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Course Title
Environmental Degradation of Materials

Module 01 Lecture 18
Broad Subject: Passivation and Mixed potential theory

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Let us continue passivation. In the passivation, the definition, it says that it's a condition of corrosion resistance because of formation of some surface film, which could be sulphate, or which could be oxide, or which could be hydroxide and that is under oxidizing condition and another point is with high anodic polarization. And we have seen the example of iron, when it is dipped in concentrated HNO_3, initially, it starts some bubble. There is some sort of bubble formation, but after that bubble formation stops, and there will be almost no reaction on the iron surface. Okay. That oxidizing condition it is clear because HNO_3 is a highly oxidizing acid.

Now we have to understand there is one more term which is basically the high resistance, high corrosion resistance that is because of large anodic polarization. Now when you consider this large anodic polarization, now let us consider this graph. Let's say this is my when polarization is zero. Now if you consider large anodic polarization, that means it should go to the positive side. This is positive N. So η would be very large, very large, and that time my anodic polarization line, which is given by η_a would be equal to β_a log of i_a by i_c, sorry, i_0, which is exchange current density of that particular reaction.

And if this is my log i axis, which is exchange current -- which is basically the current density, i_0 is exchange current density and i_a is the anodic current density and which gives you the idea of the dissolution rate. So if this η is very, very large, at the same time, if this is very large, it gives you that idea that i_a also should be very large. If this is large, this one also should be large if I go along this line.

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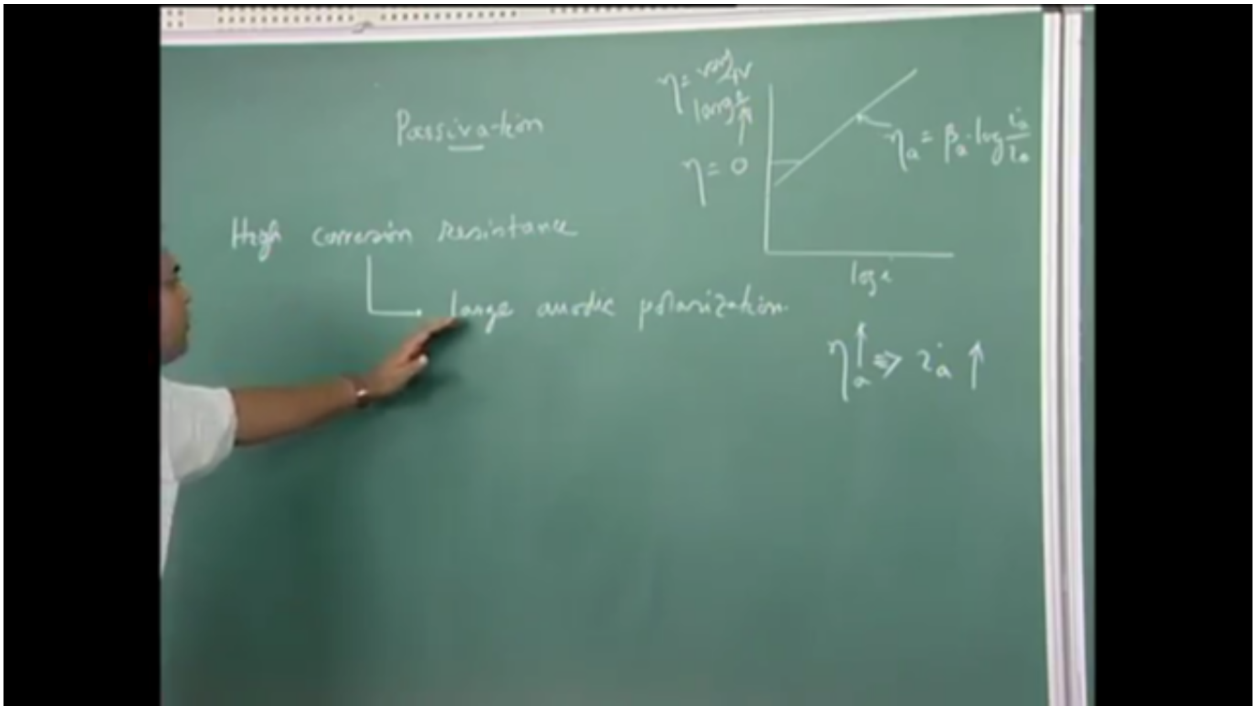

So that means if I go for large anodic polarization or large overvoltage, anodic overvoltage, in fact, I should get large current density for dissolution rate, for dissolution. So large current density for dissolution means large corrosion, but we still we get high corrosion resistance.

Now in order to understand this we need to actually perform the polarization test and that polarization will be intentionally on the positive side. So we have to take the potential from the open circuit potential to a large positive site, positive potential. Then we will see what happens.

Now let's say in case of some metal where you have this sort of phenomena, initially, the reaction would be very vigorous and then, finally, once it achieves a sort of anodic polarization, then the corrosion resistance drops or the anodic current density drops.

So for that let us perform actual test. This is my log i versus potential plot, and let's say I am at OCP or let's say plot with the potential value which this is zero or it corresponds to let's say E_0 for M^{++}/M and M is the metal. M is the metal, and the reaction which involves anodic polarization is nothing but M-2e going to M^{++}.

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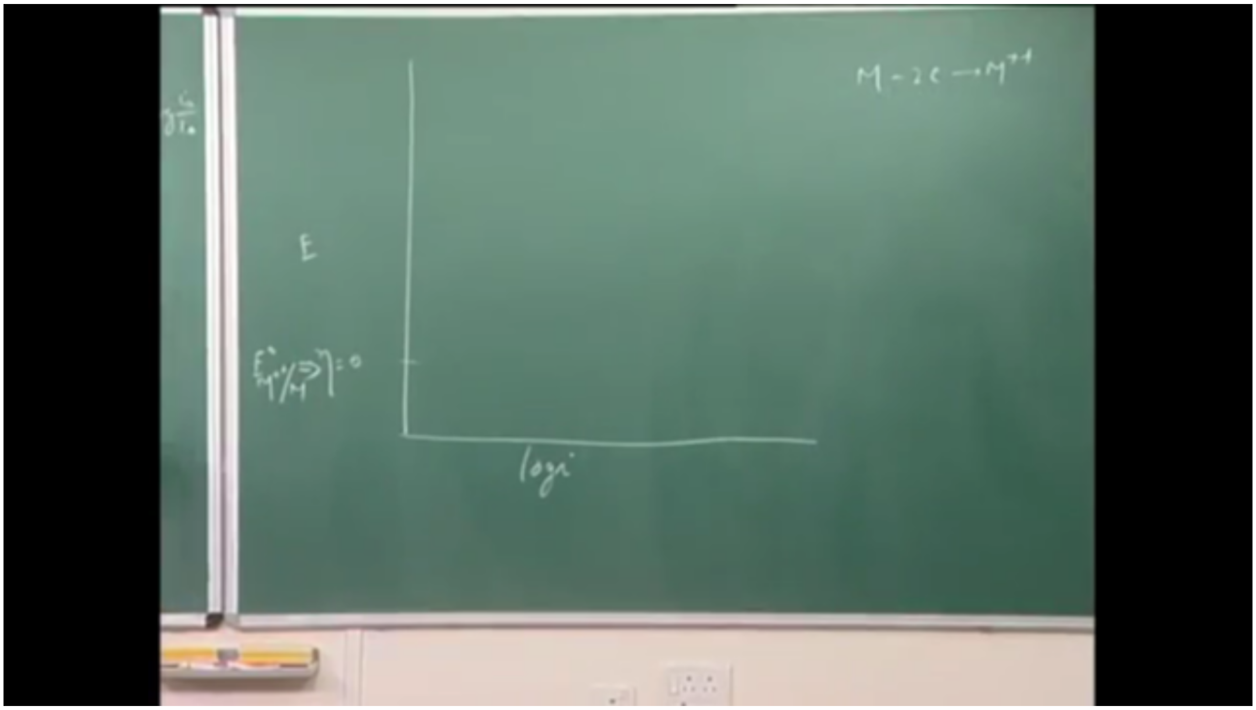

Now initial condition is that metal is dipped in some aqueous medium where you have that extra that we have the metal can achieve high anodic and high corrosion resistance at a very high anodic polarization. So that case we have to investigate the curve what we are getting.

Now the metal initially should be very clean, fresh surface with no oxide layer. So that metal immediately dipped in the solution and immediately, the anodic polarization is started. That means, intentionally, we are passing anodic current to the system so that the potential also increases and then initially, it will be here and gradually, I will see that anodic polarization is starting. At the same time, we are with the application of anodic current density or anodic current.

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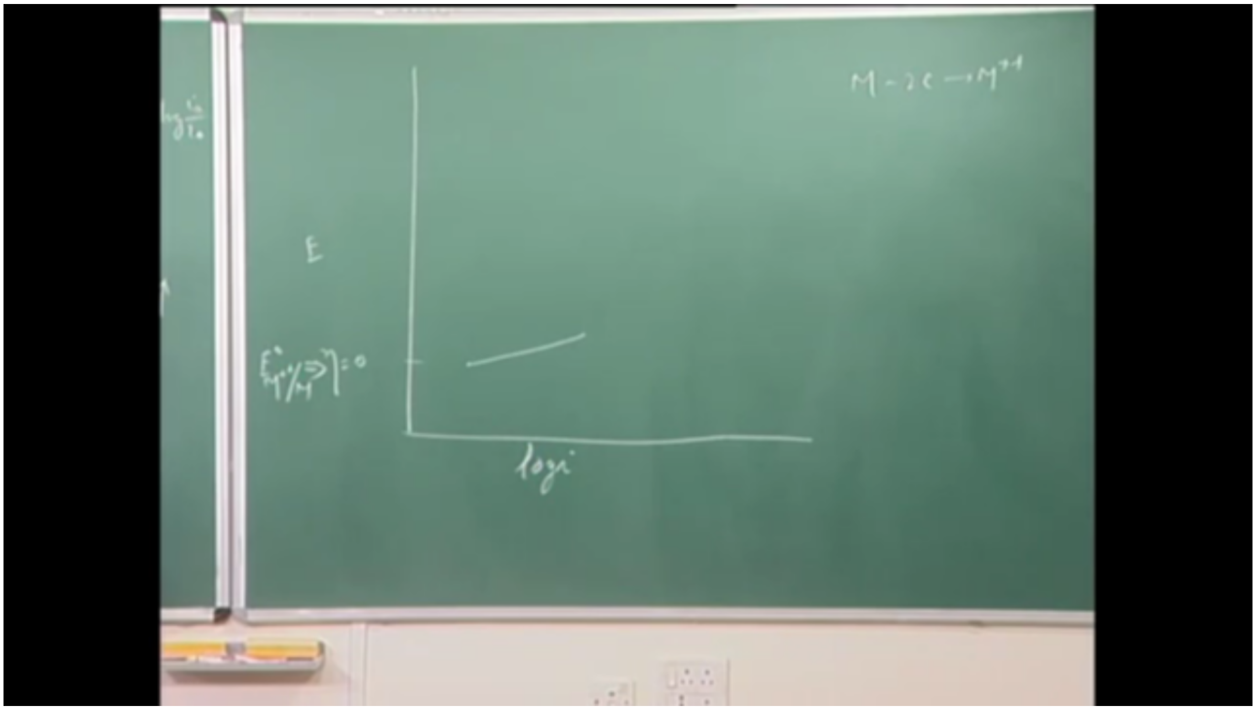

Now you will see that as the line is flowing along this, you will see at different positions if you consider you will have different current density rate $_{a1}$, i_{a2}, i_{a3} and actually, this is -- this is my relation. And what is the reaction that is happening? Reaction that is happening is this one.

Now after it achieves certain potential value because at the same time potential is also increasing, after it achieves a certain potential value, now if the potential increases as we have seen in case of Pourbaix diagram, let's say in case of nickel, nickel Pourbaix diagram is nothing -- like this where you have this is nickel, this is nickel^{++} and this is nickel oxide or nickel hydroxide.

Now if you start from here, let's say you are starting from here or you are starting from here, gradually are increasing the potential and finally, you are reaching the condition where you are having the initiation of oxide.

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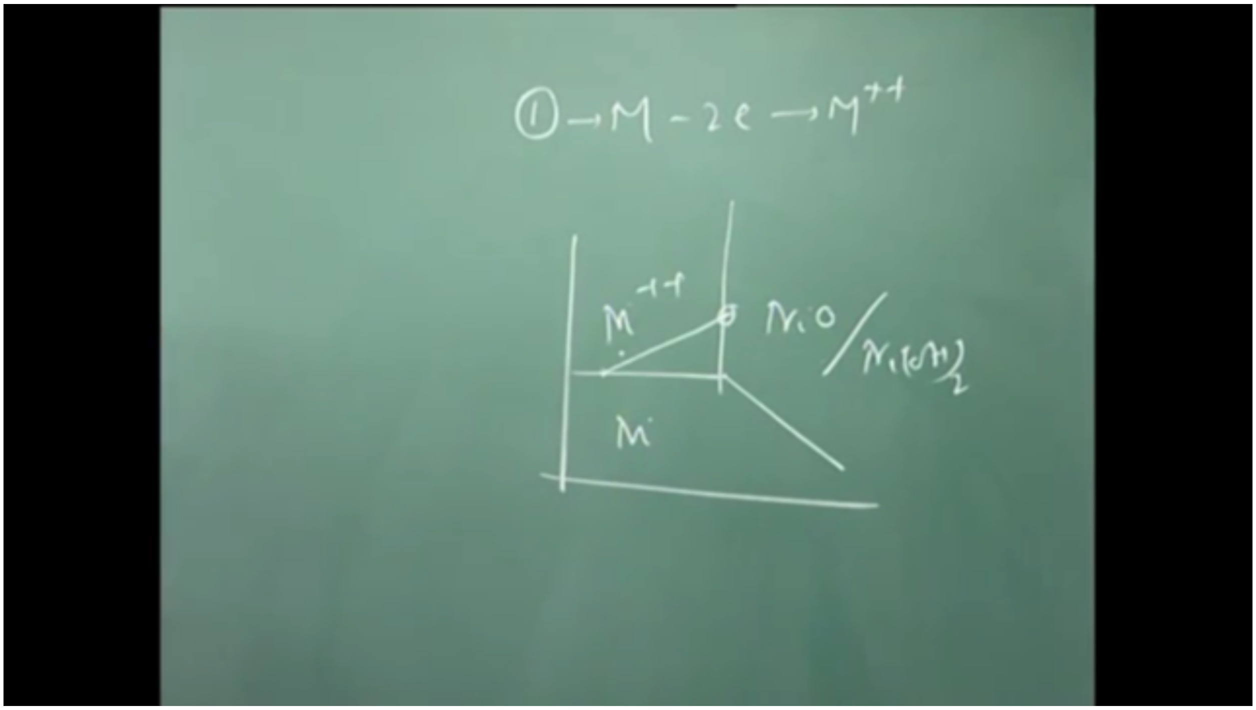

Okay, and that initiation of oxide or it may happen like this, okay, that is to be decided where from you have started this reaction. Anyhow the main thing is after some point with the pH level, this is pH, and this is potential, with the pH and potential level, there could be the possibility of oxide layer formation or oxide formation.

That oxide is forming because of this reaction, because of this reaction M + H_2O going to MO + $2H^+$ + 2e and this happens at some particular potential. For this case, this line is to be considered, and that case this is my MO. This is my H^+, which is basically pH as well as potential guided. So that point if the pH as well as potential are matching with the thermodynamic consideration for this reaction, then this reaction will initiate.

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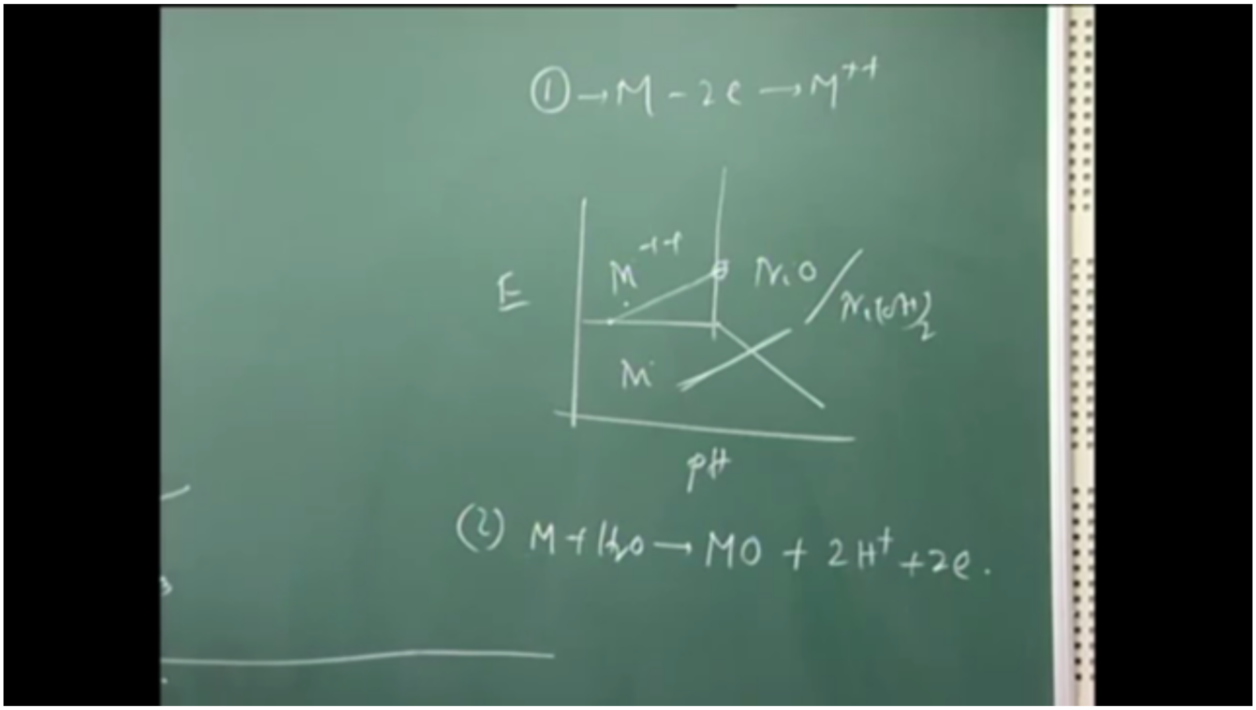

Let's say if this is my point where I have i_0 because this is my non-corroding position where potential overvoltage is zero, now let's say that this point is at this point where this is my let's say B point. At this point I am having this reaction.

