

Technology of Surface Coating
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Lecture-10
Chemical Vapor Deposition of Diamond

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Chemical vapor deposition of diamond coating. Now let us look into this significance of diamond as engineering material. If we look into these properties of diamond, the most important one and the most attractive is the thermal conductivity. And because of this, this diamond is used as a heat sink in many of the electronic application and also in the area of mechanical manufacturing.

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Now let us have a look in application of diamond in mechanical manufacturing. This is actually grinding and machining of mostly non-ferrous materials and also ceramic, plastic, composite. This is the domain where diamond has an edge over any other tool material or abrasive material.

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Now when we talk about diamond, diamond is actually available in three form. One we see natural diamond crystal, this is actually a mined diamond and non-gem quality is screened and classified as the natural diamond crystal for use as cutting tool. These tools are mostly used in all

precision machining of aluminum, copper and it is almost a very delicate cut with low feed, low depth cut and very high speed.

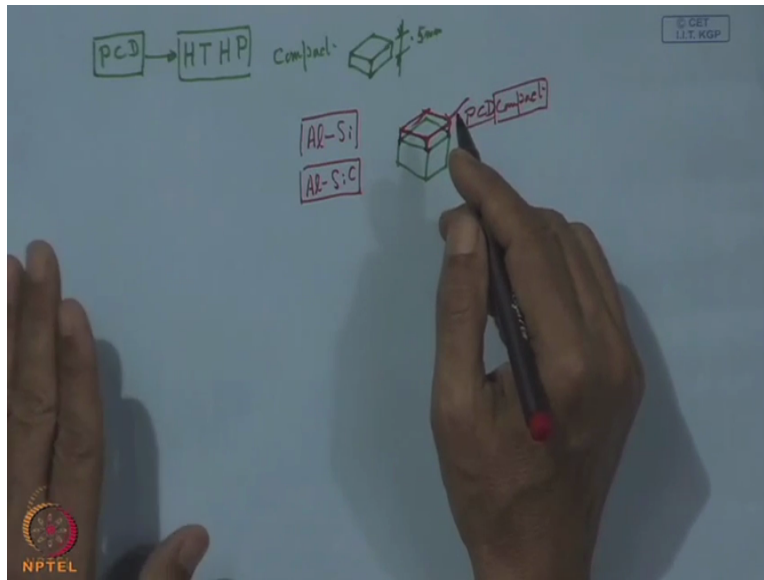
The one limitation of this natural diamond is that it has well-defined cleavage in unfortunate eventuality. If the cutting force, direction of the cutting force is aligned with any of the cleavage, in that event this diamond simply just breaks and it becomes totally unsuitable for machining. So though diamond is known for its ultra-hardness, remarkable machining performance but it has to be limited in its use only in the sophisticated areas of machining.

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Now as a substitute of this natural diamond, later on polycrystalline diamond compact that came into being.

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And this polycrystalline diamond compact, this is the polycrystalline diamond compact. This is actually the result of high temperature, high pressure technology. That means tiny particles of diamond are subjected to high pressure and high temperature to make a compact and this compact can be in the form of a round bar or it can be, can have a square shape. And this square shape maybe the thickness is around 0.5 millimeter thick.

And that can be braised on a carbide base, on this this is braised. So this actually, this is actually the tool. So this is called polycrystalline diamond, this is PCD compact. Now the greatest advantage of this PCD over natural diamond is that it has multidirectional crystallographic orientation. So average roughness, average toughness of this compact is greater than that of natural crystal.

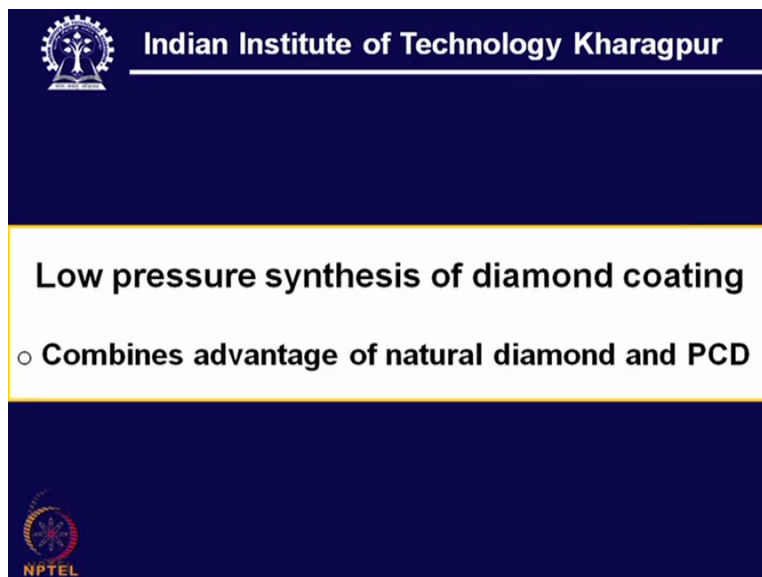
And this can be used for various heavy cut machining where we expect high cutting force and the machining task is little more difficult. So this can be used for bulk machining of material like aluminum composite, most importantly aluminum-silicon alloy or aluminum-silicon carbide alloy. And there are various others metal, ceramic composite or even some of the ceramic can also be attempted for machining using this one.

However this is also not free of weakness. The main weakness here is that often cobalt is used for binding this diamond dust or the powder. And after this compaction is made when this tool is ready for use, if this tool is elevated according to a cutting condition to a very high temperature,

then there is a risk of conversion of this diamond to graphite in presence of cobalt. So this is one of the weakness of this polycrystalline diamond compact. That means high temperature stability has certain limitation and another disadvantage of this tool, one can immediately find out its limitation in terms of tool geometry.

Now this tool of this given shape can be well used in turning, facing or in some general purpose milling cutters. But when it comes to the question of drill of different size or very small size or when mill cutters used for pocket milling having some complex geometry, then this particular compact cannot be easily given that shape to suit that geometry of the small rotary tool. So we find here that PCD has certain strength, certain limitations and natural diamond too has certain capability and certain limitation. So from this one would think of getting a new diamond, new generation of diamond combining the advantage of natural crystal and that of PCD and eliminating their shortcomings or weakness.

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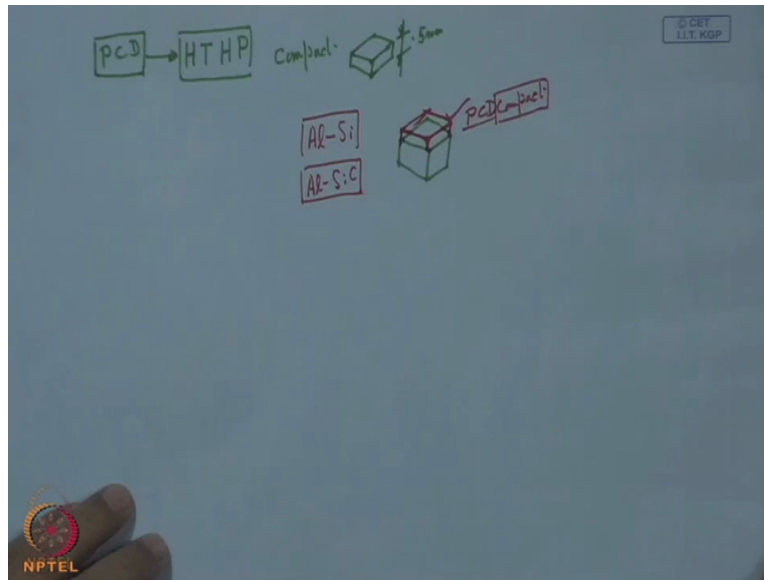
Low pressure synthesis of diamond coating

- **Combines advantage of natural diamond and PCD**

NPTEL


And as a result of that we find here diamond coating following low pressure synthesis.

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Now when we talk about this polycrystalline diamond, it can be also synthetic diamond, tiny crystals of synthetic diamond and that is converted into a compact. So this is a high pressure, high temperature technology.

