Nanostructures and Nanomaterials: Characterization and Properties

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Lecture - 44
Nano mechanics

In this lecture, we will learn about nano mechanics because of emergence of nanomaterials. It becomes important to learn about what is happening at the nanoscale and to also extract properties, mechanical properties. Such as hardness, modulus how does it vary with respect to different nano phases which are present in a bulk nanostructured material.

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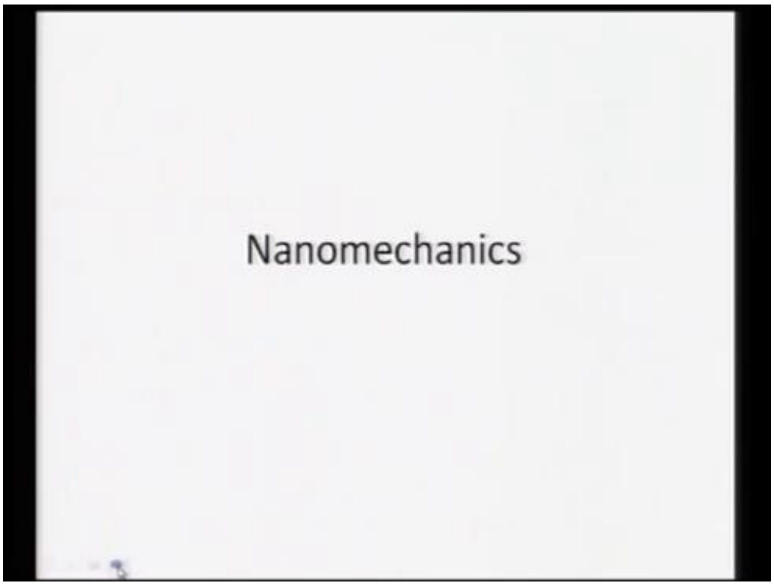

So, in this lecture we will learn about nano mechanics specifically about nano indentation. In order to extract hardness and Young’s' modulus order of these materials.

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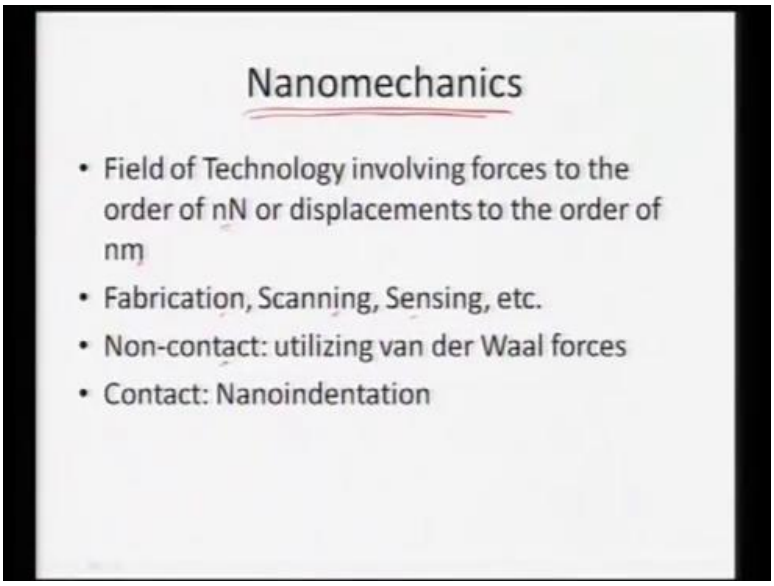

And nano mechanics is the field of technology which involves forces to in the order of nano Newtons or displacement which are to the order of nano meters. So, that is when nano mechanics will deal with materials which have a nano size, nano meter size. So, that we can specifically design this instrumentation. So, that we can precisely measure what all are the displacements or the forces which have been incurred by that particular phase, which is in the nanometer size range.

It also is utilized for fabrication of new materials or scanning a particular property out of it or scanning the morphology or the topography order of it. Also, sensing because once we talk about nanotechnology we also need to sense, what sort of forces which are incurring on a particular material. So, in order to comprise the fabrication scanning sensing the nano mechanics becomes a very, very essential tool. Learning about mechanics, nano mechanics it can again be a non-contact.

In that case, we can utilize van der Waal forces, which is again in the non-contact mode like we can utilize atomic force microscopy. And we can utilize the non-contact mode in terms of emerging the topography or it can again be in the contact mode like for nano indentation. So, coming in contact with the material and then extracting the properties or mechanical properties out of it. So, we can see nano mechanics is a very, very essential way of learning about, what is the response of a nanomaterial. It can be nano phases and how they incorporate in terms of evincing the bulk properties So, we can have a nano properties of each and every individual phase plus contribution of some additional forces. Such as, it can be just porosity or is a interaction between the 2 grains which are in nano size.

So, that can finally, evince the bulk property. So, once know what is the response at a nanometer length scale? We can somehow build them up while using multiline scale hierarchy to achieve a bulk property out of that. So, nano mechanics is a very, very essential way means of comprising what is happening in nano scale and predicting what can happen at a bulk scale. And again, as we see that it is utilized for fabrication or even scanning or even sensing. Usually, in this lecture we will learn about nano indentation.

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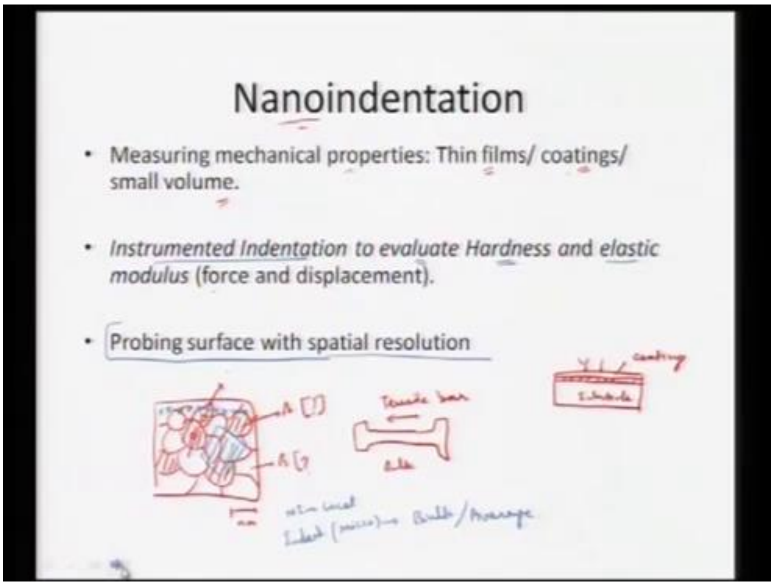

So, let us see about that, so nano indentation is utilized for measuring the mechanical properties of thin films, coatings or even smaller volumes. So, see if you have a microstructure and where the phases are in the nano regime and if you want to extract what is happening, what is the change in the mechanical property because of those particular phases. We need to see what is the property of individual phases? like we have phase A.

So, like we have phase A and we have phase B and assuming that the overall length scale is couple of nanometer. So, then once we make a tensile bar specimen it will give us a property of the bulk. So, we can get a overall property of the bulk using the tensile bar specimen, which can give provide us modulus of the system or stress strain relationship, but if you want to see what is happening, what is the contribution from each and every individual phase. Like of phase A and phase B we need to do the Nano indentation.

So, we can evince or we can extract the properties out of individual phase. Again, if you have a coating, if you have a very thin coating to the order of couple of nanometers of submicron range, it will say if it is very adherent to the substrate. So, we have a substrate and we have a coating and we want to see, what is the response of this coating with respect to the substrate? We might do a tensile bar specimen, tensile bar testing, but if you want to see what is the conclusion of coating alone.

Then it is very, very hard because even if we do indentation a micro indentation. That might actually, have some influence from the substrate also because the coating thickness has to be independent because if we are the thickness of this coating has to be independent. It has to be thick enough, so that we do not see any response from the substrate, but that may not be in reality, that may not occur in reality when we have a submicron size coating on a particular substrate.