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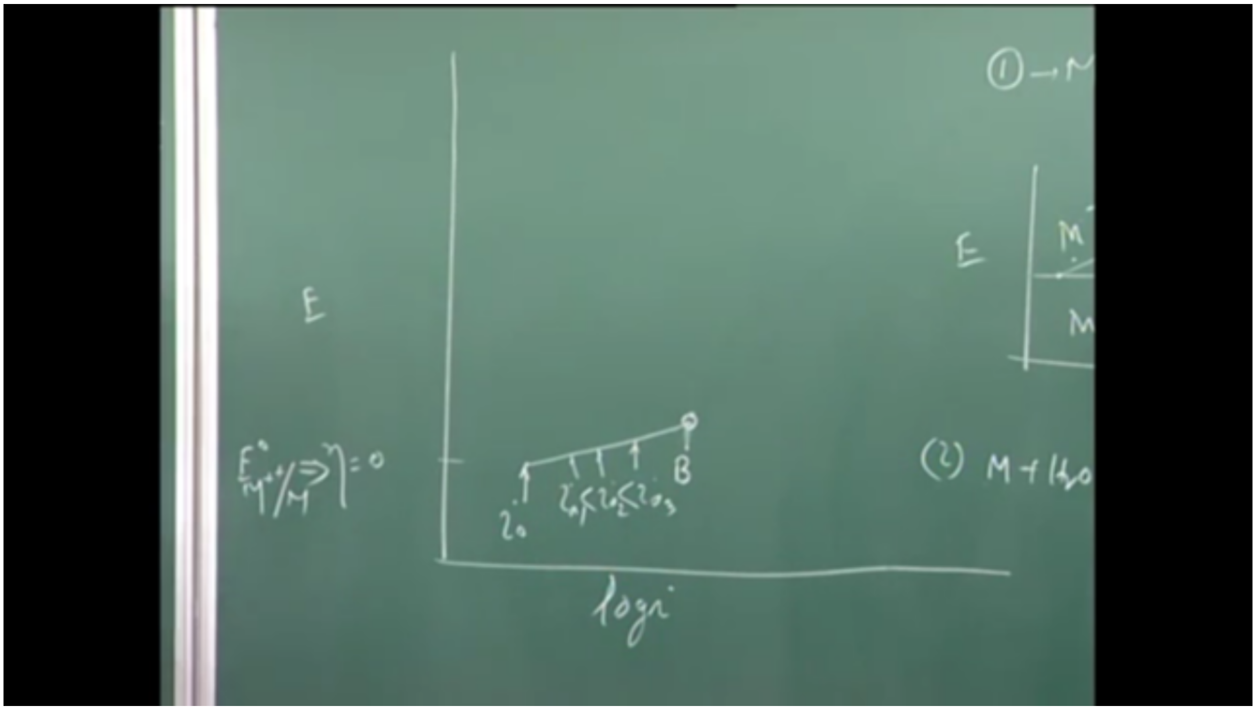

Now this reaction rate would be decided by what? The current density. At the same time, the current density also talks about the rate at which metal is dissolving. So you have some rate, at some rate this is forming. This metal oxide is forming. At some rate you are also having dissolution of that metal. So you have two different rates. One is metal dissolution, dissolution rate (a). (b) is metal oxide formation rate. And let's say the metal dissolution rate and metal oxide rate if you consider, let's say you have a surface like this, and metal oxide is forming at some points. You see that though those metal oxide wherever it's forming, it's trying to cover up the metal surface or trying to have sort of boundary layer between the solution and the metal surface. So those portions definitely we are struck -- we are sort of getting a resistance path which doesn't allow more dissolution to take place from those regions.

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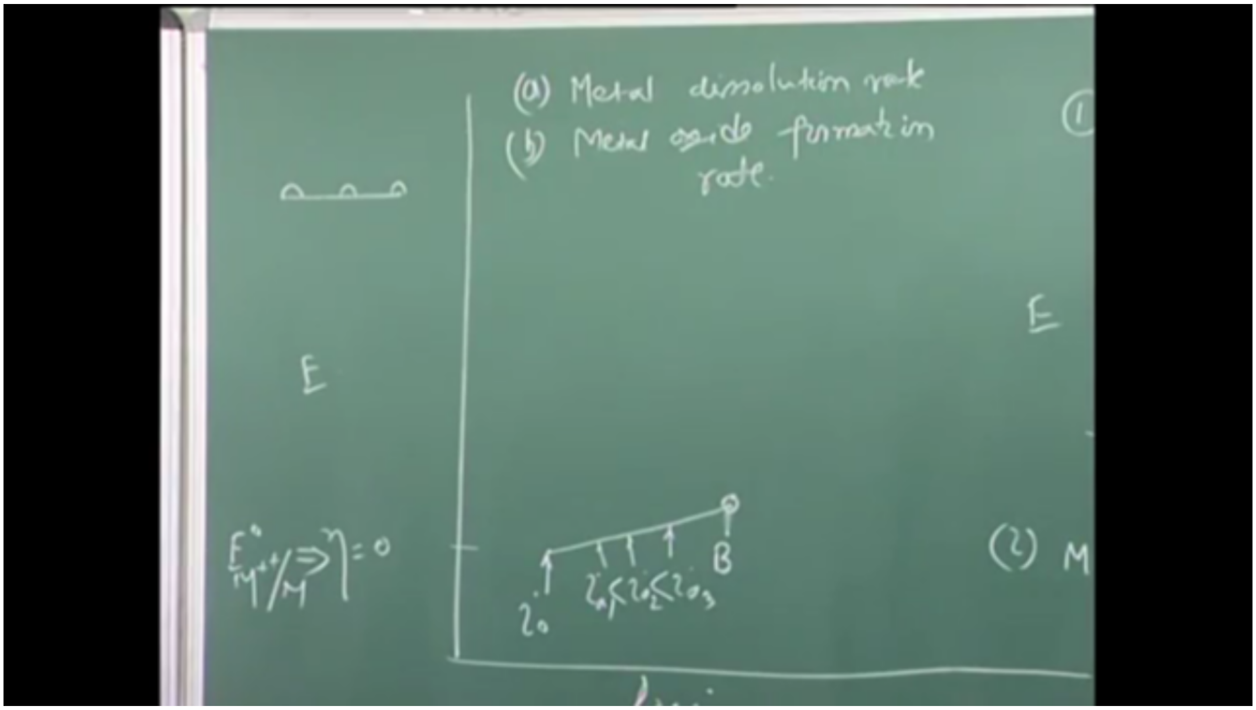

But you see the rate of metal oxide formation, if it is less than this metal formation rate, let's say R_{MO} and let's say this is $R_{dissolution}$, $R_{dissolution}$, if R_{MO} is less than less than $R_{dissolution}$, then actually, we will not get any benefit out of this metal oxide formation, but as you go ahead, so if you continue doing anodic polarization, this line also will move up, but as you go ahead to that higher and higher potential, you are coming to the single phase region in Pourbaix diagram because of thermodynamics as well as pH satisfaction. So if these two things, potential as well as pH, both are satisfied, and we are getting into the single phase region in the Pourbaix diagram.

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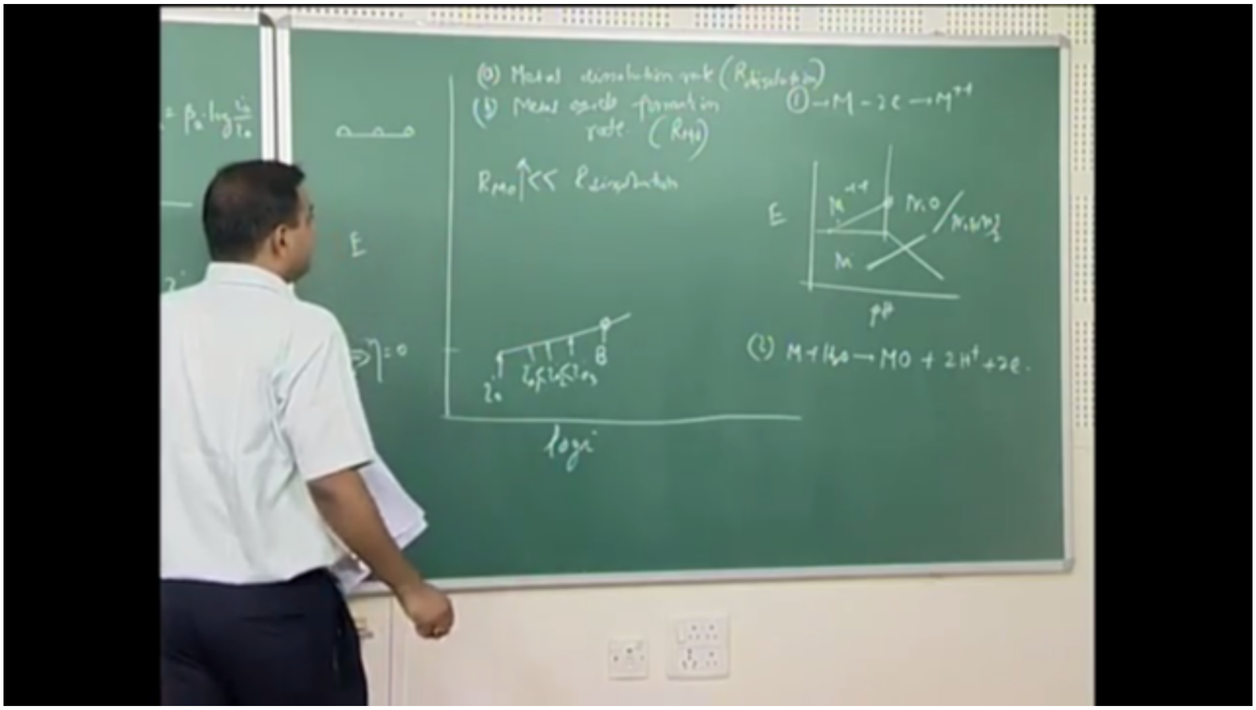

So, finally, we are also going to increase the formation rate of this. So as this one is increasing, you are basically trying to cover up more, major area, more and more areas of those metal surface. See if you are covering major, more and more metal surface, you are going to have lowering in $R_{dissolution}$, that is the rate at which dissolution occurs, but still it will go up, but it will go up at a much slower rate because you are trying to having -- we are basically, because of this reaction of and formation of this, because of this reaction and formation of metal oxide, we are trying to cover up the surface.

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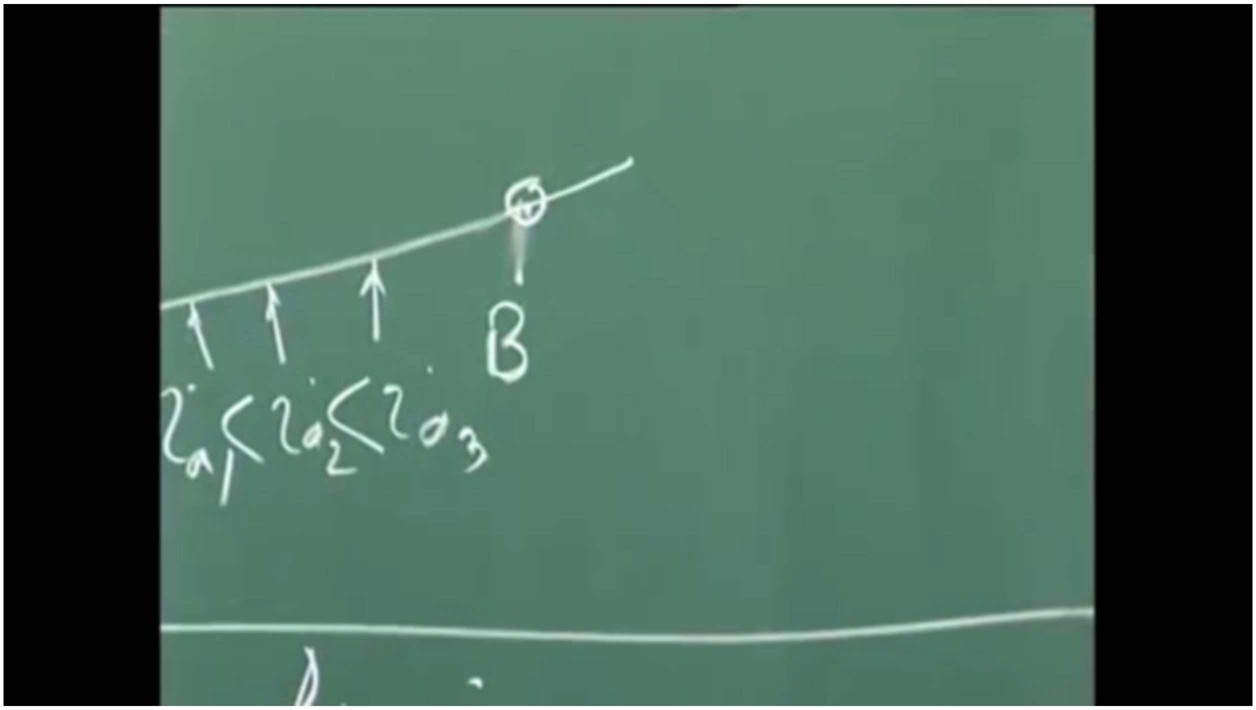

But still I will see that this is farther increasing, but the -- there is now the competition between these two rates. Now once it reaches some point where this is the relation, if this is the relation at some point along the polarization line, anodic polarization line, what would happen? The rate at which metal oxide is forming and the rate at which metal dissolution is taking place, both are equal.

Now once this is -- this point is reached, and if you further increase the polarization, because of polarization, this is increasing, and the rate of dissolution also is increasing, but the rate of increase will -- will be lower, will be very small, and gradually it becomes small, and then finally, will reach that point where these two are equal. And once you cross, once you have a little more polarization, what would happen? This one would exceed this value.

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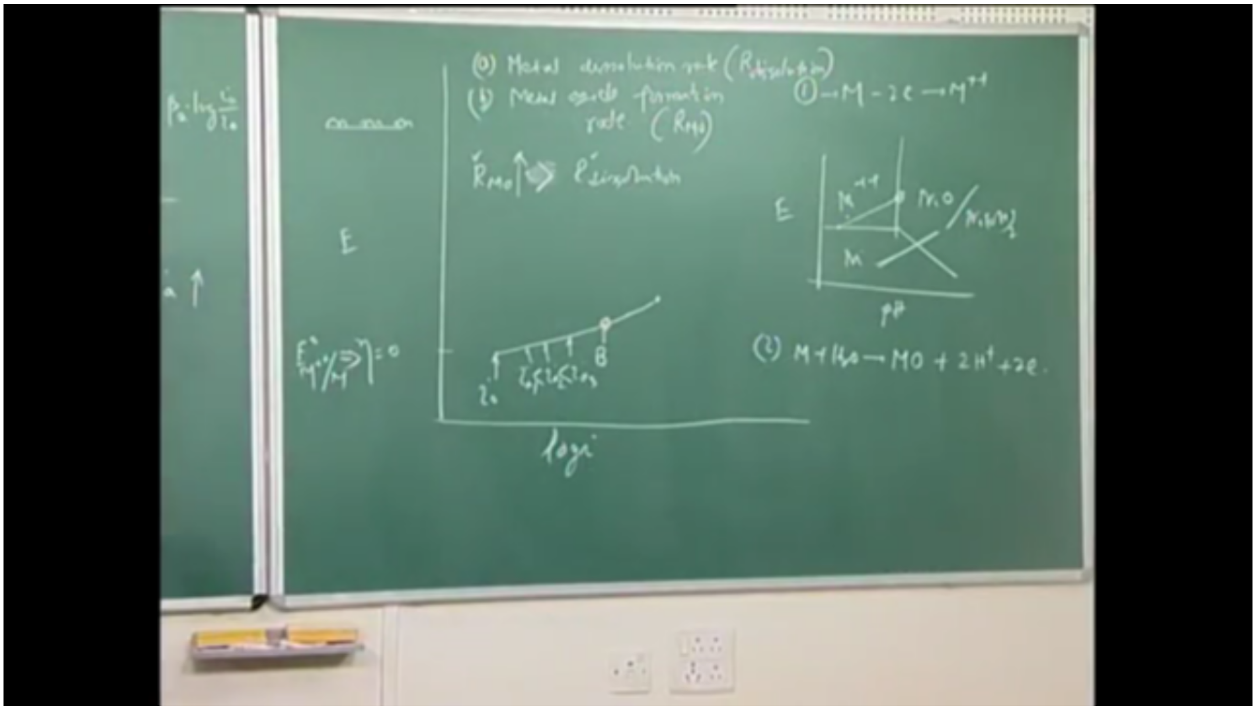

If it exceeds this value, that means the metal oxide formation rate is more than the metal dissolution rate, what would happen? You will go -- you are -- we are going to get a sort of protection mode. That means the rate of dissolution will go down. So rate at which metal oxide is forming will overseed the rate at which metal dissolution is taking place. So that point onward if you go little higher value of potential, it cannot increase further because this line, it will not go along this line. Rather since this metal oxide rate is forming, and so the major part of this surface is basically covered by this metal oxide.

