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Preperation of Materia and Their Chemical, Physical and Electrical Analysis for HEV

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Abstract— Electrical and electrochemical properties of PEO based hot pressed nanocomposite polymer electrolyte (NCPE) membrane (1-x)[70PEO:30AgNO3]:xTiO_2 where x=0-20 wt% , casted by using a novel hot pressed technique for prperation of electrochemical devices viz. supercapacitor, battery etc. have been studied. Solid Polymer Electrolyte (SPE) composition (70PEO:30AgNO_3) reported earlier identified as highest conducting film at room temperature, has been used as 1st phase host matrix and nano-size (~8nm) filler TiO_2 as 2nd phase dispersion. As a consequence of dispersal in SPE host, highest conductivity was achieved in at 5wt% of TiO_2. This composition has been referred as optimum composing composition (OCC). Addition of KOH is OCC shows further increase in conductivity. The ion transport behavior in NCPE membrane have been discussed on the basis of ionic conductivity(σ), ionic transferred number(t_{ion}) and activation energy E_a. Morphology study and compositional veriation also performs by SEM .

Keywords— Hot pressed polymer electrolyte, ionic conductivity, Activation energy,Scanning electron microscopic(SEM),

I. INTRODUCTION

The ionic conducting Solid Polymer Electrolyte (SPEs) show tremendous technological power sources viz. flexible, compact, light-weight, leak-proof, thin film micro-batteries of desirable shape and size.[1-4]. The conduction mechanism in polymer electrolyte was reported first time in 1973[5], while the first practical SPE battery based on 'Poly(ethylene oxide) (PEO)-Li^+ -ion salt complex' was demonstrated in 1979[6]. Since then, a wide variety of SPEs, involving different kinds of mobile ions viz. Li^+ , H^+ , Na^+ , K^+, Ag^+ etc. as principle charge carriers, has been reported. PEO-based polymer electrolyte is one of the most promising materials due to their good mechanical and electrochemical properties. It has been observed in general that the existence of high degree of amorphousity in polymer host(viz.PEO) supports high ionic mobility and hence, high ionic conductivity in SPEs. However, the SPE films prepared conventionally be completing the salts in polymeric host, are found to be less stable mechanically. In recent investigation, it has been observed that mechanical strength of polymer electrolyte membranes as well as the degree of amorphousity in polymer host could be enhanced substantially by dispersing nano-size particles of an inert/insulating material such TiO_2, Al_2O_3, TiO_2 etc.[7-14]. The present paper report some electrictrical viz. ionic conductivity(σ), activation energy (E_a) ,ionic transference number(t_{ion}), and electrochemical properties of hot-press synthesized new Ag^+ ion conducting nano-composite polymer electrolyte(NCPE) membrane (1-x)[70PEO:30AgNO3]:xTiO_2 where x=0-20 wt% using different experimental technique.

II. METHODOLOGY

In present studies the impedance measurement technique has been employed to evaluate the true bulk conductivity and activation energy of polymer electrolyte film as a function of salt and filler concentration. The highest conducting composition 70PEO:30AgNO_3[15] taken as base system to study the influence of TiO2 addition on it . The general formula for SPE system is (1-x)[70PEO:30AgNO_3]:xTiO_2 where 1≤x≤ 20 wt.(%), AR grade chemicals PEO (10^5 MW, Aldrich, USA, purity 98%), AgNO3(purity 98%) TiO_2 (≥99.8%,size ~8nm),and KOH were used for the synthesis of hot-pressed NSPEs. Dry powder of constituent chemicals in appropriate wt(%) ratio were homogeneously mixed for 10 min. at room temperature then heated separately about 110^{0}C for 20 min. at melting point of PEO and to remove moisture. It result into a soft lump/slurry which was then pressed (~ 2 ton/cm^2) between two SS-cold block giving rise to a uniform film. The polymer electrolyte film so obtain sandwiched between fine brass blocking electrode then it placed in a temperature regulated furnace. The impedance and corresponding phase angle θ were measure at different constant temperature for different composition using HIOKI 3532-50 LCR meter over frequency range 50Hz to 5MHz . The value of Bulk resistance R_b determine by Cole-Cole plot from which conductivity is calculated. It is found that

$[95(70PEO:30AgNO_3]5TiO_2$ is optimum conducting composition (OCC). KOH of different wt% added in OCC and conductivity calculated which shows conductivity enhancement at each wt%. The activation energy energy above melting point was calculated by the slop of Arrhenius plot., ionic transference number (t_{ion}) were determine by Weigenar polarization method. SEM analysis of material perform at IIT Kharagpur , and Pt.RSSU Raipur respectively.

