EIS measurement after each multiple charging/discharging cycles

Purpose

This test is to demonstrate coin cell battery's charging/discharging cycle with EIS measurement. If you want measure eis after multiple charging/discharging cycles, you can use batch function to do it.

You can make technique file for CC/CV cycle test using CC/CV test or CC/CC test in technique menu and prepare EIS test technique file.

This demonstration's test condition is;

- Constant current charging: 20 mA
- Constant voltage charging: 4.2V
- Constant current discharging: 20mA
- EIS measurement per 3 charging/discharge cycles
- Total 30 cycle charging discharging and 10 times PEIS measurement
- EIS measurement at each capacity interval
 - Initial frequency: 1MHz
 - Final frequency: 0.1Hz
 - Amplitude: 5mV
 - Points per decade: 10

Preparation

- ZIVE SP/MP electrochemical workstation
- 4.2V Li ion Coin cell
- 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.

SM ZIVE* Smart Manager™ 3, 1, 0, 0	1 device found.
ZIVE LAB	Ok

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. CC/CV cycle test technique file: Click New technique function icon (or select Experiment-Techniques on Experiment menu) Then you can see the following menu

Basic 🔽 EIS 🖾 Energy 🖾 Corrosion 🖾	Ecnem	* Technique - CC/CV to							Open	1
Cyclic voltammetry	^	Parameters Informat	ion							
Ru Measurement		ITEM	VALUE	1	Opt	ion			Save	
P-Ru Measurement		First action	Charge	-	L CH	n Dch		11	Save a	
G-RU Measurement ESp ackage(EIS) Static frequency scanning Potentiostatic EIS Galvanostatic EIS OCP EIS		Charge						111	Jave a	15
		-Const. charge	20m		Current		-			
		-Const. voltage(V)	4.2000e+0			5		Ш.	Apply to	C
		-Charging time(s)	1:40		Er Er	nable		111		
Pseudo gavanostatic EIS		-Limit current(A)	15m		Current		-		Close	ł
Dynamic frequency scanning		-Limit capacity(AHr)	10.000e-3		F Er	nable		111	-	_
Potentiodynamic PEIS Galvanodynamic GEIS		-Rest time(s)	10		🔽 Er	nable				
Intermittent frequency scanning		Discharge								
🖉 Intermittent potentiostatic EIS		-Const. discharge	20.000e-3		Current		-			
Intermittent galvanostatic EIS Exed frequency		-Const. voltage(V)	2.7000e+0							
Potentiostatic HFR		-Discharging time(s)	1:40		F Er	nable				
🖉 Galvanostatic HFR		-Limit current(A)	10.000e-3		Disable		•			
Potentiodynamic HFR Galvanodynamic HFR		-Limit capacity(AHr)	0.0000e+0		F Er	nable				
Multisine		-Rest time(s)	10		🔽 Er	nable				
🦳 🖉 Multisine PEIS		Cycle		3						
Multisine GEIS		Sampling								
Energy package(BAT) CC/CV Test		-Time(s)	10		🔽 Er	nable				
CC/CC Test		-Delta voltage	5.0000e-3		🔽 Er	nable				
Discharge test		-Delta current	200.00e-6		🔽 Er	nable				
Variable scan rate CV		IR Measure	🗌 On							
Pstat IV Curve	Ų	I Range(A)	1 A	-	V 1	Auto		-		

- 4. Click "Save" button to save the technique file which contains the above parameter and save it as "cccvtest1" file name.
- 5. PEIS technique file: Click New technique function icon (or select Experiment-Techniques on Experiment menu) Then you can see the following menu

