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Sample Holder for the Measurement of AC conductivity of Solid Electrolyte

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ABSTRACT

The development alternative energy source is a race among the researchers from last five decades. Even today the scientist and labs are trying to find the competitive materials which will have high reliability in energy source like Solid state battery. The materials used in solid state battery called as solid electrolytes. The high ionic conductivity is the dior and prior need of this solid electrolyte. The measurement of ionic conductivity is always a challenging for the researcher, where a good sample holder satisfying the requirement of the AC conductivity measurement is the need. The effect of pressure, frequency and temperature are the common parameters of measured to understand the properties of the solid electrolyte .In the present paper the design of sample holder and various properties related to sample holder are discussed.

Keywords : AC Conductivity, Alternative Energy Source, Solid Electrolytes

I. INTRODUCTION

The sample holder (known also as flat specimen holder) is used for holding flat, circular or square/rectangular samples. It enables contacting the sample from the front and from the back The large electrical contact with copper foil surface (not being in contact with an electrolyte) ensures optimal current collection by minimizing electrical resistance and unifying current distribution. The sample holder elements are constructed with materials that are inert to the electrochemical system. It well fits aqueous and organic solvent electrolyte requirements. The construction is well sealed which prevents the electrolyte to leak inside. It can be used wherever it is necessary to make an electrode using a thin layer of an active material deposited on a flat substrate. The effect of pressure, frequency and temperature are the common parameters of measured to understand the properties of the solid electrolyte [1-4]. In the present paper the design of sample holder and various properties related to sample holder are discussed.

II. Sample holder Assembly

Sample holder is mainly design to measure AC conductivity of the solid electrolyte materials synthesized in the laboratory. The prepared samples are either in the form of pellets or quenched one, used directly. The materials used for prepared sample holders are ceramic 12 holes pipe, alumina pipe, spring, Teflon pipe, silver electrodes, antimagnetic steel rod etc. The dimensions of the parts used for the sample holder given in the following table1.

1	**Ceramic ring**	Diameter	4.9 cm
		Thickness	1.6cm
2	**Ceramic Holder**	Length	7.7 cm
		Diameter	5.1 cm
3	**Syndhanium plate**	Length	14.5 cm
		Breath	13.4 cm
		Thickness	0.9 cm
		Diagonal	19.4 cm
4	**Silver electrode**	Diameter	1.5 cm
		Length of Ag wire	40 cm

Table 1. Dimensions of Sample Holder

The assembling stages of are shown in the figure 1 given below.

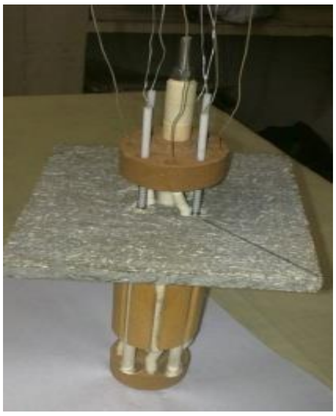

Fig1. Sample Holder

III. Parameter of the Sample holder considered and measurements carried out before using it for measurement

3.1 Contact of electrodes and its Ohmic behavior:

The pair of silver electrodes used for the measurement of electrical conductivity. The method of measurement used is two probes method where the sample is connected to a voltage source either AC or DC and the corresponding current through it is measured. Using the electronic gadgets the V-I measurement is converted into suitable parameters like R-X, C-tanδ, ε'-ε" etc. The measurement of above parameters is possible using impedance analyzer like HP4192A, Agilent 4292A in the frequency range varying from Hz to MHz While doing such measurement the most important parameter to be taken care is the Ohmic contacts. The Ohmic contacts are always confirmed by taking the V-I characteristics of the sample holder. The DC and AC (at frequency 10KHz and 100KHz) V-I measurement carried out for the sample holder over the voltage range varying from 1mV to 2 Volt found to be linear and passing through the origin as shown in the figure 2(A,B). This ensures the Ohmic contact between sample and electrodes.

