Electrochemical Impedance Spectroscopy
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Lecture – 13
Linearity, Causality, Stability, Impedance vs. Admittance, Measurement Model

What we saw yesterday was the idea that we can use, KK transform to validate data.

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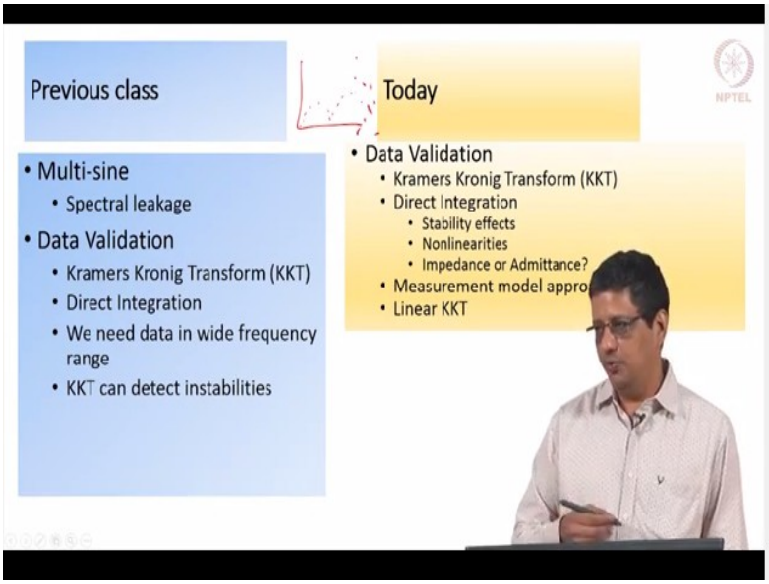

There are integral, the equations, which relate the real and imaginary parts. If you have the entire spectrum, you can use this to validate. But if you want to use the integrals, you do need to have data in the wide range of frequencies. That is ideally from 0 to ∞. In practice, you need from very low to very high. What is meant by very low to very high? Very low meaning it should settle at some level, that will be considered low. Settle meaning if I have data; at high frequency, I have data point, increase the frequency, it still stays somewhere there. Bring to low frequencies, it comes like this. At this location, if you decrease it further, it still stays around here. This is one example. Instead of this, I may have another example where it comes like this; (format) but after this, it remains there, That means it has settled there. Then I can call this as wide enough frequency range.

What if I do not have data that goes to low frequencies? I have data coming like this, it comes here and then at low frequencies, it is noisy, so I know it is not good. So I would delete this data

and say I am not able to get clear data below this. But within this limited range I have good data. It may or may not be possible to use the integration method to check when you have data in a limited range. You can use extrapolation but sometimes it will work, sometimes it will not work. We also saw one example where KKT can be used to detect instability where experimentally a potential change was superimposed deliberately to make it turn stable. So I want to continue with one or two more examples and then go to another type of problem that you would face.

Normally, we use impedance data. We get impedance data from the equipment and we use impedance data to test. But sometimes you will have to use *admittance* form. So I want to show where you have to use admittance form and what are the ways to tackle that situation. And when we realize that we have a problem that, frequency range is not wide enough, how do we handle that situation? You have to use another approach, it is called *measurement model approach*. You can use your own software to handle that. You can also use a ~~commercial, not commercial,~~ free software called linear KKT which uses a measurement model approach with certain type of constrain. So I want to show those examples also.

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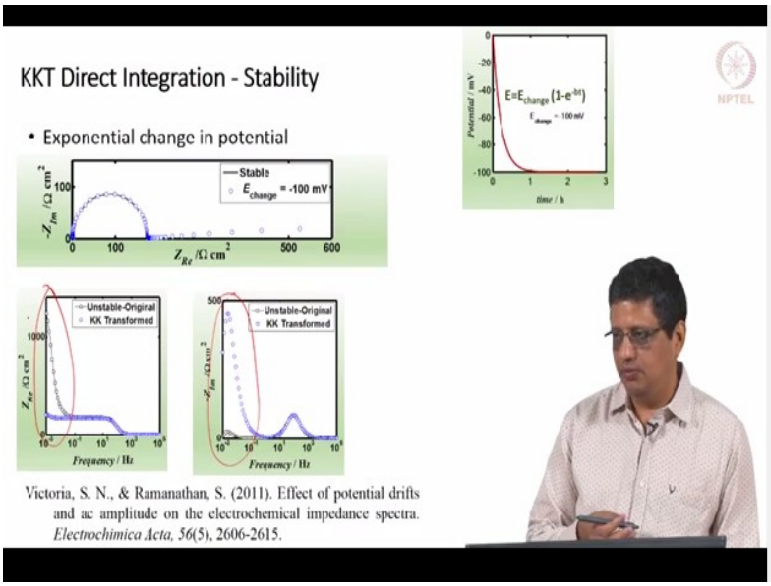

So we will continue first where we left. So in this example, this is a numerical simulation. We havelearned impedance measurement and the potential drifts, initially drifts rapidly and then it settles down. That means in the first hour, it goes from 0 to 100 mV less and then it is more or less the same. Given by this equation, I have given -100 mV, 1exp(-bt), the b will tell whether it

will go down rapidly or whether it will go down slowly.

If you take the spectrum or if you simulate the spectrum, you would get it like a semicircle, that is what you would expect if it is stable but it has a tail and sure enough when you try it with the KK transform, you would see that at low frequencies, there is a clear difference between what is transformed and what is original. This does not tell you what would happen if you do not have instability. It just tells you that the system has a problem. Of course, this is synthesized data, I have simulated this data. So I can go to whatever frequency I want. Low frequency means I can choose and get mHz, μHz without much problem.

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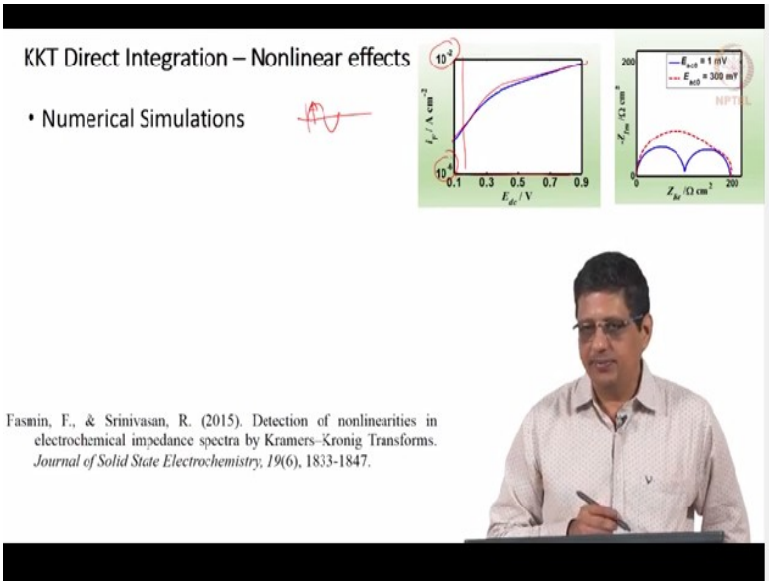

I also want to show you couple of cases where you have large amplitude perturbations. That means you are applying the sine wave but the sine wave amplitude is very large and if you look at the DC current versus potential and I have drawn this in log scale. It goes from $\mu Acm^{-2}$to 10 $mAcm^{-2}$. In log scale, it is not linear. It is nonlinear.

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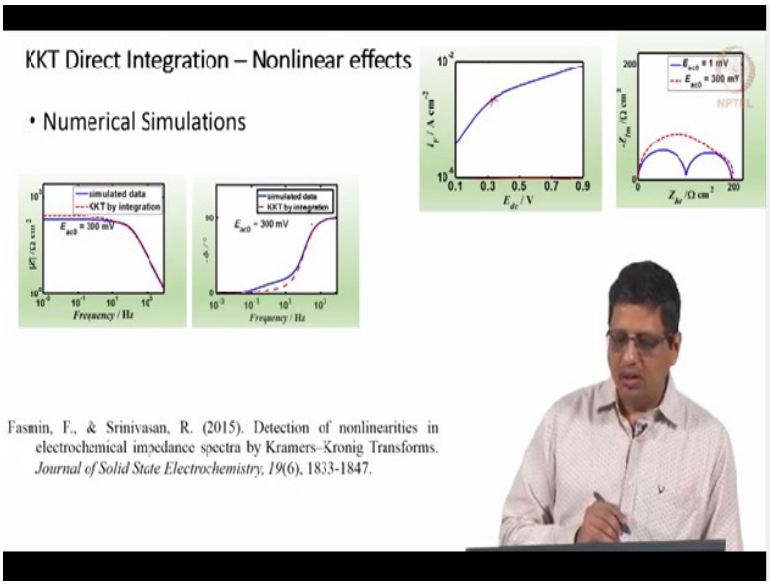

In this example, if I take a spectrum at one of the DC potentials here, I can use a small amplitude or it can be large amplitude. Nobody would use 300 mV but this is just to show what would happen when this is done in simulations. I get two different spectrum. Looking at this, you can easily say, yes, this is 1 mV, this is 300 mV, that means nonlinear effect is present.