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Low pressure synthesis of diamond coating

- **Combines advantage of natural diamond and PCD**



But as we see here from the very name, it is actually the low pressure synthesis of diamond. So it combines the advantage of natural diamond because in this case there is no need to have cobalt inside that coating. This coating can be given in the order of thickness of few micron or few tens of micron and more importantly it can be deposited on any substrate what we mean the substrate

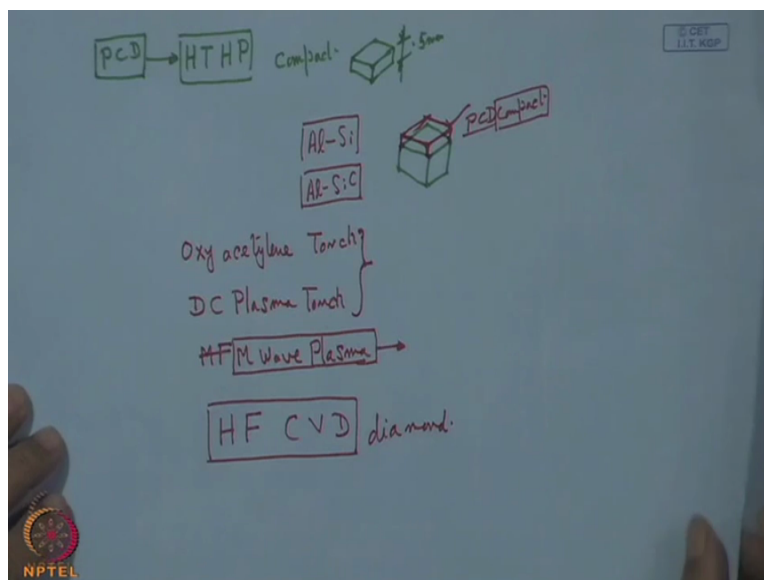
having some complex geometry. So that is its greatest advantage, that its flexibility to match the requirement of a very complicated geometry of the cutting tool base.

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Now here to make it happen, that means low pressure synthesis of diamond coating, we have various ways of doing the thing.

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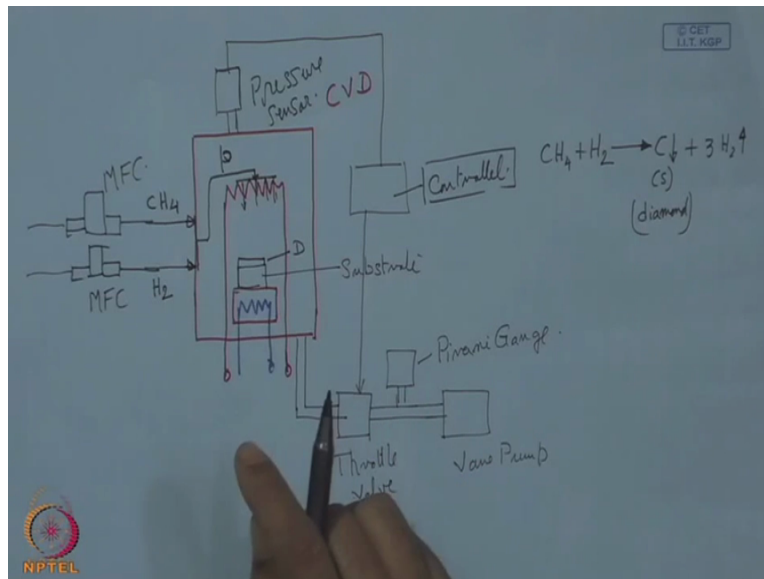


Say number one, we have oxyacetylene torch. This oxyacetylene torch or oxy-fuel torch can be used for extraction of diamond from this fuel-oxygen mixture. So this is one way of doing the

need. Then we have DC Plasma torch, that is also using this oxy-fuel and in a plasma environment. We have also microwave plasma. Now the importance of this microwave plasma is in electronics, it is strongly felt because of the capability of this process to cover a relatively large area with uniformity. That is one of its advantage and then also the quality of the coating, that means the purity of the coating which is an important consideration in electronics application.

Whereas oxyacetylene torch or DC Plasma torch, these are extremely suitable where a very high deposition rate is demanded. However it cannot provide the same homogeneity in the coating and also to cover a complicated geometry, there also this process of this oxy-fuel torch or DC Plasma torch they have certain limitation. And that is why we have the other one what we call hot filament chemical vapor deposition, hot filament chemical vapor deposition of diamond.

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Exactly what is done here, we have a chamber which we can call a CVD chamber, CVD reactor. It is eventually a cold wall reactor. And here we have to put a filament and this filament has its lead wire which are extended here and we too have a substrate, a table for supporting the substrate. So this is a table provided with a heater. So this is a hot plate like thing and where we can put this substrate.

This is actually the substrate. Now here this is the downstream side where we have necessarily a throttle valve followed by the vacuum pump, that is the vent pump. So this is a throttle valve and

this is the vent pump. Here we do have one Pirani Gauge for measuring the vacuum. Now this is the downstream side. And here at the top we can have a pressure sensor which can measure, monitor and regulate the pressure. And this can be through a controller it can be coupled with this throttle valve.

The basic idea here, this is the pressure sensor, it can be capacitive pressure sensor. The basic idea here is that if we set a pressure value, then according to that requirement this controller can regulate the opening of this throttle. It can be from 0 to 100 and it can set to a value to maintain a particular process pressure inside the chamber. Now on this upstream side, incoming are two gases. One is CH₄, that is the source of carbon and here we have hydrogen.

And they can be mixed before entry and through this it can be just like a shower. So here we can have a shower. So this gas will be showed over this filament. And on this side, obviously we have the mass flow controller, MFC. So the MFC, one for hydrogen and another for methane so this is the basic CVD setup so on this side we have the bottle.

So what happens? Here the basic principle is that this CH₄ and hydrogen they are thermally activated and after that one would expect carbon to separate out in the form of a solid and having the morphology of diamond. And this hydrogen will leave. So this is the global reaction and as a result of that we can expect this carbon to deposit on this substrate here in the form of diamond. So that is the basic principle of hot filament CVD process.

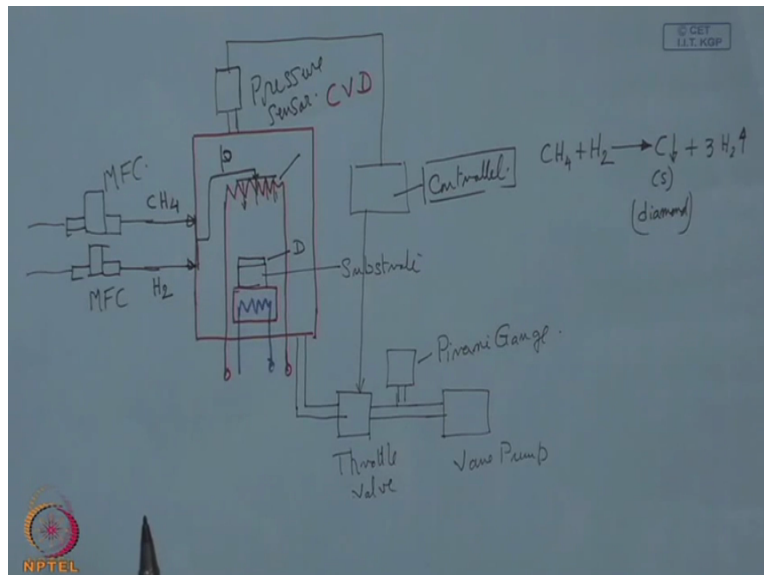
The advantages are that it is very simple to monitor and more importantly that filament can be tuned to the geometrical requirement of a substrate. It can be a turning tool, it can be a milling cutter, it can be a drill and accordingly this filament which is made of tantalum or tungsten, that can be given a particular geometry according to the need. And this side you have the controller.

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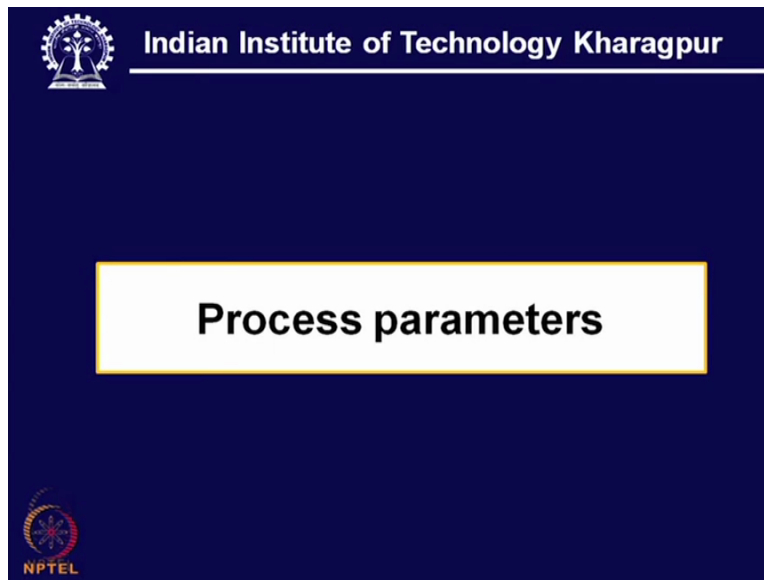
So these are the advantages of hot filament CVD.

