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Impedance Spectroscopy Analysis using CNLS Simulation for Lithium Bismuth Borate Glass containing 5 mol% d-Block Nickel & Vanadium Ions

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Abstract: In this research work, an equivalent circuit model has been designed for Lithium Bismuth Borate Glass which contains 5 mol% nickel and vanadium ions using CNLS fitting achieved with the help of LEVMW program. The equivalent circuit model is outlined on the basis of immittance spectroscopic measurements and the parameters' estimation made in the input file till a valid run is achieved. The immittance spectroscopic measurements include the variations of resistance, capacitance, conductivity, electric modulus etc. with respect to frequency and temperature.

Keywords: Immittance Spectroscopy (IS), CNLS (Complex Nonlinear Least Squares), LEVM, NVLBB3 (Nickel Vanadium Lithium Bismuth Borate Glass), DE (Distributed Elements), Standard Deviation (SD).

I. INTRODUCTION

IS is a common term that includes the computation of small-signal frequency response of semiconductors, dielectrics, single-crystal conductors, electrolytes etc. It is a broad term that involves four levels namely impedance, admittance, complex electric modulus and dielectric constant (or complex capacitance). Data at any one of the four levels can be converted into other by simple calculations. The impedance is denoted by $Z(\omega) = Z'(\omega) + iZ''(\omega)$ where single and double prime denotes the in-phase and quadrature phase components respectively and $i = (-1)^{1/2}$. The admittance is denoted by $Y(\omega) = [Z(\omega)]^{-1} = Y'(\omega) + Y''(\omega)$, complex dielectric constant by $\varepsilon(\omega) = Y(\omega)/(i\omega C_c) = \varepsilon'(\omega) - \varepsilon''(\omega)$ where C_c is the capacitance of the empty measurement cell and the complex electric modulus by $M(\omega) = [\varepsilon(\omega)]^{-1}$. Generally, measurements are made at impedance level so the term IS may also simply refer to impedance spectroscopy rather than immittance spectroscopy [1].

The most favourable investigating method for simulation of IS data to either a mathematical model or to equivalent circuits is CNLS, launched by Garber and Macdonald in 1977. In CNLS procedure, all the variables of a fitting circuit model are modified simultaneously to produce the most appropriate fit to the data [4]. CNLS fitting does not only produce approximate values of the parameters of the chosen circuit model, but it gives estimates of their standard deviations also, which are the measures of how well the parameters have been estimated by the data fit. Values of these standard deviations are of utmost importance in selecting which parameters are decisive to the model and which are of no use, or at least are not well measurable from the given data.

CNLS fits are obtained by a program that minimizes the weighted sum of squares of the real and imaginary residuals. The difference between a data value at a given frequency and the corresponding value determined from the model is called a residual. The inverses of the estimated error variance for a given real data value and that for the corresponding imaginary value are the weights used. Weighting is of prime importance although it is the most subjective part of least squares fitting but it can have critical effects on the outputs of such procedure of fitting very often [4].

The main motive of least squares simulation method is to determine a set of variables P that minimize the following sum:

$$S(P) = \sum_{1}^{N} wi[Fi - FCi(P)]^{2}$$
 -----[3]

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Where N is the total number of data points, w_i is the ith point's associated weight, F_i is the ith data point value to be fitted and FC_i(P) is the corresponding value of the fitting function computed by including the set of parameters P. This technique can be used to fit complex form of data by distributing the imaginary and real data in a single array. This array's size is two times the size of the number of data points. Therefore, if there exist N data points, data array's first element would be the first data point's real part and (N+1)th element would be the first data point's imaginary component. It is important to note that if the fitting function is of nonlinear nature in case of any of its parameters, then iterations would be required by the simulation [3].

