grain size sample preparation process what we would also otherwise maybe call a conventional grain size and the reason we say conventional grain size is because if you go to a typical hardware store or typical supplier of materials, chances are they are already giving you a sample that it is that is in this grain size range. And therefore, that is the typical material that you are going to get and that is what we are referring to as conventional grain size specimen. Please also understand that in this study, we are interested in the effect of size.

So, therefore, we need samples with grain size being uniformed within the sample. But varying from sample to sample. So, that is how you will see a size-effect. So, even if I say a 1 micron-sized sample. I need maybe 10 samples; one at 1-micron size; one at 4-micron size; one at 10-micron size; one at 20-micron size like that. Where, within that sample almost all the grains happen to be 1 micron or almost all the grains happened to be 5 microns or 20 micron or whatever it is and then so, I have these 10 samples.

Each one has the mean grain size being one particular value with minimal standard deviation and then, another sample has another grain size with many much standard deviations. So, this is what we want to do. So, for conventional grain size samples, we usually given that we are looking at metallic systems in this kind of work. You could look at other conditions, but metallic systems is what we are interested in this case. Cold rolling is a good way of going about it.

So, rolling is actually a very conventional process, metal forming process that exists in the metallurgical studies and it also is used extensively in the manufacturing industries, in automobile industries. So, if you go to any plant which is associated with providing sheets of metal, any metal in sheet form and lot of metals are provided in sheet form; you see if you go to hardware stores, you can get rolls of metal in metal rolled up in sheet form and this is used for a wide range of things.

So, any automobile body part is a rolled sheet and so, rolling is a very common process in the metallurgical industry and it basically has this idea that you see here this schematic that you see here. You have two rolls here one here and one here. So, they simply roll, they are cylindrical in shape cylindrical shape. So, you are seeing from one side of the cylinder. So, you see in the circular cross-section and then, you feed the sheet.

So, there is a cylinder; there is a cylinder and then, you feed the sheet and then, based on the gap between those cylinders. You squeeze the sheet and it comes out as a thinner sheet. So, that is basically what you are doing. So, you are having a sheet this is all exaggerated because in dimensions because this is a schematic. So, you have a sheet with this much thickness beginning to begin with. So, this is t 1, I will say thickness 1; it comes out with this thickness t 2. So, and this is the; this is the range.

So, you can see both these rolls are rotating in this direction and therefore, pushing that material through this process and what normally happens is there is this transition region here and it is in this region that it slowly gets squeezed and comes out as the squeezed material. Now, generally, when this happens, you will see that what is coming in here this is I just put some schematic here. But this may be some large grain-sized material. So, grains may be like that and here, you may have some small grain-sized distinctly smaller grain size.

So, that continues in that direction and in the transition region, slowly it goes from larger grain size to smaller grain size. So, that is basically what you start seeing. Yes, so, this is basically what you see. So, you see something that starts off as a large grain size material out here and then, as it gets squeezed it slowly becomes a small grain-sized material and comes out this way.

So, this is a typical rolling process and there are some restrictions. I mean this is a schematic. So, it looks like you can take a very thick material and roll it down to very thin material. Normally, and in fact, it is true that you can eventually do this normally you would not do this in a single pass each time, it goes through it is a pass. So, you send it through and you get it out to the other side. You normally will not do it in a single pass. There are restrictions on what you can do in a given pass given these stresses that are involved given the kind of behavior of the material and so on.

So, if you want a nice smooth sheet coming on, there are some restrictions on how much you can reduce. So, reduction per pass; there will be some restrictions. So, within those restrictions, you can do it, but you can continue this repeatedly. You can make one pass and then, you keep the rollers apart to some distance you run one pass and then, you move the rollers in slightly closer run the second pass; even closer and the third pass and so on.

And so, progressively it gets you can make it thinner and thinner and thinner. With every pass, the grain size that goes in will get reduced to a smaller grain size that comes out. So, you will get a smaller grain size here that is basically what you will have. Smaller grain size that shows up here. This is larger grain size. So, grain size reduces with each pass grain size and sheet thickness. So, grain size and sheet thickness both reduce with each pass.

So, this is how you will get something and therefore, as I said if you want a series of samples one which is a 20-micron grain size; another at 15, at 10, at 5, at 2, at 1 and so on. So, you start with a large grain size material, you roll it down and you see when you get 20 microns on average grain size. So, that is one sample. Then, you take another piece of that same sample and you roll it even further. So, you get a smaller grain size and so on and then you get this range of grain sizes.

So, this is how you would get this set of samples. Now, of course, it is also useful to keep in mind this idea that this is called cold rolling and the reason is this we are not deliberately heating the sample. More specifically, I mean even if you do heat it more specifically you do not heat, it beyond a certain point a certain temperature and that is called the recrystallization temperature. So, typically for more most metals, this temperature is approximately related to the melting point of that material in a certain way.

So, if you have a melting point, if the melting point is T subscript m, then this recrystallization temperature is typically of the order of 0.4, 40% of the melting point. So, sometimes it is 30%, sometimes 40%, sometimes 50%, so, 0.3, 0.4, 0.5 in that range of the melting point and that is the recrystallization temperature. You have to stay below this. What happens is if you cross this temperature, the small crystals that you have created they actually have higher energy.

Because you have created more grain boundaries and as grain boundaries have bonds that are not satisfied. So, if you have more grain boundaries that is a higher energy state relative to the crystal sizes which are large which, therefore, have less grain boundaries compared to the volume and if you take it to high enough temperature, 3-4 of the smaller grains will coalesce, come together and form one larger grain.

So, you will go back to the kind of grain structure that you have here. If you go to higher and higher temperatures, you will keep getting this grain structure; the sheet may be thin, but the grain structure will be large. So, if and if in some cases that is what you desire decide in some cases maybe that is what you desire decide. Then, that is. In this case, you want that small grain size to remain and therefore, it is desirable that you do not take the temperature above the recrystallization temperature and you stay below the recrystallization temperature.

