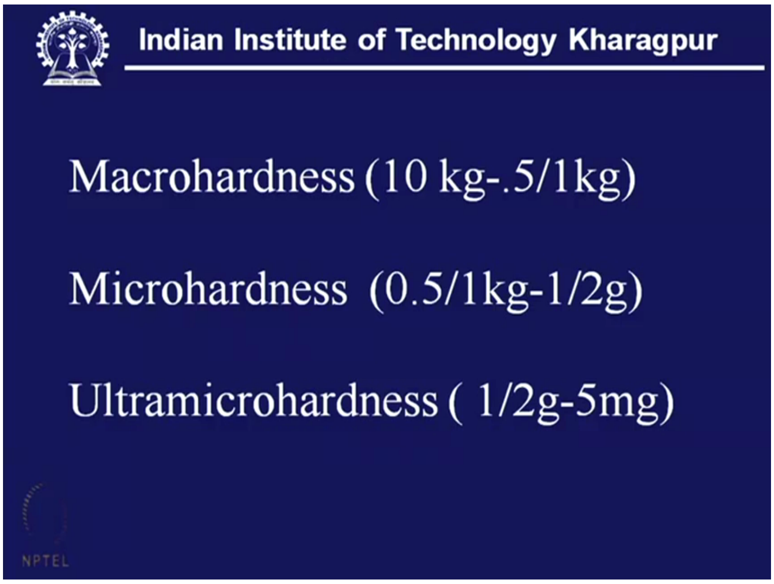
Technology of Surface Coating
Professor A.K Chattopadhyay
Department of Mechnical Engineering
Indian Institute of Technology, Kharagpur
Lecture-35
Assessment of Coating Hardness

Assessment of coating hardness, now in all coating activities particularly for those coatings which are going to be mechanically functional, there this hardness measurement is of prime importance. This is because of the simple reason that this hardness on the surface of the coating that can act like a guidance or it is an index of the performance of the coating particularly in those areas where we need wear resistance on the coating and this coating is going to be used in various kind of sliding action. Now what happens in this case, when a material takes the shape or assume any geometry there also do we need hardness on the material and that we call bulk hardness, but when this material is synthesised in the form of a coating or a film that we call the hardness of the coating.

Now this hardness of the coating this depends upon primarily on the material of construction that means the material which is going to be used for building the coating + various aspects of this coating right from physical to chemical that means the structure of the coating, its morphology then say for example, morphology within the morphology we should consider the porosity, density, the structure how it is built, whether it is a porous columnas structure or dense columnas structure or it is an equiaxed one this is one thing, whether this is sub-stoichiometry or really it is a stoichiometric coating then comes also one important thing that this stress which is built within the coating, whether the coating is too brittle, so all these issues should be considered before putting this coating in any functional application.

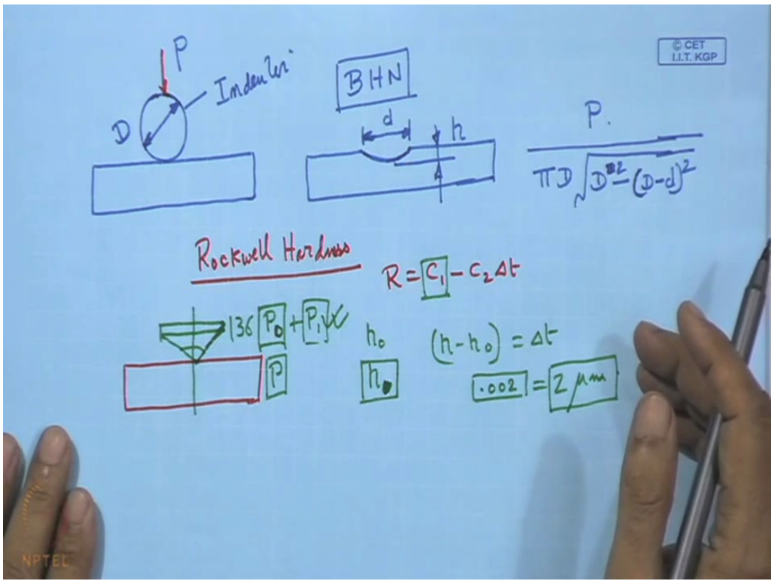
And in the whole process this measurement of surface hardness of the coating this is an important step and this is an important step in characterisation of the coating. So what we can say reasonably that this is going to be a preliminary screening of the coating and if we have adequate hardness because of film material which is already known for its bulk property so by this various coating process by particularly correct choice of the parameters we can indeed achieve a very good value of this hardness and which may not be attainable by the bulk. So here also the importance of hardness of this coating is can be understandable and this is what we can summarise a significance of surface hardness of the coating.

