

Potentiodynamic PEIS Test for Corrosion Application

Purpose

This test is to demonstrate Corrosion EIS test. This will measure PEIS by changing bias potential with staircase wave form. This complex procedure can be tested with frequency scanning potentiodynamic PEIS in technique menu.

This demonstration's test condition is;

- ◆ Start potential: -700mV vs Eoc
- ◆ Final potential: 600mV vs Eoc
- ◆ Voltage step: 20mV
- ◆ EIS measurement at each potential step
 - Initial frequency: 1MHz
 - Final frequency: 0.1Hz
 - Amplitude: 10mV
 - Points per decade: 10

Preparation

- ZIVE SP/MP electrochemical workstation
- Corrosion cell (universal cell holder)
- Coin Cell holder

Cell Connection

+ electrode(Green lead & Blue lead)

- electrode(White lead & Red lead)

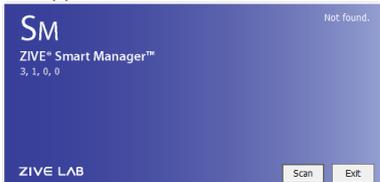


Procedure

1. Turn the Power switch on the ZIVE SP/MP electrochemical workstation
2. Open the SM software by clicking the SM icon. The following progress box will appear, and will show the progress of checking instrument configuration and communication between ZIVE SP/MP electrochemical workstation and PC.



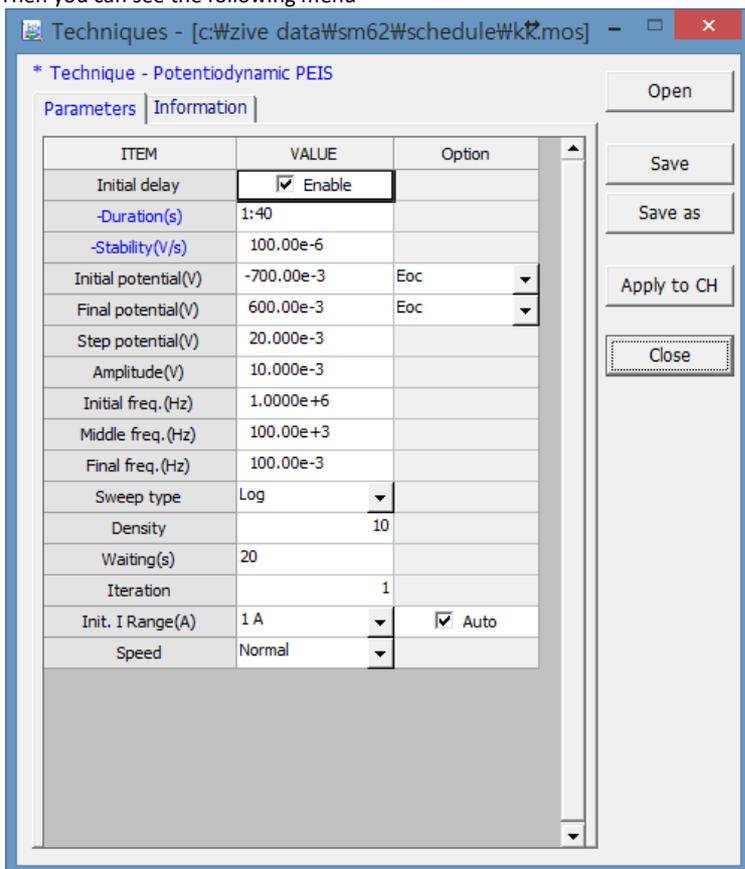
If the link is successfully connected, Click “OK” button on the box then the progress box will automatically disappear and SM software will appear. If the link failed, The following progress box will display then click the “Retry” button.



If the link failed again after clicking “Retry” button, you need to check USB cable connection.



3. Click Technique function icon (or select Technique function on Experiment menu)
Then you can see the following menu

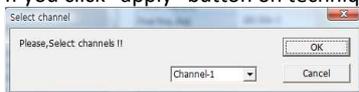


- Select Frequency scanning potentiodynamic PEIS at left technique selection region. The parameter editing region is located at right. Input figure for parameter setting as the follows..

ITEM	VALUE	Option
Initial delay	<input checked="" type="checkbox"/> Enable	
-Duration(s)	1:40	
-Stability(V/s)	100.00e-6	
Initial potential(V)	800.00e-3	ERef <input type="text"/>
Final potential(V)	3.0000e+0	ERef <input type="text"/>
Step potential(V)	50.000e-3	
Amplitude(V)	1.0000e-3	
Initial freq.(Hz)	10.000e+3	
Middle freq.(Hz)	10.000e+3	
Final freq.(Hz)	100.00e-3	
Sweep type	Log	<input type="text"/>
Density	10	
Waiting(s)	3:20	
Iteration	1	
Init. I Range(A)	2 A	<input checked="" type="checkbox"/> Auto
Speed	Normal	<input type="text"/>

- Click "Save" button to save the technique file which contains the above parameter and setting file name.
- You can assign this technique file to channel(s) which you want by click "APPLY" button or select schedule file using file selection on "Single channel control/monitor" or "multi channel control/monitor"

If you click "apply" button on technique menu, You can see the following channel selection menu.



Select channel and click OK then this technique file will be assigned on the channel. Click "Close" button or minimize technique window.

- If you want control ZIVE SP/MP electrochemical workstation using "Single channel control/monitor", you can see following windows.



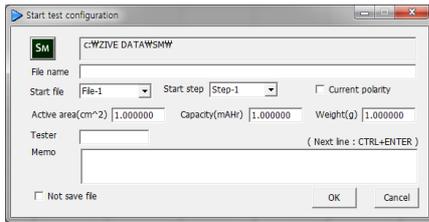
To start experiment, click Start button

If you want control ZIVE SP/MP multichannel, you can see the following window

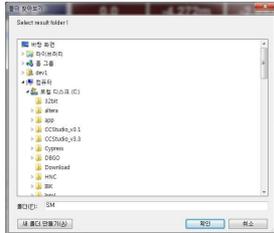
CH.	GROUP	CHANNEL STATUS				STP/CYC	SEQUENCE FILE FILE NAME	EDIT	CONTROL				TEST RESULT FILE FILE NAME	PROGRESS
		FLOW	TASK	LAST	RUNNING				CHG	RUN	FLOW	SKIP		
1	OK	READY	NONE	NONE	0:00:00	1/ 1						5512_01.SEO	0.0 KB	
2	OK	READY	NONE	NONE	0:00:00	1/ 1						5512_01.SEO	0.0 KB	
3	OK	READY	NONE	NONE	0:00:00	1/ 1						5512_01.SEO	0.0 KB	
4	OK	READY	NONE	NONE	0:00:00	1/ 1						5512_01.SEO	0.0 KB	
5	OK	READY	NONE	NONE	0:00:00	1/ 1						5512_01.SEO	0.0 KB	
6	OK	READY	NONE	NONE	0:00:00	1/ 1						5512_01.SEO	0.0 KB	
7	OK	READY	NONE	NONE	0:00:00	1/ 1						5512_01.SEO	0.0 KB	
8	OK	READY	NONE	NONE	0:00:00	1/ 1						5512_01.SEO	0.0 KB	

To start experiment for specific channel, click Start button on the channel. If you want to start experiment for multiple channel, click on Group column which you want to start and click Start button at bottom side.

