Electroceramics
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Lecture - 8

So, welcome to this new class. So, we will just review the last class in which we discussed first the intrinsic, extrinsic defects.

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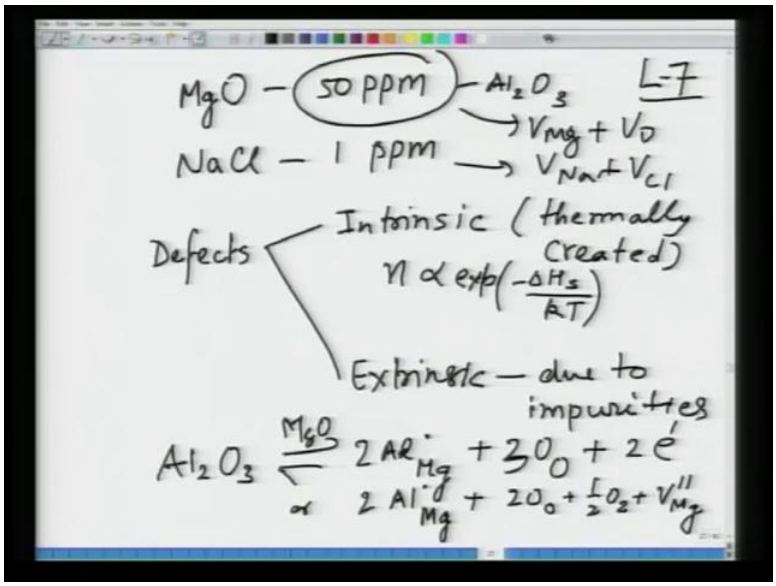

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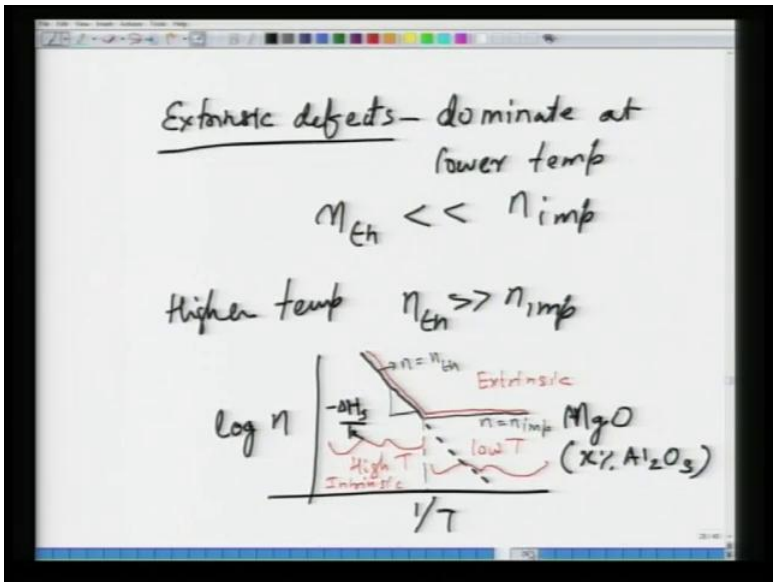

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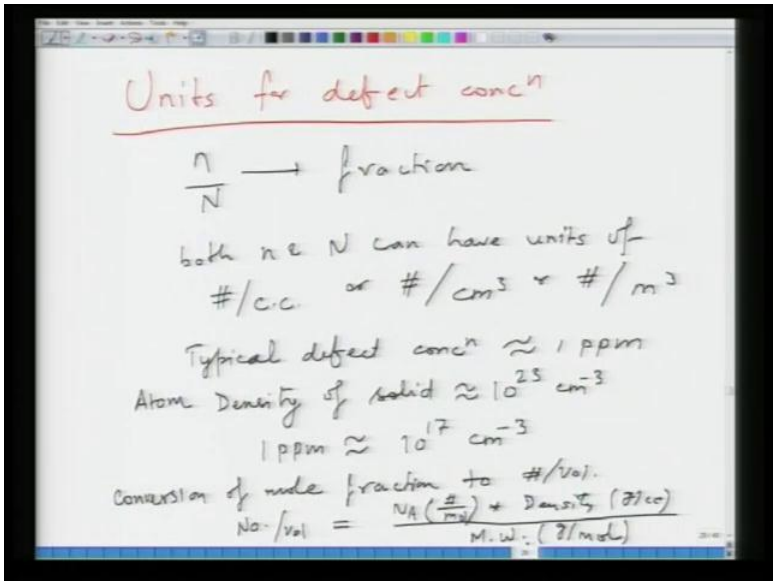

The temperature dependence and then we looked at what are the possible units for defect concentration.

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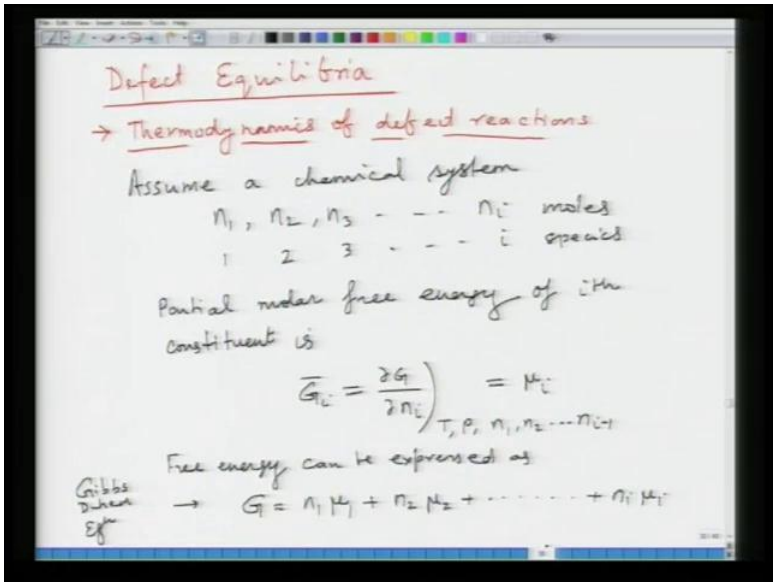

And then we moved on to a important topic of defect equilibria, which is basically the purpose of this, showing this was to basically establish the criteria that we can treat these defect reactions just like chemical reactions. So, the thermodynamics or defect reactions can be treated in a similar way as thermodynamics of a chemical reaction, in dilute solutions. So, let us say you have a chemical system with various number of species with

their respective moles, and then we worked out what was that Gibbs Duhem equation; following that we could work out what was the, we could work out that the k, which is the reaction constant, which is nothing but ratio of product of products, product concentrations divided by ratio or activities divided by product of concentrations of reactants.