If I get 1 mV and 2 mV spectrum, it will be identical, it will overlap. When I go to very large amplitude, it is clearly different. So I can say this nonlinear effect is present and I have not shown it here. If I take 1 mV data and take the blue colour line as simulated data and do KKT, they will overlap. If I take 300 mV data, we show that it does not overlap. There is a difference, which basically means KKT tells us that there is some problem with the EIS spectrum. It does not tell whether it is instability, whether it is large amplitude, whether it causality but it tells you there is a problem. But this is one example, and I want to show you another example.

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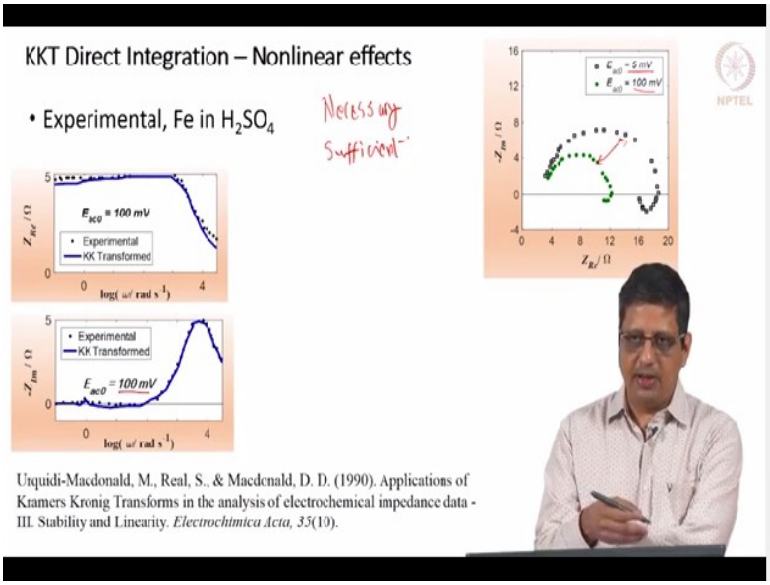

This is actually experimental data. This is taken by Professor Macdonald's group. Here the 5 mV data is the black square here and larger amplitude, 100 mV data is green round. Look at this, you can say definitely these two are different, they are not same, which means this has nonlinear effect. However, if you do the transform, you will find that they more or less overlap for the 100 mV data. 1 mV data also will show they overlap which means you can not rely on KKT to always detect the problem. Remember we have looked at the conditions and said it is not necessary and sufficient. That is if it is linear, causal and stable, it will definitely be KKT compliant. If it is nonlinear, it may or may not be KKT compliant, that means in this case, it is nonlinear but this is still KKT compliant.

But as of now, this is the best we can do. We can apply KKT and check whether it has a problem or not. If it has a problem, you will have to think about what is wrong in the system and how to get stable results or how to get results at linear regime. If it is fine, if the transformed data matches with this, then we hope that everything is fine and mostly everything will be fine. Meaning you will be in the linear regime by and large.

But there is no guarantee, that is what is given by this example. That if it matches well, because we know that data is acquired at 100 mV and we know that 5 mV data looks different, we can say yes, this nonlinear effect is not captured by this KKT. But in general, if all that you get is this data, the best you can do is to transform it and check if it works well or not. Most of the time,

you will find the problem so that is something that you should do first.

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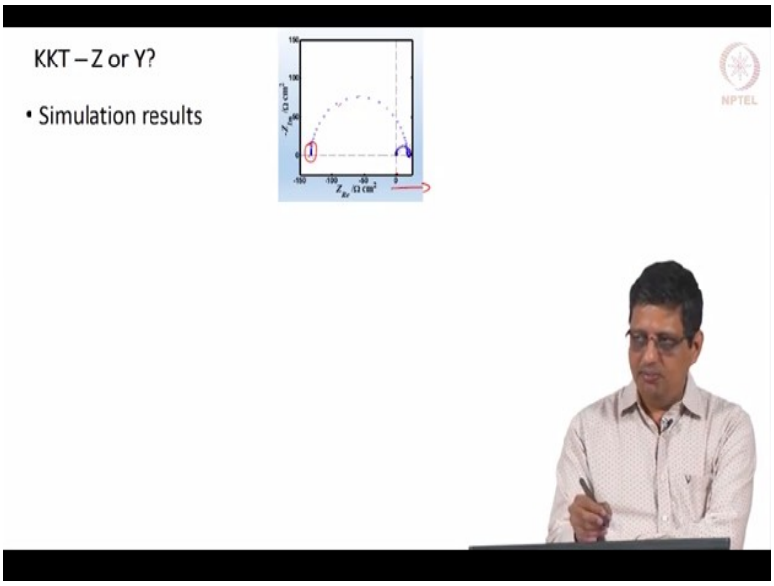

This is another example. This is simulation result where the Z_{real} starting is here and the positive values are on the right side, negative values are on the left side. And the impedance spectrum starts here at the high frequency, goes here, shows a loop and comes down and settles here. You can see there are more points here, it is denser, so it settles at this value. Right now, do not worry about the origin of this spectrum. Just believe that this spectrum is quite possible in real system.

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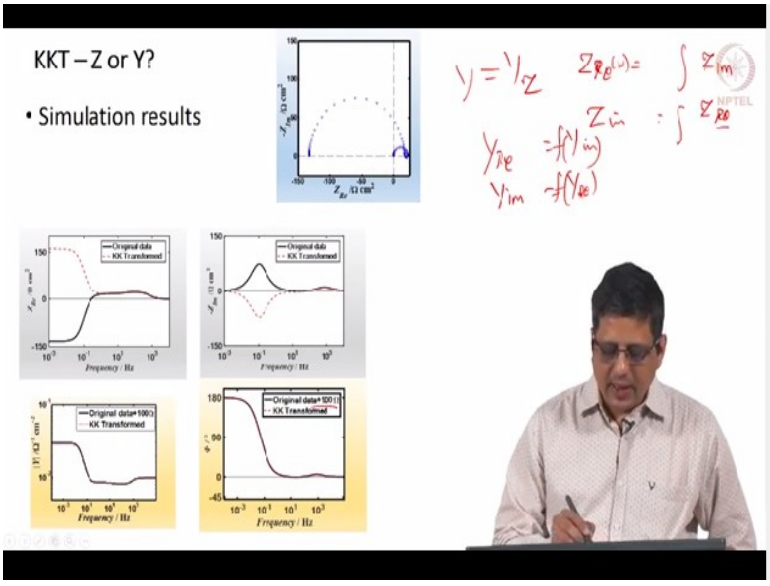

And I can show you examples where we have got data which look like this. Later, we will see what is meant by this negative resistance. Why do we get the data like this? Right now let us say

somebody gives us this data. We transform this, and you can see at the high frequencies, the red and black lines overlap. At the low frequencies, they are clearly different and in fact, they look like mirror images. This is a data simulated, synthesized by us and we have ensured that there is no instability and there is no nonlinearity in this. So we expect this to be KKT compliant. But when I use the integral, it does not show that it is compliant. So, we have a question? Is the data wrong? Data is good. Is the software doing something wrong? I do not think so. What happens is impedance is not the right form to feed this software under these conditions.

We have to take *admittance*. Admittance is 1/Z. And feed the admittance to that, that means the KKT equations which we use the rate as Z_{Re}, on the right side, you have integral, you have Z_{Re} and Z_{im}, you would have this in the imaginary side and you would have this on the real. This we have to replace with Y_{Re} and Y_{im} and of course, on the right side, you will have to have it as a function of imaginary and real.