- After click start button, you can see the following box.



The data file name is automatically determined as default value but if you want to change data file folder then click



You can make new folder which you want to save.

- ◆ If you want use your file name, input your file name on “File name”
- ◆ If you did not make file name manually, new data folder which named as sequence file name or technique file name under data folder and it’s subfolder will be created with name as date. The data file will be saved in this folder. The data file name will be determined automatically as string with sequence file name or technique file name and hour and minute and second, finally channel number will be added.

example) If you used sequence file for test and its file name is testabc.zsc and channel number is 1 and test date is 2000 year January 1st time is 15:30: 1 then the data file will be saved as

C:\Zive data\sm\data\testabc.zsc\2000010115\3001_01.sdo (If data file have DC data only)

Or

C:\Zive data\sm\data\testabc.zsc\2000010115\3001_01.sde (If data file have DC data and EIS data)

Or

C:\Zive data\sm\data\testabc.zsc\2000010115\3001_01.seo (If data file have EIS data only)

e.g.) schedule file name with extension\yearmonthdayhour\minutesecond_channel number

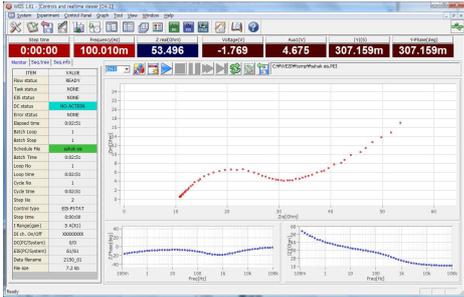
If you input file name as “tommy” then

C:\Zive data\sm\data\testabc.zsc\2000010115\tommy3001_01.sdo (sde,seo)

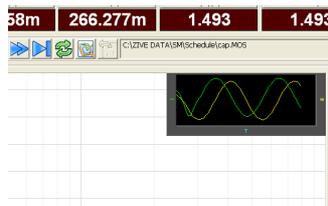
- ① Start Step: If your sequence file has several steps, you can select starting step.
 - ② Start file: If batch file is loaded, select file number
 - ③ Current polarity: If you want reverse polarity for measured current, check on this box.
 - ④ Active area: For calculation of current density
 - ⑤ Capacity: For C-rate value calculation
 - ⑥ Weight: For specific capacity calculation
 - ⑦ Tester: Operator name
 - ⑧ Memo: Text information for test (For line change, use CTRL+ENTER)
- No Save file: If you click this check box, the data will NOT be saved.

If you are ready to test, click OK button then experiment will be started.

You can see real time plot as the following.



Real time EIS graph shows sub window at right upper side. This window display sine wave or E vs. I graph to check data quality. Default display is sine wave.



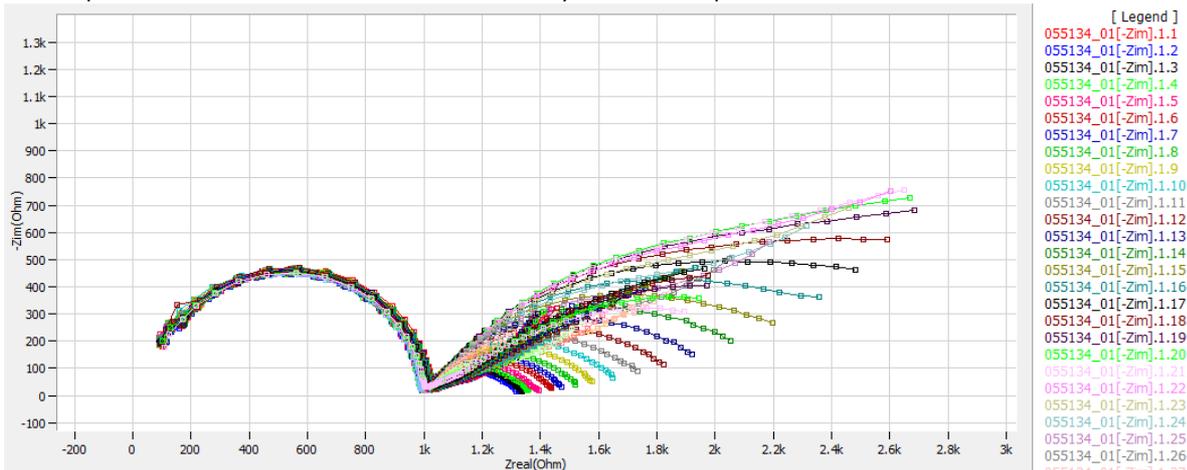
This shows two sine wave graphs. One is for current (green line) and the other is for voltage (Yellow line).

If you switch this graph to I vs E, double click on the graph then graph format will be changed.



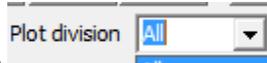
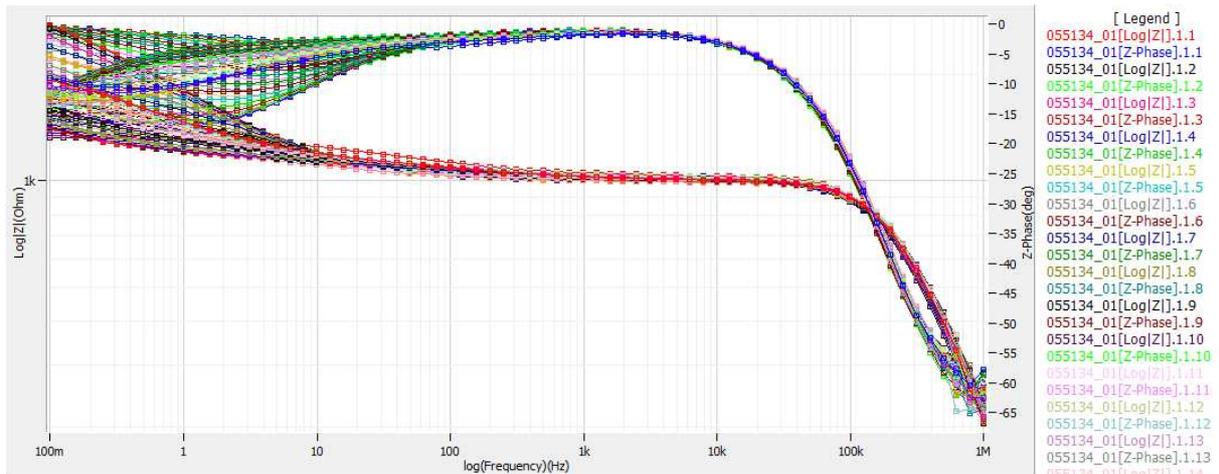
9. You can display graphic by clicking button  on tool bar

Click "Open data file" icon and select data file which you run the experiment.





You can select Bode plot by clicking  icon or Nyquist plot by clicking  icon.