Cyclic voltammetry	^	Parameters Informa	tion				Open
Fast potential sweep Ru Measurement P-Ru Measurement		ITEM	VALUE	Option		•	Save
		Initial delay	✓ Enable				Save as
G-Ru Measurement EIS package(EIS)		-Duration(s)	1:40				Sdve ds
Static frequency scanning		-Stability(V/s)	1.0000e-3				
Potentiostatic EIS		Bias potential(V)	0.0000e+0	Eoc	•		Apply to CH
Galvanostatic EIS OCP EIS Pseudo gavanostatic EIS Dynamic frequency scanning		Amplitude(V)	5.0000e-3		_		-
		Initial freq.(Hz)	1.0000e+6				Close
		Middle freq. (Hz)	1.0000e+6				-
Potentiodynamic PEIS Galvanodynamic GEIS		Final freq. (Hz)	100.00e-3				
Intermittent frequency scanning		Sweep type	Log	-			
🖉 Intermittent potentiostatic EIS		Density		10			
Intermittent galvanostatic EIS		Iteration		1			
Fixed frequency Potentiostatic HFR		Init. I Range(A)	1 A	▼ 🔽 Aub			
Galvanostatic HFR		Speed	Normal	-			
Potentodynamic HFR Galvanodynamic HFR Multisne PEIS Multisne PEIS Multisne GEIS Cover Vest CC/CC Test Occhroge test Vest Sest Vest Sest Vest Vest Vest	ŭ					Ţ	

6. Click "Save" button to save the technique file which contains the above parameter and save it as "peistest1" file name.

SM SN	ART N	Manager	- Sing	le (Channe	el V	irtual c	ontro	ol pi								
Syste	m(S)	Experim	nent(E)	Contro	ol P	anel(C)	G	apł								
X	\$ 6		quen chniq		ile Edit (T)	tor((S)	>	T								
		Ba	atch Fi	le E	ditor(E	3)											
									10 C								
						.,											
Click	"Add	" butto	n and	d se	elect	"cc	cvtes	:1.c	cv" an	d "pe	istest	1.pei"	file				
		" butto		d se	elect	"cc	cvtes	:1.c	cv" an	d "pe	istest	1.pei"	file		↔	_	
Batch		- Untitled.zb	t	d se			cvtes		cv" and		istest				↔ Delete	-	
Batch	schedule	- Untitled.zb	t								nsert[Dn]		[Up]	[-	
Batch	schedule	- Untitled.zb	it			Ар					nsert[Dn]	Insert	[Up] s)	[_	Clo
Batch	schedule Batch Fil	- Untitled.zb Save	tting Loop		as	Ар	ply to Char Chg	nel		I	nsert[Dn] Sc	Insert	[Up] s)			-	Clo
Batch	Batch Fil Enable	- Untitled.zb Save	tting Loop Next		Loop En	Ар	ply to Char Chg C:/Zive	nnel	Add	Ir	nsert[Dn] Sc 1.CCV	Insert	[Up] s)				Clo
Batch Open Index 1	Batch Fil	- Untitled.zb Save Se Count	tting Loop Next Next		Loop En Next	Ар	ply to Char Chg C:/Zive	nnel	Add	Ir	nsert[Dn] Sc 1.CCV	Insert	[Up] s)			_	
Batch Open Index 1	Batch Fil	- Untitled.zb Save Se Count	tting Loop Next Next		Loop En Next	Ар	ply to Char Chg C:/Zive	nnel	Add	Ir	nsert[Dn] Sc 1.CCV	Insert	[Up] s)				 Clos
Batch Open Index 1	Batch Fil	- Untitled.zb Save Se Count	tting Loop Next Next		Loop En Next	Ар	ply to Char Chg C:/Zive	nnel	Add	Ir	nsert[Dn] Sc 1.CCV	Insert	[Up] s)				
Batch Open Index 1	Batch Fil	- Untitled.zb Save Se Count	tting Loop Next Next		Loop En Next	Ар	ply to Char Chg C:/Zive	nnel	Add	Ir	nsert[Dn] Sc 1.CCV	Insert	[Up] s)			_	

9. Click Enable for index2 to edit cycling of this batch process and input "10" for count. Select "index1" for next column.