So, in those cases nano indentation becomes very, very essential because if you want to extract the properties. The indent or the depth of the indent should be smaller than at least 7 to 10 times of the entire coating thickness. So, that in that way we can avoid the contribution from the substrate and we can get properties only with respect to the coating and not the substrate. So, in order to calculate or measure the mechanical properties of thin films or coatings or even smaller volumes.

So, in this case if we have a phase A and phase B; these are very, very small volumes because we are talking about grain size which is to the order of couple of nanometers. So, we can individually go to a particular spot and do the indentation at those particular points. So, what we can get is property of that individual grain only otherwise, micro indentation what it will do, its size itself is so big that it will basically go on to. So, if you if you do a micro indent it might comprise a much bigger regime of indent and we get an average property.

Whereas, nano indentation will give us a local property, indent which is micron size will give us a bulk or a average property. So, that is why it becomes very, very essential to utilize nano indentation. And nano indetation through instrumented indentation it means, we are consistently measuring the load which is being incurred on a material as well as measuring the indent depth. And that can help us evaluate the hardness as well as the elastic modulus via consistently measuring the force and displacement.

Also, we can have a special resolution, so we can also probe the surface using this particular probe tip and we can also achieve a very good special resolution in this case. So, we can measure property which is very, very nearby, we can measure the properties of the nearby grains. So, that is what is meant by special resolution that it is very good special resolution. So, we can measure properties from localized region, so we can have an array of, very fine array of point where we can extract the properties and we can get very good properties along the each and every pixel as well.

So, that will give us provide a very, very good special resolution. So, nano indentation is a very essential tool in terms of extracting the properties mechanical properties of say thin films or coatings or even small volumes, using this particular technique via measurement of instrumented indentation. It means, we are consistent continuously measuring the load and the displacement and through this we can also achieve very good special resolution.

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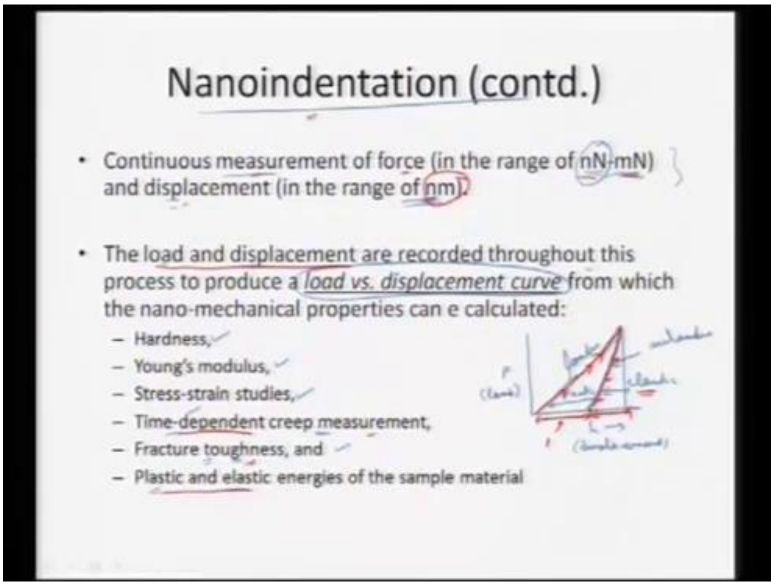

So, nano indentation is nothing but a continuous measurement of force which is to the order of nano Newton to mille Newton. So, we can utilize this nano mechanics, so that will have a load range of around nano Newton's, but we can go up to micro Newton's or even mille Newton's in case when we require. And displacements are to the order of nanometer to it can even go up to micro meter, depending on the sensitivity we require from the instrument kind of the average property we require from the substrate.

So, that is what is basically being attained by a nano indentation. And it is called nano indentation because the overall depth which are in the order of nanometers couple of 100, it can even go on a 100's or 100's or 1000's of nanometer as well, depending on the probe which we utilize or the load which we basically utilize. Again, in this case we are measuring load and displacement throughout this process as a load versus displacement curve.

So, we have a load which is given by p and displacement given by h, so we can directly measure the load as soon as we are loading it, the indentation depth keeps increasing and then we can consistently measure the load displacement curve. So, we have load versus displacement in the matter and then while unloading also, it can have various responses. It can also elastic and plastic contribution which we will talk about later on.

Through which we can also calculate certain mechanical properties such as, hardness Young's' modulus. We can also do some stress strain studies, we can also see what kind of stresses are being incurred, while using cone spherical tip. We can also convert the load into stresses and displacement to strain. So, that can provide us what is the stress strain which can be in how it can be similar to that what we can achieve from a real engineering test of a bulk sample.

We can also realize time dependent creep measurements either way I am entering the stress constant or seeing the temporal, while seeing the temporal response or measuring the strain. That can occur or the strand stress relaxation that can occur at certain load. So, it can also provide us time dependent creep measurements also, we can utilize to find fractured toughness. It means, once we are indenting a ceramic material it will eventually develop some cracking it can be middle cracking or it can be riddle cracking. So, from that we can always measure what is the toughness of the material.

So that is the semi empirical measure way of measuring the fracture toughness also, we can measure the plastic and the elastic energy of those sample. So, if we have a load displacement curve we know that this is the elastic response, so which is being recovered instantaneously. So, this is the nothing but the elastic response and this is the one which is the plastic response as it will recover by time. So, we have loading, so this this part is loading and this part is unloading.

So, once we load the sample indentation depth will keep increasing and then as soon as we unload it, we can see the unloading part is coming down like this. So, it is loading and then unloading will basically come down out here. So, this much is the strain which has been recovered, so this much strain is the one which has been recovered. So, it is the elastic part and the one which will recover later or which will remain as it is the plastic part. So, through this we can also calculate the elastic and plastic energies of the sample material.

So, we can see nano indentation it utilizes continuous measurement of forces in the range of nano Newton's to mille Newton's. And the displacements are again in the range of nanometer to the macro meter range, but mainly in the nanometer range that is why we call it nano indentation. And we continuously measure load and displacement to get this particular load displacement curve from which we can calculate such as, hardness Young's' modulus. We can also convert them into stress strain plot depending on the once, we have the area function available or how we can calculate it to the force or the stress and then the strain.

We can also utilize to nano indentation to find time displacement, creep measurements by varying temperatures, by holding the material at certain stress level or so on or seeing the stress relaxation that can occur. So, we can have many feedback loops associated with this particular testing, to calculate these properties.

We can also calculate fracture toughness as we said to basically, utilize some semi empirical formulas and see what kind of cracking is occurring in the material upon a certain loading is being applied to it. And we can also find plastic and elastic energies of the sample, so this provides the overall potential of this particular testing techniques which is called nano indentation.

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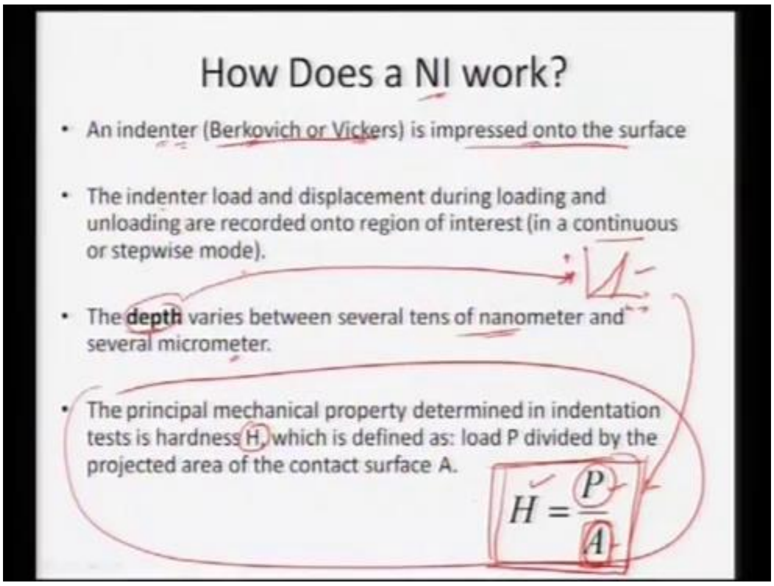

So, how does the nano indenter will work, so we have a indenter it can have a Berkovich tip, which is a pyramidal tip or Vickers tip or it can also be conospherical tip, which is being impressed on to the sample surface. And what happens is indenter load and displacement will occur and those are recorded because this is the instrumented indentation and we record the load and the displacement. And we see the loading and unloading cycle that has been recorded in a continuous or even a step wise mode.

