**Technology of Surface Coating**
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**Lecture 38**
**Assessment of Adhesion of Coating**

Assessment of Adhesion of Surface Coating, now why do we use coating that is already known to us it is only to improve the capability or functional ability of a component or a part and it can be various types of, I mean it is mechanically functional or electrically, chemically like that now this particular adhesion strength of the coating that becomes so critical in particular for all mechanically functional component or part or even a small product.

The reason is follows, that when we use this part as a wear part for example then we may expect high tangential stress and also coupled with a high temperature. So under this condition, both substrate and the coating as individual are quite capable, so that means the substrate of choice can support the coating it has bulk strength, bulk hardness, all the mechanical characteristics and at the same time the coating is rightly chosen to give all sort of performance in terms of wear resistance, chemical stability, low friction and like that.

But how to have the proper support, it is the substrate which gives that support and in between we have one discontinuity on interface and that interface is our focal point, it is the point of attention and there suddenly, all the properties just immediately properties change and there we find a discontinuity and that is why all effort should be made to have minimum effect, adverse effect of that discontinuity and we can get one of the best possible couple, that means coating substrate couple and it can work with a very high prolong service life and giving one of the best possible performance in actual application.

Now Significance of Strength of Adhesion, that we understand and now we come to this adhesion, what is mean by adhesion, it is actually a classical definition, it is known actually it is the state in which two surfaces are held together by some internal forces and which may consist of a valence force or interlocking force or even both.

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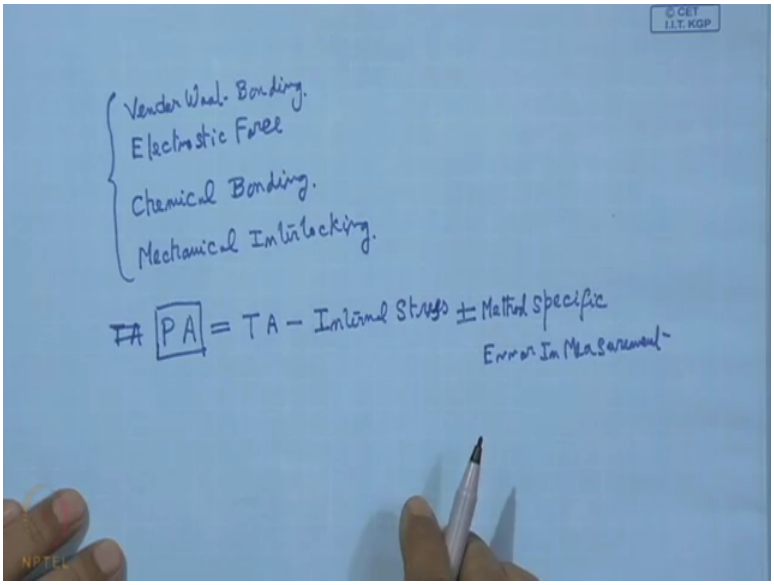


Now what we see that here actually we have various types of bonding, now one is Vendor Waal Bonding that is the physical adhesion, then we have electrostatic, electrostatic forces that also can bring them to together, then we have further to this Chemical attachment that means this chemical bonding.

So these are the various forces which can elevate this adhesion strength at this interface and in addition to that sometimes this is also very important and it presence is strongly felt, that means Mechanical Interlocking, so we can find at least four, there can be many more, but at least these four forces are present to enhance this bonding between this coating and the substrate.

Now here we have Theoretical Adhesion and Actual Adhesion, now what is the difference between this, so if write theoretical adhesion or say Practical Adhesion, PA stands for practical adhesion and this is actually theoretical adhesion minus, we have to subtract something which we call Internal Stress, that means we have to subtract a force and this comes in the picture because of the internal stress, it can be intrinsic stress because of the lattice mismatching, that is distortion or it can be thermal stress.

These are the two things should come in picture and in most of the cases that will be subtractive plus what we have, what we call Method Specific, method specific error in measurement. So what we see here and here we should put plus minus, so it is actually theoretical adhesion minus

the force which is arising out of this internal stresses and plus minus method of error, method specific error in measurement.

That means, this measurement technique that is actually in question and anybody can point to this fact about the validity of this measurement and here it can vary over a range and that can also affect the measured value so practical adhesion, because ultimately what we need, we need some sort of index value or index number or some kind of number it can be force, it can be stress, which can be a good guide for ranking the coating substrate combination, process parameters, it is actually the coating process plus the coating material and the substrate material and when it is coating process it can PVD, it can be CVD, it can be even spray process, plasma spray process or it can be flame spray process, it is also a galvanic deposition.

That means whatever may be the coating process, finally what we need, we need some sort of assessment, experimental assessment and what we call a numerical assessment here so that we can get a number and that number can be a good guidance for ranking and for also selection of this coating along with the substrate and also the process parameter variables.

