Technology of Surface Coating
Prof. A. K. Chattopadhyay
Department of Mechanical Engineering
Indian Institute of Technology, Kharagpur
Lecture-07
Chemical Vapor Deposition of Carbo-Nitride Coating

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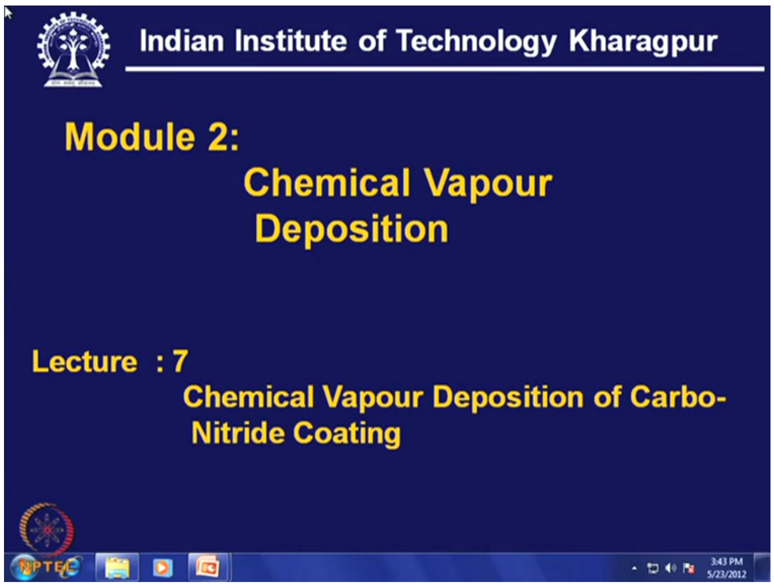

Okay. Today we shall discuss chemical vapor deposition of carbo-nitride coating.

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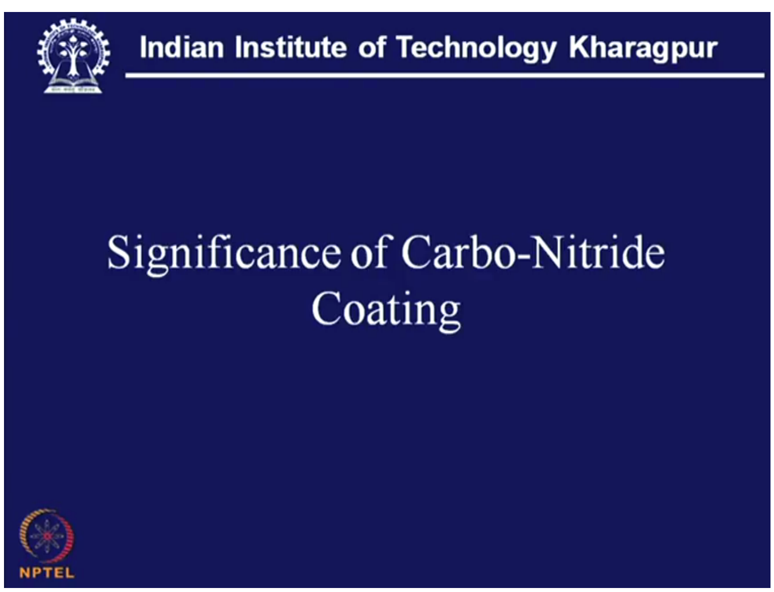

Significance of carbo-nitride coating.

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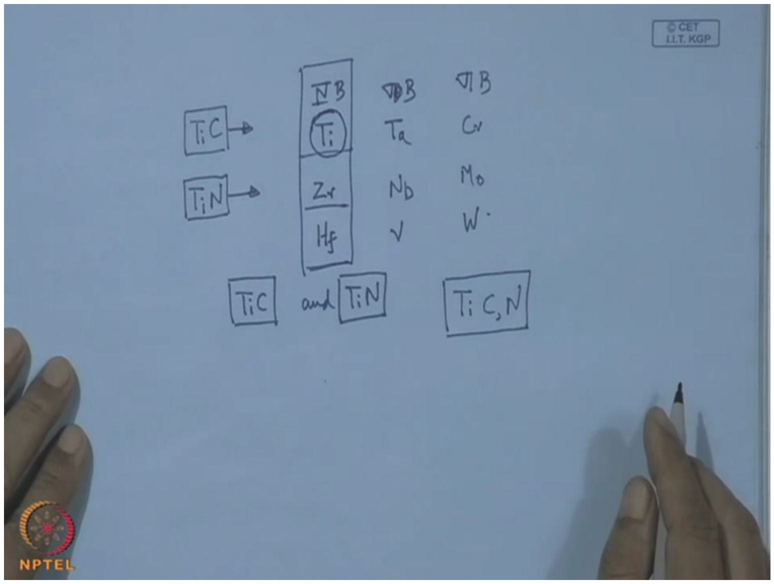

Now we know that TiC, that means this titanium carbide coating that is known for its extraordinary hardness and which means enhancement of abrasion wear resistance. And on the other side, we have also titanium nitride which is better known for its anti-welding property and also anti-adhesion property. So when titanium carbide is used, then the domain of application is well defined that means where the functional surface is subjected to abrasive wear, then we put TiC coating.

However when the application demands a coating which should be capable of combating the wear at a higher temperature, in that case the wear resistance is not just abrasion but there could be also welding or adhesion, adhesive wear that also come in picture. And in that situation, titanium nitride or the nitride of any particular transition element is, it appears to be much more effective and durable than that of carbide coating.

Now if we consider the transitional element, we can quickly have a look, these are from Group IVB, now VB is tantalum, niobium and vanadium, VB. This is VIB, chromium, molybdenum and tungsten. Now if we pay our attention on this, here we have titanium, zirconium and hafnium and their carbide. So the chemical stability of carbide that goes in this order. That means hafnium carbide is more chemically stable than zirconium carbide.

And zirconium carbide will be more chemically stable than that of Ti carbide, titanium carbide. Similarly we can also have hafnium nitride, zirconium nitride or titanium nitride. If we now choose titanium as one of the strategic material, in that case we have TiC and TiN having their well-defined domain of application. But there are situation where we may need a balance of property between titanium carbide and titanium nitride. That means the functional surface need both resistance to abrasion and resistance to adhesion. That means more of chemical stability, anti-welding property.

And in that case, a balance between TiC and TiN is found out to be quite effective and that is why for application carbon and nitrogen are brought together to make a coating, a composite coating or a multi-phase coating which is known as titanium carbo-nitride. And this titanium carbo-nitride, one of the major application is in the area of manufacturing, that means the tool for manufacturing where one can use titanium nitride, carbo-nitride coating. It is an intermediate between titanium carbide and titanium nitride giving a balanced property of abrasion, wear resistance and also anti-welding or adhesion wear resistance.

In addition to that, it can be also noted that if we like to have a top layer of TiN and it is to be done on a tungsten carbide being one of the most strategic material for cutting tool and all wear part, in that case the high temperature CVD does not allow direct deposition of titanium nitride. So in that case, that first layer just in contact with the substrate is titanium carbide and the top layer is titanium nitride.

But in between to graduate the property slowly from the interface to the top surface, titanium carbo-nitride is also used just like a bridging layer or a buffer layer.

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So this is TiCN coating. Because titanium carbide is well known since the cutting tool came into being and also titanium nitride it is known for its chemical stability, so it is titanium carbo-nitride coating which can have also well strategic application. And some of those strategic areas, carbo-nitride coating can be found out to be one of the very best candidate.

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Now characteristics of TiCN coating, that is already known to us. That means this titanium carbo-nitride is more chemically stable than that of titanium nitride while resistance to abrasion will be better than that of titanium nitride. So this is something in between two extremities, that means titanium carbide and titanium nitride.