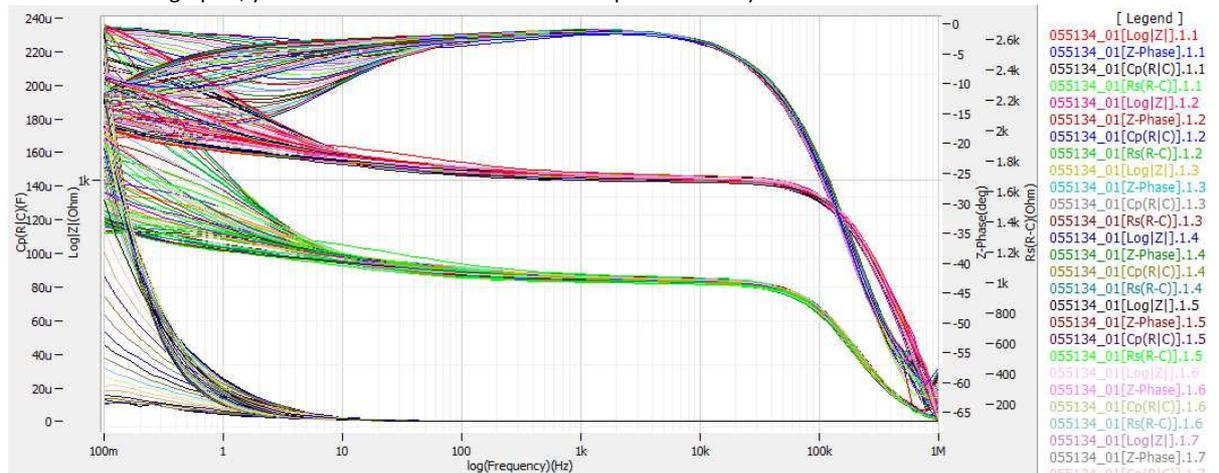


SM can split each EIS graph by selecting "step" in "division".

You can split data by file, cycle or step. Splitted data set's color was changed and graph line will be broken

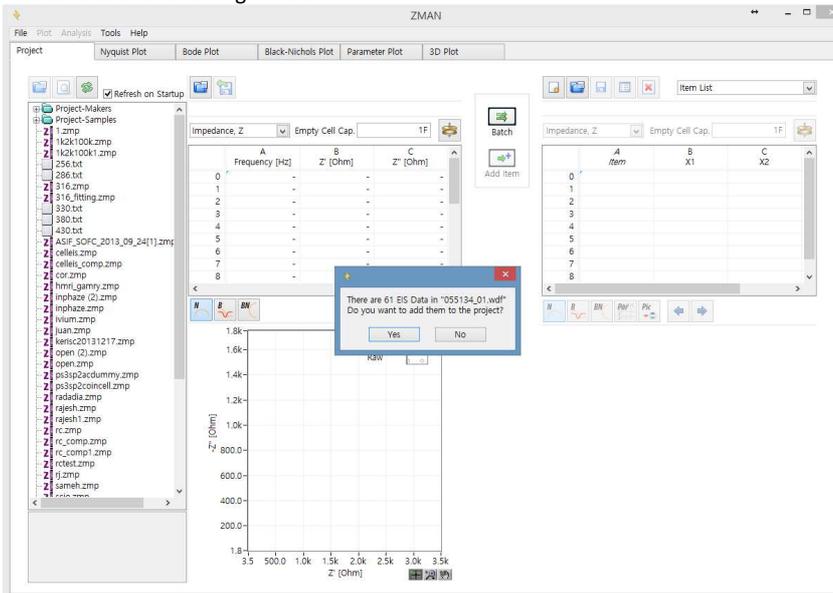
ZMAN will split each EIS data automatically if you insert "cycle" mark.

For user defined graphic, you can select X axis and Max 4 Y axis parameter as you wanted.

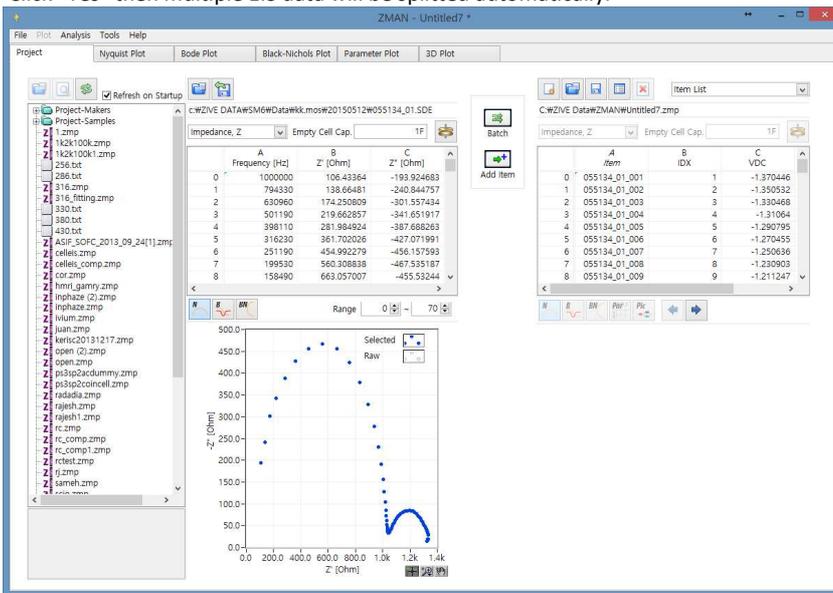


Data Analysis

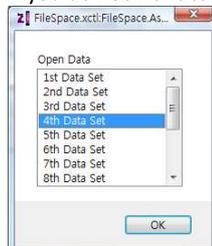
1. Open "ZMAN" by clicking ZMAN icon. ZMAN program is located c:\program files\zivlab\zman2.2 folder.
2. You can see the following window.



3. Click "Yes" then multiple EIS data will be splitted automatically.

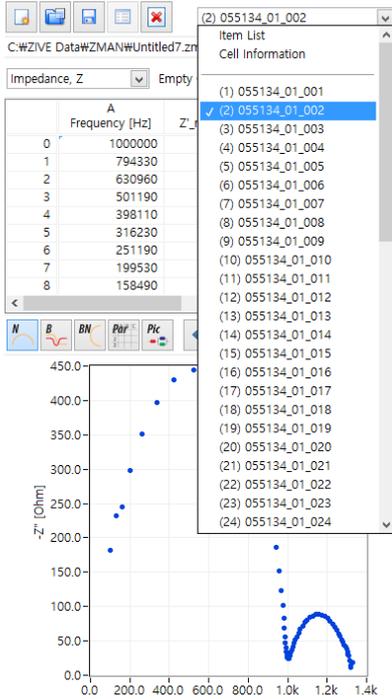


If you do not want to use all EIS data set, Click "No" then you can see select box.

