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A solid state actuator based on the PEDOT/NBR system

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Abstract

This paper reports the fabrication of a dry type conducting polymer actuator using nitrile rubber (NBR) as the base material for a solid polymer electrolyte. Thin films of NBR (150–200 μm) were prepared by using a compression molding process. A conducting polymer, poly(3,4-ethylenedioxythiophene) (PEDOT), was synthesized on the surface of the NBR layer by using a chemical oxidation polymerization technique, and room temperature ionic liquids (RTIL) based on imidazolium salts, e.g. 1-butyl-3-methyl imidazolium X [where $X = BF_4^-$, PF_6^-, $(CF_3SO_2)_2N^-$], were absorbed into the composite film. The effects of the anion-size of the ionic liquids on the displacement of the actuator were examined. The displacement increased with increasing the anion-size of the ionic liquids. The cyclic voltammetric responses and the redox switching dynamics of the actuators using different ionic liquids were examined.

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Keywords: Conducting polymer actuator; PEDOT; Nitrile rubber; Ionic liquid

1. Introduction

Electro-active polymer (EAP) actuators have attracted considerable attention on account of their high strength and large strain [1,2]. In an EAP actuator system, the applied redox potential can render ions transportable between a conducting polymer and a polymer electrolyte. The use of conventional liquid electrolytes is not practical, because the leakage or loss due to evaporation results in a limited working life time [3]. Clearly the choice of solvent can dramatically influence both the electrolyte and the anion stability but in an unpredictable manner [4].

Therefore, there is a need for improved electrolytes that satisfy the requirements of a high ionic conductivity, fast ion mobility during the redox events, and low volatility. Certain materials known as ionic liquids meet all of these requirements. Ionic liquids can be obtained in a very dry state, making them particularly suitable for applications in electrochemical systems where moisture must be excluded over a long period of operation. Recently, Cooper and Sullivan described an air- and moisture-stable room temperature ionic liquid consisting of the 1-ethyl-3-methylimidazolium (EMI) cation and trifluoromethanesulfonate (TFSI) anion, which maintains many of the required physical and electrochemical properties [5]. In addition, Fuller [6] demonstrated the utility of the $EMIBF_4$ ionic liquids as a versatile electrolyte.

The preparation and actuation of the dry type actuator, PEDOT/NBR/ionic liquid was previously reported [7–9]. In preparing the actuator system, nitrile rubber (NBR) was used as the base material for the solid polymer electrolyte. Nitrile rubber contains 40% of polyacrylonitrile (PAN), which has a high dielectric constant compared with polybutadiene, and the high polarity of PAN facilitates the absorption of a polar liquid electrolyte into the rubber matrix. It should be pointed out that the choice of the solid polymer electrolyte is critical when operating in air. It should not only have enough polarity for impregnating liquid electrolytes but also have sufficient flexibility for allowing actuation of the conducting polymer. In addition, it should have good adhesion with conducting polymer layers to prevent the delaminating problem. In our research, thin NBR film (150–200 μm) were prepared by using a compression molding process. A conducting polymer,

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poly(3,4-ethylenedioxythiophene) (PEDOT), was synthesized on the surface of the NBR by using a chemical oxidation polymerization technique. The PEDOT content was gradually decreased from the outside towards the center of the matrix film in order to yield a similar structure to that of an interpenetrating polymer network (IPN). The PEDOT/NBR film was filled with an ionic liquid, e.g. 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMITFSI) in order to take action as an solid polymer actuator. The results of this study show that the displacement rate in ionic liquids is greater than that observed in conventional electrolyte systems [9].

The actuators prepared in this study were activated in several ionic liquids: 1-butyl-3-methylimidazolium X [where X = BF_4^-, PF_6^-, $(CF_3SO_2)_2N^-$], and 1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide. This study determined the electrochemical actuation of several BMI ionic liquids. By examining three ionic liquid couples: BF_4^-, PF_6^-, and $(CF_3SO_2)_2N^-$, the anion-size of the BMIX ionic liquids was found to influence the displacement of the actuator.

2. Experimental

2.1. Materials

The NBR support was provided by Kumho Chem. Ltd. The dielectric constant of the NBR was 4.8 at 10^6 Hz. 3,4-Ethylenedioxythiophene (EDOT) was purchased from Aldrich Chemical Co. and was purified by passing it through an activated neutral aluminum oxide column. $FeCl_3$ was obtained from Aldrich Chemical Co. and was used as received.

