Nanotechnology Science and Applications
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Lecture - 13
Superplasticity and the Nanoscale: Experimental aspects

Hello. In this class, we are continuing our discussion on Superplasticity and particularly we are going to look at Experimental aspects. This is always interesting because in any of these studies in material science and for that matter any other arena of science experiments are very critical and it does not matter what theory you come up with.

So, for example, we spoke about this grain boundary sliding and there are some variations on that theory. So, theoretically you can now explain it and then maybe draw some diagrams trying to explain what might be happening, but in reality, at some fundamental level, you have to design an experiment where you can actually see that phenomenon. You can see that phenomenon; you can record that phenomenon and share that recording with a lot of other researchers.

So, that is a very important and fundamental aspect of any experimental any activity in material science and I mean work and superplasticity is no different. It is the same you have to have these really nice experiments. In fact, as part of PhD work when you work on a different area designing interesting experiments is a very important aspect. Many times, the core of your PhD is that interesting experiment that you have managed to design because you would be able to get some unique set of data which some standard experimental setups may not be able to obtain. And, that is the where you are showing your creativity as a researcher to probe some particular phenomenon.

So, we look at experimental aspects here associated with superplasticity and try to see what we can learn from this process. The experimental aspects are of course, also general in that sense. We are using it in the context of superplasticity we are also I know pointing to specific experimental techniques in the context of superplasticity, but they are not restricted to superplasticity you could potentially use it for some other application where you see a similar need to probe some phenomenon which is maybe not that easy to probe otherwise.

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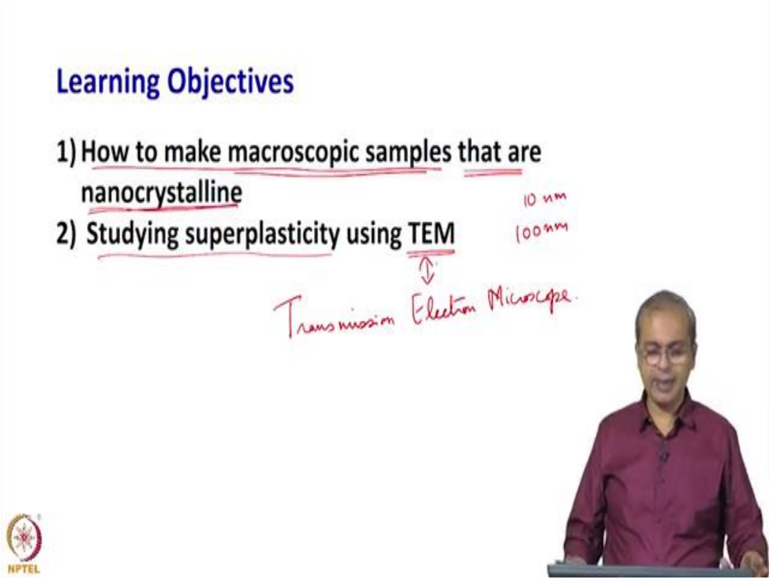

So, we will have some learning objectives here. Our first learning objective is how to make macroscopic samples that are nanocrystalline. So, we keep talking of nanotechnology, nanoscience and that means, particles which are in the several tens of nanometers, maybe 100 nanometers. So, we always think of these as individual particles small individual particles.

But real technology that we have which we use whether it is the body of a car or maybe even the body of a pen or some bio application that you are using say some artificial bone that you are using, creating and using all of these are macroscopic objects. Not I mean you may have some electronic activity going on which is in a part of a circuit which is very extremely tiny, but many of the things that we use technologically are macroscopic objects.

And, therefore, even if you say that you have found some interesting property in the nanoscale you need to make a macroscopic sample which has that nanoscale phenomenon occurring through its entirety or at least through some specific locations in that sample so that you can use it appropriately.

So, in any nanomaterial related work, this ability to make macroscopic samples which you can handle and do something with that are in the microscope showing themselves out to be nanocrystalline is very important and so, we need to think about that we need to look at some experimental techniques that help us do that. And, we will also look at what can be done with respect to studying superplasticity using the TEM which is the transmission electron microscope. So, that is your TEM.

So, these are two aspects we will look at we will also look at some related techniques and some interesting gadgets that we can work with.

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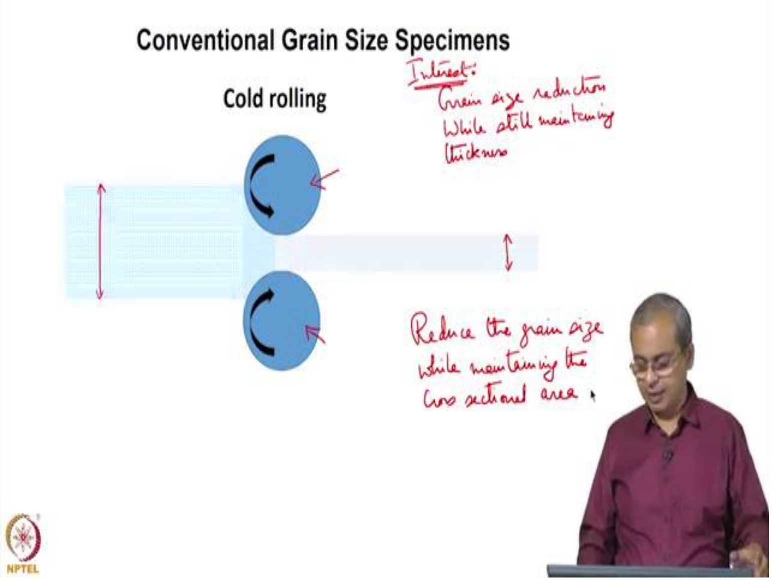