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Now this concept of putting carbon and nitrogen together, that can be extended to also incorporating oxygen.

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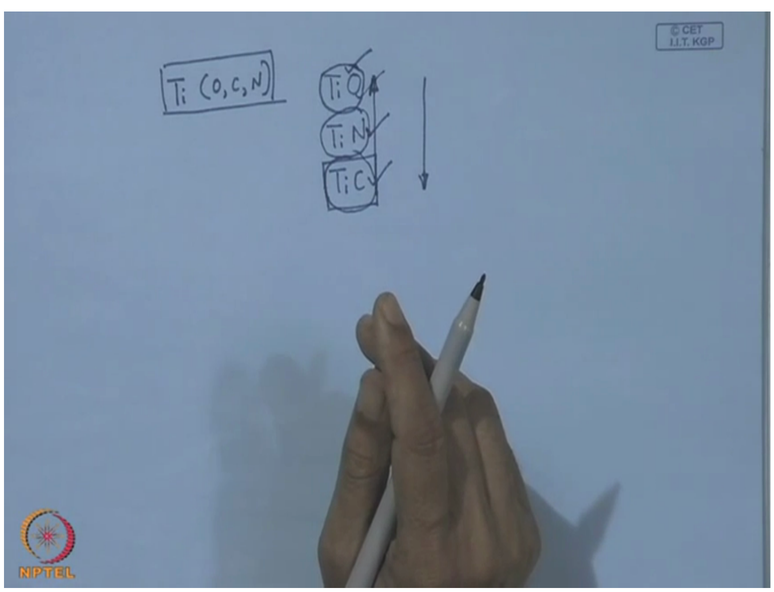

And here we get titanium oxycarbonitride. The whole idea behind this combination is that, that TiO, if we put TiN and TiC in terms of chemically chemical stability, this is the order. In terms of hardness, this is the order. That means when it is a temperature sensitive work involving the chemical stability of the material, then it is of course TiO which can have a very overriding influence.

However if we consider alone the abrasion wear resistance, in that case TiC would be the best candidate. So titanium nitride we find somewhere in between, so chemical stability of TiO that is the highest, TiN is in between and TiC at the lowest end. And when it is the hardness, it is TiC best, then TiN and TiO is that is the last choice. If the situation demands that we must have some blending of all sort of requirement, that means hardness, what does it mean? Abrasion resistance, anti-welding property and chemical stability because of the wear which is actually temperature sensitive like chemical reaction or diffusion wear.

In that case, we find that TiO would be the better candidate than TiN or TiC. Now following the same logic which was behind this coupling of carbon and nitrogen, one can keep on mixing also oxygen with carbon and nitrogen giving a new phase which is called titanium oxycarbonitride. And it has been found to be one of the effective coating particularly in those situation where temperature can be quite high promoting or inviting diffusion wear or chemical reaction type wear. And in that case it has been found that TiO is playing a decisive role in combating the wear.

However it has to be also understood in clear terms that alone chemical stability cannot serve the purpose or serve the requirement of the cutting tool. It is also abrasion wear resistance or hardness, that is also desired in the cutting tool. So we can keep both the extremities together in right proportion and we have titanium nitride in between. And putting this oxygen, carbon and nitrogen in right proportion, we can get a titanium oxycarbonitride.

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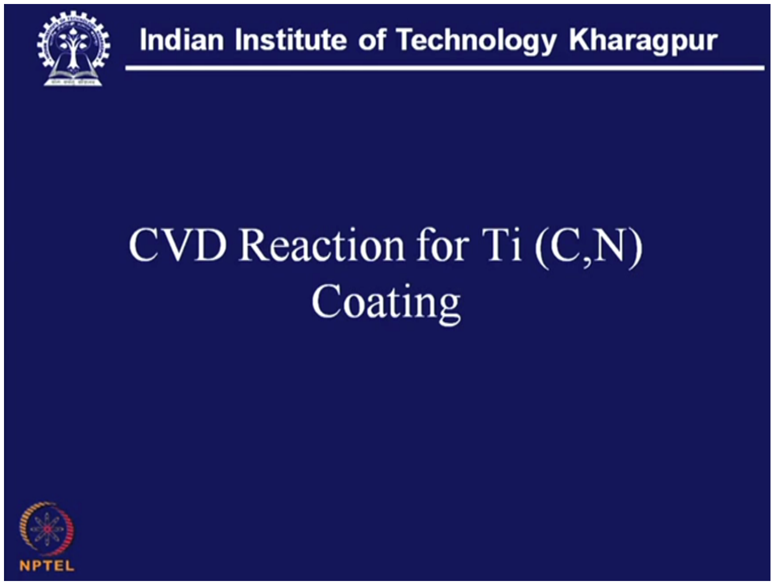

Now CVD reaction for titanium carbo-nitride. Now CVD reaction for titanium carbo-nitride, in that case we have to take if we consider the conventional CVD.

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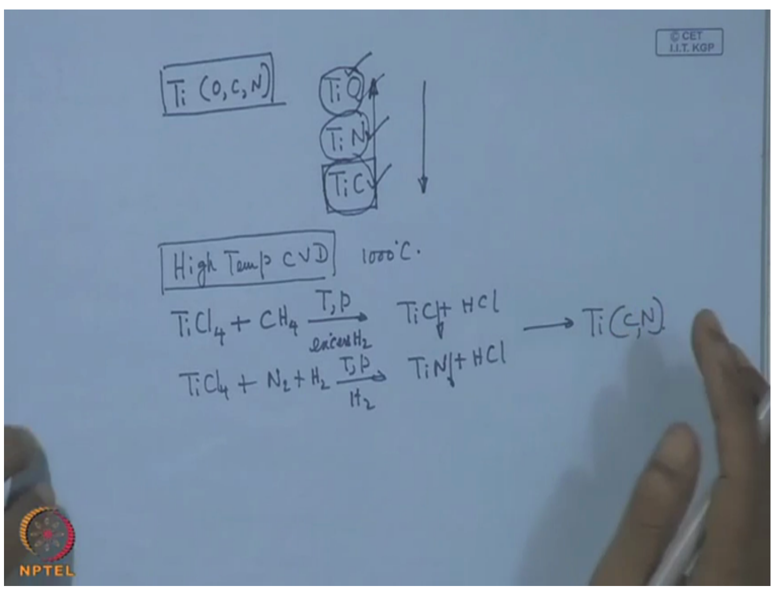

Conventional CVD means high temperature CVD. This high temperature means around 1,000. So here what are the basic reaction which can be illustrated by this equation TiCl4, CH4, here we have temperature, pressure and excess H2 as carrier gas and also to prevent reversal of the reaction. So that gives us TiC plus HCL. It has to be properly balanced.

And also we can have in simultaneous reaction N2 and H2, that can also give with pressure, temperature and with hydrogen, also here we can get TiN plus HCL. So these are the governing equation. Now if we put them together, we are going to get this TiCN in right proportion. Now the problem with this is the requirement of high temperature.