Now Assessment of Coating Adhesion, so here actually we are going to assess the coating adhesion and we can call it a numerical assessment, that means there will be some index by looking at we can at least rank the coating substrate adhesion. This is actually a bending test and this bending test is meant for assessing the coating substrate adhesion.

Now actually what happens one of the reason we find at this interface which causes this discontinuity, it is actually the modulus of elasticity of the two material and also co-efficient of thermal expansion, apart from other reasons. So these are the mechanical and thermal characteristics, physical characteristics of the material which immediately can influence this adhesion.

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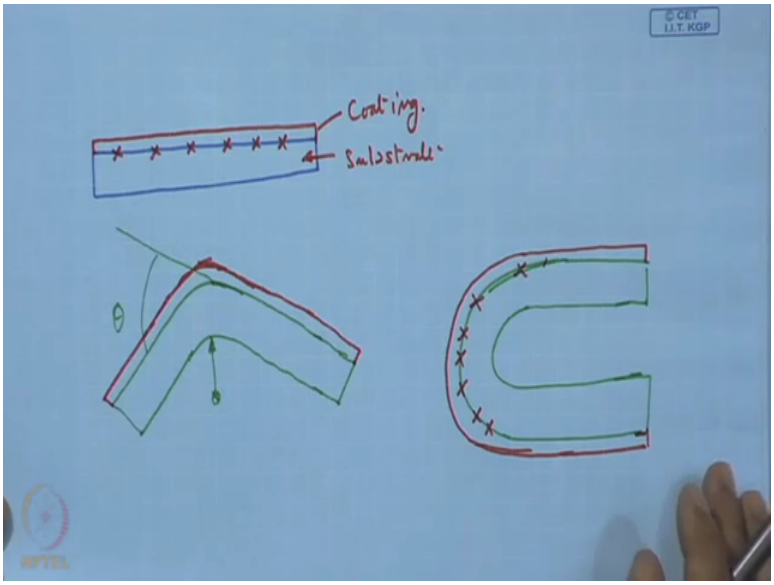


Now here let us look like this we have one substrate, flat substrate and over that we can put a coating. Now this is the interface, now how good, how strong this interface is, how to know this thing, this can be done simply by bending the whole, this piece. It is something like this, let us have a look here that we can have a bending, this is actually the bending and definitely there is some radius we have to also consider this and accordingly what happens in this case if we extend this line then we get this as the theta that means originally the theta was 0 and now we have put a certain value of theta through the this bending.

Now over this we have to also show the coating, now this coating theoretically or it is our desire that it also follows the deformation or the bending of this substrate so this is actually the substrate and this is the coating, so coating and substrate and here it has undergone a bending. Now this theta, it starts from 0 and it can go upto say extreme case extremity that is 180 degree. Say we can also consider a situation where it has undergone a bending of say 180 degree, this piece.

Now in this case, what is going to happen to this coating, now here we can also draw this line to show the top layer, however there are certain issues whether we can illustrate the very existence of coating like this. Now it depends upon how strong this interface is. Here what happens, this material and this material, their ductility may be different and more importantly their E value is also different, now when we have same level of strain here.

Naturally what we have strain is same but the stress level will be different and at this point, this interface will be heavily under tension and because of this tension it will have its own impact, there will be tension here and it may so happen that coating ruptures at this point.

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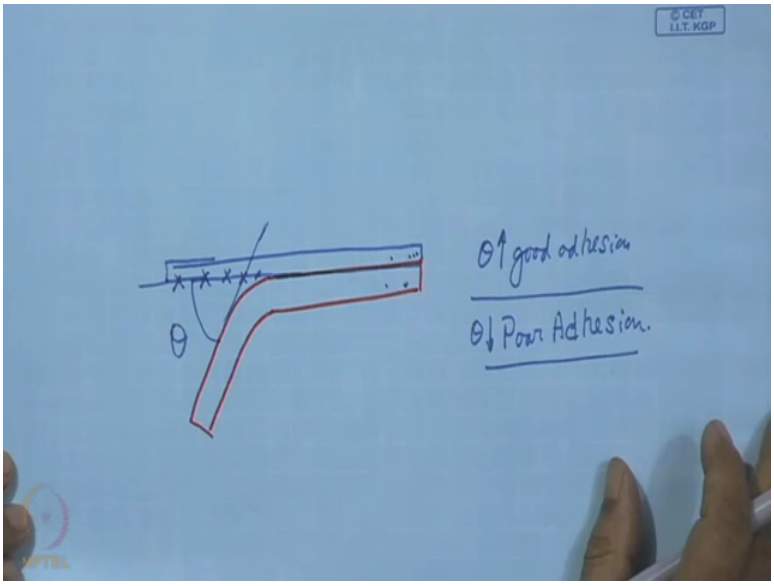


That means we can draw another figure to show that separation of the coating quickly we can show this, say this is the substrate and here it has undergone little bit of bending and however the coating does not remain with this one and it is something sort of thing which looks like this. That means from this part the coating gets separated, so this area the coating gets separated from this substrate surface.