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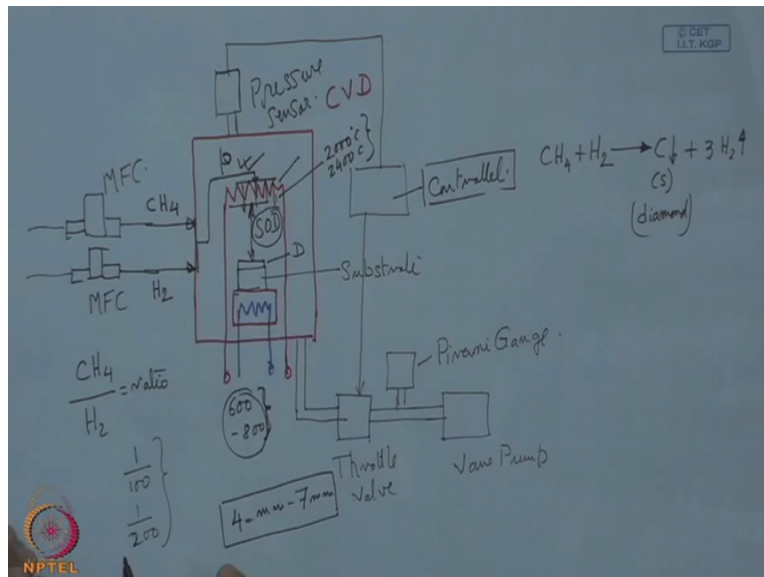


Now here the filament temperature is to be monitored. Similarly is the case with the substrate temperature. These are the two requirements. Pressure, obviously one of the parameter. And then amount of gas flow incoming of CH₄ and that of H₂ which we shall discuss. So this is the basic CVD system just we have discussed here, what are those elements we have in the whole process. So it is basically a cold wall CVD reactor because this wall is not heated.

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Now process parameters process parameter, one is, the most important process parameter is CH₄ by H₂ ratio. We mean the flow ratio. This is one of the important process parameter. All research, review of research suggest that it is mostly kept between 1 is to 100 to 1 is to 200. It depends upon certain other requirements but this is a range of work. That means if it is 100 SCCM, it is 1 or it can be even 0.5.

Now some indicative value which will be useful, the filament temperature, it is 2,000 plus and it can go up to 2,400 degrees. This is an indicative value or the range. Similarly the substrate

temperature, it can be say 600 to 800. There are of course parameter or condition which will govern the exact selection of the exact value. Pressure, pressure we can have any range but again according to this research publication or investigation it is also found that it is kept around 10 to 30 or 40 torque and that is the operational range.

Of course, there can be exception because of the simple reason that this CVD setup and its geometry, these are influencing all these parameters. So we can say that all these parametric combination and their consideration are, they are geometry specific. That means it is specific to the geometry of this CVD reactor. Another important parameter is SOD, this stand of distance. Now this stand of distance and the process pressure, they are also related.

But as we see that this parameter, SOD normally kept between 4 to, around 4 millimeter to 7 millimeter ordinarily. So these are at least we get some indicative values with which one can expect to initiate the process and can have some kind of diamond coating on a particular substrate of choice.

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Now comes the CVD reactions for diamond coating. One thing very important here to know that in this low pressure synthesis to accelerate the process or to make the process reliable, that means reproducibly to obtain this diamond coating over the substrate, one would like to have at the very starting or at the very initiation high density nucleation and that should be augmented by

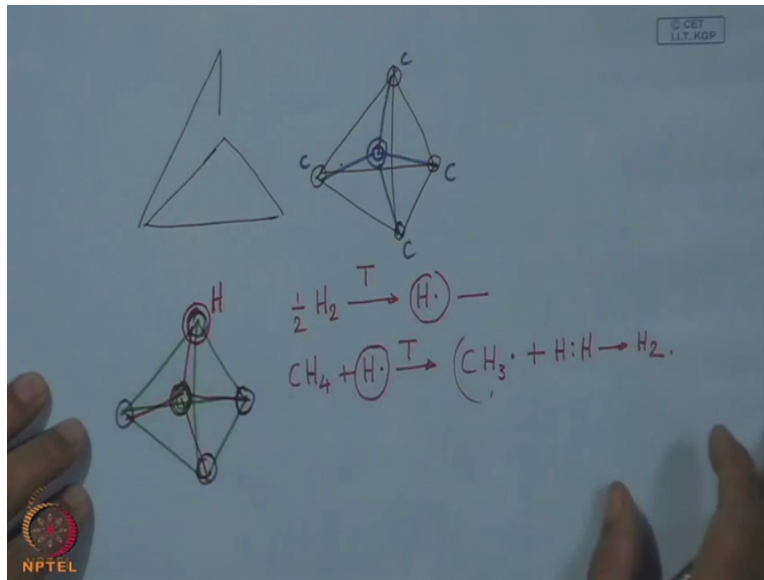
some mechanism. And this is exactly what we need here that diamond dust or diamond seed. And that we are going to also discuss.

So globally what we find that first of all what is the need of this hydrogen. Hydrogen is, one function is to act as a carrier gas, carrier for CH₄. However it has many other roles to play. This is number two function. It must strip off one hydrogen atom from this methane reducing it to a methyl radicle. Number three, it should also have sufficient energy with which it can impinge on the substrate surface assisting or facilitating restructuring of this carbon to diamond.

Then number four, it must also carry, I mean play the role of one effective agent. Now during the process of synthesis, it may so happen in unfortunate situation that this carbon which is separated from methane that can have a structure of graphite instead of diamond. So in that case this hydrogen has one of the very important role to play and it will continuously keep on etching that graphite phase and the diamond will not be that damaged because of this free energy formation of this graphite to hydrocarbon is much negative, highly negative than that of diamond.

So risk of etching of diamond by hydrogen chances are less within that domain of substrate temperature. But hydrogen must be active to have all useful, fruitful etching of graphite so that finally the diamond coating is free of graphite. There is another role of hydrogen, it is called decarburization of tungsten carbide this also one important role of hydrogen to decarburize and to reduce it to metallic tungsten that is its role.

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Now here what we find that if we look into this lattice of diamond, then one can find that it is actually a tetrahedral structure. So this is a tetrahedral structure. Here we have corner atom, four corners and they are connected by one central atom. So this is carbon, this is carbon and that is the central atom. And that is the formation, so this is actually the formation so all the four are connected by the central atom and in the next lattice, again each corner is again connected by your central atom.

So this way it spreads both in the lateral direction and in the vertical direction. Now comes the question when it is the top layer of diamond, when it is the topmost layer of diamond and after that there is no material diamond that means it is floating, it is in contact with air say. In that case this corner, so we can immediately see that for this we have here three corner atom in, corner atom for diamond and one is the central one.

However the top one, that is a vacant site, this is the vacant site and this vacant site immediately that is filled up with hydrogen and exactly that is called the dangling bond. So you have here this central atom connected by three diamond atom. But the top one, this is a vacant site which is filled up with hydrogen. Now this is the situation on the top surface of diamond which is in contact with say air.

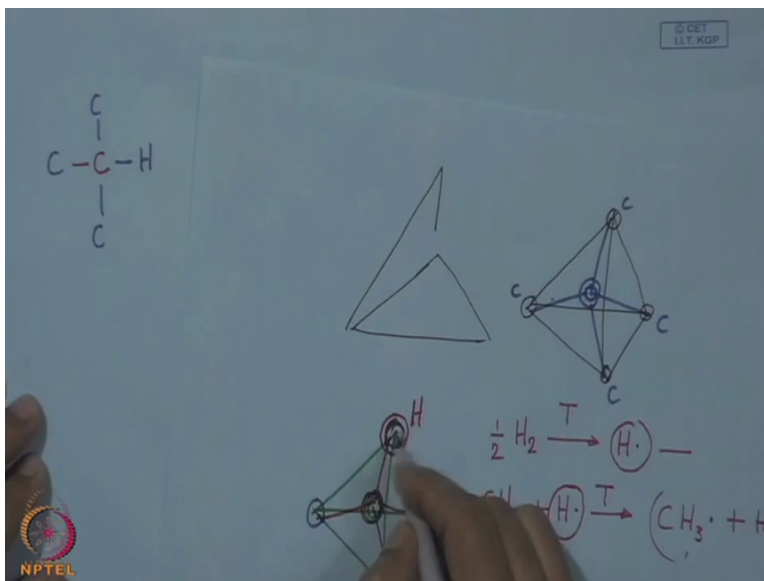
Now the whole idea here would be that when we have the seed of diamond, then this particular hydrogen will be removed from this place and it will be replaced by one carbon filling the entire

structure of this diamond with carbon. So we have now if this hydrogen is removed or stripped and it is substituted for by another carbon, then again we have a complete lattice with this tetrahedral form of diamond. And exactly in this principle the CVD process works.