So, we already saw that you can actually do a rolling process and, in that process, you can reduce the grain size of some sample. So, when you do that because you are doing some plastic deformation; so, you are starting with a sample that has a thickness that is of that value and then as the rolling proceeds you have a sample which has a thickness that is like this, significantly thinner and of course, you may do it in stages and so on.

So, this is accomplished by using two rollers which do this rolling process and then reduce this thickness and so, this is a standard metal forming process used extensively in metallurgical industries. And, what has always been seen is that you can in this process typically the grain size becomes smaller. So, potentially you can think of this as a process which you can do in stages repeated repeat this process again and again and again, and you will keep getting thinner and thinner and thinner samples which will have smaller and smaller grain size.

It is also as I mentioned it is also going to be difficult as you progress because the material is going to get stronger and more difficult to work with as you get to smaller and smaller grain sizes. So, but this is the general process by which you can do cold working as it is called and you can consider this as a process by which you can reduce the grain size. And, if depending on your starting grain size and how much rolling you do it is quite possible that you could end up with something that has nanocrystalline grain sizes in the sample. So, that is quite a possibility.

Now, generally speaking, we say especially with respect to technological applications which are little large scale and so on, not every application is going to find a thin sheet as acceptable material. So, you may actually want something solid which has 3-dimensions reasonably thick sample with which you want to do something. So, this is still a macroscopic sample you are not gone to nanometer meter dimensions, but it is it may or may not be exactly what you are interested in dimensionally.

So, there is always interest to do some grain size reduction. So, there is always interest in this to do grain size reduction while still maintaining thickness. So, or more specifically we would like to reduce the grain size while maintaining the cross-sectional area. So, we would like to reduce the grain size while maintaining the cross-sectional area and that is a very useful thing for us to have if you are looking at applications where you want refined grain size, but still some particular shape.

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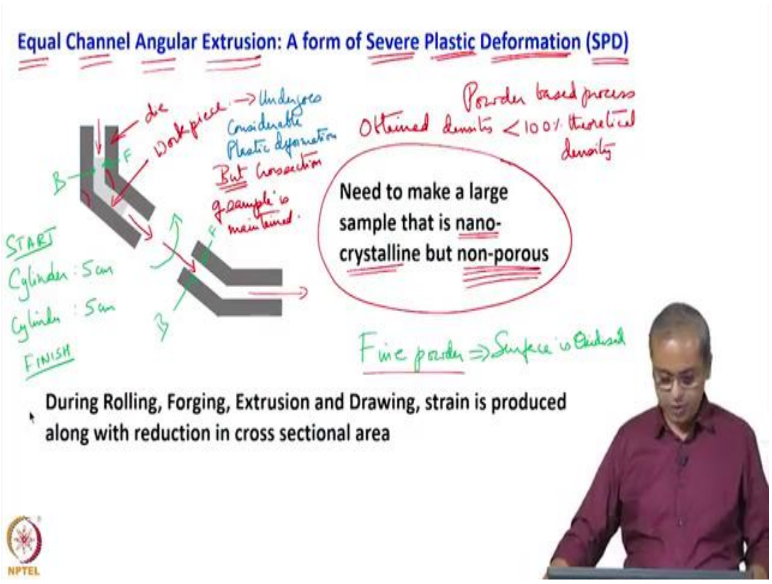

So, to do that there set of techniques that are referred to as Severe Plastic Deformation. So, Severe Plastic Deformation or in short it is referred to as SPD. So, severe plastic deformation; one version of it is this a process called the equal channel angular extrusion. So, equal channel angular extrusion is one process by which this is done.

So, again the idea is this, the purpose is this we would like to make large sample that is nanocrystalline, but at the same time it is nano porous it is sorry non-porous it is nanocrystal, but non-porous. So, you can imagine a possibility, we have also discussed this possibility in another when we are looking at say a whole patch relationship study that you can make a very fine powder, you can make extremely fine powder which is nanocrystalline and then you can compact it. You can compact it, you can do some sintering and so on, and then get a sample that is nanocrystalline and at the same time you have some sample that you can work with.

The issue is anytime you do sintering two things happen. First of all, there is a chance there is a very high chance and in fact, it does happen that the crystals grow in size. So, even if you were targeting 10-nanometer size to finish with, you will actually have to first create powders which are smaller than 10 nanometers and then when you do the sintering you will get end up getting 10 nanometers. So, this is number 1.