~~So~~ If you use it as admittance, it will transform correctly. Out here, high frequency, the impedance is close to zero because it is simulated data. If you have a solution, with solution resistance, high frequency data will not be zero, it will have some finite value. And when I use zero and when I try to calculate the admittance, it becomes very large number. It will become ∞if it is exactly zero.

If it is close to zero, it will become large number and when we do the calculation is Matlab, there are round of errors. So in order to avoid that, I have taken original data and~~,~~ added 100 Ω to that. That means the impedance that I get here will not be 0 but it will be close to 100 which means when I take the admittance, the number will be more accurate, that is all. This addition of 100 Ω is to make sure that I am not in the region of round of error in the Matlab or any other software that I am using.

And then I will calculate the admittance, transform it, and when I transform, I see that the transformed data and the original data match well. Now I have to tell you why should we use impedance, why should we use admittance. In one condition we use impedance, one condition we use admittance.

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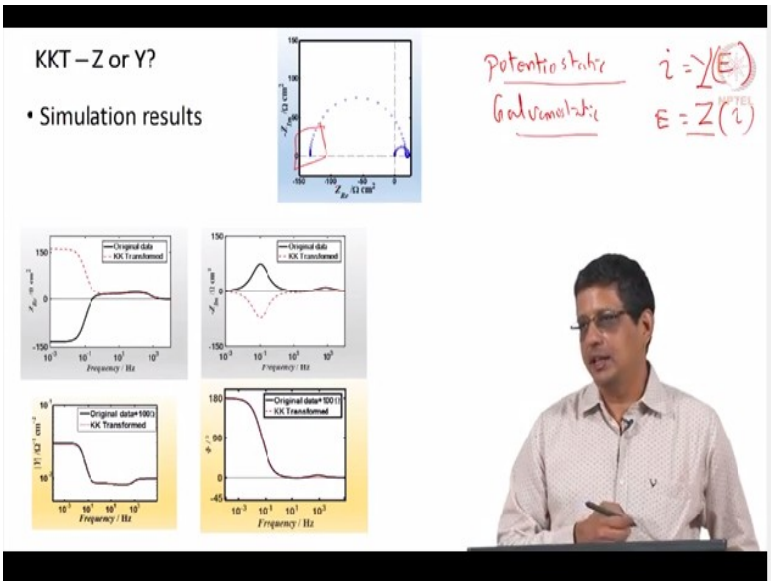

If the system is stable under potentiostatic control, sometimes called potential regulation, what we measure is current, what we control is potential. And the function that transfers the potential to current is *admittance*. Under galvanostatic control, if we measure potential oscillations, we apply sinusoidal current, we get the potential and we do FFT on it. Whatever analysis we do, and based on that we calculate the *impedance*.

So the transfer function for potentiostatic EIS acquisition is admittance. The transfer function for galvanostatic EIS measurement is impedance. If the system works well under both conditions, potentiostatic as well as galvanostatic, then you can use either admittance or impedance and in both cases, KKT will show you that it matches well as long as it is *linear, causal and stable*. If the data is acquired under potentiostatic control, the right method or right feed to the program is admittance.

Many times, it so happens that the system would be stable under both galvanostatic and potentiostatic condition. Sometimes, as an example here, the system is stable only under potentiostatic condition, it is not stable under galvanostatic condition. Right now you have to trust me but later I will show you example where you would get the spectrum and I will also describe how you would get the spectrum and I would also tell you that it is not stable under galvanostatic mode and you will be able to follow why it is not stable under galvanostatic mode.

Right now you have to trust that this system from which we have acquired this data, that is stable only under potentiostatic mode. In general, if you see the impedance going to the left side. Left side here with negative values for impedance, real part of the impedance that will be stable on under potentiostatic condition. Which means you will have to use admittance form, that is one way. You can apply one or two tricks to use this in impedance form. When you use a commercial software and get the data, what you do is, you say these are the settings, give me the impedance from this frequency to this frequency. You can say constant potential. You can say constant current, depending on what facilities are available in the instrument. You will get the data. Most of the times, you will get only impedance format. You can convert it to admittance format yourself but you will get only in impedance format. And the natural tendency is to use that format to check. If the data comes on the right side of this, it is fine. It would be stable under potentiostatic as well as galvanostatic.

So you can use impedance as it is, and that is what I have done before. In the previous example, I have not used admittance. I have given only in impedance form. Although using it in admittance form is correct way, using it in the impedance form works fine because those systems happen to be stable under galvanostatic condition.

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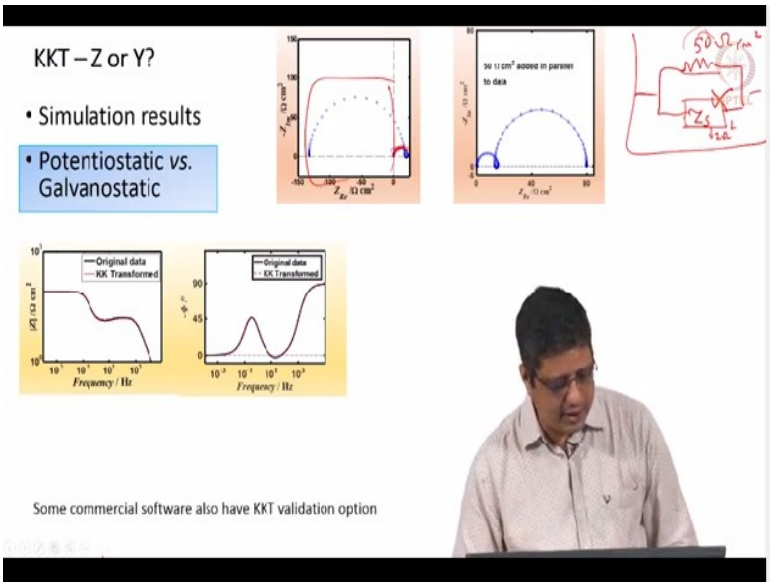

I can use a trick. I have this data and I will call this as Z_f or Z_{system}, Z_s. Now if I add a resistance in parallel to this, because the units are Ωcm^2, I am going to say $50\Omega cm^2$. What would happen is,

I can get impedance of the total system here and that would come to the right side, I will have to choose this carefully. Why do I chose 50 Ωcm^2? Because I see here that it shows some value close to -120, -130, etc. So this is large value in the magnitude. You do not worry about the sign here. And this is small value of impedance. Current will go through the smaller one. When this is small, it will go through this value. So I have to choose a value which is less than the value here. When I add this 50 ohms, added in parallel to the data, I get a spectrum which comes completely to the right side.

So the idea is this, I want to know whether this data is KKT compliant. Here of course, it is smooth, right. When you get experimental data, it may not be like this. I can either move it to the admittance form, I have to do it with care so that I do not get in round of errors and do the transformation. Or I can bring it to the impedance form. It is already in the impedance form, (otherwise) I can bring it to the impedance form where the data is on the right side of the plane. And if this system is KKT compliant, I know this is a simple resistor. So if this entire system is KKT compliant, that means this is also KKT compliant. If this system is not KKT complaint, problem must be coming only from this because this again is a simple resistor. So what I have done is to move it to the right side by adding a resistor in parallel. This data has moved to the right side.

If you look here, (the) high frequency data (which) effectively has low impedance. (The value of impedance is) 50 Ω regardless of the frequency. (The unit can be) Ω (or) Ωcm^2. Here, this is a variable, at high frequency, it is a low value. At low frequencies, it has a large magnitude. So what will happen when I go to high frequencies? This may be like 2 Ω. Then the net impedance of the system will be close to 2 Ω, little less than 2 Ω but close to 2 Ω.

So at high frequency, if data is more or less the same as original data. When I come to middle frequencies, it has moved a little bit. When I come to low frequencies, this is -100, this is +50, you can say most of the current will go through this, some of the current will go through this. It is negative, of course, I have to explain what is meant by negative resistor. Right now just say that we can do the calculation and get the value. Later, I think I can convince you that negative resistance has some meaning and this is what it means, it is not physically unreasonable. Now

what I mean by this is? I bring the impedance values to the right side of the plane. That means in the complex plane plot, the Z_{Re} which forms the x axis or abscissa, all the data is on the right side of y axis.

All the data has positive value for Z_{Re}. In this case, this data can be transformed in the impedance form itself, which is what normally you would (do). You can use commercial software for KKT validation. Some of the software, at least come with this choice. I want to show you an example.