So first task would be to have this hydrogen which is a molecular hydrogen, that is the first step of the CVD reaction and which is thermally activated and it becomes one nascent hydrogen. It is atomic state. And here we have the methane which is CH_4 plus this atomic hydrogen that will lead to one reaction leaving here a methyl radical, plus this hydrogen and the hydrogen which is already in atomic state, so it will strip one hydrogen leaving a methyl radicle. And these two will form a bond and that becomes again a molecular hydrogen. So this is one step reaction of CVD.

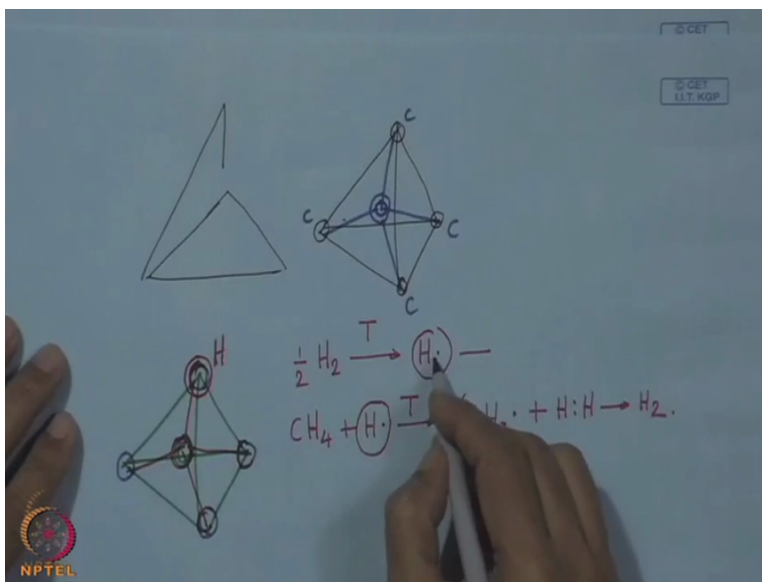
So first is that conversion of molecular hydrogen to atomic hydrogen and it follows by stripping of this methane to a methyl radicle and combining this atomic hydrogen, one of the stripped H atom and then conversion again to molecular hydrogen. This is one step of reaction. Now what we have already shown in this diagram, that means when we have such diamond seed on which this diamond film has to grow, then we can write down the second step of this reaction.

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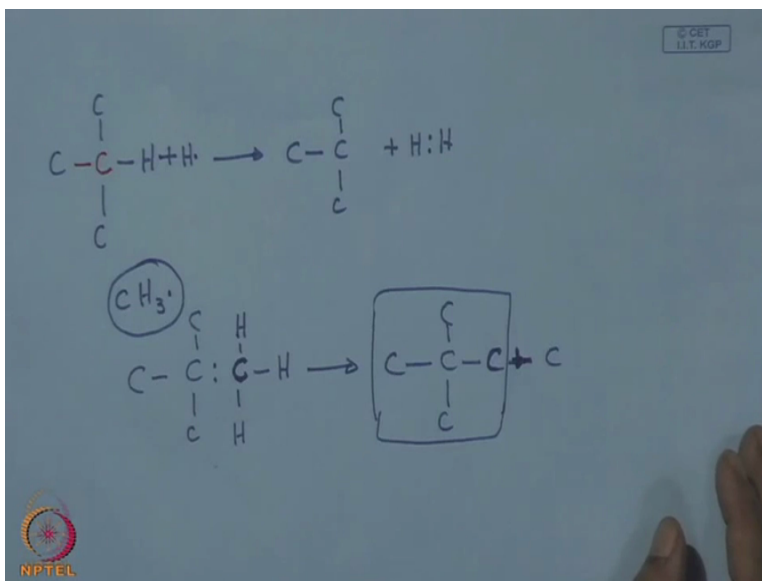
That means in this case we can write down, this is the central atom and then we have the corner atom. For example, this is the corner atom, and this is the corner atom and then we have this hydrogen. This hydrogen means actually the hydrogen which is present here.

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Now what is going to happen? This hydrogen which is in atomic state, that will participate in this reaction.

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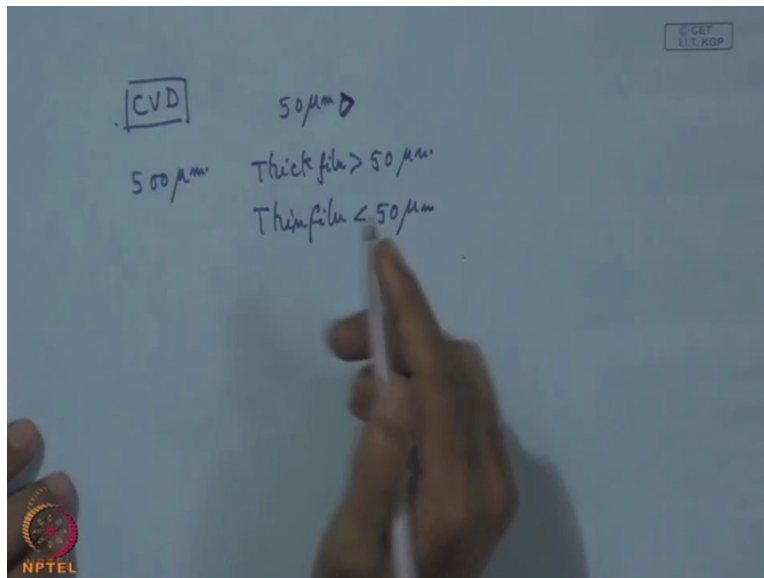


That means here it will come like this one and it will remove, that means the second step would be it is like this. So it is now free and here we have this bond formation. And this side is free, now in this side what is going to happen that methyl radical which is CH₃, so this methyl radical will occupy this position. That means with this formation where this side is free, so here that methyl radical will come and it will have this configuration, like this.

So the carbon from methyl radicle, that is occupying this position which is left vacant because of the stripping of this hydrogen which forms a dangling bond at the top. And this is removed by this atomic hydrogen. So what we see now, and this process will keep on going, it will keep on repeating. So what we finally see here that we have a complete lattice of diamond. That means this side is no more vacant and we have here this C and this will keep on moving in every direction and we get this complete filling of this atomic site.

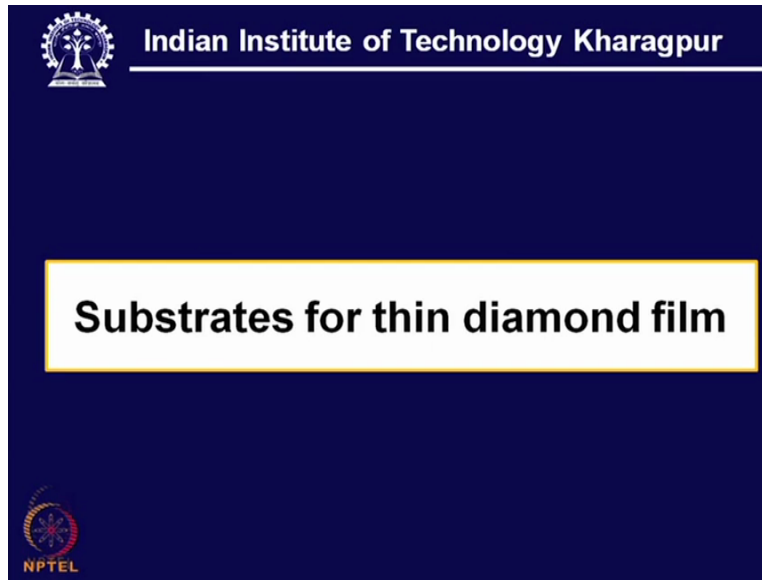
So on every location, wherever you have that removal of hydrogen in every location, that will be filled in by this methyl radicle. So this way the diamond coating will keep on growing. So this is actually what we know as CVD reaction.

