FABRICATION OF ALUMINUM OXIDE COATED CONDUCTIVE POLYURETHANE NANOFIBERS USING ELECTROCHEMICAL OXIDIZATION PROCESS

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In this research, we were succeeded in the fabrication of aluminum oxide coated poly urethane nanofibers using the combination of electro spinning and electrochemical procedures. A pristine polyurethane nanofiber doesn't have any electrical conductivity prior to its coating by aluminum oxide layer. In order to coat on the surface of PU nanofiber, we have developed a new method of fabricating aluminum oxide coated nanofiber using electrochemical procedure. We conducted cyclic voltammetry tests for evaluating electrical conductivity. Electrochemical three electrode system was composed of an electrospun nanofiber containing aluminum foil as a working electrode. Electrospun nanofiber containing aluminum foil was oxidized and aluminum oxide was coated on the surface of nanofiber. Aluminum oxide coted PU nanofiber has a high difference of current peak in I-V graph of cyclic voltammetry. The results confirms that the aluminum oxide coated PU nanofibers possess an excellent electrical conductivity that can be used for various applications.

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1. Introduction

Electro-spinning/netting, as a recently developed advanced electro hydrodynamics technique, may be considered as a variant of electrospinning process. Electrospinning have emerged as straightforward approaches to the fabrication of nanofibers with high specific surface areas, high porosities, and controllable compositions for a wide range of applications. The preparation of nanofibers using electrospinning method has attracted worldwide attention due to its versatile maneuverability of producing controlled fiber structures, porosity, orientations and dimensions.

Most of the polymers are conductive, and the charged ions in the polymer solution have a high effect on jet formation. However, like Polyurethane(PU)is non-conductive polymer even though presents a class of thermoplastic polymer that possesses a lot of excellent properties, such as good mechanical properties, excellent elastomeric properties and high durability. Many investigators have attempted to apply various degrees of control to this process in order to produce PU fibrous membranes with the designed highly conductive functions.

Here, we report the fabrication of PU nano-membranes which possess excellent electrical conductivity. A conductive aluminum oxide layer on the surfaces of PU nanofibers were deposited using an electrochemical method. Our results confirmed that such prepared aluminum oxide coated

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PU nanofibers possess an excellent electrical conductivity that can be used for various applications including biosensors.

2. Experimental

2.1 Materials and Electrospinning process

Commercially available PU pellets (Estane® Skythane® X595A-11, Lubrizol Advanced Materials, Inc., USA)were used as received. N,N dimethylformamide (DMF) and methyl ethyl ketone(2-butanone) (MEK, extra pure) were purchased from Showa Chemical Co., Ltd., Japan and Junsei, Japan, respectively, and were used as solvent. .

PU pellets were used after drying for at least 3 h at 80˚C. 10 wt% PU solution was prepared in DMF/MEK (50:50 by weight). Electrospinning was carried out using the set-up as shown in Fig. 1. The different samples of electrospun mats were fabricated using multi nozzle electrospinning system which is controlled by the spray angle for functionally graded membrane (FGM). The electrospinning parameters include 15 kV applied voltage, tip-to-collector distance of 18 cm, and solution feed rate of 1 ml/h. After electrospinning, the nanofibrous mats were dried in vacuum drying for 12 h at 30˚C.

