CHAPTER – 4
DESIGN, FABRICATION AND ASSEMBLY
OF AN EXPERIMENTAL SET-UP FOR
DIELECTRIC MEASUREMENTS AND
ANALYSIS.
4.1. Introduction

Dielectric, ferroelectric, electromechanical, piezoelectric, conductivity etc., of the ferroelectric insulators are important characteristics of such materials for their utility in industrial and other transducer applications. Ferroelectric oxides with general formula $\text{ABO}_3$ (where A is mono or divalent and B is trivalent --hexavalent) are some front runners in the area of research as well as in industrial applications. Materials of this type are potential candidates for use in transducer applications [1-9]. In order to develop materials of technological importance we need to have instruments for pursuing research on such materials. With the ever increasing use of computers in scientific research, it is a must to automate experimental set up with an objective of improving the accuracy and analysis of measurements. This may not be always available with the finished products available in market. One has, therefore, to make efforts in the augmentation of what is readily available so as to indigenously design and fabricate an experimental set up facilitating accuracy and speed in the measurements and analysis of results. To meet the requirement of research in dielectric and ferroelectric materials, it was considered very important to have an automated set up for accurate measurements and analysis of these characteristics with least dependence on manual control. In this chapter experimental set up designed,
fabricated and assembled so as to facilitate the control on heating of sample and measurement / analysis of its dielectric and ferroelectric characteristics under the variation of electric field and temperature are described and discussed. Many instruments have either been developed indigenously which are very cost effective compared to their imported versions or designed and fabricated locally in the laboratory with the purpose of creating a facility, consequently leading to training of scientific personnel in the field of instrumentation, besides scientific research. To quote a few examples Venkataraghavan et al [10] have designed and fabricated a versatile liquid phase epitaxy system. Verma et al [11] designed and developed and fabricated successfully X-ray Diffraction Topography system at NPL, New Delhi. A complete system with traversing mechanism and advanced scintillation counter and versatile turntable was established soon thereafter [12]. An inexpensive and simple atmospheric pressure chemical vapour deposition (APCVD) reactor for preparing large area transparent conducting tin oxide thin films has been designed and fabricated by Centre Electrochemical Research Institute, Karaikudi [13]. Many other systems / equipments have been developed indigenously by Indian scientists e.g., Eddy current transducer systems to locate garter spring spacers in highly radioactive coolant channels of Indian PHWR type nuclear reactors by BARC Mumbai, Vibrating sample magnetometer for magnetic multiple moment detection by Dr.R.K.Krishan, National Science Center for Biological Sciences etc [14].

Hewlett Packard impedance analyser (HP4192A) is a microprocessor based impedance measuring instrument. It can measure wide range of electrical parameters viz., impedance, reactance, capacitance, inductance, resistance, dielectric loss, quality factor as well as gain, phase and group delay. The measurements can be made in a wide range of frequency (5Hz to 13MHz) under the application of DC bias of -35 to +35V with high degree of precision. All the measurements are to be recorded and analyzed manually at
room temperature only. This makes the whole process of measurements and analysis difficult and time consuming. Moreover, effect of temperature on electrical properties is an important aspect for its application. In this direction an effort has been made to fabricate a setup in which data on all electrical properties (impedance, conductivity, dielectric etc.) of a material is directly recorded and analysed with the help of a computer. The effect of temperature and frequency of the applied a.c. field on such parameters is monitored, recorded and analysed with the help of this set up.

An effort has been made to indigenously fabricate and develop a setup in which almost all electrical properties of materials at elevated temperatures and varying frequency of the applied a.c. electrical field can be studied and the data recorded and analyzed directly with the help of a computer.

4.2. Configuration of the system

The configuration of the set-up for dielectric and other electrical measurement and analysis is shown in figure 4.1. The main unit components of the system are as under:
1) Impedance analyser (HP 4192)
2) Furnace and temperature controller.
3) Sample holder
4) Software and PC

4.2.1 Impedance analyser

Impedance analyser can measure eleven impedance parameters, including impedance (|Z|), Admittance (|Y|), phase angle (θ), resistance (R), reactance (X), conductance (G), susceptance (B), inductance (L), capacitance (C), dielectric loss (D) and quality factor (Q) over a wide range with high resolution --|Z| 0.1mΩ -1MΩ, |Y| 1nS -10S, C 0.1fF- 100mF , L 0.01nH – 1kH. Apart from these impedance parameters, it can measure four
transmission parameters viz., gain/loss, level, phase and group delay. The built-in frequency synthesizer can be set to individual frequencies or swept within the range from 5.000Hz. to 13.000000MHz. This allows stable measurement of high Q devices such as crystals. Test signal level is variable from 5mV to 1.1V (rms) with a resolution of 1mV up to 100mV and 5mV for levels higher than 100mV. It is provided with a dc internal source by which a dc bias of −35 to +35V can be applied at 10mV increments. The equipment is provided with two 4½ LED displays to display the measured values and third display to show the frequency of measurement.