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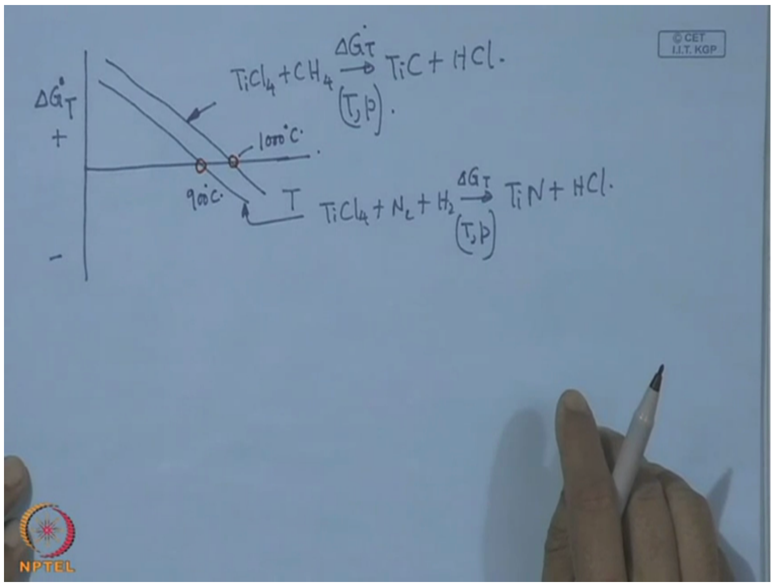

Now we can have look, quick look in this diagram, the Ellingham diagram. Ellingham diagram means we have on one side delta G0T, this is plus, this is minus and that is the temperature. Now this Ellingham diagram will give us a clear guidance how the temperature can be determined or what will be the threshold temperature for to carry forward the reaction in the, from the reaction, reactant to the product. So it should be a product favored reaction.

So in that case we can find two lines maybe something like that. This is just illustration. This one may be TiCl4 plus CH4 which gives us TiC plus HCL with proper, of course with proper balancing. And this one that will be for TiCl4, N2, H2, that gives us TiN and HCL. Now here what is important, this delta G0T or delta GT because we have a pressure which may not be standard atmospheric pressure. So this is T and P and here also T and P.

So what is important, what we understand from these two diagram that we have to really target these two points. So this is for TiC and this is for TiN. Now these are the two points. From our experience we know that if we like to conduct the classical CVD of TiC and TiN, this temperature is around 1,000 degree and this is around 900 degree centigrade.

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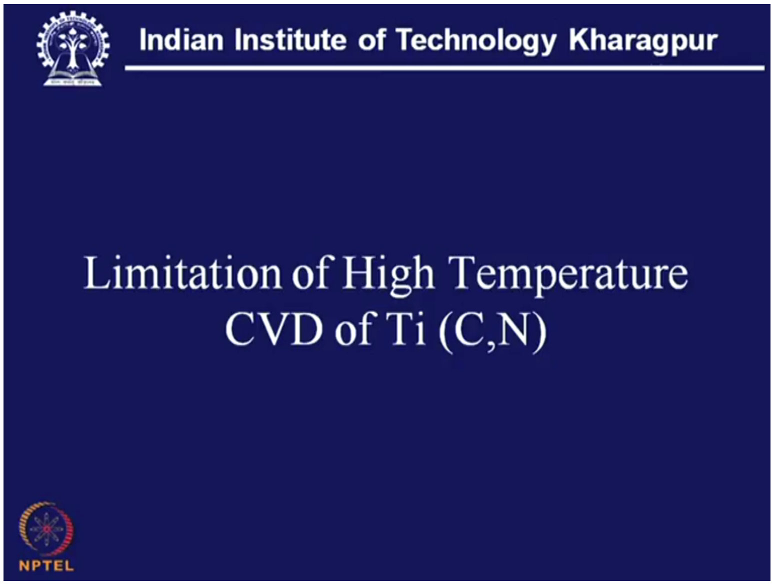

Now these 900 and 1,000, that is exactly what we understand as limitation of high temperature CVD. Straightforward HSS, tool steel, die steel or any structural material steel cannot be a good candidate as a substrate for this CVD because it will be metallurgically damaged, it will be annulled, there can be grain coarsening. So lot of post CVD processing has to be taken and this is really a challenging task. On the other hand, if it is on carbide, tungsten carbide, this tungsten carbide being a very good candidate which can support this temperature, it is free of any problem.

So one can look into the limitation of this CVD in that, this temperature does not allow the substrate to be used for receiving this coating though there is tremendous demand for this carbo-nitride coating, titanium carbo-nitride coating which is a blend of TiC and TiN.

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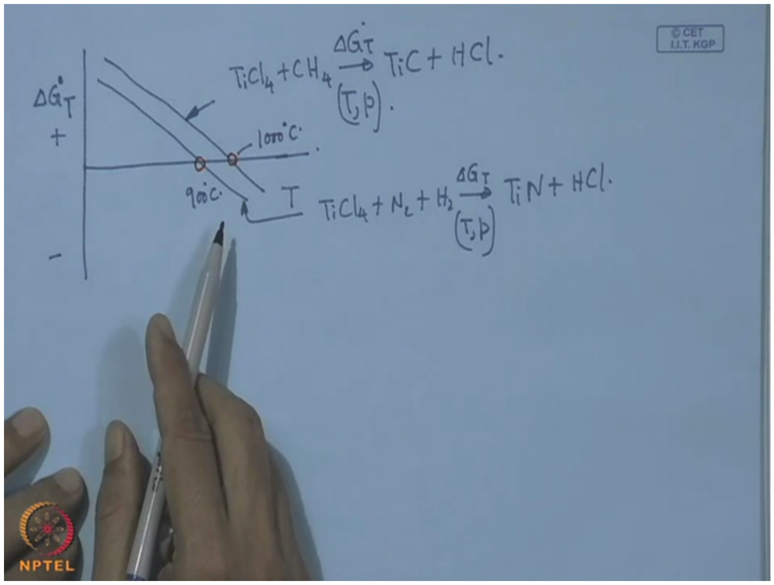

So that is why what we need to do, we need to shift this point on this side. What we can see from this diagram further that this reaction is also lot spontaneous. That means we have to raise this temperature and so that the whole delta G0T corresponding to a temperature that can be well below this side so that the reaction can proceed spontaneously in this, in the direction of the product.

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And exactly for this reason we find that there are certain materials which are actually the source of carbon and nitrogen which can allow us to go down with the temperature. That means we can bring down the temperature, lowering the temperature this titanium carbo-nitride that can be conducted in the zone which of immediate interest.

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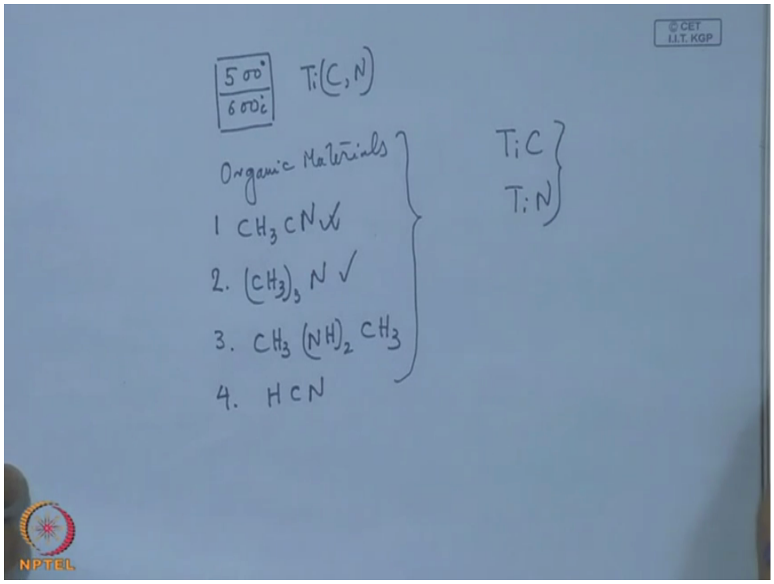

That means the zone say 500 to 600. Even if it is lower than this that will be also most welcome. But considering the limitation of this chemical reaction, one can fix a target of, a range of 500 degree to 600 degree, not above that. And if we can conduct this CVD of titanium carbo-nitride by following another path, then that would be something quite advantageous and its CVD can have an widespread application.

