Technology of Surface Coating Professor A.K Chattopadhyay Department of Mechanical Engineering Indian Institute of Technology Kharagpur Lecture 30 Special Techniques of Surface Layering and Surface Coating

(Refer Slide Time: 0:29)



Special techniques of surface layering and surface coating, now here we like to discuss this particular issue that there can be various coating techniques and processes but ultimately the objective is to come up with some utility product or utility tool which can be of some engineering use and with that objective we have different processes which can be say from CVD, PVD it can be wetting process, it can be even spray process or it can be hybrid process that means combining many of those and finally the goal of each coating technology is to find out one of the best process and one of the best coating material it can be one of phase it can be multiphase, it can be even monolayer or multilayer.

It can be not even just a continuous coating, it can be discrete coating preferentially situated at certain sites on the substrate surface and here again the whole idea would be to make the surface as one of the functional surface and as we know this mechanical engineering in this domain we have one of the widest use of the surface coating technology. (Refer Slide Time: 2:14)



Now let us look that what is this monolayering with super abrasive grit by precision brazing. Now we know that coating by wetting process, now in this coating by wetting process what we do? One layer of metal that gets attached to the substrate surface and in that we can also just not the metal but some of those non-metals say for example some abrasive particle can be also attached in this particle can be of wide use and very useful as a say tool of manufacturing.

It can be say for example as we have said here with super abrasive grit by, this super abrasive grit means these will be used for some metalworking or metal removal. Now here what we can do very interestingly and in an intelligent way?

(Refer Slide Time: 3:21)



Now ordinarily what we have a substrate and then we put one layer of this braze alloy and over that we put the grits and this is just a temporary binding, temporary attachment and later just by wetting and bonding these grits are anchored and wanted to the substrate, so this is already known and discussed but here what we like to show? Something as a regular technology for making such products and in a very reliable manner.

So what would be the idea in this case? The idea would be to have this braze alloy just not in the form of a paste but it should be an innovative technology is that we must have a strip, it is just like a tape, tape of the braze alloy and this tape means it maybe a suitable plastic bonded tape. So it is actually braze alloy in the form of a tape and it can be an adhesive tape too. So in this adhesive tape we have an adhesive material on both the surfaces, this is one surface and this is one surface.

So this has to be prepared, so this braze alloy when it is plastic bonded with plastic then this plastic should not interfere with the braze alloy during the melting and when it is become it is becoming a almost like a liquid this plastic should not interfere and this plastic also should not be a source of oxidation. So it should not affect or the wetting characteristics of this alloy.

So there this good deal of chemistry should be taken into consideration, good deal of chemistry is involved. So with this knowledge of chemistry we must have a right plastic binder which can be used, now this is from the side. Now from the top what we can see? So this is the width of the tape and what we can show here? That these grits can have some kind of setting, regular setting and we should be able to change this setting.

That means if this is say for example a cylindrical surface and our idea would be it is going to be one of the manufacturing technology and here this tape will be wrapped over the surface with this temporary binder and then what we have? We have this braze, plastic bonded braze alloy and then the top temporary binder, so the whole idea here is to just not to sprinkle this material but with screen-print technology we can have regular distribution and what is important?

Since this is going to be an abrasive tool then this will be the direction of movement motion, so that means the abrasive action will be done in this direction. So what we can have here? 2 parameters this is called the longitudinal pitch and this is called a transverse pitch, so this PL and PT these are the 2 parameters for this tool is because of the simple reason that this value of PT that will determine what will be the interference in the cut of each grit that means overlap.

How much is the overlap? Because this overlap is also necessary to increase the surface finish of the work surface, this abrasive tool is going to remove material and in that case that surface these grits have the abrasive action and this PL that is important from the point of view of chip clearance that means the chip which is removed there must be some adequate chip length, so that chip blogging does not take place and it will be wrapped over this surface just like these grits.

So this is something though it is not a continuous coating but it is actually a monolayer of abrasive grit and this is going to be used, so this PL and PT these are 2 parameters and with the screen print technology it should be possible.

(Refer Slide Time: 9:38)



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Now further to this even though we have this step with this step, what we can do additionally? That let us look, additionally what we can do? This is the tape, so this is the direction of movement, now what we can do? We can also put these grits, here we have seen here we have the rows, this is one row, this is the intermediate row this is another row but we can put in a helical pattern.