So this is a case where we do not have that best value of adhesion and it is somewhere in between, now how to quantify this thing it is not difficult in that say if we measure this angle and what I have shown already, value of theta. So this value of theta can be considered as index of adhesion and with this value of theta we can fairly compare the adhesion of a particular coating with the substrate and that comparison will be fair comparison if we have the same substrate, same size and also the coating of similar thickness and only the deposition parameter we can change.

That means to make this test, which is fairly acceptable what we have to do, the substrate is of the same material and also the coating of the same material, thickness is also of the same order

but we can change the parameters for deposition of this coating then we can have a fairly available data to make some observation on the adhesion.

So naturally higher the value of theta that means good adhesion and lower the value of theta that means poor adhesion. So when we get for some reason, if we get a value very close to 180 degree in that case we can say safely the coating is following the deformation pattern of the substrate and apart from the material characteristics of the coating it is also most importantly it is actually the adhesion between this substrate and the coating that means it is the interface and that interface decides what will be the value of theta in addition to the basic material characteristics.

We can refer to ductility or brittleness, but with the with one set of condition for the substrate and the coating, just if we keep everything constant and only parametric variables that we keep on changing then the value of theta will be a good index of good adhesion that means this is good adhesion and low value means poor adhesion.

So this is one way of assessing the coating and here this value of theta can be fairly, it can be accepted as an index of adhesion. But one thing we should understand that this is not any way it is showing any value of force or it is giving any impression of force, force of adhesion or strength of adhesion, what is a probable value that it does not give, it can only say that it is a better coating in terms of adhesion and there this scale of this theta can be a good index as for guiding.

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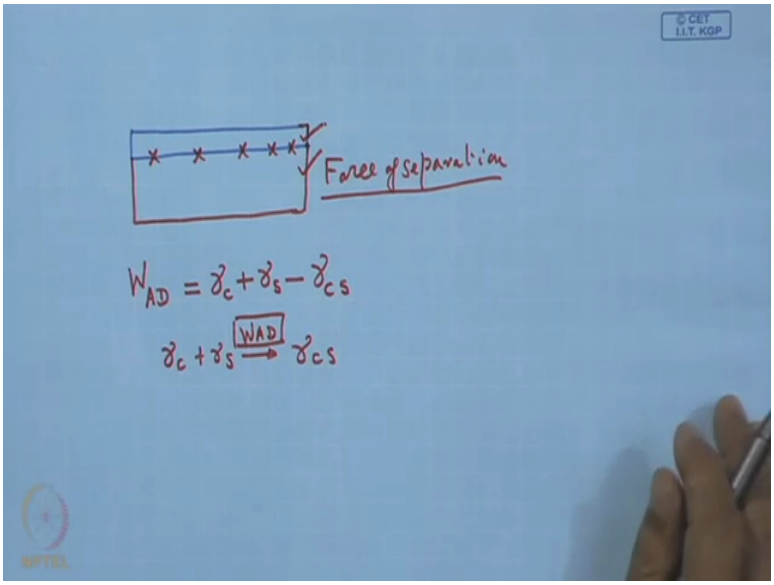


Now comes, Experimental Assessment of Adhesion, now here we have some interest in determining the value of this adhesion, either in terms of force or in terms of energy, that means if we consider this as the coating and the substrate here we have certain length and we can have the width normal to this paper, then definitely this adhesion that perhaps one can measure that this is a surface, length into the back engagement or the width.

So how much force per unit area that is required to remove this coating from this substrate, so that means that is the force of separation. So that is one index, very good index of adhesion, force of separation, this is one way one can assess and at the same time another thing can be in terms of energy. Energy means how much energy has to be spent for removing unit area of this interface that means originally we have the coating surface and we have the substrate surface.

Now they come together and to make one interface, that means two surfaces actually come together to make one interface and in this case we can also write down like this, that means this is the work of adhesion and this work of adhesion we can write like, this is gamma C that means surface free energy of the coating then this is gamma S that is surface free energy of the substrate and then we write gamma CS that is the interfacial energy, that is actually resistance offered to formation of this interface.

Naturally we have the balance of those that means here what we write that gamma C plus gamma S that is going to be gamma CS. So naturally here we have this energy available which should be

released and that comes like this gamma work of adhesion. So this we can fairly calculate and this can be also treated as one index of adhesion, so either in terms of force or in terms of energy which is required to separate to make this interface into two surfaces that is actually what we mean as the force of separation or energy for separation and that we can also treat as the index of adhesion.

So this force of separation, this can be used as one index so this is going to be a value, an indicative value and this value can be compared from one pair of coating substrate to another pair but there are certain assumptions and consideration and we have to follow those to make this test results meaningful and which can be used as a guidance for ranking and selection of the coating.

So Indentation Test means it is simply a hardness tester so here a Rockwell hardness tester is used but in this case it is not used for measurement of the hardness, it is just to leave one impression of indentation and this impression will change its geometry or pattern depending upon the adhesion between the coating and the substrate and sometimes it will be just a cavity crater that will form or there will be a cavity and around that there will be some cracked area or crack zone. Now depending upon this cracked area, there has been an attempt to assess this particular coating substrate interface.