Number 2 generally whenever you do this powder and then consolidation of the powder and then sintering process you almost always never get 100% theoretical density. So, any powder-based process will have less than 100% theoretical density. So, the density obtained will be less than 100% theoretical. So, this is the density obtained density I will put it that way obtained density will be less than 100% of the theoretical density.

So, this is maybe in some applications it is not so critical, but in many other applications especially if you are looking at mechanical behavior having porosity in the sample completely changes the behavior of your sample. The whole behavior of the sample and you know, it acts as a location where you can have a stress concentration where you may have fracture happening preferentially and a lot of things change in your sample the minute you have porosity. You can no longer treat it as a uniform sample; you are having significant variation in property through the cross-section of the sample.

And, so, what you get out of it as the property may not represent the inherent property of the material, it may rather represent the impact of those defects those that porosity on the property of that material. Therefore, while you can create nano-sized powders and consolidate them to get yourself a larger sample that does not necessarily give you the kind of sample that you will feel comfortable utilizing where you can actually get data from it that is reliable.

The other problem with a powder-based approach if you take create powders because and the reason we look at it is because you can often get fine powder and from that you can start consolidating the samples is that when you take the fine powder typically the surface of that fine powder is now exposed. So, in fact, invariably when you have a fine powder it usually means surface is oxidized.

So, the surface is oxidized. So, you were you are not going to have clean sample. So, you are typically going to have oxidized particles and those particles are going to sinter. So, first of all the sintering process is not going to be complete and on top of it you have this oxide layer which is there in significant quantity overall because you have so many fine particles. So, multiple issues with trying to do this using fine powder particles, although it appears that that is a way in which you could create a larger sample which is nanocrystalline.

So, there is interest to do to create a nanocrystalline sample that is non-porous where you do not have this issue of porosity being present and which essentially does not have oxide because you have not allowed the interior of the sample to be exposed to the atmosphere. So, you may still have a surface oxide which will form, but that is much easier to handle because you finish all the processing and you end up with the sample that has the correct crystal size or the grain size.

You can always clean this top surface; you can clean the top surface it is much easier for you to clean the top surface and then you put it to some application. It is much difficult in fact, essentially impossible for you to clean the interior of that sample and therefore, that is not particularly useful. So, the process that we use is something called equal channel angular extrusion. So, what you what we do is there is a die.

So, typically if this is just an example, I am just showing you a schematic here where there are two dies and then there is some bend in the die. So, there is some angle here. So, there is an angle here and this is your workpiece which you push through and it comes out this way. So, that is your workpiece. So, this is the die and that is your workpiece. So, you push the workpiece through and so, it changes its orientation. And so, this sample is undergoing considerable plastic deformation.

So, let us see what we have done here we have pushed this sample through it comes out and, in this process, the sample has undergone considerable plastic deformation, but the cross-section of the sample is maintained. So, the cross-section of the sample is maintained. So, you pushed something extrude something through this die and once it comes out overall after when you look at it externally it looks roughly similar to what you pushed it. It does not look like unlike a rolling process where dramatically the product is different from the material that you sent into the rolling process; it is very visibly different.

Here it is not going to be the case you extrude something out what comes out will have roughly the same dimensions as what went in. I say roughly because you are going to have some variation in the end right at the very beginning of the sample and the end of the sample because it goes through a turn. Even if you send a perfect cylinder in you are unlikely to get a perfect cylinder out, but the general cross-sectional area will be circular and it will be maintained more or less from end to end.

So, you get this in and then in principle you can say take that what comes out of here and send it into another die and then bring it out here. So, you can do this process and so, each time each pass that it goes through this one die and then goes through the second die through each pass you are having significant plastic deformation and you can do variations on this.

So, once it comes out of one die you can actually take it directly and with the same orientation so, you have that I am calling this the front side and I am calling this the backside. So, when it comes here, I can still maintain F here and B here as it goes in or I can make some rotation. So, it comes out and instead of the front going in as the front on the into the second die and back going in a back in the second die, I can make a rotation about the axis of that cylinder.

So, about the axis of the cylinder, I do a 90º rotation. So, it so it went through the first die in some orientation, once it comes out, I rotate it 90º and then I send it in to the second time and. So, this process you can do. So, people who work in this have come up with a series of such processes where they define what is a pass.

They define that in a single pass the material goes through the first die in a certain orientation and then goes through the second die in a certain other orientation and in the middle, you may have a rotation of say 90º or 180º or even 360º rotation. I mean 180 would be the thing 360 would bring it back. So, 90º or 180º and then in the second pass you again do a 180º rotation and you continue this.

So, you can define certain processes and a combination of steps and then you keep doing this again and again and again. And, you based on your experimental results and based on what you desire as the final crystal size you can say that if the sample does 10 passes through this, I will reach that crystal size. The nice thing is at the end of 10 passes the sample is still a cylinder. You started with a cylinder of radius I just to give you an example we start to the cylinder and it is 5 centimeters in the radius you finish with a cylinder also with 5-centimeter radius.