So that means these are the part of Helix, so this way also be can put oblique to this surface. So here what is going to happen? So this is one of the grit, so this is actually arranged in an oblique manner. So this is called the helix angle Theta, so what is the advantage by this orientation? Now here we can see that each grit has its cutting action in this direction. So each grit has its cutting action but here what we can see?

In the direction of motion we get more number of lines that means just by helping this angle of obliquity we have so many grits just not along this Helix line but they are little oblique to this line, Helix line, line of Helix and that is why we have for each cutting point more number of scratch marks.

(Refer Slide Time: 11:42)



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So compared to this one compared to this one we get here we get just 3 lines but here we get more number of lines with the same grit density and with that what we can achieve? We get an improved surface finish apart from free cutting action. So here what we have to have? We have the longitudinal pitch, the transverse pitch and also this value of Theta. So with this it is expected that such orientation we can have a better finish but the whole thing is centered around this that here we must have a process of getting such tape with temporary adhesive.

So that with this screen print technique we can have different pattern of the grit distribution, so it can be small size it can be larger size we can adjust the value of PL, we can adjust the value of PT that that is from the (()) (12:53-12:55) which is a part of this screen printing technique. So with that screen by changing the size by changing the orientation we can have this kind of arrangement but what is important that is tape which is coming in the form of a plastic bonded that should be useful and then fixing of the grit by wetting this brazing can be done effectively and this is going to be one of the most effective technology using the wetting property of the brazing alloy for easily, conveniently fixing those grit and in that case we need not have a randomly distributed pattern.

(Refer Slide Time: 14:01)



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So in that case the whole purpose of this tool that is seriously affected because you are the whole idea is to have one layer formation because we have said here it is a monolayering with it is a layering. So basically it is just a layering it is not just few micron coating but it is just a layer of abrasive bonded and if we can achieve this thing that will be a great leverage in the manufacturing of one of the was promising tool.

(Refer Slide Time: 14:26)



Now comes micro-diamond abrasive tool by CVD. So we know CVD technology for diamond, so this is hot filament CVD we can also have this DC plasma arc that can be also used but here what is important? It is not a continuous coating. Normally diamond coating

which means it is a substitute for half volley crystalline diamond which is the outcome of this HPHT technique that means high pressure high-temperature technology.

So that is a substitute for HPHT technology that is actually CVD diamond, so it is going to be a continuous coating that we have already seen. However it has been also noted unlike other CVD processes for say Titanium Carbide, Titanium Nitride or even Aluminium Oxide in diamond CVD technology we have seen the surfaces in comparison to other coating it is rough, so it has to be smoothen to get all sort of quality and the job then the adhesion problem premature removal of the coating, all these things can be minimized to great extent just by getting a smooth surface, so there is one of the problem of the issue.

(Refer Slide Time: 16:25)



But here we like to use this roughness of the diamond for another purpose and that is going to be one of the affective manufacturing technologies for abrasive tool. So what is done here? In fact if we see this is one just a flat surface it can be small rather we should use it for Micro tool for all sort of micro-machining, Micro working this micro-diamond abrasive tool. So let us even see consider schematically this is the receptor surface and where we like to have this deposition of diamond.

Now this surface has to be activated or passivated. So we know that for diamond it is not ordinarily the surface is active, so there are lot of steps before one can start the CVD that means this substrate preparation becomes one of the important steps before conducting this CVD. So we have 2 choices either activation of the surface or passivation of the surface. Now what we have seen?

(Refer Slide Time: 17:31)



What we like to have? It is micro-diamond abrasive tool, so we like to grow diamond in some discreet location not everywhere, some of the selected spot and that is selected spot can follow a pattern some array some chosen array, what? This diamond will be nucleated and will keep on growing, so that can be one of the innovative technology and soul that can be pursued just like the previous one where it will be more important to get this plastic bonded tape that is the layer of the braze alloy.

Here also what we like to, so there we need a bonding material but here what we like to have on a substrate, this is a substrate. On the substrate the diamond will keep on growing say we fix some of those points which are going to be the activation sites, so that itself will become a technology that means say on these processes, so these are the areas where diamond growth or nucleation that means first of all absorption of the reactive spaces that dwelling or residing on that place and then reaction that is the third step and then we have nucleation of diamond which is going to nucleated and then it will grow in volume.

So this can be some of those sites where we can have this diamond. So these are the sites, so this way one can also look for some technology, so this is going to be one diamond site and these diamonds are just not diamond of coating but these crystals are individually grown just like this crystal and this will become a self bonded, so we do not need any bonding material it need not to be neither it is braze alloy or galvanic nickel. So it is just on this substrate surface.