So the very, very small part of that metal surface is exposed to the solution and that's why the rate of dissolution is going down. So from that point it will take a reverse bend, and as it is taking reverse bend, gradually, you see the more and more metal oxide formation, and more and more coverage of this metal surface with oxide layer and gradually, the dissolution rate will go down.

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And finally, there would be a point, that point, from that point there we can achieve -- there would be a point, that point metal -- metal layer surface, metal surface will be entirely covered by the metal oxide. So then the metal oxide layer would be entirely covered by the metal oxide, and once it is covered by metal oxide, so you are going to have a protection because we are now having a sort of boundary which prevents the solution to come in contact with the metal surface.

And if you increase further the polarization, if you -- if you still go along the positive potential side, you will see that from that point onward it will take almost vertical line, parallel vertical line with respect to log i. So you are going to get this line, this line.

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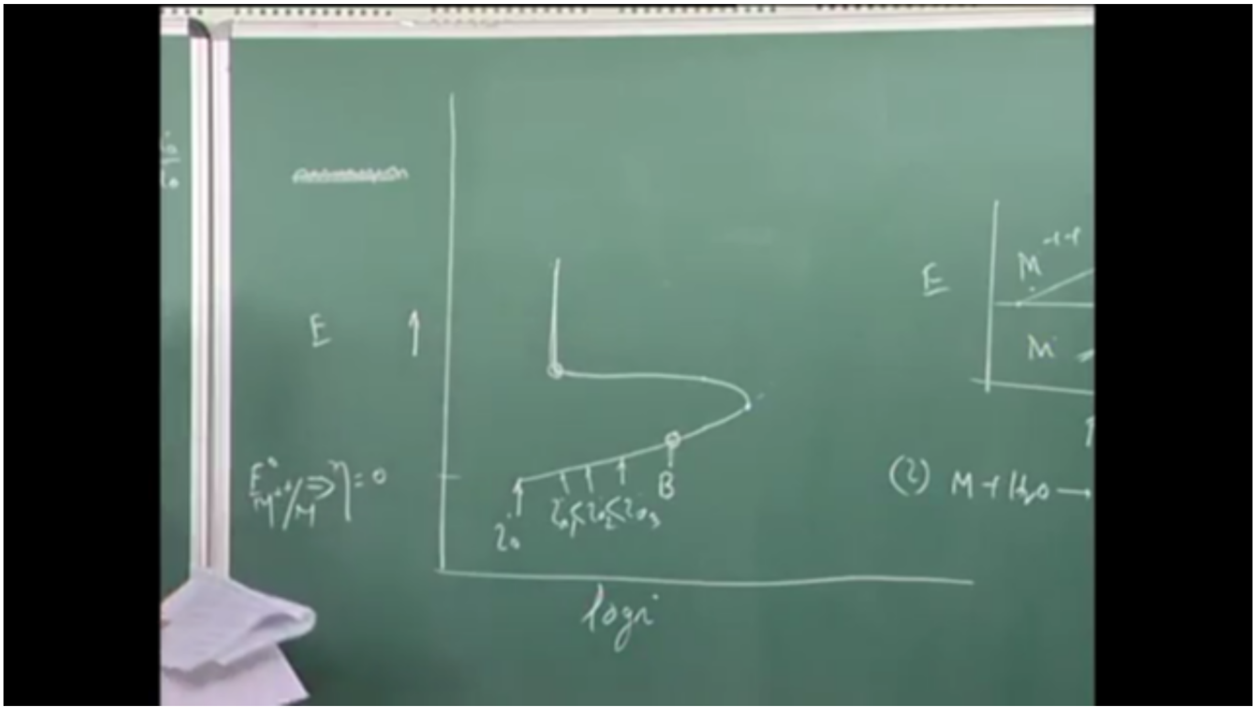

Now that means here is this point is sort of critical point where the rate of metal dissolution and the rate of metal oxide formation, both are equal, and once you further towards the positive potential side, then this line will take a reverse bend. So current density corresponds to the -- corresponding to the anodic dissolution will also go down, and finally, will reach a point where the metal surface is completely covered by metal oxide and you will get a complete protection of the surface because of metal oxide layer and from that onward, this line, if you go further to a positive potential, this line will be like this.

The polarization line would be like this, which is almost vertical to log i line -- log -- vertical to log i axis, and point is called $i_{passive}$, and this critical point and that point is called $i_{critical}$ and within this zone is basically the metal oxide, what is happening? The metal oxide is trying to cover up the entire surface because of much higher rate of generation of metal oxide compared to the metal dissolution.

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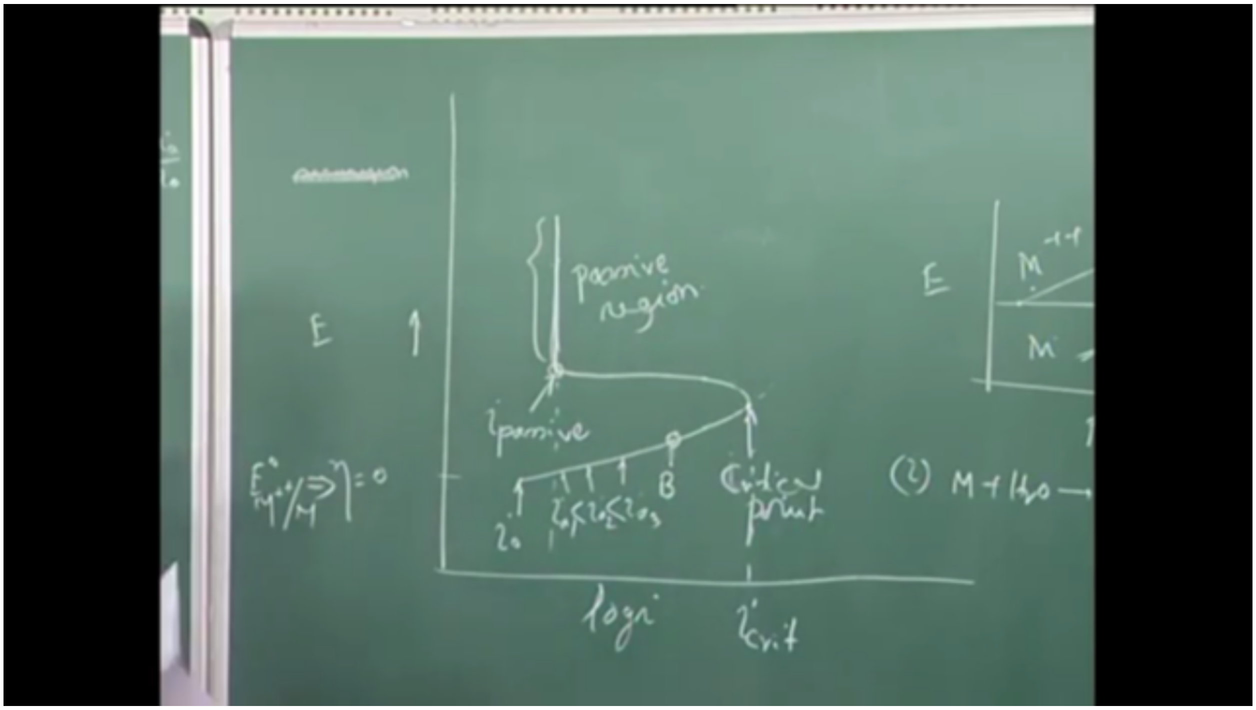

So this part a sort of unstable position and this region is passive region. Why passive region? Passive region because if you consider the current density, and this is in log scale, with the increase in polarization, anodic polarization, initially, we are having higher and higher current density or higher and higher dissolution, but once you have this formation of metal oxide and once you have this phenomena, you are achieving at some point, you are achieving this point where you have the limiting current density for dissolution because of metal oxide formation on the surface or because of surface film coverage of that surface, and then it will go like this.

And within this zone what is happening? Because still i -- there is sort of $i_{passive}$ formation, there is sort of current density, which is $i_{passive}$ and that portion if you consider the $i_{passive}$ value in the log scale, this is very, very small value, and $i_{passive}$ is nothing but dissolution rate in the passive region, and that is a very small value. And if it is a very, very small value, what does it mean? It means that it gives a much lower dissolution rate in that solution because of this oxide layer formation. Now -- and this is called passive region

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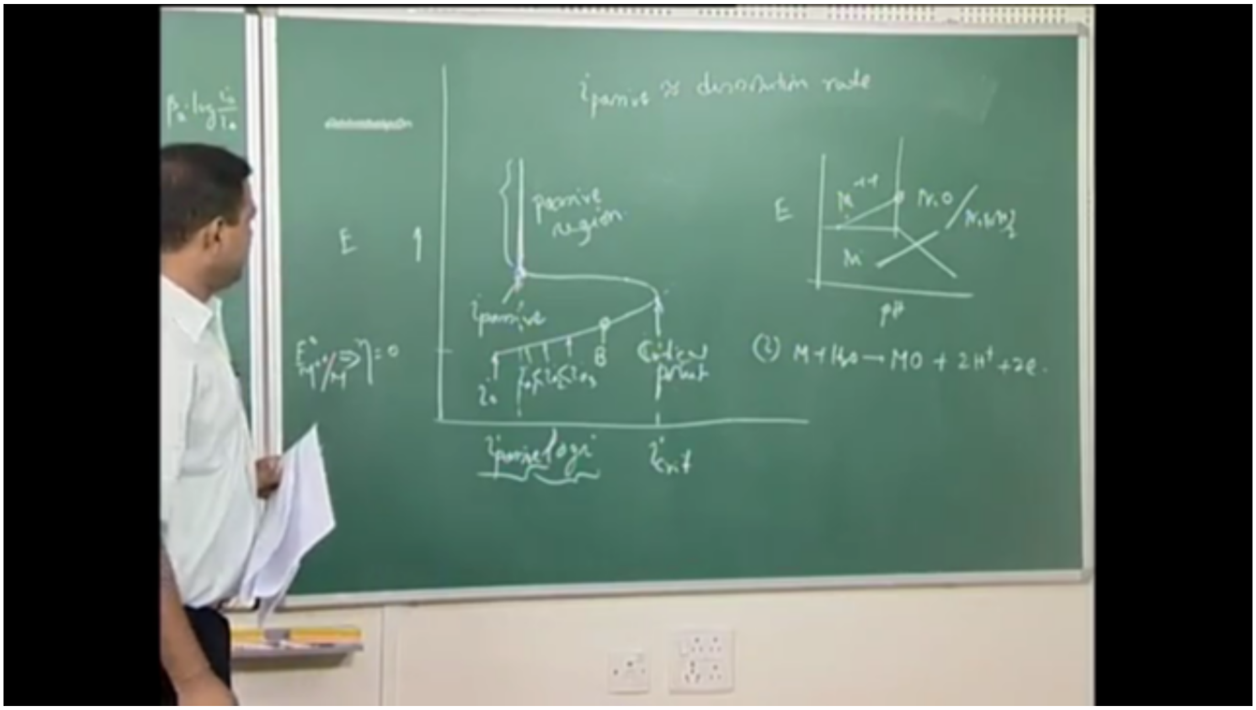

Now let us understand this large anodic polarization. You see if I am here, I see that anodic polarization is this much. Anodic polarization is this much and still corresponding to that current density where I have reached this point, this anodic polarization is pretty high, but once you let's say I have this is my anodic polarization, this is my anodic polarization from here to there where I would -- I would reach to this point, and at this point you see that the dissolution rate has decreased because $i_{passive}$ has decreased. So now large anodic polarization is leading to the formation of passive region, and that's why you are getting the benefit of corrosion resistance because of metal oxide formation.

Now if you further increase it, further go towards the positive side, what would happen? One option is we have also seen in case of -- in case of Pourbaix diagram, we know that there is a zone for H_2O stability, and this is why zone for H_2O stability and this is my line where I have oxygen formation. If I let's say I am along this line, if I reach to this level, then what would happen? Oxygen formation would be possible.

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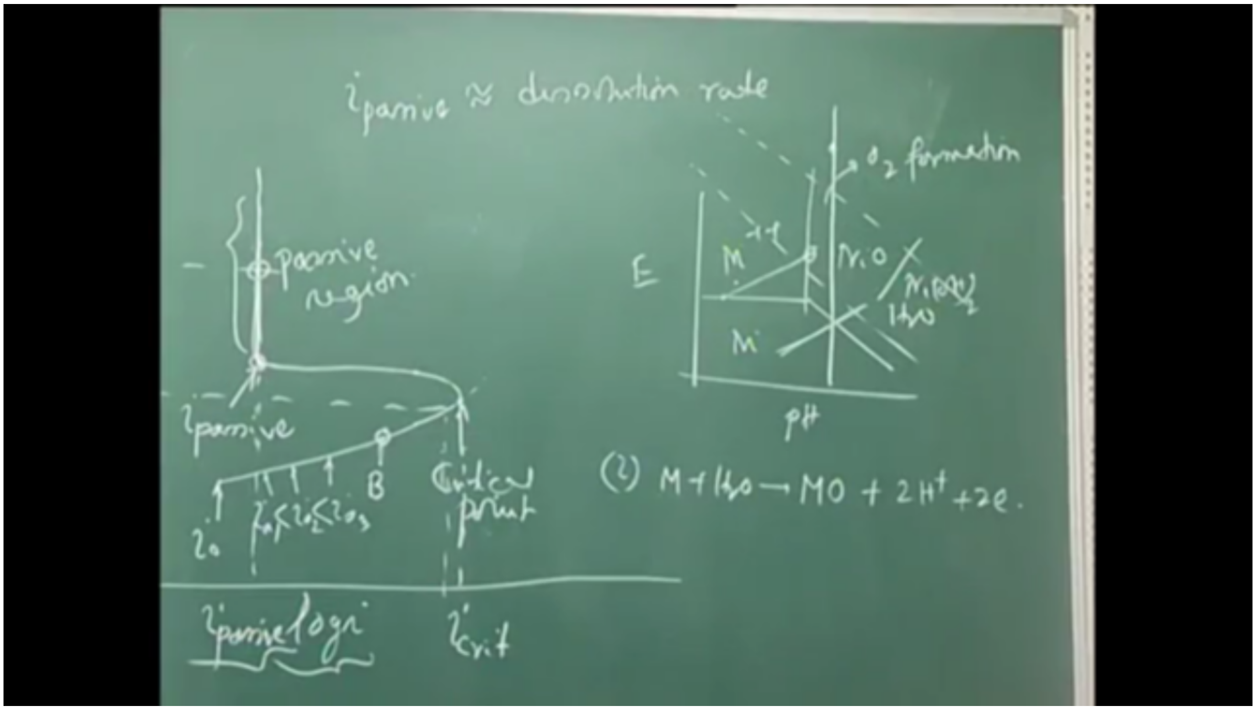

So if oxygen formation is possible, and if I reach to that level of potential and at that some pH value corresponding to this pH value, I'll see that this oxygen formation would be there because of two reactions. One is O_2 + $2H_2O$ + 4e going to $4OH^-$. So this reaction would happen. The other one would be this is a neutral solution or basic medium.

Second is if it is in acidic condition, if the pH is below seven, then this reaction can happen. This reaction can happen. So $2H_2O$ will break into O_2, and then once we reach to that potential value corresponding to that pH value, depending on whether it's a neutral solution or acidic solution, this oxygen generation would be there, and this oxygen generation rate is nothing but can be expressed in terms of current density, and if we reach to that value, this line will have a bend towards right again.