And above the recrystallization temperature, the grains sort of recrystallize into bigger grains and that is why it is called the recrystallization temperature. So, this is the experimental process for getting us getting ourselves some samples with known crystal known grain size, but on in a relatively larger regime of grain sizes of the order of few microns.

So, few micrometer-sized grains is what you are looking at some range there. So, this is one set. So, let us assume that we have know about one set of samples of this nature. We will now go to the other extreme of this spectrum, we will look at nanoscale samples how would we go about getting those nanoscale samples.

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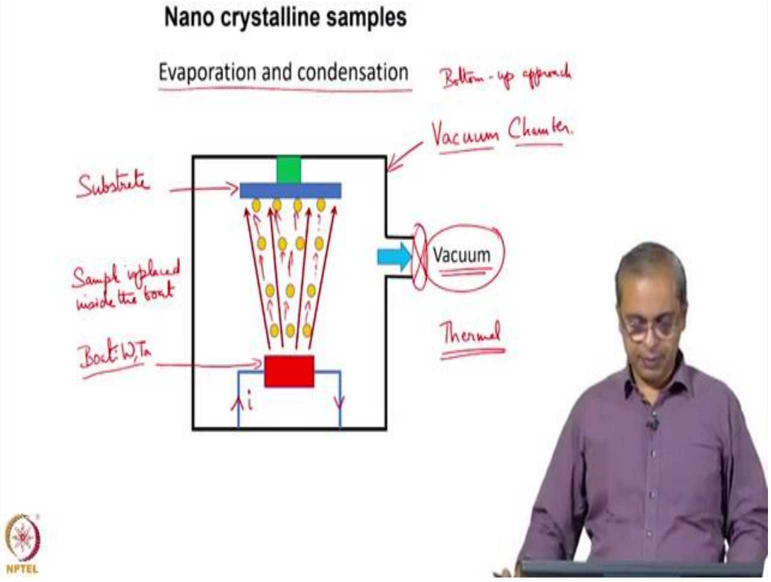

Nano crystalline samples

Evaporation and condensation

So, the general process is evaporation and condensation. So, in some sense when we looked at the rolling process ok, so the rolling process you are starting with a material which is large and then, crushing it into a material that is smaller. So, in some sense the rolling process that we saw is a top-down approach in some sense in some ways this is a top-down approach.

So, you are sort of breaking down the crystal structure and going to smaller and smaller sizes. So, this is top-down. On the other hand, what we do with these nanocrystalline samples is an evaporation and condensation process and, in some ways, this is best described as a bottom-up approach. Even here, you are starting with a sample which is large and then, bringing it down to a sample that is smaller.

But the steps that you use are such that you first get it into a vapor phase and then so at that point you think of that as your starting material, those that vapor that you have formed is the starting material which then, condenses down to these smaller crystals. And therefore, from that context you can think of it as a bottom-up approach. So, you can think of this as a bottom-up approach to sample preparation. So, in that context as I said we discussed about different possibilities. So, whether it is top-down or bottom-up.

So, this will be this would constitute a bottom-up approach. Now, evaporation and condensation can be done in multiple ways and we will see a few of the most prominent ways in which this can be done. So, the first process is a thermal process basically, thermal evaporation. So, what you what we do is there is a boat this is called a boat.

So, this is a boat. Usually, it is made of some high melting point metal. It could be tungsten, it could be tantalum so, something like that. So, it could be tungsten or tantalum for example and it is kept in a chamber. So, this is a chamber; this is a vacuum chamber and then, you draw vacuum. So, you evacuate the chamber.

So, now before you do this let us see a few parts of this system here. So, you have a vacuum chamber. This is the boat that we spoke about. The sample is placed in the boat. So, sample is placed in the boat. So, sample is placed inside the boat; then, we have a substrate.

So, this is a substrate and the substrate could be a variety of different materials. I mean it really depends on what experiment you are doing, you could have I mean you could have a piece of glass there, you could have quads glass there, you could have some ceramic material there, you could have some other metal surface there. Basically, it is the surface on which this condensation is going to happen. So, that is what it is.

So, based on your study, maybe that substrate is also very important; maybe you are trying to understand the relationship between the deposit and the substrate, in which case you have to select that appropriate surface substrate. Sometimes maybe this substrate is not very important for us because we are maybe going to scrape the deposit away from that surface. So, in which case substrate may have still have some impact, but we are not very concerned about that substrate.

So, there is some decision to be made there on what that surface is. So, this is the general setup. So, what we do is we draw a vacuum here. So, the chamber gets evacuated and then, you can send current which goes through this boat. This boat is a high resistance boat; tungsten and tantalum have high resistance.

So, you have high heating. So, I^2R heating will happen. You would have considerable amount of heating. So, if you actually see these setups, you can actually have a fairly large chamber. It really depends on what your setup is. You can also have a small appropriate material glass chamber kind of the vacuum chamber.

And in that you draw a vacuum. So, you can actually see this boat, inside it or at least there will be a window through which you can see the boat and then when you heat it you will see it slowly go from being dull red to red to yellow, it starts getting close to white-hot. So, it becomes really hot like no different than a filament in your older style bulbs. So, it becomes that hot. So, whatever material you keep in it, usually note tungsten tantalum and all are a very high melting point.

So, there is a good chance that you are going well past the melting point and even the vaporization point of that material. So, you have to select those materials where this is possible and that those are the materials with which you can use this technique. Then, that material evaporates. So, now, this is material what you see here going upwards are evaporated sort of atoms and molecules of whatever was the sit as the sample. So, these evaporate and move up. So, they actually evaporate and sort of fill the chamber.

So, it is like you are creating an atmosphere in there where you fill the chamber. Of course, what you normally do is after you draw a vacuum, you close a valve here. So, you use a valve here and you close it. So, that otherwise if you keep drawing vacuum then whatever you evaporate will also go off into the vacuum chamber into the vacuum pump. So, you draw vacuum for some time and then, once you have reached a very low vacuum, there is a condition, you just close the valve there and then, now we evaporate this material. This evaporated material starts filling the chamber and it will also go towards this substrate.