Open	Open Batch File Save Save as Apply to Channel Add Insert[Dn											
Index		S	etting Loop				5					
Index	Enable Count Next Loop End						Chg					
1		1	Next	-	Next	Ŧ		C:/Zive Data/SM6/schedule/cccvtest1.CCV				
2	•	10	Index-1	-	Next	Ŧ		C:/Zive Data/SM6/schedule/peistest1.PEI				

🐻 Batch schedule - Untitled.zbt

- 10. Click "Save" button to save this batch file using "test1" batch file name
- 11. Select test1.zbt file by clicking "assign schedule file" icon

710712(1)	Sam S(rm)	Em rid (onny	Exprire(onny
3.979	9.073m	0.000	0.000
	N 😵 🔯 🔃 🕬	'zive data₩sm6₩schedule₩te:	t1.zbt
T			

To start experiment, click Start button

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

Start test co	nfiguration				
Sм	c:WZIVE DATAWS	M₩			
File name					
Start file	File-1	Start step Step-1	•	Current p	olarity
Active area	(cm^2) 1.000000	Capacity(mAHr)	1.000000	Weight(g)	.000000
Tester				(Next line : CT	RL+ENTER)
Memo					
🗆 Not sa	ve file			ок	Cancel

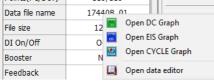
You can see real time plot as the following.

Test time		Voltage(V)	Current(A)	Aux1(V)	Sum Q(Ah)	Lin. Ru(Ohm)	Exp. Ru(Ohm)	2
0:32:3	39	4.201	17.589m	2.799	-8.229m	0.000	0.000	
Anitar Seq.tree	Seq.info	Channel St						_
ITEM	VALUE	0	utematik 🗉 💉 🕺 🛅	3 🕨 🗖 🚺 🔊	• N 🥵 🔯 🖄 🚥	ve catamsmomschedulewces	1.200	
Flow status	RUN							
Task status	RUN STEP							
EIS status	NONE	4.2-						
DC status	Charge	4-						
Error status	NONE							
Elapsed time	0:32:39	3.8-						
Batch Step	1	\$3.6-						
Schedule File	cccvtest1	(0.6- (0.6- (0.4-	- North Contraction of the Institute of					
Batch Time	0:32:39	83.4-		A CONTRACTOR OF			1	
Loop No	1	3.2-			POOP OF CONTRACTOR OF CONT			
Loop time	0:32:39	3-				Networkson .		
Cycle No	2	3-				Contraction of the local division of the loc		
Cycle time	0:03:51	2.8-					or other designation of the local division o	
Step No	1	2.6-					and the second	
Control type	CONSTANT-CC-C	V	- L - L -					
Step time	0:03:51	0	im 2m	3m 4m	Sm 6m (QI(Ah)	7m 8m	9m	10m
1 Range	100mA(X1)				180/107			
V Range	10 V(X1)			6	20m-		-	
Points(PC/Dev)	453/453	- 1 S			210m-			
Data file name	104623_01	(Alass-		1				
File size	40.6 kb	≥ 3-		~	310m- -20m			
01 On/0#	0000							
Booster	None	0:00:00.00001		:28:48.00001	0:00:00.00001	0:28:48		

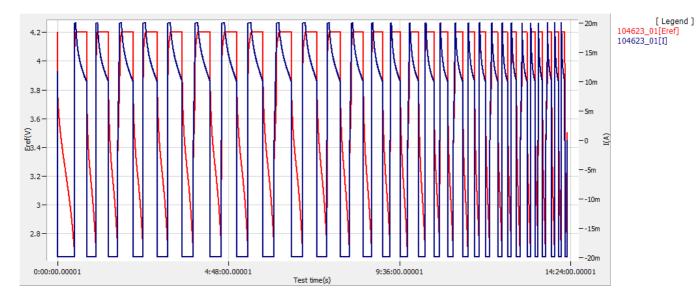
charge-discharge cycling



13. You can display graphic or data editor by clicking right mouse on data file name

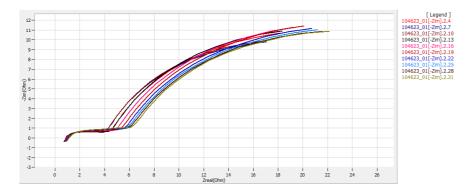


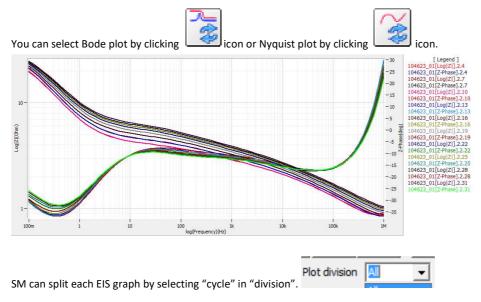
14. You can CC/CV charging-CC discharging profile when you select Open DC graph





15. You can display graphic by clicking button **Less** on tool bar Click "Open data file" icon and select data file which you run the experiment.