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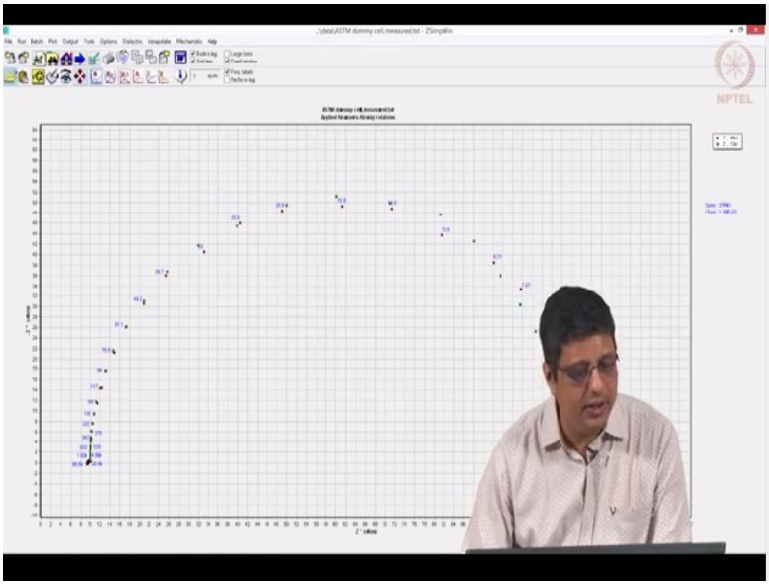

This is of course some dummy data. This is one example, Depending on what software you have, you may have different menu choice is there. In this, if you go to the menu and extrapolate and for whatever reason, they had given it as calculated impedance. It would show you the transformed data and the original data. The green colour is the calculated value, red colour is the original value, and you can give it in different formats; bode plot, real and imaginary (values) in the normal Nyquist or complex plane plot.

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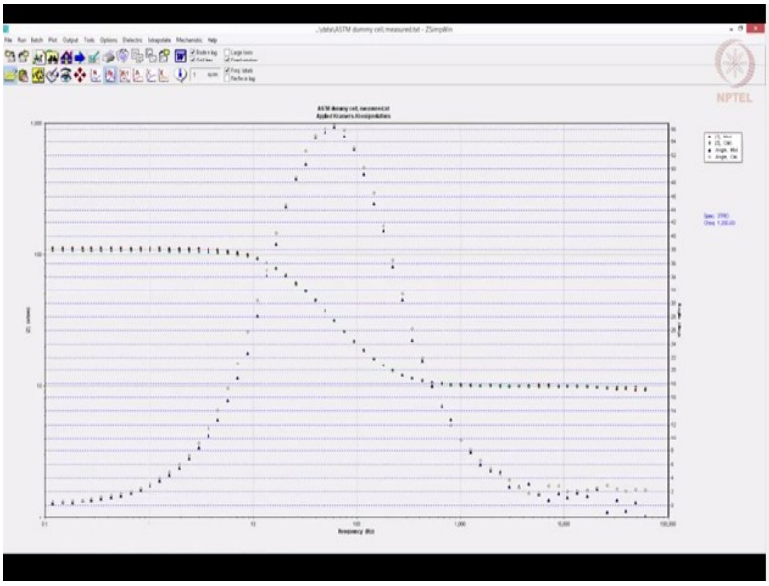

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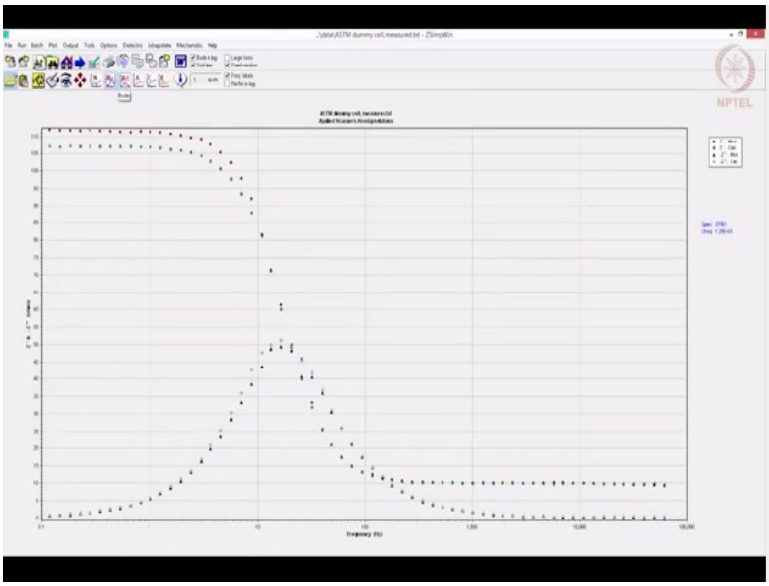

However, here if you take a data that goes to the negative side, you cannot ask it to do the transform in the admittance form, and they do not even tell you how they do this. They just do the transform by some method and give you the result. And for whatever reason, if you say Kramer Kronig routine, that does not do the KK transform here.

That does an extrapolation using KKT, extrapolation to lower frequencies. So if you want to check for KKT, you should use calculate impedance in the system. Likewise, other software depending on whichever commercial software you are using, will have, mostly will have some way of the doing the KKT. But I think by and large, they expect that you have the data in the

correct format which in their feed, it is going to be only in the impedance format.

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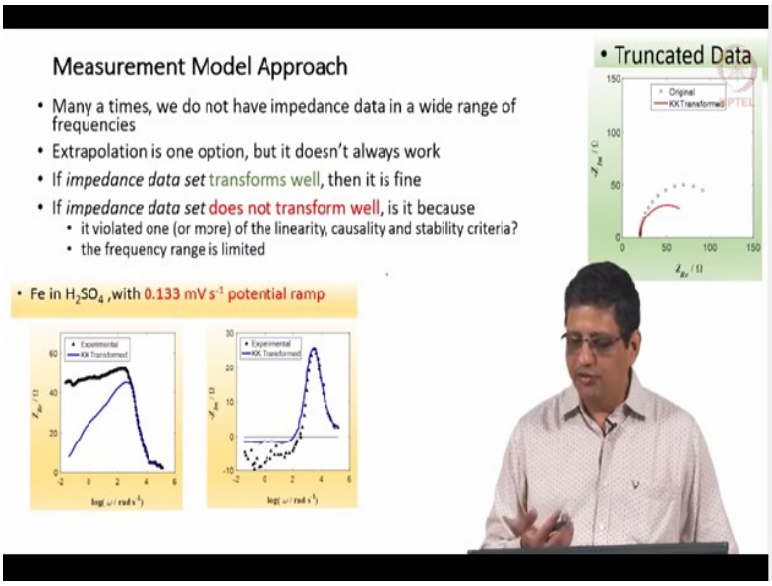

However, when you have a problem with the result, we want to know. We do not have impedance data in the wide range of frequencies. The extrapolation will work sometimes, it may not work sometimes. Now you have a data, you transform it, it transforms well, you are happy. If it does not transform well, you have questions. Is it because the data is not KKT complaint because it has violated one or more of the constraints?

This is an example here, here it does not transform well. And that is because it is unstable. Is it because the frequency range is limited? Another example which we saw before. It actually comes from a clean system but we cut the data or truncated the data and then when we transform, it does not match well. Here we know what we are doing. But in general, you get a data, you hope you have done the experiment correctly and you got the data or somebody has sent you the data. You hope that they have done it correctly. Is the system unstable? Have they used large amplitude? Have they saved the data? By mistake they have saved some wrong information? We do not know that, and if it is truncated, you cannot really do much about that. Meaning, I cannot extrapolate it reliably all the times. So there is another way to handle this.