Now this substrate surface obviously that is a surface its chemistry is very important because it should be pointed automatically by natural bonding, so that the surface must be from that point of view from chemistry point of view it should be Carbide former it should have equal very close matching of thermal coefficient of expansion E value should not be too very, so if we can satisfy those things then it will be also another promising technology for using the CVD process.

But not for getting a continuous coating but a coating over the substrate surface and with some selective growth, so these are the surfaces, so to say these are the nucleation sites or nucleation centres where we have this growth of this diamond and here as usual we can keep this longitudinal pitch we can also keep transverse pitch and here this is going to be a tool. So this diamond can be grown, so growth of the diamond that is in the hand of the operator of the CVD.

So how long this CVD process is going to be continued? So it can be a small grown diamond and that can be even say 50 micron or 30 micron per hundred micron it depends upon the requirement that in that case that is really going to be a Micro tool for all micro-application particularly micro-machining of very sensitive material for delicate material where utmost care has to be taken for slotting, cutting, drilling and this can be one upcoming technology for constructing for manufacture of a single layer diamond tool for all this micro-processing operation.

(Refer Slide Time: 22:24)



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Now comes this PACVD, plasma assisted CVD. Now what happens? Suppose we have one reactive spaces where this is going to be the metal donor and this is going to be the (()) (22:48), so that need to be decomposed but when we have this may be thermal decomposition but when we have some electron there, so we can also write A plus X plus e, so it is actually this what we call that coalitional disassociation that means inside the CVD chamber we must have sufficient electron and during its course of movement towards the anode it may interact with the neutral molecules of the gases and thereby finally this gases will be ionised and in this ionic state their reactivity will be much more stronger, vigorous and that will be facilitated because of this ionic state.

(Refer Slide Time: 24:04)



So the whole idea here is to have sufficient quantity of electron and for that what is done? In a CVD chamber maybe we have a substrate and then we have one cathode and there will be one anode, so this substrate can be one anode or we can have an additional anode adjacent to this one. So this is actually cathode, this is the substrate, so this will be polarised it can be RF also.

So this is cathode, now here we have the entry of this gas and as a result of this will be ionised and this reaction will be stronger. Sometimes it is also possible, so what is the advantage? Low-temperature, so the reaction which may take place otherwise if it is say for example 800 degrees for thermal decomposition. Normally by thermal decomposition but with this plasma activation, with this e we may have this thing done in 400 to 300 degree centigrade so that is the greatest advantage and then definitely temperature sensitive material can be used as substrate and apart from that if we can also polarise this thing with this negative polarisation of the substrate what is done?

In routine PVD, in routine PVD that means sputtering then ion plating biased evaporation, all sort of biased sputtering we routinely use substrate bias and that is only to have impingement by this positive recharge particle ion, so the whole idea here to have some improvement in the morphology of the coating and also on the substrate adhesion. So cohesion adhesion these problems if we like to properly address then this attraction of this positive ions we must attract this positive ions towards this substrate surface and that is why substrate is biased.

So even with this CVD it is also possible to have this substrate biasing and in that case this substrate will be there will be impingement with this ion of course the negative polarity has to be properly chosen. So that there should not be resputtering of the entire material. However the whole material will be processed in that its densification then minimization of the porosity induction of some compressive stress, elimination of the tensile stress.

And last but not the least a good interface say at the very beginning of coating where 2 materials from 2 sides are going to make one interface that is the most critical stage in the whole development. So during that process if we have in a controlled manner even in this CVD process if we have a controlled manner ion impingement in that case definitely there will be a game in the quality of the coating. So this is exactly PACVD what we have seen.

(Refer Slide Time: 28:02)



Now this is also very important PVD of cBN. We know that diamond and cBN these are called super hard material. Now we know that diamond is already coating low-pressure synthesis of diamond that is already one established technology there are at least 3-4 technologies for doing that thing but looking at diamond one would be also interested in getting a CVD coating.

(Refer Slide Time: 28:43)



Normally the CBN though it is man-made what we get as PcBN poly crystalline cBN which comes like a lamina which comes like a lamina of about 0.5 millimetre thick layer by using the particle of cBN and by compaction with a high pressure and high-temperature we can get

these laminates of PcBN that is poly crystalline it is not a single crystal, so this we call high pressure high-temperature technology but it is just having some limitations just like PCD poly crystalline diamond.