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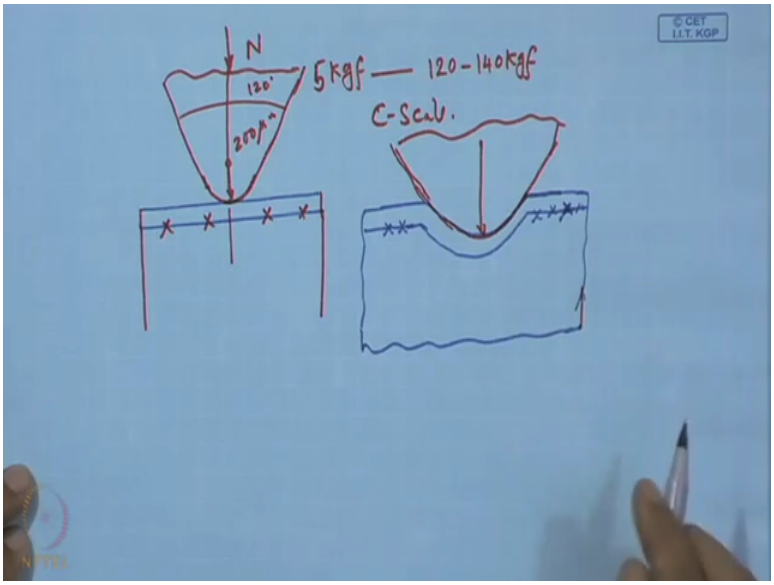

Now let us look how does it work, now say this is actually the coating and here we have the substrate, now on that this diamond brale of the Rockwell C-scale, so it is actually Rockwell c-scale that indenter and this is actually this diamond brale that means that it is a cone of diamond having a tip in the form of a hemisphere and this angle appears to be 120 degree.

So that is a well known indenter used routinely for this hardness measurement, so it has a tip radius of 200 micron and this angle is 120 degree. Now this will be pressed against this coated surface with a normal load and this normal load normally we can keep say from it can be 5 kilogram, force 5 kg or it can go as 120 or 140 kg also for a well adherent coating. It depends upon the coating substrate interface how strong the interface is and what is the hardness of the substrate, hardness of the coating.

But important thing here is it that because of this action, this will change its pattern that means there will be some deformation so we can also show this deformation this way, this is the deformation and, so this we can show as the deformation and here we can show this tip, so this is going to be the tip which is having 120 degree, which is now, so this is actually the tip.

Now comes very important thing whether the coating will follow the deformation of the substrate, definitely the coating is harder than that of the substrate so substrate deformation pattern that one can see here from this curvature, it is like a dent, it is a cavity formation, however if this adhesion is good, very good adhesion, very strong adhesion, we should not have any problem and then this coating can follow this deformation and only we are left with this cavity which has certain depth and diameter, which are inter-related.

However if it is not the case in that, in that situation because of the poor adhesion what can happen, a crack may propagate that means in this case what is going to happen the coating is following the deformation of the substrate and in this case because of the very high level of adhesion it can follow that deformation and there is no separation at this interface. So that is the weakest link.

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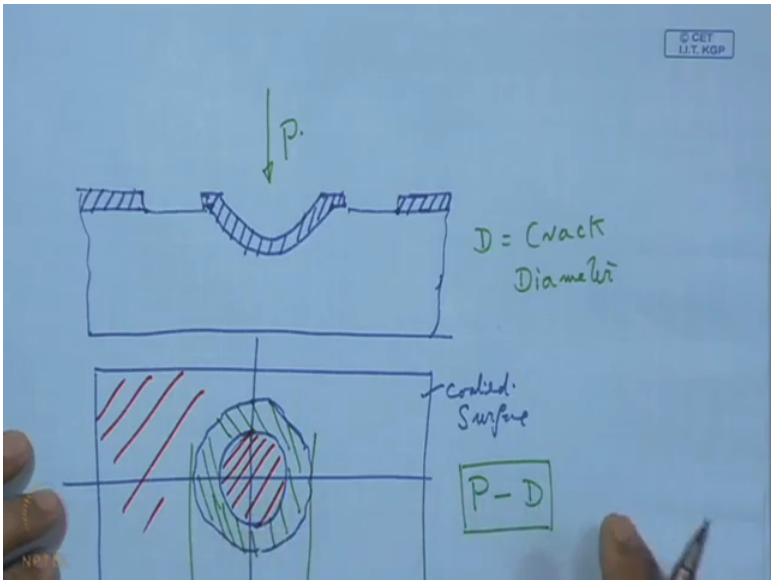


And in this case what is going to happen in case of poor adhesion, what we can find a portion which will be deformed so that means this is going to be a portion which is deformed like this. However, this will be the coating and here one can find out that means a crack formation that means there will be at this point the substrate will be exposed and low coating will remain so this will break and this will be separated from the substrate.

