



The ionic conductivity, mechanical performance and morphology of two-phase structural electrolytes based on polyethylene glycol, epoxy resin and nano-silica



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ABSTRACT

As one of significant parts of structural power composites, structural electrolytes have desirable mechanical properties like structural resins while integrating enough ionic conductivity to work as electrolytes. Here, a series of polyethylene glycol (PEG)-epoxy-based electrolytes filled with nano-silica were prepared. The ionic conductivity and mechanical performance were studied as functions of PEG content, lithium salt concentration, nano-silica content and different curing agents. It was found that, PEG-600 and PEG-2000 content in the epoxy electrolyte system had a significant effect on their ionic conductivity. Furthermore, increasing the nano-silica content in the system induced increased ionic conductivity, decreased glass transition temperature and mechanical properties, and more interconnected irregular network in the cured systems. The introduction of rigid *m*-xylylenediamine resulted in enhanced mechanical properties and reasonably decreased ionic conductivity. As a result, these two-phase epoxy structural electrolytes have great potential to be used in the multifunctional energy storage devices.

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1. Introduction

Lightweight has been more and more attractive in material design and fabrication for the promotion of energy efficiency in many applications [1]. The structural power composites (SPCs), a series of multifunctional materials simultaneously realizing the goal of load bearing and energy storage, are believed to be one of the most promising alternatives for conventional structural materials in manufacturing energy storage devices [2–4]. As vital components in SPCs, structural electrolytes play a critical role in providing high ionic conductivity and transferring mechanical loads [5–7]. In order for the optimization of structural electrolytes, not only mechanical properties, but also the electrochemical performance, i.e., the ionic conductivity, was considered to realize a better multifunctional performance [8–11].

The preparation of structural electrolytes mainly focused on two major strategies: one is the polymerization of certain kinds of monomers to form the capabilities of ionic-conductivity and load-bearing simultaneously, the other is blending ionic-conducting components with structural components to combine these two properties together. Snyder and co-workers [12,13] synthesized homopolymers and copolymers of the vinyl ester deriva-

tives containing polyethylene glycol (PEG), with a structural scaffold generated through crosslinking and the PEG side chains to enhance their ion transport capability. Willgert et al. [14,15] synthesized polyethylene oxide (PEO)-dimethacrylate/PEO-mono methacrylate lithium salt mixtures by photo-initiated curing, and the relationship between these polymers' ionic conductivity and mechanical properties was investigated. However, despite their manufacture process is simple and convenient, the trade-off between mechanical and conductive properties makes it difficult to realize an acceptable multifunctional performance for the SPC [12–15].

For another strategy, epoxy resin, a class of structural resin with high mechanical modulus and strength, have been widely used as structural polymer matrix. And the addition of the immiscible electrolyte into epoxy/amine system can form the two-phase phase structure, with the epoxy networks bearing loads and the electrolyte phase achieving ionic conductivity [16–18]. Recently, ionic liquid, propylene carbonate (PC) were used as additives to generate a bi-component structural electrolyte [17]. Their mechanical performance was relatively low because of plasticization of the structural network.

In addition, PEO or PEG was blended with epoxy and curing agent to study their mechanical properties, miscibility, and morphology [19–22]. Lu et al. [19] investigated the PEO/epoxy/amine system after curing and found that the molecular weight of PEO

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had a crucial influence on the components' miscibility. Hu et al. [20] found that for PEO-20000/epoxy blends cured by 1,3,5-trihydroxybenzene, the crystallization of PEO affected the forming kinetics and morphology of crosslinking network. The morphology and kinetics of the PEO crystallization were in turn influenced by the content and chemical structural of epoxy/amine system [21]. Feng et al. [22] found that the addition of PEG simultaneously enhanced the cryogenic tensile strength, ductility of epoxy resins. However, to our best knowledge, few literatures available have ever discussed the relationship between ionic conductivity and mechanical properties of PEG/epoxy systems though PEG has very high ionic conductivity, which merits further study to optimize the multifunctional performance of structural electrolytes.

Because bisphenol A epoxy resin contains the aromatic rings which present good mechanical performance after curing, and is miscible with PEG [23,24], bisphenol A epoxy resin was blended with PEG, lithium salt, nano-silica and curing agent to form the two-phase microstructures in this paper. Two different curing agents, polyetheramine D-400 and m-xylylenediamine, were used to form the crosslinking network for load-bearing, and PEG mixed with lithium salt acted as the ionic conductive pathways. Nano-silica was added into the system and the influence of lithium salt concentration and nano-silica content on ionic conductivity, mechanical performance and morphology of the structural electrolytes were discussed. Our results suggest that these two-phase structural electrolytes containing PEG, epoxy resin and nano-silica have great potential to be used as structural electrolytes in the multifunctional energy storage devices.