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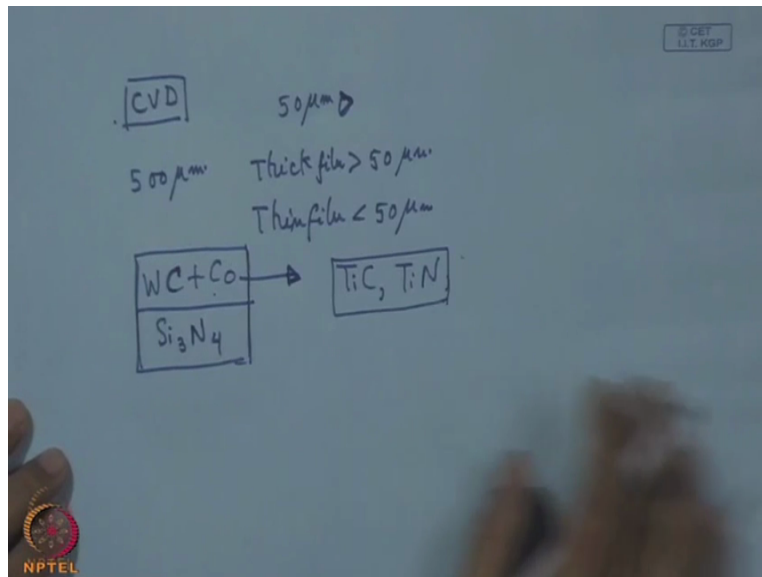


Now here we find we have both thick film and thin film. Now thick film means we can have this CVD grown diamond which can be about 500 micron, that is we call thick film. That means a film which is thicker than 50 micron, that we call greater than 50 micron, that we call thick film. So a thick film means greater than 50 micron. And thin film, less than 50 micron. So this thin film we are interested in to put directly as the coating on the substrate.

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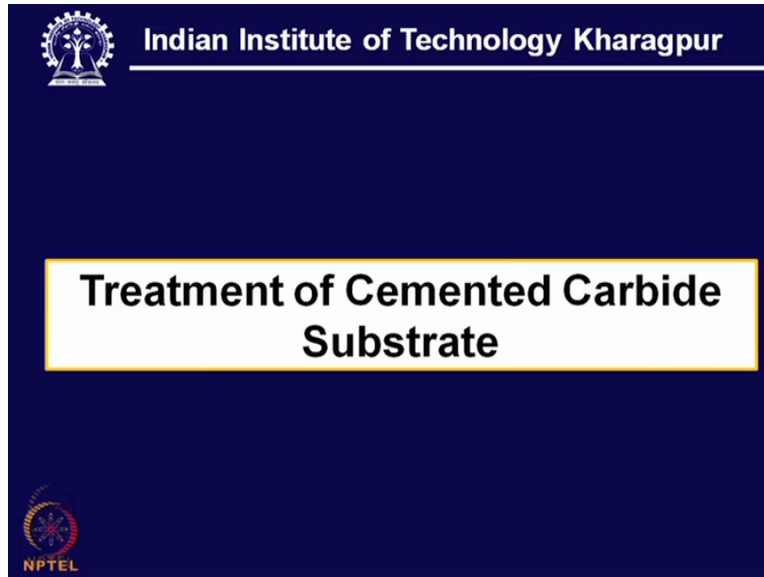


Now this substrate for thin film. Here we can have immediately tungsten plus cobalt plus silicon nitride, Si_3N_4 . These are the two substrate but considering the toughness and other requirements as silicon carbide, as tungsten carbide plus cobalt that is used as a substrate for all other type of hard coating like TiC , TiN . Immediately the tool manufacturer will find it one of the substrate of immediate interest.

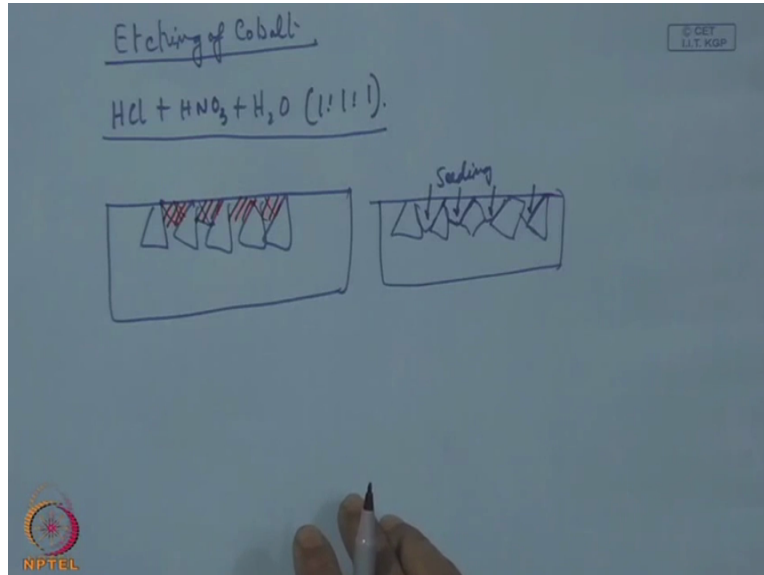
However the problem is here with the cobalt. Now when we have cobalt, then the diamond which is deposited on this surface, that cannot be a stable diamond and this cobalt try to make

this conversion of diamond to graphite or nucleation and synthesis, further growth of diamond becomes extremely difficult on cobalt surface. So here the whole idea is to remove this cobalt.

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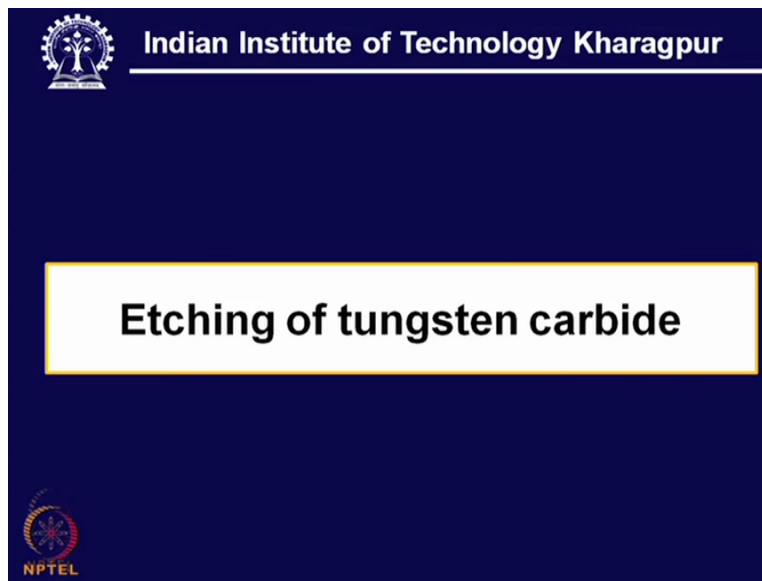


So treatment of cemented carbide substrate so here one way of doing the thing is etching of cobalt. So etching of cobalt, this is normally done by use of a mixture of HCL, HNO₃ and H₂O in equal proportion, 1 is to 1 is to 1. And that is used for etching of cobalt. And this not only, suppose you have here tungsten carbide particle and then this from this position these are the

area where cobalt is very much present. So those areas cobalt should be etched and as a result we are left with a configuration like this.

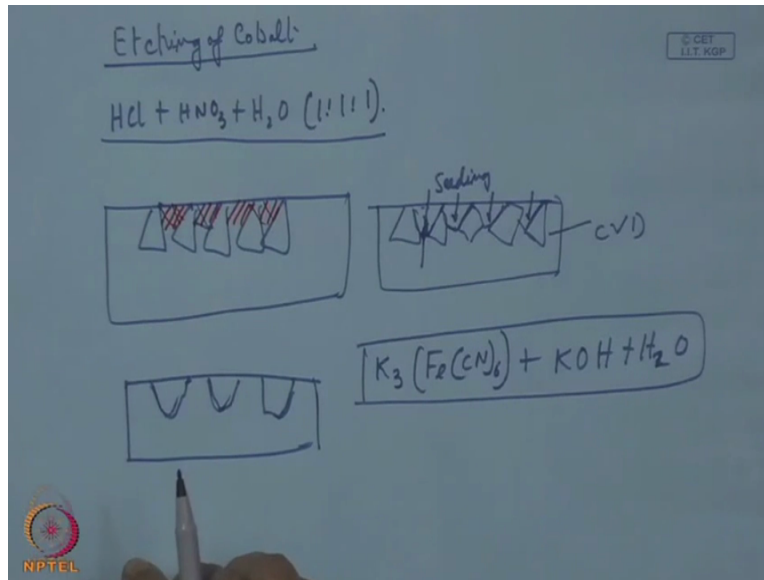
And this area is actually cavity. And this cavity, now we have some advantage in that, this cavity which are formed because of the removal of this thing marked by this hash mark. So here this is the cavity. And here we go for some kind of diamond seeding, so here we go for this diamond seeding. So this diamond dust will fill this space and which will be later used for further growth of diamond. So this is one way of doing the thing.