(Refer Slide Time: 4:15)



Now here what we find that this hardness this is actually a technique of measurement and in this technique of measurement this is already well as well-established technique what we are showing here that means macro hardness then we have micro hardness and finally it comes to the ultra micro hardness. So macro hardness that is mostly applicable for a bulk material that means the material is created by some metallurgical process and after that we should assess its bulk hardness and in that there are standard processes like Brinell hardness testing or Rockwell hardness testing, so these are the routine thing for measuring or assessing the macro hardness.

(Refer Slide Time: 5:49)



So it is just to introduce this thing how it is done so it is a ball which is going to be the indenter and then we have the material which is going to be tested that means this is the material and then loading has to be done from this side and then we expect little bit of deformation on this specimen and this is done by this ball so this ball curvature that will be produced here and this is going to be the deformation. And this deformation is an index of this hardness but in Brinell hardness testing one number is introduced and that is we call Brinell hardness number and that is given by this load divided by this area under indentation.

So if this is the diameter of indentation then this d and h they have certain relation with a given diameter of the ball so this can be reasonably written as Pie into D root over D – sorry so this is actually going to be the value of h and from the spell of h and this diameter of the ball this is this denominator is going to give this area under indentation. So it is actually pie D into h and this h is geometrically related by this capital D square – within bracket capital D – small d Whole Square so this is actually the Brinell hardness number in fact, this is load divided by area under indentation.

Obviously the accuracy of this measurement depends upon the rigidity of the ball which is going to be a hardened steel ball so obviously this hardness of the surface and the hardness of the ball there is some restriction in use of this process so if the surface is very hard then this process is not recommended because with high value of load there could be some local deformation or bulk deformation of the ball and the whole process there may be some error may equip in, so these are the limitations so this is load divided by area under indentation. Now here what we can find that Rockwell, Rockwell number Rockwell hardness this is not load divided by area under indentation but this is a number which can be given in the form C 1 – C 2 into Delta t.

Now this Delta t is actually this is the surface and on that we have one indenter and this indenter is actually sharp point and in this case it is actually a diamond cone which is called the Brill so this diamond cone with 136 degree this cone angle that is pressed against this material and in this case what we measure so there is one minor load and the major load so this is actually minor load and then there is a major load by adding someone some load here. So when this P 0 + P 1 added later on that becomes the major load, so we have actually penetration because of P O and then penetration because of h, penetration h because of this total load P O + P 1 and that gives us h.

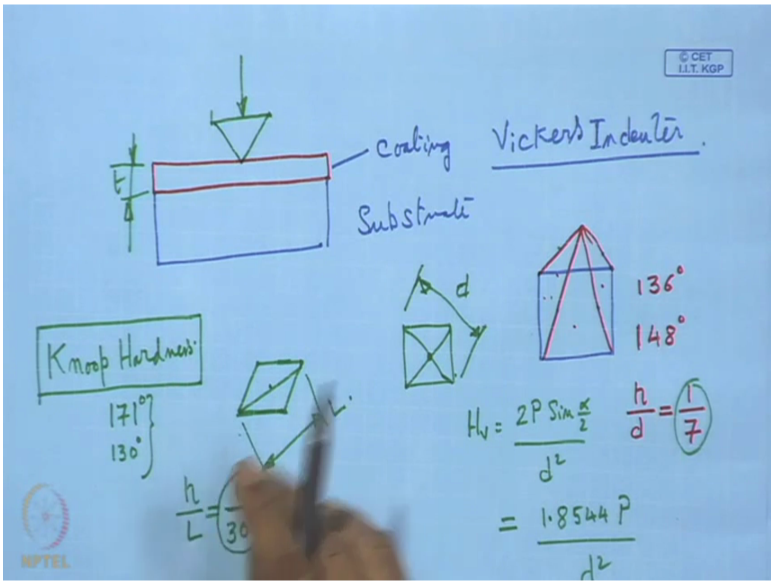
And this penetration is measured after removal of this additional load but retaining the original primary load, so this is the initial load remains there and then we measure h. So this h – h O that is actually the penetration and that is in fact that equal to Delta t so this is actually penetration. However, Brinell number is given following this equation that means higher the penetration, lower will be the number and lower the penetration higher will be the number and this equation can show that number. Now here each one unit of this Rockwell hardness that is equivalent to 0.002 millimetres that means this is equal to 2 microns that means a penetration of 2 micron that is equivalent to one unit of this Rockwell hardness number.