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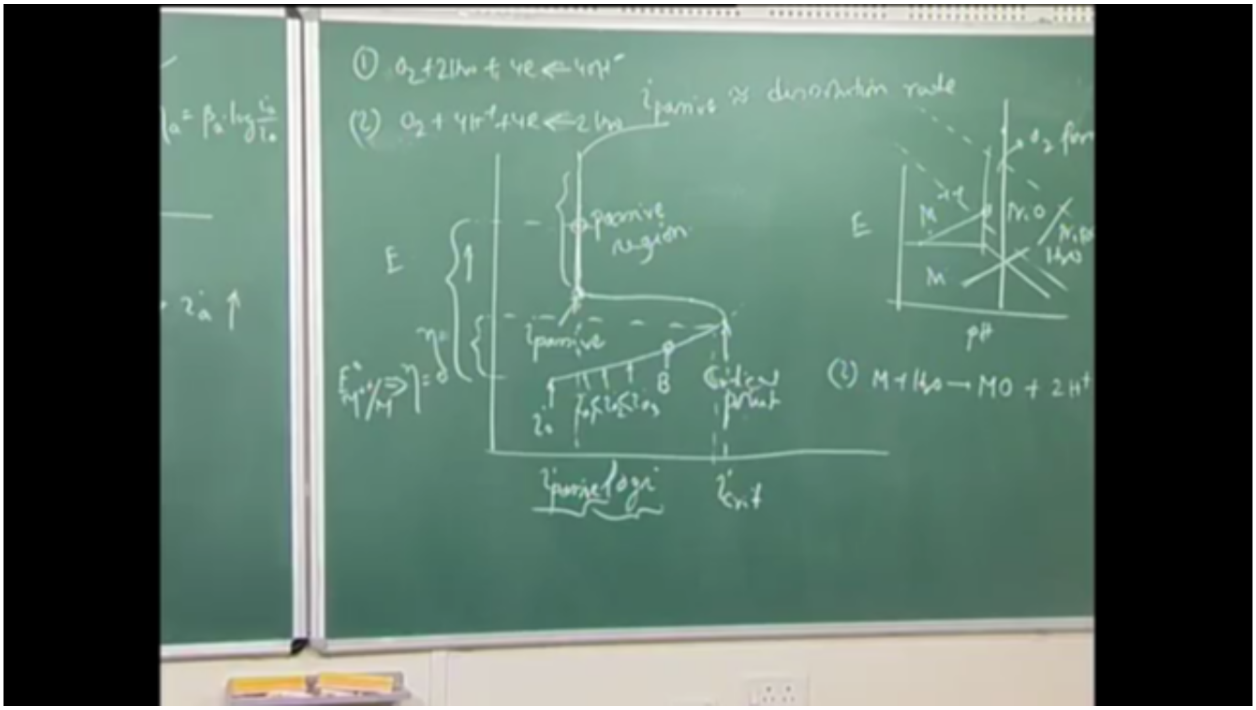

And this one corresponds to oxygen generation. So this potential corresponds to potential around this region or rather this -- this one so where you just reach to that potential value, and gradually, if you go higher anodic potential, you are going to have larger and larger oxygen generation rate. So this is sort of break in this line.

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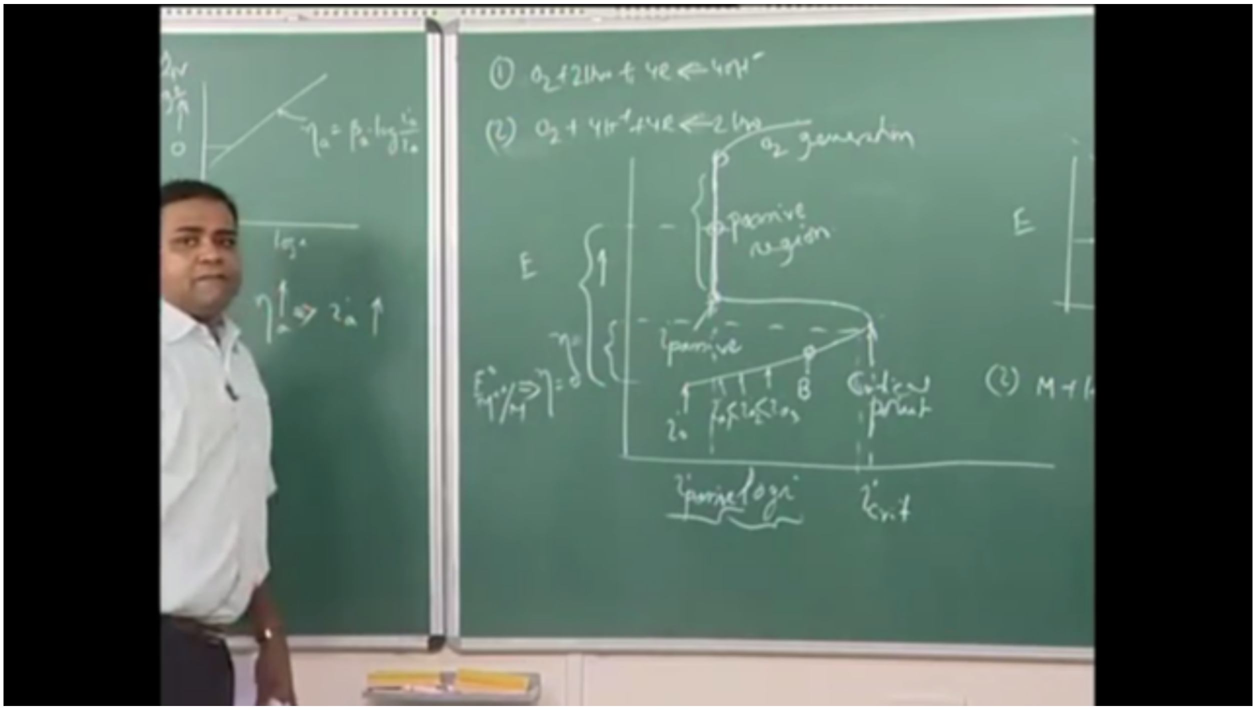

This line, which is the passive region which is breaking down, this line can be broken if there is chloride ion or halides ion. That halide ion can break this line, or we can also have a sort of breaking of those passive layer or the oxide layer because of chloride attack, and that can happen in this mechanism.

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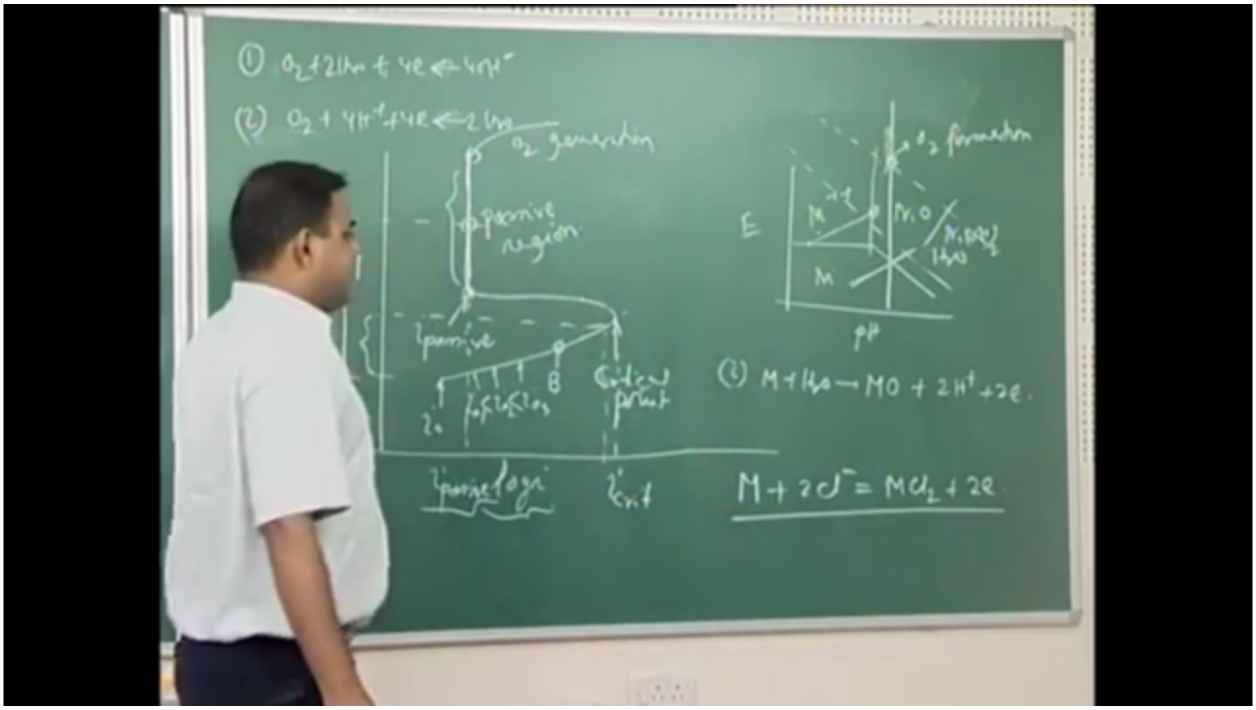

So one that can also be possible, let's say this reaction can happen also. This reaction can happen, and if metal -- if metal reacts with chlorine ion, it can form metal chloride, and this metal chloride is soluble, and where it forms depending on the potential, that point all over the surface layer will be broken and there would be a break like this. So this point corresponds to $E_{breakdown}$ or $E_{pitting}$ because chloride ion leads to pitting of the metal. So this is one -- one way.

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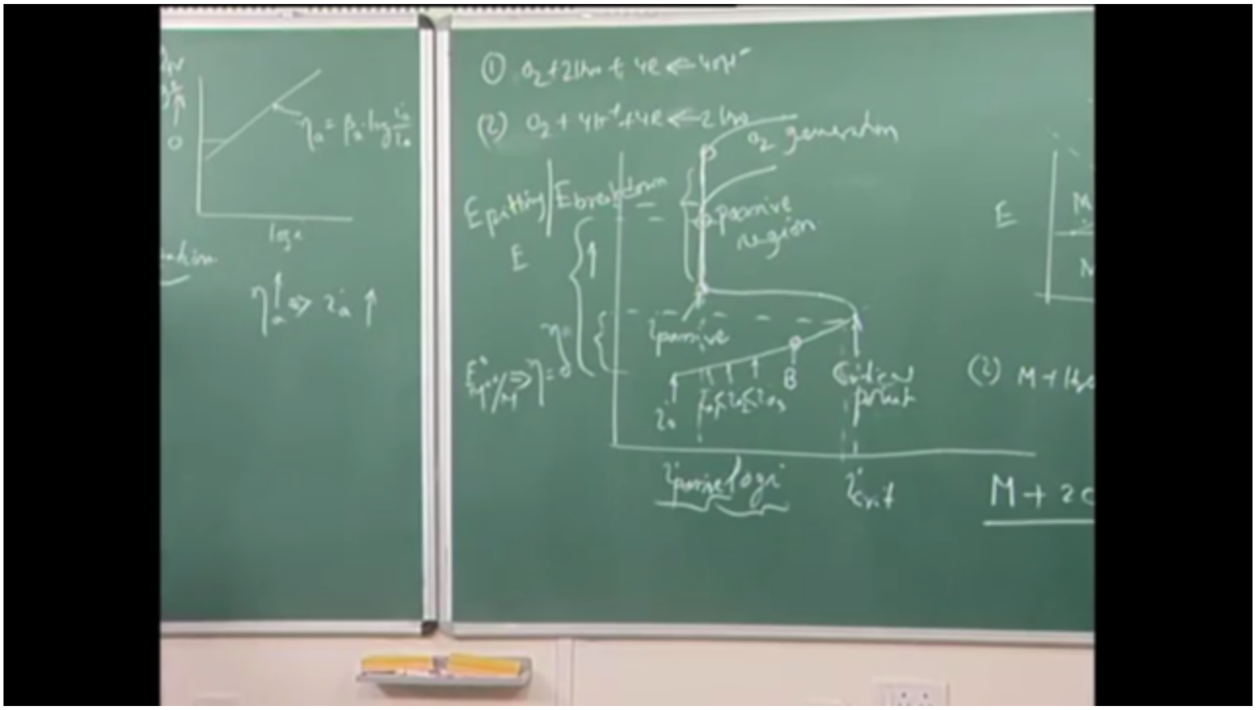

Another way is this passive region can be broken at much higher potential. Let's say in case of chromium, let's in case of chromium that chromium, let's say chromium^{3+} ion is forming because of whatever small dissolution chromium surface, this Cr_2, Cr^{+3} can react with H_2O and going to Cr_2O_7 minus +14 H^+ plus 6e. At a very, very high potential for this, at very, very high potential, this reaction can also take this. And if you carefully observe this, at a very, very high potential, you have Cr_2O_3 layer, and this Cr_2O_3 layer, we have Cr^{+3} ion, and that ion can react with H_2O and form Cr_2O_7, which is a complex ion, but it can dissolve in water.

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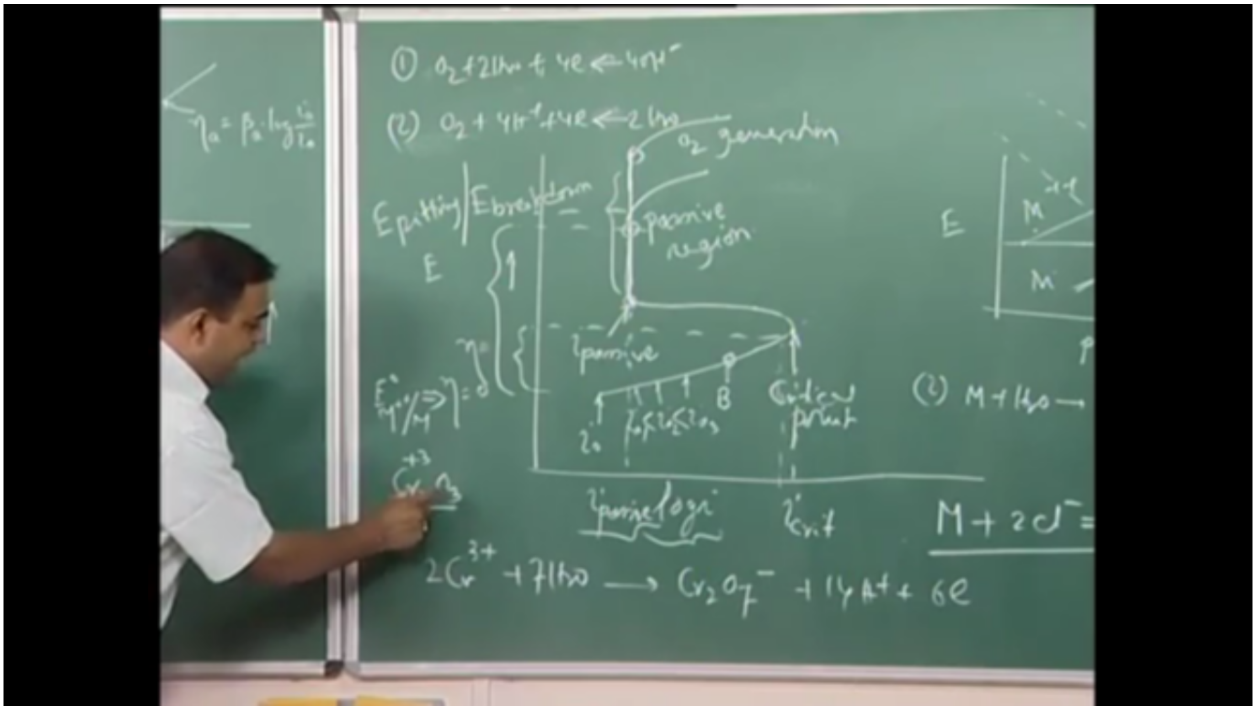

So that means you have oxide layer. That oxide layer is reacting with H_2O and forming a complex ion of $Cr_2O_7^-$, which is soluble. What does it mean? The protection is over because oxide layer is eaten away by this reaction, and that can happen at a very high potential depending on the standard reduction potential of this reaction. So, again, you can have another break, and that break because of different reason, because of this. In case of chromium, it can happen.

Another break that is possible in case of aluminum, that aluminum can also form a sort of Al_2O_3 layer, which is porous. Why it's porous? At a very high polarization, it can have porous aluminum oxide layer because as you are increasing the potential, what you are doing? You are basically increasing the potential field across the metal surface and the solution surface.

So you have -- you have metal surface. You have oxide layer and this is solution. This is metal oxide and this is metal. So as you go higher and higher potential we are going to have, we are going to create the potential difference or the potential gradient between this and this surface.