So, it at that point there is no specific direction in which it is headed it is just headed up in different directions and it hits the substrate. If the substrate is at a distinctly lower temperature ok, so and we will talk about this lower temperature distinctly lower temperature. Then, this vapor that you have released will go and condense on it. When it condenses, it forms fine particles of whatever is evaporating and coming and convincing there and based on the nucleation and growth kinetics.

So, nucleation and growth kinetics of each of those systems that you are interested in, you will see multiple nuclei which will form and they will grow and you will form a wide range of a small number of means wide large number of small crystals is what you will form on this substrate.

And then, you can sometimes again depending on the substrate and how it adheres how this material adheres to the substrate, you may end up getting a thin film of the material that you have evaporated or you will get a powdery deposit on the substrate. In either case you will have some deposit there which later you can then, scrape remove and then use for any subsequent study that you want.

So, this is a system which you can use. Again, this can be made if you have the right kind of parts. You can make it in your lab, it is simply you have to get the right kind of materials though because I mean you cannot just take any glass and use it. There will be specific glass materials available, jars available which can handle high vacuum and that kind of a jar you have to have or you can even make a vacuum chamber; a proper vacuum chamber stainless steel based vacuum chamber with enough wall thickness that it does not get crushed under the one atmosphere pressure difference that is there.

And then, but it has to have some window through which you can see what is going on inside that window will again we know covered with an appropriate kind of glass. So, that you can end it sealed well. So, that you can still see without loss of vacuum and then, you can see this evaporation process you will see this is the condensation process. So, normally this is just as an experiment, the main duration that is involved here is the time taken for the vacuum to reach an acceptable level. If the vacuum does not reach an acceptable level, then as the vapor forms it will react with whatever is left in the atmosphere.

So, if you have oxygen there, you will form instead of sending the metal, you will form a metal oxide particle which is probably not what you want in your experiment. So, you have to get very good vacuum so that you have removed essentially scrubbed out all the oxygen that is present and then you do this evaporation so that you get a clean deposit and so, this jar can be small it can be large and so on.

And you have this simple circuit through which you can send this current and then, evaporation takes place and then, condensation occurs. And so, you can make a small tabletop setup, you can make a large setup, you just need a power supply not very different from what you would use for welding power supplies that is capable of reasonably high amounts of current, not a very significant amount of voltage. So, that is really what you have to get. You can also get commercial systems where you can do this.

So, this is an evaporation and condensation process. This is a thermal evaporation process and that is what we have described in this slide here and it helps you get samples with small crystal size. We will continue with the evaporation and condensation, but there are a couple of other variations on it which I will talk about, they all also get you these small crystal size samples.

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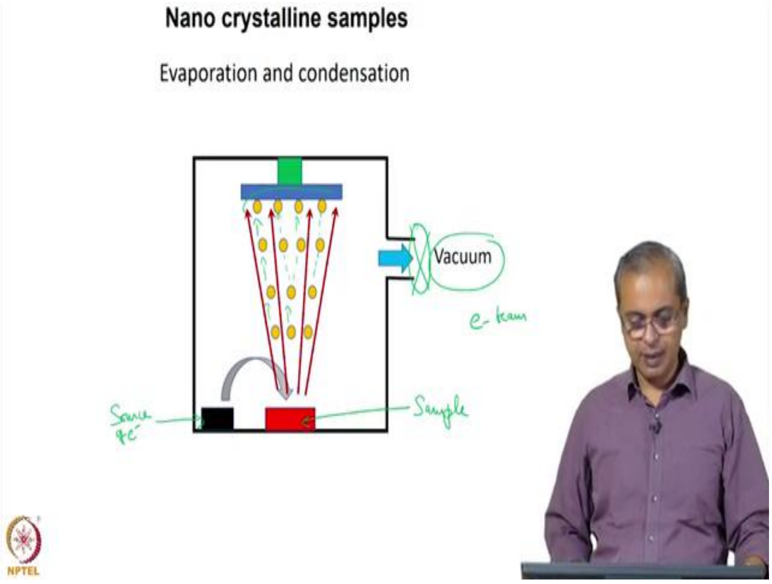

Nano crystalline samples

Evaporation and condensation

So, the next variation on this is one where you have an electron beam based evaporation process. So, you will see here. So, this is a sample now. So, this is the sample and this is a source of electrons. So, if you charge the sample versus the source of electrons appropriately, electrons will get off the source of electrons and get accelerated and will hit the sample which is a positive potential. You keep it will come and accelerate and it will hit that sample. So, when you do that forcefully enough you will find that it will eject material from that sample and again you will have to do this in a vacuum.

So, you have a vacuum system here. So, that process is still the same. Put a vacuum here; you put a valve here and once the vacuum has been drawn, you close that valve and then you start sending current to your filament. So, there will be some filament which is the source of electrons. From that filament, you will have electrons ejected. They will raise towards the sample and they will strike the sample and when they strike the sample, they will eject some atoms from that sample and that sample ejected atoms then begin to move up which is what you see here and once again you get this conversation.

So, this is an evaporation and condensation process which uses an electron beam. So, this is called an e-beam evaporation; an e-beam based evaporation process. So, that is how we get this evaporation condensation process.