Now what is this moderate temperature CVD? The key factor behind this moderate temperature CVD is the source material for carbon and nitrogen. These are actually organic materials for C and N. That means this acetonitrile, number one, CH3CN. Number two, trimethylamine, this is CH3, trimethylamine. Then we have the third one, dimethylhydrazine, this is dimethylhydrazine and then hydrogen cyanide. So some serious investigation has been carried out to look for certain source materials for carbon and nitrogen which can be effective donor of carbon and nitrogen and at the same time titanium will be supplied by the conventional source.

That means titanium tetrachloride and putting them together perhaps we can see that the reaction can be conducted in a temperature lower than that which is required to have TiC and TiN by the conventional CVD.

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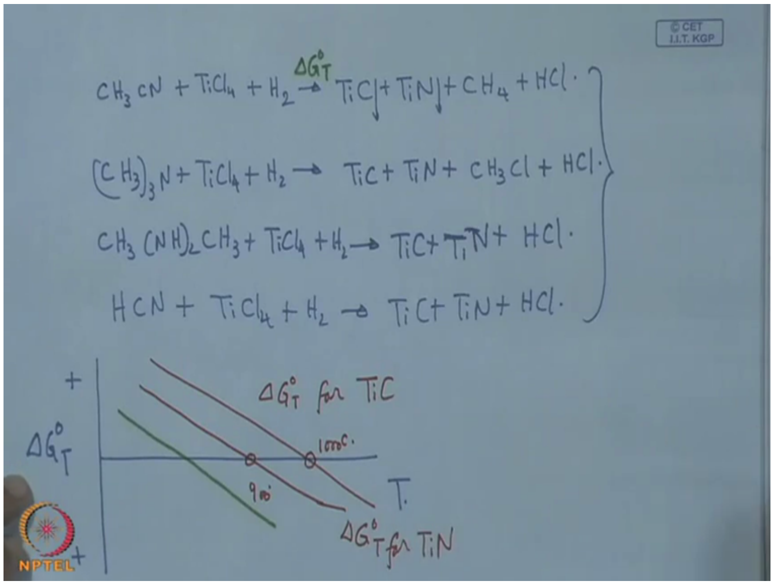

So if we follow the reaction, then acetonitrile, that means CH3CN plus what we have TiCl4 plus H2, that gives us TiC plus TiN, CH4 and HCL. This is one mode of the reaction which can be carried in the forward direction and we get simultaneously TiC and TiN and with some byproduct of CH4 and HCL. So this is acetonitrile. We can also look for the possibility of using trimethylamine.

So in this case we can also write and we have to put TiCl4 with hydrogen and that gives us TiC, TiN. But it has been found out and proved that it is, it leads to production of methyl chloride and also HCL. Then we have dimethylhydrazine and this is exactly NH2 which can straightway participate in this chemical reaction, plus H2 and this case we have TiC plus TiN plus HCL and the last one which can be also a good candidate for this chemical reaction, so this gives us straightforward TiC plus TiN and HCL.

So we can see that at least there are four chemical routes which can lead to formation of TiC, TiN and exact requirement will be served by this Ellingham diagram. That means if we see here this one, so this is temperature delta G0T, that means at the standard state plus and minus. Now here we can put this one say for normal change of free energy for reaction leading to TiC. Then we have another one which is also delta G0T for the reaction TiN.

Just now we have concluded and here we have around 1,000 that is the threshold. And this is around 900. Now if we put them together, we can find out that this is shifted quite remarkably on this direction and exact line that will depend upon the delta G0T and that will be, that will depend on respective enthalpy of formation and entropy of formation. And that will be guided by the governing equation.

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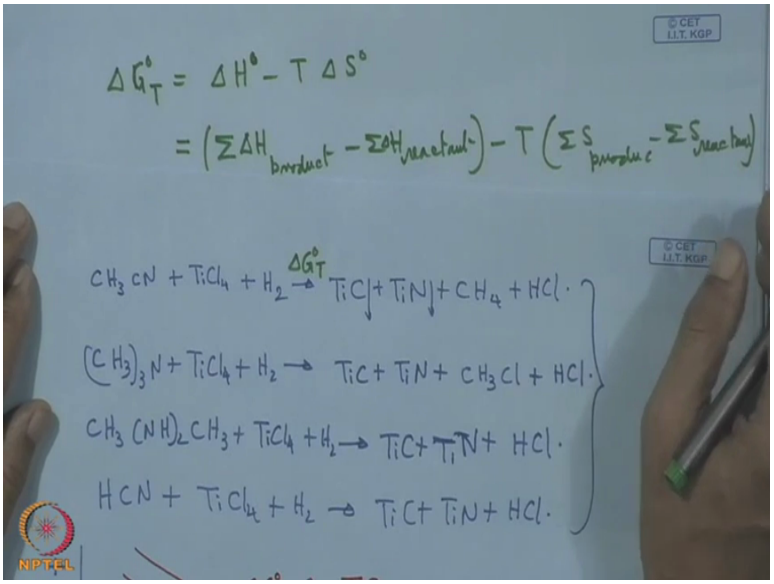

Say, what we can write here, governing equation means this delta G0T, that is actually delta H0 minus T into delta S. Now here this should be the actually summation of delta H product minus delta H reactant minus T this sigma S product minus sigma S reactant. So one has to really using two, here these terms for product, taken all the product, putting it here, taken all the reactants and putting in this second place of this parenthesis and then respective value of entropy of the reactant and product, one should be able to find out that what is the threshold temperature.

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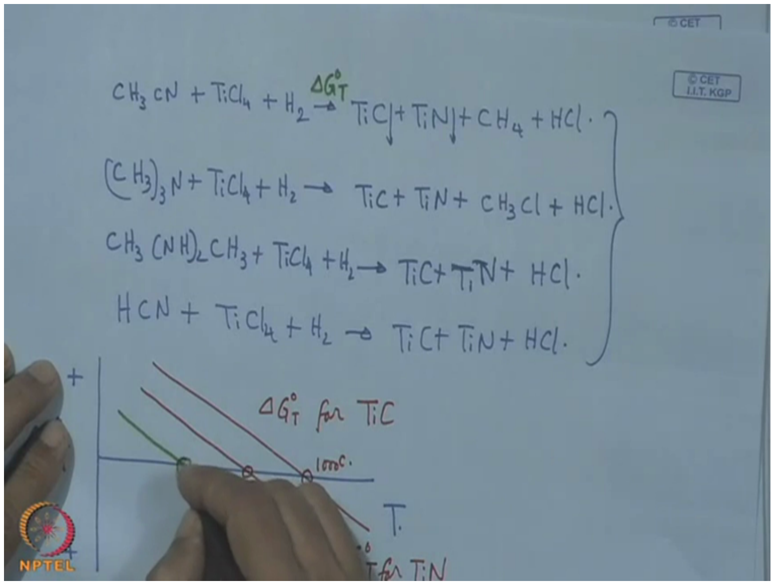

Because at the threshold temperature means this is the point. So this is the point.