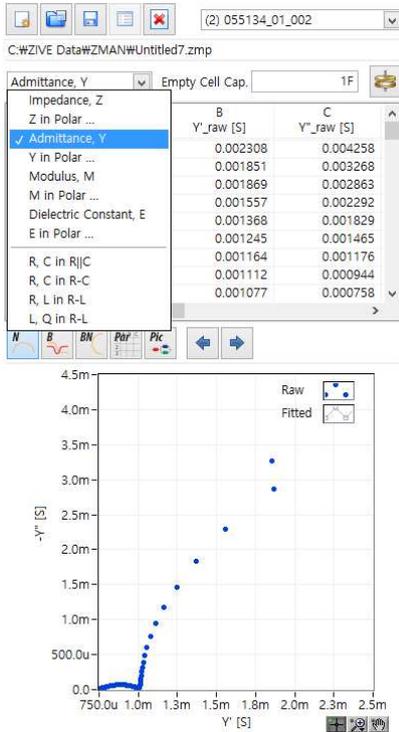


You can select one EIS data for analysis.

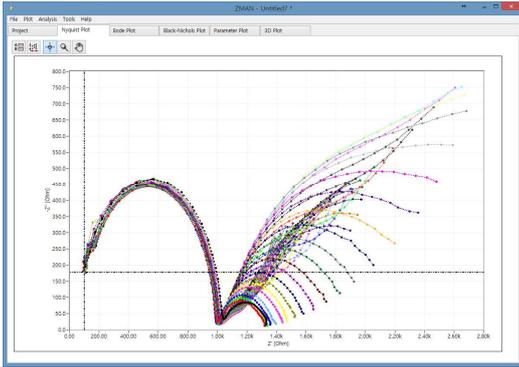
4. You can see Impedance, Admittance, Modulus, Dielectric Constant by selecting as the following
 a) Select data set



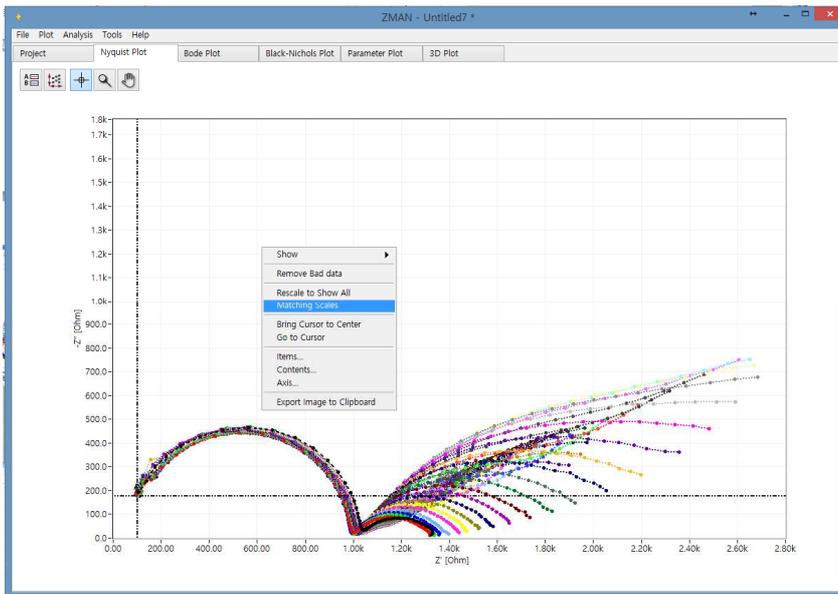
- b) Select Impedance, Z in polar, Admittance, Y in Polar, Modulus, M in polar, Dielectric Constant, E in polar. (in polar = in Polar Coordinate)



5. Click 2D nyquist plot

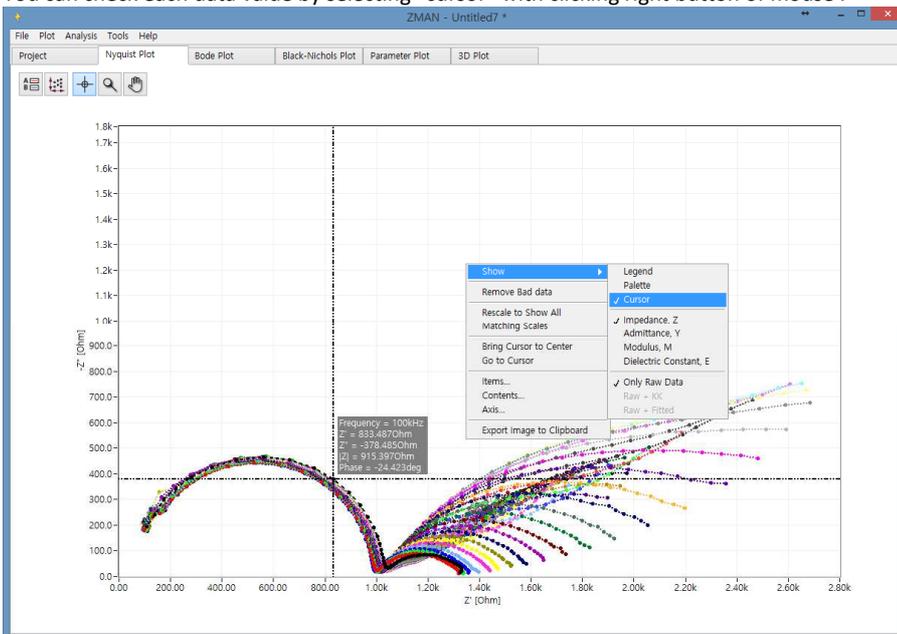


You can show plot as 1:1 scale for nyquist plot by clicking right button of mouse and select “matching scale”



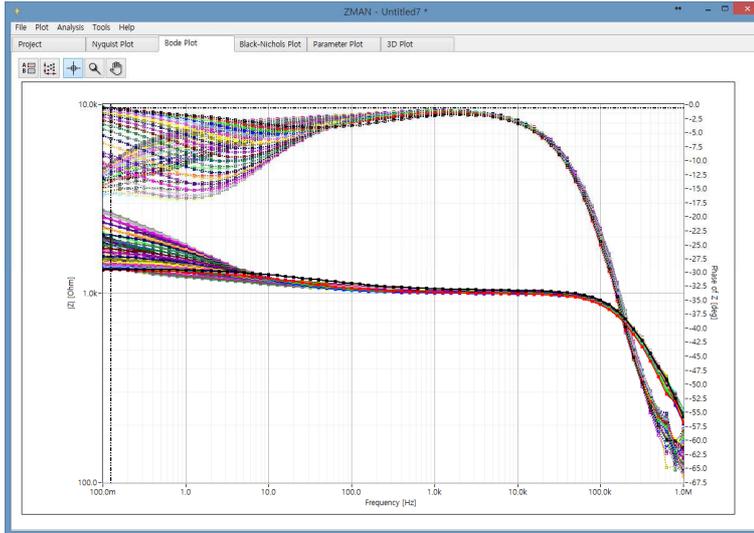
Matching scale is X axis scale and Y axis scale on graph is same.

You can check each data value by selecting “cursor” with clicking right button of mouse .



You can move the point by dragging the mouse after check cursor icon .