In order to operate the system directly with help of PC for data recording and analysis we need to have a link between the PC and the impedance analyser. This impedance analyser (4192A) has the option for PC interface through Hewlett Packard Interface Bus (HP IB). Hewlett Packard Interface Bus (HP IB) is Hewlett Packard’s implementation of IEEE-488 communication interface. To interface the impedance analyser we made use of personal computer interface card (HP 82341D). This card supports HP!B parallel interface with data speed of 750kB/sec. The card is configured with the system and desired address for system chosen. A HP IB compatible cable of 2 meters (HP 10833B) has been used for interface between PC and the impedance analyser.

4.2.2 Furnace and temperature controller.

A muffle furnace with a cavity size 10X10 cms, 20 cms in depth and power consumption of 2kW at 220V was designed and fabricated. The design of the furnace is illustrated in figure 4.2. To ensure good thermal insulation between the cavity and external walls of the furnace, enough space is provided for insertion of insulating material between the cavity and the external walls as shown in figure 4.2. A circular hole of 3 cms is provided in the top lid of the furnace for the sample holder (described in the following section) so that the latter can be inserted in the furnace with ease. The
dimensions of the holder are chosen so that its water jacket (to ensure circulation of water within the sample holder for safety of leads against the rising temperature of the furnace) rests on the lid while the pedestal of the holder (where the sample is kept) is just above the bottom of the furnace cavity. To make measurements at a specified temperature in the furnace with large thermal mass, we need to have a system which can continuously control and monitor the temperature accurately. This was realized by using a universal digital temperature and controller (UDC-1000) and power controller. Universal digital controller (UDC-1000) is a compact and microprocessor based PID (proportional integral differential) controller incorporating the latest surface mount technology. The temperature is sensed by using a 'K' type thermocouple connected to the controller through proper compensating wires. The temperature at which measurements have to be made and the rate of rise of temperature (1°C/hour to 9999 °C/hour) can be programmed by universal digital controller (UDC). The controller senses the temperature and the rate of rise of temperature and compares it with the programmed values and accordingly generates an out signal (4–20 mA) that is fed to the power controller which controls the power supplied to the furnace. The power controller and the furnace are connected in series across the ac mains (230V). The power controller has a pair of thyristors connected in anti-parallel (back-to-back) connection. The power supplied to the furnace is controlled by controlling the ON/OFF duration (duty cycle) of the thyristors. The temperature controller (UDC-1000) is provided with RS485 serial communication (ASCII or MODBUS protocol) port. This port has been used to interface the controller with a computer by using RS485 interface card, which facilitates direct programming and reading of temperature and other parameters of the temperature controller directly with the help of PC. The furnace designed and used by us has a maximum working temperature of 800 °C.
4.2.3 Sample holder

Fig 4.3 is a schematic diagram of the indigenously designed and fabricated sample holder. The sample under study is placed between the upper and lower electrodes of the sample holder through a window shown in the figure. The upper electrode is connected in series with a spring by which pressure is mounted on the sample to keep contacts firm and prevent the displacement of the sample during handling. The electrical contact with sample holder is made through BNC connector. The sample holder is placed in the furnace for measurements at temperatures above the room temperature. Since the sample holder gets heated at high temperatures, it can result in damaging of insulation of the chord connecting BNC connector. To prevent it, one has to devise means of removing heat of the sample holder ensuring better safety of the leads against rising temperature. A steel jacket is provided around the sample holder (with water inlet and outlet as shown in the figure) on its top end towards BNC connector. Water is circulated through it while working at high temperatures which prevents the damage of the connecting lead due to overheating. Fig. 4.4 shows the actual sample holder designed and fabricated as per the above description. Fig. 4.5 shows the whole experimental set-up known as "Automated Impedance Analyser" designed and assembled according to the above description.

4.2.4 Software

Software for the overall control of the system and recording data directly with help of PC has been developed using Hewlett Packard Visual Engineering Environment version 4.0 (HP VEE 4.0). HP VEE is visual programming language optimized for building test and measurement application, especially programs with operator interface. With the HP VEE, programs are created connecting icons together using mouse; with textual languages one has to use keywords following rules of syntax. The result in HP VEE program resembles a flow diagram which is easier to use, understand and modify than traditional lines of code. The software developed
as Windows 95 based. Figure 4.6(a) and 4.6(b) respectively show the main screen and the one of the sub screens of the software developed.