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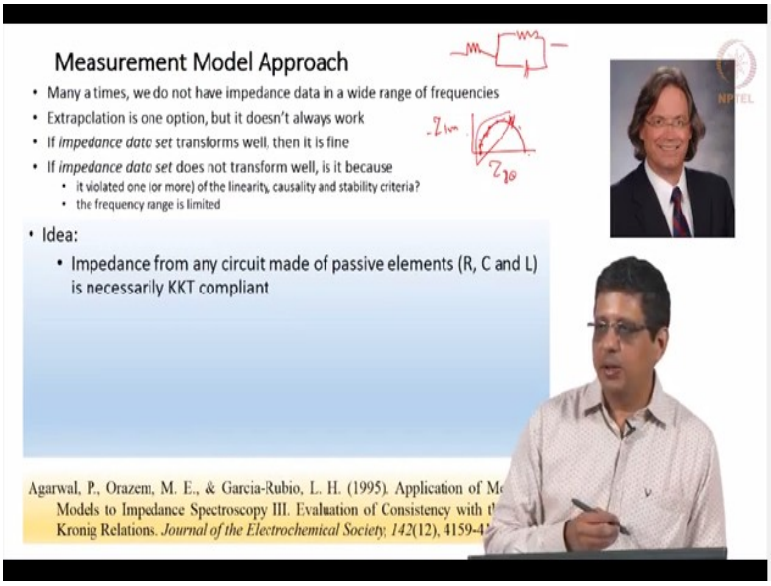

This idea was proposed by Professor Mark Orazem from University of Florida. The idea is this, we have some data. I want to get a system which I know is KKT compatible and see if I can generate the same data. If this data which we have got, whatever form it is in if that can be reproduced by another system which is known to be KKT compatible, then I can say this data has come from some system which is KKT compliant.

Now if I take impedance from a system which contains resistance, capacitance and inductance, they are called *passive elements*. All the spectrum that comes from them, will be KKT compliant. So in such cases, let us say you have an RC system, RC meaning resistor and capacitor which looks like this and we know in the complex plane plot, if I take data, it is going to look like a semicircle. From high frequency to low frequency, if I take data at many frequency, this is how it looks like. This is $-Z_{Im}$, this is Z_{Re}. But let us say I have taken data only up to this. So I have partial data, still I know the system is good. System is KKT compliant, and this I can model using this circuit and I know the circuit is KKT compliant.

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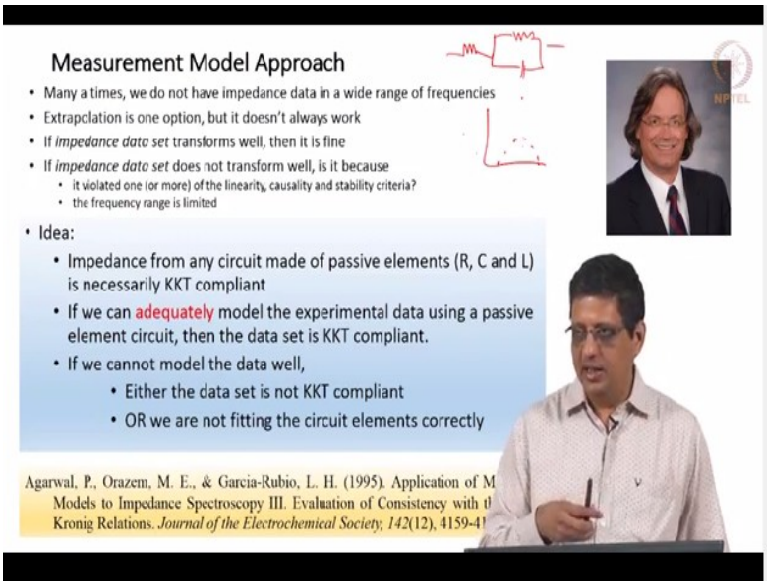

~~So~~ This approach is called *measurement model approach*. So we say if we can model the experimental data using a circuit which contains only passive elements, then this data is KKT compliant. We model it well, model it adequately, we have to tell what is meant by that. Now if we cannot model the data, if I try modelling it and it does not fit well, I have two choices. One data set is not KKT compliant. Another may be I am not doing it correctly in terms of modelling. So if take this data; of course, this data most of the software will model correctly. But if I take some other set of data and I come up with a circuit that I think will model this data well and I know the circuit is KKT compliant, I am not modelling it correctly, meaning they are not matching. May be I did not do it correctly.

If I use different choice of circuit, I might get this correctly. If I use different initial values for the guesses, I might model it well. So that doubt is always there and we will see how to overcome them. How to ensure that we have done our best in terms of modelling, then if it does not match well, you can say data has some problem.

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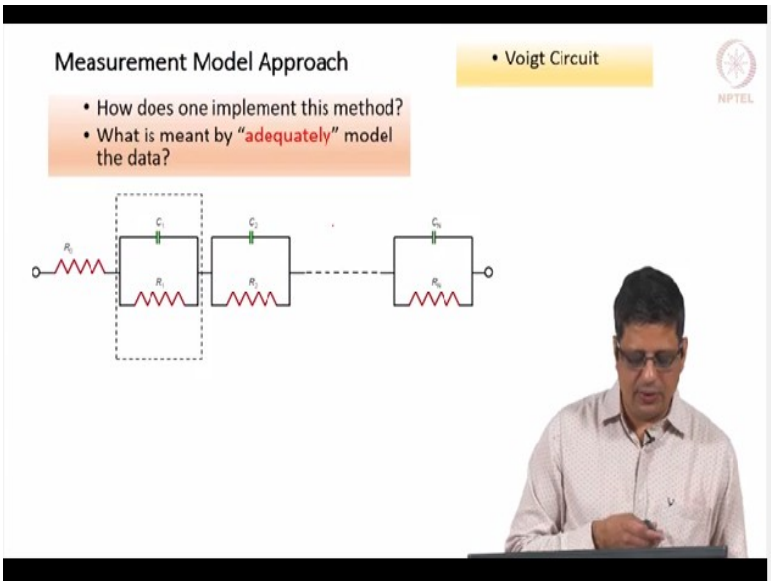

Now I have to tell you how to implement this method. It is one thing to say yes, you take the circuit and then it has to model it well, then we say it is KKT compliant. But how will you go about implementing it and what do you mean by adequately model it or model it well? So I want to show you type of circuits that can be used for this.

One you have a resistance which can be thought of as a solution resistance, and you have one capacitor and resistor in parallel. This type of circuit is called *Voigt circuit*. You can add more number of them and keep increasing that number, 1, 2, 3, 4, n. This type of modelling is with Voigt circuits.

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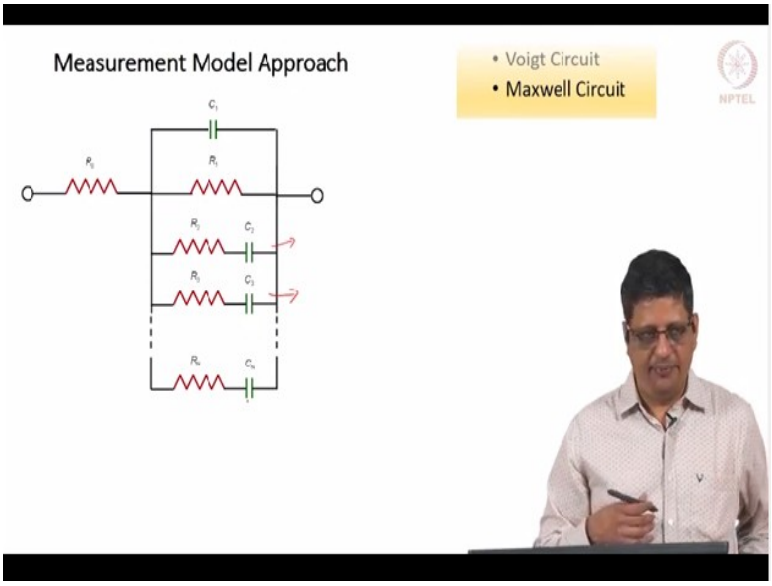

Another type is to use the capacitor and resistor in parallel for the simplest circuit. And when we want to add more and more elements, we will call, this is second, Maxwell pair. This is third Maxwell pair and so on. We can keep increasing the number. And by the way, these are equivalent, meaning from Voigt circuit, I can move to Maxwell circuit and back.