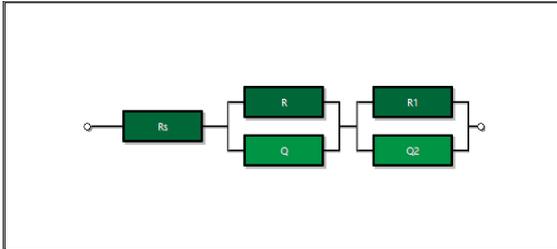
6. Click 2D Bode



Analysis

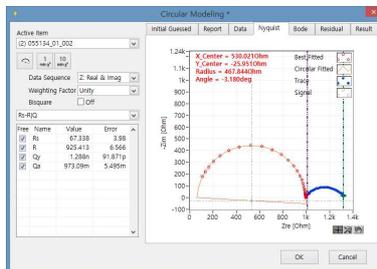
A. Two circles Nyquist spectra (model fitting using circular fit: Manual Modeling)

1. You can select fitting method by your purpose Circular fit or Modeling & fitting

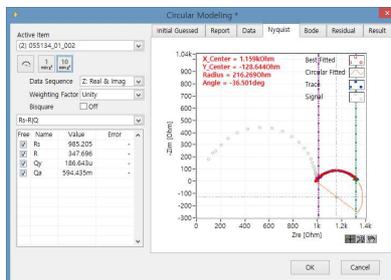


a. Circular fit

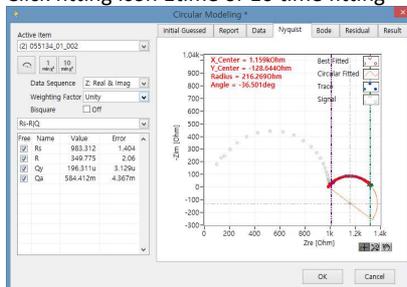
a. Select circular fit on Analysis and Move left(biolet) & right cursor(green line) into 2st circle region



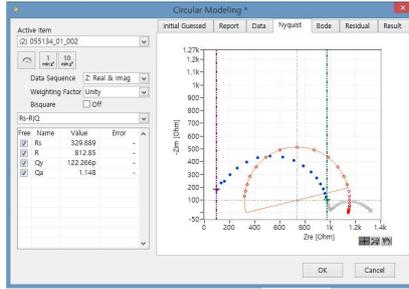
b. click Initial guessing icon



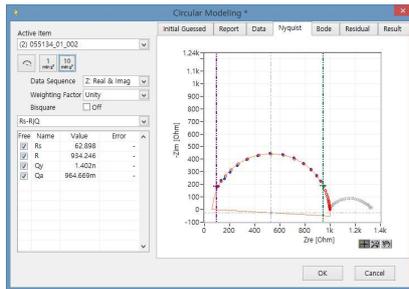
c. Click fitting icon 1time or 10 time fitting



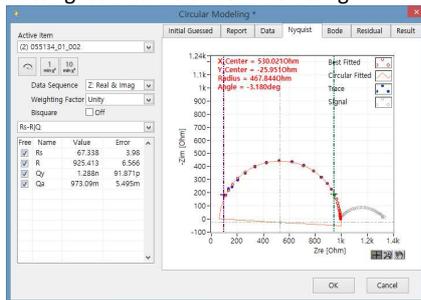
d. Move right cursor (green line) into 1st circle region



e. B click Initial guessing icon

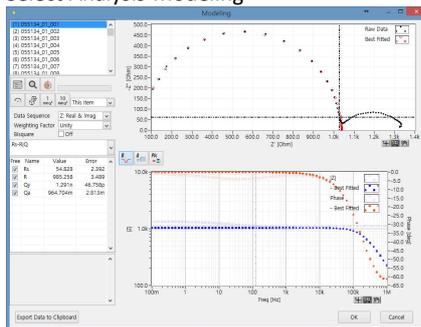


f. Click fitting icon 1time or 10 time fitting

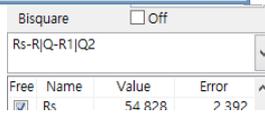


g. Click OK button

h. Select Analysis-Modeling



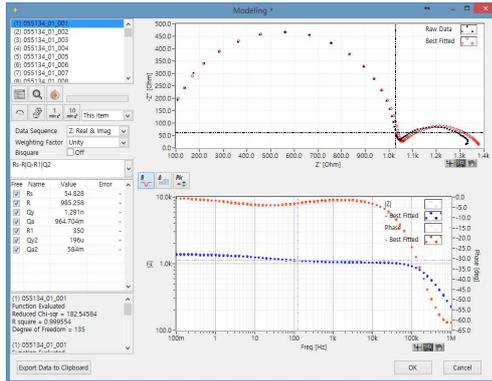
i. Input model as



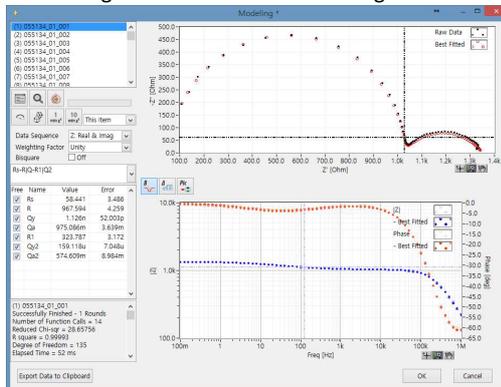
j. Input value for R1 and Q2 which you got the value in circular fitting.

Free	Name	Value	Error
<input checked="" type="checkbox"/>	Rs	983.312	1.404
<input checked="" type="checkbox"/>	R	349.775	2.06
<input checked="" type="checkbox"/>	Qy	196.311u	3.129u
<input checked="" type="checkbox"/>	Qa	584.412m	4.367m

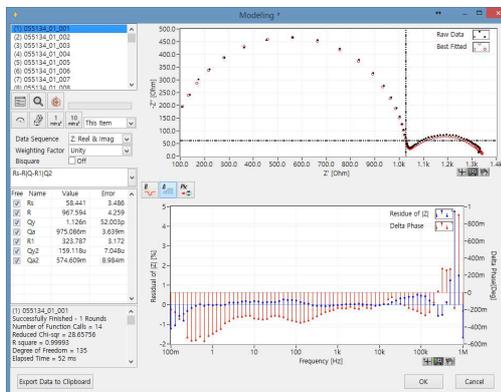
Free	Name	Value	Error
<input checked="" type="checkbox"/>	Rs	54.828	-
<input checked="" type="checkbox"/>	R	985.258	-
<input checked="" type="checkbox"/>	Qy	1.291n	-
<input checked="" type="checkbox"/>	Qa	964.704m	-
<input checked="" type="checkbox"/>	R1	350	-
<input checked="" type="checkbox"/>	Qy2	196u	-
<input checked="" type="checkbox"/>	Qa2	584m	-

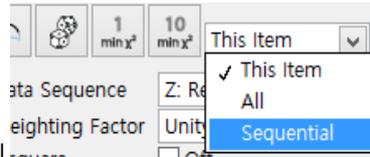


k. Click fitting icon 1 time or 10 time fitting

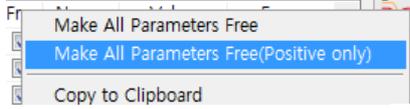


Free	Name	Value	Error
<input checked="" type="checkbox"/>	Rs	58.441	3.486
<input checked="" type="checkbox"/>	R	967.594	4.259
<input checked="" type="checkbox"/>	Qy	1.126n	52.003p
<input checked="" type="checkbox"/>	Qa	975.086m	3.639m
<input checked="" type="checkbox"/>	R1	323.787	3.172
<input checked="" type="checkbox"/>	Qy2	159.118u	7.048u
<input checked="" type="checkbox"/>	Qa2	574.609m	8.984m