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..

Data Analysis

2.

3.

1. Open "ZMAN" by clicking ZMAN icon. ZMAN program is located c:\program files\zivelab\zman2.2 folder.

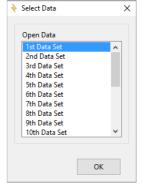
×

	Rp(R)C) <u> </u>		Division \boxed{C} $10 - 26 \qquad \boxed{C} \propto 10^{-26}$	
			-24 ♥ α -22 ♥ α ♥ α	
	e following window.			
*	×			
Yes Click "Yes" ther	No N	itted aut	omatically.	
ZMAN - Untitled2 * File Plot Analysis Tools Help				-
Project Nyquist Plot	Bode Plot Black-Nichols Plot Parameter Plot 3D	Not		
🔛 🧕 🛸 🗌 Refresh on S	tartup 🔛 😭		🕞 🔛 🗔 🔳 🗶 Item List	
	c:\ZIVE DATA\SM6\Data\coinpdeis.MOS\20160212\coincellpdeis_01	SDE	C:\Zive Data\Untitled2.zmp	
	Impedance, Z Empty Cell Cap. 1F	😂 Batch	Impedance, Z 🚽 Empty Cell Cap.	1F
	Frequency [Hz] Z' [Ohm] Z' [Ohm]	Add Item	A B Item IDX	VDC
	0 1000000 0.559139 0.48646 1 794330 0.580035 0.34432	1	0 incellpdeis_01_001 1 1 incellpdeis_01_002 2	3.28 3.338
	2 630960 0.599289 0.22775 3 501190 0.619817 0.13074		2 vincellpdeis_01_003 3 3 vincellpdeis_01_004 4	3.387 3.438
	4 398110 0.643495 0.04739	5	4 sincellpdeis_01_005 5	3.488
	5 316230 0.670846 -0.02400 6 251190 0.702283 -0.08599		5 vincellpdeis_01_006 6 6 vincellpdeis_01_007 7	3.538
	7 199530 0.738408 -0.14065	4	7 vincellpdeis_01_008 8	3.638
	8 158490 0.779181 -0.18916	5 v >	8 vincellpdeis_01_009 9	3.688
			N B BN PNY PK	
	Range 0 🕹 ~ 7			
	13.0-	1		
	13.0- 12.0- Selected			
	13.0-			
	13.0 12.0 12.0 10.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0			
	V Kringe Vig r 130 Setecget - - 11.0 Sever - - 10.0 Sever - - 0.0 - - - -			
	V Kringe Vig r 130 Setecget - - 11.0 Sever - - 10.0 Sever - - 0.0 - - - -			
	V Arrige Vig n 130 Selected 120 Selected 100 Bave 00			
	Kringe Kringe<			
	V Krige Via r 130 Setecget - - 110- Bave - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100- 00- - - 100-			
٤	Kringe Kringe<			
<u>c</u>	Kringe Kringe<			

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

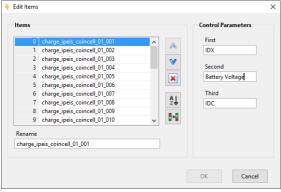
15.0 20.0

10.0 Z' [Ohm] 5.0



You can select one EIS data for analysis.