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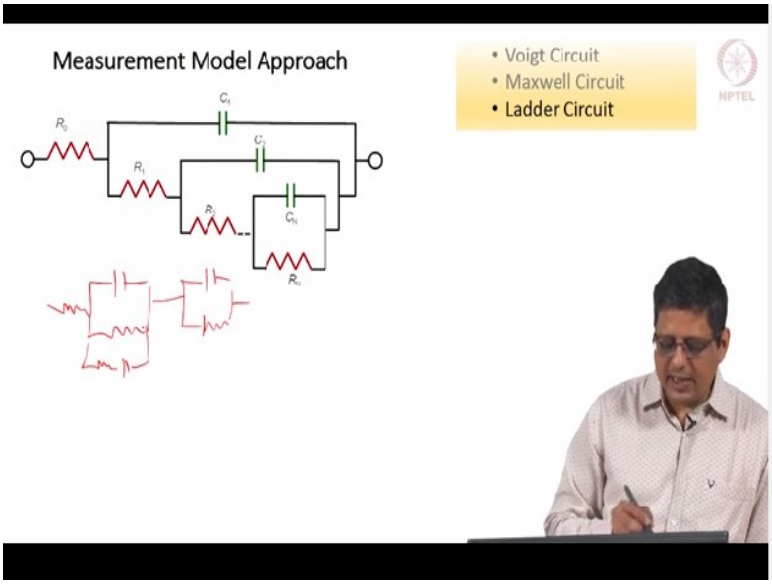

Third possibility is to use what is called *Ladder circuit* where you have the solution resistance. This typically, it will be used to model a capacitor, double layer capacitor. Simplest case I will not have C_2, R_2, etc. I will just have C_1, R_1 and that is the same. So the simplest circuit of resistor, capacitor and another resistor, you can think of it as a Voigt circuit, you can think of it as the simplest Maxwell, you can think of that as the simplest Ladder. The moment I add one more RC, depending on where I add it. If I add it here, I would call this as the second level Voigt. If I add it here, I will call it as a Maxwell. And if I say I am going to add it here, I will call it as a Ladder.

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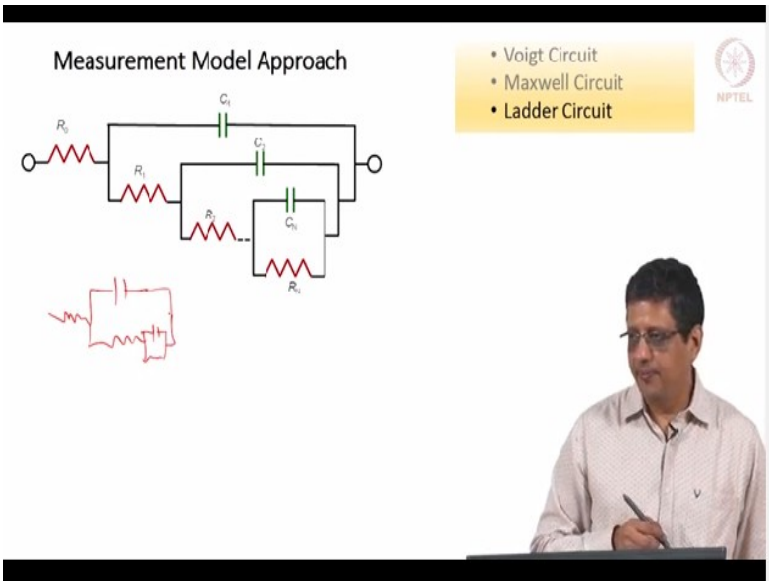

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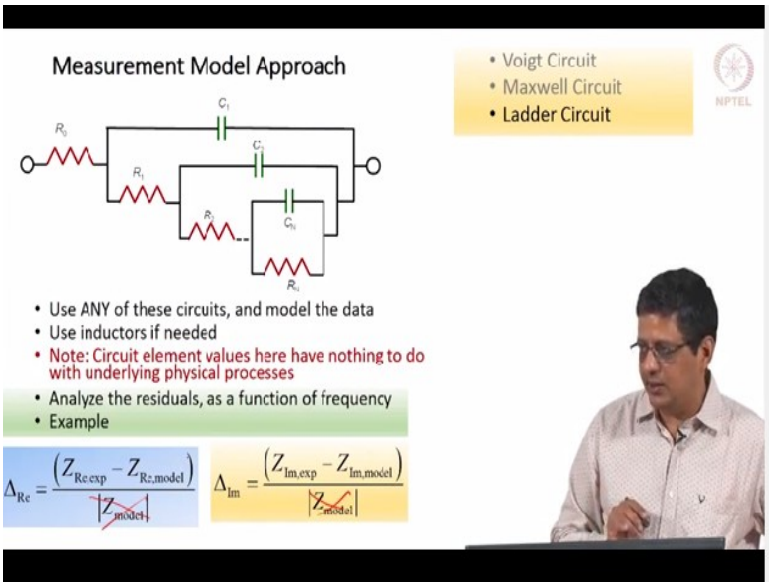

And just like capacitance, I can add an inductor, that is also possible. If any of the circuits with any n, model the data well, we can say the data is KKT compliant. You can use inductor if you want, if it is necessary. Now the thing is if I can model the data with n Maxwell pairs, it does not mean I know what is happening in the system and each of these element has some physical significance.

All that we want to do here is to say this data has come and this KKT compliant system can produce this data, that is all that we are checking here. We are not trying to find what is the meaning of R_1 value being 100 Ω or what is the meaning of C_2 being 20 μF. What is the meaning

of C_1 being 10 mF. None of these things. We just want to see whether data can come from a system that is KKT compliant.

So if you have to model this with 10 elements, it does not mean when you go for physical interpretation of the data, you have to go for 10 elements. It just says that the data is KKT complaint or not. So that I want you to remember. First time when we do the data validation, you can use any of these circuits. Check it, if (gives propeor results), next when you go for actual modelling, you will have to think what is the system, how do I think it can be represented and then model as per that idea.

Now when we model, when I say it models well, what do I mean? When we model this, you will get a simulated data, you have the actual data. The difference we will call it as *residual*. For each data point, you will get a residual. In fact, one data point is a complex number. It has real and imaginary part. You can get the residual as real part; this is the residual, imaginary part, this is the residual.

And plot it as a function of frequency. This is an example. One way to define a residual is to say Exp here is the experimental, so I will take the real part. (Experimental-Model) / The absolute value of the complex number given in the model. That is one way. I can use this and say I will not normalize it. It will just use the residual as it is.

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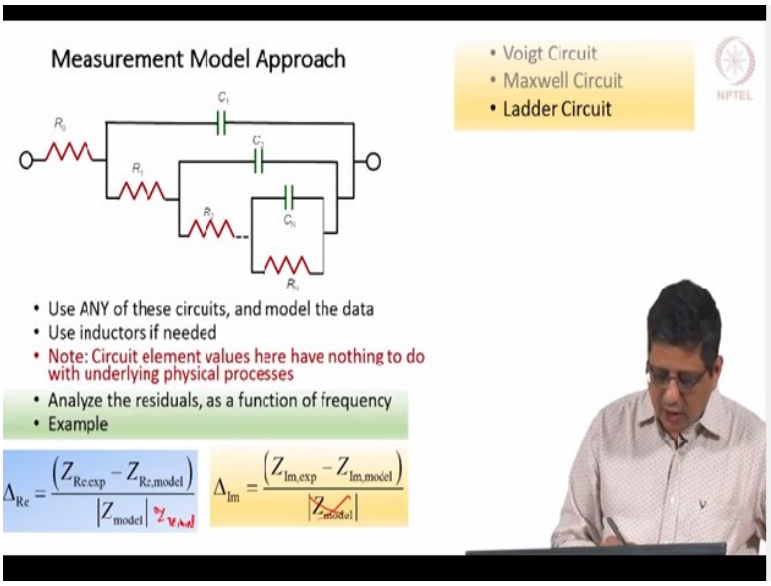

I can say this, instead of saying absolute value here, I will use Z_{Reexp}as normalization or $Z_{Remodel}$ as a normalization factor. This is all fine. You have to be careful that you are not going to a value which is very small. At the high frequency, do not use a very small value for the denominator. If it is close to zero, it is going to disproportionately push this up. So at high frequency, you may see that residual is very high.

You have to check is it because my impedance is very close to zero, I am having this problem or is it because genuinely the model and the experimental points are different. So this is one form of definition. You can have your own definitions, as long as they are meaningful, it is okay.