And the depth starts varying around 2 or around several nanometers to, it can go even to micrometer. So, we as I said earlier we have loading and unloading which is occurring and that has been recorded continuously by the instrument. And then we can measure the hardness which is given by the load divided by the projected area. So, we can measure the hardness through the measurement of the load and the area. So, through this we can calculate the hardness very easily. This is a very normal principle of measuring the hardness.

So, the first property is that we utilize a particular indenter it can be Berkovich Vicker or it can be cono spherical indenter as well. That is impressed on a surface we measure the continuous loading and the unloading out here. And right now, we do not know what is the area of the indenter which has being impressed, so it require certain measurements as we will see later on. So, measurement of area or the contact area is not easy as we can see in a regular bulk testing because once we talk about a tip Berkovich or Vickers' or even a conospherical tip, they may not have a perfect geometry.

So, to take care of that we always utilize the standardization of calibration of that particular tip to measure the contact area with respect to the depth of the material. So, that is very much required in any nano indentation instrumentation and the depth is physically varying. That is how we need to measure what is the final depth or the depth which is, what is the relation of p versus h? So, through this we can calculate the hardness which is obtained by the applied load divided by the projected area. So, we can calculate the hardness using nano indentation.

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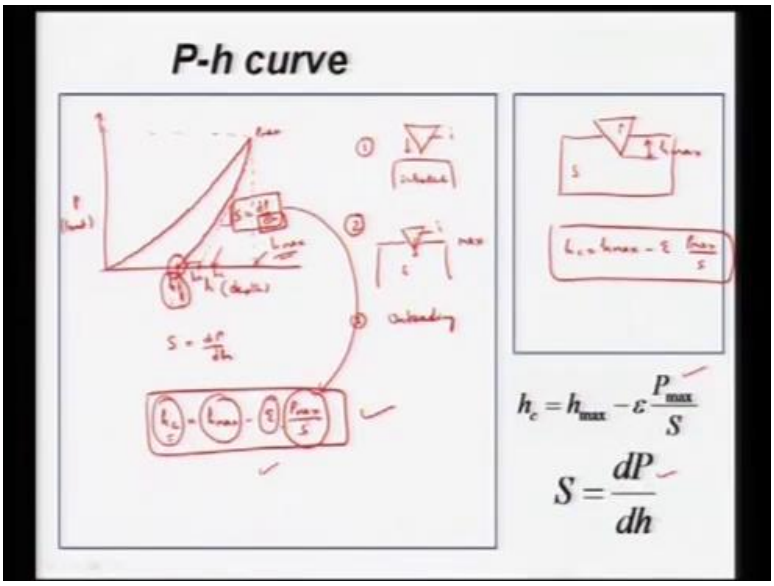

So, typical p h curve basically will look like that we have loading, so we can see that so what do we have an indenter initially. So, we have indenter, so assuming the geometry is like this and this is a substrate. So, we have this indenter and the substrate so initially, it will come in contact so once we start impressing this indenter on the substrate. So, we have a load we have a indentation depth. So, once we start impressing it will start getting into the substrate and it will reach a substrate and this is a indenter.

So, we start loading it so as the load increases the indentation depth also starts increasing. So, we are impressing this indenter on the substrate and it will reach a certain depth. So, that is called h max this is once it has reached a maximum depth it is called h max. And geometry of this particular the curvature of this one will depend on the material it can vary from material to material. So, for very brittle materials it might look like this, for little softer or some hardened material it might look will look very different. So, it can have various geometries as it is, so let us take a typical shape something like this.

So, we have reached now h max and what will happen and then we start unloading it. So, in the first stage we are loading it and if you want to hold it the load might remain constant. So, in this case we might require to relax the material in certain cases to avoid the viscos elastic effects. So, what right now let us not consider that, let us just consider that once we have impressed the sample we are unloading it. So, second step that it has reached a maximum and then third step we start unloading it.

So, in first case we have impresser samples so we have certain maximum depth. So, let us see this is the, so we have a substrate and this is a indenter and this height is nothing but h max. Now, what happens is we start unloading the sample, we start unloading the material. So, what do we have we receive a unloading part like this. So, the slope of this one if we take the slope of this one and while the length it has recovered it is called h final, this point is called h final.

So, what do we have, we had h max after unloading we have received which is called h final and then the slope of this curve is given by S, that is the compliance part of it so we receive d p by d h. And incorporating this we can also see that also the as soon as we the materials recovered instantaneously some to certain h f, but the overall contact depth. The overall contact because what has happened this depth has been achieved when the indenter has been recovered. At the same time it has also gone under some plastic recovery as well.

So, we can see that the elastic just taking the elastic recovery part, we can see that ideally it should have gone out here, but so h c is given as h max minus the strain it has incorporated into p max by s. So, actually this is the h c and incorporating this particular formula, we can see that the h c is basically nothing but it might come up somewhere here. So, what is happening is once we indent the material we are seeing the h max or this is the p max and h max is being achieved here? We indent sample, so we achieve h max it undergoes a recovery which is the final depth which is called h f.

And elastic part is basically given by h c, but the d l h c which is the overall contact depth that is being given by h c. So, we have this particular slope, which is given by d p by d p the elastic part plus the strain which is incurred in the material due to plasticity is also being incorporated. So, we have h max minus epsilon p max by s. So, this is the contact depth which is achieved after the indentation, when materials has undergone a maximum depth of h max. It is the compliance of s and the overall h which is being also taken from here is basically d p by d a. So, that part is being incorporated here with respect to strain.

So, we can see the total h c is given by h max minus epsilon p max by s, so that is the criteria for the finding the h c. So, we can see then the by the load indentation depth we can see what is happening, that we are first taking the indenter impressing it on the material. It is achieving a maximum depth at a maximum load, then while it is recovering it recovers, it is also recovering both elastic as well as the plastic recovery.

So, some part of it has been recovered that is given by h f that comprises of both the elastic as well as plastic part. So, the elastic part is given by h c, so we also reduce that particular part from the actual contact depth. So, h c will be equal to h max minus epsilon d p by d s or p by s. So, that part is basically been given out here. So, this is the other formula which we have utilized earlier on.