So, you started with 5-centimeter radius, you finished with 5-centimeter radius, but in the process, you have done a lot of plastic deformation and created a lot of small crystals and then in the process you have a very fine grain structure. So, this is called equal channel angular extrusion and it is a very nice way of a creating a sample of a very fine crystal size while still maintaining the external dimensions to be similar. Now, as I mentioned I know this is nice because it maintains the same cross-section.

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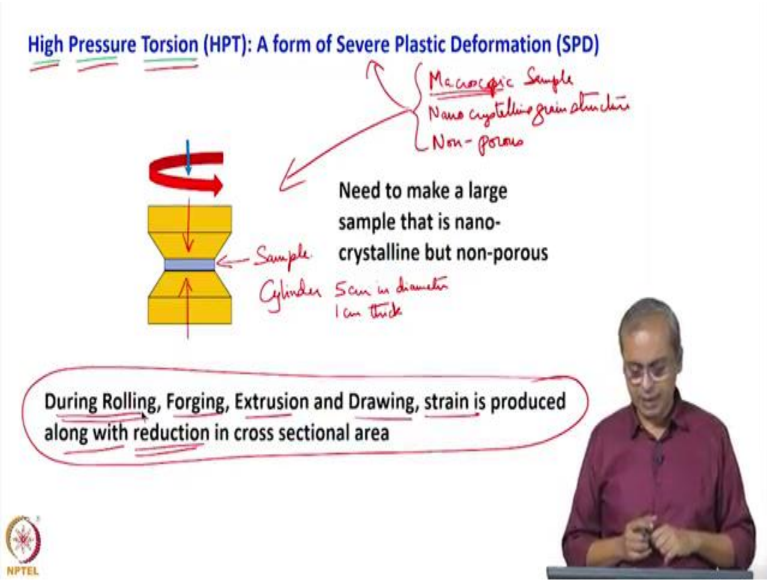

We will also see another version of severe plastic deformation and that is called high-pressure torsion. So, this is high-pressure torsion. And, so, as the name suggests you can see here this is your sample what is sitting here is your sample and what is done is it is pressed between two pieces here. So, it is pressed there and those two pieces are much stronger than this sample.

And, then under pressure, you create enough friction there because of the high pressure that you are of applying from the top and the bottom and then you start twisting the top and the bottom. By twisting it you are doing torsion you are doing a lot of torsion on the sample. So, continuously you are changing you are moving material in the sample. So, you are doing plastic deformation on the sample except that that sample is now constrained to one location.

So, again if you start with a cylinder and this is a some I mean say cylinder, I do not know the same thing we will say 5 centimeters in diameter and or even 1 centimeter in diameter it depends on what you are trying to do and say 1 centimeter thick. This is just an example. you are dealing with something like this then from the beginning of your experiment till the end of your experiment it will continue to maintain that dimension you are not really changing the dimension any way you are continuously doing torsion.

Please remember that anytime you do mechanical deformation of this nature you will generate a lot of heat. So, you always even rolling process etcetera at all those cases you will find because considerable amount of material is moving, solid material is moving significant amount of heat is actually being generated. So, you have to be conscious of it. You have to know what you are doing if you are if it is very important for you to maintain some temperature and not exceed some boundary, then you have to keep that in mind when you do this kind of activity. If necessary, you may have to do some passes, then wait till the temperature of the sample cools down and then continue this process.

So, both these high-pressure torsions, as well as the equal channel angular extrusion both of them are helping us accomplish this particular aspect which is that you are able to do this significant plastic deformation without a change or without a reduction in a cross-sectional area without any reduction in cross-sectional area. Whereas, typical conventional processes like rolling, forging, extrusion and drawing will produce you will produce strain in the sample, but that will be along with reduction and cross-sectional area.

Now, having said this so, this is a nice way of creating samples as I said we want to create a macroscopic sample which has nanocrystalline grain structure at the same time it is non-porous. So, this is the combination you want to do you want to create macroscopic sample with the nanocrystalline grain structure that is non-porous this is what we want to create. So, this is being accomplished herein and in any form of severe plastic deformation. So, this is good and we can utilize it.

Having come upon this particular technique and having discussed it, it is also important to know what limitations this might have. Generally speaking, this is a process that is going to require a lot of energy. You are going to spend a lot of energy trying to get this done because you have to put a lot of energy to force materials especially metallic materials to undergo plastic deformation. So, a lot of energy is going to be spent. And, on top of it, this is not ideally suited for industrial production.

So, while you can create definitely macroscopic samples, I mean samples that you can handle with your hand at the end of once it is cool etcetera you can handle with your hand. So, it is a microscopic sample in that sense, but it is not going to be large enough that you can put it to some industrial application, by this process I find some very strong material that I have made.

Now, it is difficult for me to scale up this process to create a car body using this particular process it is not tailor-made for that; whereas our traditional processes such as rolling are tailor-made for that I mean tailor-made for creating this large sheets with which you can do any number of different things. So, so, there are pros and cons when you look at different techniques.

So, our requirement is for validating some model in say superplasticity in this case associated with some phenomenon that is occurring at the nanoscale in this in this case. So, for that combination, these kinds of techniques, the severe plastic deformation technique either equal channel angular extrusion or high-pressure torsion enables us to get those kinds of samples. So, these are very useful technique to have in your lab and then you can do different experiments with it.