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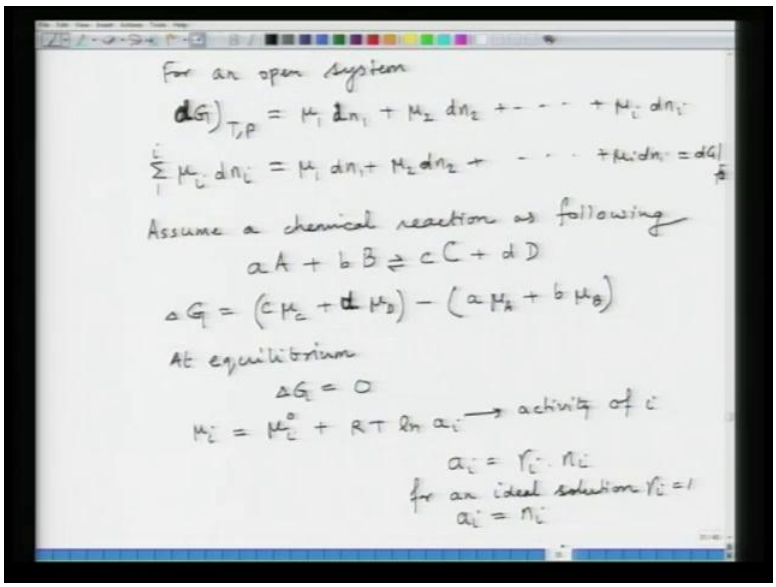

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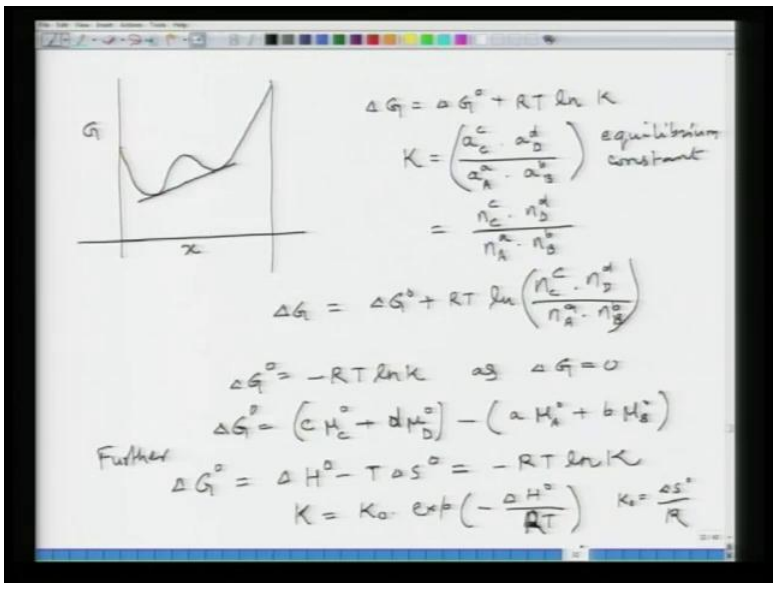

Now so, this is k but, we also saw that k is also equal to k is also equal to k naught exponential minus H naught by k T or R T which explains the temperature dependence

of k. So, this this helps us in establish the relations later on and we want to make a defect so, so you you can see the defect concentration now, not only will depend upon the temperature and now, we will establish that it also depends upon things like oxygen partial pressure.

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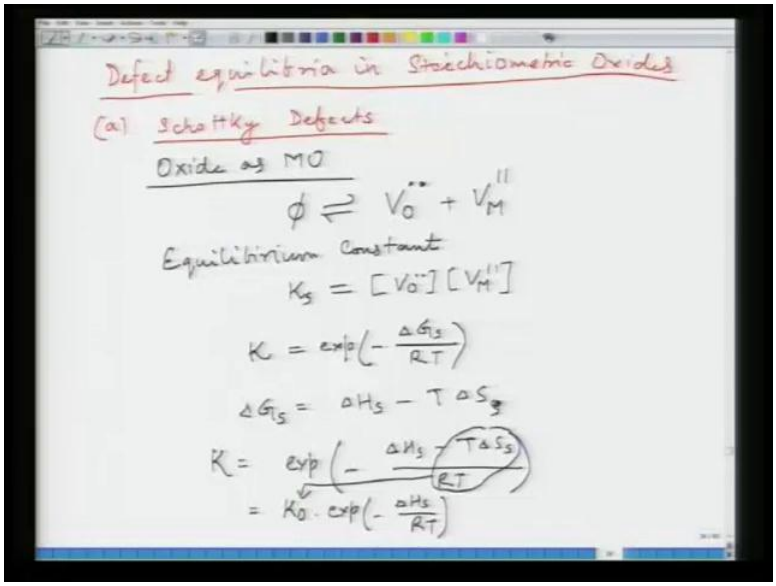

So, then we looks at the defect equilibria and stoichiometric oxides considering defect reactions like chemical reactions, where we assume that defects are very small in numbers hence these can be considered as solute in a solvent or dilute solution, solid can be considered as a dilute solution, where solvent is the host lattice and solute is the defect. So, first we took the case of Schottky defects and considered an example of M O and in that we worked out the reaction constant and we find that there is no temperature dependence of concentration in case of Schottky defects and concentration is purely dependent upon temperature and magnitude of delta H S.

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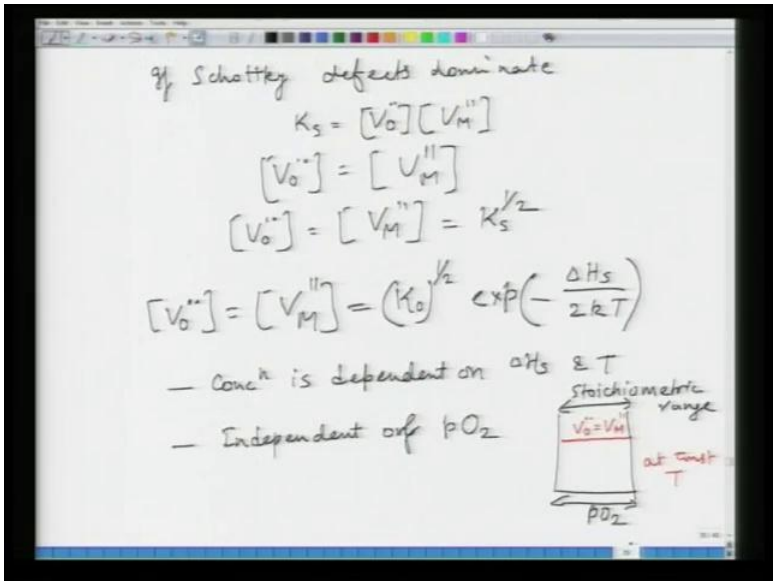

So, and this typically happens at intermediate partial pressures for oxygen and in these ranges your V O is equal to V M.

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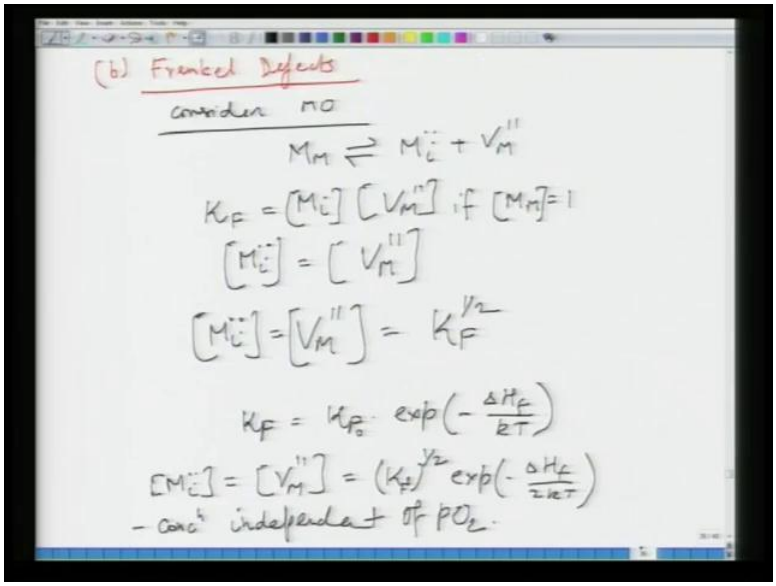

So, similarly, at the same was true for Frenkel defects as well, again the concentration is independent of partial pressure of oxygen and it is dependent upon temperature and defect formation energy. So, both the formalisms are similar with the conclusion that in case of Schottky in case of stoichiometric solids the defect concentration does not depend upon the partial pressure of oxygen, it only depends upon the temperature

because and then we looked at what is the equilibrium in equilibrium concentration in non-stoichiometric solids.