So harder the material less will be the penetration, naturally since C 1 is positive so that will give us a higher value. So these are the 2 though they are useful but they are in the domain of macro hardness and in the macro hardness we have also seen that the load is quite high and with this load normally the bulk hardness is assessed. Now comes the micro hardness, so micro hardness actually it is how this whole load is controlled, what is the magnitude of the load and what is the penetration depth say for example, this is penetration depth or here this is the penetration depth and this is just not few micron but micro hardness when we talk about micro hardness.

This penetration depth that may be within few tens of microns or it can be even reduced or confined within 10s of a micron, the whole idea here is that in a material after the heat treatment or as a as such the material can have different faces on the top surface and each face has its own characteristics and one of the characteristics will be some mechanical property and that can be very well identified as one of the hardness so one face may show one level of hardness while the another face that can be quite high or quite low so with this micro hardness technique or the apparatus one should be able to determine the hardness of each face so that area can be well-recognised and focused under the microscope and with this diamond pointed indenter with a particular load it should be possible to determine the micro hardness in a particular location indicating one particular face.

So this is number 1 and what is number 2? In a manufacturer surface which is already machined, ground and already attained some finish which is totally complete, the job is complete in every respect and in that case if one like to assess the hardness of the finished product, in that case also this micro hardness can find its use because this is not going to make any dent on the surface, superficial or very visual so it is just in the micron level but quality control can be very well done and by this process also the surface mechanical hardness value can be very well assessed on a otherwise complete job. Now the question is we are talking about coating, the coating means here we are not referring to measurement of hardness on a bulk geometric but on a coating.

(Refer Slide Time: 16:21)



So if we put this illustration, this is a coating and then just beneath it we have the substrate so this is coating and substrate, in that case this Micro hardness indenter which we call Vickers indenter and it is actually a square pyramid that means in Brinell, a ball is used say 10 millimetre diameter ball, in Rockwell we use a diamond cone, but in this case a square pyramid that is used as one indenter and we can show this thing it is having this shape, it is like a square pyramid and these are the slant surfaces and this pyramid has certain geometry that means between the slander faces we have one angle between the faces, between the faces it is 136 degree and between the edges it is 148 degree.

So these are the geometrical characteristics and at the same time if we consider this diagonal of this square base then this indentation that means h by d is going to be about 1 upon 7 that means if we make one impression on any surface and if we measure the diagonal d, in that case the penetration depth will be one seventh of that diagonal of this indentation so that is the ratio which is governed by this geometry. Anyway, this square base pyramid if it is is used for indenting than what is going to happen, this is the pyramid and in this case we expect an indentation almost like this leaving this impression and one can measure this diagonal this is d.

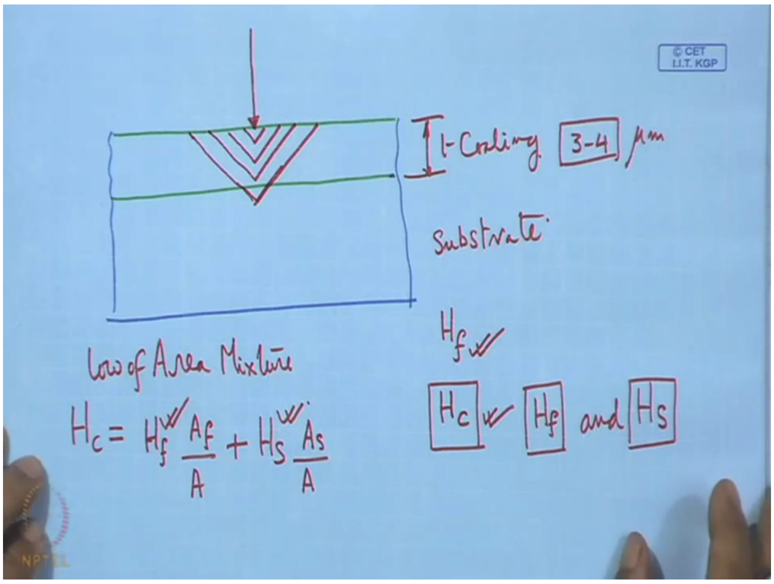
We have 2 diagonals so in all practical for all practical purpose we have to take the mean of these 2 diagonals and after that the hardness value can be written as 2 P Sin Alpha by 2, d square and where this Alpha is this 136 degree that means between these 2 faces and this can be rewritten 1.8544, it comes out to be 1.8544 into P by d square, so d is the diagonal length, P is the applied load which can be say 1 kilogram to it can go up to few kilogram. So it can start with say 10 grams, 5 grams, and it can go up to 1 kilogram of force, not to exceed this value but for most measurement it is within a kilo may be 50 grams or 10 grams that is the limit where all this surface hardness measurement has to be done.