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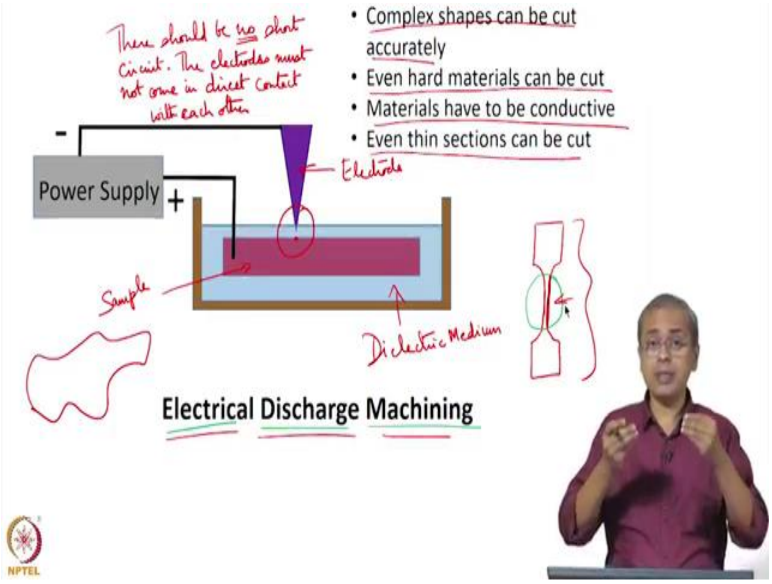

So, now you have made a sample that has nanocrystalline grain size you want to do some mechanical studies on it. So, let us look at some two steps ahead here. What we want to do is that we want to actually create a sample which we can do a tensile test on because we are looking for mechanical properties particularly, we want to see superplastic behavior and at the same time we also want to actually see the grains rotate. So, we want to see the grains reorient themselves, we want to see them sliding grain boundary sliding we want to see. So, that concept we have to have in mind and then like try to see what techniques we can put together experimentally so that we can go towards actually doing that kind of an experiment.

So, having made some sample which is of some reasonable physical dimensions which you can actually handle, you have to now cut a sample out of it which has the dog bone shape you want something like that. So, maybe that is not very well drawn, but that is basically what you want to do. So, this is what we want to create. This is just a schematic, but this is what you will want to create. So, that is how your sample would be. And so, we need to machine this out of the sample that disc that we got from high pressure on torsion we got a disc and the disc has very nanocrystalline grain structure to it from that disc we want to machine this out.

Now, anytime you do this kind of mechanical testing you have to be very careful to ensure that you do not have any defects on the surface. So, this the surface that is having this region which is what you are going to test, it should not have significant cuts on it because then that is the source of stress concentration. And, so, when you do the test you may you will actually prematurely the sample will fail because a crack will start propagating from that cut from that scratch.

So, we want nicely finished samples which are where the surface finish is good so that when you do the test you have a dependable sort of result from it. So, one of the techniques that is used to do that is, this thing called Electrical Discharge Machining ok; so, EDM; so, in fact, EDM is the technique that is called Electrical Discharge Machining, and the idea is quite simple. So, it is you have a workpiece which is what is here. So, that is your sample that you are trying to cut. So, that is here and it acts as an electrode.

So, it is a so, it is, in this case, it is attached to be positive of the power supply and you have another electrode on top this electrode is attached to the negative of the power supply and so, you can build charge and the important thing is these are both conductive materials both of them are the conductive materials in this case and therefore, can be I mean effectively have to be metallic. You cannot have a ceramic material in this particular situation and you are basically going to develop charge.

And, importantly they should not be in direct contact with each other. So, you should not create a short circuit. So, you should not there be no short circuit. So, the electrodes must not come in direct contact with each other. So, that is our requirement. The electrode should not come in direct contact with each other, but at the same time the electrodes must come very close. So, here the electrodes will come extremely close to each other and this is a dielectric medium.

So, by definition, it is a medium that is not a conductive material usually some liquid which can be used there, which is not an electron conductor. So, what happens is we start building charge between the electrode out here. So, the machining electrode and the sample and this charge will actually locally break down that dielectric material and during that break down a spark is generated.

So, if that spark is what erodes the material. So, it will do some small erosion here at that spot. It do a small erosion at that spot and momentarily because you will be pulsing the power supply momentarily that the buildup will halt and the liquid will flow again, the electrolyte will flow again, in this case, the dielectric will flow again and fill the gap. And, you can even do some flushing there to because some debris will be created due to the spark and that is the whole process.

You are creating a spark and that spark is locally damaging your sample and that damage results in some breakdown of that material tiny bit of the material is broken off and then that is washed away from that location and then you create another spark. So, you do this spark by spark by spark and then you can do a machining of that sample. The nice thing is you can create complex shapes in a very accurate way.

So, once you do once you attach this to some computer-aided movement so to speak of the electrode you can create some complex shape. So, some extremely complex shape you want to cut you can actually cut using this. It will create exactly that shape pretty accurately it will create that shape and it is very well done and the nice thing is it can even cut hard materials. Because this is not a physical hit you are not taking a hammer and breaking this sample instead you are creating sparks and the spark is what is doing the cut. And, so, that spark depends really on the voltage that you are applying, the dielectric medium that is present and so on.