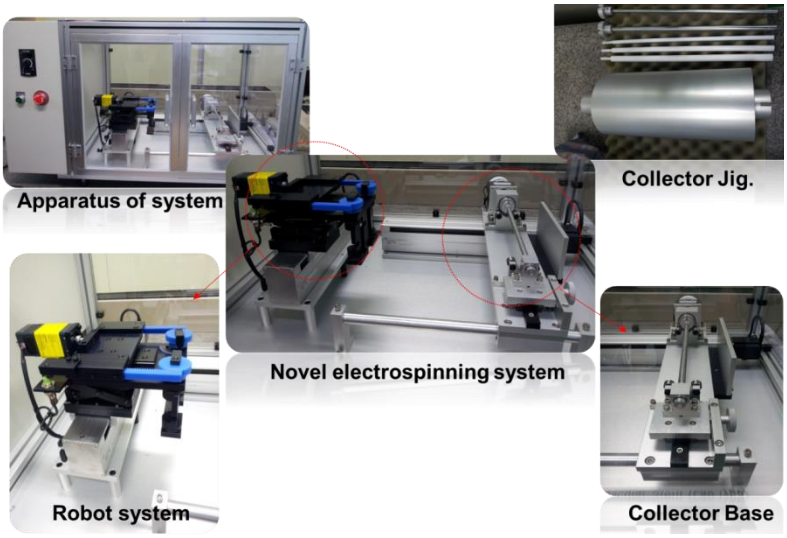


Fig. 1 Schematic of experiment equipment

2.2 Cyclic voltammetry experimental system

The Fig. 2 illustrates the main components of the electrochemical experiment cell setup. The cell is composed of a quartz crystal and two Viton O-rings were used to prevent leakage of test solution. Bowl shaped Pyrex glass tube was used to feed test solution to the quartz crystal. The cell case was made of Teflon (PTFE). The whole setup is connected to a data acquisition system for automatic data acquisition and storage. The circular quartz crystal has a diameter of 1.4 cm. At the center of the crystal used for working electrode, a concentric circular metal surface of diameter 0.5 cm was prepared by means of plasma vacuum coating. The crystals can be modified with any of the metals including Cu, Au, Ni, Cr, Ti, and Al for testing. In the reference electrode, a similar circular metal surface of Au/Ti was used. In setting up the cell, two rubber O-rings were used to prevent leakage between the circular quartz crystal and the connecting glass tube used for supplying the test solution. The crystal was carefully mounted onto holders, which were used to connect an AC power source for excitation of the crystal. The schematic diagram of key component in EQCN were shown if Figure. 2.The reference and testing cells were positioned side-by-side, and a frequency of 5 MHz was applied to both crystals.

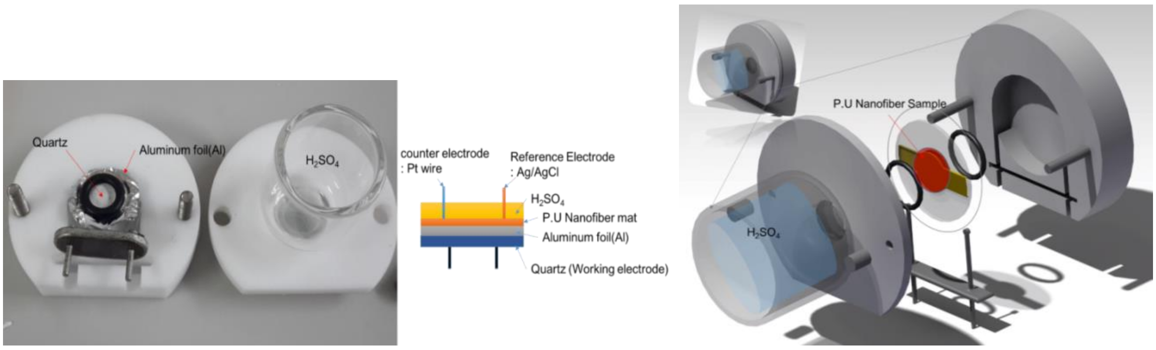


Fig. 2. Real electrochemical experiment configuration using QCM

2.3 Characterization

The surface structure and morphology of the present Al_2O_3nanofiber composite mat were studied by field emission scanning electron microscope (FESEM, S-7400, Hitachi, Japan). All the prepared samples were used directly as electrodes for electrochemical analysis. The electrochemical properties of the samples were measured on a potentiostat (Won-A tech, South Korea), which was examined using a three-electrodetesting system. The system consisted of 1M H_2SO_4 as the electrolyte, a platinum plate and Ag/AgCl as the counter and reference electrode, respectively. Cyclic voltammetry (CV) of the electrodes was conducted at scan rates of 10, 20, 30, 40, and 50 mV s^{-1} at potential windows between e 0.2 and 0.8 V.