So this is going to be a situation if we have poor adhesion that means referring to this previous figure, what we can see because of the good adhesion very high level of adhesion, there was no crack propagation but in this case this deformation, that means there is a stored energy which is released through this crack propagation and this happens only because of poor adhesion and at this interface.

So what is going to happen this is coating, this is the crater, inside the crater we have also coating, this side also we have coating, now this is the gap, so this can be illustrated by another diagram which we can show here, that means this is actually the cavity, inside we have the coating, but outside what happens, the whole surface, the top surface that is the coated surface, this is the coated surface. So this is actually the front sectional view and that is the top view.

So what we are going to expect here, it can be a surface something like this. So what we see we have coating in this area, in that cavity, we have coating, this here too we have coating but only this is the area where we have lost the coating so these are the area zone where we have lost the

coating. So this diameter that is actually one index that means this D, what we call average crack diameter, so there are two issues one is that with what load, what load has been applied and the load versus this diameter, that means P versus D, we can have one variation, this is number one.

And also what is number two, that means the minimum value of load P which is applied from through this intender, this diamond brale, that means this is the minimum value where just the first sign of this crack propagation is seen, that means below that there is no crack formation and it can be like this what we can see here that in this situation if we leave it, it will be just like a deformation without any damage so in that case we have a cavity without such area zone from where coating has disappeared. So that will not happen, had it been a very good adherence.

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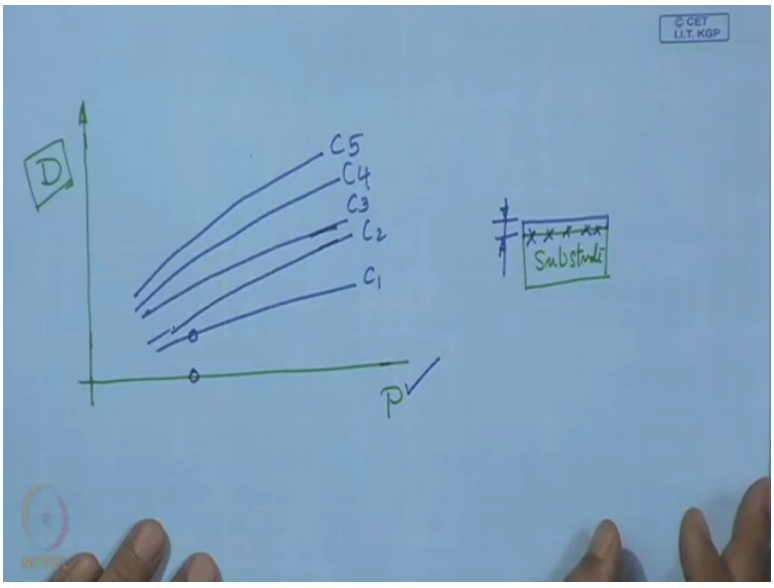


So from this what we can say that there are two things, say if we can have a plot like this. That means this is actually a load which is the indentation load and this is actually the diameter of the, average crack diameter, so from this if we like to compare two coating or a series of coating which are deposited under various conditions and keeping the substrate material, substrate we do not change the substrate because its hardness and modulus of elasticity that affects this value of this cracked area, cracked zone and at the same time coating thickness that is also there.

So if we like to compare various process conditions (())(33:09) like to compare the conditions of coating and which condition gives the best value of D, that means or the best value of P whatever may be the case, in that we must keep a constant uniform coating thickness and also the same

substrate, but if we like to consider the effect of coating thickness on adhesion in that case we do not change the process variables, we can only have a thin coating, a thick coating with one intermediate value, another one reasonably thick coating and that is just a question of deposition time.

So keeping all those parameters constant and we can compare also the adhesion of the coating substrate interface with this thickness. So which thickness we have more or less reasonably acceptable coating because the simple reason it has been already said that coating thickness is definitely one of the thing of interest because the wear resistance and what will be the service life that will depend upon the thickness of the coating.

If it is, if it only undergoes simply wear, however this is not always the case in certain situation, we can also have unfortunately a premature delamination of the coating and in that case naturally coating thickness has to be restricted because of the thickness also causes lot of discontinuity and variation in all sort of interfacial property which ultimately lead to poor adhesion.

So just comparison is possible in this way, so here we can find out graph like this so these are the graphs, so what these do indicate, it simply indicates if we have conditions, various condition say C1, C2, C3, C4 and C5. So all process variable skipping this side constant, so in that case obviously we can see a curve which is followed by condition C1 that is always the best because for a particular value of P, we get a small diameter, crack diameter and that is an index of good coating.

Now here we can refer to some of the physical process or chemical process like CVD, now when it is a CVD process, we are interested in TIC or TIN or Aluminium oxide coating. Now in those cases obviously with a given thickness of the coating, if we keep the same thickness of the coating, it is mostly the temperature of the position that matters, that influences the coating surface interfacial adhesion because of the residual stress.