So, those are the things that are controlling that spark and the amount of energy that is coming out there. And, therefore, relatively hard material and also you are taking off tiny material at a time tiny amount of material at a time. And, so, you can actually use this for even hard materials except that both the I mean that material has to be electrically conducting. So, that is the most important thing. It has to be electrically conducting because you are going to build up charge using that as an electrode.

So, the materials have to be conductive, you cannot have insulating material there. And, then if the process is nice because it sort of localizes everything to that spot so, even a thin section can be cut. So, even if you have a thin sheet you can cut it very nicely using this process and typically it does a very clean job of the machining. So, that when you are done with that sample you do not have any burrs. You do not have any burring of the sample. So, you do not have rough edges. You have very nice smooth well-defined edges and that is very nice in this process.

So, that is a process we can use for creating samples that we can use for say mechanical testing and which are small in size because bigger samples can be machined in some other way, but which are small in size and are very well finished. So, for that combination of things to happen electrical discharge machining is used.

So, this helps us between the previous step of doing severe plastic deformation and doing this step of electrical discharge machining. We get a fine-grained sample which is nanocrystalline in nature which has got no oxides inside, it has got no porosity inside and it has got the correct kind of shape and dimensions required for us to do a tensile test, but the sample is also relative small. So, all these set of things we can accomplish using this; so, the set of processes.

So, now, we have got the sample. We have now got a sample; we would still like to see we have to see a movement of these grains to see what the superplastic behavior is actually happening in that sample. So, there is something more to be done before for us to see that and typically what is done is you have to thin the sample. So, you have to take the sample and you have to thin it so that for you to see in the electron microscope it has to be thin enough that electrons can go through. And, so that is a very tricky aspect here.

So, you have to create a sample that is thin enough at in this region it is thin enough with excuse me, it is thin enough in this region that electrons will go through. So, you can actually see the grains under the microscope and the idea is that we would like to do a tensile test inside an electron microscope. So, that is the idea and if to the extent that you can do that, you can actually see deformation as it is happening in the electron microscope.

And, this is very tricky because like with anybody working in any area associated with transmission electron microscopy, this requires a lot of patience for you to create a sample of this nature. Because, you can create samples, but by the time you make it thin enough for it to be fit to be seen inside an electron microscope chances are you will do some damage to the sample and it will make your experiment useless.

So, invariably one of the biggest challenges for people working with nanomaterials to the extent that they are trying to use the electron microscope for this activity one of the biggest challenges is for them to make a successful sample and make a successful experiment. So, for an a experiment that you see published in a journal, the amount of experiments that they may have done which failed simply because there was some crack in it or the it was not thin enough or something else was not right about it the number of failures they have to face before they show managed one or two samples that are where they are successful is remarkable.

And, so, that requires a lot of work, it requires a lot of patience, a lot of skill their application of learning from one experiment to the next experiment etcetera and so on and so, then you get this process. So, thinning is actually done by multiple ways. So, we will not get into that in great detail again there is a separate course on electron microscopy in NPTEL set of course, where I think sample preparation is discussed in great detail. There is a process called focused ion beam based thinning.

So, there is some thinning that you do ahead of time before the sample even gets into the microscope and there are ways in which you can use another equipment called FIB a Focused Ion Beam based equipment through which you can thin the sample to some electron thick transparent thicknesses and then that sample is what you will see in your electron microscope.

So, you made this it goes into the electron microscope and you are able to see something from it through it, but that does not complete our process because this is now a static thing. You have you are able to see so if it at a nanocrystalline sample you will see nanocrystals. So, that is fine, that that is one step in it, but we want to see superplasticity. We want to see a mechanism that we are proposing for superplasticity in the microscope. So, for that we have to do more.

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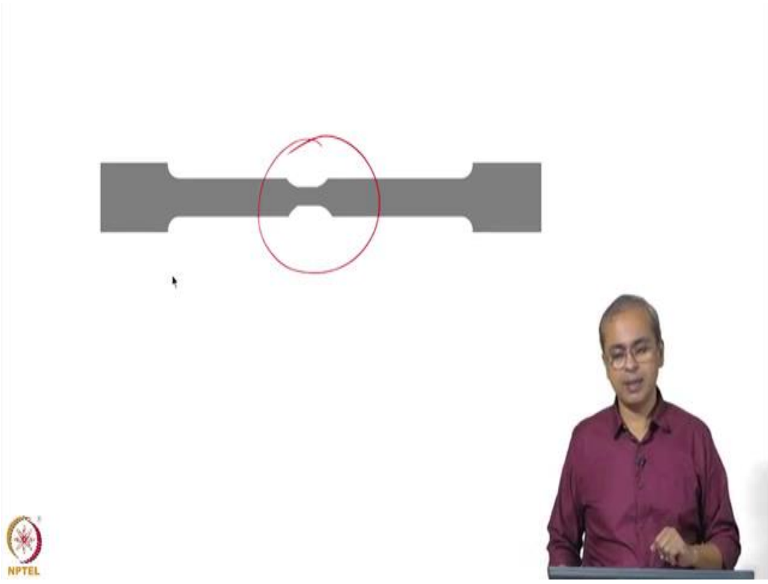

So, if you see here. So, this is your dog bone sample and this is how your test will look. So, this is how the sample expands, necking happens and it starts expanding. So, we want to actually explore this area. So, we want to explore that area and see that in the electron microscope and try to understand what is happening in that area as the deformation process happens.