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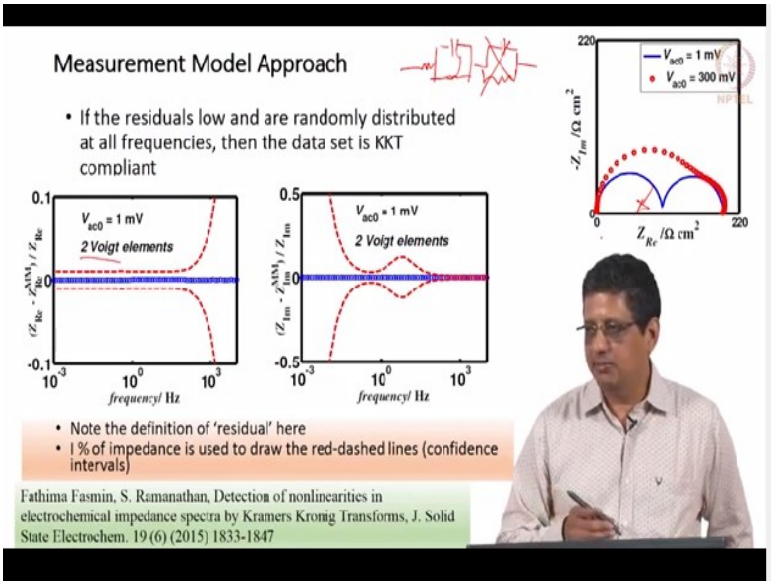

Now if the residual values are small and they are randomly distributed, then you can say this data is by and large model correctly. If the residual values are large and they show systematic distribution, meaning they are not randomly distributed, then you know that model is not capturing the data correctly and there is a clean difference. Just verify that you are modelling it to the best of your abilities and then if it still does not match, system has a problem, it is not KKT compliant.

So this is one example. So I have 1 mV data and 300 mV data here. 1 mV data I have fitted this to Voigt circuit which goes like this and I have denoted this as 2 Voigt elements. If I fit this data to 1 Voigt element, I will not be able to fit it. Only one semicircle will fit. This RC can capture one time constant. This RC can capture another time constant which is given by this loop. So two semicircles, I can easily capture using two Voigt elements. Residuals are pretty much zero given by the blue colour, circles here. The way I have defined the residuals is $Z_{real} - Z_{real\ model}/Z_{realexp}$. And I have made sure that at this level it has not come close to 0, so it looks very low. This is simulated data, so I do not really have a standard deviation here.

If I have an experimental data, I should take few sets of repeats and then say at these frequencies, these are the standard deviation and these are the average values for impedance. Here I have used 1% of impedance and said that 1% of the impedance value gives us the confidence interval. So if the residuals are very low and they are well within the confidence interval, I can say this data is clean data, 1 mV data that means if I fit this to 2 Voigt element circuit and I do it correctly, I get lower significant amount of residuals. So this data probably arises from a KKT compliant system.

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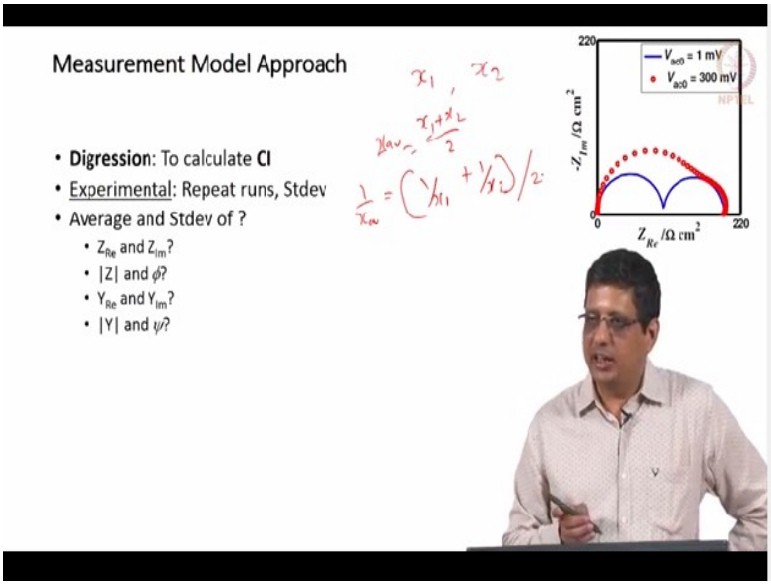

I want to calculate the confidence interval. Normally, I will run experimental repeat runs and when I calculate the confidence interval, what do I do? Do I take the average and standard deviation of impedance, real and imaginary values? Do I take the average in standard deviation of magnitude and phase? Should I do it for admittance, real and imaginary, or magnitude and phase? All of this will give you a different value by the way. If I have value, just take simple real numbers, x_1, x_2 and I take the average, I will write it as $(x_1+x_2)/2$. If I take the inverse and I write this, I will get a different $x_{average}$. Of course, divide by 2, correct. Do you understand. You will not get the same number by and large. If you are lucky, you might get same number or close to the same number. Standard deviation again will be different.

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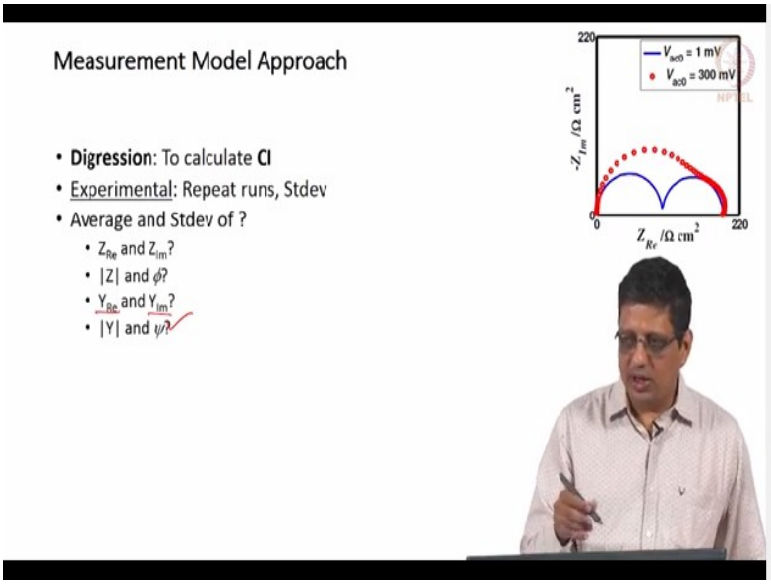

What one should do is to find in which format the data acquired. If you apply potential sinusoidal waves and get data in the current, if you do it in pseudopotentiostatic mode, what you are getting is admittance. And if it is done with FFT, what you get is magnitude and phase. If it is done using lock-in amplifier or cross correlation, what you get is usually real and imaginary. However, what we end up doing most of the time is to use the impedance anyway.

That what we see in the software, we click and get impedance and we usually plot it. Of course, we plot it in all formats but commonly used format is the Nyquist plot where you plot real and imaginary values.

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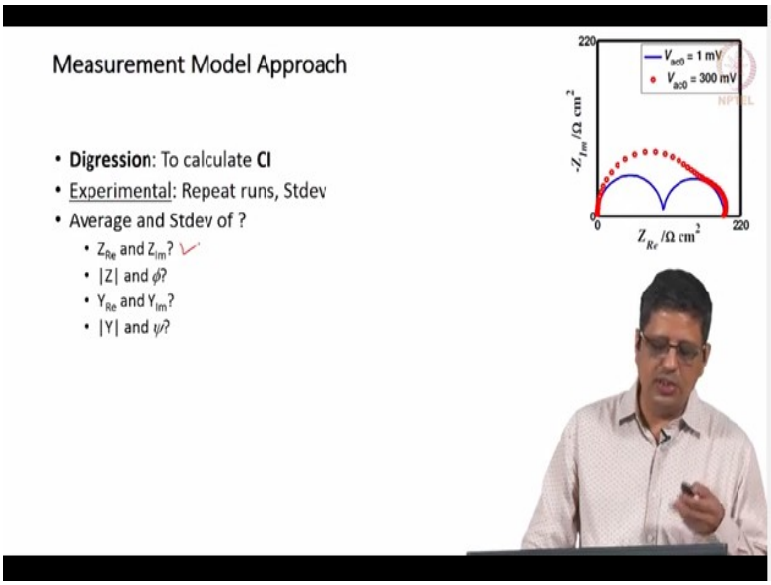

So this is what we end up using or most of the people end up using but if you want to do it correctly, strictly speaking, you should use it in the format in which data was acquired.