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But there is another school of thought, etching the tungsten carbide.

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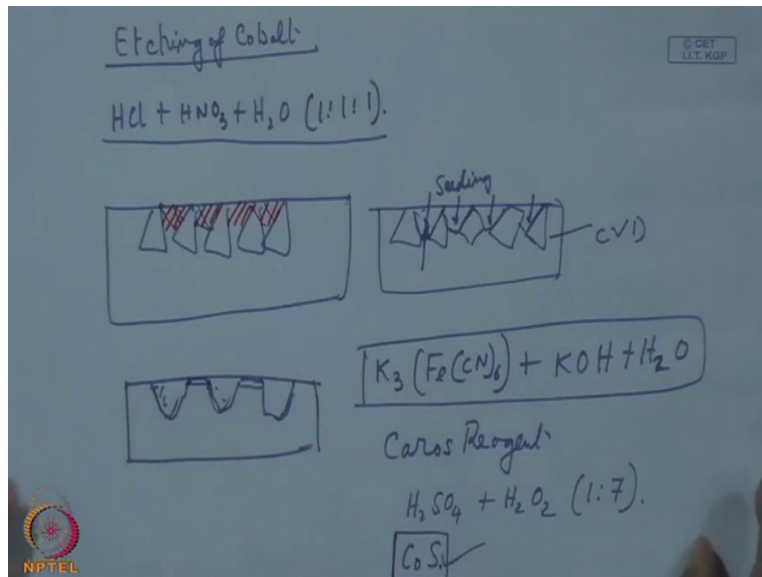
The whole idea here is that though we are etching cobalt but it may be, may not be very useful because during the high temperature CVD this cobalt can also diffuse and come to the surface and the whole purpose of etching the cobalt is lost. So here the whole idea is that we can also etch tungsten carbide to leave these cavities. That means in this case this tungsten carbide will be etched, so like this this will be etched leaving large cavities.

And this can be done by one solution, it is called $K_3Fe(CN)_6$ (38:45) reagent. And then we have KOH plus H₂O, this is the standard reagent for etching the carbide and that can be intelligently used for removing this. So here we have these cavities. That means the whole idea here only, not only to get rid of this cobalt but also to make some pocket for reception, for receiving this diamond dust. And it will be acting like seed. But here it is not just etching of tungsten carbide, it is actually combined etching.

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Combined etching means now we have pocket but still cobalt is not yet deactivated. So for deactivated, for deactivation of the cobalt what is used, Caros reagent and Caros reagent means here we have H_2SO_4 and H_2O_2 and that is a mixture in 1 is to 7 and that becomes one reagent for deactivating this cobalt. Actually what is going to happen? Because of this reaction, we can have cobalt sulfate and which will be converted finally into cobalt sulfide.

So final aim is in this etching process which is a part of substrate preparation, so it is one of the very, very important step in preparing the substrate for receiving that carbon in the form of

diamond. So this is actually cobalt sulfide which should form here in addition to this cavity formation. So this part where originally cobalt is present, that should be transformed into cobalt sulfide. And this cobalt sulfide is chemically extremely stable. And this is also very, very passive. That means this cobalt cannot now really do any damage to this diamond surface and it is well protected.

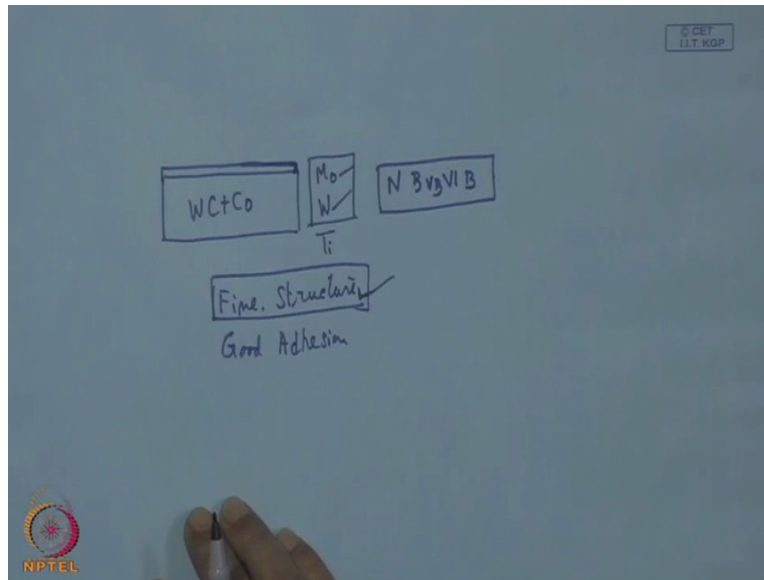
So this is another way of handling the thing. And as we have already seen that it is substrate preparation step which is unlike that of other CVD coating process, as for example CVD of TiC or TiN or aluminum oxide, there we do not require that special treatment or special attention so far as this substrate chemistry is concerned. So now here what we see, this is actually seeding and this seeding is done in this pocket with this totally passivation of this layer.

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There are various processes which has been successfully attempted and this is one of those, interlayering.

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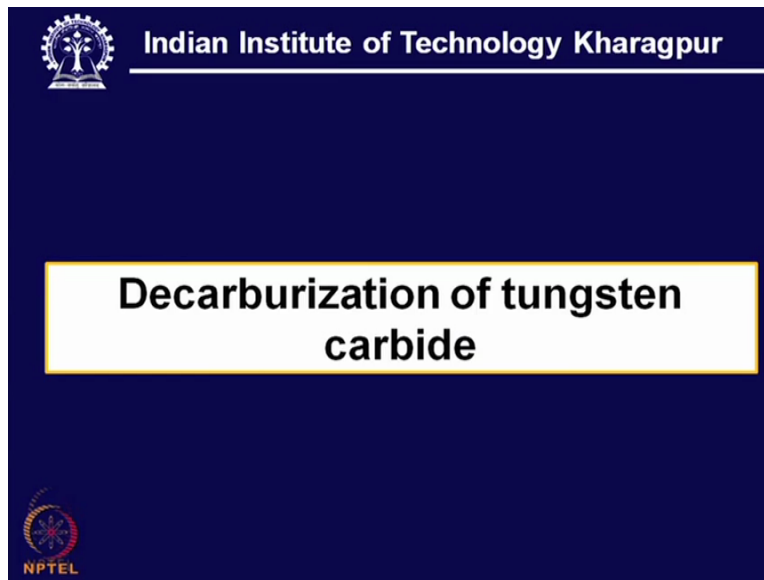


Interlayering means even if we have tungsten carbide plus cobalt, but we can protect, we can suppress this cobalt, any adverse action of this cobalt by interlayering with a metal. Say for example, molybdenum or tungsten or titanium, these are mostly found from this group IVB, VIB and VB. These are all the transitional elements but all, it is not that all are compatible. But if we look at all those practices done previously, it is mostly molybdenum and tungsten, they are found to be very, very effective in not only suppressing the cobalt layer but also enhancing the nucleation density of diamond during the CVD deposition process.

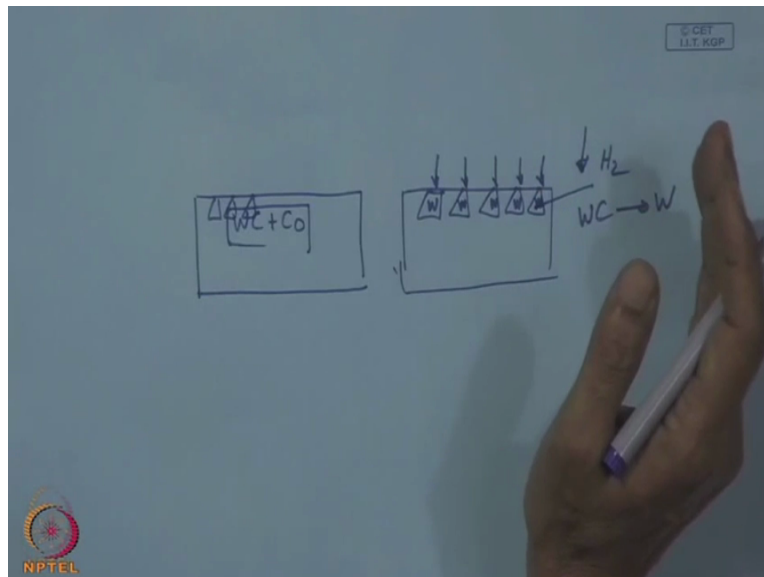
Because one of the important requirement in this is that fine grain structure of diamond, fine grain, fine structure, next is good adhesion and for that this so much care has to be taken for preparation of the substrate, good adhesion. So fine structure is important if we like to use it for finishing operation. That means surface of the, working surface of the tool or the coating that must have that smooth surfaces smoothness. And the third one is that this coating process is capable enough to have the deposition in a complex geometry with all uniformity of thickness and uniformity of the property.