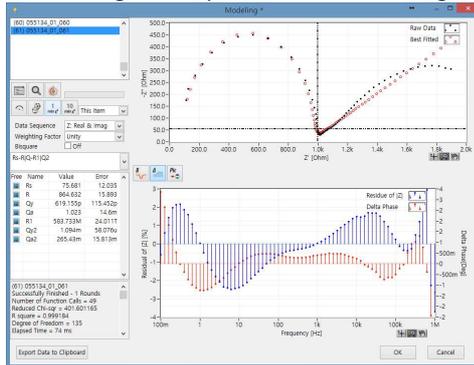




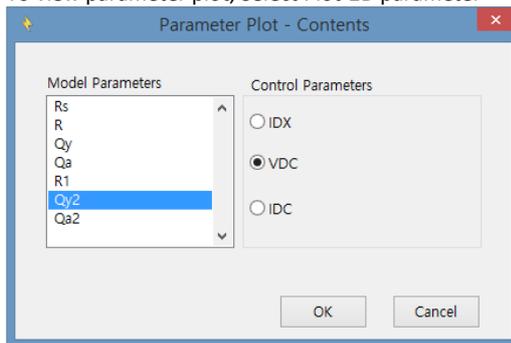
- l. To fit other impedance data with this model, select sequential
- m. Select parameter as positive one by clicking right mouse on "Free" location



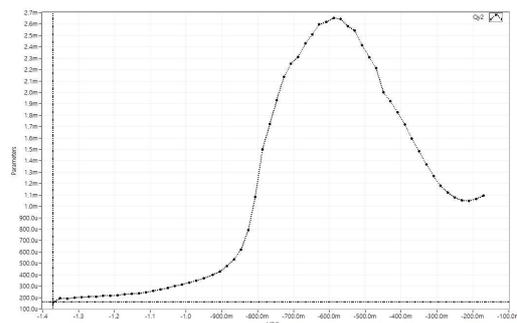
- n. Click fitting icon 1time or 10 time fitting then ZMAN software will fit other impedance data sequentially.
- o. When fitting is over, you can see the following



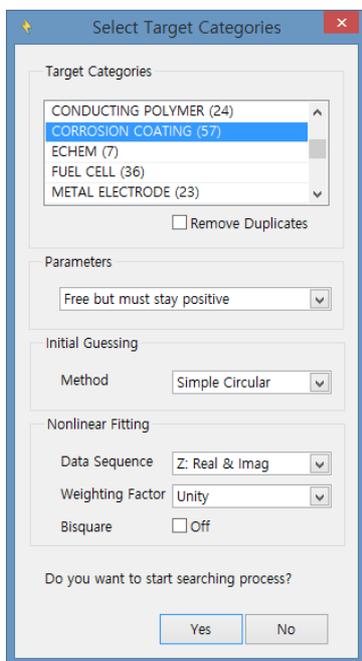
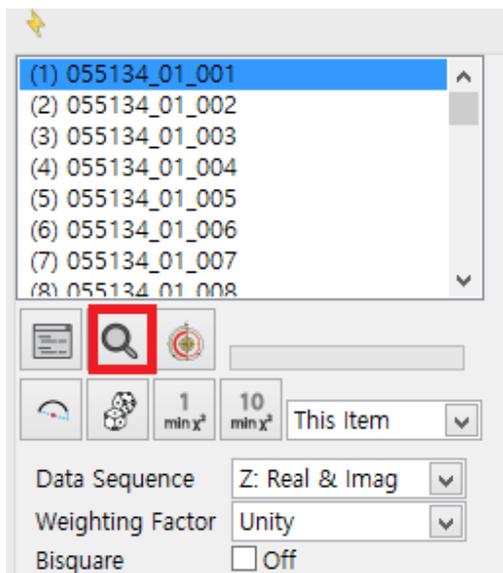
- p. To view parameter plot, Select Plot-2D parameter

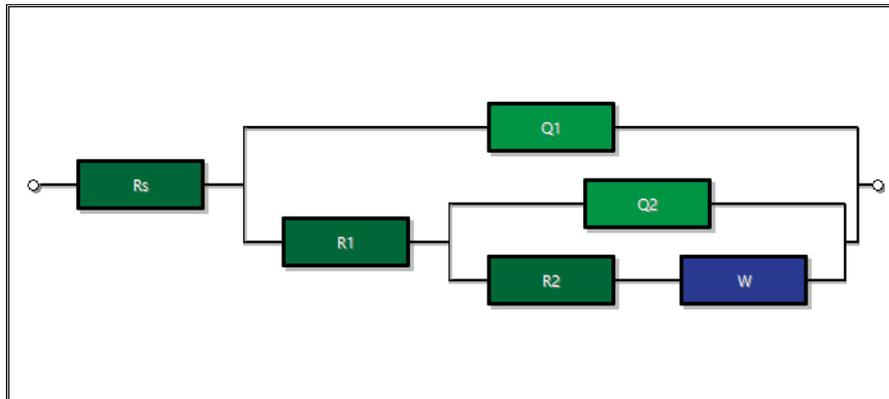
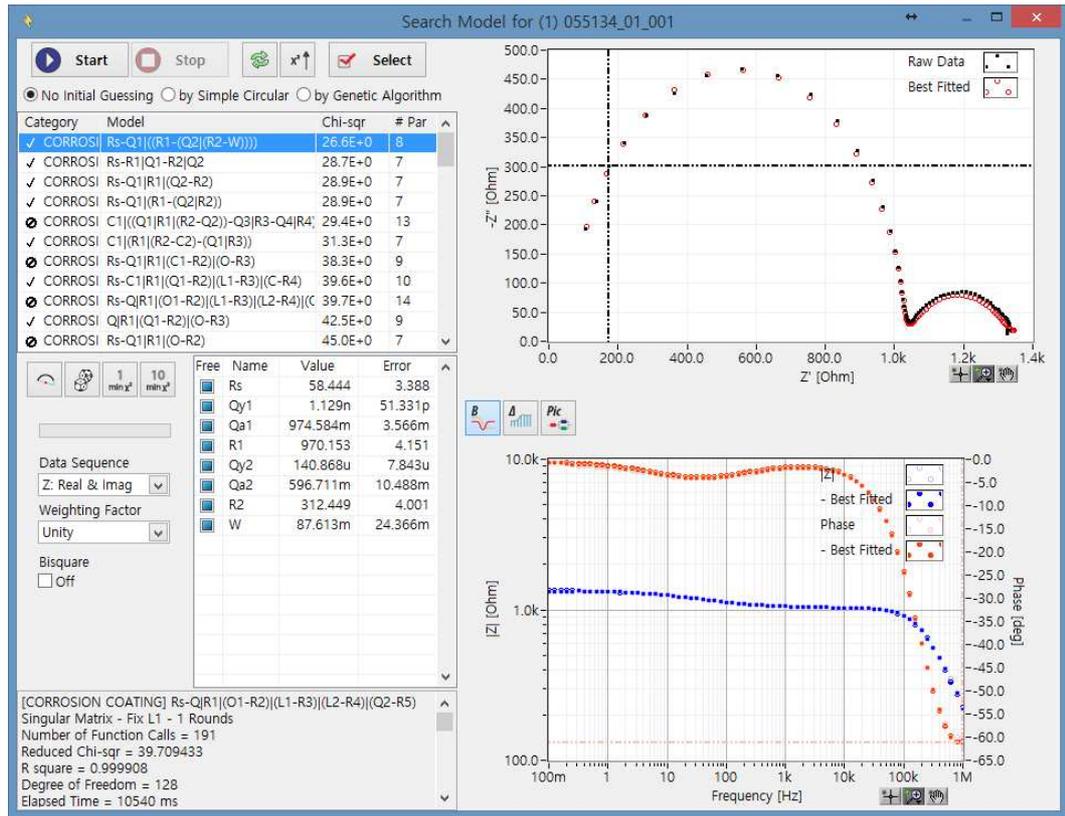


