Investigations on ion transport properties of and battery discharge characteristic studies on hot-pressed Ag\textsuperscript{+}-ion-conducting nano-composite polymer electrolytes: \((1-x)\left[90\text{PEO} : 10\text{AgNO}_3\right] : x\text{SiO}_2\)

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**Abstract.** Investigations on the ion transport properties of Ag\textsuperscript{+}-ion-conducting nano-composite polymeric electrolytes (NCPEs): \((1-x)[90\text{PEO} : 10\text{AgNO}_3] : x\text{SiO}_2\), where \(x = 0, 1, 2, 3, 5, 10\) or 15 (wt\%) and PEO is poly(ethylene oxide), are reported here. Films of NCPEs were cast using a novel hot-press/solvent-free/dry technique instead of the usual solution cast method. Nano-size (~8 nm) SiO\textsubscript{2} particles, as the second dispersoid phase, were dispersed into the first-phase conventional solid polymer electrolyte (SPE) host: \((90\text{PEO} : 10\text{AgNO}_3)\). The SPE host, also prepared by hot-pressing the homogeneously mixed composition: PEO : AgNO\textsubscript{3} :: 90 : 10 (wt\%), has been identified as the film with the highest room temperature conductivity \(\sigma \sim 4 \times 10^{-6}\) S cm\textsuperscript{-1}, exhibiting a \(\sigma\) increase of more than three orders of magnitude from that of the pure PEO. Dispersal of SiO\textsubscript{2} nano-particles in the SPE host resulted in a 2-fold conductivity enhancement in the NCPE film: 95(90PEO : 10AgNO\textsubscript{3}) : 5SiO\textsubscript{2} (wt\%). This has been referred to as the ‘optimum conducting composition (OCC)’. The ion transport behavior in NCPE has been characterized on the basis of experimental studies on some basic ionic parameters, viz. ionic conductivity \((\sigma)\), ionic mobility \((\mu)\), mobile ion concentration \((n)\), ionic transference number \((t_{\text{ion}})\), etc. The temperature-dependent measurements on \(\sigma\), \(\mu\) and \(n\) in the NCPE OCC film provided quantitative information on the energies involved in different

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thermally activated processes. A thin-film solid-state battery has been fabricated using the NCPE OCC membrane as the electrolyte to test the cell performance under a fixed load condition at temperatures below/above room temperature.

1. Introduction

Solid polymer electrolytes (SPEs) show tremendous technological promise to develop all-solid-state electrochemical power sources, viz. flexible/compact/light-weight/leak-proof thin film micro-batteries of desirable shapes/sizes [1]–[4]. Ion conduction in polymers was reported for the first time in 1973 [5], whereas the first practical SPE battery based on the ‘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 the principal charge carriers, have been reported. SPE films are prepared usually by the solution-cast method. The majority of SPEs reported so far are PEO-based Li$^+$-ion-conducting electrolytes. Practically, polymer electrolytes with room temperature conductivity $\gtrsim 10^{-4}$ S cm$^{-1}$ are considered good candidates for battery applications. It has been observed, in general, that the existence of a high degree of amorphousity in the polymer host (viz PEO) supports high ionic mobility and hence high ionic conduction in SPEs. However, the SPE films, prepared conventionally by complexing the salts in the polymeric host, are found to be relatively less stable mechanically. In a recent investigation, it has been observed that the mechanical strength of the polymer electrolyte membranes as well as the degree of amorphousity in the polymer host could be enhanced substantially by dispersing nano-size particles of an inert/insulating material such as Al$_2$O$_3$, SiO$_2$, TiO$_2$ [7]–[9]. Such systems are referred to as nano-composite polymer electrolytes (NCPEs). The size of the filler particles plays a significant role in improving these physical properties of SPEs [10, 11]. Very recently, in place of the solution-cast method, a novel hot-press (extrusion) technique has been developed for casting SPE membranes [12, 13]. This is a relatively inexpensive, more rapid, solvent-free procedure as compared to the traditional solution-cast method. The present paper reports the hot-press casting of new Ag$^+$-ion-conducting NCPE membranes: $(1-x)[90\text{PEO} : 10\text{AgNO}_3] : x\text{SiO}_2$. The SPE film composition $[90\text{PEO} : 10\text{AgNO}_3]$ was employed as the first-phase polymer electrolyte host for dispersal of nano particles of SiO$_2$ used as second dispersoid phase. In fact, the SPE host has been identified as one of the high conducting and relatively more stable films. Further, an NCPE film exhibiting optimum conducting composition (OCC) has been identified. The ion transport properties have been characterized on the basis of basic ionic parameters, viz. conductivity ($\sigma$),