In addition to this Vickers indenter we have also what we call Knoop hardness. So in this case instead of this square base pyramid what is taken, in this case it is going to be a rhomboidal so it is a rhombus and in this case in this rhombus we have 2 edges like these are the 2 edges or these are the 2 edges and this edge angle where is so the edge angle will be in one case it comes out to be around 171 degree and the lowest one is around 130 degree and with this that is the proportion and with that this h by L, and here we introduce another term what we call L that means length of the longer diagonal. Length of the longer diagonal that we have to consider and with that this ratio this h by here we have to write L that comes out to be as high as 1 upon 30.

So this is 1 upon 7 and this is 1 upon 30, now what is the advantage with this Knoop hardness what we can see that this penetration depth is less with the same load so with that it would be advantageous in measuring the hardness of the coating whose thickness is also specified. So this is one consideration one can make while measuring the surface hardness of the coating either with this Knoop hardness or Vickers indenter but here this will be the first choice, this is number 1. And number 2 since this penetration depth is less for a harder coating say it is a ceramic coating, so chances of crack formation or crack propagation because of this indentation force, it is actually it is causing some plastic deformation.

In fact, hardness means resistance to penetration or resistance to plastic deformation. Now if we allow too much of indentation depth then there is every chance risk that this ceramic coating or ultra hard coating can open up just by through a crack propagation, so for that also this Knoop hardness this technique is found to be advantages. However, one thing one has to look that this technique whether it is Rockwell, Vickers hardness tester or Knoop hardness tester that this depth of penetration that should not exceed the coating thickness so that is one of the issue one has to look.

(Refer Slide Time: 24:45)



So this is the coating which is shown by this line and just below this we have the substrate so this is coating substrate composite. Now here penetration means we have to restrict this thing, suppose we consider here 1 square based pyramid as an indenter so this is actually the coating thickness coating and this part this is substrate . Now in this case as the load is applied gradually what will happen, so it will keep on increasing the penetration depth and it depends upon the load, smoothness of the coating, it should be also flat that means this should be at right angle to the direction of loading otherwise, what we can show here from the previous picture this diagonal it will not be a square diagonal but it can have distortion.

So this is also another difficulty in measurement if the coating surface is not smooth or it is not properly aligned that there is a problem in detecting the diagonal and then the length of the diagonal that measurement becomes more complicated. Anyway, so what we see that given the coating thickness now say coating thickness of a hard coating say for all it is a reasonable coating 3 to 4 micron and if it is a well-developed coating with all sort of addition and then what we have densification, stoichiometry, then the crystallographic orientation if everything is perfectly done, we expect a high-value hardness or wear resistance even with this 3-4 micron coating.

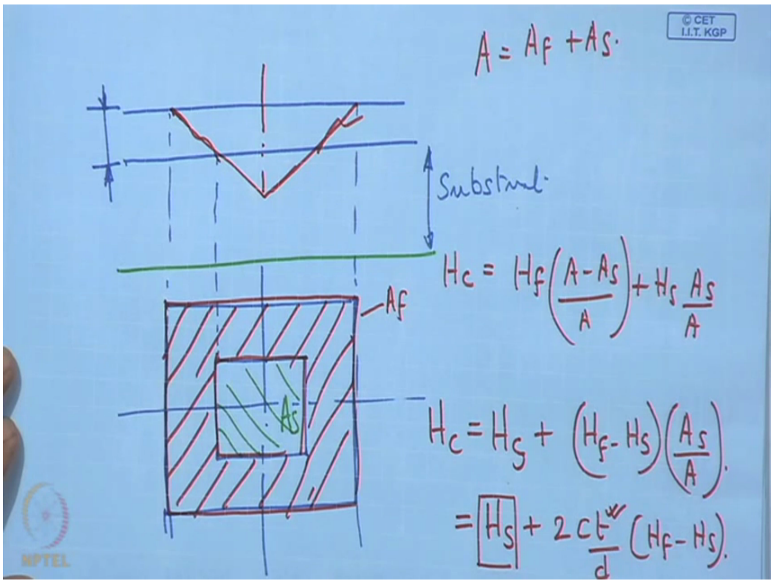
However, before application one would be interested to assess its hardness now with this existing Vickers or Knoop there is every chance that this coating depth that will be exhibited by the depth of penetration and in that case what exactly we measure, it is not the hardness of the film or the coating but we would say it is mostly it is the hardness taking the combination that means this coating and substrate because this part this is the influence of the substrate and this part it is the contribution of the coating.