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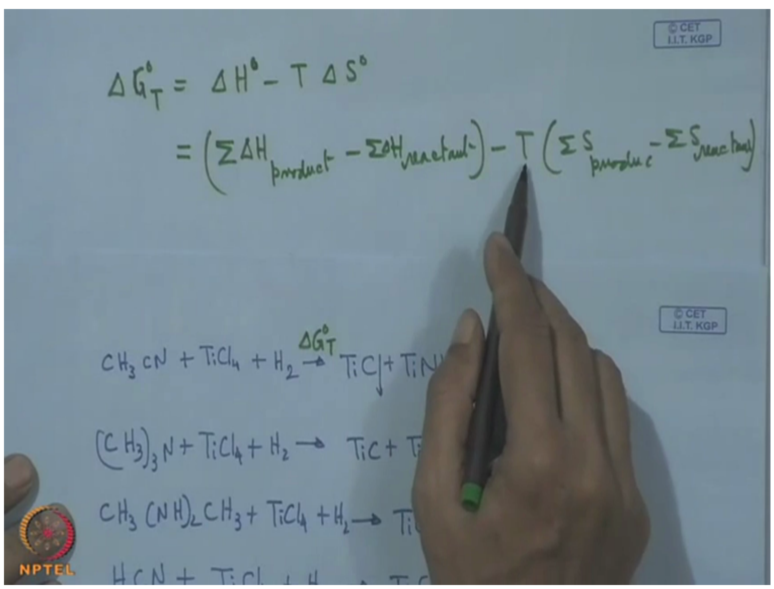

And for this if we put it to 0, then from this thermodynamic property T can be found out.

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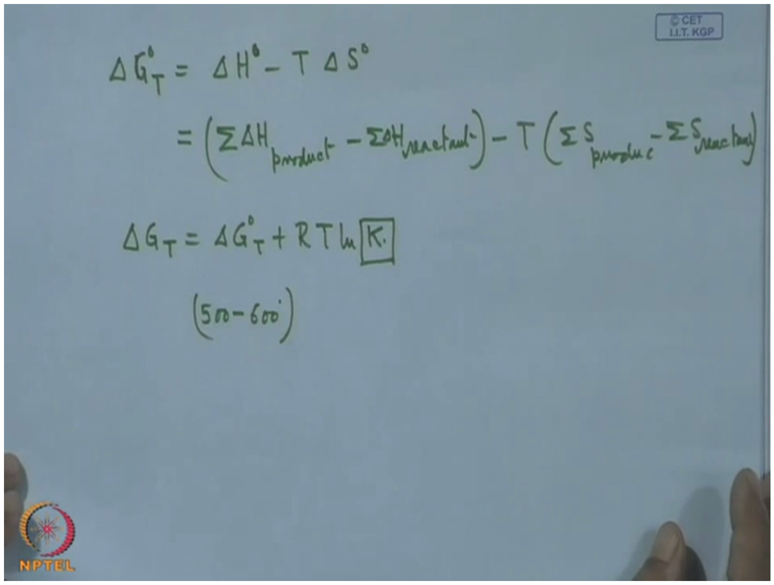

However if the reaction is conducted at a pressure other than standard atmospheric pressure, then we can write further to this. So this is actually at the particular pressure in question where the CVD is conducted, is equal to delta G0T plus RT into ln K where K is the equilibrium constant. So this has to be taken care of considering the stoichiometric coefficients and respective thermodynamic property. And with that, we can find out what is the temperature.

And as per the documentation it is confirmed, it is reported that around 500 to 600 all these reactions which are explained here by these four equation that can take place within this temperature of 500 to 600 and which will be not only of academic interest but will be of theoretical or industrial importance.

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So this way we can see that this use of organic source material which are supplier of carbon and nitrogen instead of conventionally chosen methane or acetylene for carbon and molecular nitrogen or methane for nitrogen. Instead of that if we use acetonitrile, trimethylamine, dimethylhydrazine or hydrogen cyanide, there is ample opportunity of conducting the same CVD and this can be done well within 500 to 600 degree centigrade which will be of immediate interest. In that, it will be an useful technology process for depositing this material in the wide range of steel family starting from HSS, die steel, tool steel and many other, light alloy steel.

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So CVD reaction with organic C-N compound that has been narrated and here we find out the role of this delta G of the reactant which is an organic compound of carbon and nitrogen and its value is so strategic and it can have a real overriding influence in bringing down the temperature required for this CVD reaction.

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Now factor influencing the growth rate of TiCN coating now naturally one would be interested to know what are those factors because there are certain operational parameter once the CVD is established we have to control those operational parameters. And those parameter can be summarized as follows.

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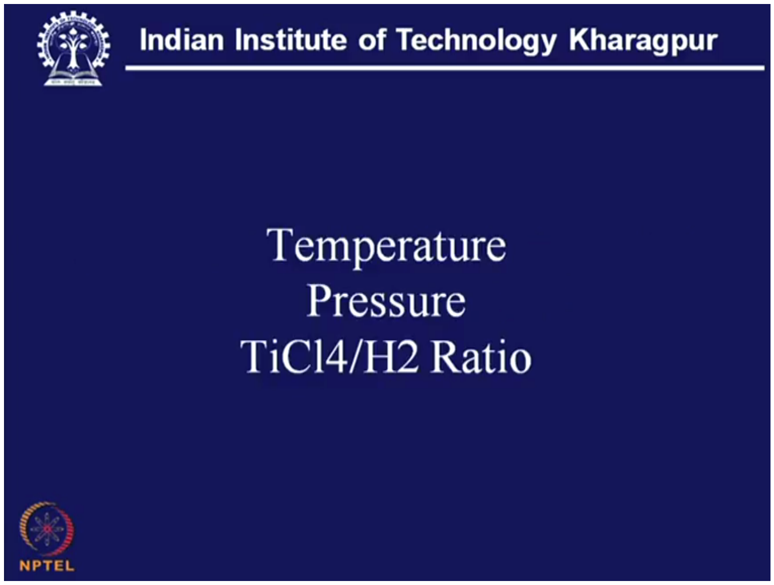

Number one, immediate, of immediate attention is temperature. Then comes the process pressure and finally it is TiCl4 to hydrogen ratio.

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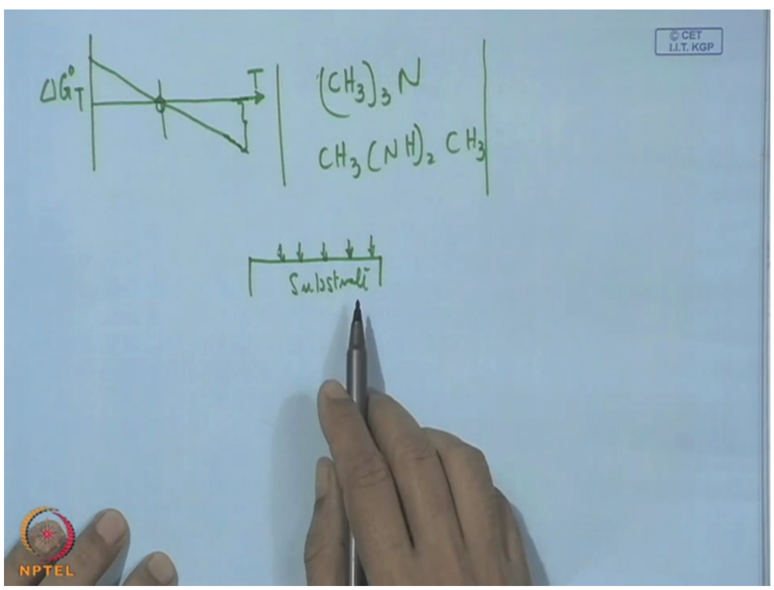

Now we understand that temperature, the role of temperature can be well understood because the graph of delta G variation with temperature, we understand that this reaction is not spontaneous and we have to really cross this threshold value to have a substantial negative difference so that the reaction can proceed in the forward direction and it becomes really a product favored reaction.

However it is interesting to know from the research result that particularly this is true for trimethylamine and this dimethylhydrazine. If we go beyond certain temperature, of course here the one interesting point is to keep the temperature as low as possible. But if somebody is interested in higher growth rate, and to use this process for certain substrate which can allow little rise of the temperature, then he can elevate this temperature and the reaction can go at a faster rate. So that provision is already there.

