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Multifunctional enhancement of woven carbon fiber/ZnO nanotube-based structural supercapacitor and polyester resin-domain solid-polymer electrolytes

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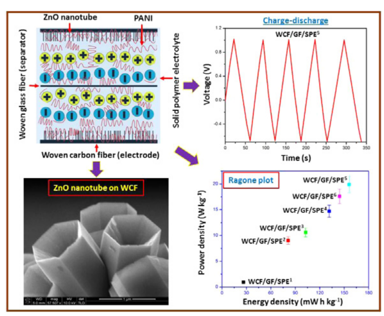
HIGHLIGHTS

- We fabricated WCF/ZnO nanotube electrodes based structural supercapacitor.
- We developed a solid polymer electrolyte of $EMIMBF_4$, LiTf and polyaniline nanofiber.
- The specific capacitance (up to 18.8 F g^{-1}) was substantially improved.
- Energy and power densities (up to 19.87 W kg^{-1}) were significantly improved.

GRAPHICAL ABSTRACT



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ABSTRACT

Structural supercapacitors can be considered as next-generation energy storage devices that have significant simultaneous performance characteristics in both structural applications and battery-like functions. In this study, we report the development of novel structural supercapacitors for the first time based on ZnO nanotubes, grown on woven carbon fiber electrodes, with a glass fiber separator. A solid polymer electrolyte is developed by mixing an ionic liquid ($EMIMBF_4$), a lithium salt (LiTf), and polyaniline nanofiber with a polyester resin matrix. The supercapacitor is fabricated by a vacuum-assisted resin transfer molding process that is both effective and eco-friendly. The specific capacitance of the supercapacitor enhances to 18.8 F g^{-1}, versus 0.2 F g^{-1} for a bare carbon-fiber supercapacitor. Large increases in energy (156.2 mW h kg^{-1}) and power density (19.87 W kg^{-1}) are also achieved, with exceptional tensile strength (325 MPa) and modulus (21 GPa) values. The device demonstrates strong multifunctional performance so that it can be used confidently for energy storage in electric vehicles and unmanned aerial vehicles, and in the aerospace industry generally.

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1. Introduction

In recent years, due to shrinking of fossil fuel reserves, energy harvesting has become an active area of research [1,2]. In the meantime, batteries and supercapacitors work as alternate energy storage devices that have high applicability due to their superb performance [3]. While slow kinetics during redox processes restricts the power density of fuel cells and solar batteries despite their high energy densities [4], supercapacitors have attracted substantial attention due to wide ranges of possible energy and power densities among them as storage devices in various applications, such as hybrid electric cars and portable electronics, and in the aerospace industry [5,6].

The replacement of ‘conventional’ metallic components by fiber-reinforced polymeric composite counterparts in the automobile, aerospace, and defense industries is a well-known practice

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[7,8]. It offers multiple benefits, such as higher mechanical strength, lower fuel consumption, and outstanding durability. Structural components made of glass fibers and carbon fibers offer high tensile strength, reasonable failure strength, and excellent compressive strength [9,10]. Carbon fiber (CF)-based polymeric composites are also excellent candidates for the automotive and aerospace sectors because of their light weight, low cost, straightforward manufacturing technology, and outstanding capabilities to improve specific strength, thermal and electrical conductivity, and especially corrosion resistance [11,12].

Recently, an outstanding novel manufacturing process has been developed that may change the entire body of an automobile into an energy storage device that can work in the absence of fuel and still support the required mechanical stresses in the body [13]. In this process, a structural composite has been developed on the basis of the principle of a supercapacitor by combining two carbon fiber layers sandwiched with one glass fiber layer [14]. A significant characteristic of the glass fiber separator is that it should allow the passage of ions from one side to other, while it should be an insulator to electrons [15]. Although some polymeric membranes, such as porous polypropylene film, polyethylene-terephthalate film, and polycarbonate film, have high capacitance values, they do not provide sufficient load-bearing capacity [16]. The use of glass fiber can satisfy both the required electrical properties and high mechanical stiffness and load-bearing capacity.

Among the various challenges in the development process, an important issue is to increase the surface area of the carbon fiber electrodes. Of the different processes available, surface activation by basic solutions, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), increases the surface area only minimally [17,18]. Researchers have also assessed the process of growing carbon nanotubes on the surface of carbon fibers, with reasonable enhancement of surface active sites. Moreover, applying a monolithic carbon aerogel on the surface of carbon fiber can result in high surface area improvement [19]. Recently, our group used a new method to enhance the active surface area by growing metal oxide nanorods on the surface of carbon fibers [20]. These metallic nanorods greatly increase mechanical properties and provide reasonable improvements in the capacitance of the carbon fiber sheets.