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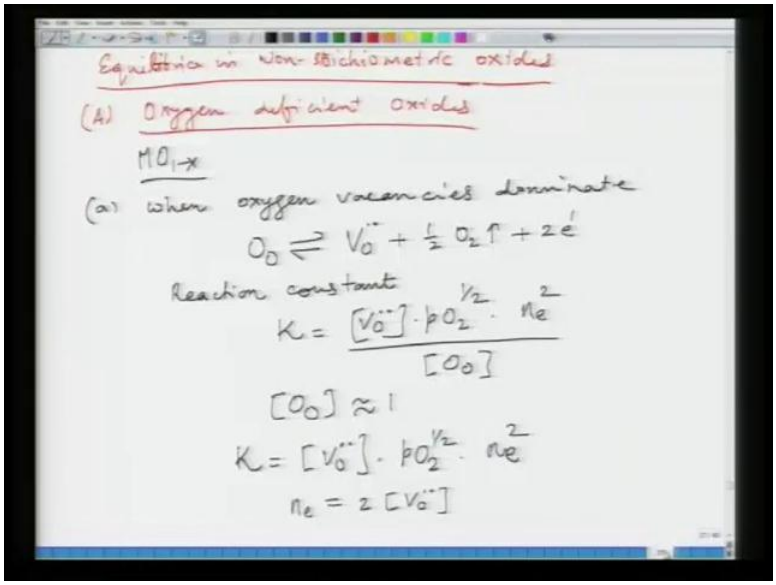

So, first we considered the case of oxygen deficient oxides. Example of let us say taking example of M O 1 minus x so, some oxygen is missing from the host lattice, those sites are empty. So, when oxygen so first case we took was when oxygen vacancies dominate in such a situation your the defect reaction can be written as like this and so O O gives rise to V O plus half O 2 plus 2 electrons and then we worked out that using the charge neutrality condition n e is equal to 2 V O and we can also write the defect we can also write the reaction constant which has dependence not only on concentrations and also on partial pressure of oxygen.

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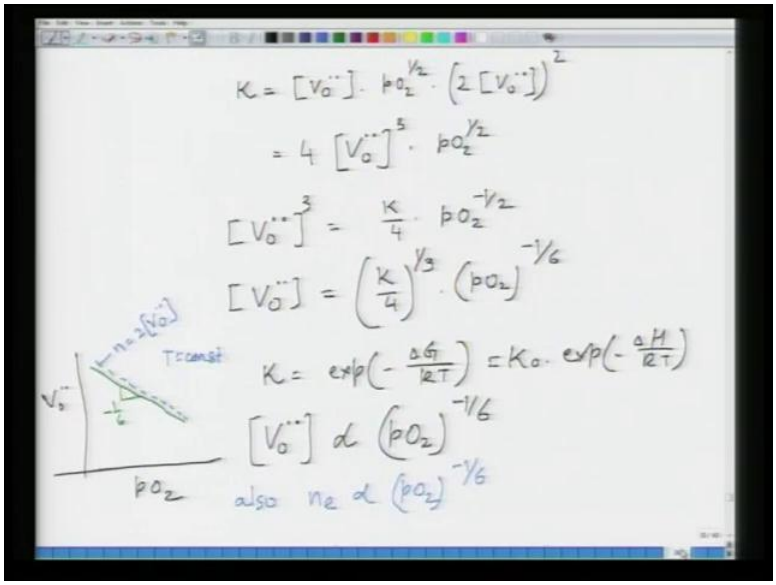

And if we just interchange various equations what we work out is the concentration of defects or electrons is proportional to p O to the power minus 1 by 6. Temperature dependence is of course, given by k now, at a fixed temperature what it means is that so, not only the concentration will depend upon k that is temperature as exponential minus 1 by 2 to the power 2 to the power 1 by 3, it will also depend upon partial pressure of oxygen to the power minus 1 by 6 so, if you plot these so V O is plotted as as a line with slope of minus 1 by 6, we know from n e charge equality that n e is equal to twice of V O. So, n e line would like just on top of this V O line and that will be 2 of v o and then we did the similar analysis for conditions when metal excess prevails for an oxygen deficient solid in such a condition your M i is dominate.

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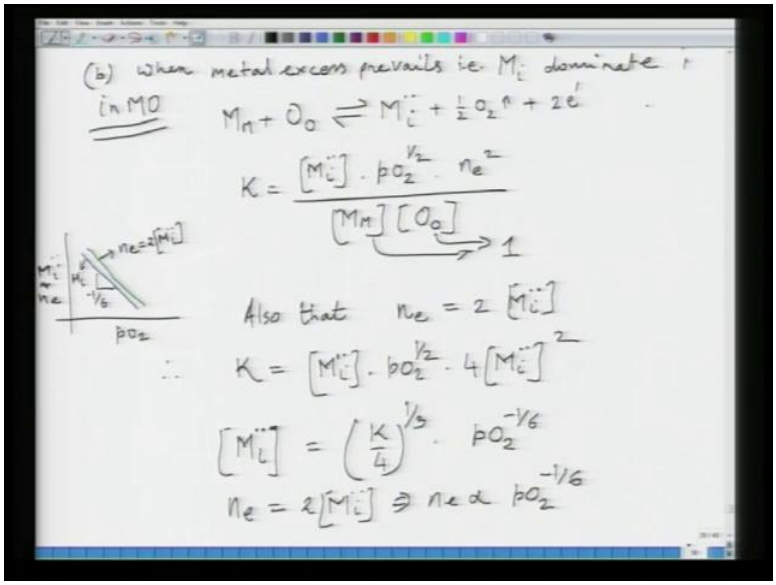

And again you can write the equation for k and again you can see that n e is proportional n e and M i both are proportional to p O to the power minus 1 by 6 and again you can plot the charge concentration, if defect concentration as a function of partial pressure of oxygen so, we were here now now the next case that we will take is in this lecture.

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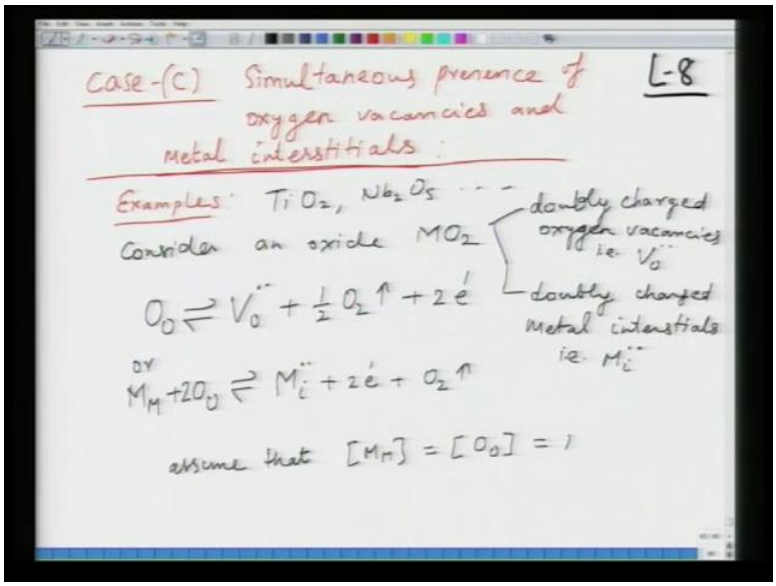

The next case now we will take is, case three or c I do not know what we used let us see if we used in the previous slide c so I will just rub three case c okay. So, in in this case we will take simultaneous presence of now so, far we have considered that only one kind

of defect can be present at a time now in this case you will consider simultaneous presence of 2 defects, which is oxygen vacancies and metal interstitials. So, this is the again for oxygen deficient solid now, examples of such a scenario this is a very practical scenario so, examples of such a such scenario is oxides like the T i o 2 or N B 2 O 5 so all these oxide shown not only oxygen vacancies but, also metal interstitials and this is simply because metal ions tends to be smaller titanium is a smaller cation so, as a result it tends to go to vacant interstitial sides.