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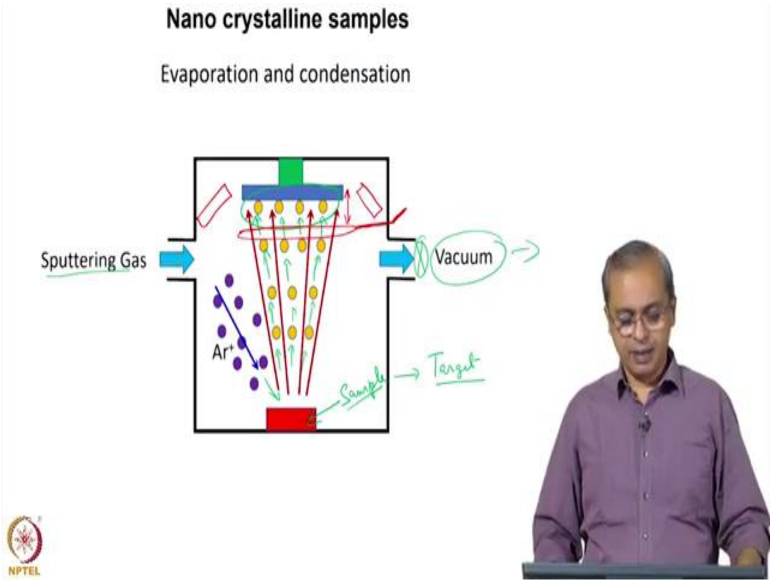

Nano crystalline samples

Evaporation and condensation

One more variation we can use which is called a sputtering. So, in sputtering what we do is there is a very similar process again, you have a valve here and then you have a vacuum. So, you draw vacuum and then once the vacuum system has developed low enough vacuum conditions, you send in a sputtering gas. So, argon comes in and then you set the right kind of conditions, it gets ionized.

It comes and strikes the sample and then when it strikes again atoms are ejected and they go and condense on the sample. So, this is the general process. So, you can see in all these three techniques that I showed you, at some fundamental level you have the similarity is that you have a vacuum chamber, you generate enough vacuum and you create a situation where the material of interest evaporates.

So, you either use thermal energy or you use electron beam or you use argon ions and you evaporate this material, then it floats into the chamber and then it goes and settles on the substrate and so, this is how you and so, you collect enough on the substrate and then, you stop your experiment. And at that point, you can scrape off whatever you want. In these three, if you see relatively speaking the thermal evaporation process is very fast.

So, normally your vacuum chamber may take half an hour or 45 minutes or even an hour to reach the low vacuum that you are interested in. Once you reach that low vacuum if you just in the thermal evaporation process if you start raising the current. Almost immediately within the matter of if you want to do it kind of gradually.

So, you do not have non-uniform heating that is going on. If you do it gradually in about 30 seconds to a minute, you will have that boat getting close to white-hot past the red-hot condition. It will start looking close to white-hot and the material will evaporate. Evaporation will happen almost immediately if you have the again material of the right kind of melting point in it and I mean boiling point in it, it will evaporate out and it will condense.

So, that process of that experiment is a very short duration at that point. Once you have reached the vacuum you just raise the current in about 2 to 3 minutes, your experiment is over. On the other hand, if you use a sputtering process or you use the e-beam evaporation, even after you reach the low vacuum, you have to continue this it is a very slow process. It is gradually removing material and that material eventually ends up in the substrate that you are condensing.

So, it takes some time based on how many samples you want to evaporate. In the thermal evaporation process, all of the sample the boat remains intact whatever sample you put inside almost instantaneously evaporates. So, if you put a few grams all of that is in vapor form immediately, it condenses. In the electron beam process as far as the sputtering process, the sample is used as a target. It is also called a target because it is hit by the argon-ion or the electron beam.

And so, usually if you go for these kinds of sputtering units, commercially available sputtering units will also sell targets for you. They will sell separately, they will sell high quality maybe platinum target, palladium target, copper target etcetera. They will sell that to you and that is the kind of target that you will use it will be. So, you actually do not evaporate the entire target; you are basically removing only a few microns of the target and then, you make it settle on your sample.

And in fact, you will take care because these targets are going to be sitting outside and therefore, they may have an oxide layer or something on it which you do not want. So, usually in fact, in all these cases what you will do is there will be a mask of some sort which I am which are just shown here, some kind of a mask which will sit in front. It will sit in front blocking the which will block the substrate from your target. So, it is between the substrate on the target and it blocks whatever is coming from the target from reaching the substrate.

So, usually what will happen is in these experiments, we start the process we allow some amount of sputtering to go on with this mask blocking the path and so, at that point any impurity that is getting released from that sample will not reach the substrate. It will get blocked by this mask. It will maybe form a layer on the mask and that is useless for us. So, it just continues to form for some point in time. So, based on how fast this process is occurring and how deep we want to go into the sample before we start the sputtering process, we let it runs with this mask in place and then, you should have this attached in some form to a lever outside; some lever here which you can then rotate.

So, that when you rotate this mask will now go away from that position and then, it will expose the substrate and at that point whatever you are evaporating starts condensing on the substrate. So, this kind of small details are there in this process which you have to look into plus there is a fair amount of variation on what is the temperature at various locations and what is the angle at which the material is arriving etcetera.

So, having a substrate at that location may be may give you different results than having a substrate in this location or in this location and so on and so, some level of experimental work, you have to do to satisfy yourself that the kind of sample you are getting is the kind of sample you are interested and that is what you would do with this system. And in fact, with these units because the process is a bit slow.

In fact, its good idea for you to think ahead of time what kind of samples you want and put multiple substrates in position so that you can get all of the samples together in one sputtering process. And so, that is why so, those kinds of logistical details you have to pay attention to for you to fully maximize the utility of a sputtering process and get yourself these samples. So, we have seen some ways of getting us as microcrystalline samples, we have seen some ways of getting us as nanocrystalline samples.

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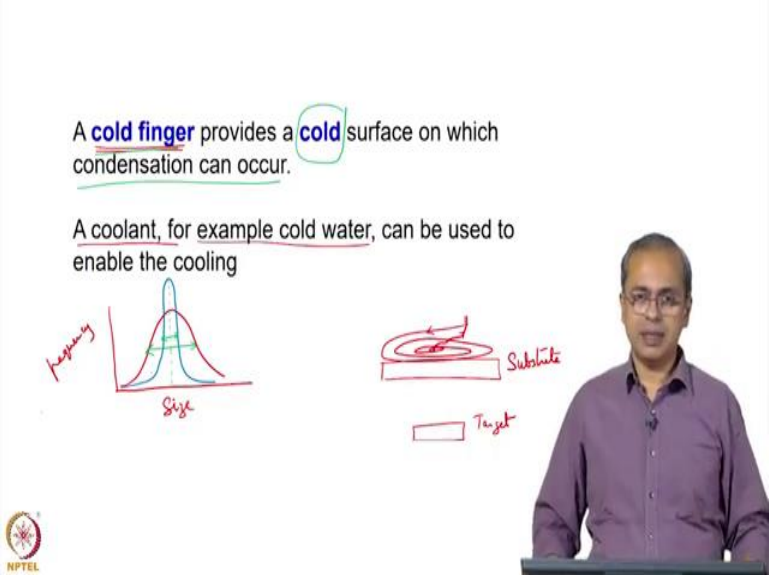

A **cold finger** provides a **cold** surface on which condensation can occur.