Glasses with general composition $x(2NiO.V_2O_5).(30-x)Li_2O.50B_2O_3.20Bi_2O_3$ and x = 5 called Nickel Vanadium Lithium Bismuth Borate (NVLBB3) were fabricated by using melt- quenching technique, which is a very old technique for fabrication of amorphous solids like glass[2]. The datasheet consisting of impedance spectroscopic measurements for this material was provided by Dr. Satish Khasa.

II. LEVMW TOOL

LEVMW is a profound, flexible and powerful complex nonlinear least squares program for simulation, fitting and inversion of time and frequency domain data sets. LEVM is a name not an acronym and LEVMW is its windows version. It has been optimized for exploring dielectric, electrochemical, or high-resistivity conductive-system data. The LEVM tool involves four types of main functions. It may be utilized to fit transient-response or frequency-response data by nonlinear least squares (NLS) or CNLS firstly. It may also be deployed for simulation of various circuits and for Kronig-Kramers testing and also for other model response functions. At last but not the least, it may be utilized to approximate distributions of activation energies (DAE) or relaxation times (DRT) from transient or frequency response data. All outputs may be plotted in 2-D or 3-D form at any of the four immittance levels mentioned earlier [3].

It runs on a PC that has minimum 3MB of extended memory, required for proper operation and has been compiled using the Microsoft FORTRAN Powerstation V.1 compiler, resulting in the executable file LEVM.EXE, which is provided in the tool's LEVM folder. This .EXE file permits up to 42 free parameters and 1001 data values presently. The greater the number of free parameters and the higher the number of data values in a fit, the tool will take longer time to converge to a final least squares solution[3].

Some Features of LEVM:

- It consists of almost hundreds of thousands built-in circuits already ready to use and various new circuits can also be added to it.
- There are various weighting choices (>10).
- Fitting of complex, real or imaginary part is possible.
- Various distributed circuit elements (DCE's) (>30) are available there for circuits.
- Input data may be in any of Z, Y, M or E form.
- Simulation may be at any of the four immittance levels.
- Input/output may be in polar or log-polar form and rectangular form.
- It can be applied to dielectric, conductive or mixed systems.
- It includes powerful 2-D or 3-D plotting programs.
- Effects of selected circuit elements are possible to subtract.

How to use LEVMW:

- To deploy LEVMW for CNLS fitting, there must be an input file available which contains two parts.
- The header (or top) part of the input file (10 to 13 lines) is utilized to set choices which decide how LEVM explores the data, and the data is available in the bottom part of the file.
- Then selecting a fitting circuit and a fitting model to represent the data is a very crucial choice.
- The program is not fully interactive; thus before invoking LEVM the input file must be prepared, although some changes are permitted to be made in it when operating LEVM tool.
- The data should be organized in form of three columns: frequency, real part, imaginary part.

Input parameters for LEVM:

The input parameters for this simulation tool have a power to determine a wide range of program's functions, namely data conversions, data types, choices of fitting model and other parameters of NLLS/CNLS fit. All the input parameters are entered in formatted FORTRAN line-images, therefore their precise and accurate spacing and positioning are very important to ensure valid outputs. The parameter having a default value may be left blank. The primary output of LEVM will appear in the PNTOUTL file and the secondary output in AUXPNTL file [3].



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PNTOUTL file:

By examining this file, firstly the circuit model used is found out and then the parameter values are paired with the real elements in the circuit utilized in the simulation by using the identification list for that circuit. During the simulation, only those parameters with a value 1 or 2 succeeding them are free. The output quantity XS is the chi-squared statistic for the fit whose square root is the estimated standard deviation of the fit, SIGMAF. One more parameter, fit quality factor (FQF) helps in the comparison of two fits. The lesser the FQF is algebraically, the better the fit is generally, however the difference may or may not be significant statistically. The quantities, PRSDAV and PRSDRMS are of primary importance as they evaluate the overall adequacy of a fit. It is a poor fit if these quantities exceed 0.2 or 0.3. These are expected to be less than or equal to 0.03 for a good fit. The quantity SSE in this output file is a measure of negative entropy of goodness of fit [3].