3. Results and discussion

3.1 characterization of composite fiber containing Al2O3

FE-SEM images are illustrated in Fig. 1. Sphere shaped Al_2O_3 particles were incorporated into PU by the electrochemical process. The morphology of the pristine PU showed a smooth and porous structure and the fibers were distributed into a wide range of 240-600 nm in a diameter while the morphology of the PU/Al_2O_3 composite showed the deposition of particles into the nanofibers.Al_2O_3 nanoparticles contributed to improve electrical conductivity.

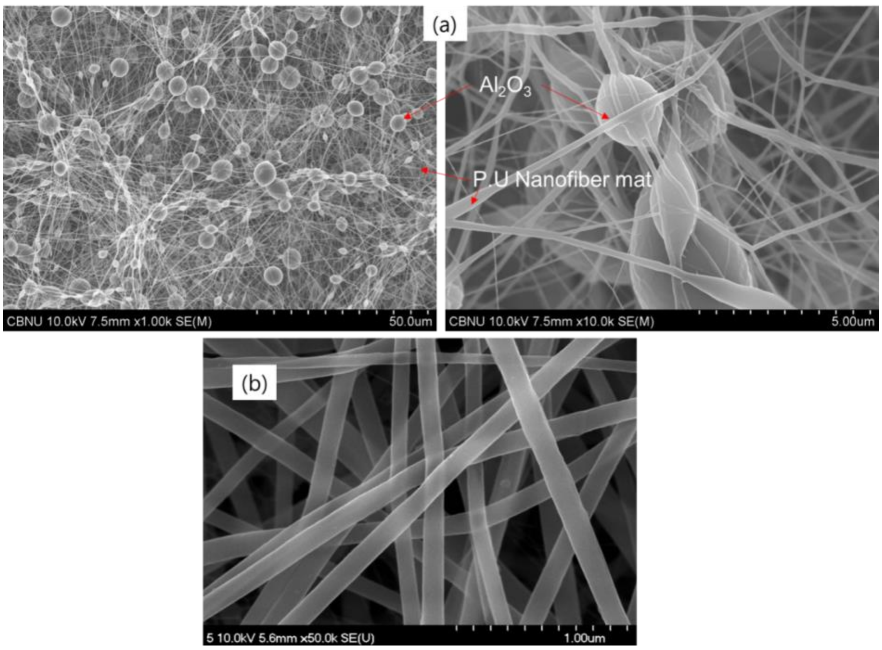


Fig. 3 FE-SEM images of (a) PU/Al2O3 composite mat, (b) Pristine PU

3.2Cyclic voltammetry (CV) analysis

Fig. 4 illustrate I-V graph of cyclic voltammetry. Pristine PU (4a) has no specific oxidation or reduction peak. It behaves like an insulator, showing relatively low current peak difference compared to PU/Al_2O_3 composite mat. On the other hand PU/Al_2O_3 composite mat shows high current peak difference (4b). As cycle number increased, current peak difference increased larger. It means much more current flows through the interface between PU and Al_2O_3nanoparticles than pristine PU. Figure 4c shows it clear that current increased through the repeating of oxidation of aluminum and we could ratiocinate that electrical conductivity increased.