Now so, we will take an example of so consider an oxide let us say M O 2 and this oxide has doubly charged oxygen vacancies, that is V O dot dot and it has doubly charged metal interstitials, that is M i dot dot. So, we have not taken completely charged metal interstitials for a convenience and I will explain you what the convenience would be, because if you take this M i 4 dot then you may not appreciate the exercise that we are going to do.

So, so this is the situation so for the first scenario when you have so, what is the defect reaction for the formation of these vacancies. So, oxygen at oxygen side goes out giving rise to 2 vacancies plus half of oxygen going out and as a result you create 2 electrons so, this is the reaction for oxygen going out or other reaction in case of metal interstitials would be M M plus 2 O O because it is M O 2. So, you have M i metal going to interstitial side. oh sorry I just take so, it is not completely ionised I will only take 2 electrons so, compensating that you will have 2 electrons and both the oxygens go out so, that maintains the side balance so this is the situation.

Now, let us say that defects are a smaller in number as compared to host lattice, assume we can assume that M M is equal to O O is equal to 1, assuming that it is a dilute solution. So, now we can write the defect reactions for we can write the reaction constant and now reaction constant assuming that M M and O were equal to 1. k 1 for the first reaction would be so, I can write this as k V O. Let us say, just to avoid the confusion I will just let us go to previous slide.

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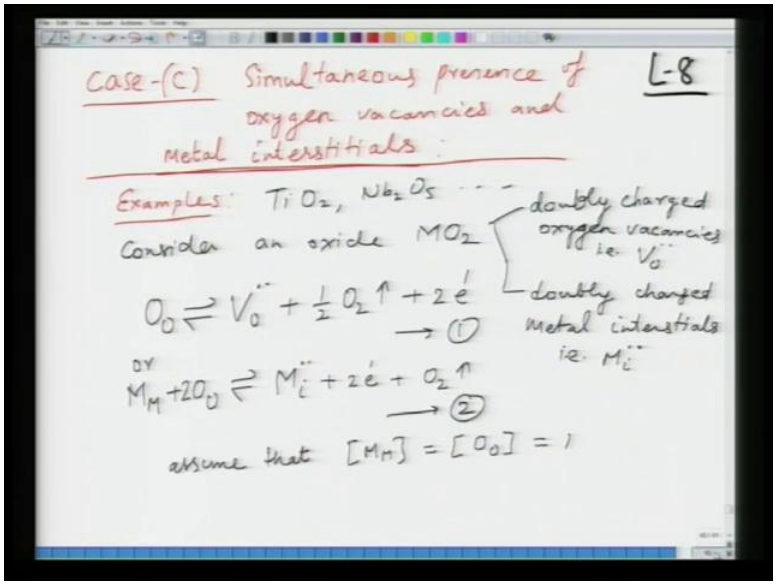

So, this is let us this is this is let us say reaction one and this is reaction two. So that makes it better.

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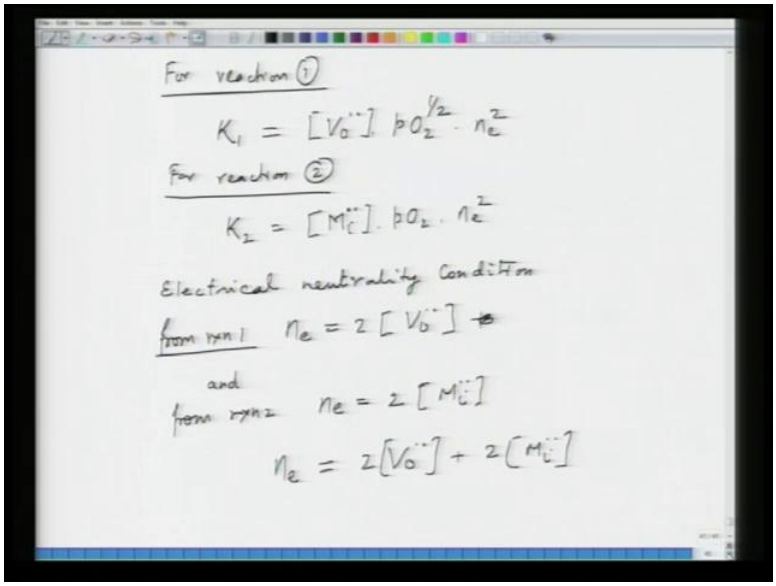

So, for reaction one we can write k 1 and k 1 will be equal to V O multiplied by p O to the power half multiplied by n e square and for reaction two you will have k 2 which is equal to M i multiplied by p O 2 multiplied by n e square. So, these are two expressions that we get. Now, what is the electrical neutrality condition? That says that end of reaction one number of electrons n e is equal to 2 of V O and for the second case so, this

will total basically and for the second case so, from this is from reaction one and from reaction two n e is equal to 2 M i, right. So, if both for simultaneously present then n e would be equal to 2 V O plus 2 M i so, the concentration of n e would depend upon the total concentration of these 2 so, let us say we take two limiting cases.

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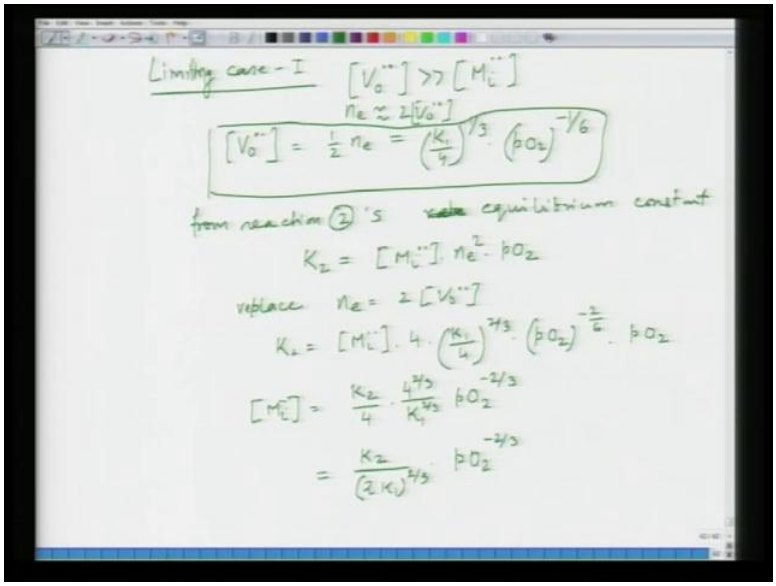

Limiting limiting case one now, this limiting case one is so you can have both V O and M i in various proportions so, we are taking two extreme conditions one condition is when V O is large in quantity another condition is when M i is in is in large quantity so, we will take first case which is V O is much larger as compared to M i in concentration. So, if that is the case then we of course, the reaction one would be more appropriate reaction so, that gives us the concentration of V O is equal to half n e is equal to because from the electrical inequality condition in that in such a situation n e will be equal to 2 V O so, V O will be half n e which will be equal to your k 1 you can work out from that is expression k 1 to the power 1 by 3 to the in multiplied by p O to the power minus 1 by 6.

So, you just go to previous expression so, this is what you have k 1 is equal to V O p O to the power half n e square, you replace the expression for n e which is equal to 2 V O so, it will become 4 V O square so, if you take p O p O 2 on the left hand side and just invert the equation then then it will become V O cube is equal to k 1 by 4 multiplied by p O to the power minus 1 by 2 and then you just so, V O cube was equal to that. So, when

when you want to take for V O the right hand side will become to the power 1 by 3 so, this is the expression for V O you get here. Okay.