So obviously it comes to the point that it depends upon the thickness of the coating in one hand and on the other it is actually the loader, how much load is applied that means it is also a limitation of the apparatus the coating and in the hardness tester how precisely we can control this loading arrangement that means the rate of loading, how much duration the load should be kept here and also with what precision the magnitude of the load can be controlled, so these are the few issues must be properly addressed and before really we go for measurement of coating. Now at the outset what we can say by this technique in most of the cases, hardly we can measure this film hardness but what we measure that is actually composite coating that means it is actually influence of H f and influence of H s.

H f means has it been just coating then its hardness and it is substrate alone so their influence with certain weightage that is actually playing the role so it is actually a certain percentage of H f and certain percentage of H s they are playing their role and finally what we measure superficially externally with this apparatus that is H c. Now here 2 attempts have been made to measure this hardness by this Vickers indenter or Vickers hardness tester just by modelling this hardness of the composite that means here it is written as H c = H f into A f by A + H s into A s divided by A.

So that is actually called Area mixture approach that means this macro scale measurement, microscale measurement and microscale assessment of coating hardness that what we are discussing so it is actually the area mixture approach that means it is actually called law of mixture, so it is actually low of area mixture so it is law of area mixture. And this is actually the law connecting this composite hardness with coating hardness and substrate hardness.

(Refer Slide Time: 31:53)



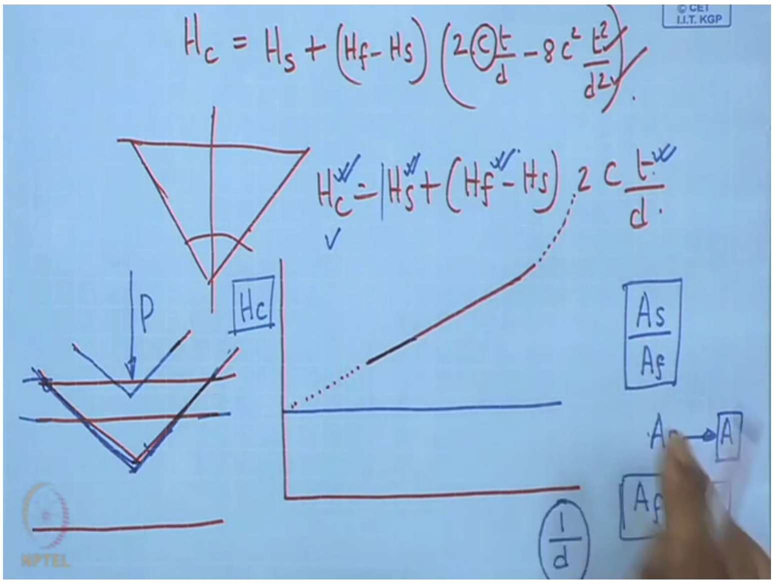
The whole idea here is that let us have a quick look in another illustration that this is the coating and just beneath it we have a substrate and here we have this indenter. So indenter has come to this depth, this is the depth of indentation and this one the impression, so the impression so impression by given by this indenter that we have shown by this redline and these 2 blue parallel lines that is the coating zone that is the coating and this part that is substrate. So what we can see here that this area under indentation that has two portion; one portion that is well within the coating and another portion that has gone deep into substrate.

Now this can be further illustrated by another simple diagram say we can have a quick look here so this is the total impression, this is the square pyramid impression and here we can also transfer this line to show these 2 segments clear segments. Now after having this impression under the microscope we can clearly distinguish between 2 zones, one zone that will be that means this zone this zone this which is illustrated by this hatch mark that is the slide area and this is actually this zone from this point to this point so this is just like a peripheral area, which is the area provided by the coating that means this area is actually A f and what is H s? H s is actually the load hardness of this substrate and this area this is actually A s so this is actually A s and this is actually A f.