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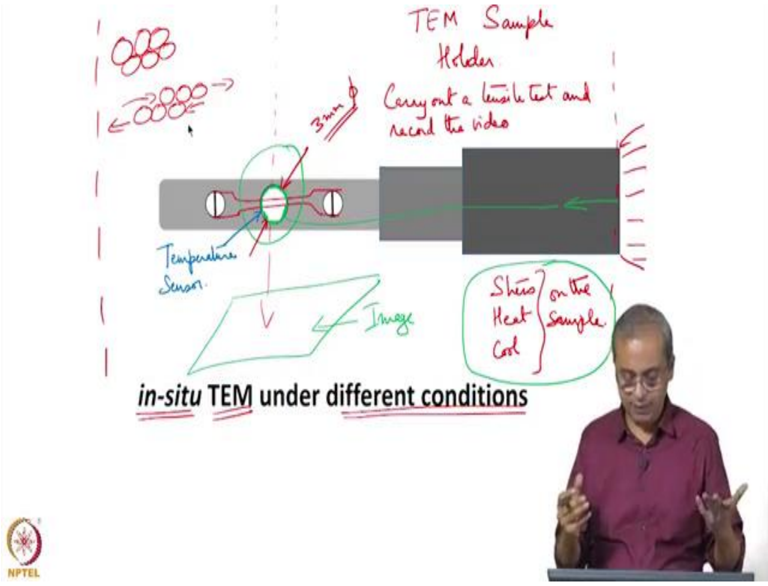

So, for that, it is very useful for us to use do an in-situ experiment in the transmission electron microscope under different conditions. So, the transmission electron microscope usually will have a sample holder and what I am showing you on screen is a; so, this is a TEM sample holder. This is a section of it because actually outside of the screen we will still have something else going out.

So, this part that you see here will now sort of come out of the electron microscope sort of what you see here will all be inside the microscope. This region will now be inside the microscope and the electron beam will come from the top and it will go through this sample this is. So, this is where your sample will sit.

So, usually, we will have a grid here. So, typical TEM experiments where we are dealing with just where the purpose is only to look at say the microstructure, the crystalline structure on over the grain structure of that sample you are going to actually make that sample sit on top of this grid. So, on this grid the sample will sit. So, some sample will sit here and that sample will be seen through the microscope and you will have an image here outside you will see an image and that image can be recorded.

Now, our purpose is different. Our purpose is actually to see superplastic phenomena happening here. So, we have to do something more. So, let us just clear the space a little bit here. So, what we need is a situation where we will actually have a tensile sample sitting here. So, something like that. So, that is our tensile sample and this is the region of that sample that we are going to see. So, this region of that sample is what we are going to see.

So, it turns out many of these electron microscope manufacturers have understood that material scientist; I mean they work with material scientists many of them are I mean they employ material scientists because they are seeing trying to understand what are new things that scientists are trying to do and therefore, they would like to make their microscope capable of doing those things.

So, there are a lot of people who would like to do in-situ in the electron microscope tensile testing. So, therefore, the microscope manufacturers have created stages where you can control various things on the sample. You can control stress on the sample, you can control the temperature of the sample, you can heat the sample or cool the sample. So, all these things you can do. You can apply stress on the sample, you can heat the sample or you can cool the sample. So, all of these you can do and it is actually very small region in which they do it. So, that is why it is very fascinating for us to see how they managed to do it.

So, for example, although I have drawn a circle here for you typically this is only about a 3m 3 millimeters in diameter that a region there is only 3 millimeters in diameter. So, your sample that you are doing a tensile test on is going to be in that range in that size scale. 3 millimeters is the sample width also or less that may be where you are doing your test. In this region this small 3 3-millimeter diameter region you have to do these things which I have just indicated. Any of these things that you want to do.

If you want apply stress that has to happen in that 3-millimetre region; if you have to apply heat it has to happen there; if you have to apply create a cool atmosphere you have to happen make it happen there. So, usually, in fact, they will have a heater here. So, they will have an annular heater which is around the edge and using that they will heat that region. So, with that you send some electricity from outside you are sending electricity into this sample holder. So, that electricity reaches this location here and then you are able to create some heating locally you can create the heating.

You have to also have a temperature sensor because you want to do this experiment at a 600ºC or 700ºC. You cannot just randomly heat the sample you have to ensure that the sample reaches that 700ºC, it stays at 700ºC and you are running the test at 700ºC. So, you have to have a temperature sensor. So, you would also have a temperature sensor there.