Now, what will be the concentration of now, let we are saying that V O is very large in number, but still what is the concentration of M i so, M i concentration will still be given by reaction two, but in that reaction now, n e is governed by concentration of V O so, n e is no longer a function of M i. So, from reaction two we know from reaction two's rate constant or reaction constant or equilibrium constant, we know that k 2 was equal to M i into n e square multiplied by p O 2 so, here this. So, you replace n e is equal to 2 V O because in this condition n e is not dependant upon M i it is only depended upon V O because V O is much larger than M i now, k 2 will become M i dot dot multiplied by what is n e n e square so, it will become 4 into k 1 to the power 2 by 3 into p O to the power minus 2 by 6 into p O 2.

Now, all you have to do is that you have to rearrange so, M i will become k 2 divided by 4 into so, this will become 1 by 3 so, this will be p O 2 the power 2 by 3 which on the other side if it goes it will become p O to the power minus 2 by 3 and this so, k 2 k 2 k 2 divided by 4 and then this becomes 4 to the power 2 by 3 divided by k 1 to the power 2 by 3. So, I have just taken everything on the other side and later next question for M i M i dot dot, which will reduce to k 2 divided by 2 k 1 to the power 2 by 3 so, this is basically 4 to the power 1 by 3 so, 4 to the power 1 minus 4 to the power will be 4 to the power 2 by 2 will be equal to 2 to the power 2 by 3.

So, so k 2 and k 1 so, M i so in this regime when V O is much larger than M i then V O is equal to k 1 by 4 to the power 1 by 3 into p to the power minus 1 by 6 so, V O varies log V O varies as with the slope with respect to p O 2 with the slope of minus 1 by 6 on the other hand M i varies with the slope of minus 2 by 3 and what is the boundary condition? The the condition beyond which this is valid so, we are saying that if you want to determine that partial pressure of oxygen beyond which this is valid in that case we are saying that since V O is greater than M i so, what is V O? V O is k 1 by 4 to the power 1 by 3 into p O to the power minus 1 by 6 is greater than k 2 divided by 2 k 1 to the power 2 by 3 multiplied by p O to the power minus 2 by 3.

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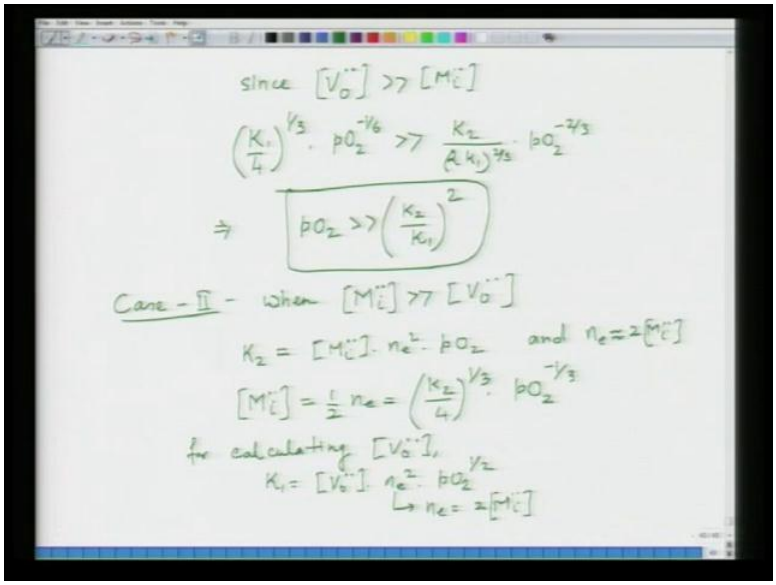

So, this is the condition that we establish and so, if we now rearrange this what you will find is that p O 2 is greater than k 2 divided by k 1 square so, this is the pressure beyond which this condition will satisfy. So, this was case one, limiting case one when V O is greater than M i second case we will take is when M i is greater than V O so, we have to determine this condition so, naturally this condition will prevail under this so p O 2 has to be lesser than k 2 by k 1 square. You can verify that using again this equality when you calculate the concentration.

So, now we know from so, first we will calculate what is M i now so, from reaction two we know k 2 was equal to M i multiplied by n e square into p O 2 so, M i now will be equal to and under such a condition n e will be equal to 2 of M i because it is much more larger than V O so, M i M i under such a condition will be equal to half of n e 3. This now you can do, I am not going to do the whole exercise so, you all you need to do is, that you need to put this expression here and just rework the parameters on either side to work out an expression for M i in terms of p O 2.

So, this is one expression and now for calculating V O we know that k 1 was equal to V O into n e square multiplied by p O to the power half and now, this n e now is equal to 2 of M i so, we will just move to next slide.

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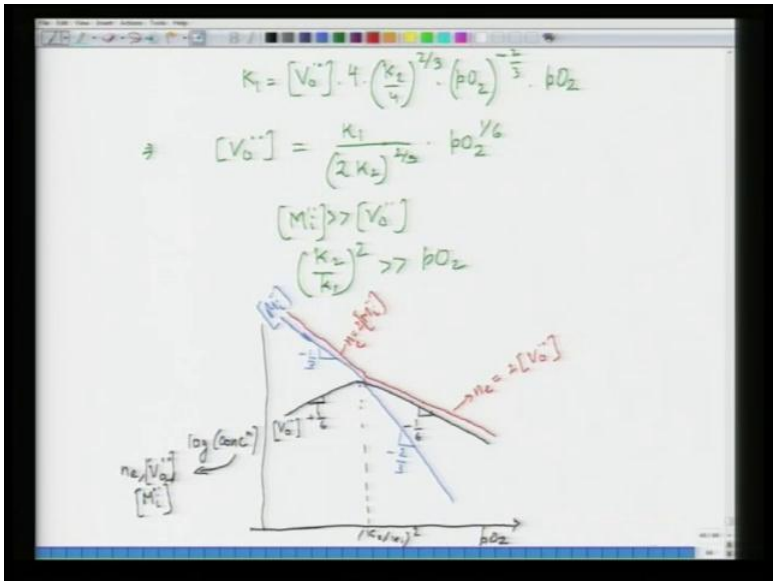

So, we know that k 1 is equal to V O into n e n e square so, n e square will be 4 M i square and M i square will be k 2 divided by 4 to the power 2 by 3 multiply by p O multiplied by p O to the power minus 2 by 3 multiplied by if you go to previous expression previous page k 1 was V O we have replaced n i n e with two M i and M i is equal to this. So, we have just replaced this so k 2 by 4 to the power 2 by 3 into p O to the power minus 2 by 3 multiplied by 4 multiplied by p O 2 now, if you work out the expression in for V O in terms of p O 2 what you will find is V O is equal to k 1 divided by 2 k 2 to the power 2 by 3 into p O 2 the power 1 by 6.

So, we can see here that in this regime i, m i has a pressure dependence of minus 1 by 3 to the p O to the power minus 1 by 3 and v o has a dependence of p O to the power 1 by 6. So, again now you can calculate the boundary so, we know that since M i is larger than V O I am just putting the value of V i and M i and V O and what you will find is k 2 by k 1 square is greater than p O 2 which is nothing but you worked out from the previous expression now, you can so here you can see that v o increases as the partial pressure of oxygen decreases and but at different rate as compared to previous exercise sorry V O increases as you increase the partial pressure of oxygen up to certain point and then it decreases when V O is much larger.

So, we will just now plot this so, this is log of any kind of concentration so, which means concentration will mean your n e V O and M i and this will be your p O 2. So, the

boundary we get it is at so, let us say we start at a arbitrary point and this boundary is equal to k 2 by k 1 square. Now, what we see is that p O 2 larger than k 2 by k 1 is when k when k 2 by k 1 square is larger than p O 2 which means the smaller p O two's in that case what we have is our V O varies as this so, this will be plus 1 by 6 for V O and how does how does V O varies how does the V O vary after when when p O 2 is larger, in that case it varies as like this. So, this will be minus 1 by 6 this is the V O line now how does let us say blue line now, how does M i vary? M i in this region if you go to previous slide it varies as minus 1 by 3 if you go to previous slide it varies as minus 2 by 3.