Now from this we can further right that A = A f + A s, so we can have a substitution for this thing that that A f so what we can write here this A f = A – A s divided by A and that is equal to H s by A s A. So this is going to actually what we can write in this case we can also write in this way that H c is going to be H s + H f – H s into A s by A, so from this relation we can find out that this one H s so we can put this way we can have a substitute for H s and from that we can ride H c is equal to this one and this can be written as this is H s into 2 c t by d into H f – H s. So it is actually an equation which connects hardness of the substrate + this difference of hardness of the film – hardness of the substrate then it comes the thickness of the coating and also impression of this diagonal.

So here this diagonal means actually this point, so from this point to this point if we consider we can show it that this is going to be the length of the diagonal so this is going to be the length of the diagonal and this is we have put this d here and this is c is a constant and this constant depends upon this portion which is actually the portion which is bearing the load and this is well within the coating.

(Refer Slide Time: 37:43)



So from this relation what we can write in fact, actual relation comes in this form composite coating is equal to H s + H f – H s into 2 c t by d – c square into t square by d square. So it is actually elimination of those area that means writing this area which is actually A f and A s so these are eliminated, so here what we can see this elimination of A f, A s and A in terms of in clear terms of t and d, thickness of the coating, length of the diagonal, and this c that depends upon the this cone angle here that means the angle of this pyramid, so this pyramid angle, angle between the 2 faces, so this is actually simplified as H c = H s + H f – H s into c into t.

Now this can be represented in a form of a graph, so what we are actually measuring with this Vickers hardness tester that is not H f, we are actually measuring H c so just if it is a bear uncoated sample, for the uncoated sample we can show this value so this is H c and this is actually 1 upon d so 1 upon d because for a given thickness of the coating, this composite hardness is going to maintain a linear relation if we follow this equation. Now here what we can find that with increase of for a certain coating thickness we can get a value of this type, we can get a value of this type so this is the coating thickness and this can be extended on the side and this can go like this, now the shape of the curve why does it take such a shape?

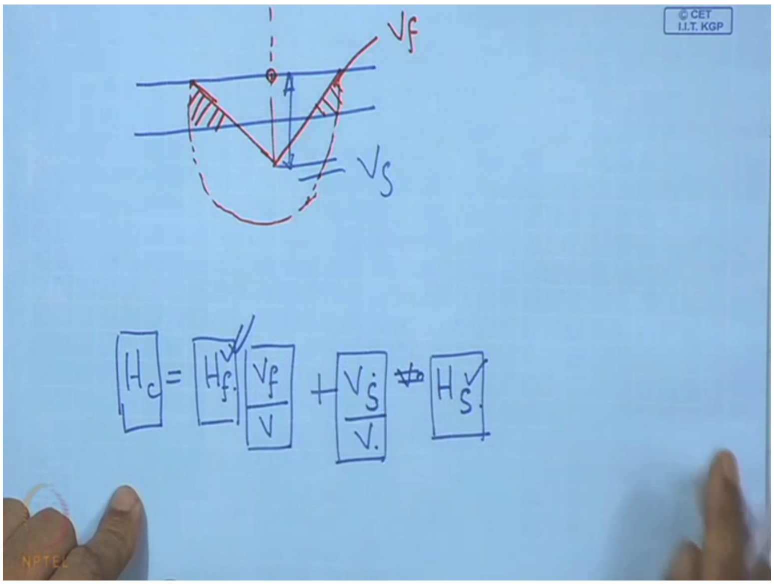
It can be explained this way, for a coating say here we have this coating and this is the substrate, now what happens we measure this diagonal, now the length of the diagonal it depends upon the load so 1 upon d that is 1 upon d large means d is rather small, and where 1 upon d is small that means d is large so we can have 2 extreme situations so this blue one is showing 1 indentation by this Vickers indenter on a coated area and here say for example this indentation is limited within the coating. So in that case what we find here that this d is quite high and in that case the composite hardness then the contribution of this h s that is going to be quite low and in that case the composite hardness that will show a hardness value which is almost approaching that of the film.