2.2. Preparation of NBR film

The virgin NBR resin is a copolymer consisting of 60 mol% butadiene and 40 mol% acrylonitrile. NBR films used in actuators were synthesized by the following method. The virgin NBR resin was mixed with proper amounts of ZnO, S, and stearic acid in a Haake mixer at 90 °C for 5 min and then in a two roll mixer at 100 °C for 2 min. The resulting NBR compound was pressed into a mold at 160 °C for 20 min under a pressure of $100\,kg_f/cm^2$ to complete the vulcanization process. The films were 150–200 μm in thickness.

2.3. Preparation of PEDOT/NBR/BMITFSI composite film

The NBR films were soaked in pure EDOT for 10 min, and then their surface was wiped off with filter paper. The oxidative polymerization of swollen films was induced by immersing in a 1 M $FeCl_3$ solution for 6 h. The films were then washed several times with methanol. The resulting PEDOT-NBR films were dried in a vacuum at 25 °C for 24 h. After the edges were cut off, the PEDOT-NBR films were immersed in a 50/50 CH_2Cl_2/BMITFSI solution for 24 h at room temperature. After evaporating the CH_2Cl_2, the incorporated BMITFSI produced a 10–15% increase in weight. The composite films were stored in air.

2.4. Synthesis of ionic liquid

The ionic liquids were synthesized according to standard procedures and were dried prior to use [10–12]. The following ionic liquids were synthesized: 1-butyl-3-methyl imidazolium tetrafluoroborate ($BMIBF_4$), 1-butyl-3-methyl imidazolium hexafluorophosphate ($BMIPF_6$), 1-butyl-3-methyl imidazolium bis(trifluoromethyl sulfonyl)imide (BMITFSI), and *N*-butyl-3-methyl pyrrolidinium bis(trifluoromethyl sulfonyl)imide (BMPTFSI).

2.5. Instrumentation

The ionic conductivity of all the films was determined by using AC impedance spectroscopy. Two blocking stainless steel electrodes were used to hold the film and a Teflon spacer was included in order to fix the sample thickness. The impedance tests were carried out over the 10 mHz to 100 kHz frequency range by using an impedance/gain phase analyzer, Solarton SI model 1260.

The displacement of the actuator was measured by laser beam radiation using a Keyence LK-081 (Keyence Co., Japan). The point of the laser radiation was focused on the bottom of the film which was 30 mm long, 3 mm wide, and 200 °C thick. Cyclic voltammetry (CV) was performed by the cyclic scanning of the potential between −10 and 10 V with a sweep rate of $100–500\,mV\,s^{-1}$ in the solid electrode.

Dynamic mechanical analysis were measured on the original NBR and the ionic liquid swollen NBR films ($L \times w \times t = 10\,mm \times 5\,mm \times 0.158\,mm$) with Exstar 6000 (Seiko Instruments, Japan). The experiments were preformed at a frequency of 1 Hz and under 150 mN pre-tension and with a heating rate of $2\,°C\,min^{-1}$ from −100 to +50 °C. This setup allows measurements of the storage and loss moduli (E' and E'') and the damping parameter or loss factor ($\tan\delta$) defined as the ratio $\tan\delta = E''/E'$.

3. Results and discussion

The characteristics of the solid electrolyte films produced by the impregnation of an ionic liquid into the NBR matrix films was determined by measuring the ionic conductivity and the dynamic mechanical analysis (DMA) technique. The NBR films were activated for 24 h in a different ionic liquid of $BMIBF_4$, $BMIPF_6$, and BMITFSI. The variation in ionic conductivity as a function of the different ionic size for the imidazolium salts were as follows: $0.524 \times 10^{-4}\,S\,cm^{-1}$ for the NBR/$BMIBF_4$, $1.26 \times 10^{-4}\,S\,cm^{-1}$ for NBR/$BMIPF_6$, and $2.08 \times 10^{-4}\,S\,cm^{-1}$ for BMITFSI. The variation in ionic conductivity was examined as a function of the immersion time and the amount of BMITFSI loaded in the NBR was examined. The amount can be controlled by the swelling time.

Fig. 1 shows the absorption kinetics of BMITFSI into NBR. A plateau value was observed at approximately 18% (w/w) ionic liquid uptake was observed after 24 h exposure. The uptake of different ionic liquids, such as $BMIBF_4$ and $BMIPF_6$, were similar to that of BMITFSI because of similar polarity. No significant

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