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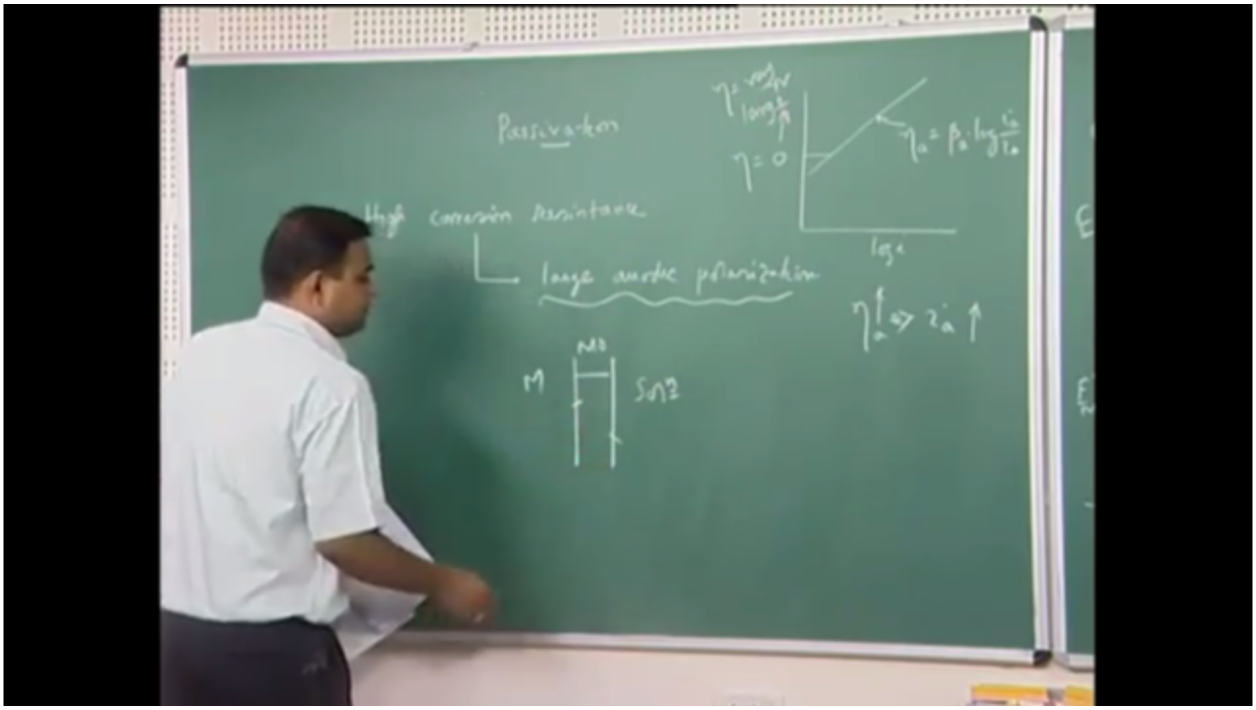

We are going to increase the potential gradient because as we go up and up, we are going to have a larger potential gradient between this layer and this layer. So as you go higher and higher potential, you are going to have large potential gradient between this metal surface and solution surface, and these potential gradient will draw this metal ion towards this metal surface, this -- this surface. This higher potential gradient will draw this metal ion -- metal ion towards this surface, and this here it can react with OH forming $M(OH)_2$ or it can also form MO. So, gradually, as you go higher and higher potential this layer will thicken.

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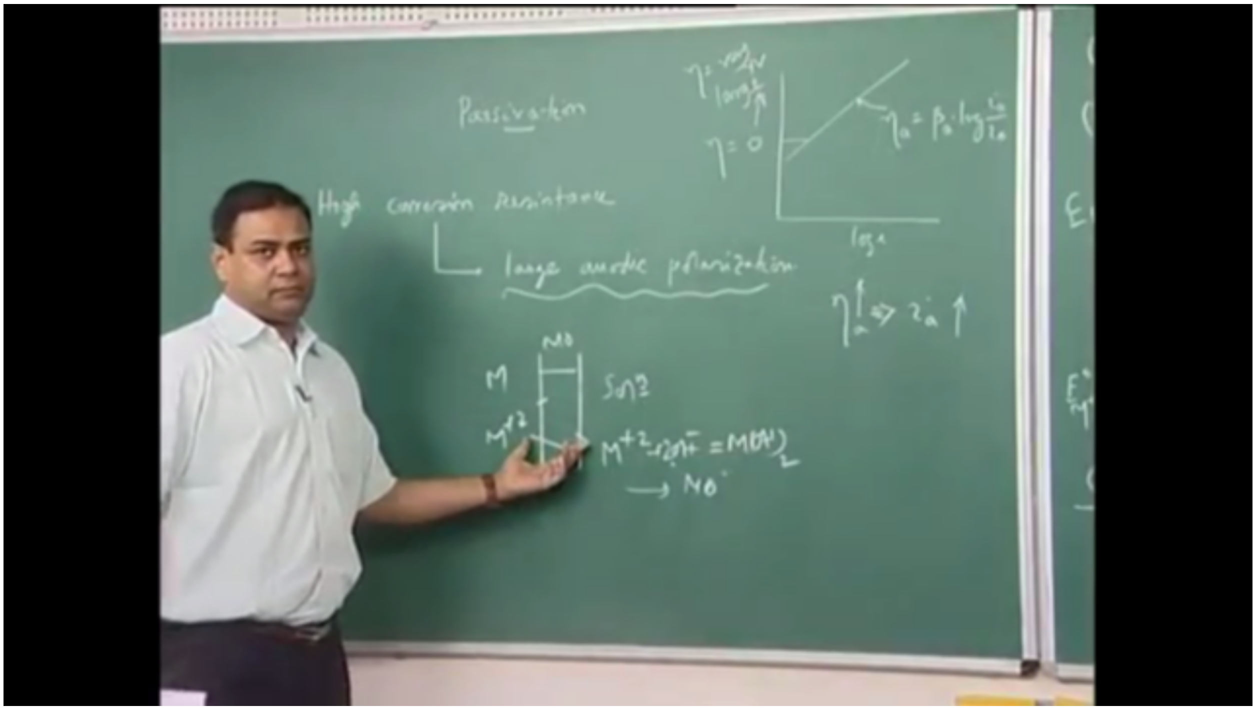

But in case of aluminum what happens? If you go a very, very large potential, the rate at which this metal ion is getting drawn from this surface to this surface will be much larger than the rate of which this combination reaction is happening. So what would happen? Because of that phenomena, this -- this rate of -- rate at which metal oxide is drawn from the metal surface and towards the surface, because of this, the surface, this oxide surface will become porous and that is called anodizing.

So that case since we have a porous layer and then, again, you will see that there would be a break in this. There will be a break in this region. So it can happen because of chloride attack, because of oxygen generation. It can happen. This break -- this break in the passive region, it can happen because of passive -- because of chloride attack. This is chloride attack. It can happen because of oxygen generation. It can happen because of complex ion formation at higher potential, higher anodic potential. It can also happen because of anodizing in case of aluminum. So this is on the way overall this acid curve for a passive metal in a particular solution where the passivity can be achieved.

So now if we draw this curve here this is log I, this is potential, and that this is -- this is 0, and let's say this is my i_0, and it will have this kind of behavior and where this region -- this region up to $i_{critical}$ is called active -- active region from. This to this, it's called passive. And when -- when the passive film is broken, that part from above this point above, this is potential axis, it's called transpassive -- transpassive.

So active region, passive region, and transpassive and there is a small region here. This region we have already seen that the phenomena of coverage of the metal surface with the metal oxide is taking place. So that region is basically a kind of unstable zone -- unstable zone. So these are the different parts of the passive curve in case of a passive metal in a solution where you are going to experience this kind of behavior.

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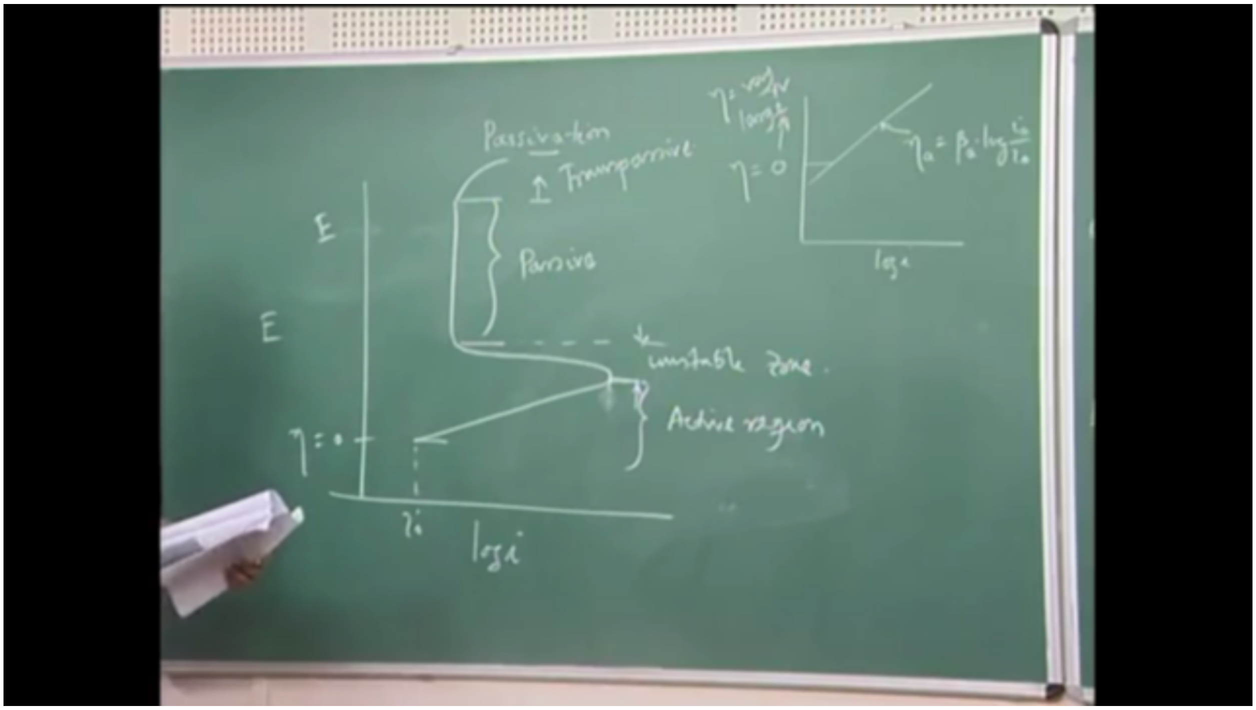

For example, if you dip iron freshly -- freshly prepared surface of -- freshly prepared iron, if you dip it in HNO_3, you will experience curve like this, but if you have the same freshly prepared iron and dip it in dilute HNO_3, you will not experience this because there you can only experience the active part. The passive region will not be reached because you don't satisfy the condition of metal oxide formation or metal hydroxide formation. Fine.

So it's also solution specific. One is metal specific. Second is solution specific, and there are few other issues. For example, temperature specific. Few other variables which can -- which can guide whether this particular nature can be achieved or not, temperature -- temperature. Then you have reducing atmosphere -- atmosphere. Then you can also have abrasion -- abrasion. We have already seen in case of iron, if, from concentrated HNO_3, if we take the metal one and put it in diluted HNO_3, we don't see any reaction, but if we scratch it, then we'll see that there will be a vigorous reaction in the dilute HNO_3. So abrasion is one important issue whether this passive layer will be maintained or not. Then you have cathodic polarization -- cathodic polarization.

Now we'll see that importance of cathodic polarization whether this cathodic polarization actually will guide the system to reach to the passive region or it will also guide whether the system will still remain in active region. So this part let us now start discussing.

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So we have to start the effect of cathodic polarization. Now when you have this graph, this e versus log i, you have this graph.

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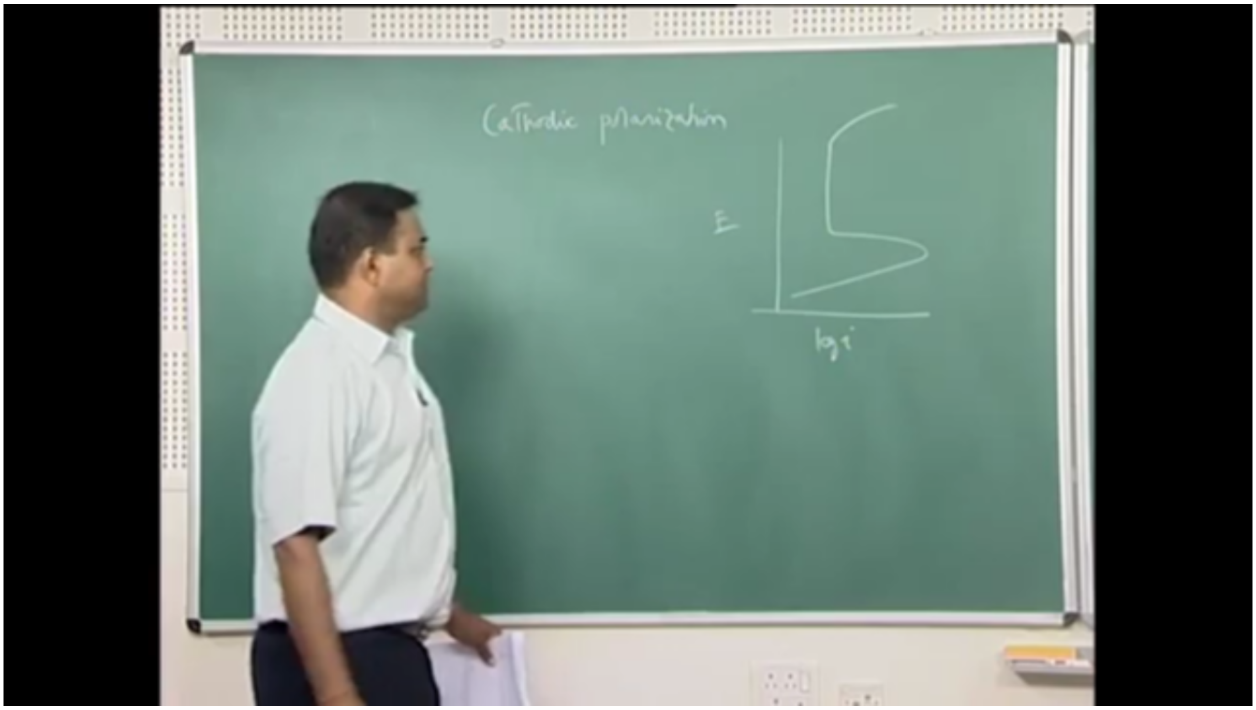

What we are doing here? We are taking the potential to a higher and higher potential value sending anodic current to the metal. Now in this is basically a sort of external stimulant we are using. That is current density or anodic current density, which you are passing towards to the metal surface, but in actual system, let's say you have a freshly prepared iron surface and dipped in HNO_3 where you are not -- you have not connected it to any electrical circuit. So how would it achieve that passivity? Now that passivity achievement will be decided by the cathodic polarization that is taking place in the system.

So let's say I can have a cathodic reaction like this. I can have a cathodic reaction like this. So now which point the system will remain? The system will remain at that point where you have the rate at which the equal -- equality between the rate at which electrons are getting generated and the rate at which electrons are getting consumed. So system will remain at this point if you have the cathodic reaction like this. So until unless I reach to this level, I will not get the passivity, but here the system is remaining at this point which is in the active zone -- active zone.

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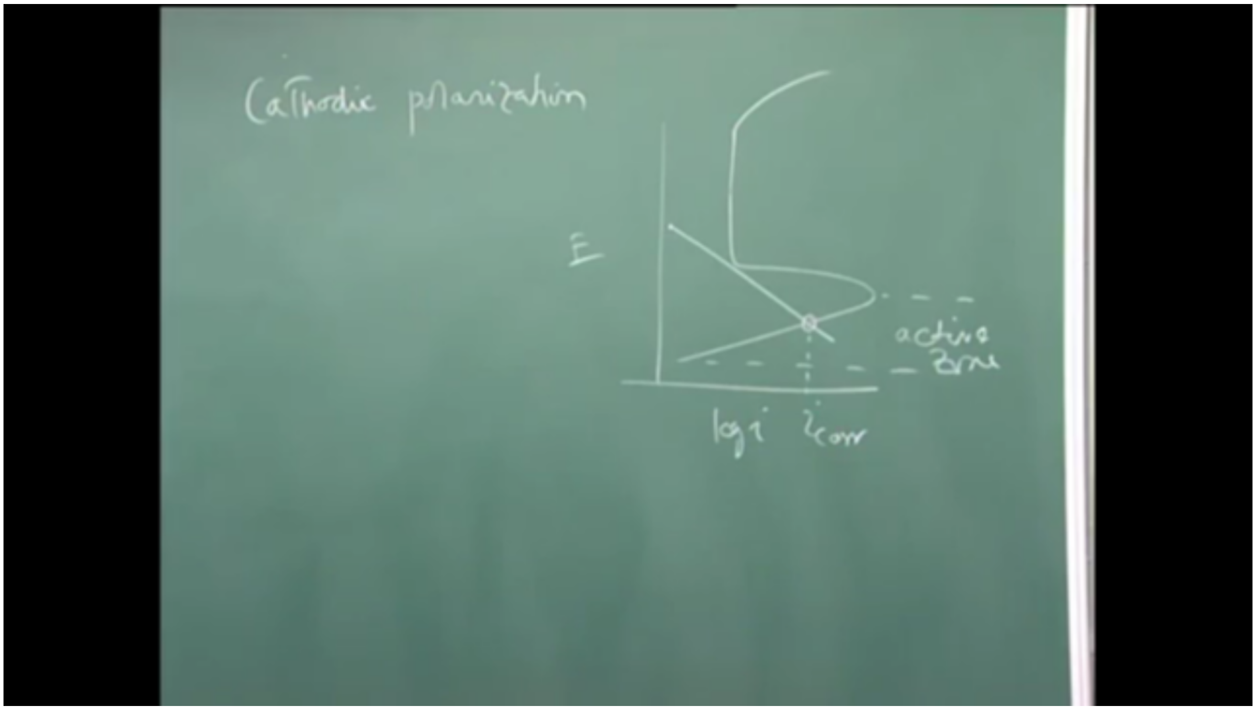

So system will have the corrosion current corresponding to this. Now let's say I have some cathodic reaction in the system which is happening like this. So if it is happening like this, the point where you see the rate at which electrons are getting generated and the rate at which electrons are consumed, that point is nothing but this where here i_c equal to i_a equal to i_{corr}.