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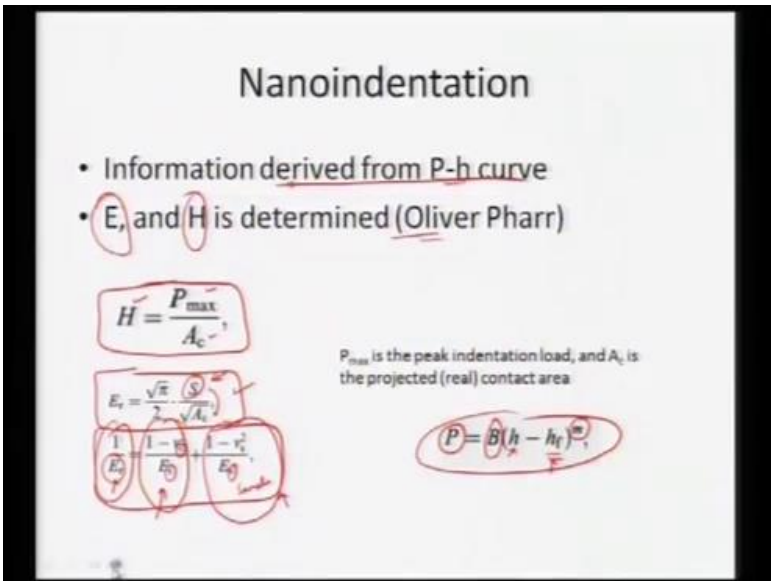

The nano indentation it is very, very essential that we derive the information from p h curve and also, what is happening at just at the recovery is that is the elastic portion. The true elastic part is nothing but the part which is just been released. So, this is the real elastic part which is basically specific to this p-h curve. So, we need to derive information from the p-h curve and E or the Young’s' modulus, elastic modulus and the hardness is derived from the Oliver Pharr method.

So, we first have to have a relationship between the h f and the p. So, we have instantaneous depth h, h f is the final depth of the contact depth and B n m are the fitting parameters. And through that we saw that we can calculate hardness via P max by A c or the contact or the projected area. And the reduced modulus is given by root pi by 2 as it is the compliance and then pi divided by under root A c. So, that part we can see we can achieve it out by that method and again we can get the reduced modulus.

Reduced modulus given by 1 minus nu i square of the indenter divided by the modulus of the indenter, which is that for a diamond and then for plus 1 minus nu s square divided by E s that is for the sample. So, this is called reduced modulus, because it is comprising this formulation for indenter as well that for a sample. So, essentially the samples modulus should be little higher than the E r value. So, that is why we call it as a reduced modulus which is being obtained from this particular equation. So, this is the part we can see out here, that we have reduced modulus which can be obtained from this particular part. To assess nothing but the stiffness which is being obtained from this particular equation.

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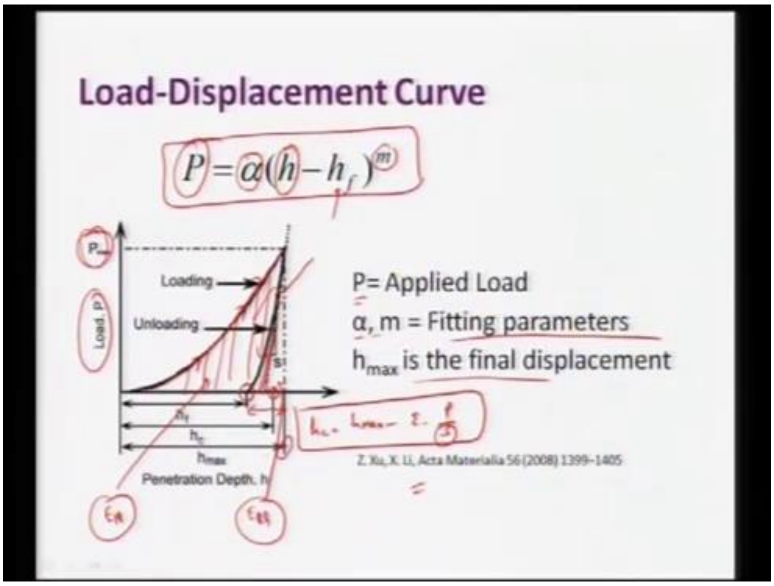

So, we can see that p is given by alpha h minus h f to the power m. Actually the instantaneous indentation penetration depth h f is the final depth, p is the load so m and alpha are nothing but fitting parameters as we saw earlier. So, we have p as the applied load, alpha and m are the fitting parameters and h max is the final displacement, which is given by this particular term and this h c which is actually out here. So, we have a elastic part and then we can identify h c h max minus epsilon p by s. So, we can see the stiffness part is being in the denominator load and everything else.

So, this is the overall relationship between the load displacement curves for the Nano indentation. So, we can see once we apply the load, we have a loading part which basically goes on like this and the unloading part. So, this is the part which is instantaneously recovered this is the elastic region and this is the plastic region. So, plastic, so plastic energy can also be calculated by calculating area and other curve.

So, we can see that this area is nothing but energy, plastic energy and this energy is called the elastic. So, we can also compare the energies of this particular these two regions to basically, compare two different materials how much elastic energy they can store or how much plastic energy they can store. Also while keeping the relationship constant, so for a particular loading or particular dis penetration depth we need to have this comparison.

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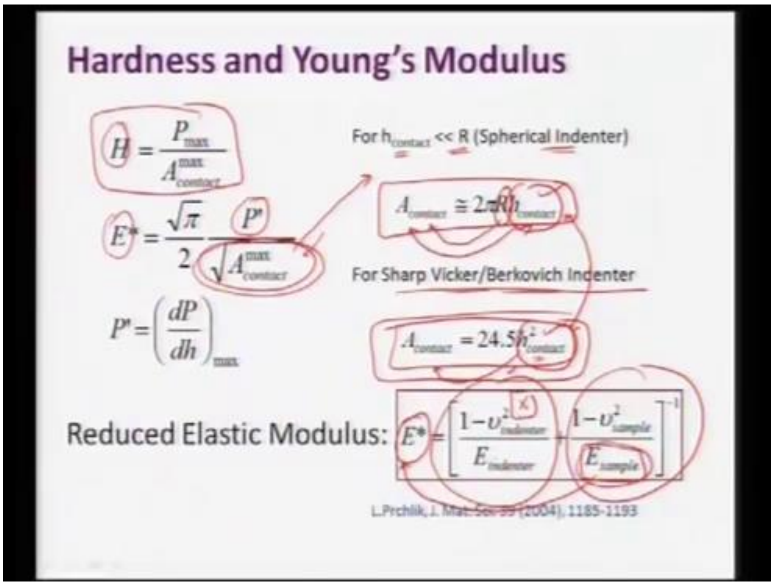

And just stating it forward calculation of area or the contact area is very, very typical very, very critical for this hardness and modulus evaluation. Hardness is given by p maximum by A maximum contact and modulus is given by root pi by 2 p and the stiffness is again given by, p dash is nothing but d p by d p by d h and A is the area of contact for this one. So, what is happening is calculating this part is pretty much difficult and that depends on the geometry of the indenter as well.

So, for a spherical indenter h of contact is much lesser than the radius of the indenter. So, we can get A contact is equal to 2 pi approximately 2 pi r h of the contact depth. So, this thing is little bit easier, but for sharp and Berkovich indenter A contact area comes out to be 24.5 h square. So, we can see that from depth or penetration depth itself we can now, calculate the area of contact. So, from depth of contact we can calculate the area of contact.

Also we can see that for a spherical indenter the contact area depends on the radius, where in the case, of Vickers or Berkovich indenter it is independent of the any other entity. So, just by once we know the h of contact we can always calculate area of contact, but these are for a pure or a real or a for a any defect free Vickers or Berkovich indenter, but in reality what happens is the overall geometry of this indenter is not always perfect. So, what happens is we also add some correction terms, some higher order correction terms as well in this case for Berkovich or Berkovich indenter or even spherical indenter.

So, we will see that we are not incorporating it out here, but the calibration always requires that we also add some correction terms along with this particular A contact. So, eventually what we get is the reduced modulus which comprises 1 by modulus of the indenter as well as 1 by modulus of the sample. So, this is one more typical part of it because then E sample will be little higher than the modulus value what we are getting finally, because once the response is being obtained it is the response of sample plus that of a indenter.

So, we if your calculating the modulus the kind of resistance which is being offered is by both by the tip as well as by the sample. So, we always need to extract or subtract the contribution which is coming from the tip. So, that is the reason we also need to subtract this part from the reduced modulus. So, what we get is little higher modulus for the sample.