Gradient outputs:

As per least squares condition, as iteration steps in the direction of convergence, the gradients of free parameters should approach the value of zero ideally. So, generally as the iterations progress, each gradient should decrease in magnitude. However, if at every iteration, any parameter's gradient results in zero, then it indicates that parameter is not suitable (or may be not set free to vary) in the fitted circuit. All parameters' gradient values are much smaller (usually less than 10^{-6}) at the end of iteration as compared to the beginning. If this is not the case, then input guesses need better modifications [3].

III. RESEARCH METHODOLOGY

There are five major levels in the methodology of the proposed work:

- 1. Data Acquisition
- 2. Input File Preparation
- 3. Pre-analysis
- 4. Remodeling of parameters
- 5. Equivalent Circuit Model Determination



Fig.1 Different levels used in the analysis process

Data Acquisition:

The datasheet required consists of the measurements of different parameters of impedance spectroscopy of lithium bismuth borate glass namely, series and parallel resistance, series and parallel capacitance, free space capacitance, dielectric loss tangent, reactance, complex electric modulus, AC conductivity, complex dielectric constant in accordance with different frequencies and temperature. An impedance analyzer (HIOKI IM 3570) was used to carry out the impedance spectroscopic measurements over the temperature range of $100^{\circ}C$ -400°C and frequency range 1Hz-5MHz. The experimental errors in the capacitance and resistance measurements were estimated to be ±0.1 pF and ±10 Ω respectively [2]. This datasheet was directly provided by the physics department.



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Input File Preparation:

LEVMW software uses a specific type of input file (in FORTRAN language) which contains a top part that includes fitting choices, followed by a bottom part that consists of three data columns. Input file preparation in Windows is very straightforward. It must be specified in the input file which one of the many available circuits of the software is to be used for simulation.

8		EDIT : LEVMRUN
Save Save As Load Close	Close & Load	
MYDATA E CKT. EXACT PAR. VALU		FREO: BAD/S
0 ZZRR E .1000D+01C	0.0000D+00 0 -11	x
26 40 299 0 2	0 1 2.0000D+00 .2	740D-14 .9441D+00
.44628958D+09 .20499393D-0	.00000000D+00 .1000000D+01	.2000000D+01
.96808304D+01 .76168335D-0	.00000000D+00 .10026896D+01	.2000000D+01
.000000000+00 .0000000000+0	.00000000D+00 .0000000D+00	.0000000D+00
.00000000D+00 .0000000D+0	.0000000D+00 .0000000D+00	.0000000D+00
.00000000D+00 .0000000D+0	.00000000D+00 .0000000D+00	.0000000D+00
.0000000D+00 .0000000D+0	.0000000D+00 .87958428D-07	.0000000D+00
.0000000D+00 .63382710D+0	.0000000D+001000000D+01	.0000000D+00
.0000000D+00 .0000000D+0	.0000000D+00 .0000000D+00	.0000000D+00
110001101000000000000000000000000000000	000000000	
1 .628571400000D+0	.96800000000D+01	333520000000D+09
2 .691428600000D+0	.96800000000D+01	303200000000D+09
3 .817142900000D+0	.96800000000D+01	256560000000D+09
4 .942857100000D+0	.96800000000D+01	222350000000D+09
5 .106857100000D+0	.96800000000D+01	196190000000D+09
6 .119428600000D+0	.96800000000D+01	175540000000D+09
7 .138285700000D+0	.96800000000D+01	333520000000D+09
8 .157142900000D+0	.96800000000D+01	133410000000D+09
9 .182285700000D+0	.96800000000D+01	115010000000D+09
10 .207428600000D+0	.96800000000D+01	101070000000D+09
11 .238857100000D+0	.96800000000D+01	877700000000D+08
12 .270285700000D+0	.14000000000D+07	149790000000D+07
13 .308000000000D+0	.14000000000D+07	131450000000D+07
14 .352000000000D+0	a111000000000D+07	18095000000D+06
15 .4022857000000D+0	.73800000000D+06	.887780000000D+06
16 .458857100000D+0	98200000000D+06	.981680000000D+06
17 .521714300000D+0	.13600000000D+07	144120000000D+07
18 .5971429000000D+0	.23300000000D+07	126870000000D+08
19 .685142900000D+0	33200000000D+07	18960000000D+07
20 .779428600000D+0	33200000000D+07	16660000000D+07
21 .892571400000D+0	.1500000000D+07	378500000000D+07
22 .101828600000D+0	.78300000000D+07	.577670000000D+07
23 .116285700000D+0	.16500000000D+08	.319680000000D+07
24 .132628600000D+0	41600000000D+07	330700000000D+07
25 .1514857000000D+0	309000000000D+07	181400000000D+07
26 .1728571000000D+0	11700000000D+08	635030000000D+07