Number two also we find that if we have the gas phase nucleation rather than the nucleation at the surface of the substrate that also can lead to this poor adhesion then also comes another very important issue that means that substrate attack by the reactant or the product. So from this, from looking at this diagram, one can find out the answer that how this CVD coating is affecting the adhesion and accordingly corrective measures have be taken in to consideration.

Say for diamond coating, we also know how to use this graph, so for diamond coating, the percentage of cobalt which is present on the top surface of the substrate that is one deciding parameter altering the adhesion or influencing the adhesion of the coating, so in that case also we can expect various position of these graph co-relating this indentation load on one side and the crack diameter on the other side and there obviously this one, this corresponds maybe a percentage of residual cobalt and that should be one of the best percentage of cobalt one should consider before going for the actual coating.

And when we consider the C5, C5 maybe another substrate treatment to receive this diamond coating on the substrate and in this case perhaps cobalt percentage is quite high which does not allow to have good adhesion and it leads to very large crack diameter with the same magnitude of load. Similarly say for a PVD coating, what we can also see, say Titanium Nitrite PVD coating, in this case also we can utilize such graph and here if we have four or five such graph showing the variation of P verses D.

There also we can see that cathode current and substrate ion current, these are the two thing which can effects the adhesion strong interface at the substrate surface and there we can also have similar graph showing a particular condition under which we get one of the graph which is holding the lowest position is almost flat and where as another where the rate of increase of this diameter is quite high and it simply applies that, indicate that in this case the proper interfacing has not been done and proper care has to be taken to bridge this interface with all sort of favorable conditions.

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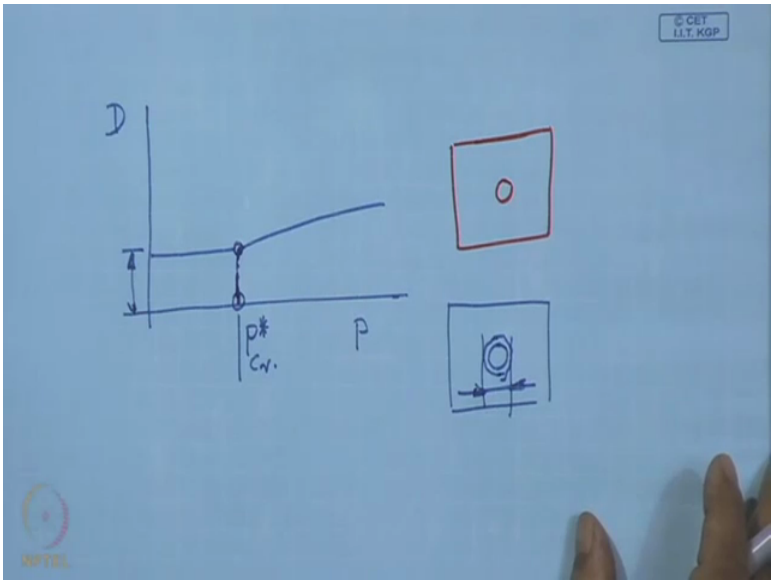


So this is about indentation test, now this Principles of Indentation Test that we have discussed and how we can use this PD characteristics and here one point we can also see, one point we can also mention that PD. So definitely as we get this curve, there may a point from where we get a crack diameter, so actually what happens below this we do not have any crack diameter so it is only indentation and from this point, so that means here the load is increasing.

Though the load is increasing what we can quickly see that though load is increasing we have just a cavity formation and there is no crack and ultimately what happens, as this load goes to this value, this value which is P critical, and on that we can see a sign of crack so this an indentation and over that we can see some crack formation and this diameter maybe this one and that is actually a load which we can call a critical load which causes the, first we can see the appearance of this crack area and that corresponds to this value of P critical.

Now Scratch Test, so this is also another way of working and in this what we see that by this scratch also we can assess the adhesion now this scratch test that has been known in (())(42:29) and this was one of the oldest technique just by fingernail or with a sharp point, metal point or a knife, one can assess the adhesion of a paint on a substrate and this has been used since long.

However this can be used with must refinement and this scratching technique, now it is well established, a well established technique for assessing the adhesion and the data one can gather

or procure from this can be meaningfully used for doing lot of investigation on this coating substrate interface and how to built one of the strongest interface.

Now this scratching technique can be in the macro level, micro level and nano level. Only the difference is in the tip radius of the indenter and also the magnitude of the load. So these are the two things which makes this difference in macro level, micro level or nano level.