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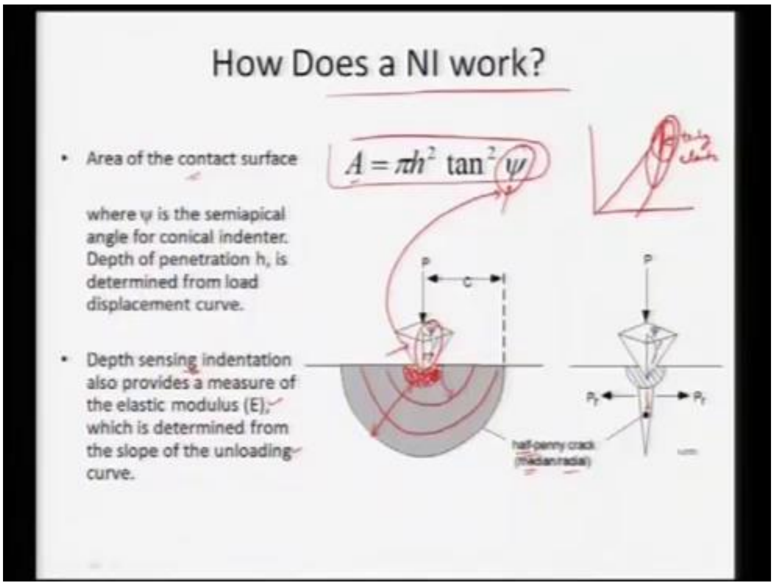

So, how does the nano indentation works initially, we need to measure the overall contact surface area. So, we have certain equation available for that semi where is size is the semi apical angle for the conical indenter. So, in case of Berkovich indenter we always need to have a equivalent conical indenter, so this is for a conical indenter this value Psi. So, for a Berkovich indenter we always have a comparative value of Psi for a conical indenter.

So, depending on the angularity of or the geometry of this indenter Berkovich or Vickers indenter will always have Psi for a comparative conical. So, that it behaves like a conical indenter as well. So, we can see the A depends on the Phi h square 10 square Psi and through this we can through depth sensing we can always get the value of E, which is basically determined from the unloading part. So, we can see for loading and unloading this is the region which is truly elastic, so this region is truly elastic.

So, from this region we can always identify what is the slope and we can convert it back to the reduced modulus from this particular dependence. And also once we have this indenter we can also generate half penny cracks, which can be median or radial. So, we can see once we have a indenter it will generate a very dramatic or drastic plastic field around it. So, that part again depends on the geometry of the indenter.

So, in case of Berkovich or Vickers indenter this plastic region has certain length certain depth as well or it can offer a brittle material it can directly initiate a crack as well. So, we can either have some sort of a plastic or an elastic regime with certain depths. Which are being already been modeled by certain researches or it can also generate crack when the material is very, very brittle. So, it can also help opening the crack as well, so that is given by this particular force or the load which is required up to open this particular crack.

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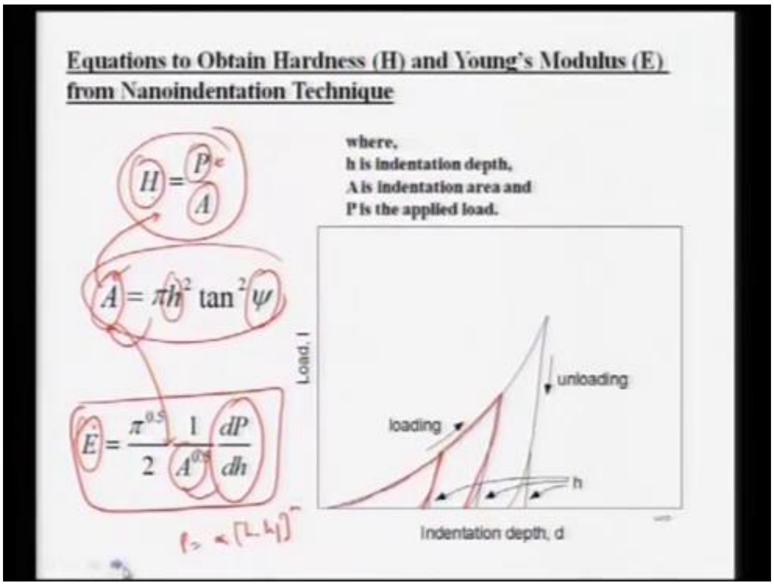

So, we can see this once we are loading it we can unload it loading, unloading. So, from that we can always find the relationship or the hardness dependence of the load. So, we can also achieve very marginal difference in the hardness, because of the kind of load which we utilize and that is given by the hardness is given by load by the area. And then from that we can always identify what is the A value from the semi apical angle for a conical indenter.

So, from the depth we can always find what is the contact area? And through that we can always find what is the hardness. And again, the modulus can also be obtained once we have this particular relationship available of p versus h minus h max. So, we have p equal to some fitting parameter h minus h final to the power of say some m. So, through that we can always identify this relationship of d p by d h and once we know A, we can always fit in here to find the modulus and this is nothing but the reduced modulus. So, we always need to back calculate the modulus of the sample also. So, this is the overall idea of obtaining the hardness and Young’s' modulus from the nano indentation.

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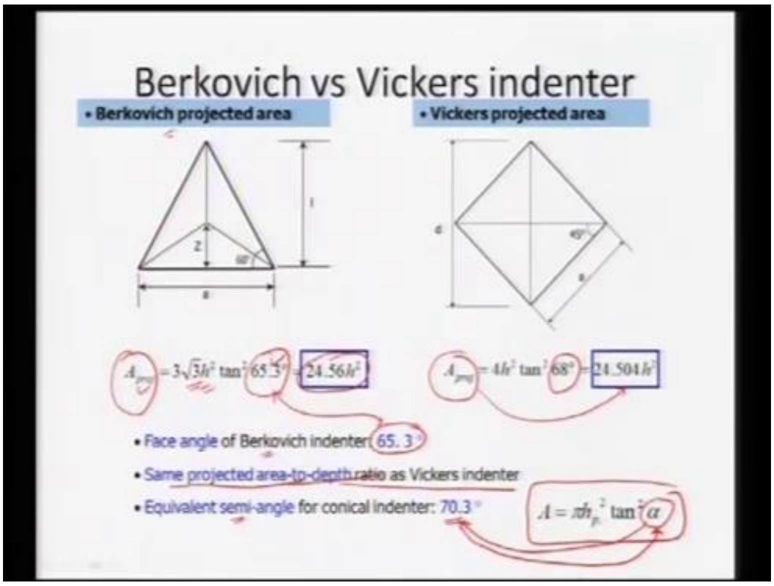

From just two say, how what sort of constants we can achieve from A projected we saw that it was 24.5 h square. So, for a Berkovich projected area we can always go about calculating the Psi, which is for a Berkovich tip it the equivalent Psi angle is 65.3 for Vickers it is 68. So, depending on the geometry we can always find, what is the projection area for a Berkovich strip which comes out 24.56 h square? Whereas for Vickers it comes out to be around 24.504 h square.

And for Berkovich tip as I said it is 65.3 is the equivalent semi apical angle, which is for conical indenter. And equivalent semi angle for conical indenter is around 70.3 as well. So, semi angle is different than the semi apical angle and this provides a similar projected area to depth ratio as it for a Vickers indenter. So, by utilizing this particular geometry we can also see that, we can also utilize the conical angle with respect to alpha. So, we can get alpha of 70.3 as well in this particular case.