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ionic mobility ($\mu$) and mobile ion concentration ($n$). To confirm salt complexation in the polymeric host, x-ray diffraction (XRD) analysis has been done. The ionic transference number ($t_{\text{ion}}$), a quantitative measure of ionic contribution to the total conductivity, has been evaluated by a dc polarization technique. The newly synthesized hot-pressed NCPE OCC membrane was used as the electrolyte, to fabricate a thin-film solid-state battery and to test the cell performance under a fixed load condition at different temperatures.

2. Experimental

The hot-press casting of Ag$^+$-ion-conducting NCPE membranes: $(1-x)[90\text{PEO} : 10\text{AgNO}_3] : x\text{SiO}_2$, where $x = 0, 1, 2, 3, 5, 10$ or $15$ (wt%), was done using AR-grade precursor chemicals: PEO ($10^5$ MW; Aldrich, USA), AgNO$_3$ (purity > 98%; Reidel, India), SiO$_2$ (>99.8%; Sigma, USA; particle size $\sim$8 nm). Firstly, films of the conventional SPE: (PEO : AgNO$_3$), in varying salt concentrations viz. 10, 20, 30, 40, 50, 60, 70 and 80 (wt%) were hot-press casted. Dry powders of PEO and AgNO$_3$ were mixed thoroughly at room temperature for $\sim$30 min, heated at $\sim$70$^\circ$C (close to the melting point of PEO) for about 15 min with mixing continued to form a homogeneous slurry and then hot-pressed ($\sim$1.25 tonne cm$^{-2}$) between two SS blocks. As a result, a thin blackish membrane of thickness $\sim$0.014 cm was obtained. The SPE film composition (90PEO : 10AgNO$_3$) was identified as the highest conducting composition from the salt-concentration-dependent conductivity study, as mentioned, and employed as the first-phase polymer electrolyte host for casting NCPE films. Dry powders of PEO : AgNO$_3$ in 90:10 (wt%) (as the first-phase) and nano-sized SiO$_2$ in different wt% ratios (as the second-phase dispersoid) were mixed homogeneously, heated at $\sim$70$^\circ$C and then hot-pressed, as above. The NCPE membrane exhibiting OCC was identified further from the SiO$_2$-concentration-dependent conductivity measurements. An LCR bridge (Hioki 3520-01, Japan) was used for conductivity measurements at a fixed frequency (i.e. 5 kHz). The salt-complexation and the presence of the second-phase dispersoid in the polymeric host were identified by XRD using Cu-K$_\alpha$ radiation (Shimadzu, model 6000). The ionic mobility ($\mu$) and ionic transference number ($t_{\text{ion}}$) in the NCPE OCC membrane were directly determined at room temperature using a dc polarization transient ionic current (TIC) technique [14, 15]. Subsequently, the mobile ion concentration ($n$) was evaluated from $\sigma$ and $\mu$ data. Temperature-dependent measurements on $\sigma$, $\mu$ and $n$ for the NCPE OCC film were also carried out and the various energies governing the ionic transport phenomenon in the system were computed from the linear least square fitting of the data obtained from the respective Arrhenius plots: ‘log $\sigma - 1/T$’, ‘log $\mu - 1/T$’ and ‘log $n - 1/T$’. Finally, using the NCPE OCC membrane as the electrolyte, thin-film solid-state batteries were fabricated in the following cell configuration:

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Ag-metal foil | 95[90PEO:10AgNO$_3$] + 5SiO$_2$ | (C + I$_2$ + Electrolyte) | (C + I$_2$ + Electrolyte) (Hot-pressed cathode film) |
(Anode) | (Hot-pressed NCPE OCC membrane) |
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The cathode in the film form was prepared by hot-pressing the homogeneous physical mixture of elemental iodine (I$_2$), the conducting graphite (C) and NCPE in 1 : 1 : 1 weight ratios at $\sim$70$^\circ$C. The cell performance was studied under 100 k$\Omega$ load conditions at three different temperatures: 15, 27 and 40 $^\circ$C. Some important cell parameters were calculated from the plateau region of the cell–potential discharge profiles.
3. Results and discussion