However if we cross that limit, what can happen that in that case instead of heterogeneous reaction, we may end up with homogenous reaction. One precondition of CVD is that for all these coating process is that coating should be synthesized on this substrate. Whatever may be the reaction, the reaction should take place on the substrate surface and the product should also synthesized its nucleation growth and lateral growth, vertical growth, everything should takes place on this solid surface and it should not be a homogeneous reaction.

But as we have mentioned that if we go to that temperature that may be counterproductive in that, that reaction may not, no longer be heterogeneous but it becomes homogeneous. And then the whole purpose of CVD is lost because in that case in the gas phase reaction takes place and we get dust, powder, flakes of titanium carbo-nitride instead of a coating, a uniform well-adherent, well-coherent coating on the substrate surface. So the temperature rise should be also considered with that particular care.

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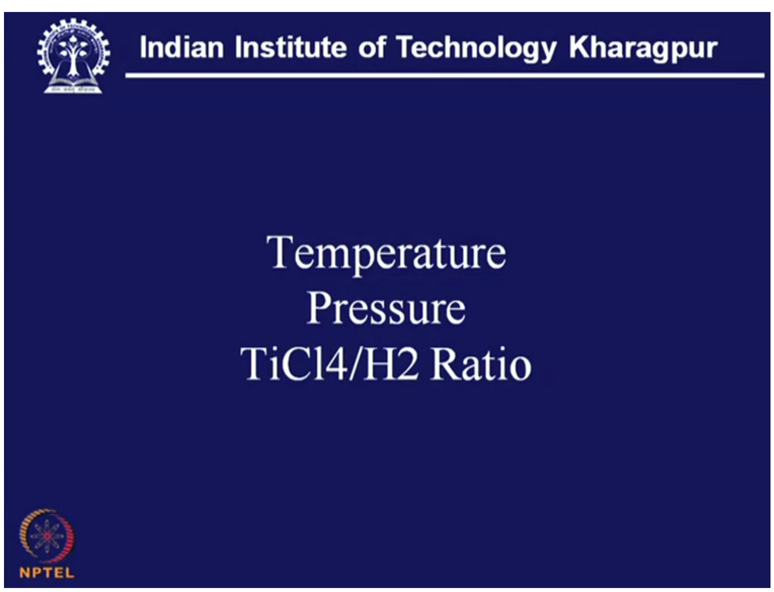

Then comes the pressure. Now definitely pressure has a role.

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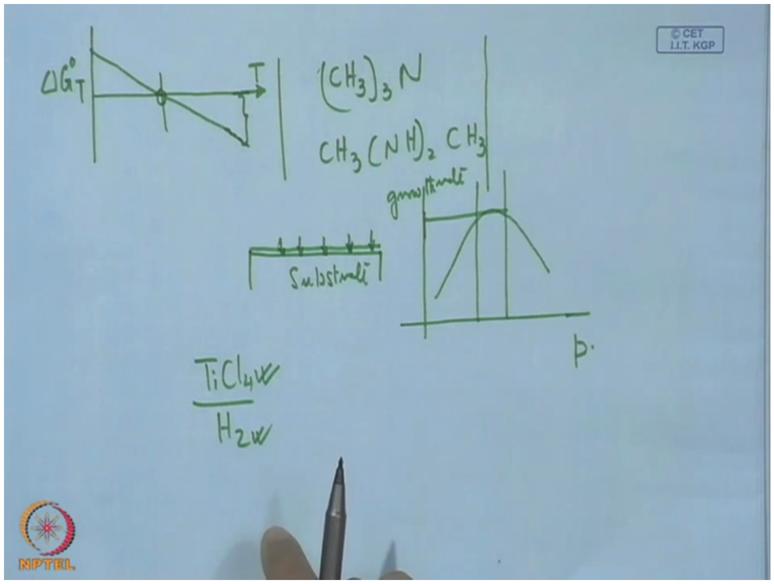

Now with increase of pressure, it has been found that the growth rate increases with pressure. Now this can be something like this. This is a typical nature, it is typical nature of any synthesis process. Now this is actually a point which gives the maximum yield. And on this side, if we have a low pressure, high stream velocity on this side and this is pressure and that is the growth rate. Growth rate of the coating, it can be milligram per hour or it can be micron per hour, increase of mass or increase of the thickness. However if we go beyond that point, what is going to happen?

Because of this high pressure, then mean free path will be shorter and that will invite collision and reaction in the gas phase. So in that case not only the growth rate will fall, that means there will be no reaction on this surface but reaction will take place in the space above the substrate surface and the coating growth rate will fall. Then comes this TiCl4, H2 ratio, this is already known that if we like to increase supply of titanium by increasing titanium tetrachloride mass flow, then proportionally H2 has to be also increased.

If it is not done, then we may have excess of HCL and this HCL may revert the reaction and then the reaction instead of being product favored it becomes reaction favored. So there also we have to pay attention that how to increase the TiCl4 with the increase of H2 if we like to have enhancement of the growth rate of this coating.

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Now coating composition one thing will be very important and interesting here is that TiCN.

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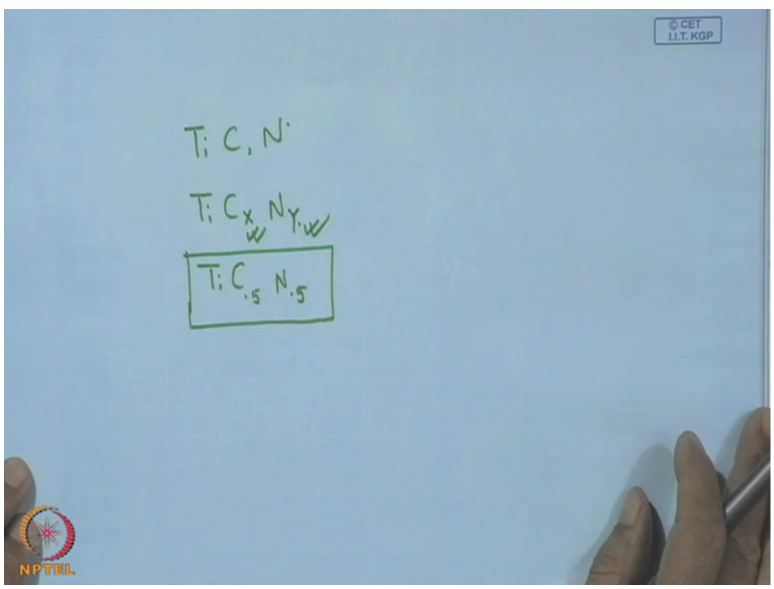

TiCN, now more generally we can express this as TiCNy. Now for all practical purpose, it is also the experience that if we have like 0.5 and 0.5, it is some kind of a equal blending of carbon and nitrogen in the lattice side with titanium. However this percentage of this 1 is to 1 of carbon to nitrogen that also depends upon the basic composition of the carbon, nitrogen source. That means those organic materials which are used for deposition of this carbo-nitride coating, there we have to look in what is basic composition of carbon and nitrogen.

And that basic ratio of carbon and nitrogen which is present in the source material, that will also influence the final outcome here in the value of x and y. So this point can be taken into consideration in selection of the source material apart from the fundamental consideration or most important consideration is the temperature, threshold temperature where we can initiate the reaction and that temperature is obviously of immediate interest considering the temperature sensitiveness of various substrate.