A coolant, for example cold water, can be used to enable the cooling

For the condensation process, there is something called a cold finger. So, in many of these condensation process, they will say that they have used a cold finger. So, the idea is simply this that you can actually have a substrate which is just sitting there which when you loaded was sitting at room temperature and then, as the evaporation process happens and condensation process happens based on how the chamber is operating based on any other heat that is getting generated in the process, the substrate may stage around room temperature that is basically what you are likely to see.

But you can assist the condensation process by deliberately keeping that surface at a colder temperature and to that extent, you typically will have coolant and for example, cold water would suffice which can go into a small loop, which goes just behind that substrate. So, you have a substrate and this is where your target is; below is the target and this is your substrate. So, in the chamber, you will have some pipe which does this loop and goes out and you send some cold water in this and it just comes out this way and then finds its way out.

So, this way you can cool the substrate and keep it at a much colder temperature and you can maintain the temperature. More specifically, you can maintain the temperature; you can confidently say that all of the condensations happened at 5ºC something like that you can say that and you can maintain that. It is important because that impacts the extent of these nucleation and growth kinetics processes.

And therefore, it impacts the crystal size that you will get. It also impact the distribution of crystal sizes that you get; like I always said if you have the size distribution and this is frequency. For us for the nanoscale work, this kind of distribution is not particularly useful for us rather we need a distribution that looks like this something like that.

So, much narrower distribution, you will notice that both of these have the same average size. So, the mean is the same. In both these cases, the mean is essentially the same it is here. But the distribution in one case is this wide distribution, in the other case is this wide and we want a narrower distribution.

So, if you want such things to be taken care of, then it is very important that you keep good control on the temperature of the substrate. So, that that it did not change temperature and because of that the kinetics did not change and you had good control on the kinetics and therefore, by maintaining the temperature, you got good control on your kinetics. And therefore, greater with greater confidence you can expect that the crystal sizes will be uniform and they will be very close to the value that you are interested in. So, that is why we would do this.

And so often we in these kinds of activities, they use something called a cold finger which basically provides a cold surface on which the condensation can occur. So, this is the scheme of things how we make macroscopic scale samples; how you make nanoscale samples and then how you control some aspects associated with it.

So, this deals with the nanoscale sample synthesis and in this context where you are trying to look at the Hall - Petch effect you want a range of sizes. So, in both of these cases, you can get a range of sizes based on the conditions under which you are preparing the samples. You may need to do some heat treatment after you have done the sample synthesis to more accurately get the kind of sample sizes that you are interested in, but basically this is what you would have to do.

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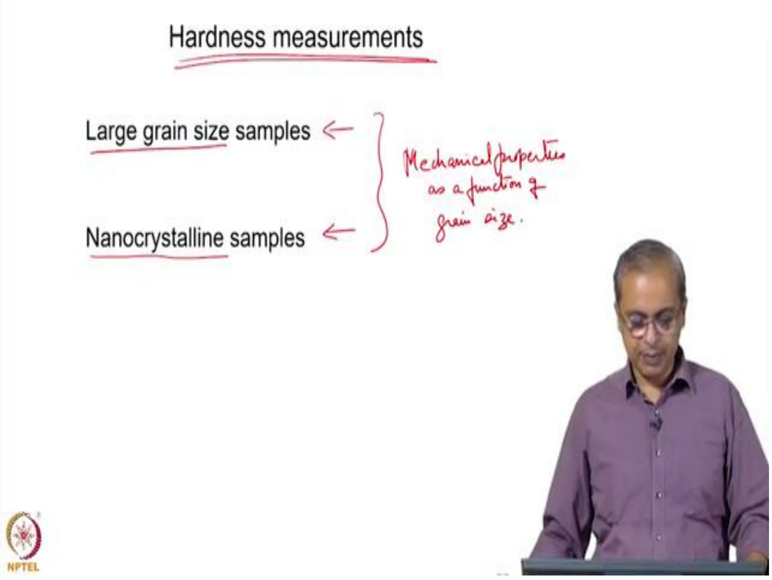

Hardness measurements

Large grain size samples ←

Nanocrystalline samples ←

Mechanical properties
as a function of
grain size.

So, now having gotten the samples, we want to make some estimate or actually some measurement which tells us something about the mechanical property of these samples and that is the idea here. So, both for large grain samples as well as small grain samples, we want mechanical properties as a function of size.

So, this is what is desired. So, the way in which this is done is to make hardness measurements. So, when we actually look at the original work, where they did this you will see what they have done, but in principle, this is the idea that they have used that they use this hardness measurement process, where they are able to get using the hardness. The mechanical properties of a large grain size sample as a function of grain size and also the mechanical properties of nanocrystalline samples as a function of grain size.

So, this is what there was done and therefore, we will take a few minutes to look at what is this hardness measurement and how do we make it how do we make the measurement.