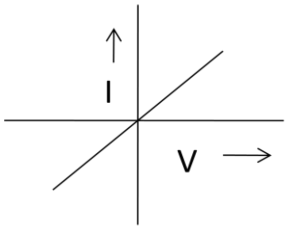

Fig 2A (DC)

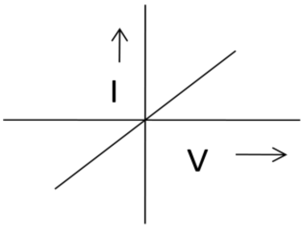

Fig 2B (AC)

Fig 2: V-I characteristics of the sample holder

When the V-I characteristics is not a straight line (as shown above) then contact is not Ohmic. To ensure the Ohmic contact different conducting paints are used depending upon the composition of the sample. Using different conducting paints for various sample compositions is given in the following table 2[5].

Table 2 : The use of conducting paint for different solid electrolytes

	Nature of Solid electrolyte	
Type of Electrolyte	**Crystal line**	**Amorp hous**
Silver (Ag) Based	Silver (Ag) / Graphite (Gr)	Silver (Ag) / Graphite (Gr)
Lithium (Li) Based	Silver(Ag) /Aluminum (Al) / Graphite (Gr)	Silver(Ag) /Aluminum (Al) / Graphite (Gr)
Sodium (Na) Based	Silver(Ag) /Aluminum (Al) / Graphite (Gr)	Silver(Ag) /Aluminum (Al) / Graphite (Gr)
Potassium (K) Based	Silver(Ag) /Aluminum (Al) / Graphite (Gr)	Silver(Ag) /Aluminum (Al) / Graphite (Gr)
OH^- Based	Graphite(Gr)	Graphite(Gr)
Protonic Conductor	Aluminum (Al) /Graphite (Gr)	Aluminum (Al) /Graphite (Gr)

The testing the sample holder for Ohmic contact is needed for the reason of usage of blocking electrodes [5]. The blocking electrodes (silver electrodes) mean which do not have the common ions as that of the solid electrolyte used for the measurement of the conductivity. The best contacts can be established only after making the contact of non-blocking electrodes. The use of non-blocking electrodes is not possible every time, like lithium based solid electrolytes require lithium as electrodes. Handling the lithium has many constraints as it needs spec pure inert atmosphere and sample holder of different design for holding the sample,

3.1 Impedance and capacitance measurement of sample holder with sample and without Sample.

The Impedance measurement of glassy solid electrolyte is carried out using Agilent4292A impedance analyzer over the frequency range of 20Hz to 2MHz. The Impedance plot for the same is shown in figure 3.

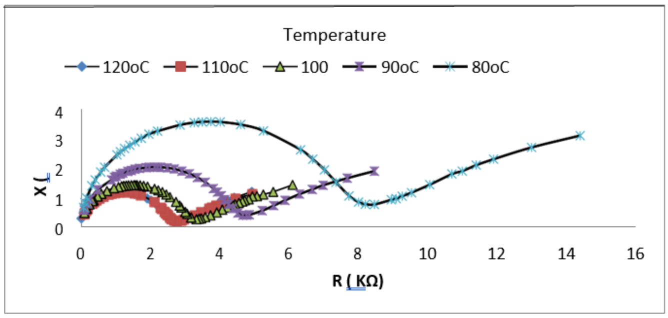

Fig 3 : Complex impedance lot of amorphous potassium doped lithium Borate Solid electrolyte at different temperature.

The impedance plot reveals the characteristics of sample and electrode-sample contact. The amorphous solid electrolyte does not contribute to grain boundary therefore one can get only the contribution of sample electrode-sample contact. Many researchers has developed the conductivity modelsRef for amorphous solid electrolyte and given the possible equivalent circuit for it.

The AC equivalent circuit [6] for solid electrolyte (amorphous) is shown in the figure 4.