4. Parameter for voltage name can be changed .for example by clicking "edit item" button



5. Change Idx to cycle No.

ŧ	Edit Items					×
	Items				Control Parameters	
	0	104623_01_001	^		First	
	1	104623_01_002		A	Cycle No.	
	2	104623_01_003		V		
	3	104623_01_004		•	Second	
	4	104623_01_005		×	VDC	
	5	104623_01_006				
	6	104623_01_007		A 1	Third	
	7	104623_01_008		₽↓	IDC	
	8	104623_01_009				
	9	104623_01_010	~	••		
	Rename					
	104623_0	1_001				
					OK Canad	
					OK Cancel	

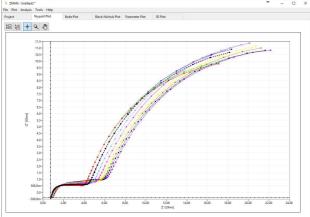
C. LEIVE Data (Ontitied2.200)

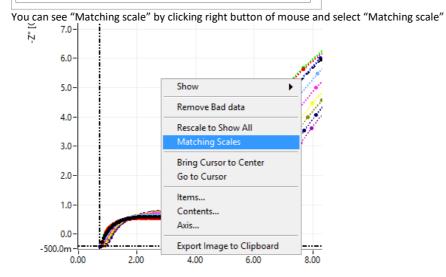
	A	В	С	~
	ltem	ID	Сору	
0	eis_coincell_01_001		Paste	
1	eis_coincell_01_002	-		
2	eis_coincell_01_003		Copy Table	
3	eis_coincell_01_004		Export To Excel	
4	eis_coincell_01_005	-	Select All	
5	eis_coincell_01_006			
6	eis_coincell_01_007		Select Col	
7	eis_coincell_01_008		Select Row	
8	eis_coincell_01_009		Set Column Value	- v

🔶 S	et Column Value					>	×
	B[i] =			×	Cols		
	(i+1)*3;			^ 🗢	A[i]: Item Functions	~	
		From	0 🜩 To	9 🕈	abs(x)	\sim	
				J •			
					ОК	Cancel	

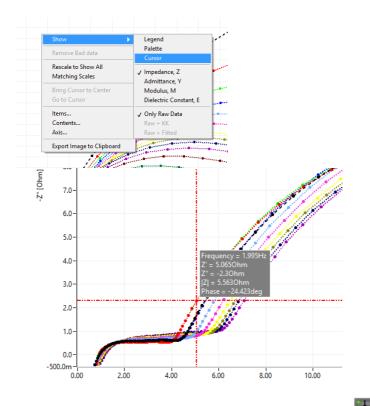
	A	В	С
	ltem	Cycle No.	VDC
0	104623_01_001	3	3.591088
1	104623_01_002	6	3.554358
2	104623_01_003	9	3.540634
3	104623_01_004	12	3.582112
4	104623_01_005	15	3.61628
5	104623_01_006	18	3.641113
6	104623_01_007	21	3.663592
7	104623_01_008	24	3.674739
8	104623_01_009	27	3.685508
<			

6. Click 2D nyquist plot



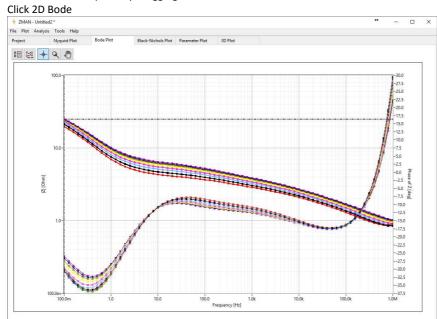


Matching scale is X axis scale and Y axis scale on Nyquist graph is same. You can check each data value by selecting "cursor" with clicking right button of mouse .



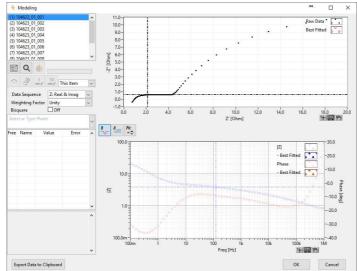
You can move the point by dragging the mouse after check cursor icon

7.