Fig. 2 Prepared Input File for NVLBB3 at 100°C by using circuit E

Pre-analysis:

At this level, the output obtained as a result of the initial guesses of the parameters' values (representing the circuit elements) in the top portion of the prepared input file is examined. The quantities, PRSDAV and PRSDRMS are expected to be 0.03 or less for a good fit.

Remodeling of parameters:

If the values of the quantities, PRSDAV or PRSDRMS in the output file are not less than or equal to 0.03, then the various parameters in the top portion of the prepared input file, representing the selected fitting model are estimated again and again based on the values of free parameters' SDs mentioned in the previously obtained output file till these above mentioned quantities come within range. Also, some of the parameters' choices in Line 2 and Line 3 of the input file can also be altered to achieve a good fit for the chosen circuit and fitting model.

Equivalent Circuit Model Determination:

If the values of the quantities, PRSDAV or PRSDRMS in the output file are less than or equal to 0.03, then based upon the chosen fitting model parameters' estimation in the top portion of the prepared input file, an equivalent circuit model can be designed. LEVM program consists of fifteen such circuits, namely circuit A, B,...T in the folder FITTESTS.

IV. RESULTS

Figure 3 shows the output PNTOUTL file obtained for the input file shown in figure 2. The quantities PRSDAV and PRSDRMS are 1.9676D-02 and 2.6856D-02 respectively. Both values are less than 0.03 which indicates that the fit obtained is a good one. Also, the FQF has a value of -3.275D+03 which is very small as required for a good fit. Hence, based on the final parameter guesses, especially those representing the chosen circuit elements, an equivalent circuit model has been designed shown in figure 6. The impedance versus frequency curves for NVLBB3 at 100°C temperature is also shown in figure 5.