So, now we just take it so it it varies at minus 2 by 3 here and and this region it varies at minus 1 by 3. So, this will be your M i and n is nothing but if you want to plot n so, n is nothing but in this region 2 of M i and in this region it is 2 of V O. So, up to this point n is equal to 2 of M i and in this region it is equal to n e is equal to 2 of V O. So, this is kind of an equilibrium diagram where you get a boundary the the the transition point and the conditions away from the boundaries in both the directions. In one condition your M i is a dominating defect in another condition your V O is the dominating defect and in between both of them exist together.

So, basically what it means is that you start drawing the lines from the far of regions and then let them meet at the at the juncture junction which is nothing but p O 2 is equal to k 2 by k 1 square. So, I hope this gives you an idea about how to establish these various equilibrium conditions, you can take single defect situation, you can take multi defect situation and see how they co-exist under various conditions of partial pressures of oxygen.

So, next we will take into account what is the scenario in case of metal deficient oxide. So, far we have considered scenarios of defect we have worked out the defect equilibria only for a stoichiometric only for oxygen deficient oxides and stoichiometric oxides now what is remaining is metal deficient oxides and we will do it very briefly because now, that you understand the fundamentals of how to work out defect equilibria in case of oxygen deficient oxide the approach is similar in case of metal deficient oxide. So, we will only take one case in such a such condition. So, here now let us see how we were we had the numbering so, this is case c so A capital A so, we will take capital B here.

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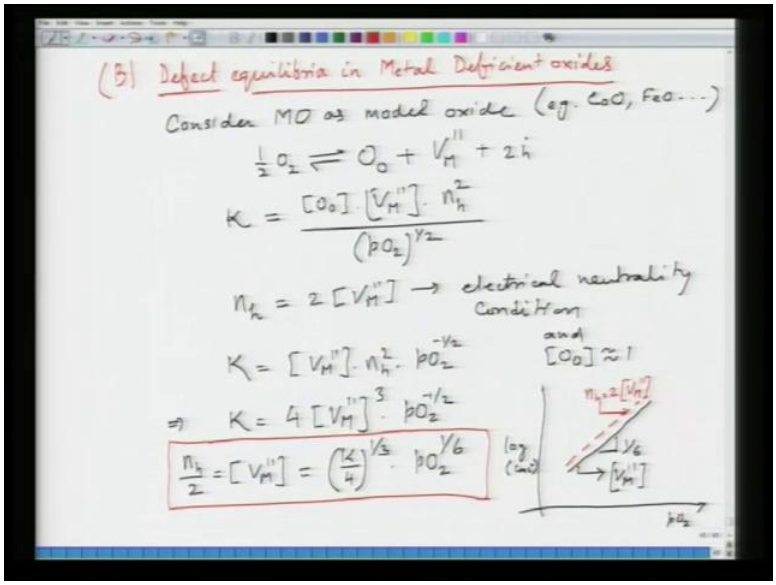

And we will take defect equilibria in metal deficient oxides. So, again consider for example, M O as model oxide all right. So, as now assuming complete ionization of vacancies for the formation of metal vacancies what you write is half O 2 now, this will happen in for example, example our C O O, F e o etc. So, you write half O 2 is equal to O O so, oxygen has gone to oxygen site in order to maintain the side balance you need to create a metal side.

However, there is since it is metal deficient that will be metal vacancy and that would be doubly charged and that would be compensated by creation of 2 holes and the equilibrium constant for this will be k to be equal to O O multiplied by V M dash dash multiplied by n p n H square divided by p O to the power half. Now, we know that from electrical neutrality condition n H is equal to 2 of V M so, as a result your k will become and and also assume that and O O is equal to 1 because it is very large in number.

So, as compared to defect concentration so, it becomes k becomes V M double dash multiplied by n H square multiplied by p O 2 and so, now what you can do is that k in such a situation will be n H if you replace by 4 V M cube multiplied by p O 2 sorry p O to the power minus half minus half so, V M double dash. So, just see there was a mistake here it should be so, if it goes there now it will become k by 4 to the power 1 by 3 into p O to the power 1 by 6 so, this is the expression for V M and this is also equal to since n H is equal to 2 V so, this is n H by 2 so, this is the expression for pressure dependence

for whole concentration. So, if you just draw a plot here, plot would be something like this so, you have log of concentration and this p O 2, so this is your oops sorry it is positive slope so, if this is your V M line then your whole line will be right on top of this V M so, this is how it will vary the scales are arbitrary we will see they are relative to what is beyond or what is after.

So, what is before, what is after. So, we will see that so, this is the case in case of metal deficient oxides when when metal vacancies are dominating defects you can do the similar exercise for conditions when so I will just leave it to you.

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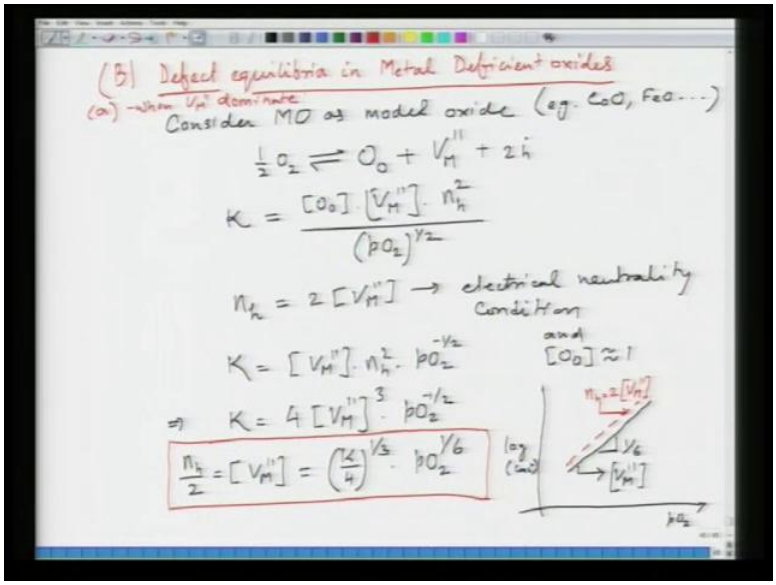

For example, I will just mention the condition here condition a when V M dominate and I will leave it to you as an exercise, solve it do the defect equilibria calculation for condition 2 when O i dominate and condition c when mixed presence of O i and V M.

