



Effect of spinning condition and fiber alignment on performance of ionic polymer gel actuators with nanofiber mat electrodes



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ABSTRACT

We developed a novel type of actuator using conductive nanofiber mat electrodes, and investigated the relationship between the spinning solution concentration and the displacement of the actuator. The nanofiber mat was prepared using the electro-spinning method, and its conductivity was enhanced through the application of a conductive polymer coating. We used poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF/HFP) and poly(3,4-ethylenedioxythiophene) (PEDOT) for the nanofiber mat and the conductive polymer coating, respectively. It was found that the nanofiber diameter decreased with decreasing spinning solution concentration, resulting in an increase in the specific surface area of the nanofiber mat. The displacement of the actuator increased with the decreasing spinning solution concentration of the nanofiber mat electrode (i.e., as the specific surface area increased). In addition, we developed an actuator containing aligned nanofiber mat electrodes, which could lead to anisotropic deformation.

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1. Introduction

Actuators employing various conjugated polymers, such as polyaniline [1,2], polypyrrole (PPy) [3–5], and poly(3,4-ethylenedioxythiophene) (PEDOT) [6–8], have previously been studied. When an electric current is applied, these actuators deform through the volume change caused by the electrolyte ions passing in and out of the conjugated polymer [4,5]. Increasing the interfacial area between the electrolyte and the conjugated polymer should therefore lead to a larger volume change. Torop et al. recently reported on a novel type of actuator that employed a carbide-derived carbon, combined with a PPy film [9]. Through the addition of this carbide-derived carbon, they enlarged the specific surface area of the PPy film, resulting in an increase in performance. In addition, Ismal et al. studied the use of microfibrillar silk fibroin coated with polyaniline as a reactive sensor [1]. The microfibrillar structure of the fiber also led to an enlargement of the specific surface.

In this study, we employed a nanofiber mat as our electrode material. By processing a polymer into a nanofiber mat, a large surface area can be obtained. Electro-spinning is one of the popular methods for fabricating nanofiber mats, as it can

be easily used in the processing of polymers into nanofiber mats by applying a high voltage between the grounding plate (collector) and the spinning nozzle. Several authors have previously reported on fabricated nanofibers covered with metals and conducting polymers [10–12], while there are few papers detailing their application to actuators, with the exception of the piezoelectric actuator containing poly(vinylidene fluoride) (PVDF) nanofibers, fabricated using the direct-write electro-spinning technique [13,14]. Among the various conductive nanofibers, PEDOT is a chemically and thermally stable polymer and exhibits a high conductivity. By using a PEDOT coated nanofiber mat, both flexibility and a large specific surface area should ideally be obtained, resulting in a higher actuator performance. A Tetra-PEG ion-gel was used as the electrolyte material, which is known to be a highly ion-conductive and mechanically tough gel [15]. The Tetra-PEG gel was composed of two types of tetra-armed poly(ethylene glycol) prepolymers, tetra-amine-terminated PEG (TAPEG) and tetra-*N*-hydroxysuccinimide-terminated PEG [16]. In this study, we prepared a Tetra-PEG ion-gel using the “re-swollen method”, in which dried Tetra-PEG hydrogel is swollen using an ionic liquid, as reported in our previous work [17].

Previous reports suggest that nanofiber mat conductivity can be controlled using the orientation of the nanofibers [18]. An aligned nanofiber mat can be obtained through the electro-spinning method by using a rotating collector [19]. In this study, by applying

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an aligned nanofiber mat to the electrodes, we aimed to prepare a novel type of actuator that is able to drive anisotropically.

2. Experiment

2.1. Materials

Poly(vinylidene fluoride-hexafluoropropylene) (PVDF/HFP) and dicumyl peroxide (DCP) was purchased from SIGMA-ALDRICH. The average molecular weight (M_w) of the PVDF/HFP was approximately 400 kg/mol. 1-Ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide ([EMI⁺][TFSA⁻]) and 3,4-ethylenedioxythiophene (EDOT) were purchased from Tokyo Chemical Industry Co., Ltd. (Tokyo, Japan). *N,N*-dimethylformamide (DMF) was purchased from Nacalai Tesque, Inc. (Kyoto, Japan). Two types of Tetra-PEG prepolymers, TAPEG and TNPEG (both 40 kg/mol), were purchased from the NOF Corporation. These materials were all used as received.

2.2. Preparation of the electrode using a nanofiber mat

PVDF/HFP was dissolved in DMF at 80 °C. The various concentrations of the solution used were 20 wt%, 24 wt%, 27 wt%, and 30 wt%. After completely dissolving the PVDF/HFP, we added DCP as a cross-linker, and stirred at room temperature. The concentration of the DCP was 0.4 wt% for all solutions. The resulting solutions were then placed in a syringe and electro-spinning was carried out. A 21 gauge spinning nozzle with a 0.51 mm inner diameter was employed. The spinning voltage, temperature, humidity, feed rate, and the nozzle-collector distance were 19 kV, 23 °C, 41%, 0.6 mL/h, and 23 cm, respectively. The collector was a copper plate covered with aluminum foil, and the spun nanofiber mat was easy to peel away from the collector.