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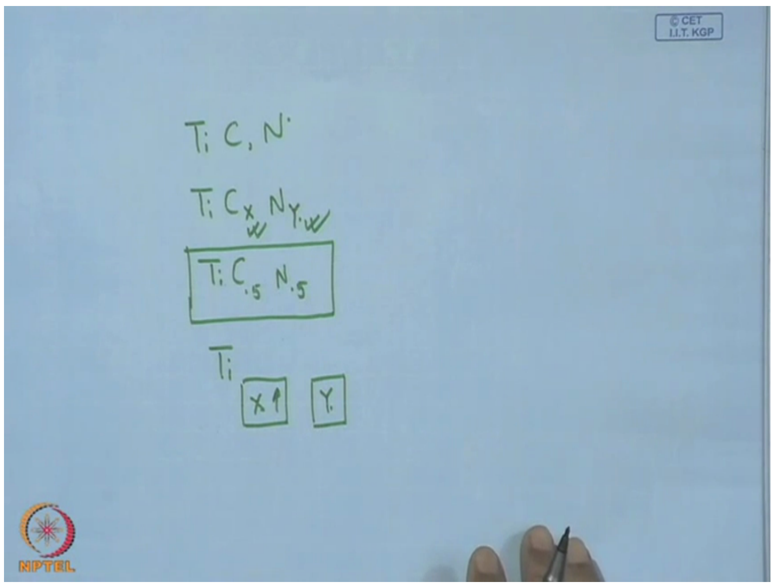

Another thing is also in our observation that if the temperature is raised little bit in the CVD process, then the percentage of this x, x goes up in comparison to percentage of y. That means that high temperature promotes formation of TiC, more carbon and in proportion to nitrogen and as a result we have a titanium carbo-nitride where it will be more than 50 and that will be less than 50. And this is also another experience that with slight increase of temperature of deposition, hardness of the coating also increases.

And this can be explained by the concentration of, higher concentration of carbon in this coating because titanium carbide is basically harder than that of titanium nitride. Now pressure can also have some influence. But with rise of pressure what is the experience that coating becomes porous, less adherent and in that the influence of pressure should be also considered if one has to make a good quality on the coating in terms of high density, good adhesion and adequate hardness of the coating.

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Now this is very important aspect of this coating, that means the performance of the coating. The performance of the coating depends upon number one, wear resistance of the coating.

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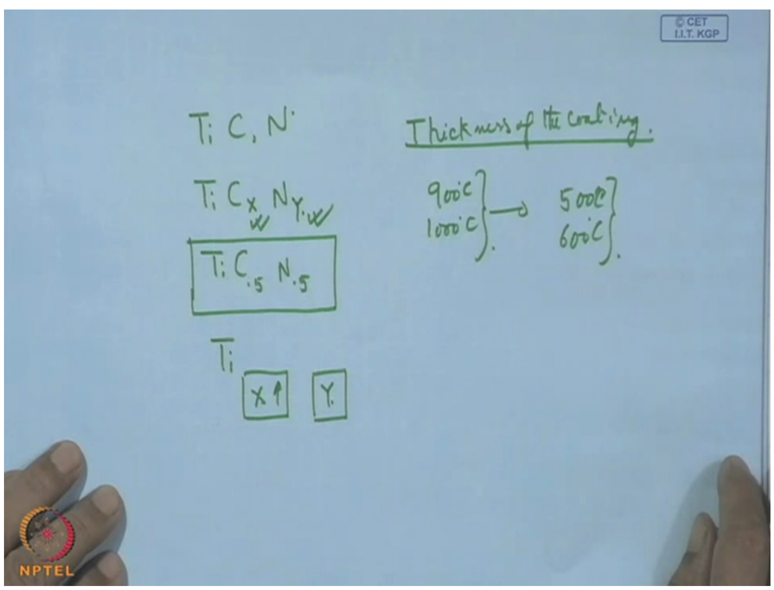

Now here one issue can be raised that what is the thickness of the coating. Thickness of the coating, this is very important issue because of the simple reason in conventional CVD using the temperature in the range of 900 to 1,000, our experience is that hardly we can keep the coating more than 5 micron, hardly it goes to 6 to 10 micron. The reason is as follows:

That coating is conducted, deposition is conducted at a very high temperature. And when it is brought down from high temperature to the room temperature because of this delta T, high value of delta T, residual stress generated at the interface is quite high, and there is every chance of separation of the coating from the substrate and failure of the interface. It is not the cohesion, not the density of the coating but it is just the separation at the interface because of this high stress buildup. And that is why we have to keep it within that range.

But here the situation is little different because of the simple reason that this deposition now can be conducted in the range of 500 to 600 degree centigrade. So this gives us a clear edge or a leverage of increasing the coating thickness. By our common sense, we understand if we have a thicker layer, obviously life of the coating will be longer. And if it is a thin coating, life of the coating will be also shorter.

So thicker coating means high performance coating provided it can be retained with adequate adhesion with the substrate and there this high temperature is the greatest obstacle. But for this moderate temperature coating, that obstacle is clearly removed. So one can very reasonably make an attempt to look how this thickness of the coating and the life of the coating they are correlated.

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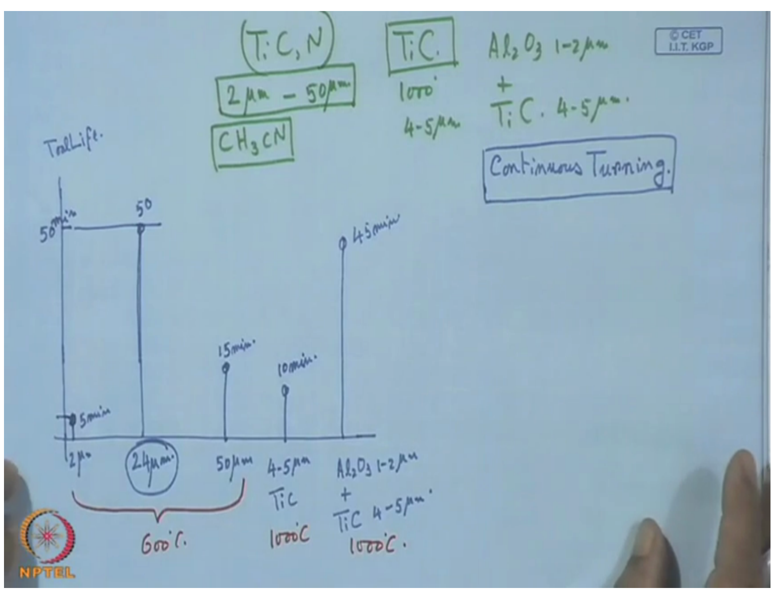

Now performance of the coating clearly means here that this coating of TiCN it has to compete with the conventional coating that means conventional TiC coating or conventional aluminum oxide plus TiC coating. Now this coating can be put in from a coating thickness of 2 micron to as high as 50 micron without any difficulty. At least after the deposition there was no such visible mark of failure at the interface from the visual inspection.

So the coating has been deposited in steps of 2 micron, from 2 to 50 micron. Now conventional TiC coating which we normally deposit at 1,000 degree that is 4 to 5 micron and this TiCN has been deposited using acetonitrile as the source of carbon and nitrogen. Now when it is aluminum oxide and TiC, that is a multilayer coating, we have here 1 to 2 micron and here also 4 to 5 micron.

Now if we now display the tool life versus thickness of the coating, some interesting observation can be made. Now this is the case of continuous turning, this is continuous turning. In a lead, it is a ductile material and industrial speeds, feed, depth of cut that has been chosen. And here if we put tool life, that is in minute. And here the coating, three coating, that means one is this TiCN with different thickness and then we have TiC and this multilayer coating.

So the plot is something like that. Let us try to illustrate. If this is a 50 minute tool life, we can have a thickness of this, this is about 24 micron. What does it mean? A coating of TiCN which is a product of this moderate temperature CVD and if it has a thickness of 24 micron, then it can give a life 50 minutes in continuous turning. Now if we have a coating of 2 micron, just 2 micron, this life will be very low if it is even less than 10 minutes. So these are the two things.