III. RESULTS AND DISCUSSION

The Cole- Cole plot for different (1-x)[70PEO:30AgNO3]:$xTiO_2$ where $0 \le x \le 20$ and 95[70PEO:30AgNO3]:$5TiO_2$+xKOH where x = 10,20,30,40 wt% is shown in Figure 1 and Figure 2 respectively. Which shows variation of real and imAgNO3nary part of impedance. Blocking impedance R_b calculated by intercepting point of semicircle on real axis.

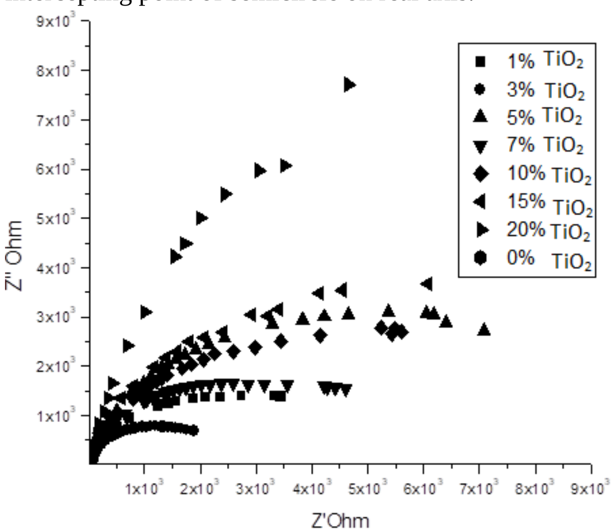


Figure1.Cole-cole plot (1-x)[70PEO:30AgNO3]:$xTiO_2$

By measuring the thickness t, area of sample A, and blocking impedance R_b form experimental Cole-Cole plot the conductivity values can be calculated from the equation,

$$\sigma = \frac{t}{R_b A} \qquad (1)$$

The filler concentration conductivity variation at room temperature for hot press nanocomposite polymer electrolyte membranes(1-x)[70PEO:30AgNO3]:xTiO2 is shown in figure 3.

The room temperature conductivity increases initially with increase in nano size filler concentration in SPE host($70PEO:30AgNO_3$). The conductivity (σ) maxima