These are the three basic requirements they must be met in order that this coating become one of the successful surface, coated surface for the machining application.

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This is actually decarburization of the tungsten carbide. So some attempts has been also made here that we can see that this is actually tungsten carbide and cobalt, so that is uniformly distributed. But what one would expect at the top where you have tungsten carbide particle and the whole idea here is to have decarburization by strong hydrogen etching. So with this hydrogen etching the whole idea is that to reduce this tungsten carbide to metallic tungsten.

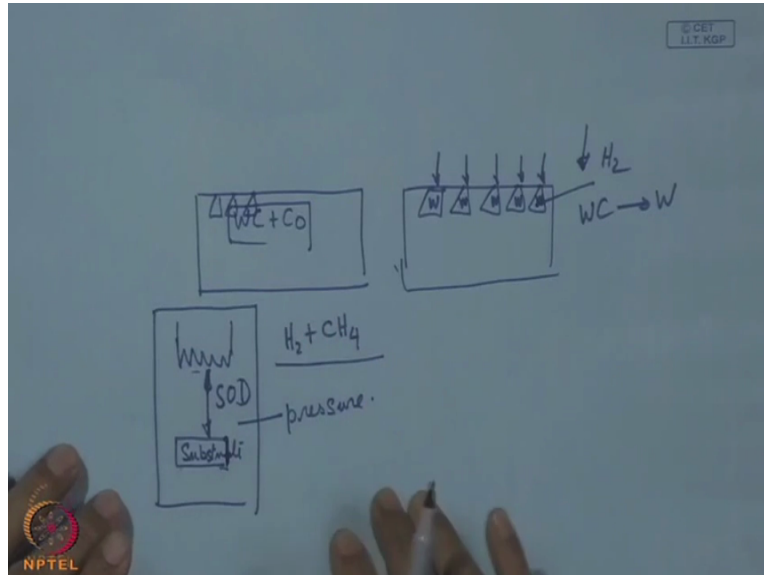
And now this tungsten will be very, very useful as a receptor surface which will receive carbon and also have some chemical attachment and thereby we can have very good adhesion. So this is

one way of doing the thing, that means preparing the surface by just reducing this tungsten carbide to a metallic carbide.

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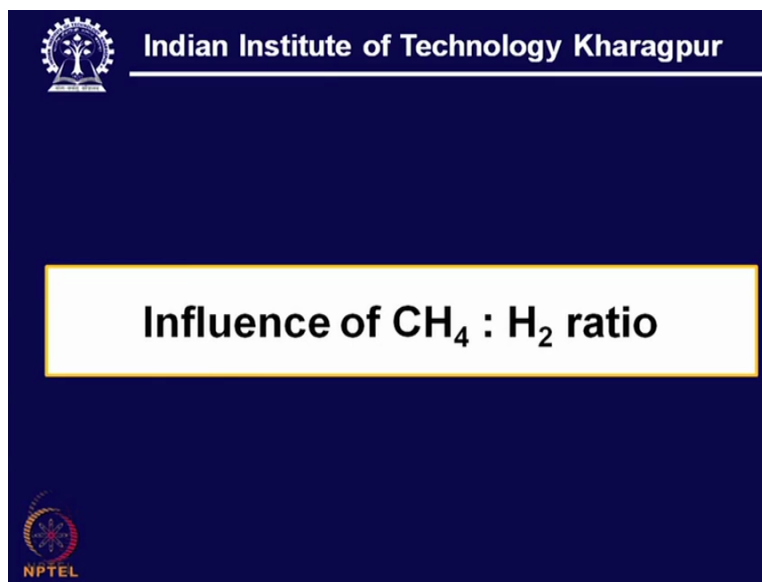
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Now here we find that influence of SOD. This, what we have seen, this is the filament and this is the substrate. How to choose this SOD? In fact, when this is H_2 and CH_4 incoming after all sort of chemical reaction, this is the mainstream which is moving towards this with certain velocity. Now what is important here that they should arrive at this surface with certain energy. To have that, we should not promote unwanted collision before they arrive at the substrate surface.

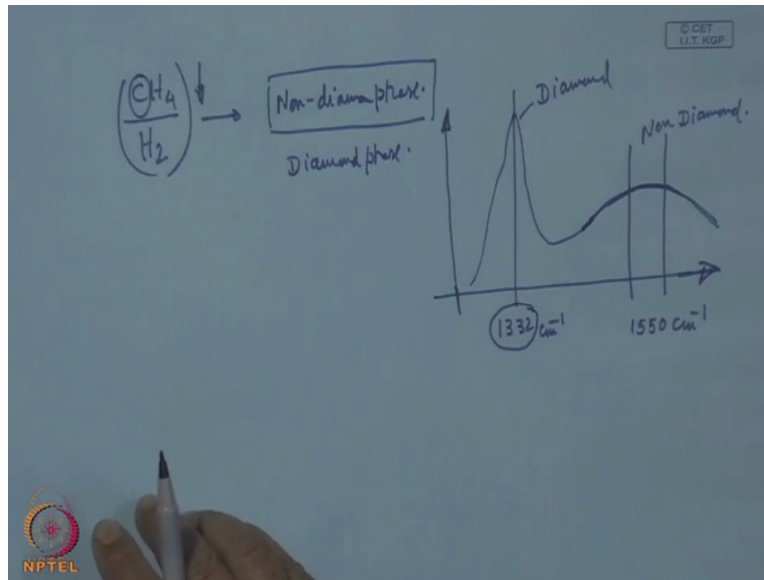
This is the substrate, so naturally selection of this stand of this distance and the selection of pressure, they are not independent. And accordingly the pressure and the substrate distance, that has to be also controlled or that has to be also considered in concurrence with the selection of the pressure. So pressure and SOD, they have to be considered in their totality. The whole idea again is that to have that impingement of this gas stream over this substrate over sufficient energy, further necessary conversion or structuring of the diamond morphology and not to have unwanted collision in the midway so that energy is spent or energy is lost.

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Influence of CH₄ to H₂ this is very important consideration, CH₄ to H₂. Now CH₄ concentration means higher percentage of carbon, so we have more amount of carbon available for the growth of the coating. However it is found that if we have too much of methane in proportion to hydrogen, we may end up with non-diamond phase.

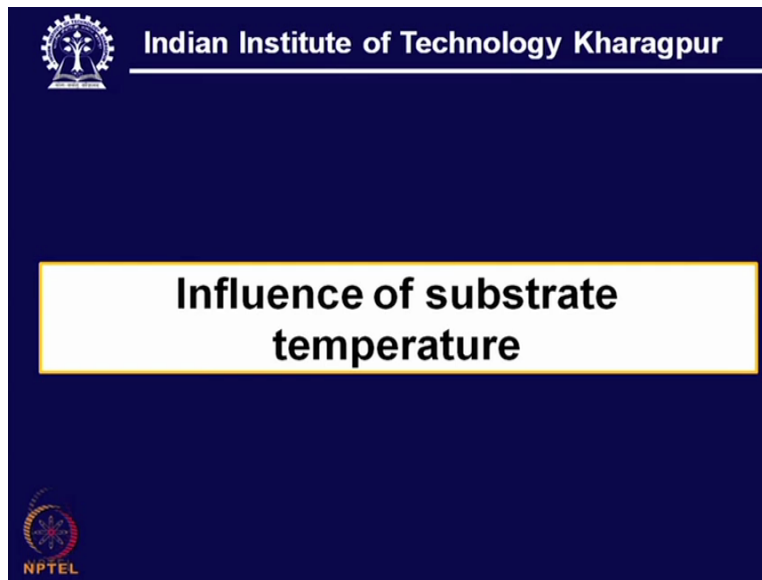
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Non-diamond phase, what we want? We want diamond phase. And this we can check with, this is one of the most important characterization tool is that Raman shift. So if we consider the wave number in this x axis and here it is the intensity in arbitrary scale, then we can have a sharp pick here. And this is something one may expect or may happen. And this is exactly around 1,332 per centimeter. So this is one typical pick for this Raman shift characterizing the very existence of diamond.