What is quite interesting, so it is even just 10 minute. But if we have a coating thickness of 50 micron, then of course the coating reveals its weakness, here the life is just, it is 50 minute, it is 15 minute. So this is about 5 minute, 2 micron. 24 micron give 50 minute and 50 micron give 15 minute. So we also find here that even in this coating which is deposited in the range of 600 degree centigrade, there also if we increase the thickness of the coating, then also we have this premature wear on the coating, fast wear on the coating, delamination of the coating and it is not just gradual natural wear of the coating.

But it should be flaking delamination and premature separation of the coating from this surface. That means with the thickness of the coating, the stress at the coating-substrate interface has gone up and that was responsible for early separation of the coating from the substrate and that leads to shorter life of the tool.

But when we put 4 to 5 micron coating of TiC, then its life is just 10 micron. 4 to 5 micron TiC and in that case it is just 10 minute. However if you put this multilayer coating of aluminum oxide, that means this is AL2O3 plus a bottom layer, this is top layer, that is the buffer layer in contact with the substrate. This is 1 to 2 micron and this is 4 to 5 micron. This is high temperature. So this is actually the low temperature coating. So this is actually the low temperature coating, this is moderate temperature at 600 degree centigrade. This is at 1000 degree centigrade, this is also at 1000 degree centigrade.

So what we find, this is around 45 minute. So what is interesting thing that a coating of 1 to 2 micron aluminum oxide with a buffer of TiC 4 to 5 micron, it can give a service life of very close to 45 minutes. Whereas a coating of TiCN which is less wear resistant than aluminum oxide, which can give, offer a service life of 50 minute and this is possible because of the larger thickness of the coating. And this larger thickness of the coating means actually here it offers wear resistance over a large thickness and as a result the duration of the coating it lasted to 50 minutes.

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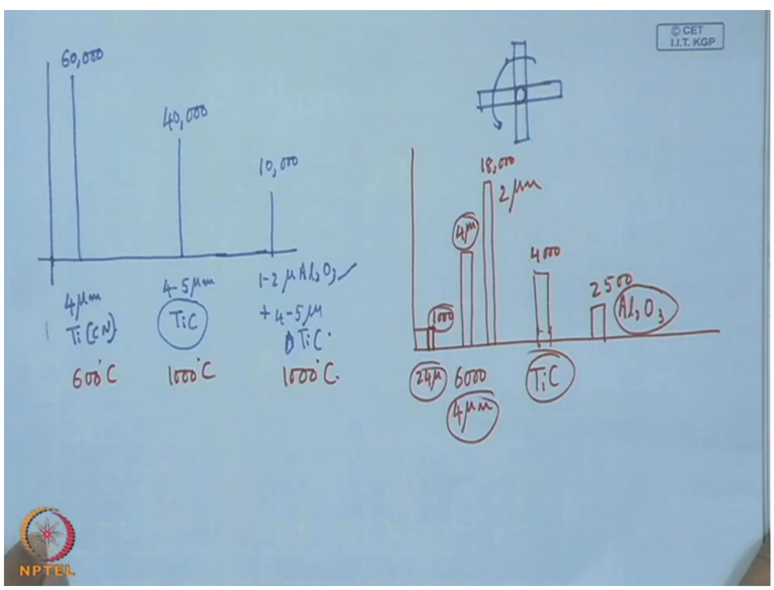

Now when it is interrupted cutting, we can see another interesting picture, it is an interrupted cutting, that means it is just like a cross shaped job which is allowed to rotate and it gives an impact on this surface of the tool. And in this case what we find that this 4 micron thick TiCN coating and then we have 4 to 5 micron TiC coating and then 1 to 2 micron AL2O3 plus 4 to 5 micron TiC. And if we see their performance, number of heats it receives, here it can go up to straightforward 60,000. This can go up to 40,000. These are some indicative figure.

And this is just 10,000. That means what happens? This is a test of toughness of the coating. Now you can find out that since it is aluminum oxide, naturally toughness will be less than that of TiC. And that is why we find that this can receive 40,000 impacts and then only it shows the fracture or edge chipping. Whereas this 4 micron of equal thickness which can go straight to 4 micron and in this case it can receive shock up to 60,000 times, 60,000 impacts it can receive before it shows some sign of edge chipping.

So from this it is also evident that this coating which is a low temperature coating deposited at 600 degree centigrade, this is 1,000 and this is 1,000 and which are comparable in terms of thickness. But this has a clear edge over this high temperature coating and it shows its strength in machining situation, in machining environment where the material has some non-symmetry in its geometry and while rotating it can give some kind of interruption in cut and the coating with the cutting edge can survive more number of impacts compared to the conventional high temperature coating.

Another figure, performance figure can be also shown here. And this is simply okay, this is simply, it is a, it is in the milling where we have 6,000. It is a fly milling, with one milling insert we can also conduct this test. And here when it is 2 micron thickness, it can go to 18,000 impact. But when it is 4 micron, it will receive only 6,000 impact. And then when it is 24 micron thick, it can receive only 1,000 impact.

But when it is an aluminum titanium carbide coating, this is 4 micron, and this is TiC, that is also 4,000 impact. And when it is aluminum oxide, it is actually 2,500 impact. So this is high temperature coating and this is a low temperature coating of equivalent thickness, but when these inserts are used in milling action with a fly milling mode, then also this low temperature coating shows its strength.

And this is at 4 micron but when it is 2 micron, it is 18,000. But when it is 24 micron, it is very low to 1,000. So from this performance test also we can see that the strength or durability or capability of this coating is much better than the conventional coating and it is going to be one of the best candidate for all sort of machining application whether it is continuous cut or interrupted cut or in also milling operation.

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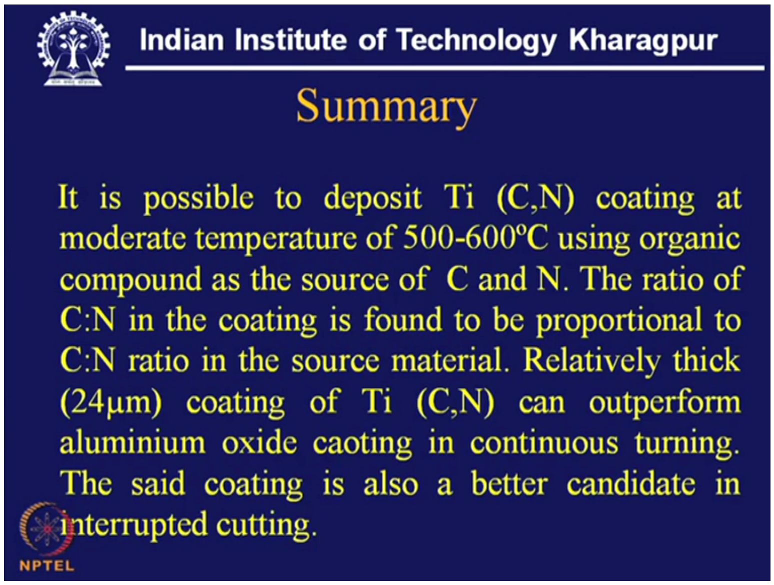

So we can summarize this discussion. In that, it is possible to deposit titanium carbo-nitride coating at moderate temperature of 500 to 600 using organic compound as the source of carbon and nitrogen. The ratio of carbon to nitrogen in the coating is found to be proportional to C:N ratio in the source material. Relatively thick coating of titanium carbo-nitride can outperform aluminum oxide coating in continuous turning. The said coating is also a better candidate in interrupted cutting.