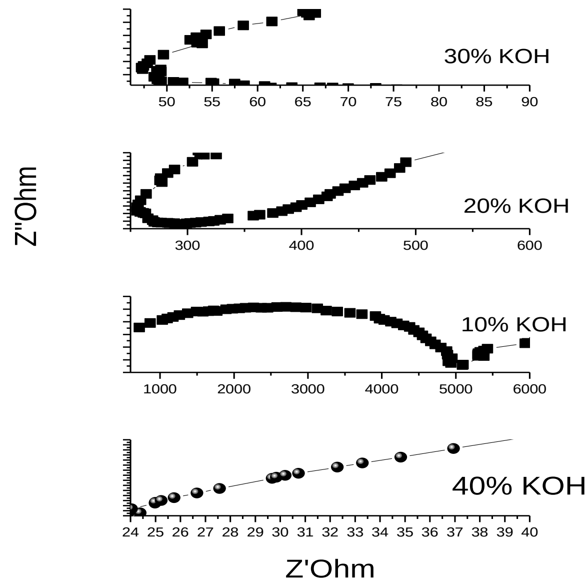


Figure 2. Cole-cole plot for 95[70PEO:30AgNO3]:$5TiO_2$ +xKOH

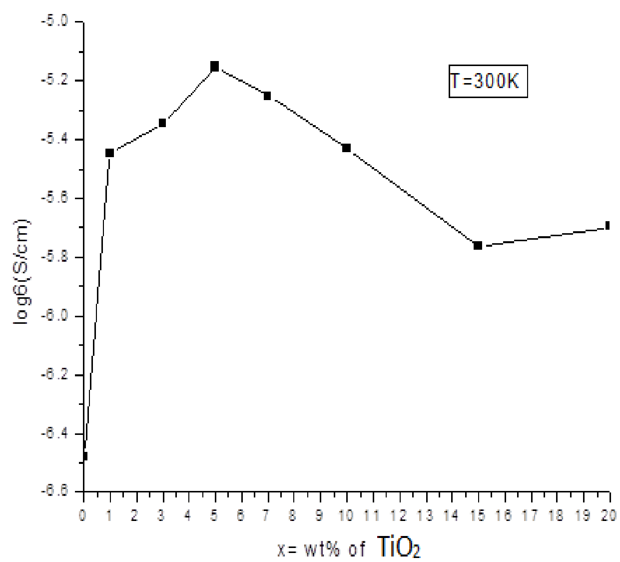


Figure 3. Variation of Conductivity with wt% of TiO_2

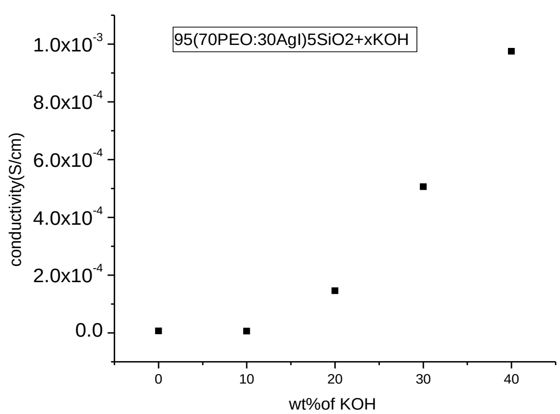


Figure 4. Variation of Conductivity with wt% KOH in OCC

The room temperature conductivity of OCC also increases with the increases in wt% of KOH. Which is shown in Figure 4, which shows that maximum conductivity ($1.4625x10^{-3}$ S/cm) is obtain at 40 wt% of KOH, further increases in percentage of KOH the electrolyte converted in gel form. Knowing the ionic conductivities at different temperature, for each composition, the Arrhenius plot can be fitted form which the activation energy calculated. The Arrhenius equation gives the quantitative basis of relationship between the activation energy E_a and the rate at which a reaction proceeds. The Arrhenius equation is

$$\sigma = \sigma^0 e^{\frac{-E_a}{KT}} \qquad (2)$$

The experimental Arrhenius plot with different wt(%) TiO2 in $70PEO:30AgNO_3$ is shown in Figure 5

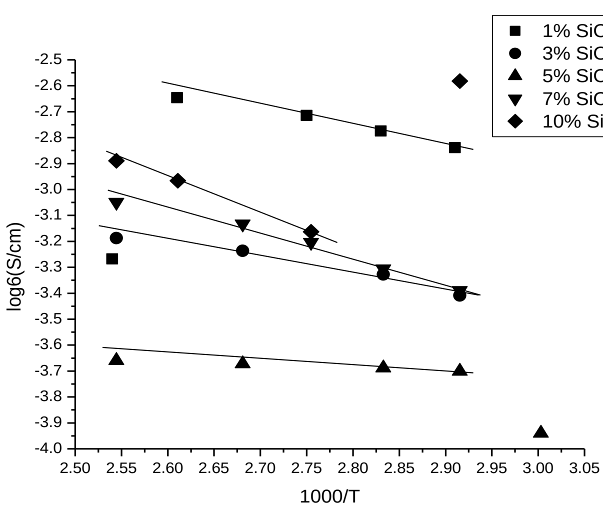


Figure 5. Arrhenius plot for wt% of TiO_2 in PEO

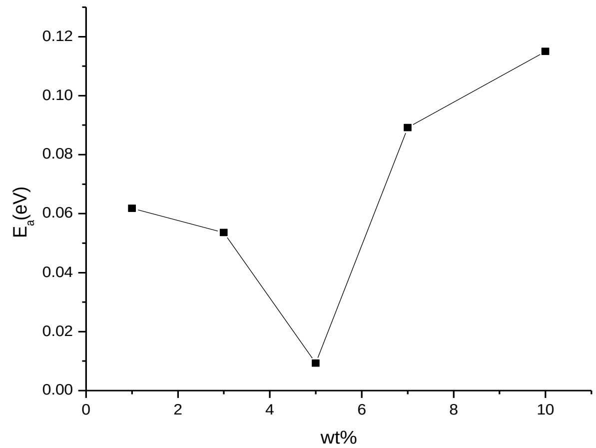


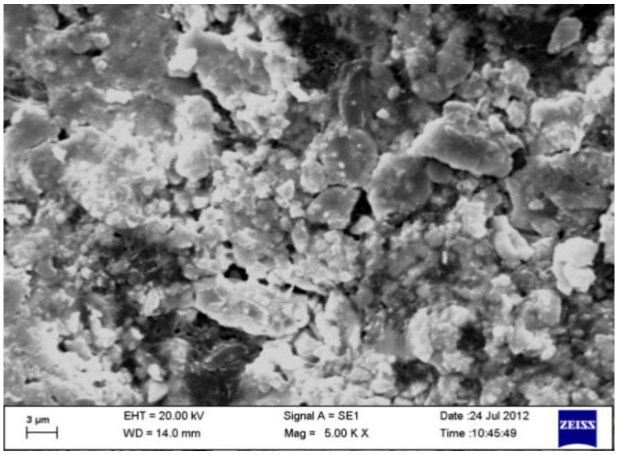
Figure 6. Activation energy vs wt% of TiO_2