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EVMVIEW	- 🗆 🗙						
File Help							
Graphics Complex-plane plot Numerical data PNTOUTL AUXPNTL							
LEVM : COMPLEX NONLINEAR LEAST SQUARES IMMITTANCE DATA FITTING PROGRAM	^						
VERSION 8.11 - 2011							
MYDATA E CKT. EXACT PAR. VALUES: ZC(2D6,1D0,0.3),1D+6,1D-12 FREQ: RAD/S							
DATA ENTERED IN ZR FORMAT TO BE USED IN ZR FIT; CIRCUIT MODEL : E							
****** FIT OF COMPLEX DATA ********							
- # OF DATA POINTS = 26 WEIGHT: IRCH = 2 # OF FREE PARAMETERS = 6 - PRINTS FUERY & ITERATIONS MAX # ITERATIONS=299 MAIN WEUSES- DATA							
TO THE POWER 6.338270+00							
INITIAL PARAMETER GUESSES AND FIXED (0) OR FREE (1 OR 2)STATUS							
P(1) = 4.4628960+08 1 P(17) = 0.0000000+00 0 P(2) = 2.0499390-03 1 P(18) = 0.0000000+00 0							
P(3) = 0.0000000+00 0 P(19) = 0.00000000+00 0 P(19) = 0.0000000+00 0 P(19) = 0.00000000+00 0 P(19) = 0.00000000+00 0 P(19) = 0.00000000+00 0 P(19) = 0.00000000+00 0 P(19) = 0.0000000000000000000000000000000000							
P(5) = 2.000000+00 0 P(21) = 0.000000+00 0							
P(0) = 9.680830D+00 1 P(22) = 0.000000D+00 0 P(7) = 7.616833D-09 1 P(23) = 0.000000D+00 0							
P(8) = 0.0000000+00 0 P(24) = 0.0000000+00 0 P(9) = 1.0026900+00 1 P(25) = 0.0000000+00 0							
P(10) = 2.0000000+00 0 P(26) = 0.0000000+00 0 P(11) = 0.00000000+00 0 P(27) = 0.0000000+00 0 P(27) = 0.00000000+00 0 P(27) = 0.000000000+00 0 P(27) = 0.0000000000000000000000000000000000							
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P(13) = 0.0000000+00 0 P(29) = 8.795843D-08 1 P(14) = 0.0000000+00 0 P(29) = 0.0000000+00 0 0 P(30) = 0.0000000+00 0 0 P(30) = 0.0000000+00 0 0 P(30) = 0.00000000+00 0 0 P(30) = 0.000000000000000000000000000000000							
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P(35) = 0.000000000000 P(39) = 0.0000000000000000000000000000000000							
-NO TRANSFORMATION MADE: DATA IN ZE FORMAT							
TERMINATION: XTOL IS TOO SMALL, NO FURTHER IMPROVEMENT IN THE FREE							
PARAMETERS IS POSSIBLE.							
FINAL L2 NORM OF THE RESIDUALS 0.18807940-13 NUMBER OF FUNCTION EVALUATIONS 32							
NUMBER OF JACOBIAN EVALUATIONS 8							
WEIGHTED SUM OF SQUARES, S: 3.5373857123504D-28 SIGMAF SQUARED ESTIMATE, XS: 7.6899689398923D-30							
SIGMAF ESTIMATE: 2.7730793244861D-15 FIT QUALITY FACTOR: -3.275D+03							
NO. DEGREES OF FREEDOM: 46							
ORIGINAL PARAMETER NUMBERS PARAMETER ESTIMATES	^						
ESTIMATED STD DEU OF PARAMETERS							
ESTIMATED KELHTIDE SID DEV OF PAKAMETERS							
1 2 6 7 9 29							
4.0732D+08 2.0360D-03 9.6808D+00 8.3899D-09 1.0025D+00 8.7059D-08 2.1905D+07 5.5943D-05 2.6894D-05 1.3907D-10 8.1037D-05 1.7531D-00							
5.3779D-02 2.7477D-02 2.7780D-06 1.6576D-02 8.1733D-05 2.0137D-02							
PUHV= 1.9676D-02 PDRMS= 2.6856D-02							
ESTIMATED PARAMETER CORRELATION MATRIX 0.1000D+01							
0.19560+00 -0.32480+00 0.33300+00 0.10000+01							
-v.2y320+vv v.1005D+00 0.4253D+00 -0.3793D+00 0.1000D+01 -0.1885D+00 0.9465D-01 0.5396D+00 0.9402D-01 0.8236D+00 0.1000D+01							
AU ARR SD WR SD WI SD WC AU ART # TT							
5.769D-01 4.206D-15 1.920D-29 2.777D-15 1.000D+00 0							
AURR SDRR SDRI SDRC SSE # OPIT							
2.1150-01 8.660D-01 1.140D+00 9.441D-01 4.757D-01 0							
**************************************	×						
	Exit						