The development of a multifunctional matrix, commonly referred to as a solid polymer electrolyte (SPE), is another challenge in the development of a structural supercapacitor. As most structural polymers are generally non-conducting in nature, to use such polymers in an SPE, researchers have used ionic liquids and different cationic salts with the base polymer [21]. A problem arises at this point because the conductivity and mechanical properties of SPEs are typically inversely proportional [22]. Thus, the addition of ionic liquids or other additives to improve the conductivity should be tuned in such a way that the mechanical properties are also retained at a maximal level.

In this study, ZnO nanotubes were grown on the surface of carbon fibers to enhance their surface area. Woven carbon fiber/ZnO nanotubes composites were developed using a vacuum-assisted resin transfer molding (VARTM) process, which was economical, eco-friendly, and time-saving, and which provided consistent mechanical properties [23]. The electrochemical and mechanical properties of these composite based supercapacitors were evaluated.

2. Experimental section

2.1. Materials

The woven carbon fibers (T-300, density 1.76 g cm^{-3}, 3 K plain weave) used for electrode development were supplied by Amoco Corporation (Chicago, IL, USA). The woven glass fiber separator (thickness ~0.2 mm, plain weave, weight ~ 200 g m^{-2}) was provided by JMC Corporation, South Korea. Analytical-grade zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$) and sodium hydroxide (NaOH) for seeding processes were from Sigma-Aldrich (St. Louis, MO, USA). Zinc oxide nanorods on the WCF surface were synthesized with zinc nitrate tetrahydrate ($Zn(NO_3)_2{\cdot}4H_2O$) and hexamethylene tetramine ($C_6H_{12}N_4$), from Sigma–Aldrich. The unsaturated polyester resin (LSP-8020B) was from CCP Composites (Jeollabuk-do, South Korea) and the curing agent, methyl ethyl ketone peroxide (MEKP), was from Arkema (Kunsan City, South Korea). Ionic conductivity was enhanced using 1-ethyl-3-methylimidazolium tetrafluoroborate ($EMIMBF_4$; reagent-grade; C-Tri, Chuncheon City, South Korea) and lithium trifluoromethanesulfonate (LiTf; Sigma–Aldrich, Seoul, South Korea). Other chemicals and reagents were of analytical grade and were used as received.

2.2. Hydrothermal synthesis of ZnO nanotubes on WCF

The process of growing zinc oxide nanotubes on a WCF surface was carried out in several steps. First, WCF samples (80 × 80 mm^2) were washed with ethanol solution to remove impurities and induce the growth of nanorods. In the next step, ZnO seeding was performed on the treated WCF to increase the interaction of nanorods with the carbon fiber surface. For this, 10 mM zinc acetate dihydrate (98%, $Zn(CH_3COO)_2{\cdot}2H_2O$) solution was prepared with sodium hydroxide solution. The WCF samples were then dipped in the seeding solution, followed by annealing (150 °C, in an oven, 10 min). This cycle was repeated four times. These samples were used for the growing of nanorods in the next step. For nanorod synthesis, equimolar amounts of hexamethylenetetramine (HMTA; $C_6H_{12}N_4$) and zinc nitrate tetrahydrate ($Zn(NO_3)_2{\cdot}4H_2O$) were dissolved separately in distilled water at room temperature for 10 min. This was followed by mixing the solutions and stirring for 30 min. The pH of the solution was maintained at 6–8. This growth solution (100 mM) was used to treat the WCF samples in a stainless steel autoclave (100 °C, atmospheric pressure). The autoclave was opened after 8 h, and the WCF samples were washed with distilled water and used in further steps for the nanotube synthesis. The entire process of ZnO nanorod synthesis can be summarized as follows [24]:

$$C_6H_{12}N_4 + 6H_2O \leftrightarrow 6HCHO + 4NH_3 \tag{1}$$

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \tag{2}$$

$$2OH^- + Zn^{2+} \leftrightarrow Zn(OH)_2 \tag{3}$$

$$Zn(OH)_2 \leftrightarrow ZnO + H_2O \tag{4}$$

In the next step, the ZnO nanorods grown on the WCF samples were dipped in 0.18 M KOH solution at 70 °C [25]. After 2, 4, 5, 5.5, and 6 h, samples were removed from the solution, washed with distilled water, and dried (room temperature, overnight).

2.3. Preparation of the ZnO nanotube-WCF/polyester composite capacitor

The supercapacitor was developed using a VARTM process [23]. In this, the carbon fiber electrodes, along with the glass fiber separator, were placed inside the VARTM chamber. A schematic representation of the supercapacitor, its internal microstructure, and the VARTM set-up is shown in Fig. 1. Two copper electrodes were attached to both sides of the carbon fiber sheets. There were inlet and outlet vaults at the chamber for the flow of resin. The VARTM

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