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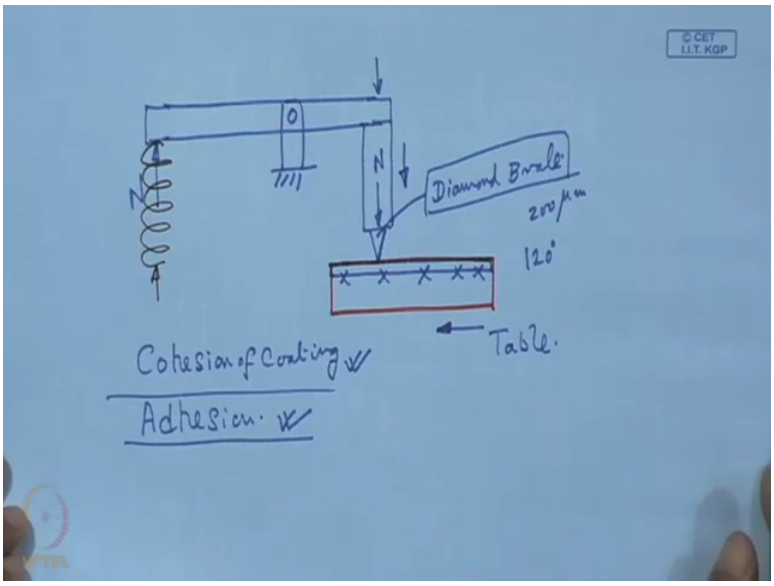


So what exactly we see here principle of scratch testing. Now in this principle of scratch testing what we see actually there is one arm, this is the apparatus, there is one arm which is pivoted at this point and then what we have here, we have one stylus and this is going to be an indenter, this is also a standardize that is also this Rockwell C-Scale, that means this diamond brale.

So that is the indenter which is used for measuring hardness in Rockwell C-Scale, that is with a tip radius of 200 micron and the cone angle of 120 degree, so that is the geometrical features, now on that what we can see here we have to place the substrate on this point and on this side what is done, we have to apply the load that is the normal load, so the normal load will be transmitted on this side.

So with this normal load it will make a deeper scratch, it will try to go inside the coating so here we have a coating, so this is coating and this is the substrate, so it will go deep into this coating, now here we can expect two type of problem, one is what we called Cohesion of Coating,

another is Adhesion. Now when we have a coating which undergoes in actual field, when it in actual field when it undergoes normal wear which is quit long this time taking process, this wearing, it is a time taking process and in that case it is actually one of the best coating, that means in that case we have good cohesion and good adhesion.

That means the coating does not have grain pull-out and which is grain pull-out premature grain pull-out from the coating that is one indication of poor cohesion, now if this coating as a whole get detached from this interface, that is actually poor adhesion both of this can be observed and both of this can be assessed depending upon the quality of coating, how this interface is built, how this synthesis has been done and this synthesis of the coating after building on this interface that means on the substrate surface.

So that is the top portion of the coating, it is mostly cohesion and the interface that is going to be the adhesion. Now in the process what can be done, it can be a constant load instead of this lever we can also have a constant load but in this machine particularly with the help of a spring, with the help of a spring, a load can be given.

That means the spring will be compressed and this table will move in this direction. So that will be the table movement or the substrate movement. That coated sample will move in this direction and this stylus with this indenter that will also have a deeper penetration.

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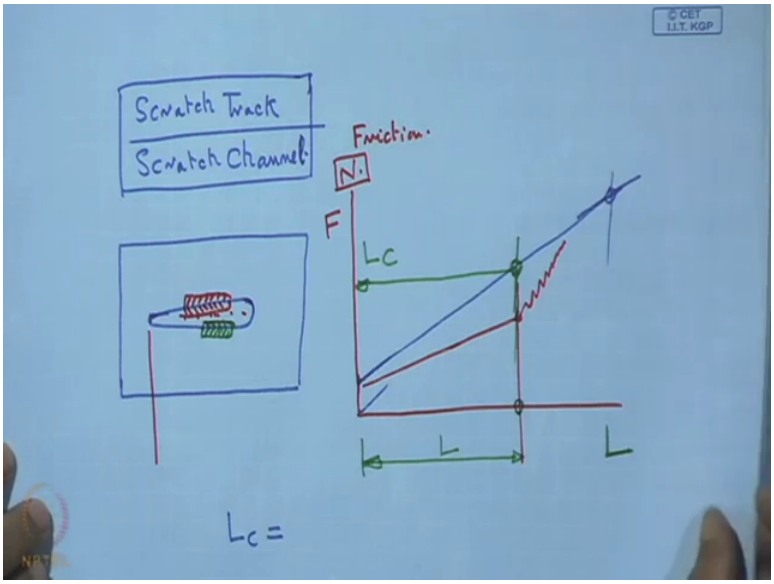

So in this process what is going to happen, we can have a scratch channel, scratch track or a scratch channel. So this scratch track and scratch channel, this observation in this zone is very important, so what we see in this case, say this is the top surface of the coated specimen and as it starts its movement travel looking at this picture, it start from this point and it is moving, so here the loading rate, loading rate means it can be a speed, speed is defined that table speed that is defined it is so loading rate is also defined that means increase of load per millimeter that can be also defined, it can be also defined per minute.