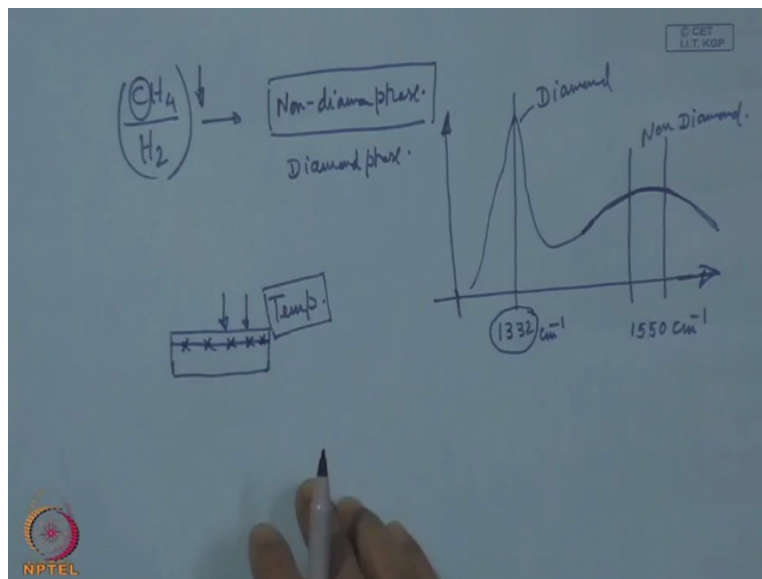
But if we have a broad wide hump and which we see in the zone around 1,550 per centimeter, then this is actually characteristics of the non-diamond phase. So this is actually the diamond and this is a non-diamond. So after deposition by this Raman spectroscopy and Raman shift, one would come to know whether this coating is having the diamond phase or it is having non-diamond phase. So this is one of the thing which should be decided here in selecting this CH_4 , H_2 . So this is one important consideration.

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The slide features the Indian Institute of Technology Kharagpur logo at the top left and the NPTEL logo at the bottom left. The main title, "Influence of substrate temperature", is centered in a white box with a yellow border. The background is dark blue.

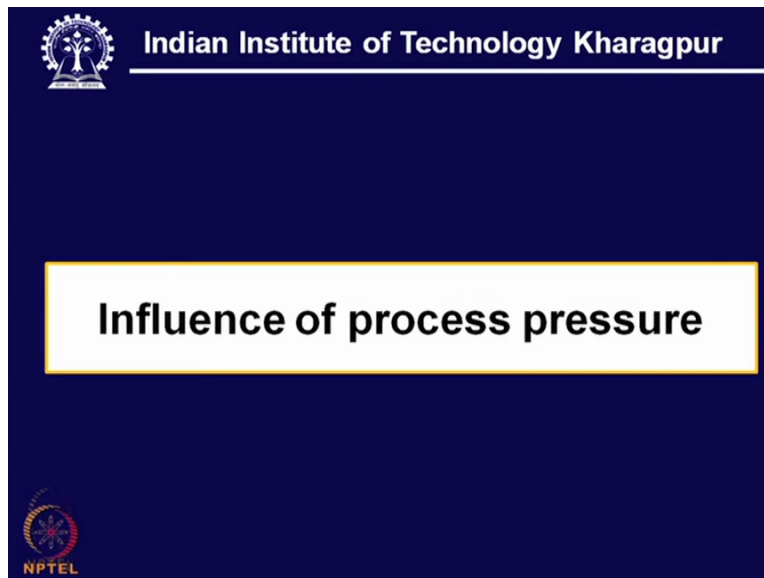
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Influence of substrate temperature, now this substrate temperature, one way it is helping in the nucleation and growth of diamond. But at the same time it can be also counterproductive if we have too high temperature where it will be thermally activated. And then by this hydrogen also we can have etching of diamond. So this temperature, it is somewhere in between too extreme which should be chosen properly to have a diamond with a growth, reasonable growth.

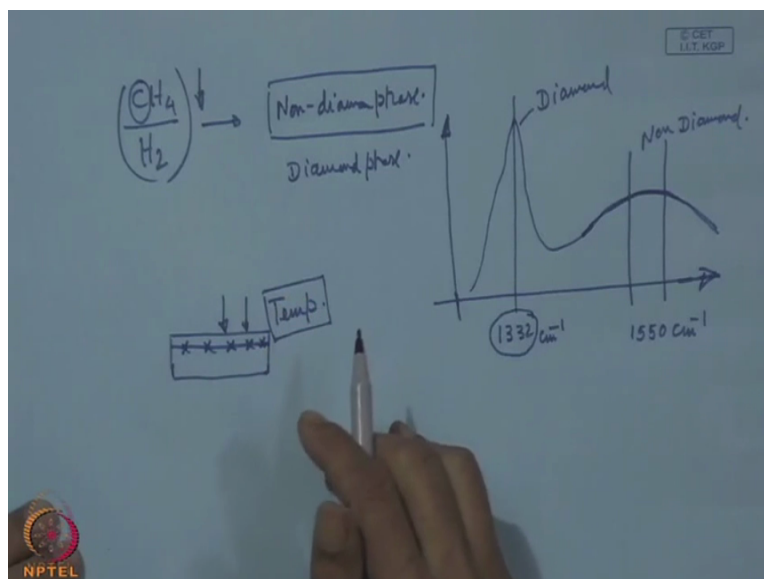
And more importantly that if we have too high temperature deposition, like any CVD we may also here adhesion problem because of the too much of stress discontinuity. So this point should be also looked in.

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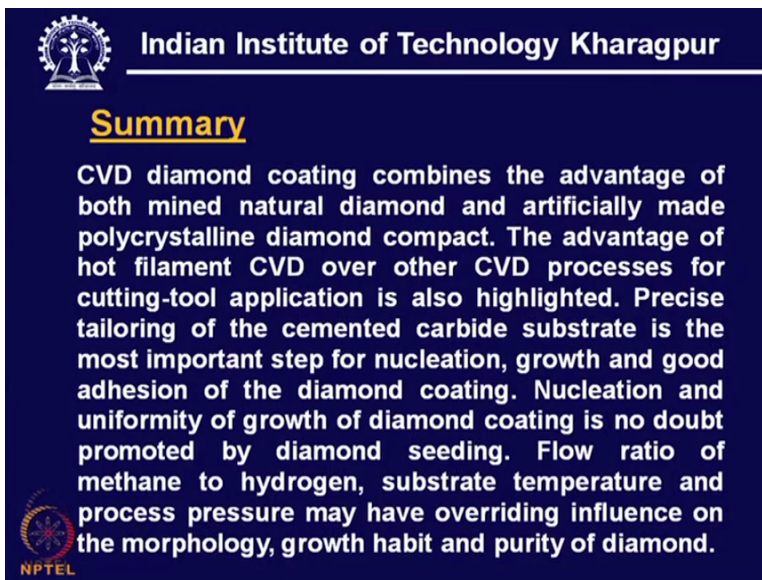
Influence of process pressure, this also we have mentioned while considering the SOD, that means the process pressure. If it is too low, then we can find the residence time will be also not adequate for this adsorption on this surface.

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But if it is too high, then we invite the collision in the midway between the filament, hot filament and the substrate and there will be much loss of energy. So that part also to be taken into consideration. So this way we have seen that this methane to hydrogen ratio, then substrate temperature and pressure and SOD, so they have to be considered in there totality to have a well-adherent, well-nucleated and pure diamond which is having good adhesion and at the same time purity, high purity diamond. So this is one of the issue one should look in.

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Indian Institute of Technology Kharagpur

Summary

CVD diamond coating combines the advantage of both mined natural diamond and artificially made polycrystalline diamond compact. The advantage of hot filament CVD over other CVD processes for cutting-tool application is also highlighted. Precise tailoring of the cemented carbide substrate is the most important step for nucleation, growth and good adhesion of the diamond coating. Nucleation and uniformity of growth of diamond coating is no doubt promoted by diamond seeding. Flow ratio of methane to hydrogen, substrate temperature and process pressure may have overriding influence on the morphology, growth habit and purity of diamond.

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Now coming to the summary what we can find that diamond coating combines the advantage of both mined natural diamond and artificially made polycrystalline diamond compact. The advantage of hot filament CVD over other CVD process for cutting tool application is also highlighted. Precise tailoring of the cemented carbide substrate is the most important step for nucleation growth and good adhesion of the diamond coating. Nucleation and uniformity of growth of diamond coating is no doubt promoted by diamond seeding.

So here we find two issues. One is to accelerate the process and to act like a catalyzer, this diamond seeding is one of the important step. Flow ratio of methane to hydrogen, substrate temperature, process pressure may have also overriding influence on the morphology, growth habit and purity of the diamond. But what is important here that finally it is well-adherent diamond with fine structure and with a process capability which can facilitate deposition of this

diamond coating over any complex geometry. That will decide how this process would be useful in practical application.