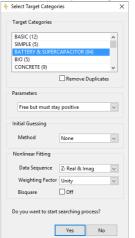


Anaysis

a. Select data



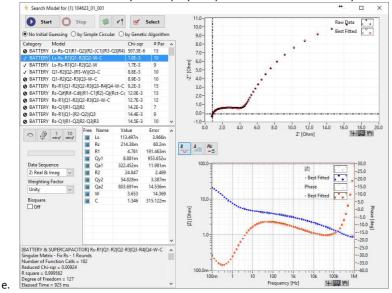
b. Search Model by clicking "search" button



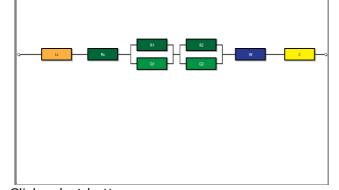
c. Select Battery library and None for Method.

d. Click Yes buton

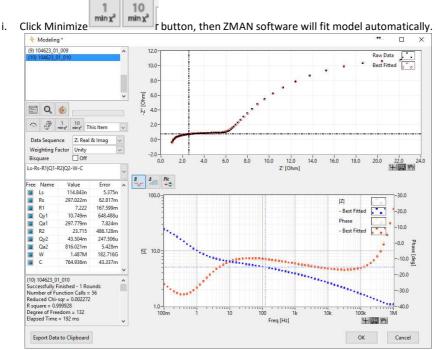
ZMAN software will try to find proper equivalent circuit automatically.



f. Select the simplest model on upper row.



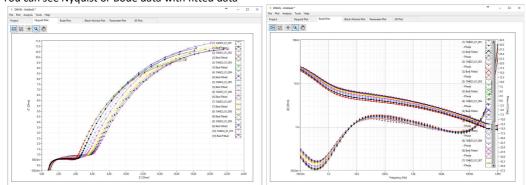
- g. Click select button
- h. Select sequential to fit other eis data set.

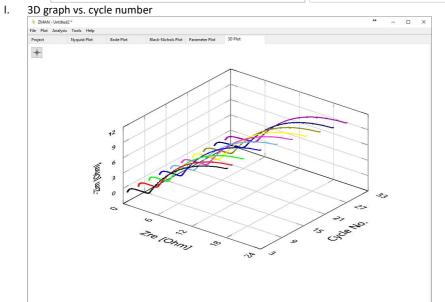


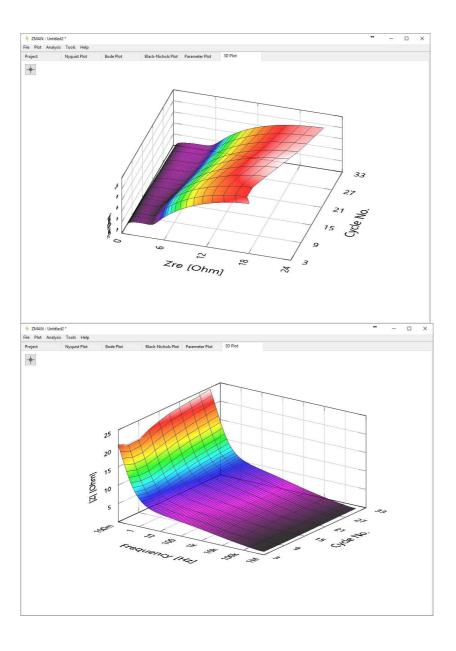
j. If you want overlay raw data and fitted data, Plot detail in Plot menu

(1) 104623(2) 104623			^
(3) 104623			
(4) 104623	01_004		
(5) 104623			
(6) 104623			
(7) 104623			
(8) 104623(9) 104623			
O Show O	nly Raw D	ata	
O Show R	aw and KK	Data	
Show Ri	aw and Fit	ted Data	

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

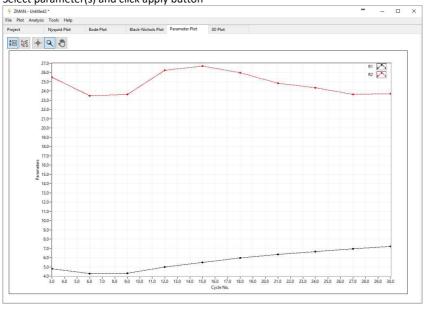






m. To view parameter plot, Select Plot-2D parameter

Ls	^	Control Parameters
Rs		Ocycle No.
R1		0
Qy1 Qa1		OVDC
R2		0.00
Qy2		
Qa2	~	



Select parameter(s) and click apply button



Copyrighto 2011 ZiveLab