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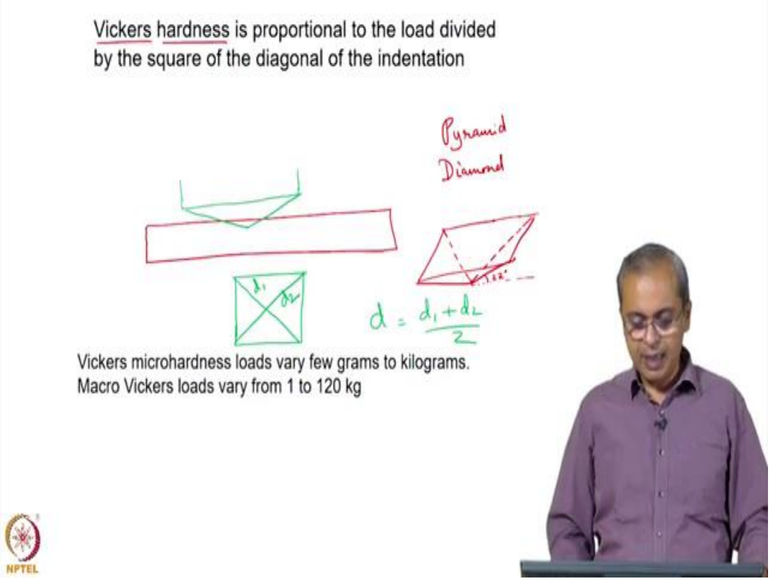

Vickers hardness is proportional to the load divided by the square of the diagonal of the indentation

Vickers microhardness loads vary few grams to kilograms.
Macro Vickers loads vary from 1 to 120 kg

So, to do that there are actually different types of hardness that can be measured in a sample and one of the common ways in which hardness is measured is called Vickers Hardness and to do this, all you have to do is your samples here and on this sample, we are trying to make an indentation. We are trying to force a sharp object into the sample.

So, and we try to see that if you put a certain amount of force, how deep into the sample you are able to go? The softer the sample is with the same amount of force; this sharp object will go at greater depth. If it is a harder object, it will go only a shallow depth. So, that is the basic idea and that is the Vickers Hardness measurement to standardize it the shape of the sharp object is fixed and the material it is made off is also fixed.

So, typically it is a pyramid made of diamond and its angle will be something like this. So, that is a pyramid and so, this pyramidal angle is set such that this is about 22°. So, that is the angle that we have 22° and that is the pyramid that you use. So, if you see it sideways this pyramid will come here and it will form an indentation on the sample and then, that indentation is seen made on the sample.

So, once you get done making the indentation, if you remove this indenter you can look down on that indentation and you will see an indentation that looks like this. Square shape roughly square shape and it will have two diagonals. So, now, usually given that it is a pyramid, you should get an exact square. But you may or may not get an exact square based on some maybe some lateral variation in properties of that sample and so on.

So, what is typically done is to approximate it as a square and so, what we do is look at the two diagonals and with the dimensions d1 and d2 and you take an average you take. So, d averaged is (d1+d2)/2 is just the average of the two diagonals and that is used to help us do some calculations and essentially, the hardness value Vickers Hardness is given as force over area.

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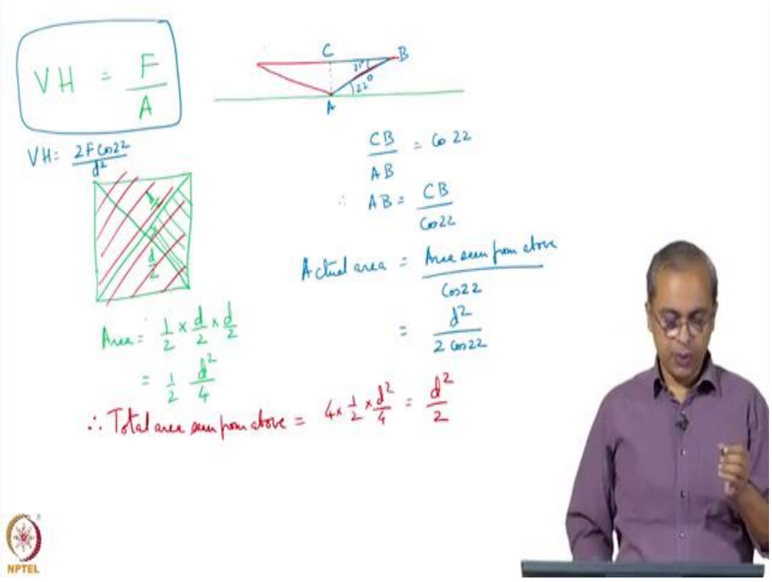

$$\boxed{VH = \frac{F}{A}}$$

$$VH = \frac{2F\cos 22}{d^2}$$

$$\frac{CB}{AB} = \cos 22$$

$$\therefore AB = \frac{CB}{\cos 22}$$

$$\text{Actual area} = \frac{\text{Area seen from above}}{\cos 22} = \frac{d^2}{2\cos 22}$$

$$\text{Area} = \frac{1}{2} \times \frac{d}{2} \times \frac{d}{2} = \frac{1}{2}\,\frac{d^2}{4}$$

$$\therefore \text{Total area seen from above} = 4 \times \frac{1}{2} \times \frac{d^2}{4} = \frac{d^2}{2}$$

So, that is all that we are trying to do and the force is that weight that you put on the indenter and so, that we can figure out. So, we are interested in knowing what is that area that surface area that was created by that indentation. So, now, if you look at it top-down you have the square and you have these two diagonals. To the extent that this is a square this is going to be 90º and if that is 90º and this whole diagonal is the diagonal is dimension is d.

So, then, therefore, this half the diagonal is d/2; half the diagonal here is also d/2. So, if you look at the area of this triangle; this triangle here, the area is simply half into base into height. So,

$$area = d^2/8$$

That is the area of one that one triangle that I have shaded there and therefore if you look at the area of the entire indentation, this is just the plan area of it without looking at the shape of the indentation, simply looking it look at looking at it vertically downwards.

If you take the area of this entire thing, clearly that is simply 4 times the area of the small triangle that we have there. So, therefore,

$$total\ area = \frac{d^2 \times 4}{2 \times 4} = \frac{d^2}{2}$$

So, that is the total area of this indentation as seen from above. Now, we also know that the indentation has happened at an angle and that angle is 22°.

So, if you see the indenter and this is the surface of your sample that is the indenter and this is the surface of a sample then, this angle is 22°. Therefore, if you see what you are getting here, you are actually seeing the indentation from the top. And therefore, you are missing the fact that you are actually having and having the surface at an angle and therefore, that has more area.