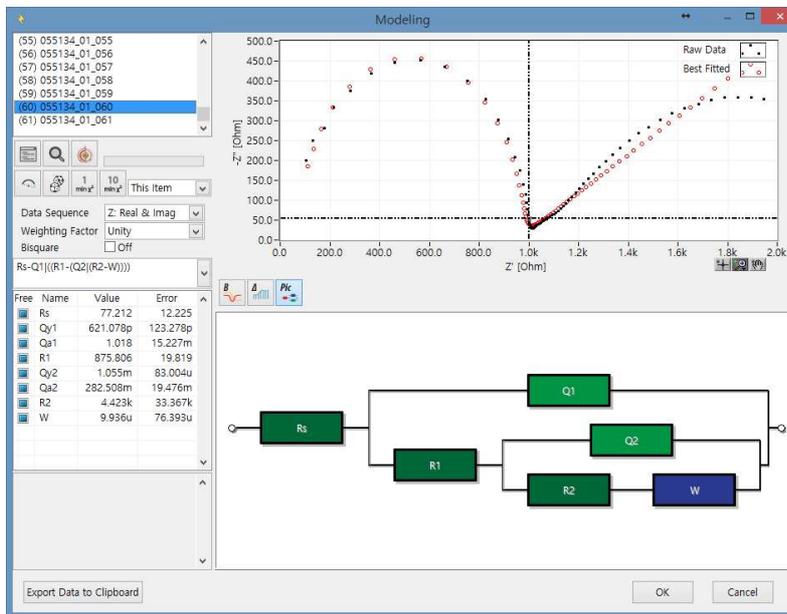
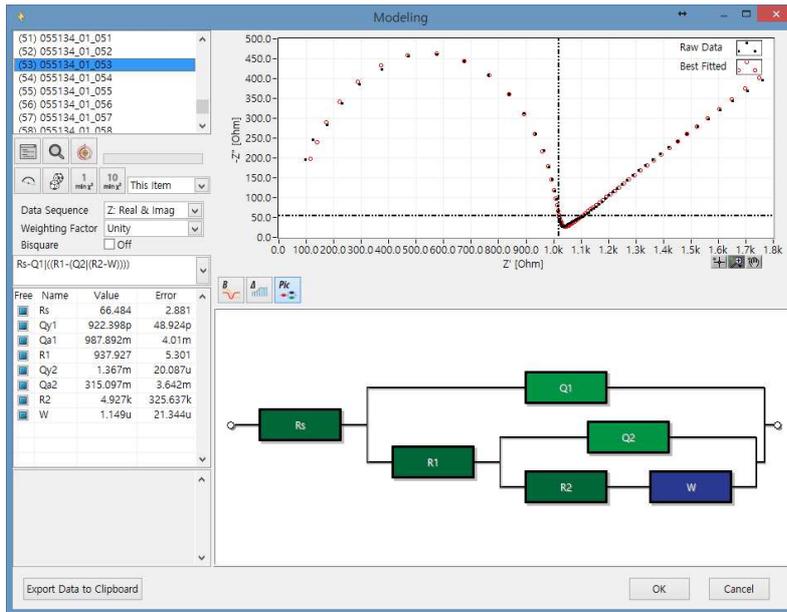
Select parameter(s) and click apply button