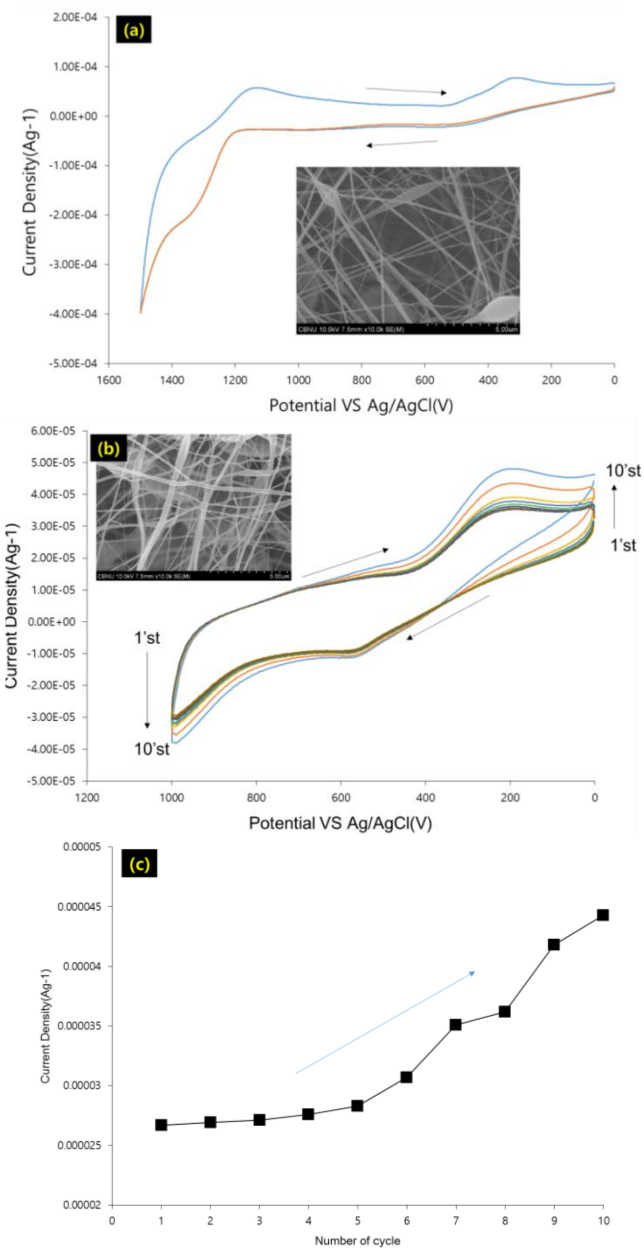
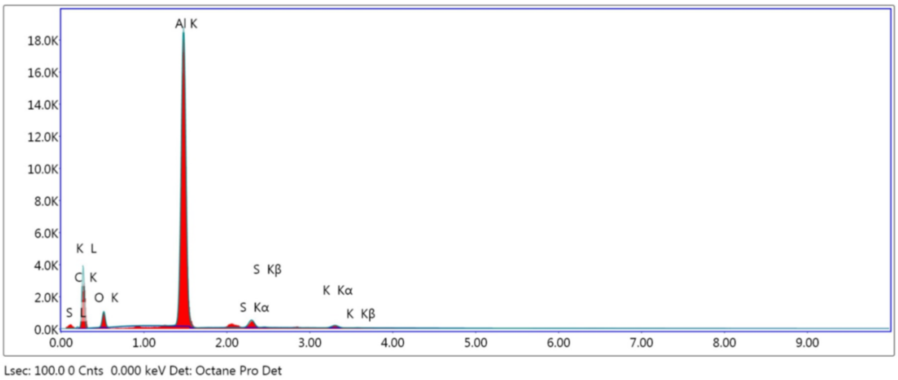


Fig. 4. CV curves (a) Galvanostatic charge-discharge curves

3.3EDAX Analysis

EDAX was performed for Qualitative analysis of PU/Al_2O_3 composite. Aluminum and oxide peak indicate presence of Al_2O_3 on the surface of PU nanofiber.



Element	Weight%	Atomic%	Net Int.	Error%
C K	49.14	66.45	263.97	10.00
O K	7.9	8.02	87.43	10.61
Al K	40.33	24.28	2538.11	2.31
S K	1.72	0.87	72.75	7.22
K K	0.91	0.38	28.71	10.71

Fig. 5. EDAX Results and table

4.Conclusions

In this research, the electrical conductivity of a non-conducting polyurethane nanofibers were improved through the electrochemical oxidization process. PU nanofibers coupled with aluminum oxide nanoparticles were fabricated using our current strategy. Aluminum nanoparticles is a key material for increasing the electrical conductivity since it form the interfaces between PU and aluminum foil. As number of cycles increased, current peak difference increased to higher levels. Consequently, PU/Al_2O_3 composite mat which was fabricated by electrochemical method, shown much high electrical conductivity and it can be successfully applied for biosensors and bioelectronics fields.

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