So, you have to actually account for the fact that you are only seeing the projection of that area on the flat surface. So, if you look at this line here and this line here, this angle is also 22°. So, let me say this is A, this is B and this is C. So, CB by AB is equal to Cos20 Cos22 right there for AB=CB/Cos22.

So, that is the linear dimension. The same thing will happen for the area. So, for the area if you take the area flat and you want to see its projection at a 22° angle that area. So, actual area equals area seen from above divided by cost 22. So, therefore,

$$Actuval\ area = \frac{t^2}{2 \times \cos 22}$$

So, this is the actual area that you get and we see here Vickers Hardness is forced by area. So, therefore,

$$Vickers\ Hardness = \frac{2 \times F \times \cos 22}{d^2}$$

So, it is simply this actual area, I have simply done forced by the actual area. So, force is F/d^2/2Cos22. So, if you rearrange it you have to F×Cos22/d^2. So, this is Vickers Hardness and based on the units, you use for force and the units that you use for d, you can get Vickers Hardness in different units. Typically, they are using the force in kilogram-force and the d in millimeter.

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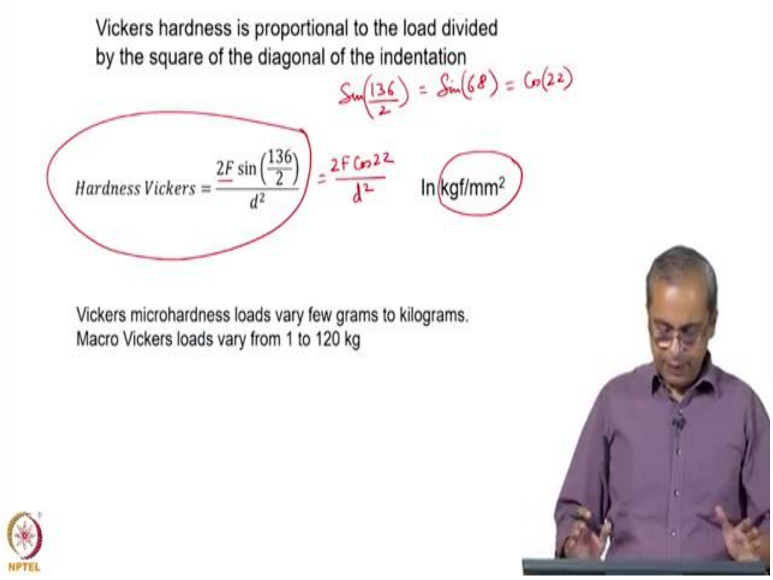

Vickers hardness is proportional to the load divided by the square of the diagonal of the indentation

$\sin\left(\frac{136}{2}\right) = \sin(68) = \cos(22)$

$$Hardness\ Vickers = \frac{2F\sin\left(\frac{136}{2}\right)}{d^2} = \frac{2F\cos 22}{d^2}$$

In kgf/mm^2

Vickers microhardness loads vary few grams to kilograms.
Macro Vickers loads vary from 1 to 120 kg

So, you will have units which look I mean not so very standard. So, you get units in this. The unit you end up getting is kilogram-force per millimeter square. So, this is the formula here? It suddenly shows up here as Sin (136/2). I had it as Cos22 there, but if you see here Sin136 divided by 2 is the same as Sin68 which is 136/2 and that is the same as Cos22 because Sin is Cos (90-θ). So, that is basically all we have here. Otherwise you have the same thing here 23F I had 2F. So, this is the same as 2F×Cos22 by d square which is what we derived in our previous slide. So, this is the hardness and this is how we get Vickers Hardness measurement done.

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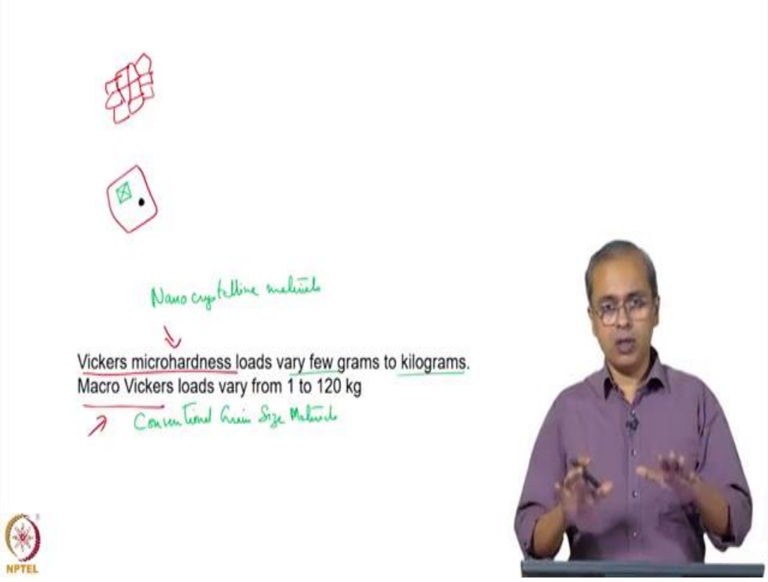

Vickers microhardness loads vary few grams to kilograms.
Macro Vickers loads vary from 1 to 120 kg

And the other point that we need to just keep in mind here as we close this discussion is that there is something called Vickers microhardness and there is something called macro hardness. Macro Vickers Hardness and typically in this kind of a situation for the nanoscale materials, we would have to use this microhardness technique.

And for the conventional materials, we would use to have to use the macro Vickers Hardness technique. Primarily, the idea is to get an indentation that is significant enough that you can make a good measurement and it also conveys to us the property of that material that is basically the idea that we are looking at. Now, when you go to nanocrystalline materials when the crystal size is very small and you have very small crystals there.

It sometimes helps to have small indentation. So, I mean you may not be able to do this, but normally what people try to do is with micro Vickers Hardness, they put much smaller loads. So, like few grams to kilograms, you can say and typically it will be in the gram range if you are looking for a small grain size sample. The reason is then you want that indentation to stay limited to a particular region.