So here this value of H s will be less contributory and less insignificant compared to the value of the film that means if we have a very large value of 1 upon d that means d is small, so in that case the value of H c that will approach the value of H f. However, if we increase the load, if we have a very high value of the load and which is represented here by this depth of penetration which has gone well within the coating, well within the substrate and in that case what we can find then 1 upon d that will have a smaller magnitude that means d will be quite high and in that case this composite hardness though it is the composite hardness, composite hardness means it is actually the hardness of both coating and that of the substrate which are contributory but in this case hardness of the substrate has a greater contribution than that of the coating.

And this way we can find that the value is coming more towards the side, this can be physically also represented here that means this is the area which is the load bearing area for this load which is applied here and this is actually the load bearing area on the substrate. So with a thin coating with the supplied load naturally the load bearing area that will be higher in proportion that means this A s by A f so if A s is more compared to A f or if A s is approaching A that means that is the total area of indentation then the composite hardness will be showing, almost it is coming very close to the substrate. On the other side, if we have A f that is approaching A that means it is very close to the border but not it has not gone too much inside the substrate.

So in that case we can say the bearing area on the film that is almost approaching that actual area of indentation and in that case composite hardness will be on the higher side and it will approach that of the film. So this is one way of handling the problem and this is law of mixture and this is actually the area law of nature and this area law of mixture that can be utilised in this case for assessing the hardness. So we can measure H c and from this knowing the value of t and d, H s is known, we can determine the value of H f.

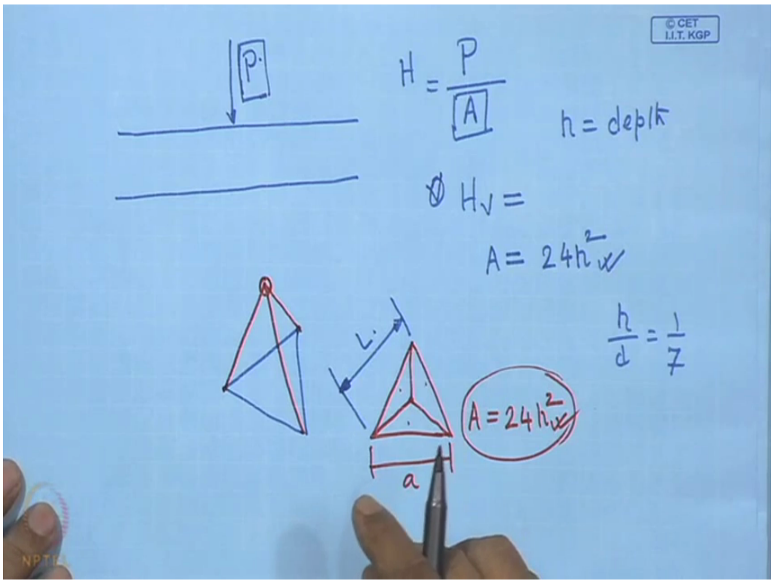
(Refer Slide Time: 46:19)



Then there is another way of working that is called volume law mixtures, volume law. So in this case we can write that this H c, H c = H f into V f by V + V s by V into H s, so this is the contribution of the film hardness and which is governed by this law of mixture for volume V f by V and this is actually contribution of substrate hardness and this is governed by this law of mixture substrate volume divided by total volume. Now this is just illustrated here, so if we consider this is the area of indentation then what we can consider further to this just by considering this as the centre and considering half this diagonal as this radius of the hemisphere we can consider one hemisphere like this and in here we can make apportionment.

So apportionment would be that this area which is shaded here so that will be actually V f and the rest of the area that is going to be V s, so with this V f and V s that is also another consideration forming another model for this determination of H c. So by this volume law of mixture it is also possible to determine the value of H f because we are going to measure H c from the actual measurement and depending upon this depth of impression that means this depth, geometrically we can find out what is the volume of that portion on the coating which is bearing that load and what is the volume of that portion inside the substrate which is also contributing in bearing that load and from there if we know H s then it is also possible to determine the value of H f.