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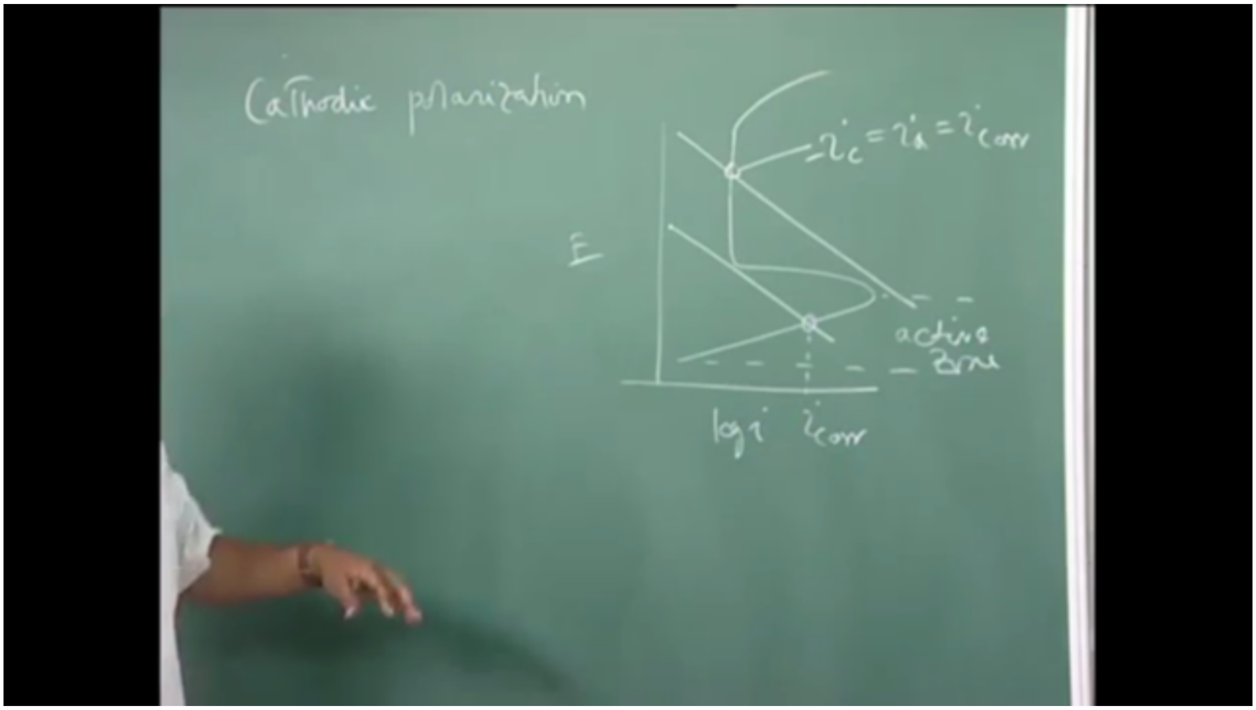

Now if the cathodic reaction is happening like this, if the cathodic polarization is happening like this, and this is my anodic polarization, so system will reach there. Now the point is then which -- what guides the system to have more and more anodic polarization and to reach this point? Because if you would like to -- if the system has to reach to this point, which is the equilibrium point between the charge transfer rate, that means the electron consumption and the electron generation, so this cathodic reaction actually will take the potential of the anode or the metal dissolution. That potential will -- will be lifted to this level. So, actually, the cathodic polarization is taking the potential of that metal towards to this level or so that means this cathodic polarization actually decides whether the system will remain here or the system will remain here. If it remains here so it will be just like some active metal.

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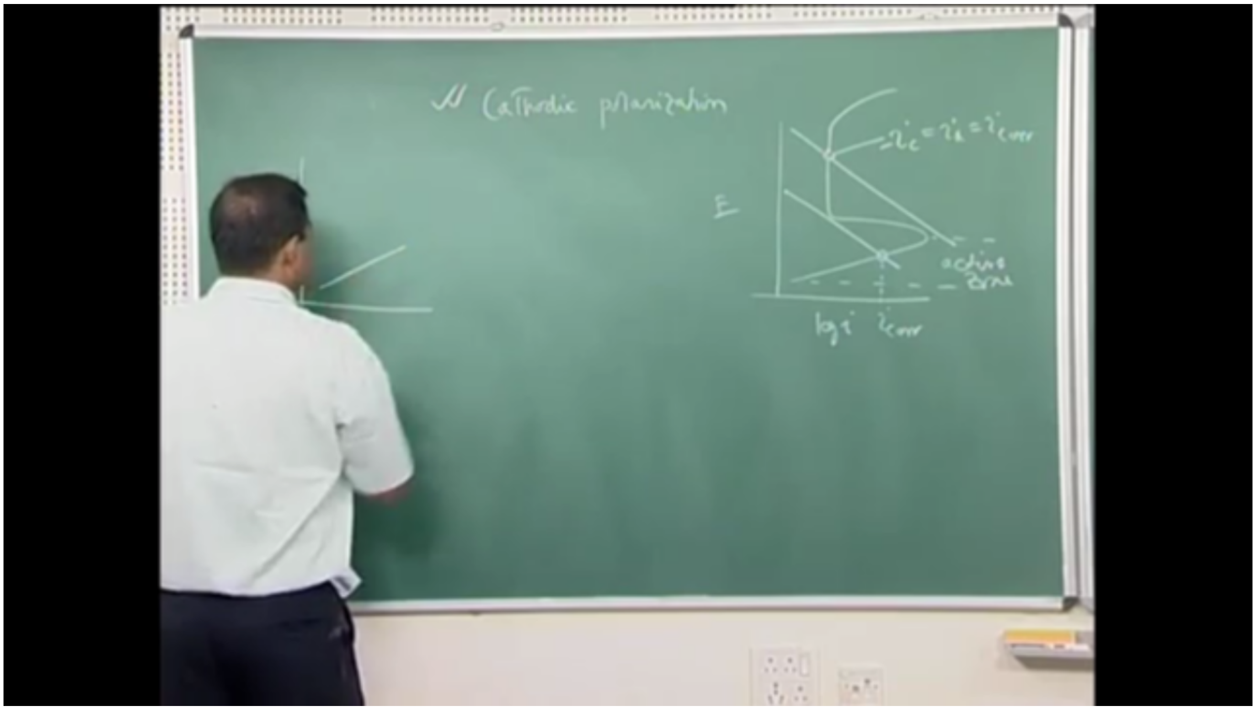

We have seen in case of zinc in HCl, we see that this is my anodic polarization line, and this is my cathodic polarization line, and this is my point where I have i_{corr}, which one basically nothing but i_c equal to i_e, and this is e. This is log i.

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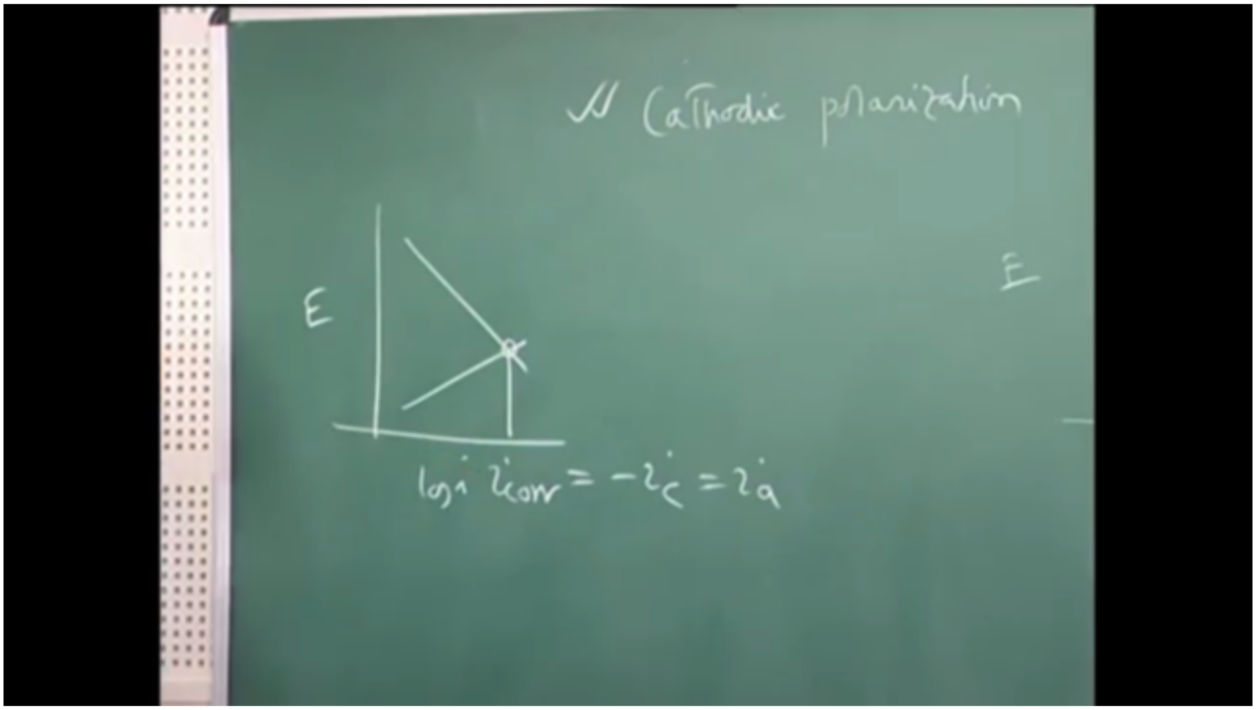

So this is a normal circumstance in case of active metal. So system will remain in active zone, but if it is -- if it has to reach to the passive level, then your cathodic polarization line should be placed like this. So that means here we get to the passive region. So this is passivity and this is active corrosion. This is passivity. This is active corrosion and the catholic actually is helping to reach to the passive level.

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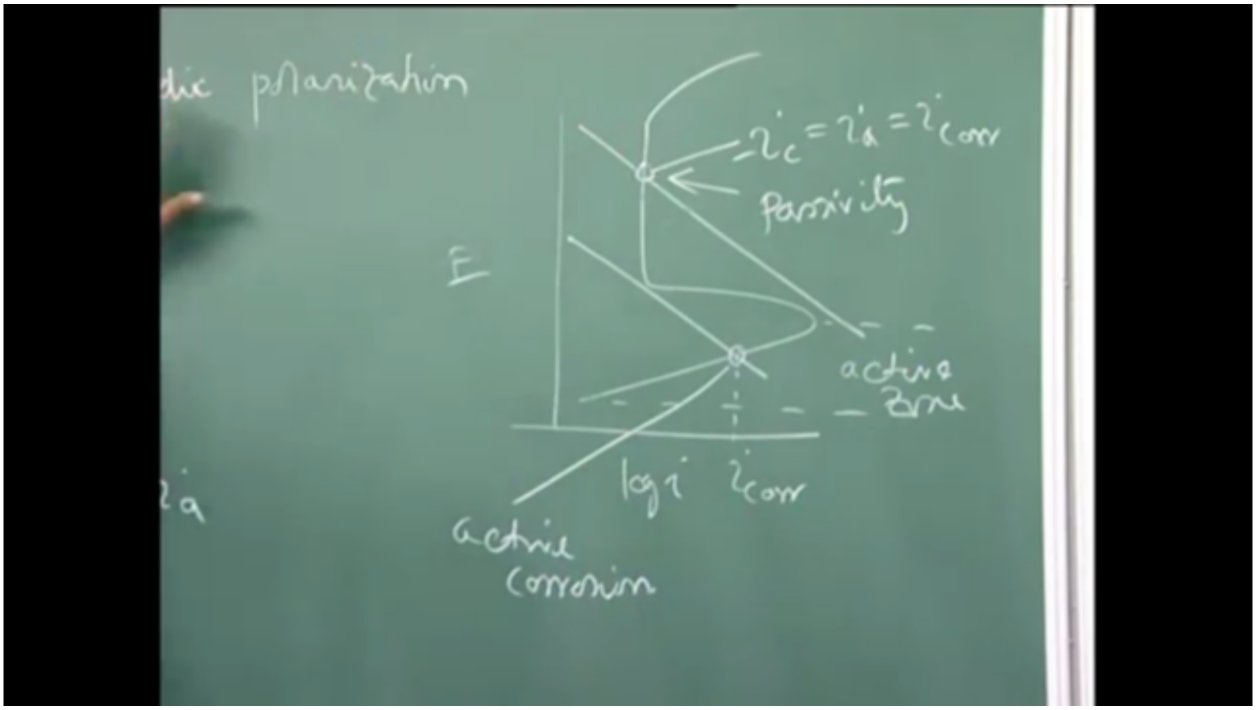

Now if you see these two lines, there are major -- two major observations. One major observation is when this one is going, this Catholic polarization is being followed, we are bisecting, we are basically going -- this line is not touching this position where you have attainment of passivity. So it's basically little away from this region, and here also if you see this line is not touching this line. This anodic polarization line is not touched by this cathodic polarization line at any positions except this position. So it is also this line is also going away from this line. So these two are very, very important observations.

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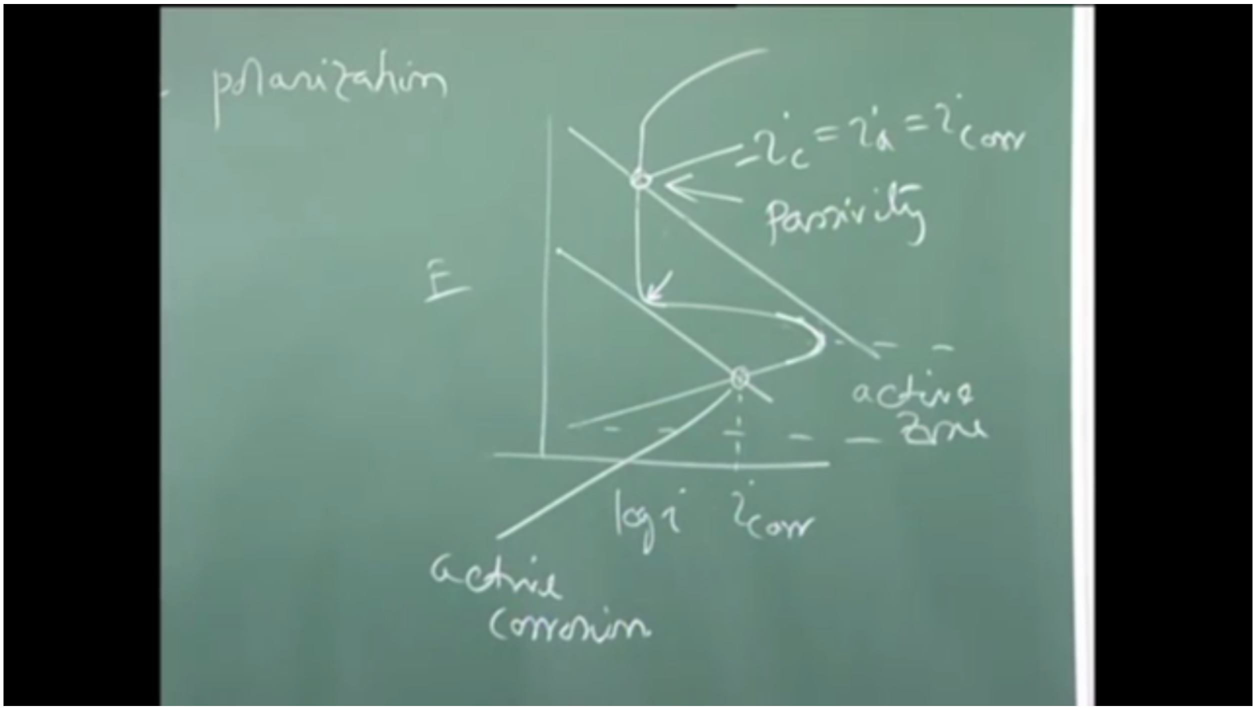

Now let us see what happens if it touches there. Now, initially, we have started with a freshly prepared surface. So the surface is not having any surface layer or the oxide layer, and this let's say in this case, if it is happening like this, the system will go along this line. It is though it -- I am drawing it, but the system actually following this track and then finding this point.