The resulting nanofiber mat was then heat pressed at 140 °C for 1 h using a 100 μm spacer, in order to add cross-linking points. EDOT was then applied to the cross-linked nanofiber mat, and the nanofiber mat was immersed in 35 wt% FeCl₃·6H₂O aqueous solution for 17 h in order to polymerize the EDOT on the nanofiber. The amounts of EDOT were 10 wt%, 30 wt%, and 63 wt% against the weight of the nanofiber mat. The resulting conductive nanofiber mat electrode coated with PEDOT was then repeatedly washed with methanol and dried completely.

2.3. Preparation of the actuator

The preparation method is illustrated in Fig. 1. First, a 1 mm thick silicon sheet containing a rectangular hole was attached to a slide glass and used as a mold for the actuator. Second, a nanofiber mat electrode of the same dimensions of the hole in the silicon sheet was adhered to the slide glass by wetting with distilled water (Fig. 1(a)). Next, the Tetra-PEG solution, a mixture containing an equal amount of the TAPEG and TNPEG aqueous buffer solutions [20], was pored over the nanofiber mat electrode. Another slide glass, to which a second nanofiber electrode was adhered by wetting with distilled water, covered over the Tetra-PEG solution before it became a gel (Fig. 1(b)). Fig. 1(c) shows the cross-section of a specimen after the process described in Fig. 1(b). The Tetra-PEG solution was sandwiched between two nanofiber mat electrodes. After the complete gelation of the Tetra-PEG, the upper piece of slide glass was removed, and the sample was allowed to dry completely. A [EMI⁺][TFSA⁻] ionic liquid was then absorbed for 5 h, using an amount of up to 80 wt% of the sample weight. The sample was finally cut into the shape of a 15 mm × 3 mm rectangular specimen. The thickness of the specimen was measured using a

thickness gauge (TCM-N1010, Toyo Seiki Seisaku-syo, Ltd., Tokyo, Japan).

2.4. Measurements

SEM observations were carried out using a Keyence scanning electron microscope (VE-9800, Keyence Co. Ltd., Osaka, Japan). The fiber samples were gold-sputter coated using an ion coater (SC-701, Sanyu Electron Co., Ltd., Tokyo, Japan). The average fiber diameter and standard deviation were determined from 100 measurements using the Adobe Photoshop CS3 extended program.

Specific surface area measurements were carried out using a Micromeritics Automatic Surface Area and Porosimetry Analyzer (TriStar 3000, Shimadzu Co., Ltd., Kyoto, Japan) using N₂.

The electrical conductivity of the nanofiber mat electrode was measured using a four-point probe resistivity meter (Lorester-AX MCP-T370, Mitsubishi Chemical Analytech Co., Ltd., Kanagawa, Japan).

The displacement of the actuator was measured at room temperature under a rectangular waveform voltage (±1.5 V, 0.05 Hz), using a laser displacement meter (LK-G85, Keyence Co., Ltd., Osaka, Japan).

Tensile measurements were performed using a mechanical testing apparatus (Tensilon UTM-III, Toyo Baldwin Co., Ltd., Tokyo, Japan) at room temperature. The rectangular samples (15 mm × 3 mm) were used and the tensile speed was 10 mm/min. The initial length was 10 mm.

2.5. Preparation of the aligned nanofiber mat

A schematic illustration of the electro-spinning apparatus using a rotating collector for the preparation of aligned nanofiber mats is shown in Fig. S1. The spinning solution concentration was 20 wt%, 24 wt%, 27 wt%, and 30 wt%. The DCP content was 0.4 wt%. The rotating speed was 3500 rpm, and the nozzle-to-collector distance was 7 cm. The diameter of the rotating collector was 16 cm. The spinning was carried out at a temperature of 22 °C, a humidity of 37%, and a voltage of 20 kV. The feed rate was 0.6 mL/h, and we used the same spinning nozzle as was used in the case of the disordered nanofiber mat (Section 2.2).

After the spinning process, the aligned as-spun nanofiber mat was heat-pressed at 140 °C without a spacer, and then coated with PEDOT in the same way as the nanofiber mat described above (Section 2.2). The resulting conductive aligned nanofiber mat was then applied to the actuator electrodes.

3. Results and discussion

3.1. Effect of spinning solution and EDOT concentration on the resulting nanofiber mat structure and properties

First, we describe and explain the structure and properties of the nanofiber mat electrodes prepared in this study. SEM images of the nanofiber mat prepared using various concentrations of both the spinning solution and EDOT are given in Fig. 2. It can be seen that the fiber diameter decreased with decreasing spinning solution concentration. The surface of the as-spun nanofibers appeared to be smooth, but the PEDOT coated nanofibers exhibited a rough surface structure. A similar rough structure was also reported by Temmer et al. for PEDOT coated PVDF membranes [8], while other studies reported a smoother and more uniform PEDOT coating [21]. This difference in coating properties may be attributed to differences in the PEDOT coating method employed. In our study, the PEDOT coating was carried out by immersing a cross-linked nanofiber mat in FeCl₃ solution, after dipping in an EDOT monomer solution. This method was reported by Temmer et al. [8]. However, the smooth

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