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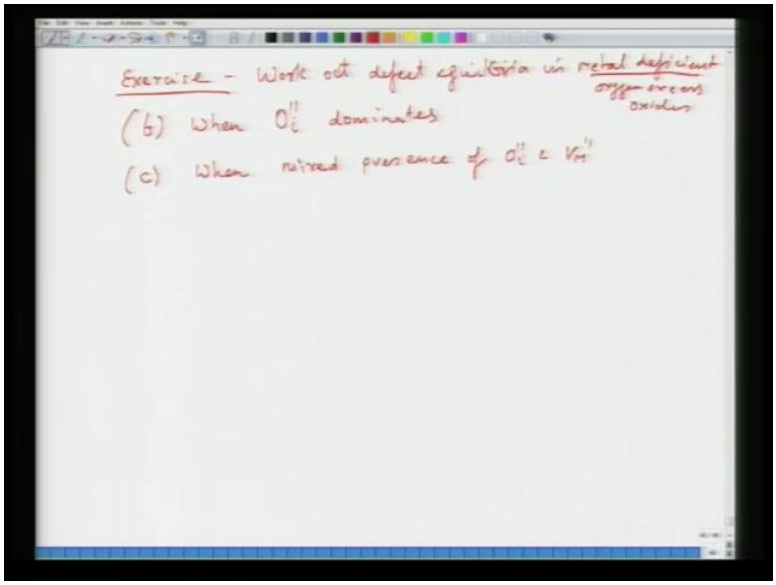

So, the analysis is very similar to what we have done in a previous case for oxygen deficient oxides and this would be similar in case here as well. Now, if you remember in the in the case of if you remember in the case of, yes in this case we choose an oxide M O 2 with when we took simultaneous presence of oxygen vacancies and metal interstitials, we took an oxide M O 2 that was for an purpose. What happens is that in these materials if you had M i completely ionised which means M i 4 dots present or if you had taken an oxide M O which is completely ionised defects then you would not see this transition as a result when you make the measurements conduct now these as we will see later on these are all all these plots which I am plotting at the moment they can be obtained by making conductivity measurements as a function of pressure oxygen partial pressure so, when you when you make those conductivity measurements you are not able to ascertain, you you would not be able to get this sort of those sorts of differentiation which you get in these cases.

So, this kind of analysis is applicable in cases where it is very explicit that how what kind of defects are present at what kind of oxygen partial pressures. So, just looking from the analysis or the derivation point of view, it is easy to derive it for cases like these where such as in M O 2 you have M i dot dot etc, but it does not work the approach similar approach does not work for for instance you want to do for M O, where both the defects are completely ionised or even for M O 2 when where both the defects are completely ionised.

So, one has to be careful, if you if you want to go into details of this you can go you can go through the book of per Kofstad which is known as stoichiometry in oxides which I have listed in the bibliography so, but this is a very useful approach you can see where you can find out the differentiation and where you cannot, so that you can work out yourself by taking various examples of various oxides. So, now so this is as a exercise I left it to you so basically work out defect equilibria in metal deficient slash oxygen excess oxides in under following conditions.

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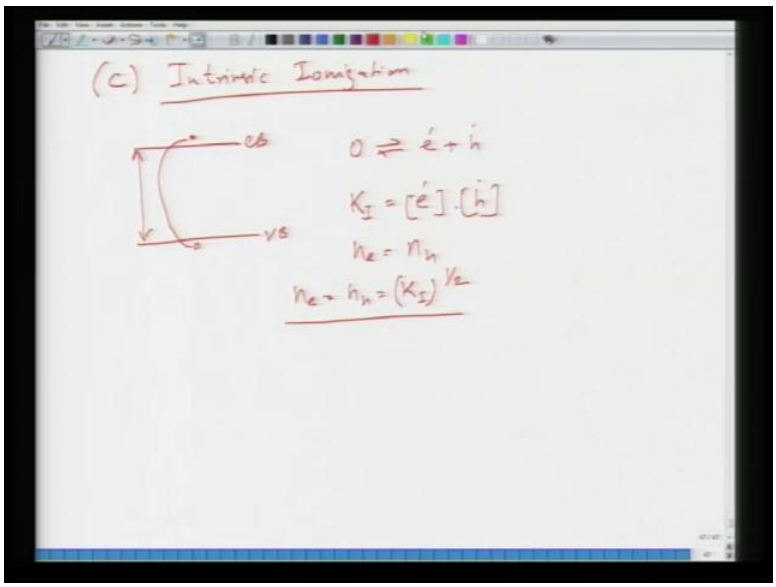

Now, the third case in this series would be intrinsic ionisation. An intrinsic ionisation as we have seen it has nothing but formation of electrons and holes. So, basically you have this band gap of the material n e oxide so, the electrons from valence band so, this is your valence band this is your conduction band. So, electrons from here jump to valence band gives rise to electron in the conduction band and hole in the valence band so this leads to formation of electron hole pair and the reaction would be null electron and hole and equilibrium constant for this would be e dot h and since n e is equal to n h, n e is equal to n h will be equal to k I to the power half. So, you can see in such a condition when this happens at moderate rate partial partial pressures of oxygen under such conditions you can see that n e or n h is only dependent upon k I which is temperature dependence as you know and it is independent of partial pressure of oxygen.

So, now what we will do is that so far what we have seen is that we have seen oxides of specific kind. We have considered cases of metal deficient, we have considered the cases of oxygen deficient, we have considered the cases of stoichiometric conditions, typically as you can intuitively think that metal deficiency or oxygen excess would tend to occur at higher oxygen pressures. Oxygen deficiency would tend to occur at lower oxygen pressures and intermediate oxygen pressures would determine the stoichiometric condition. Now, what we will do is that we will consider one particular oxide and we will look at the defect equilibria for this particular oxide under three conditions of oxygen partial pressure.

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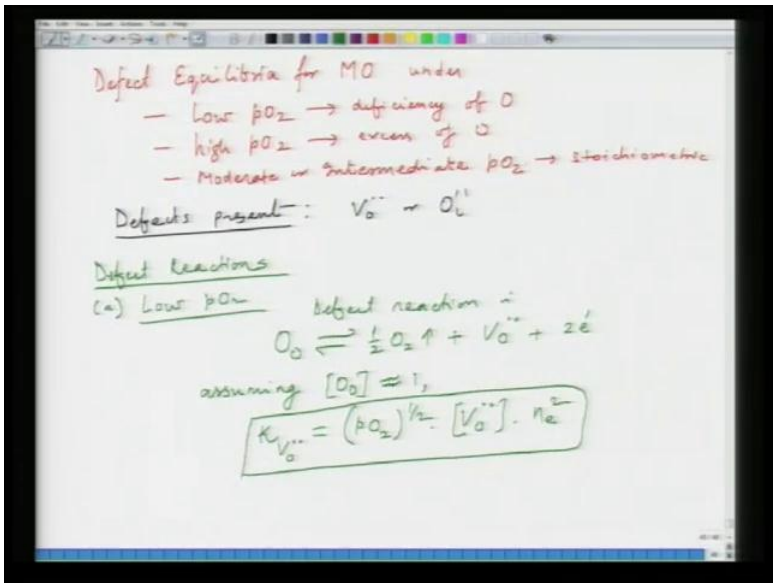

So, the so basically so, we will consider defect equilibria for an oxide M O for M O under number 1 low p O 2 number 2 high p O 2, number 3 moderate or intermediate p O 2 which is typically in most cases atmospheric pressure intermediate p O 2. So, this condition would typically determine deficiency of oxygen, this condition would typically determine excess of oxygen and this condition would typically determine stoichiometric condition. Now, deficiency of oxygen will also mean excess of metal and excess of oxygen will also mean automatically not automatically but it would be metal vacancy. So, assume that the defects which are presents are either oxygen vacancies or oxygen interstitials and let us say both are doubly charged completely ionised.

So, let us say what is what are the defect reactions first. So, first we will see just change the colour so, defect reactions so first we will write for low p O 2 case so at low p O 2, I am not considering M so if this was V O then you would have complementary defect M i M i or you can have V M so, we are just considering oxygen at the moment you can consider others as well but let us not complicate let us just simplify otherwise it becomes little complicated so just to simplify, we have chosen only one kind of dominating defect in one region. So, at low p O 2 the defect reaction would be O O which means oxygen present in the lattice going out giving rise to oxygen vacancy plus two extra electrons.

Now, assuming O O was equal to 1 almost equal to 1 and so this would be so, we can write k for this reaction. So, let us say this is k V O so k V O would be p O 2 to the power half multiplied by V O dot dot multiplied by n e square, I am not writing it in terms of n e, I am just writing the whole without replacing for n e so, this is the first expression that you get.