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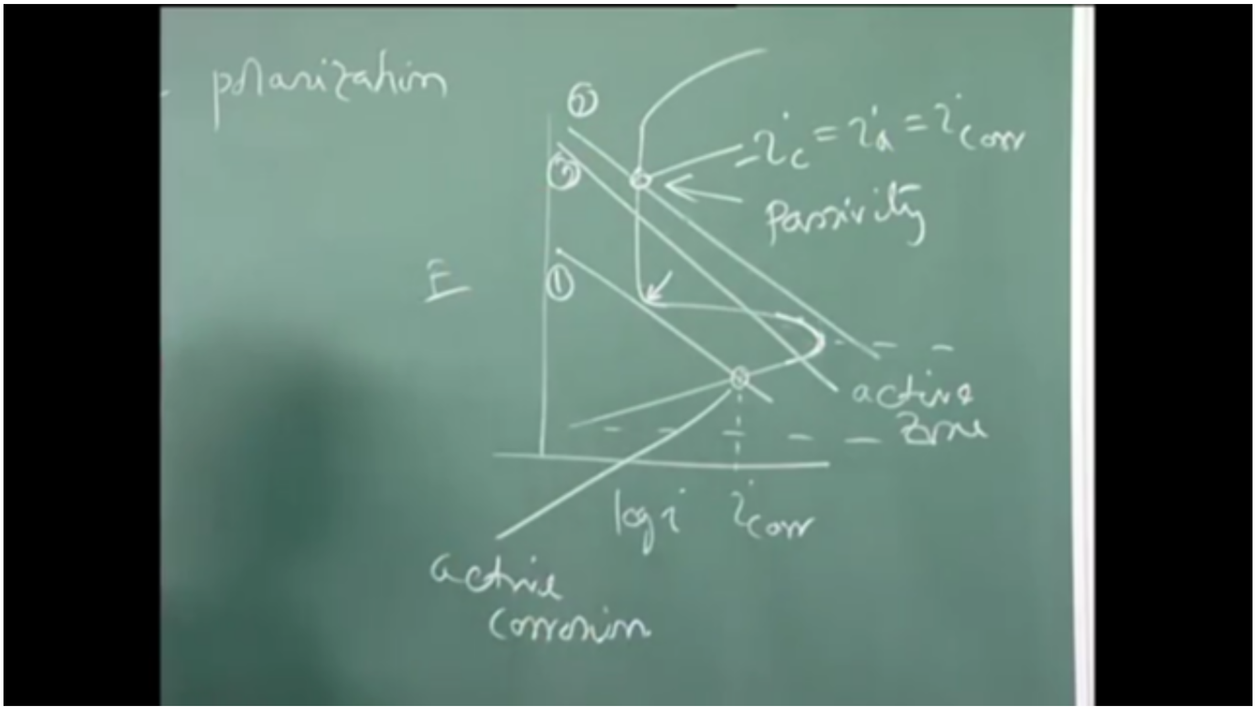

But let's say this line -- this line, let's say I have one more reaction cathodic reaction, this is let us say 1, this is 2, and this is 3. So what would happen in this case? I am very clear that this point because this is not touching here, so this point will remain here. This is not touching here. This point will remain here, but in this case what would happen? Now that part we have to see.

So let us say I have this reaction, this cathodic reaction is taking place in this case, first case. Let us consider the first case where the cathodic reaction is this, some cathodic reaction, which is going like this. Let's say hydrogen evolution reaction. It is happening like this and this is my line. So if I do polarization in case of zinc case, zinc case in HCl, I have this and if I would like to do the polarization, the polarization curve would look like this.

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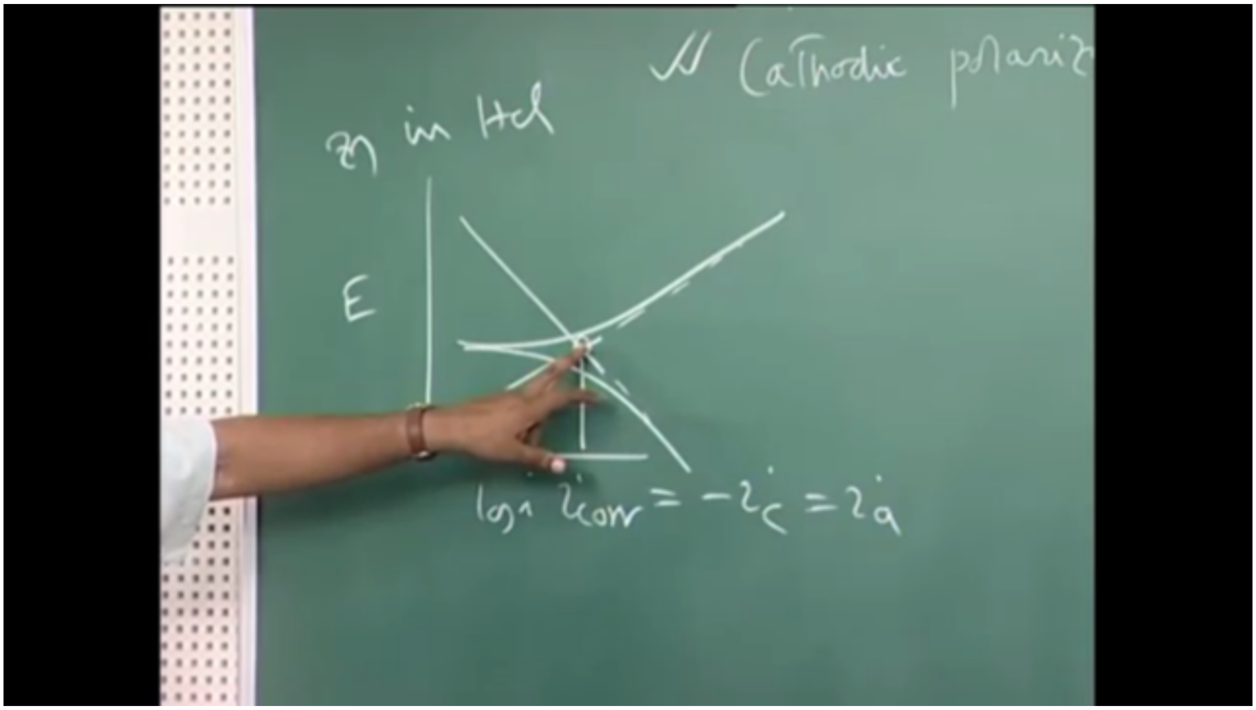

So polarization will start from this point because this is my new point of equilibrium. So from that point, I have to take the potential to the higher side or lower side, and accordingly, I will get this polarization line, polarization curve. This would be my new plot if I do the polarization. So that case, this line, how would we get this line? This line is nothing but this is my i_a minus i_c mod value. i_a is this line, and i_c is this line, and I am putting a mod because I am just taking the difference of these two values and that value is falling along this line.

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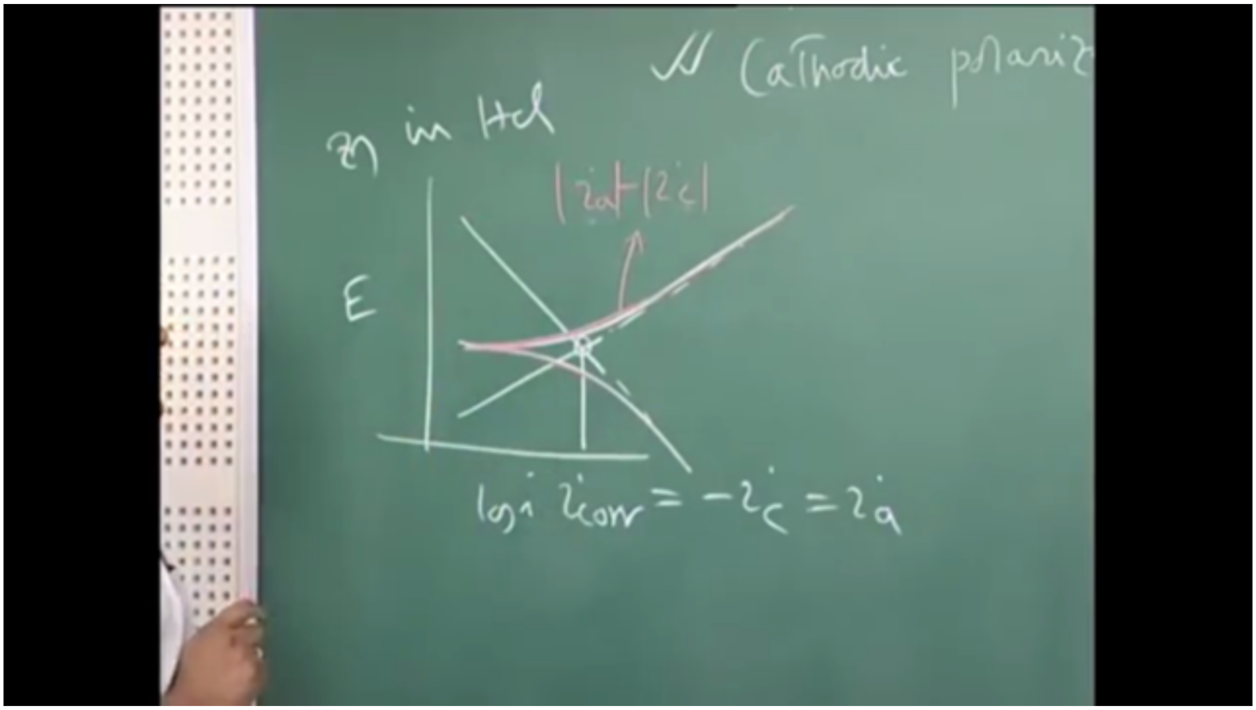

We have already seen in case of normal polarization, we have seen that this curve is basically the combination, addition of these two currents. Similarly, this is nothing but i_c minus i_a because the value of i_c along this line if you consider is more than i_a and this difference is coming like this.

So if you do the same operation in this case, so where from we'll start our polarization? We will start our polarization from this point because this is my new equilibrium point, and if I take this polarization, let's say from negative side, this would be my reference point then, this point if we go to the negative side as well as if I go to the positive side, how would my polarization curve look like? I'll see the polarization line like this. I'll see the polarization like this. Let me -- potential. This is log i. I will now next to that I'll plot the polarization.

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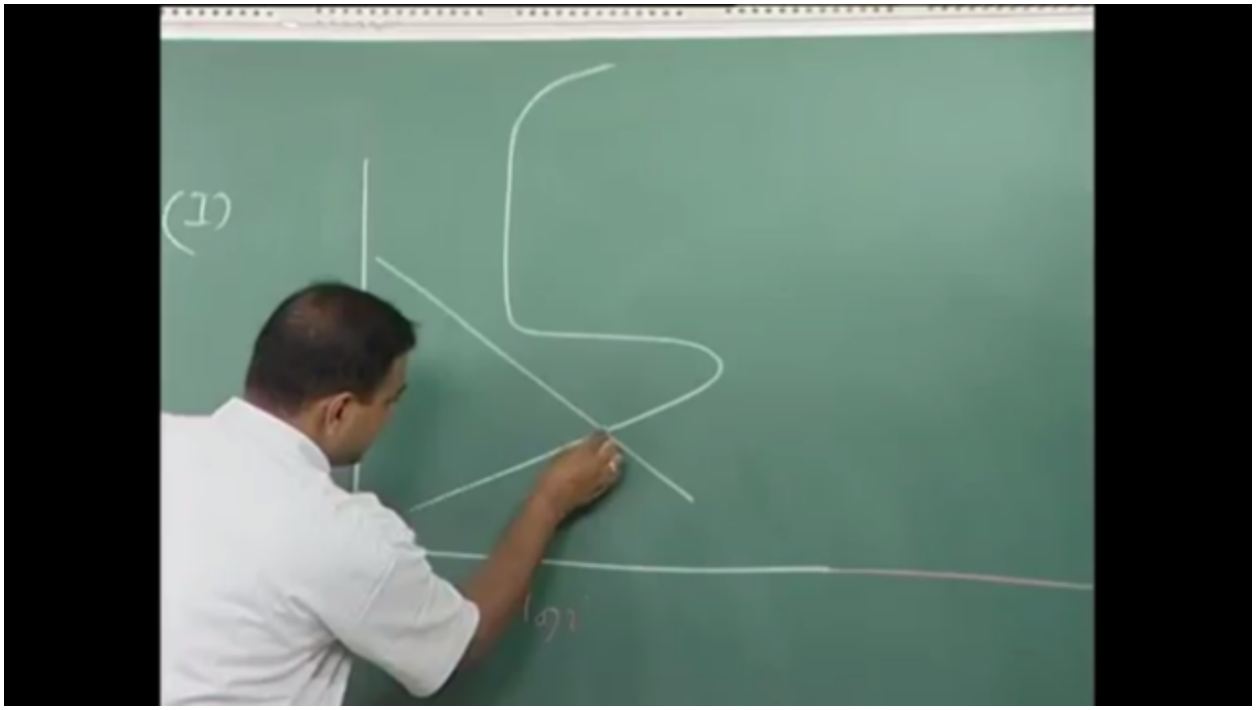

So this is my starting point and from that point, if I go to the negative side, this is my line and this is my reference line. This is my reference line or let's say zero line because this is my equilibrium point and this plot is this. So this is nothing but which one is $I_{a/c}$? This is my i_c and this is my i_a. So this is basically i_c mod - i_a and this curve will look like this.

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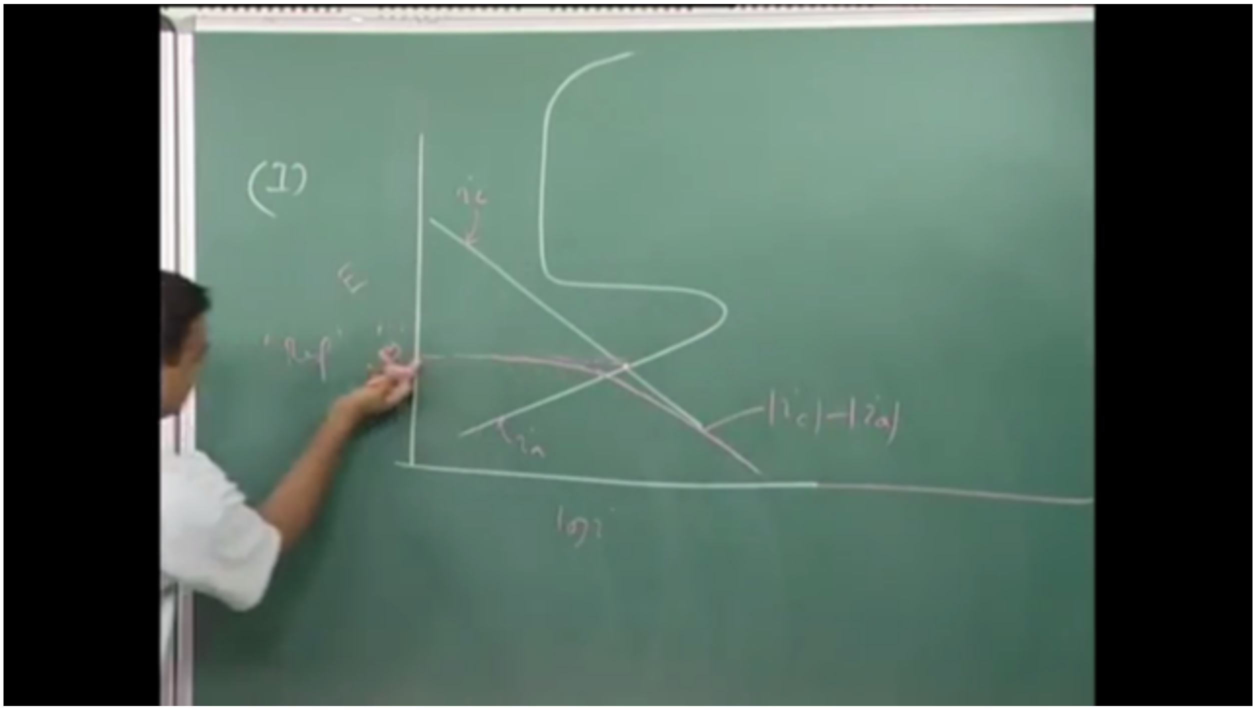

What would happen to the positive side? So you have to just make a difference between these two halves. So this point at different level we have to measure i_a - i_c. So the plot would be it will go like this, like this. Basically, this line is basically difference between these two lines. So if I would like to plot the polarization curve, log i guy versus potential, it would look like this. This is just like coming like this.

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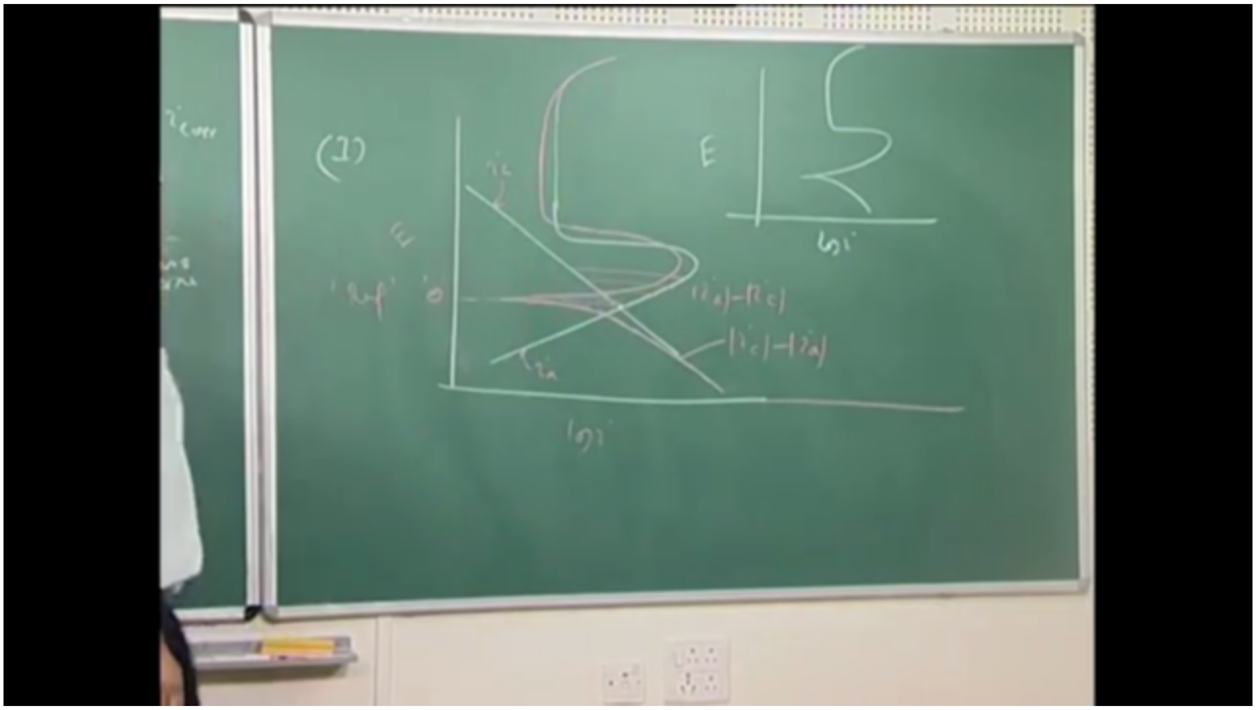

Now if the system is like this, second case, let me plot the second case here. If I plot the second case, which one is my equilibrium point or the reference point? This is my reference point from that because system will reach to that level.