This is particularly useful if you have like a 2-phase microstructure, you have a matrix and you have some precipitate. If you want the hardness of only the matrix and you do not want the hardness of the precipitate clouding the data, then you want the indentation to be small enough that it stays limited to the matrix.

So, supposing you have a matrix that is that big and then, in that you have a precipitate sitting here. So, now, you want an indent indentation that is only this size so that you can make this measurement and then get your hardness value out of it. So, that is the reason why there is something called a Micro Vickers Hardness and there is a Macro Vickers Hardness. So, this is used for nanocrystalline materials, in this case, is better suited for it and this is used for conventional materials.

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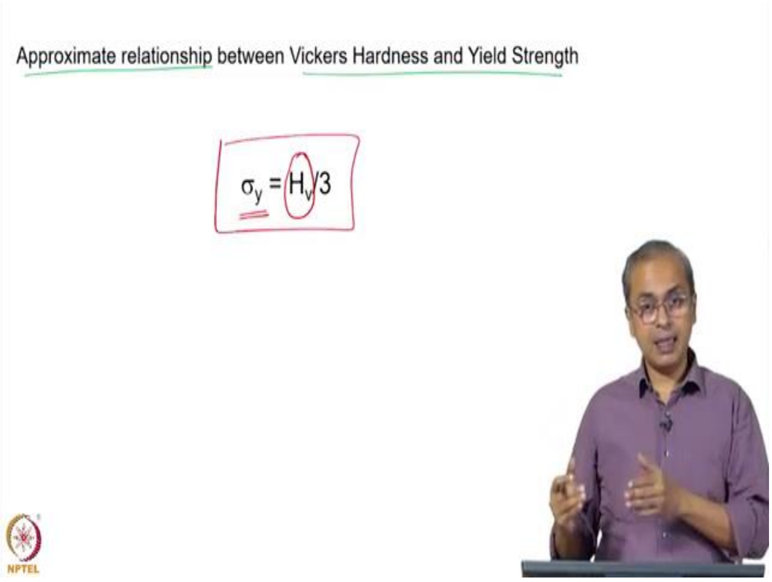
Approximate relationship between Vickers Hardness and Yield Strength

$$\boxed{\sigma_y = H_v/3}$$

Now, having gotten the hardness value, the one other detail that I would like to add before we close is the fact that from the hardness value, there is because of an approximate relationship between Vickers Hardness and Yield Strength, the yield strength can be approximated as showed here. So, using this approximation, we can get the yield strength of that material based on the hardness value we have obtained for the material.

So, therefore, the yield strength is as we discussed earlier, yield strength is typically a parameter that we associate with mechanical properties and it helps us compare materials with different yield strength etcetera. So, that is the kind of information that we are more interested in arriving at and in this case, we arrive at it indirectly by first getting the hardness and for an indentation to occur also the material has to yield.

So, therefore, the hardness is related to the yield strength and then, we use this formula of approximately the yield strength is one-third the hardness value and in this process, you can get the yield strength. So, we have seen in this class how to make microcrystalline materials; how to make nanocrystalline materials and how to make get the mechanical property of it using hardness.

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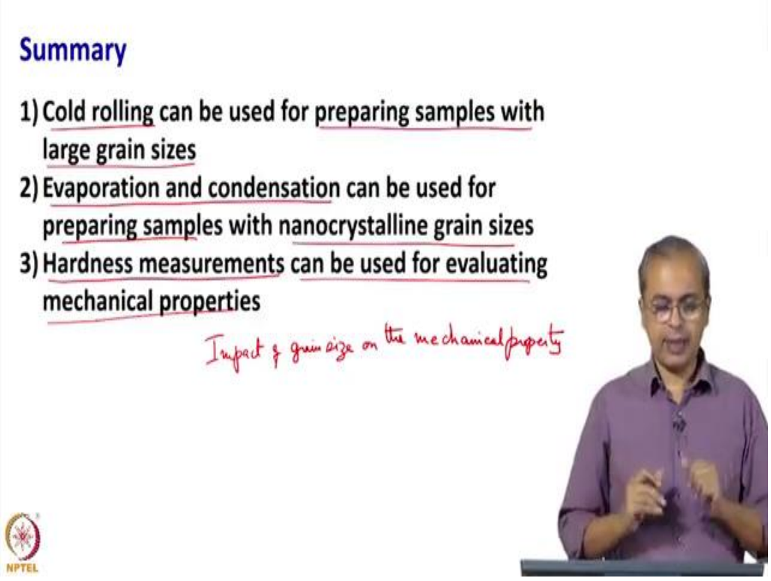

Summary

1) Cold rolling can be used for preparing samples with large grain sizes
2) Evaporation and condensation can be used for preparing samples with nanocrystalline grain sizes
3) Hardness measurements can be used for evaluating mechanical properties

Impact of grain size on the mechanical property

So, in summary, cold rolling can be used for preparing samples with large grain size, microcrystalline kind of grain size. Wide types of different types of evaporation and condensation processes can be used and based on your sample the system that you are working with different types of evaporation and condensation processes can be used. For preparing samples with the nanocrystalline grain size and between the first step and the second step, you will get a range of samples of narrow grain size distribution, but of different values of main grain size in the macro scale as well as the nanoscale.

Having got that having obtained those kinds of samples, we can take hardness measurements using Vickers Hardness testing process for which we just saw what is the process; how the indentation is formed; what kind of calculations are done with it and we also saw that it is approximately related to the yield strength and therefore, we can use that for evaluating properties. So, between these set of steps experimentally, we can evaluate or we can get the data together on the impact of grain size on the mechanical property of the material. So, this was our particular aim. So, we have seen the whole process of how this can be accomplished experimentally.

In our next class, we will actually look at what is seen as results in this kind of material systems with these kinds of crystals structure sorry crystal size, grain size variations. And we will see what happens to trends in those mechanical properties in the large crystal size or grain size range and the small grain size range and we will try to understand the theory that explains what you see in that data. So, with that, we will conclude this class.

Thank you.