(Refer Slide Time: 49:48)



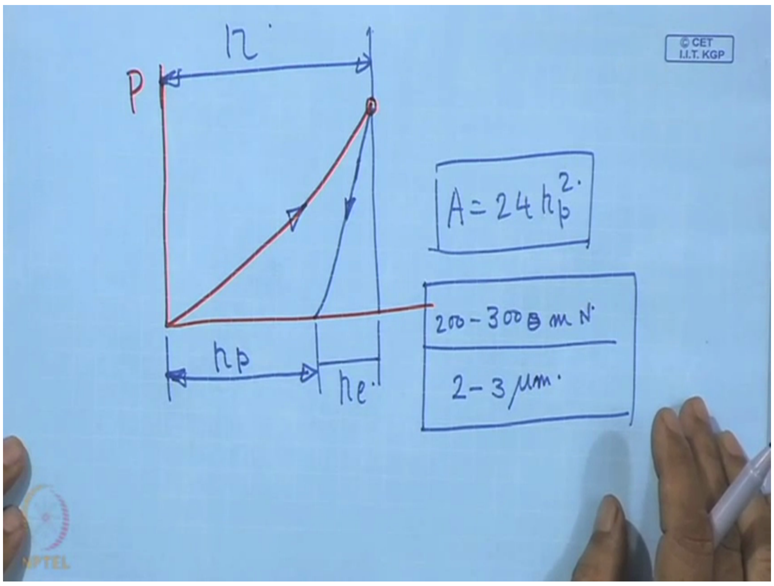
Now it is coming, we are coming to this nanoscale measurement, now in nanoscale measurement the whole idea is that not to go beyond this coating thickness to restrict the penetration within this thickness. So this is actually called the nanoscale measurement and this is the apparatus which controls the loading and also the depth of penetration so it is actually a close loop control so the force is monitored and the depth of penetration that is also correctly assessed. Now in all those cases except in Rockwell what we have measured, it is actually always the hardness value is load divided by area under indentation so that is the basic principle, load divided by area under impression and in this case.

And in this case what we measure, we measure actually the diameter of the indication or diameter of indentation or the diagonal length of the pyramid. However in this case we measure the depth of penetration depth of penetration so that is the depth of penetration so that is the penetration depth and when we have this Vickers hardness measurement, in that case what we have in that case the area under indentation that can be equated by this penetration depth by the relation, it comes very close to this 24 h square and in this case this diagonal length and h they are related by this h by d as 1 upon 7, so in this case measurement of penetration that is done but it is not optical measurement of the diagonal or that of the diameter of indentation.

And this is actually measure and in this case instead of the square pyramid what is taken it is actually a triangular pyramid, it is going to be an equilateral triangle so with this equilateral triangle one can put this way this is the equilateral triangle apex and it looks like this way it is the triangle and here we have the apex point, this way it is the triangle and here we have the apex point. So this triangle or this triangular base pyramid is made in such a manner that in this case also this is called dimension a and this is actually the dimension L.

So in this case also the pyramid is designed in such a manner that here also this ratio A = 24 h square that (())(53:06) that means in simple terms what we understand whether it is a square-base pyramid or if it is a triangular base pyramid with the same load it will give the same penetration and the same hardness number, so here it is actually the penetration depth that is measured. And triangular pyramid that is used because of the simple reason that in this case manufacturing is easier making manufacturing precision is better because these are the 3 non-coplanar planes they are anyway going to meet at a point. But when this is the 4 sides of a square base pyramid, this cannot meet it is not always it is not necessarily true that all these 4 sides will meet at a point and that becomes an error which may creep in, in the indenter itself and that can also affect the precision of measurement okay.

(Refer Slide Time: 54:34)



So with this, what we can see her that this loading in this case it is actually the loading, this way it is the loading point, this is the load it is elastically and then when it is the when the load is released, it comes like that and that is going to be the recovery. So this is the total deflection which is known as h and then this is going to be elastic and this is going to be h p. So this h p is actually measured so here what we write this 24 h p square and from this measurement this value of h p, it is possible to measure the hardness of the coating and in this case the load can be kept as low as 200 to 300 milli newton.

And the penetration depth that can be also restricted to 2 to 3 micron and with that it is possible to localise the entire penetration within the coating and in that case directly the hardness of the coating can be measured with a much better accuracy and that is why now for all sort of hard coating this has become this nano-scale level measurement that becomes one of the most useful and usable technique of hardness measurement.

(Refer Slide Time: 56:21)

So we can summarise today's discussion, hardness is often used as the index of wear resistance of mechanically functional coating. Traditional hardness tester cannot directly measure hardness of the coating because of the depth of indentation often exceeds the thickness of the coating. However, hardness of coating may be determined from measured composite hardness with proper modelling. A newly developed nanoscale indentation technique allows application of load in few hundred million range and depth of indentation in 1 to 2 micron range thus, facilitating direct assessment of coating hardness.