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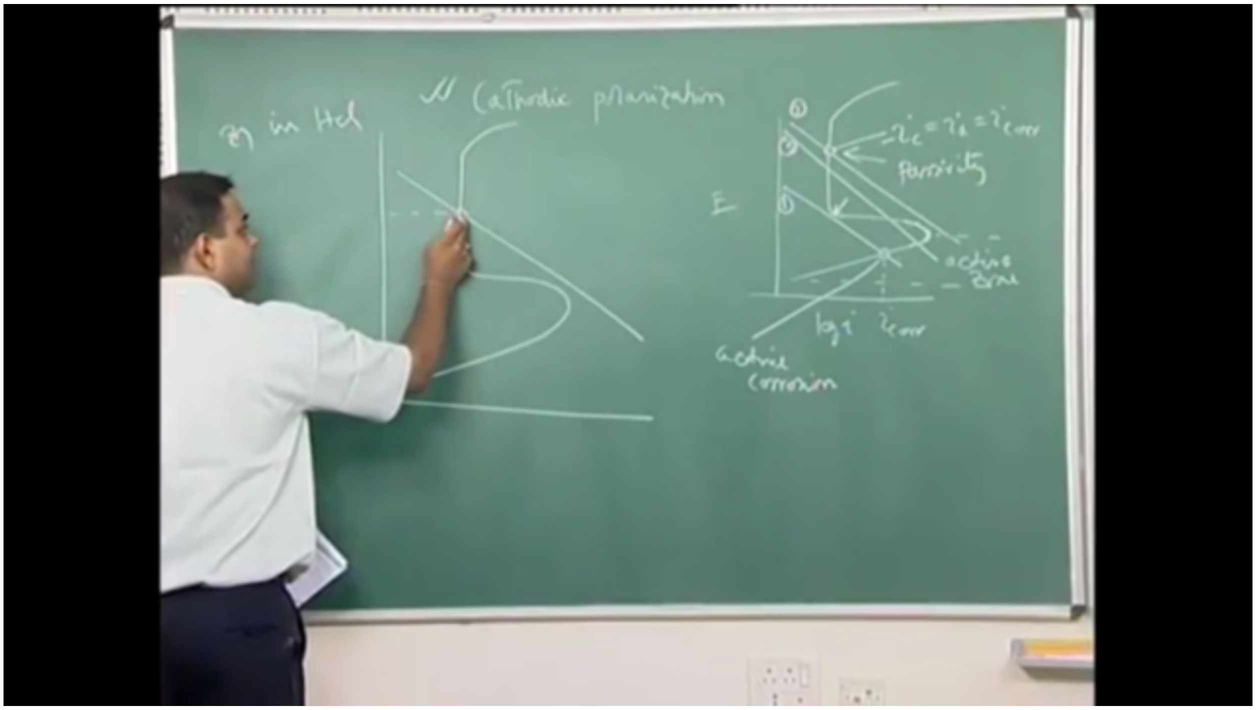

And now if you'd after that if you do the polarization, if you go to the negative side, the line -- line would move like this. So you have -- you have to see how this line is coming because of this difference between i_c and i_a because this line is nothing but i_c minus i_a.

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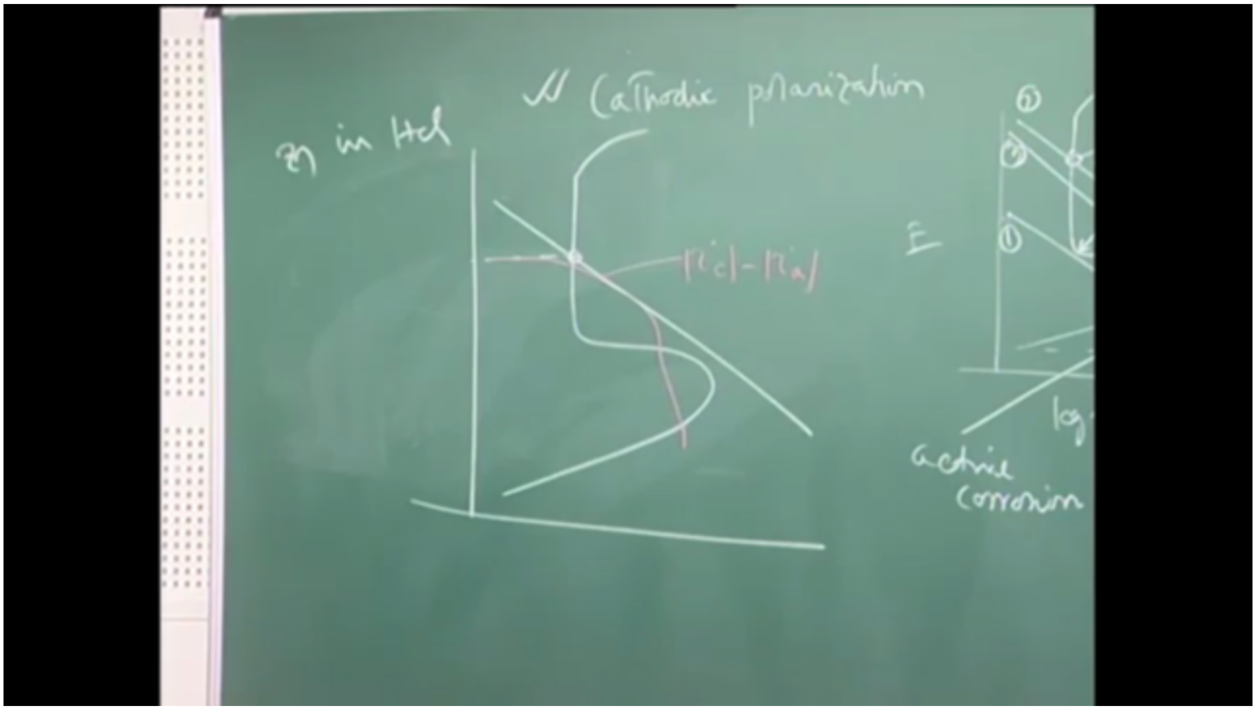

Now what would be the other side? Other side you have to just make a difference between i_a because this is ia, so this is i_a, this is i_c, so this other line would move like this. Fine. So that if you see the curve, this is log i, E. It would come like this.

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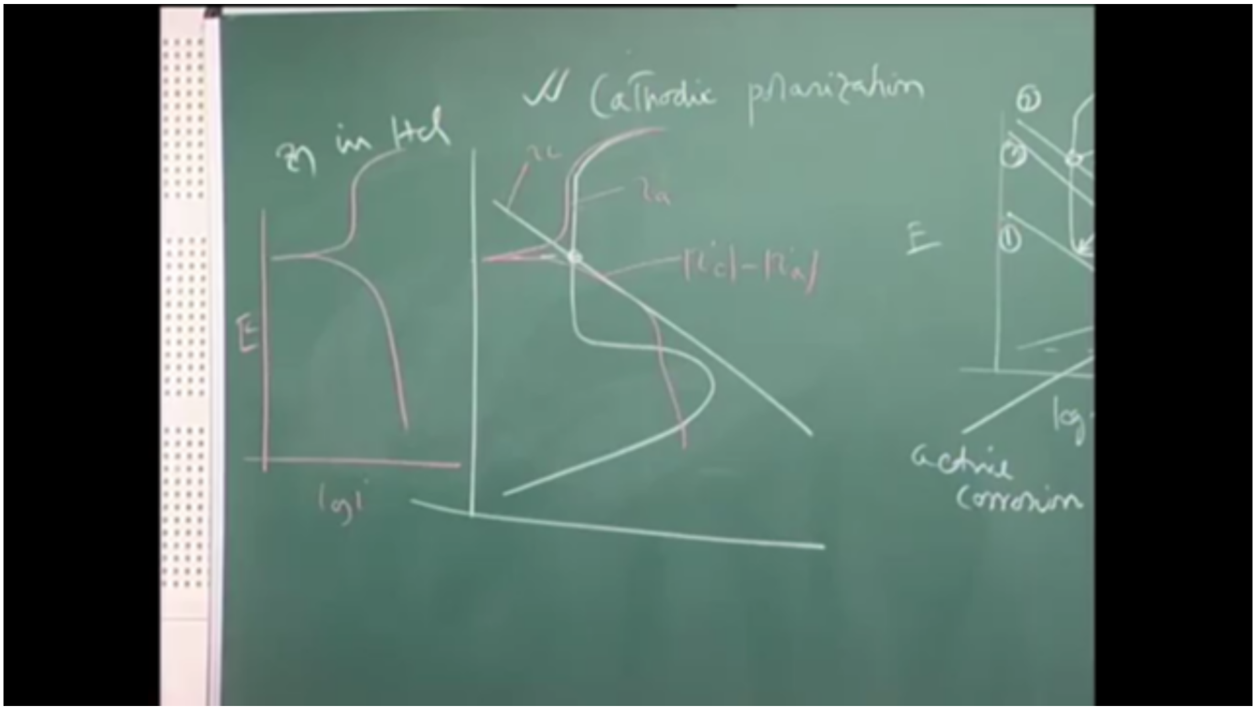

So this 2, this is second case. This is case 2. Now if you have this situation, if this cathodic polarization line cuts the anodic polarization line at three different points 1, 2, 3, then I have a peculiarity because I can take this is as reference, this is as reference, and this is as reference. So the curve would be little complex. So that case let me remove this line. Let me remove this part and let me see what happens if the cathodic polarization line cuts at three different points. This is log i. This is E. Now I have this is the situation.

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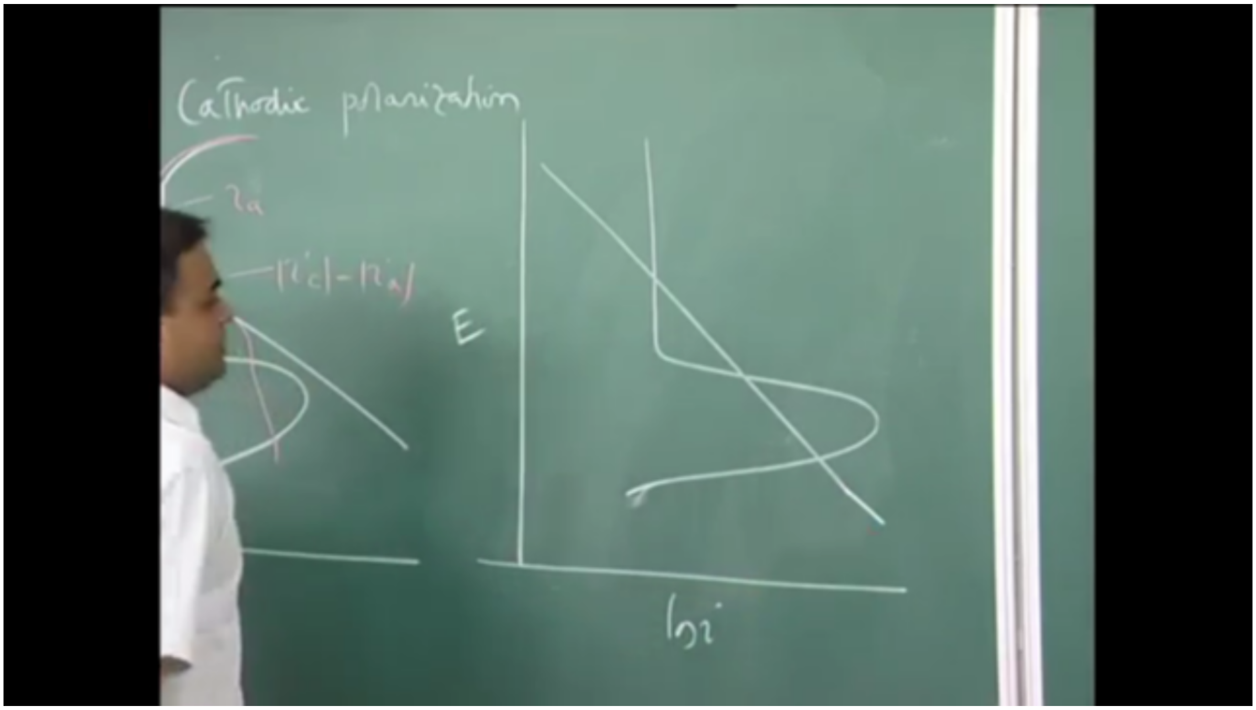

Now let me start this one as a reference. I will get one curve like this, and move like this, and once it crosses, once it finds this reference, then again, it will come back, and it will go up again depending on those two values. So this cannot go beyond this line. So this is again coming back and again, I am seeing another reference. So I am having one more loop like this.

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So my plot is becoming like this like this. So this region is called cathodic loop. This is stable passivity. This is active, but here this cathodic loop is coming because this cathodic polarization line is cutting this anodic polarization lines at three -- line at three different positions, and this is also a sort of unstable passivity.

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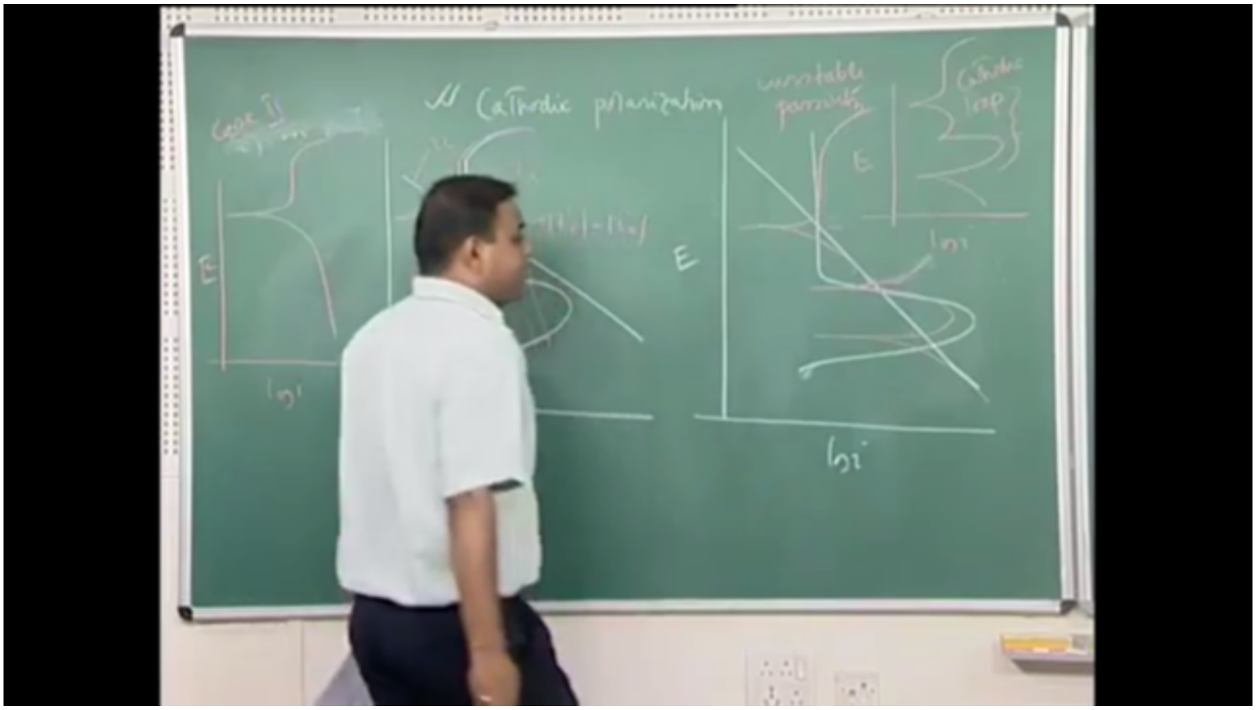

Why it is unstable passivity? Because it is cutting at active region. It is cutting in the passive region. It is also cutting in the unstable region. So the system will not decide where to be -- where to remain. It can remain here, it can remain here, or it can remain at this point. This point is it can remain either this point or this point because this is unstable zone. So if it remains there the system either will try to go there or try to come here.

Now whether it will come here that would be decided whether the system has stayed with the fresh surface or it can go there if the system will decide what system has come from the passive side already. If you -- if you bring the system from this side, then system will come here or if you bring the system -- bring the -- if you start the equation system -- if you start the polarization from here, then system will remain here. Fine.

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