2. Experimental

2.1. Materials

The epoxy resin used in this study was liquid diglycidyl ether of bisphenol A (DGEBA) type epoxy resin E51 with an epoxy value of 0.51 purchased from Jinan Sunny Chemical Technology Co., Ltd. Nano-silica (purity: 99.5%, particle size: 30 nm) was from Shanghai Maikun Chemical Co. Ltd. PEG-2000 (molecular weight: 2000 Da) and PEG-600 (molecular weight: 600 Da) were purchased from TCI Shanghai. Lithium trifluoromethanesulfonate (LiTF) was from China Ship building Industry Corporation. Polyetheramine D-400 and m-Xylylenediamine (MXDA) were bought from Aladdin. The chemical structures of DGEBA, Polyetheramine D-400 and MXDA were shown in Fig. 1. All materials were dried in an ambient vacuum oven prior to use to remove moisture. All other chemicals were purchased from Beijing Chemical Works and used as received.

2.2. Preparation of samples

First, epoxy systems with different PEG content were prepared, the molar ratio of lithium and etheric oxygen in PEG ([Li]/[EO]) and the content of nano-silica were kept for 0.1 and 2.5 wt%, respectively. Then, the composition of PEG was changed to the mixture of PEG-2000 and PEG-600, the ratio of [Li]/[EO] and nano-silica content were still kept for 0.1 and 2.5 wt%, respectively. Furthermore, epoxy systems with varied [Li]/[EO] and nano-silica content were prepared.

The general preparation procedure was as follows: First, PEG and LiTF were mixed and ultra-sonicated until homogeneous liquid was formed and no precipitate was left. Then nano-silica and E51 were added into the mixture, followed by transferring them into the planetary mixer (ZYMC-200, Shenzhen ZYE Technology Limited). And they were mixed with a speed of 2000 rpm until a homogeneous mixture was formed. Finally, curing agents were added into the mixture and evacuated to degas until a uniform mixture was formed. Two different curing agents, polyetheramine D-400 and MXDA, were used, respectively. And the molar ratio between epoxy group in E51 and active hydrogen in curing agent for all systems was kept constant for 1.

Three different shapes of samples, disc samples for electrochemical impedance spectroscopy and morphology, bars for dynamic mechanical analysis (DMA) and dumbbell samples for tensile testing, were made by different Teflon molds. The mixture was poured into different preheated molds and degassed in vacuum oven at 60 °C for 20 min. The curing cycle was 80 °C for 2 h, 100 °C for 2 h and 120 °C for 2 h, the rate at which all the heating and cooling cycle was 2 °C/min. Macroscopic phase separation cannot be observed during the curing process. After demolding, the samples were machined to be smooth for characterizations. All samples should be kept in an ambient temperature vacuum drier prior to characterization to remove absorbing moisture.

2.3. Characterizations

2.3.1. Electrochemical impedance spectroscopy

The ionic conductivity of the cured disc samples (2–3 mm thickness, 20 mm diameter) was tested with CHI660E (Shanghai Chenhua Device Company, China) electrochemical workstation by electrochemical impedance analysis, which was carried out at ambient temperature (23–25 °C) using an amplitude of 10 mV in the frequency range of 1 Hz to 1 MHz. Samples were placed into a fixer for clamping with two stainless steel electrodes, and the whole fixer was sealed into an incubator. The high frequency arc was associated with the ionic conduction process in the bulk of the electrolyte system [25]. The low frequency part was attributed to blocking double layer capacitance near the electrode–electrolyte

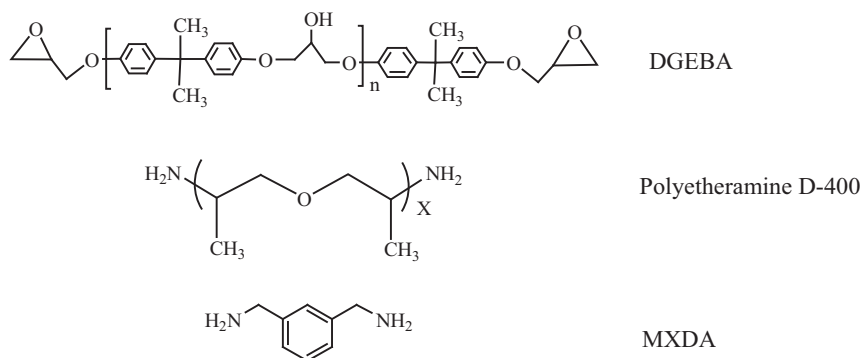


Fig. 1. Chemical structures of DGEBA, polyetheramine D-400 and MXDA.

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