So that is why from PCD to we have CVD of diamond, similarly there is a logic that why cannot we go from PcBN to CVD or CVD of cBN but before that CVD there has been lot of attempts to use this PVD of cBN that means the PVD technology that is already known, so here the whole idea is if this is the target and there we have the substrate, so this is substrate this is a target and this target is actually made of hBN hexagonal Boron Nitride.

So it is made of hBN, so with hBN it is possible to have this target but what we get? During this process hBN will be sputtered but this is also, so this is a, this is actually RF this is also going to be one RF radio frequency that was the existing technology at that point. However sputtering is possible and by this ion impingement by this ion bombardment towards the substrate which is also negatively polarised, it was possible to have some sort of cubic formation but it was not a total success but more importantly the main problem was that in this BN it was not BN that means it is no more B by N is 1 is to 1 but it is less than 1.



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So it is actually BNX where X is less than 1, so what is to be done to take a rectification of this? What is done? That you have partial admittance of with this process, this is substrate and this is entry of Nitrogen and this Nitrogen may make up this deficiency, what we find as BNX, so the problem is that it was not a total conversion, partial conversion of this hBN to cBN and also it is deficient in Nitrogen.

So following this issue there had been another method of doing this thing that is actually a substrate is here and what we have? Here it is actually a crucible consisting of hBN it can be a rod or it can be powder of hBN and what we have here it is actually electron beam gun. So an electron beam gun, so this is actually EBG electron beam gun that is focused on this surface to have evaporation of hBN and it can be hBN or it can be even Boron.

So one way would be to have HBN but the idea here is to have just Boron because its melting point will be below that. So it can be even Boron in this, so it is Boron and at the same time we have entry of Nitrogen and this is exactly what we call ion plating, this ion plating and this is going to be reactive ion plating that means here we have Boron evaporation and admittance of N2.

So with that here of course just like the conventional ion plating we must also bring a probe a ring with a positively polarised. So that electrons will be drawn on this surface and there this will be also ionised this Nitrogen the whole result will be that we expect this Boron should go in this direction and it is vapor of Boron and we have Nitrogen. So it is going to be Boron Nitride and at the same time this is also going to be polarised it is AC and here we have grounded.

So with this ion impingement we expect this Boron Nitride in cubic form. So here it was much useful much effective then the process what we have just now described. So it is actually reactive ion plating of Boron Nitride and because of this impingement with high-energy this is going to be a cBN structure but it also has lot of I mean huge high magnitude compressive stress when it is deposited finally on the substrate.

So this is the substrate and that is why it has premature (()) (36:23). So because of this compressive stress it is very brittle, so will use point of view it was not that success. However it would have been possible to change this structure to this cubic form.

Now here comes, so far what we have described those are all PVD. So now first one is a hBN solid target and the second one is reactive evaporation this way also sputtering is also possible that means sputtering is possible just using Boron as the cathode and then admitting Nitrogen just like reactive sputtering with proper biasing of the substrate, in that case we expect conversion of this Boron and Nitrogen into Boron Nitride and with this high impingement that will assume a cubic structure.

(Refer Slide Time: 37:54)



Now what we can show here that is actually use of this plasma enhanced CVD of CVD of cBN. So what is done in this case? Is actually a substrate putting like this and this is a CVD chamber and we have admittance of gas and this and this is obvious we have all sort of whether we biasing wherever it is necessary plus vacuum pumping system on the side this vacuum system those are the peripheral elements of the system, peripheral support.

So here what is admitted it is actually a mixture of Diborane and Ammonia because in this case it is much more reactive compared to Nitrogen and you can have Hydrogen as the carrier gas. So with this expectedly, we expect here BN to form and then H2 and here of course we have to have Hydrogen as the carrier gas, so this BN that is going to be an hBN, so whatever is deposited that is hBN.

So this is hBN but important thing here is that now comes is most important thing is generation of ion and which will be attracted towards this surface that means inside we have to have a cathode. Now from this cathode secondary electron will emit and this secondary electron during it movement towards the anode it will be repelled by this cathode which is also charged negatively.

So in this case what is going to happen? It will oscillate and in the process it can take some of the neutrals liberating ion. So finally it is ion which is going to impinge on this surface, so it is ion impingement. So it is striking with ion and that is the high-energy, so with that high-energy it is possible to have this cBN structure over this coating even in the range of 300 to my 300 to 400 degree centigrade.