The variation of activation energy above melting point with wt% of TiO2 is shown in Figure 6.The NCPE OCC were identified at 30 wt% of TiO2 in SPE corresponds to lowest activation energy calculated by the use of Aurreheneous plot which is 0.00932eV. The ionic transference number (t_{ion}) of different SPE were determine at room temperature by using Weginar dc polarization TIC technique[16-19]. The room temperature measurement values of all these ionic parameter σ, t_{ion}, and activation energy E_a are listed in Table 1

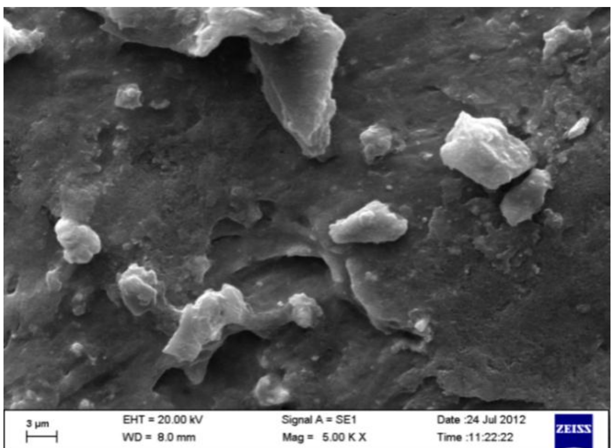
Table 1: Some important parameter of the hot-pressed SPE host and NCPE-OCC at room temperature

Film	σ (S/cm)	t_{ion}	E_a (eV)
$70PEO:30AgNO_3$	$3.3 x10^{-5}$	~95	0.069[15]
$95[70PEO:30AgNO_3]: 5TiO_2$	$7.032x10^{-4}$	~94	0.00932
$95[70PEO:30AgNO_3]: 5TiO_2$+40%KOH	$1.4625x10^{-3}$	~94	

The SEM examination of four samples showed greatly varying surface morphologies. Image of polymer electrolyte at $5x10^3$ magnification with salt and different wt% TiO_2 and KOH is shown in Figure 7(a). It shows smooth appearance is generally associated with the lowering of crystallinity in the presence of salts. The presence of TiO_2 particle in this case had therefore caused a lowerning of polymer crystallization, but to a limited extent. Further the more dramatic refinements of the surface morphology were made possible with the incorporation of more TiO2 [20,21]. Figure 7 (b) shows that the addition of KOH smoothen the surface of electrolyte which provide path for the ion transport due to which mobility and hence conductivity increases.



(a) $70PEO{:}30AgNO_3$



(b) $95[70PEO{:}30AgNO_3]5TiO2$ +40% KOH

Figure 7. SEM analysis of prepared SPE

IV. Conclusion and Future Scope

A new solid polymer electrolyte membrane (1-x)[70PEO:30AgNO3]:xTiO2 with different wt% of TiO2 has been investigated for the purpose of fabricating solid state thin film electrochemical devices viz. supercapacitor, batteries. A novel hot-press technique has been used for the film casting. The maximum conductivity of NCPE-OCC obtain at 5% TiO2. With the increase of KOH in NCPE-OCC conductivity increases. The SEM analysis of different polymer electrolyte shows the conductivity enhancement is due to smoothness of surface. The ion transport mechanism of NCPE-OCC has been studied in terms of basic ionic parameters σ, E_a, t_{ion}. The very low activation energy of NCPE-OCC shows very good conducting property of electrolyte, the value of t_{ion} close to unity shows that the electrolyte is pure ionic. The conductivity was found to increase with increase at a limited extent of dopant concentration of salt and filler with temperature in pure PEO. Lowest activitation energy confirm optimum conductivity of material. Ionic transfer number close to unity shows that material is pure ionic rather than electronics.

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Authors Profile

Mr Satpreet Singh Gill pursuing M.Tech. in power electrinics form Kalinga University under the guidence of Proff. Manish Kurrey.
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Mr. Manish Kurre, presently Assistant Professor in Kalinga University Raipur. His work in the field of power engineering are highly appereaciable. He has guided many M. Tech. students at the level of research in the field of power engineering. Mr. Satpreet Singh Gill is one of the students working unider him..