3.1. Ionic transport parameters and materials characterization studies

Figure 1 shows the room temperature conductivity ($\sigma$) as a function of salt concentration for the hot-pressed conventional SPE membranes: (PEO : AgNO$_3$). $\sigma$ increased abruptly ($>10^3$ times) as AgNO$_3$ concentration increased initially, then remained almost unaltered on further addition of the salt. A moderate-sized $\sigma$-maxima with room temperature conductivity ($\sigma \sim 4 \times 10^{-6}$ S cm$^{-1}$) was observed at 10 wt% of AgNO$_3$ (i.e. for the composition 90PEO : 10AgNO$_3$). Chandra et al [16] also investigated similar SPE films formed by the usual solution cast method and reported almost analogous salt-concentration-dependent conductivity variation with maximum conductivity ($\sigma \sim 4 \times 10^{-7}$ S cm$^{-1}$) for the SPE film of the same composition: (90PEO : 10AgNO$_3$). According to them, the addition of salt in PEO resulted in an increase in the degree of amorphousity, which in turn gave rise to an abrupt increase in conductivity [16]. Figure 2 shows SiO$_2$-concentration-dependent conductivity variation at room temperature for the hot-press Ag$^+$-ion-conducting NCPE membranes: $(1-x)[90$PEO : 10AgNO$_3$] : xSiO$_2$. As a consequence of dispersal of nano-size ($\sim 8$ nm) filler particles as the second-phase dispersoid into the first-phase polymeric electrolyte host: (90PEO : 10AgNO$_3$), the room temperature conductivity increased initially as the SiO$_2$ ratio increased, reached a peak value at 5 wt% and then decreased gradually. A conductivity variation of this kind has been invariably witnessed in the majority of two-phase inorganic composite electrolytes, and various models, based on space charge double layer effect [17, 18], have been proposed in order to explain the conductivity enhancements in these systems. The increasing/decreasing trend in conductivity with the increase in dispersoid concentration can be understood on the basis of the percolation model [19]. These models can also convincingly explain the mechanism of ion transport in composite polymeric...
Figure 2. Room temperature conductivity of hot-pressed NCPE membranes: \([(1 - x)(90\text{PEO}: 10\text{AgNO}_3): x\text{SiO}_2]\), as a function of SiO\(_2\) concentration (wt%).

(organic) electrolytes. However, due to the absence of exact structure–property correlations in the polymer electrolyte systems, a complete understanding of the ion conduction phenomenon is still lacking. Nonetheless, to explain the mechanistic aspects of ion transport in micro/nano-composite polymer electrolyte systems, a working hypothesis has been suggested. Accordingly, the dispersal of submicron-size filler particles, containing large surface area, into the SPE host creates a high degree of amorphousity and/or lowers the degree of crystallinity, which may also be thought to be due to Lewis-acid–base interaction between ceramic surface states and polymer segments [3, 9, 20, 21]. Hence, in addition to the usual space charge effects of the dispersoid particles, the increased amorphousity would also support the conductivity enhancement in terms of increased ionic mobility through the amorphous phase. As a consequence of the dispersal of nano-sized SiO\(_2\) particles, a 2-fold increase in the room temperature conductivity was achieved further in the NCPE membrane: 95[90\text{PEO} : 10\text{AgNO}_3] : 5\text{SiO}_2 with \(\sigma \sim 8.8 \times 10^{-6}\) S cm\(^{-1}\). This has been referred to as the OCC NCPE membrane, as mentioned. The decrease in the ionic conductivity for SiO\(_2\) concentrations higher than 5 wt% can be attributed to the usual blocking effect of the filler particles [19]. Physically, the NCPE OCC film appeared relatively more stable/flexible mechanically than the SPE-host film. Improvements in the physical properties in composite polymer electrolytes due to dispersal of filler particles have already been reported [7]–[9]. In order to identify the reason for conductivity enhancement in NCPE OCC further, ionic mobility (\(\mu\)) was determined at room temperature directly by the TIC technique and mobile ion concentration (\(n\)) was evaluated from \(\sigma\) and \(\mu\) data, as mentioned. In addition to this, from the ‘current versus time’ plot, obtained in the TIC experiment, the ionic transference number (\(t_{\text{ion}}\)) was also determined. For details of the experimental procedures used for the measurement of \(\mu\), \(n\) and \(t_{\text{ion}}\), reference may be made to the original papers [14, 15, 22]. Table 1 lists the room temperature values of \(\sigma\), \(\mu\), \(n\) and \(t_{\text{ion}}\) obtained for NCPE OCC as well as SPE host membranes. One can clearly note that the overall increase in \(\sigma\) of NCPE OCC is due to a
Table 1. Room temperature ($27^\circ$C) values of some important ion transport parameters for the hot-pressed SPE host: (90PEO : 10AgNO$_3$) and NCPE OCC : 95(90PEO : 10AgNO$_3$) : 5SiO$_2$.