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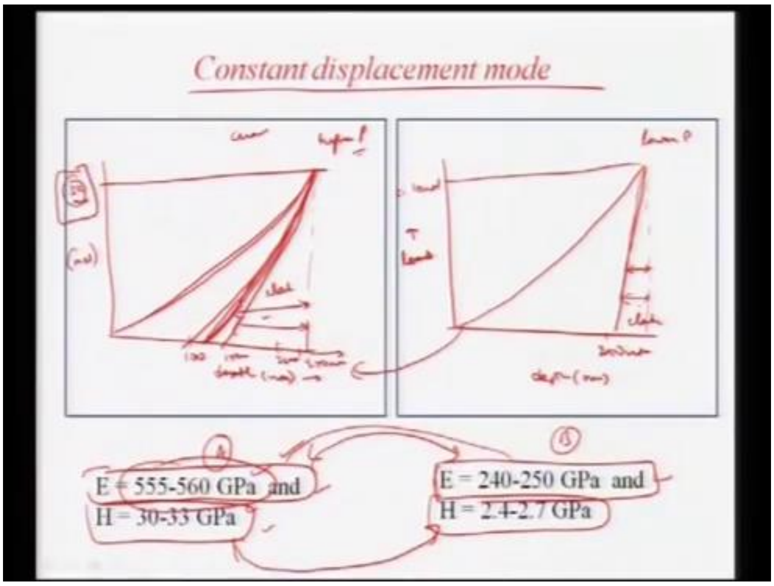

And just by saying that by measuring the constant displacement mode also. Say, if you have two different materials, material A with much higher modulus and much higher hardness. And then second material is B, which is much lower modulus and very low hardness as well. So, just comparing them we can see that how how different of a p-h curve we can obtain in these two cases.

So, in this case if we are obtaining say just for example, say if you have a load of to the order of mille Newton’s and displacement to the order of couple of 100’s. So, we can see 100 200 nano meter and say nano say, we can go up to say around 50’s of mille Newton’s. So, we can see that let us say around 200 250 we can achieve a curve and then it will also revert back.

So, what we are seeing in this case is that for a very high load we are getting a very low displacement. So, we have h nanometer we have a depth of around 150 nano meter or so for a load of 50 mille Newton's. So, in this case we can get very high modulus as well as very high hardness because the maximum depth also is very, very low in this particular case. So, it is to the order of couple of say around 250 nano meter for a load as high as 50 mille Newton's.

So, in this case we can see it as it is might be true for a ceramic material also because the kind of recovery it is undergoing it is also very, very elastic in nature. So, we can see that much of the elastic, much of the load depth has been recovered so it is more like a elastic material. So, that is why we can say much of it has been recovered as well, but in the second case what we can see that even smaller nodes say around 10 10's of mille Newton can generate very high displacement also, the recovery also is very, very low.

So, we can see say the curve is like this, so in this case we are achieving say similar sort of displacement, but in this case we can see it is around 300 or 250 nano meter and around say 10's of 5 to 10's of mille Newton's. So, what we are seeing here is this load and this is the depth nanometer. So, we can see that very low loads are creating the similar sort of displacement. So, in this case we in the first case where we had 50 mille Newton's to give us displacement of 250.

In the second case, 5 to 10 mille Newtons are giving as the displacement of around 250 to 300 nanometer. Also, the recovery is very, very low the displacement is very, very high that means, the hardness is very, very low and second case the recovery is pretty less it means the slope is very, very less. So, in that case slope is little bit high, so in that case we can see that E value is pretty low it means, the recovery is very, very less. So, when slope is high the compliance is pretty high it means, the material is lesser elastic so that that part we can see here.

So, we can see that there is much more very, very less of elasticity in comparison to that of the first case. So, we can see that difference in the modulus and difference in the hardness. Difference in hard hardness is arising because of similar loads say in this case similar, displacement this for a lower is achieved in lower load in this case, it is achieved at higher load.

So, that is why we can see that in constant displacement mode say in this case, we had displacement constant at say around 250 to 300 nano meter. So, for a lower load we can achieve that particular depth in the first it is at much more higher load. So, that is telling the difference in the p-h curve itself what we can achieve for two different material. So, in first case we have high modulus we see very lower slope. In second case, it is very high slope, unloading slope also the recovery is pretty high in the first case in comparison to second case. So, that can tell us about the difference between two different materials.

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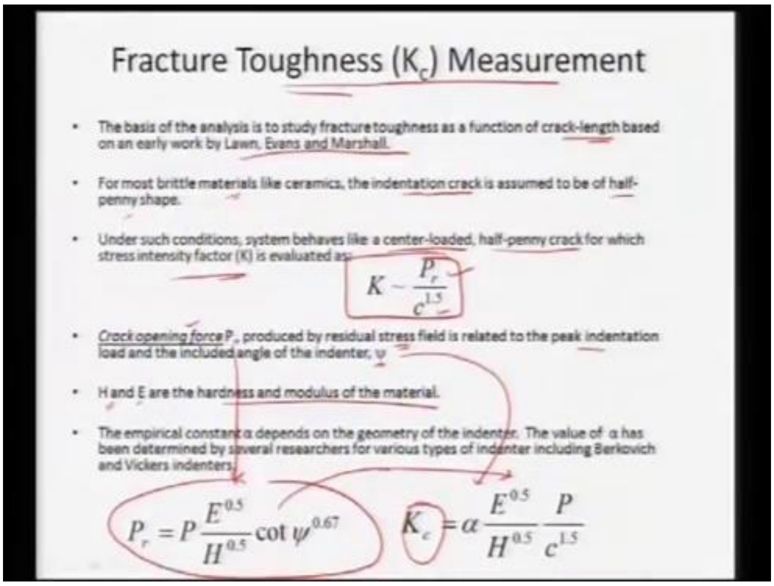

So, fracture toughness measurement can be done by this particular technique as well nano indentation. So, basically we people utilize this Lawns, Evans and Marshalls equation, which is basically been earlier done by these three scientists. That basically, takes care of the crack length that emanates when an indentation is being done. So, mainly for brittle ceramics the indentation crack which is being generated is assumed to be around half penny shape.

And under those conditions system will behave like a center loaded half penny crack from which the stress intensity factor K can be evaluated as P r divided by C to the power 1.5. That is, P r is the crack opening force and C is the crack length that is being generated. So, crack opening force P r which is produced by the residual stress field. That results when the peak indentation load is starts to relax and that is being matched by the fractured toughness of the material itself. And Psi is nothing but a semi angle or the semi apical angle of the indenter and hard H and E are the hardness and modulus of the material.

So, through that we can also see that how P r can be related to the modulus and hardness and that is being utilized in calculating the he fractured toughness of the material as well. So, through that we can calculate what is the crack opening force that is being resulting out from here and then from that we can always calculate, what is the fractured toughness which can which is responsible for this crack destruction.

So, overall fracture toughness we indent the material a crack is effectively generated and when the crack is being restricted that is the correctness of the material, that for a particular load when the stress intensity factor matches the fracture toughness. Basically, the crack will tend to stop and by measuring that particular entity we can always find, what is the fracture toughness of the material?

And for this ceramics we always assume that the crack has half penny crack and then we require certain load to stretch it further. And again, the value of certain constant which is like alpha which is already been determined for say Berkovich or Vickers indenter by using certain semi empirical equations. And again, they depend on the geometry of the indenter also because they induce a different stress field at the tip of the indenter. So, evaluating that also is very, very critical for measuring the fracture toughness.