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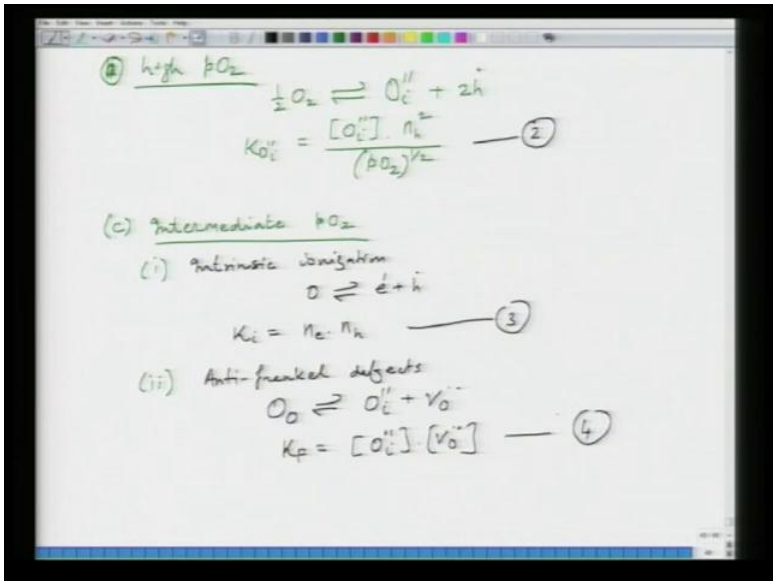

Now, the second expression we will write at at high p O 2 so, at high p O 2 the defect reaction is half O 2 is equal to O i plus 2 h and as a result the reaction constant you can work out as k O i to be equal to O O i dash dash dash dash multiplied by n h square divided by p O to the power half and third condition is oops change the order here a this should be b this should be c intermediate, what am I writing then I get p O 2 so under intermediate p O 2 there are two possible scenarios. Under intermediate scenario is so

number first number 1 is intrinsic ionization, given the two creation of electrons and holes well reaction in this case would be e plus h. So, under such a condition k i would be equal to n e multiplied by n h and the second condition that is possible is, so either you can have intrinsic ionisation which dominates so, stoichiometric defects could be of electrons and holes or the stoichiometric defects could defects could be Frenkel defect or anti Frenkel defects.

So, the reaction here would be O O is equal to O i plus V O. So, basically what you can see here is the defects that we have considered at higher and lower p O 2 is they have to be in accordance with the defects that are going to be present at in stoichiometric conditions as well so, that you get a continuity in defect concentration as a function of partial pressure of oxygen. So, you can see that intuitively now, if you had chosen Schottky defects here Schottky defects would give rise to V M and V O so you would have continuity for a V O but V M would not have so, either you replace O i here with the V M so, if you had Schottky defects dominating in that intermediate pressure range in that case you would have to choose V M in the higher pressure regime. So, you understand what the logic is? The logic is whatever defects you choose in the intermediate low and high pressure regions there has to be continuity in the defect concentration.

So, if you choose one particular kind of stoichiometric defect in stoichiometric region then that particular defect grows or reduces in the concentration at higher or lower pressures. So, that that makes sense and that also makes our life little easier so, for this reaction we can write k F is equal to O i multiplied by V O and so from these 4 expressions for so this is let us say 1 just let us say it is 2 let us say it is 3 and this let us say is 4 so, if you combine these expressions combining above expressions gives us k F multiplied by k i square is equal to k V O multiplied by k O i just a second, show you some that is easy to verify. You can do that yourself, so this could be equation number 5.

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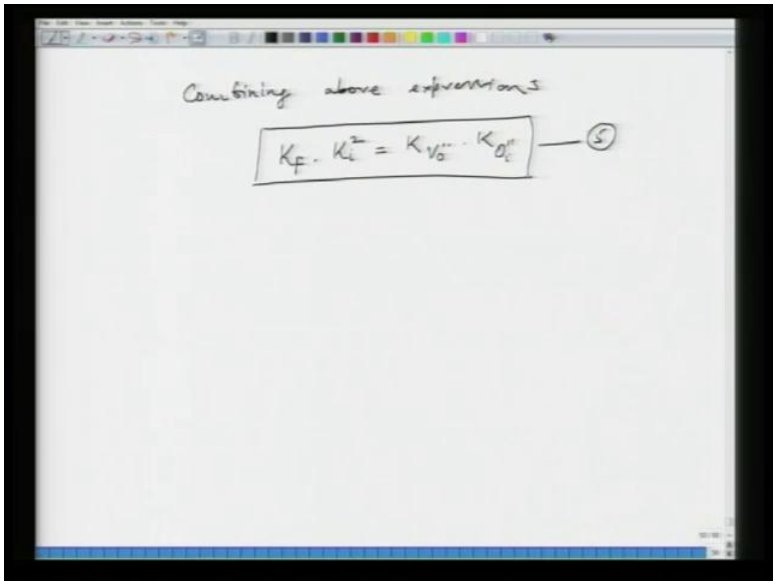

Now, what we will do is that we will work out the limiting conditions for each of these cases.

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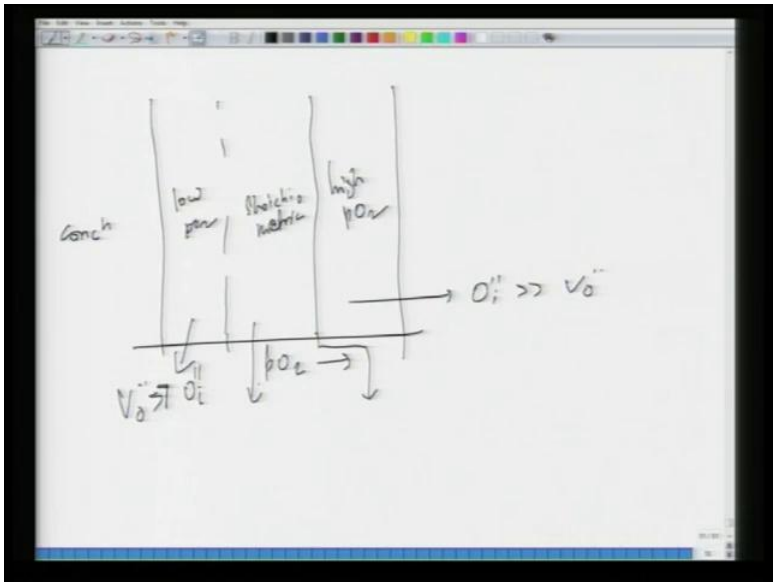

Which means if we are making a plot for example of p O 2 versus concentration so, of course, this is the low pressure regime, this is the high pressure regime and this is the stoichiometric regime. So, we know that these are three regions but we do not know where the boundary occurs. So, we need to define the boundaries, where these

boundaries are? So, what basically it would mean is how we would define boundaries are this region would be a region when you have O i lot greater than V O.

In this region we will assume that V O is a lot greater than O i and in this region we would assume either intrinsic ionization or anti Frenkel defects. We will take both the cases which one each of domination of each of these and we will see how these concentrations then vary, as a function of partial pressure of oxygen and which defects dominate in which region. So, we will finish here we will take these limiting conditions in the next class followed by followed by we will, we will calculate the concentrations of defects in each of these regions based on these limiting conditions and then we will draw the plots of concentration of defects versus partial pressure of oxygen, which are also called as Brouwer's diagram, these are very important diagrams because at any given temperature you can see what the defect concentration versus partial pressures of oxygen is going to look like and these plots also replicate when you measure electrical conductivity versus temperature. You do not know the relation of electrical conductivity with respect of to to partial pressure of oxygen or concentration yet.

But as we will see later on that these plots are very useful to know because you get the similar plots by plotting d c conductivity versus partial pressure of oxygen as a at a given temperature. So, we will do this all this in next class and and then we will move on to next topics after this.

Thank you.