The complete system has been working successfully ever since its fabrication and assemblage and many materials have been analysed using this system. Most of the results have been published in scientific journals [15]. Some important data collected and analyzed through the automated dielectric measuring and analysis system as assembled in the laboratory is described and discussed here in the subsequent sections as examples of its potential in accurate measurements and analysis of dielectric parameters. The whole assembly as installed in the laboratory is shown in figure 4.5. The major components of the assembly are indicated in the figure. Fig 4.4 is a picture of the two terminal sample holder designed on the basis of what is schematically shown in figure 4.3.

4.3 Analysis of the data on dielectric characteristics

The successful operation of the experimental set-up has been put to test and some significant results obtained on measurement and analysis are described here as examples though these examples are not connected with the actual topic of this thesis. However, it is considered important to reproduce some of the results obtained on this set-up as examples of its successful operation.

Fig.4.7(a,b,c) shows variation of dielectric constant, tangent loss and conductivity of praseodymium heptamolybdate with temperature under the varying frequency of the applied a.c. field. The data was recorded on a pallet prepared by powdering the crystalline material followed by pressing in to a disc in a hydraulic press under a load of 10 tons and using silver paste as electrodes. The transition temperature turns out to be 150 °C. The results obtained on praseodymium heptamolybdate as presented here have been by using the automated experimental set-up under discussion here [16]. Similar variations have been observed in other crystalline materials like Gd-
Ba molybdates bearing formula Gd$_2$Ba$_{20}$Mo$_{42}$O$_{153}$; the transition temperature turns out to be 128°C (the detailed results are discussed elsewhere [17]). The results obtained in melt grown KMgF$_3$ crystals using Czochralski pulling technique are compiled in the graphical analysis shown in figure 4.8. The transition temperature of KMgF$_3$ turns out to be 787°C. The detailed results as obtained on this material and their analysis will be published elsewhere.

The dielectric properties of samarium modified lead titanate ceramics bearing composition Pb$_{1-x}$Sm$_x$Ti$_{1.2}$W$_y$Fe$_{1-y}$O$_3$ with $x=0.02$, 0.05, 0.10, 0.20 any $y=0.02$ prepared by high temperature solid state reaction method were investigated using the automated setup. The results of dielectric constant Vs temperature at different frequencies of the applied a.c. field ranging from 1kHz to 1MHz for the composition containing 10 mol% samarium ($x=0.10$) is depicted in figure 4.9. It is observed that transition temperature is independent of frequency of the applied a.c. field. That the addition of samarium drastically changes the transition temperature of lead titanate ceramics, is clearly reflected by the results shown in figure 4.10. Dependence of dielectric loss on temperature at different frequencies of the applied a.c. field for composition containing 10 mol% samarium modified lead titanate ceramics as obtained by the set-up is illustrated in figure 4.11. The complete data obtained on these samarium modified lead titanate ceramics is analyzed and discussed in chapter-9 of this thesis.

The above said results are presented here with the aim of providing examples of the success of the automated experimental set up in the measurement and analysis of dielectric / ferroelectric characteristics of electronic materials. Baring the results on modified lead titanate ceramics, all the above results on heptamolybdates and KMgF$_3$ are presented here through personal communication and consent from my other co-workers of the Crystal Growth & Materials Research Group, Dept. of Physics and Electronics, University of Jammu.
4.4 Conclusion

Experimental set-up consisting of impedance analyser, furnace, temperature controller, a two terminal sample holder with a water jacket, a personal computer and a suitable software as designed, fabricated and assembled during the present investigations provides a convenient, fast and accurate measuring and analyzing system to investigate the dielectric and ferroelectric characteristics of materials.
References

2. T. Takahashi, Ceram Bull 69 (1990) 691
Fig. 4.1 Block diagram of the automated set-up for measurement of dielectric and other parameters of materials.
Fig. 4.2 Design of muffle furnace.
Fig. 4.3 Design of the two terminal sample holder used for holding the material to be analysed.
Fig. 4.6 (a) Main screen of the software (b) One of the sub screens of the software for parameter selection of the Impedance Analyzer
Fig.4.7(a) Variation of dielectric constant with temperature in praseodymium heptamolybdates (Courtesy Dr. S.Pandita).
Fig. 4.7(b) Variation of dielectric loss with temperature in praseodymium heptamolybdates.
Fig. 4.7(c) Variation of a.c conductivity with temperature in praseodymium heptamolybdates
Fig. 4.8 Variation of dielectric constant with temperature in KMgF3
(Courtesy B.L. Gupta)
Fig. 4.9 Variation of dielectric constant with temperature in 10 mol% samarium modified lead titanate.
Fig. 4.10 Variation of dielectric constant with temperature in samarium modified lead titanate at 1kHz.
Fig. 4.11 Variation of dielectric loss with temperature in 10 mol% samarium modified lead titanate.