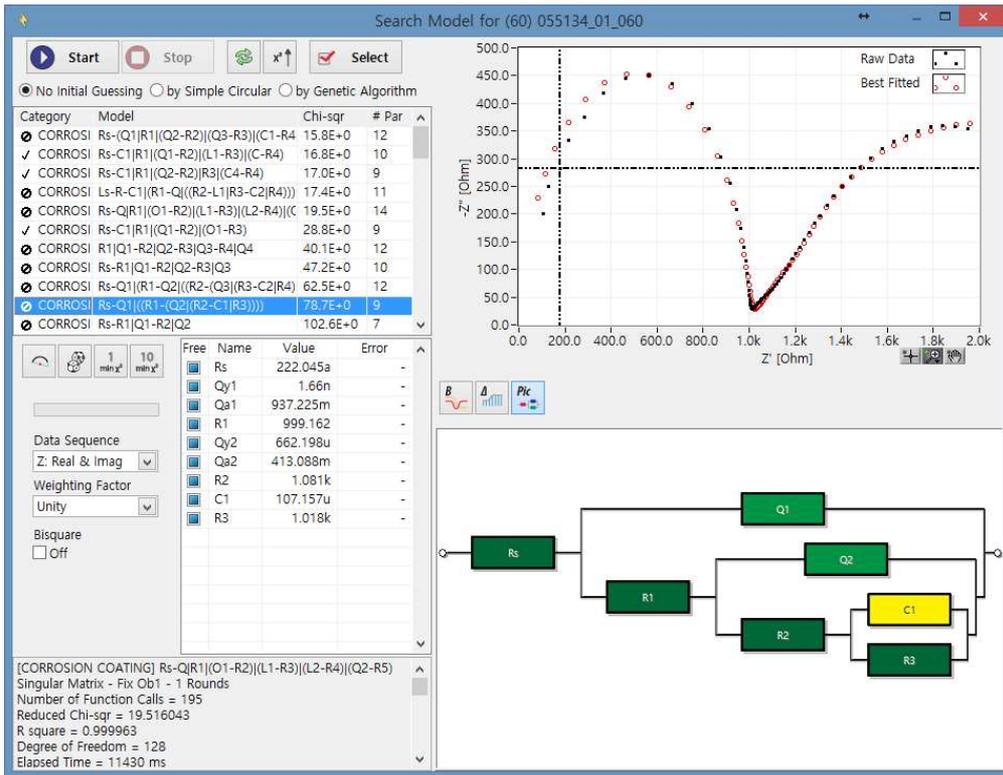


B. Automatic Model Searching

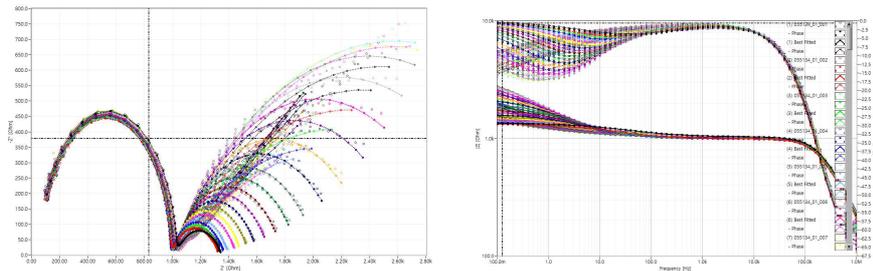




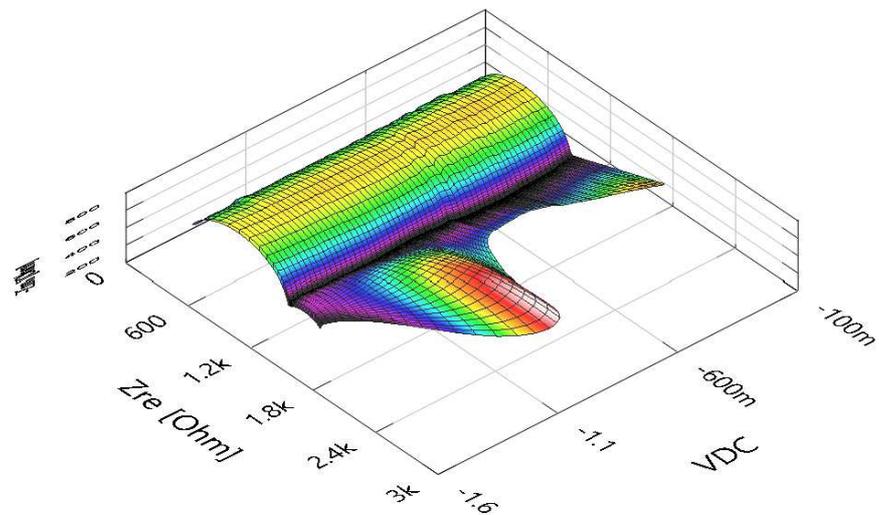




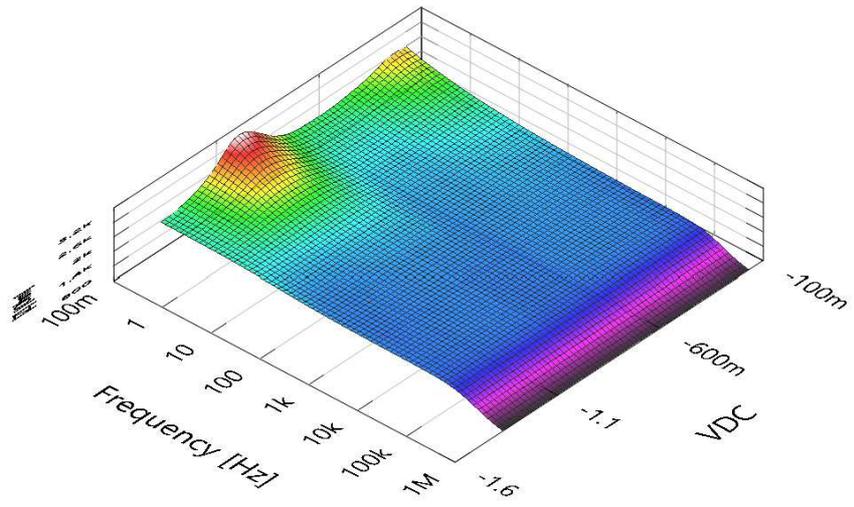
a. You can see Nyquist or Bode data with fitted data



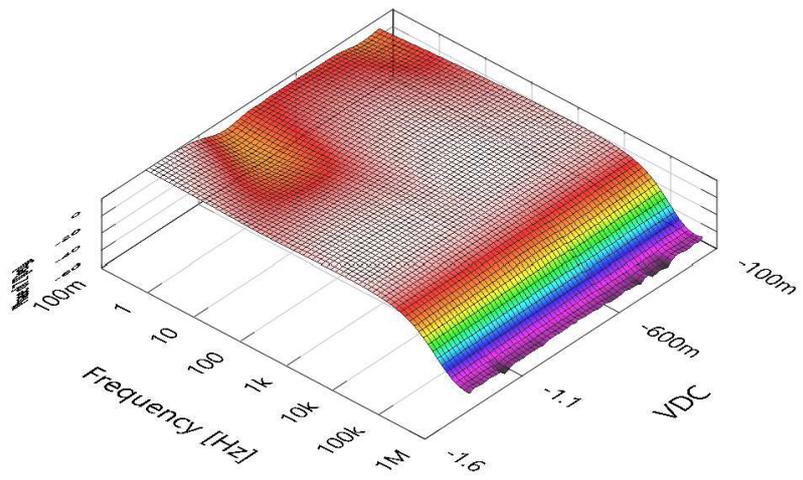
b. 3D graph



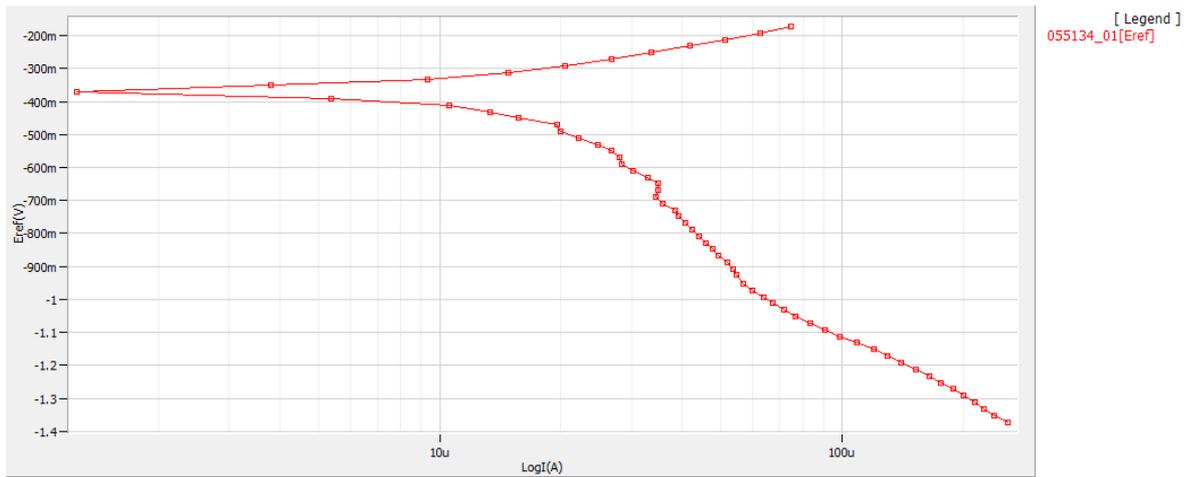
Nyquist plot vs potential



Bode Impedance vs potential



Bode phase vs potential



Tafel plot during DC potential scanning