So, once you put all of these together, you have a very interesting situation where you can run a tensile test inside the electron microscope inside the transmission electron microscope and you can watch the sample undergoes say tensile test while watching the location of the grains. Again, you have to do one more thing you because this is you do not know exactly when you are doing the tensile test you do not know exactly when which grain is going to start sliding if you are looking for grain boundary sliding, and you do not know how many grains are going to slide.

You do not know as if several of them are going to slide together or you simply going to have individual grains sliding around you going to have one or two twisting around etcetera. So, usually it is an experimental challenge and some of the ways in which people have dealt with this is that they then record the video. Today, these days it is much easier for us because we have all these digital ways of capturing data; so, typically this you do a tensile test and record the video. Carry out a tensile test and record the video.

So, when you do that you have now recorded you are watching, so, the image will be now showing you a lot of crystals you are going to see what is happening to the crystals as the experiment of this tensile test is happening and presumably eventually you will see if you are looking for grain boundary sliding you will start seeing this grain sliding. You will be able to figure out the difference between plastic deformation where a crystal is actually deforming and then changing shape versus a situation which is because you are going to see the crystal change its shape.

You will be able to easily distinguish that from a situation where simply the crystal may be or the grain reorients a little bit with respect to its neighbouring grain and then suddenly slides. And, so, if you had a bunch of grains like that and then after the test you see these lower grains are here and the grains on top have shifted. Then, you can you are able to watch them you were able to watch them move like that and this like this and that moment you are able to actually see in the microscope as it happens.

So, usually they it is the way to do it would be then to record the video and then you can do screen capture of specific short single scenes that you capture where you can see one set of grains holding some orientation and then another one screen capture where the grains have shifted and so, this is the way in which you would do this. So, this is a set of experimental steps that I have described to you.

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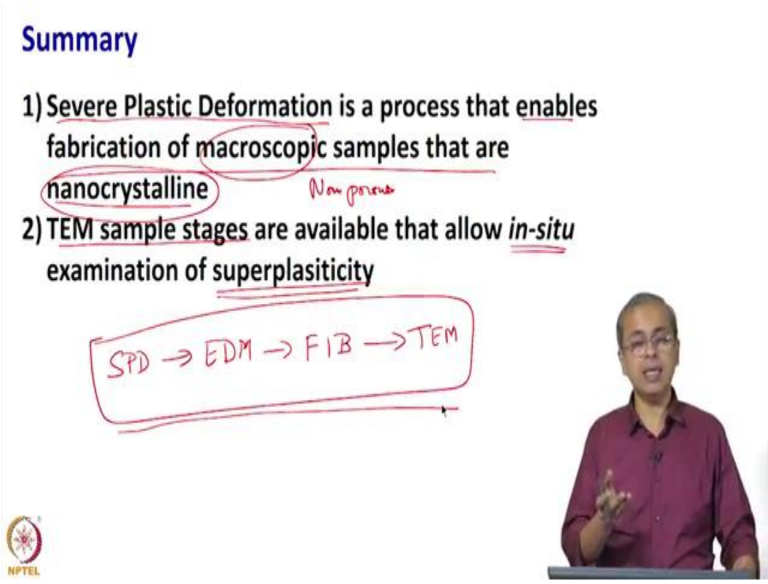

In summary, we used in this particular discussion the possibility that we can use severe plastic deformation as a process that will enable us to fabricate macroscopic samples that are nanocrystalline. So, all these combinations are important that it is macroscopic and, but it is nanocrystalline and also non-porous. So, all of this is accomplished in this technique. And, so, if was trying to study superplasticity inside the electron microscope this is a nice way of going about it.

And, then you have to do electrical discharge machining to get yourself a sample that is a burr-free of accurate shape and at the same time a smooth finish etcetera. So, that is how you will have to start with this SPD superplastic deformation, then you do this EDM that gets you the shape that you want then you use some sample preparation process of which

FIB maybe one focused ion beam technique may be one step in the process that gets you an electron thin sample and then you use the TEM.

So, this combination is a set of experimental processes that you can use for you to study the impact of nanoscale on superplasticity. And, as I pointed out we have the manufacturers give us also a wide range of interesting stages they are called stages they are basically a stage in which you can hold a sample which allows us to do different kinds of in-situ experiments. And, they also will allow us to do an in-situ tensile test which helps us probe the process of the phenomenon of superplasticity based on the material system that we have selected and based on the grain size that we have selected.

So, that is the summary of our class today. We looked at an experimental process to understand how you can probe superplasticity, where you are looking for phenomena that are occurring at the nanocrystalline scale. But you would like to provide proof that in fact, the description that you are giving for the phenomena is that is the actual event that is happening inside the sample.

So, this combination of I mean each of this is a fairly sophisticated technique and potentially as a first-time user of any of these techniques, there are any number of things that can go wrong. So, clearly, it is a lot of work for you to manage to get samples that are good, that are consistent, that are really representative of the system and will bring out the phenomenon that you are trying to prove. But, if you do it you have a very nice result that you can show and share with the rest of the community. So, that is a nice thing that we can do.

So, with this, we will close this class. We will pick up other discussions later.

Thank you.