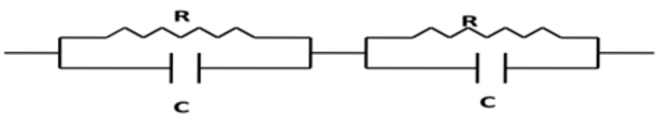

Fig 4 : R-C Equivalent Circuit

The equivalent circuit comprises the effect of solid electrolyte sample, contact between sample and electrodes during the impedance measurement.

What is important in this is the net contribution of electrodes to impedance and it should reflect uniformity in its value over temperature range of measurement. This can be understood in the following manner.

The semicircular part of the impedance plot in figure 3 is for sample under measurement; the increase in the value of reactance at the peak maxima and at the onset of the impedance plot with decrease in temperature has been extensively studied and explained by many authors Ref. This is the reactance of the sample arising due capacitive effect as seen from equivalent circuit. The linear nature of the impedance plot is due to the sample-electrode contact impedance. Moreover the linearity in the graph remains unchanged with temperature this reflects the uniform contribution of impedance arising due to sample –electrode contact.

This linear nature is then further analyzed to understand the net contribution of resistance and capacitance of sample holder electrodes to the contact. The equivalent circuit shown above is the parallel combination of R and C, the resultant reactance of the circuit is given by equation

$$Requ = \frac{R\ Xc}{R + Xc}$$

The Requ decreases with increase in frequency as shown in figure 5. The range of the Requ varies from 2.758KΩ to 0.4389KΩ over the frequency. When the contribution of the Xc is isolated from it, it is seen that it decreases with frequency as shown in figure 6. This behavior goes well with that observed for parallel plate capacitor with air as dielectric.

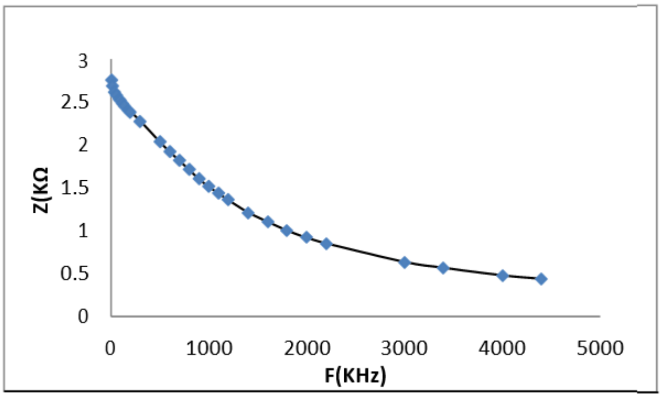

Fig : 5

The R and X, C and tanδ are measured as a function of frequency and temperature without sample (keeping the separation of electrodes same as that of sample thickness) has carried out. The variation in R-X with frequency and temperature is not reflected in the measurement. Therefore it has been cleared that the contribution of electrodes alone to the impedance is negligible. Whereas the C – tanδ measurements as a function of frequency shows similar nature as that is observed for air. This reflects that the electrodes contribution towards R-X and C can be ignored during the AC measurements of the sample. Therefore the net effects of the electrodes of the sample holder do not contribute to the conductivity measurement of the solid electrolyte sample under study.

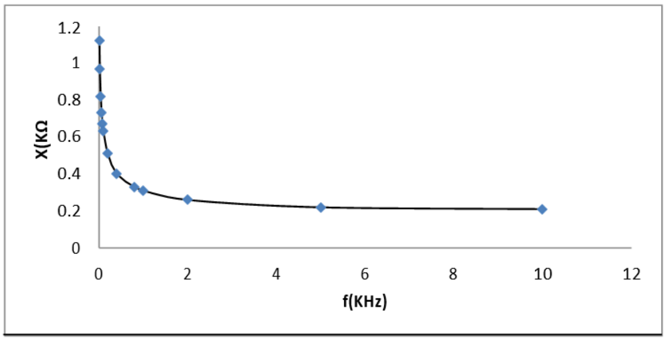

Fig 6

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