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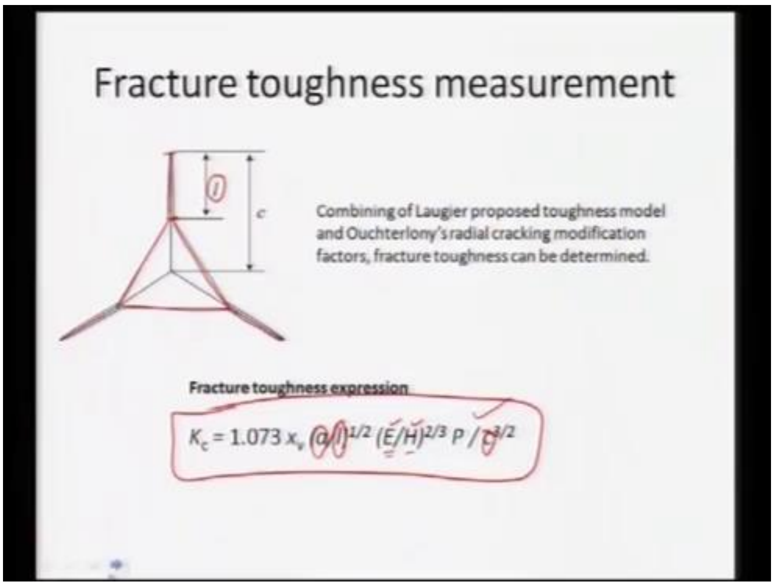

So, in this case again there have been some more modifications to decide what is happening exactly in case of a indentation crack. So, we can see for a Berkovich tip we get an impression and then we also start seeing some little, which can emanate from the corners. And l is the length of the crack from the edge and then C is the length from the center and utilizing those formulation we can see that l and C are being utilized in the equation, alpha is again the geometrical constant and E and H are the modulus and the hardness of the material.

So, fracture toughness can be calculated using by using this particular semi empirical equation, which utilizes the crack which is emanating from the edges and also it is length from the center. So, we can always find the fracture toughness of the material by using nano indentation technique by utilizing a force enough to initiate the crack and that crack is being basically being restricted.

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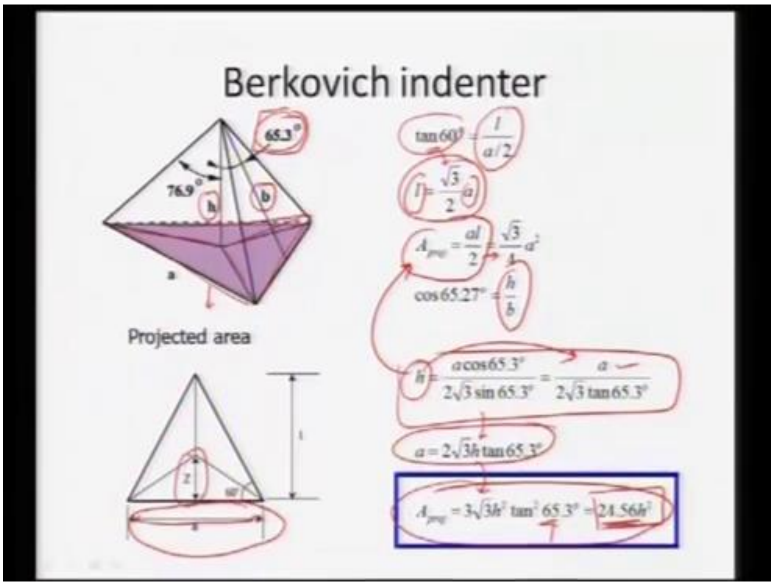

And again, just coming to the Berkovich the semi apical angle of 65.3 or equivalent angle of 70.3 also is out there. So, we can basically what we can see here is the overall a projection area, how it is dependent on the semi apical angles that is 65.3 and it comes out to be 24.56. So, just to show a relation what is happening is once we have a indenter kind of projected area, which is the projected area on this particular part. So, we can see the overall projected area will come out basically for this particular field and then we can see, what is the overall relation between all of them?

And then, measuring the h in terms of A what is the width of this particular edge and from that we can always calculate, what is the projected area that is being resulting from this Berkovich indenter and that comes out to be around 24.56. So, seeing the relations out here we have 10 60 that is l by a by 2 and then we can always have 10 60 in terms of the values. And then we can see relationship between l and a and then the projected area is given by a l by 2.

So, that results the overall geometry and then we also relate b and h by utilizing this semi cone, semi apical angle that is h by b. And then we can always relate what is h and then relate it to the and achieve it is value and then finally, find the projection area from that by utilizing h and a. And then we can finally, get the overall relationship out here.

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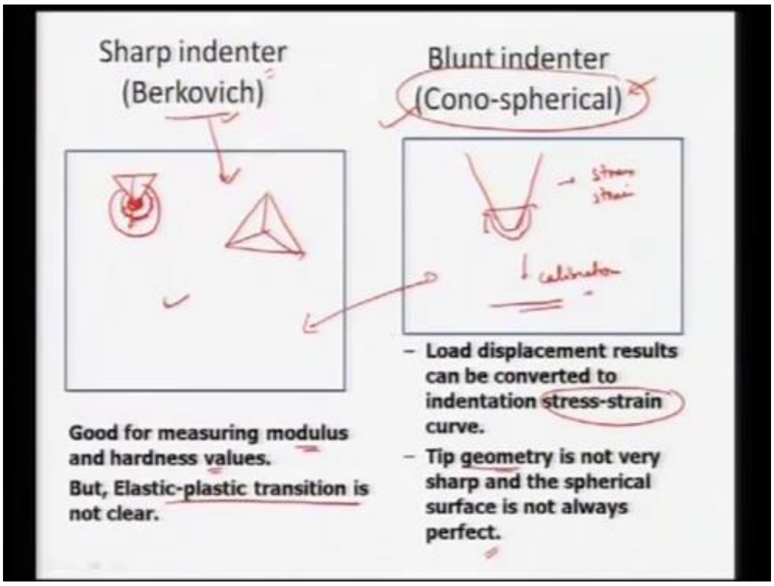

Again, there are many indenters which are also available like Berkovich or cono spherical and they have their own functionality or the own dependence, why because what is happening is. Like in Berkovich tip we have very sharp tip and very sharp tip will induce very high plastic strains in case of a hardening material. It can induce cracking very effectively in brittle ceramics as well. So, basically it is very good for measuring modulus and hardness values, but in this case elastic to plastic transition is not clear because once we have plastic fields around it and elastic fields around it. The transition when it is going from plastic to elastic will be very, very unclear or it is not clear in the case of Berkovich tip, but we can get very nice indents, but for Berkovich it will be it will be like pyramidal. So, it will be more like this.

So, we can see very nice impressions using the Berkovich tip and it will be very, very sharp. So, this can provide us a very good estimation of the elastic and plastic the hardness and the modulus values. So, that is very good for measurement of the modulus and the hardness values, but sometimes many times we also need to see what is happening, peculiar to the material. If you want to utilize what is the stress strain relationship, then we want to go for a cono spherical material which actually appears more like this.

That gives us the precise indication of precise relationship between the depth and the contact. So, we can always estimate what is the stress and strain that is emanating from the tip with respect to the material. In this case, the stress field is so high that is very high to predict the relationship between the stress that is being generated via implication of this or impression of this particular type of tip in case, of Berkovich tip. Whereas, in cono spherical tip it is much more easier and tip geometry is not very sharp also the spherical surface is not always perfect.

So, that also is some problem with that so we always require some calibration to be done for measurement of the contact area, but this is again very, very good for doing for imparting very low stress value on a sample surface. So, like if you have to measure a very soft sample like polymers or biological sample cono spherical tip might be very, very effective in measuring the modulus or hardness of that particular material, whereas sharp Berkovich indenter tip will just start to penetrate those soft materials. So, evaluating soft materials it might be much better to use the cono spherical tip, but for little harder materials such as, ceramics or metals Berkovich tip might work much better in comparison to cono spherical tips. So, there are certain gives and takes between the kind of geometries which we geometry of indenter that we finally tap, so this is also very, very essential component to be considered.