| System                                      | $\sigma$ ($S\ cm^{-1}$) | $\mu$ (cm$^2\ V^{-1}\ s^{-1}$) | $n$ (cm$^{-3}$) | $t$          |
|---------------------------------------------|--------------------------|----------------------------------|-----------------|-------------|
| SPE host: (90PEO : 10AgNO$_3$)              | $4.03 \times 10^{-6}$    | $(3.6 \pm 1) \times 10^{-3}$    | $6.99 \times 10^{15}$ | $\sim 0.90$ |
| NCPE OCC: 95(90PEO : 10AgNO$_3$) : 5SiO$_2$ | $8.8 \times 10^{-6}$    | $(4.8 \pm 1) \times 10^{-3}$    | $1.12 \times 10^{16}$ | $\sim 0.90$ |

Figure 3. XRD patterns for film samples: (a) pure PEO, (b) SPE host: (90PEO : 10AgNO$_3$) and (c) NCPE OCC: [95(90PEO : 10AgNO$_3$) : 5SiO$_2$].

A moderate increase in $\mu$ (indicative of an increase in the degree of amorphousity in the polymeric electrolyte host) and an approximately one order of magnitude increase in $n$ (indicative of the usual space charge double layer effect and/or dissociation of more number of Ag$^+$ ions from the salt). The ionic transference number ($t_{\text{ion}} \sim 0.9$), obtained for both SPE and NCPE OCC membranes, indicated that the majority of Ag$^+$ ions do take part in the conduction process. In order to confirm the salt complexation in the polymeric electrolyte host, XRD analysis was done on the film samples of NCPE OCC, SPE host and pure PEO. XRD patterns for these membranes are shown in figure 3. On closer inspection, it can be noted that some of the peaks of pure PEO became relatively broader as well as less prominent/feeble after salt complexation/SiO$_2$ dispersal. This is usually attributed to the increase (decrease) in the degree of amorphosity (crystallinity) and hence, in turn, confirmed the salt complexation as well as, to some extent, the dispersal of nano-SiO$_2$ in the polymeric host.

However, the peaks for AgNO$_3$ and SiO$_2$ could not be distinctly seen in the XRD pattern, which may probably be due to their presence in a relatively low percentage in the system as compared to PEO and/or overlapping of some of their main peaks with those of PEO. Figure 4 shows the temperature-dependent conductivity variations for different NCPE membranes: $(1 - x)[90\text{PEO} : 10\text{AgNO}_3] : x\text{SiO}_2$ including the SPE host: (90PEO : 10AgNO$_3$). The equation governing the straight line portion of the Arrhenius plot
Figure 4. The ‘log $\sigma$–1/$T$’ plots for the SPE host: [90PEO : 10AgNO$_3$] (■); NCPE membranes: $(1 - x)$(90PEO : 10AgNO$_3$) : $x$SiO$_2$; $x = 1$ (●), 2 (△), 3 (□), 5 (○), 10 (+) or 15 (×). Top inset: variation of activation energy ($E_a$) as a function of $x$.

‘log $\sigma$–1/$T$’ for the NCPE OCC film can be expressed as:

$$\sigma(T) = 2.88 \times 10^9 \exp(-0.32/kT) \quad \text{(S cm}^{-1})$$

where $E_a = 0.32$ eV is the activation energy. The activation energy values for other NCPE membranes as a function of SiO$_2$ concentration, computed likewise from the respective ‘log $\sigma$–1/$T$’ plots, are plotted in the inset of figure 4. It can be clearly noted that the activation energy for NCPE OCC is not only minimum but has also been reduced substantially from that of the pure SPE host. This is indicative of a relatively easier ion migration in the NCPE OCC.