Whatever maybe the case, since it is going deeper with time, what we expect to see a scratch channel something like this and this is actually the impression of this cone now this is going to be a surface and from the whole surface the coating may be removed from the substrate. So that means from the surface, this coating may be removed from this surface, from this scratch channel coating may be removed.

Now what is going to happen in this case here we have actually measurement of the force of friction. Since this is moving in this direction, we have also force of friction which is also opposing this movement. So force of friction and normal force, they can be monitored and depending upon the state at this scratch channel, that means whether the coating has undergone just a cohesive failure or the coating has undergone a adhesive failure.

There will be a change in coating condition, that means the friction in this scratch channel that is going to change because of this contact of the indenter with the substrate material and this happens if we have poor adhesion and that may take place depending upon a particular load. So what we can illustrate here, that means say this is the starting point and here we have normal load, we can also present the frictional force or say tangential force, so normal load will keep on going from this zero level or it can be a step input also.

We can also give a step input like this and along with this, the force of friction will also keep on increasing and depending upon where the coating fells, there will be a change in co-efficient of friction and it can go like this, so actually this is the point where this coating can fell in the scratch channel or also on the side that means here also we can have failure that means this is also the coating which is removed from this area, we can also have removal of the coating from this surface.

So we have also removal of the coating from this surface, now where we see a change in co-efficient of friction, what we see actually that point we have to record we have to mark and that point also we can see what is the corresponding normal load and since this normal load it is given varying with this length of travel, so from this length of travel, we can find out what is going to be the normal load and that normal load we call as the critical load.

So from this critical load, we call that this is actually the parameter or index of adhesion of this particular coating instead of we can measure friction but if we know this length which can be found out from the graph and exactly at this length we can see also damage on the coating because this damage actually that caused the change in friction and this change in friction is indicated by this transducer.

So this way we can find out the value of LC and this LC actually is known as the critical load where the first damage in the coating we can determine. So obviously if one has to improve the adhesion, then definitely one can look into a higher value of this normal load where only this crack will appear. So naturally one has to take all the necessary steps and condition only to increase this adhesion so that this frictional change characteristic which happens because of the damage that take place at a much higher load.

Now this thing can be also done by Acoustic Emission, so in this diagram what we see, here we have one acoustic emission detector and with this acoustic emission detector we can also record the acoustic emission, that noise because of this deformation and cracking by that also it can be determine.

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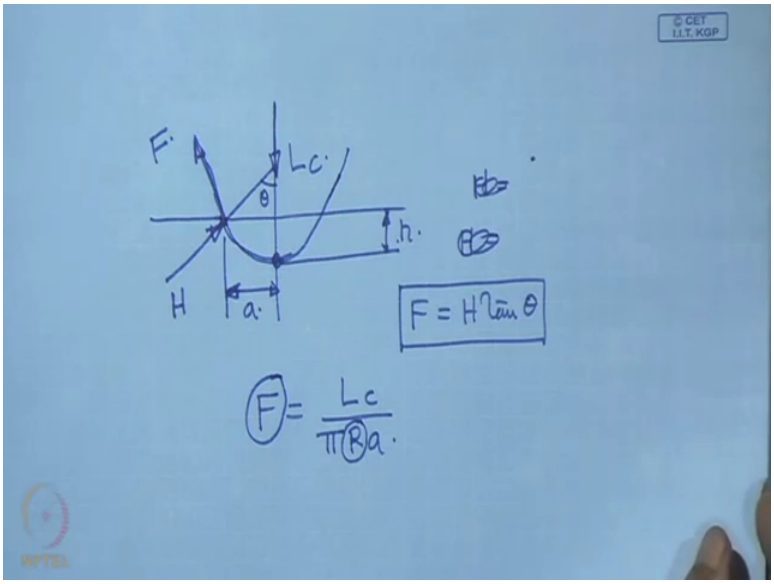


Now this value of LC that can be, that is a parameter but if we like to have the adhesion of the coating then we can show this thing quickly here that adhesion strength, say this is actually this tip radius and here we have this load and this is the thickness that is the thickness of the coating, the tip has already reached the substrate and here we have the LC, that is the critical load LC and here we have what is the tangential that is actually the hardness, so this hardness is given by this LC that is theta so this H is actually given by, this is actually the adhesive strength and this is actually given by this relation F is equal to H tan theta, so from this what we can find out that this H is equal to, from this value of LC.

So this F is equal to H tan theta and from this what we can write further to this, this F is actually given by LC into pi into R into A. So what is R, R is tip radius of this indenter and A is the contact radius. So A is the contact radius and R is the tip radius, so from this we can determine this value of F and which is the value of F that is actually called the adhesive strength and that can be compared depending upon different coating which are deposited at different condition.

So with that we can summarize that high strength of adhesion at coating-substrate interface is of primary importance for prolong service life of the coated component. This is particularly true for mechanical parts subjected to high tangential stress associated with high temperature. Indentation or scratch technique can conveniently used for evaluation of adhesion at coating-substrate

interface and influence of the variables of a coating process can well judged from numerical assessment of adhesion.