(Refer Slide Time: 41:53)



So that is the whole idea here that this ion will be high-energy ion, energetic ion because of this negative polarisation, so this is just like a biased substrate and this biased substrate will be impinged by this ion and that is why we can have a change in structure to cBN. So here it is unlike that of all PVD, so here we use this CVD reaction followed by ion bombardment on the substrate surface and then convert in these 2 cBN. So this is actually plasma assisted CVD of cBN what we have looked into.

(Refer Slide Time: 42:11)

HPH pressw -m B Props 300 4000

Now the idea is that this CVD coating, now we know that PCD diamond coating and this is low pressure. So this is actually low-pressure technology and this is high-pressure technology similarly if we consider PcBN that is a high-pressure technology HPHT but it has some limited use, very costly, very complicated and very limited use but from there we have also now that low-pressure synthesis of cBN.

So just like now what we have? We have just like TiC, TiN, Aluminium Oxide, Chromium Nitride all sort of coating which comes like a smooth coating on a substrate surface on an Carbide tool. So here also we can get even diamond coating just like diamond coating we can have a continuous coverage over the cutting tool insert and that can be well used for various cutting action particularly for those areas where normal tools, normal coating cannot be used.

Say machining of inconel, nimonic or machining of say heat treated material, high-strength material where cBN grit PcBN as a cutting tool, cBN grain in grinding wheel have shown their remarkable performance now with the available of coating we have the user has much more flexibility, so he can also choose this coating and this coating can be put in various complex geometry of the cutting tool.

So now we can have cutting tool even with complex geometry just like end mill cutters with this cBN coating which is the result of this low-pressure low-temperature synthesis and this can be handled in say around 300 to 400 degrees centigrade. So that is if essence the specialty of this process.



(Refer Slide Time: 44:42)

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Now Tungsten Disulphide coating, now this is actually a super lubricious coating WS2. Now recent time's people have lot of interest on WS2 because it is superior then MoS2 terms of its thermal stability and resistance to oxidation its performance is better than MoS2. However it is also our experience that when we use a target of WS2 for sputtering, direct sputtering it using say RF or pulse DC.

Now if we like to push the coating rate, deposition rates by increasing the WS2 cathode current then we encounter a problem of resputtering of Sulphur from the coating from the coated surface. So ultimately the coating what we receive that on the on the substrate surface that is deficient in sulphur, so it is Wx where x is less than 2.

Now the question is how far we can allow the value of X which is less than 2? Now here we have to see that this preferable (()) (46:13) plane which is 002 plane, if we have this plane parallel to this substrate surface then there is such no complication will arrive but one thing also we must take into consideration that if we have a too low value of X in that case this place will be occupied by oxygen and that will be counter-productive.

This coating will lose its all lubricious characteristics and which will be not used of much use in getting superlubricity which is a demand for various mechanical application using micromovements and dry lubrication. So the idea here is to have this WS2 where we have some deficiency in Sulphur to have. (Refer Slide Time: 47:27)



In to have admittance of, so this is substrate and here we have Sulphur that means in the form of H2S and that is admitted to have make-up this deficiency. So this is one way but another weight can be also done just by using Tungsten as the cathode and having H2S and this is reactive sputtering.

(Refer Slide Time: 48:21)



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Now this is one of the most upcoming coatings that mean here what we have? We have metrics of carbon, so it is a co-deposition of tungsten. So it is something like this, so it is Tungsten target and we have admittance of H2S and CH4 inside the substrate, inside this deposition chamber and this is the sputtering of the tungsten. So during the process what we have?

So we have this reaction with WC that will be one form then we can also have WS2. So it can be done this way that in a metrics of carbon we can have nano particles of Tungsten Carbide and Tungsten Disulphide and that can give required hardness from Tungsten Carbide and the super lubricious property from Tungsten Disulphide and that is well distributed with fine particle in a metrics of carbon which is amorphous.

So this is going to be one of the coatings with a good promise of giving one of the best performances in terms of hardness and also lubricity. So this is going to be one important hard lube coating for many applications. (Refer Slide Time: 50:30)



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Now comes one important thing super lubricious and super hard coating. Now this is also another attempt is made, so as we know in the normal diamond coating with a CVD diamond coating, the surface is not smooth as we get which CVD, TiC or TiN which are all conventional well established routine these are all routine coating, so these are rather smooth but diamond coating these are the crystals, one can clearly visualize those crystals and this gives a roughness.