Figure 5 shows ‘log $\mu$–1/$T$’ and ‘log $n$–1/$T$’ plots for the hot-pressed NCPE OCC membrane: 95[90PEO : 10AgNO$_3$] : 5SiO$_2$. The Arrhenius equations governing these variations can be expressed by:

$$\mu(T) = 2.12 \times 10^{-11} \exp(+0.49/kT) \quad \text{(cm}^2\text{V}^{-1}\text{s}^{-1})$$

$$n(T) = 3.22 \times 10^{29} \exp(-0.79/kT) \quad \text{(cm}^{-3})$$

where the numerals 0.47 and 0.78 in eV are the energies involved in the two thermally activated processes and can be designated as the energy of migration ($E_m$) and the energy of formation ($E_f$), respectively. The (−)-ve or (+)-ve signs appearing in the argument of the exponential terms indicate the increase or decrease, respectively, of the factor on the left-hand side of the equation with increase in temperature. It can be obviously noted from figure 5 that $\mu$ ($n$) decreases (increases) as the temperature increases. The decrease in $\mu$ with increasing temperature is probably due to the decrease in the preferred conducting pathways and/or decrease in the degree of amorphousity at higher temperatures. However, the increase in $n$ is most probably
Figure 5. The ‘log $\mu - 1/T$’ (□) and ‘log $n - 1/T$’ (●) plots for NCPE OCC: 95(90PEO : 10AgNO$_3$) : 5SiO$_2$.

Figure 6. Cell potential discharge profiles for a thin-film solid-state battery: Ag/95(90PEO : 10AgNO$_3$) : 5SiO$_2$/(C + I$_2$ + NCPE) under a 100 kΩ load at three different temperatures: 15 °C (▲), 27 °C (●) and 40 °C (■).

a consequence of dissociation of Ag$^+$ ions from the ionic salt AgNO$_3$ in larger numbers as the temperature increases.

3.2. Thin-film solid-state battery application

Figure 6 shows the cell potential decay profiles for the thin-film solid-state batteries, mentioned in section 2, when discharged through a 100 kΩ load at three different temperatures: 15, 27 and 40 °C. An open circuit voltage (OCV) of $\sim$0.61 V was obtained for all the batteries. This is fairly low as compared to the theoretical OCV of $\sim$0.687 V with the Ag/I$_2$ electrode couple.
Table 2. Some important cell parameters calculated at the plateau region of the discharge profiles of figure 6 obtained at three different temperatures: 15, 27 and 40 °C.

| Load (kΩ) | Working voltage (V) | Current density (µA cm⁻²) | Discharge capacity (µA h) | Power density (mW kg⁻¹) | Energy density (mWh kg⁻¹) |
|-----------|---------------------|---------------------------|---------------------------|-------------------------|---------------------------|
| 100       | 0.36 (40 °C)        | 0.29                      | 23.4                      | 0.19                    | 12.96                     |
| 0.34 (27 °C) | 0.27              | 18.7                      | 0.16                      |                         | 8.96                      |
| 0.30 (15 °C) | 0.24              | 10.8                      | 0.15                      |                         | 6.75                      |

The reason for the low OCV may be due to the mixing of NCPE in the cathode material (C + I₂) [23]. The cell potential remained practically stable at ~0.3–0.4 V for ~60 h except for an initial drop which was due to the usual cell polarization effect. However, it can be clearly seen that the performance of the battery has been relatively superior at higher temperatures. Table 2 lists some important cell parameters calculated in the plateau region of the discharge profiles of figure 6. The ionic transference number \( t_\text{ion} \) in NCPE OCC was also evaluated alternatively using the electrochemical cell potential method with the help of the following equation [23]:

\[
t_\text{ion} = \frac{E'}{E},
\]

where \( E' \) and \( E \) are the measured and theoretical OCV values, respectively. Substituting \( E' \) and \( E \) into the equation, \( t_\text{ion} \sim 0.88 \), which is very close to the value obtained earlier through the dc polarization method.

4. Conclusion

A new Ag⁺-ion-conducting NCPE membrane: 95(90PEO : 10AgNO₃) : 5SiO₂ (wt%), has been synthesized employing a novel hot-press/solvent-free/dry technique. Dispersal of nano-SiO₂ particles into the polymer electrolyte host (SPE): (90PEO : 10AgNO₃) resulted in an enhancement in the room temperature conductivity as well as improvement in the mechanical stability/flexibility of the film. Ionic mobility \( (µ) \) and mobile ion concentration \( (n) \) studies on NCPEs and SPE indicated that the increase in room temperature conductivity is predominantly due to the increase in \( n \). The ionic transference number measurements by dc polarization and electrochemical cell potential methods indicated the fact that approximately 90% of the total Ag⁺ ions take part in the conduction process in the newly synthesized NCPE material. Thin-film solid-state batteries were fabricated using NCPE as the electrolyte and the cell potential discharge characteristics have been studied under fixed load conditions at different temperatures.

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