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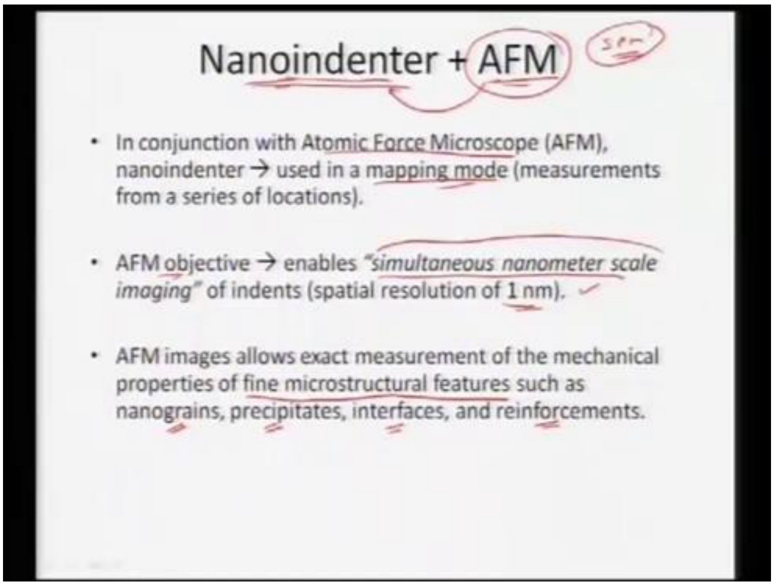

And some cases, we also require mapping of the surface like we can have a indentation done. Then we also want to see, what is the topography of that particular indent? So, we can utilize other something called atomic force microscopy or we can also utilize the same tip as imaging source by via using scanning probe microscope. So, we can use the same tip and we can scan it more like an SEM, instead of electron beam here we are just sending the indenter itself to scan the surface.

So, in conjunction with atomic force microscope the nano indentation can be very, very essential for mapping the surface. So, we can see where we have indent in the surface and which surface or which phase is resulting that particular mechanical property. Also, it also in AFM objective also enables to simultaneously measure a nanometer length scale because once we utilizing the scanning probe microscope that might be very hard that might also damage the surface. Whereas, in AFM we can utilize very low forces and image the surfaces as low as 1 nano meter special resolution.

So, they can provide us very good pictures also of the indent also of the surface. So, a simultaneous nano meter scale type of measuring can be done via using atomic force microscope. And those limits can be little coarser when we utilize a scanning probe microscope. At the same time we can also know exactly where the measurement is coming from. If you scan the surface using AFM we can exactly pinpoint which finer micro structural feature it is resulting that particular mechanical property.

Whether it is coming from a nano grain, coming from a precipitate, coming from an interface or coming from a reinforcement. So, we can exactly pinpoint what the AFM information is providing, we can exactly pinpoint by seeing the indentation where it is coming from. So, that addition of AFM becomes very, very important in nano indenter though we are getting the property mechanical property using Nano indenter AFM is telling us exactly where is coming from.

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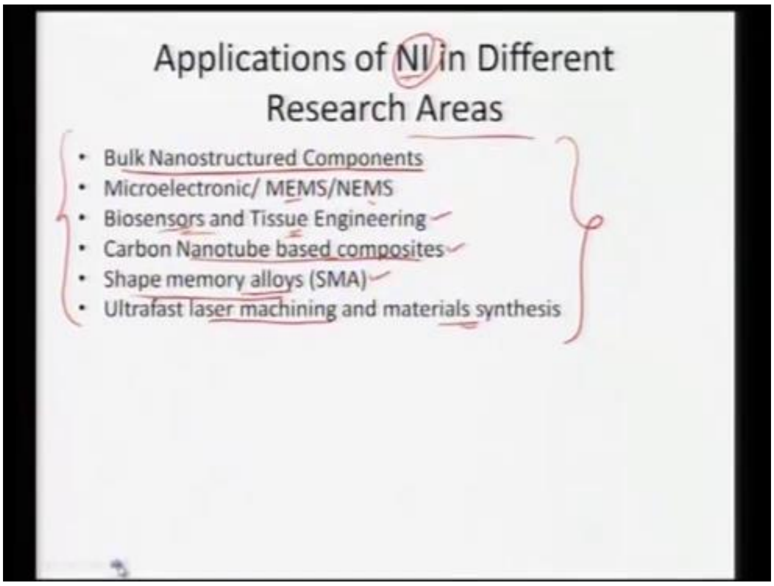

And also, nano indentation has been utilized in many, many different research areas for evaluating mechanical properties of bulk nanostructured components. In specifically for microelectronic mechanical systems or nano electronic mechanical systems, biosensors even tissue engineering. So, we can test the mechanical properties of a say healthy tissues or rough cartilages or say toucan beak and so many things of biological entities. Also, to utilize mechanical properties of finding mechanical properties of carbon nano tube based composites.

So, once we have a reinforcement of a very fine entity such as, carbon nano tube we want to incorporate what is happening at the interface, what is the role of carbon nano tube in improving the mechanical properties which is modulus or hardness? Those have been widely utilized or illustrated by various researches. In shape memory alloys again we can have some sort of martensitic transformation or twinning that can occur. So, those can also be very well identified by using nano indentation also less ultra-fast laser machining and material synthesizes.

So, what sort of changes that might occur that can also be interacted by the Nano indentation? So, we can see there are myriad applications of nano indentation which can be utilized in the exact application of these particular fields and then engineering. Basically, engineering it in terms of certain applications, such as biosensors or even tissue engineering or utilizing these composites for certain applications nems, mems or saying about the quality control by utilizing this particular technique.

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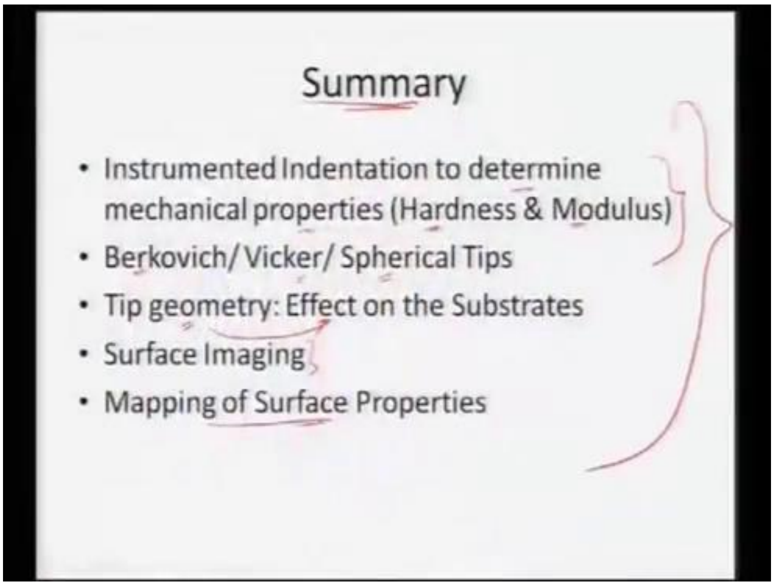

So, in summary we can see that instrument indentation is utilized to determine the mechanical properties, which is hardness and modulus by using Oliver Pharr method. And depending, on the geometry of this particular tip we can utilize Berkovich, Vickers or cono spherical tip for certain various applications. Mainly, the indentation is utilized for evaluating the mechanical properties, hardness modulus we can map the surface once we have an attachment of say AFM atomic force microscope.

We can also see what is the role of particular phase or particular grain or particular precipitate in dictating the overall mechanical property? We can also read about, we can also learn about the stress relaxation that is occurring or even the creep deformation that is occurring in the material. So, this is a very strong technique to give us provide us certain mechanical property evaluation of these nanomaterials. Also, tip geometry as we saw it can have very strong effect on the substrate and that like Berkovich tip is very good for metal and ceramics, whereas cono spherical tips might be good for polymers and biological material for extracting the modulus and hardness. We can also do surface imaging by using a AFM or atomic force microscope or even scanning probe microscope and that will tell us exactly where the data is coming from to map the surface. So, we can once we map the surface we can somehow relate it to the multi scale hierarchy like if you have nano entities, how they club up together, how do they combine together to give us a mechanical property at the bulk scale. So, with this I will end my lecture.

Thank you.