So with that roughness there are 2 problems, one say during machining of Aluminium base alloy that Aluminium chip can get entrapped into those voids in between the crystals that means in the valleys number 1 and number 2 when this flank surface of this cutting tool which is not very smooth, it rubs over the surface of the work material though diamond is extremely inert but because of this high points it cannot give that expected surface roughness.

So what is the current attempt? That means over this diamond coating, coating of MoS2 or WS2 that can be deposited and that can feel that (()) (52:05-52:07) and giving a smooth surface, so that coating can be inert towards the material like Aluminium and that the same time this coating's get a very hard support it is because it is diamond. So with this lubricious coating with a hard support of diamond that can also be one of the good candidates I mean prospective candidate in many applications, mechanical applications mostly involving cutting tool.

(Refer Slide Time: 52:47)



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Now comes super Nitride coating using high ionisation sputtering and high ionisation pulsing, so it is actually Nitride coating which is indeed a very promising tool, coating that is given by this Titanium Aluminium Nitride. Now here comes the question what is the percentage? This is x and this is y and this is N. Now in fact if we look into the equilibrium diagram of TiN and TiAln then we see that there is a solubility gas, so they cannot be put into one club like that and that is normally they are not soluble to each other. So that is in the normal equilibrium state.

However thing is that if we can change the pressure temperature combination during the processing it can have a metastable state that means during the conversion from one stable state to more stable. Now if this is the path, so one stable position to more stable but if it is so that because of the processing condition it can go through one metastable state and it becomes stagnant there and further transformation or transition does not take place.

So this depends upon the particular coating condition but in fact it is the temperature and pressure with that it is possible to have a metastable state and that is quite stable in that the further movement to more stable state that direction that does not occurred. It is exactly that principle is used that means just by having the sputtering condition it is possible to have this kind of combination.

(Refer Slide Time: 55:44)

I.I.T. KGP (Ti.46 Al. 54) N BI Cubical. (Ti.35 Al.65) N Hexagonal.

Now for the moment what we find that a condition in which we have say Ti4.6 and Aluminium 0.54 Nitrogen and this is one combination but here it is important to have that this is more or less a B1 type cubical structure but if we like to improve this performance of this coating then one has to go for this (()) (56:16) structure hexagonal, hexagon will (()) (56:23) structure and that can be given by 0.35 and Aluminium 0.65 and to do this what we need?

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Cubical Hexagonal Callton 1-3 KW/Cut Pulseveli 50-150 Q1/2S 800-1500V

We need here this super Nitride and this coating in every respect harder than this one, denser than this one, resistance to oxidation that is better than this one. So this coating in every respect is superior coating compared to this one and this is also going to be a metastable state that means that is metastable compared to this one but we can keep this thing without any change and we can keep this with this proportion Titanium and Aluminium with Nitrogen by high ionisation sputtering or by high ionisation pulsing.

So in this case what we see? That this energy density is this cathode energy power density that is actually 1 to 3 kilowatt per centimetre square and the pulse rate, pulse width that is that is 50 to 150 microseconds and voltage, cathode voltage 80 to 150 volts.

(Refer Slide Time: 58:11)



So it is if we compare with a normal pulse DC, in a normal pulse DC what we have? We have say 10 milliseconds and here 20 milliseconds and in between what we have here? We have such a high ionisation sputtering with high energy and it is giving high impact but when it is normal pulse DC, here we have at least 20 pulses, 20 pulses over the surface, so that is the basic difference.

So this is conventional pulse DC and here what we have? This is actually another spike, so this spikes come with this period of rest and then it is active.

CET LLT. KGP Cubical 1:35 Hexagonal. Calture 1-3 KW/Cm² Pulsevuli 50-150 01/25. Voltage 800-1500V

(Refer Slide Time: 59:03)

So with this it is possible to have such kind of coating which has a much better property compared to conventional Titanium Aluminium Nitride and this is called the super Nitride coating.

(Refer Slide Time: 59:15)



So with this we can summarize the whole thing that means there are certain upcoming technology for layering or for having a coating, now the whole idea of all those modern and upcoming technology will be to improve the ultimate implement or useful product whether it is in metalworking or whether it is in wear part. Ultimately the whole idea here is to have a process which can be technologically easy to handle, easy to operate but at the same time the end product of this process or outcome of this process will be a high performing product.