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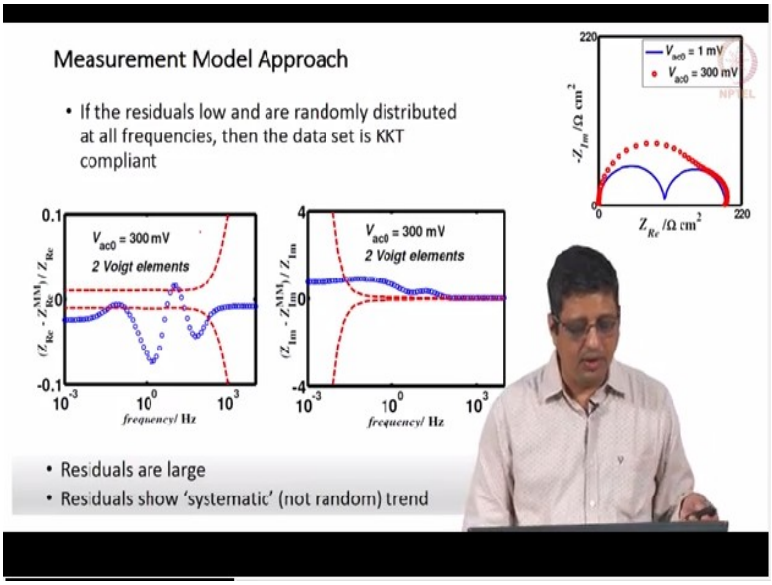

Now I have taken the 300 millivolt data and then I subjected this to model fit. I use two Voigt elements. Blue circle show that they are not random. They are not small and they are well beyond the confidence interval. And right now trust me when I say if we use three or more elements, you will not get any better (fit) which means I can tell this data is not KKT compliant. I cannot use it now but in this case of course, I know, I will have to reduce the amplitude and get a better data. Residuals are large and they show a non-random distribution.

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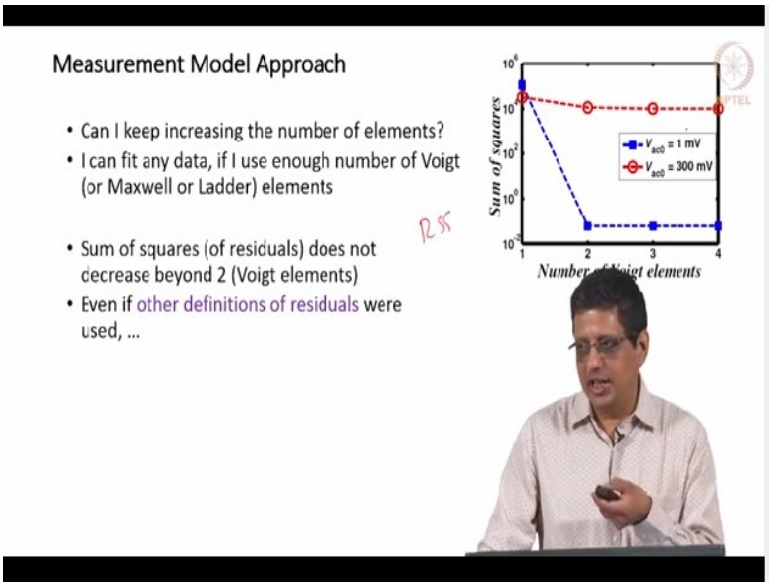

Now can I keep increasing the number of elements and there is always a question or a statement saying, I can fit any data. If you give me enough number of elements, I will fit any data. You allow me resistance, capacitance and inductance in a Maxwell, Ladder, or any of this, I think I

can fit any data. Meaning you are using 2 elements to fit this circuit but if I use 10 elements, I will definitely fit it.

May be you are not doing it correctly. If you take the residuals and calculate their sum of squares, sometimes it is called RSS, *residual sum square*. For 1 millivolt data, 1 element does not fit well, 2 elements fits very well and after that there is no change. For 300 millivolt data, in fact the residuals are little less at 1 element and then they decrease a little, they remain the same.

This is for 1 type of definition. Even if you use another definition of residual, the value will change but you can check for yourself that you will not be able to fit it better by going with more number of elements. So it is not true that you can fit anything. You will not be able to fit anything but you will get better confidence if you actually try this.

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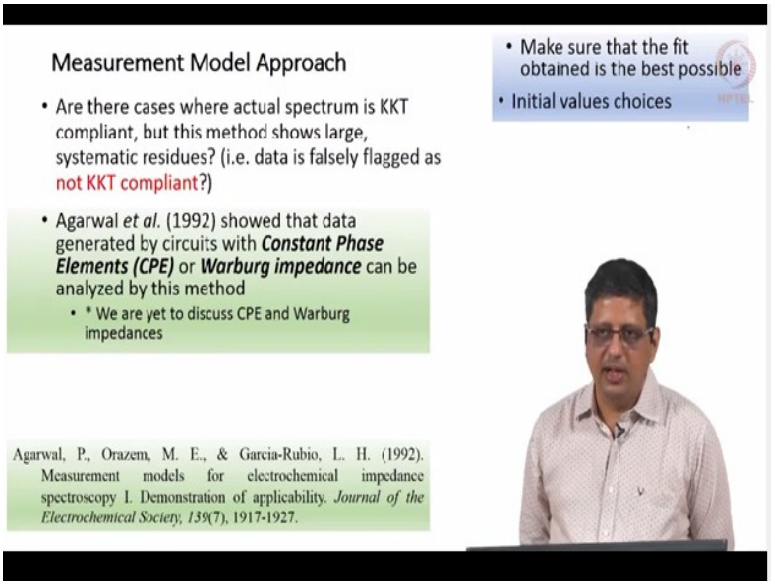

Now another question: are there cases where spectrum is actually good but the model says I am not able to fit; therefore, it is not KKT compliant? So if data is falsely flagged as, to the extent we know it is not possible. Even if we use elements called *constant phase elements* or *Warburg impedance* which normally are not fitted with RC circuit, I have not discussed them yet. So we will discuss them later. Even if I get other elements which are not normally modelled using RC, if you put enough number of RC's, if the data is KKT compliant, you will find that system will capture it. That means you are not likely to get false information saying the data is not compliant.

And of course, it is up to us to make sure that the fit is the best possible. Later when we go to electrical circuit fit, I will also tell how to chose the initial values if they does not fit well in the first attempt. Can you do something to make it fit better?

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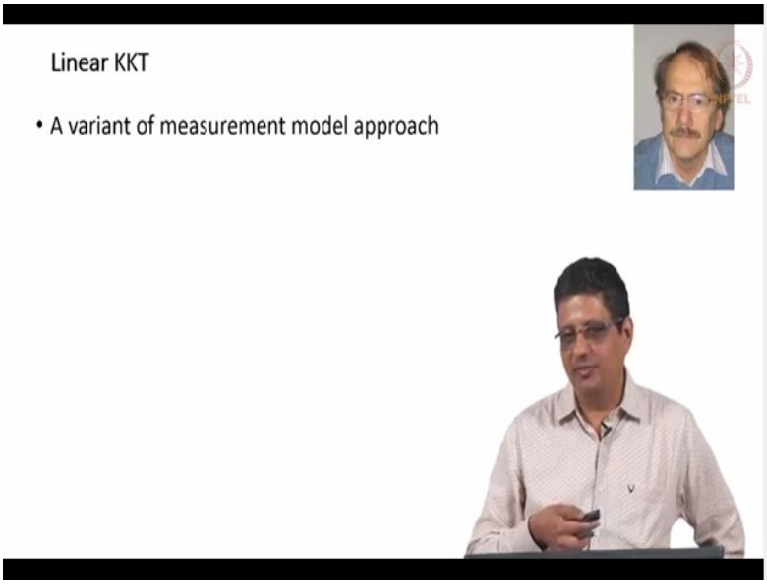

This software is given by Professor Boukamp in University of Twente. It is called *Linear KKT*, It is a variant of the measurement model approach. It makes it easy for us to apply this approach, measurement model approach and check. So when you ask do I have to keep putting more and more elements, do you want me to first model with one RC. Let us fit another RC, one more and then calculate the residuals and do all these things, do I really have to do this or is there any other easy method to do?

I will describe that to you tomorrow. So I wrote to Professor Boukamp and to professor Macdonald, got permission from them to give the software to you. When you use it, you will get a better handle or how to take a real data and apply the KKT on this. When I show you here, you will get some idea but you will forget it quickly. If you use it, you are likely to remember it for a longer time. We will stop here today.