Fig. 3 PNTOUTL File obtained for the fit

	LEVMVIEW						
File Help							
Graphics Complex-plane	plot Numerica	I data PNTO	UTL AUXPNT				
- Evaluations			Correlation m	atrix of parame	ters		
Final L2 norm of the residuals	1.880	794e-014					
Number of function evaluations	;	32					
Number of jacobian evaluation	s	8					
Fit quality Weighted sum of squares, S Sigmal equared estimate, XS Sigmal estimate Fit quality factor No. degrees of freedom	3.537385 7.689968 2.773079	7123504e-028 3338923e-030 3244861e-015 3275 46	p[1] 1.0 p[2] -0.6 p[6] 0.4 p[7] 0.1 p[9] -0.2 p[29] -0.1	p11 p12 0000 -0.6374 0. 374 1.0000 -0. 223 -0.7871 1. 956 -0.3244 0. 9332 0.1005 0. 9355 0.0946 0.	p(6) p(7) 4223 0.1956 -0. 7871 -0.3248 0. 0000 0.3330 0. 3330 1.0000 -0. 4253 -0.3793 1. 5396 0.0940 0.	p[9] p[29] 2932 -0.1885 1005 0.0946 4253 0.5396 3793 0.0940 0000 0.8236 8236 1.0000	
Free parameters							
	p[1]	p[2]	p[6]	p[7]	p[9]	p[29	
Parameter estimates	4.073200e+008	2.036000e-003	9.680800e+000	8.389900e-009	1.002500e+000	8.705900e-008	
Belative std dev	2.190500e+007 5.377900e-002	2.004300e-005 2.747700e-002	2.689400e-005 2.778000e-006	1.350700e-010 1.657600e-002	8.193700e-005 8.173300e-005	1./53100e-005 2.013700e-002	
							Exit

Fig. 4 Numerical data output for the fit performed

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Figure 4 shows the correlation matrix of the parameters which have been set free to vary. It also consists of the values of weighted sum of squares, XS, FQF, standard deviation of free parameters etc.



Fig.5a) Impedance vs. frequency curve of NVLBB3 at 100°C; b) Residuals vs. frequency curve for NVLBB3 at 100°C

Figure 5 shows direct graphical output obtained from LEVMW CNLS fitting of the material used at temperature 100°C. The left two plots show the input data as dots and fitting result as solid lines. The right two plots show the real part residuals and relative residuals by red lines and imaginary part by blue lines. These are simply plotted by using the residuals yielded in the output file AUXPNTL.



Fig. 6 Equivalent Circuit Model for NVLBB3 at 100°C using circuit E

Figure 6 shows the equivalent circuit model for NVLBB3 for the given data at 100°C. It consists of two distributed elements in parallel to a capacitance. Each DE has five associated parameters which determine the element's characteristic. These are RDE, TDE, UDE, PDE and NDE (NELEM parameter). This equivalent circuit model has been designed on the basis of 40 parameters values obtained in the final run of the good fit obtained. Here, C_P has a value of 0.87958428D-07 F and in DE₁, RDE equals to 0.44628958D+09, TDE (here capacitance) has a value of 0.20499393D-02, UDE is zero, PDE is 1 and NELEM is 2 (so that the DE be simply a combination of resistor and capacitor in parallel). For DE₂, the parameter RDE is 0.96808304D+01, TDE is 0.76168335D-08, UDE is zero, PDE is 1.0026896 and NELEM is 2 again.

V. CONCLUSION

This paper deals with the designing of an equivalent circuit model for a glass NVLBB3 whose impedance spectroscopic measurements are given at various frequencies and temperature values. The simulation tool used for this purpose i.e. LEVMW consists of fifteen general circuits and various circuit models. Out of these, circuit E has been chosen in the

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input file and thus based on the general circuit E elements, the model parameters in the input file are guessed again and again by using the standard deviation values of all the free parameters obtained in each run till a good fit is achieved. The parameters with a value zero have been omitted from the general circuit and all the non-zero parameters then decide the final equivalent circuit model.

VI. FUTURE SCOPE

Future scope of this research work is to implement the obtained equivalent circuit model in hardware so that it can be used in various practical applications of glasses with vanadium content such as solid state batteries, memory and switching devices etc.

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