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Fabrication of Ceramic Nanofibers Using Atrane Precursor

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1. Introduction

One-dimensional (1D) ceramic nanostructures, such as nanofibers, nanotubes, nanorods, nanowires, nanorings, nanobelts, and nanowhiskers, have been a very exciting and promising research topic in the last decades. This interest has been aroused by their unique properties and a wide variety of potential applications in future nanoelectronics and functional nanodevices, such as optoelectronics, photonics, cosmetics, health, bioengineering, gas and humidity sensors, mechanics, and catalysis (Xia et al., 2003; Wang, 2004; Carotta et al., 2007; Kumar & Ramaprabhu, 2007; Luo et al., 2007). A large number of techniques are available to prepare these materials, including vapour-phase method (Pan et al., 2001; Zhang et al., 2002), vapour-liquid-solid method (Morales & Lieber, 1998; Duan & Lieber, 2000), solution-liquid-solid method (Trentler et al., 1995; Markowitz et al., 2001), template-assisted method (Martin, 1994; Han et al., 1997; Zach et al., 2000; Barbic et al., 2002), solvothermal method (Heath & LeGoues, 1993; Wang & Li, 2002), arc discharge (Iijima, 1991), pulsed laser ablation (Sasaki et al., 2004), and precursor thermal decomposition (Wang et al., 2002a). Nevertheless, developing simpler and versatile approaches to the synthesis of one-dimensional ceramic nanostructures still remains a challenge. Among various approaches, electrostatic spinning (electrospinning) seems to be the simplest and most versatile technique capable of fabricating one-dimensional nanostructures including polymer, ceramic and composite nanofibers (Huang et al., 2003, Shao et al., 2003, Ksapabutr et al., 2005a ; Ksapabutr et al., 2005b; Li et al., 2006; Sigmund et al., 2006; Panapoy et al., 2008a; Panapoy et al., 2008b). Like a number of conventional spinning processes, electrospinning involves the ejection of a viscous solution (or melt) from an orifice and the subsequent drawing and solidification of the jet to form thin fibers. Unlike the conventional spinning processes, the exclusive driving force for the formation of ultrafine fiber in the electrospinning process is the electrostatic interaction. The fundamental principle of electrospinning is that a Taylor cone is formed by applying an electrical field to the viscous solution or melt hanging from a capillary tip, which causes jets of an electrically charged solution to be emitted when the applied electrostatic force exceeds the surface tension of the solution. A liquid jet shoots away from the capillary tip towards an oppositely charged electrode and solidifies with evaporation of solvent to form a mat on the grounded

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collector (Wang et al., 2002b; Berkland et al., 2004; Seong et al., 2005; Kalayci et al., 2005). Because of the small pore size and high surface to volume ratio inherent in electrospun nanofibers, these fibers show promise for interesting applications in fields of filtration, protective clothing, self-cleaning, drug delivery, tissue engineering scaffolds, wound dressings, biomimetic materials, composite reinforcement, electronic, optical and photonic devices, etc. (Remant et al., 2000; Guan et al., 2003; Li et al., 2003; Yuh et al., 2005; Zhang et al., 2007; Qi et al., 2008).

Zirconia (ZrO_2) is a well-known material with extreme refractoriness, high mechanical strength and fracture toughness, thermal insulation, wear and erosion resistance, chemical durability and alkali resistance. It has received intensive attention, due to its potential application in diverse fields, particularly in functional ceramics, electronic ceramics, constructional ceramics, biological ceramics, functional high-temperature coatings, heterogeneous catalysis processes, fuel cell technology, and so on (Cheung & Gates, 1997; Chadwick, 2000; Park et al., 2000; Feng et al., 2004). Its applications range from thin films to particles, platelets, whiskers and fibers. One-dimensional zirconia nanofibers have attracted considerable attention because of their surface dependant and size dependant properties and wide potential applications.

A variety of methods for the preparation of zirconia fibers have been reported, including relic process, in which organic polymer fibers are impregnated with zirconium salts, dried, and heated to burn out organic material and form zirconia fibers, spinning of the fiber precursor from organic polymer solution containing zirconium salt or fine zirconia particles with subsequent calcination process, and dry spinning of polyzirconoxane, or sol solutions prepared by sol-gel processing of zirconium acetate or alkoxide followed by calcination (Abe et al., 1998; Li et al., 2007). However, many efforts have been made to synthesize zirconia nanofibers by the electrospinning using zirconium salt or fine zirconia particles followed by calcinations process. For example, Shao et al. (2004) were the first to prepare zirconia nanofibers with diameters of 50–200 nm using the electrospun thin fibres of poly(vinyl alcohol)/zirconium oxychloride composites as precursor and through calcination treatment at 800°C. The viscous gel was prepared from zirconium oxychloride, poly(vinyl alcohol), and distilled water in the weight ratio of 1:2:20. Jing et al. (2005) prepared electrospun zirconia nanofibers having an average diameter less than 40 nm after calcination at 500°C using a viscous solution of zirconium oxychloride / poly(vinyl pyrrolidone) / ethyl alcohol / distilled water in the weight ratio of 1:1:3:3. Zhang and Edirisinghe (2006) also prepared zirconia nanofibers down to about 200 nm by electrospinning a mixture of a zirconia suspension and a poly(ethylene glycol)–poly(ethylene oxide) solution after calcinations at 600°–1200°C. An acetate-stabilized suspension containing 20 wt% of 5–10 nm size zirconia particles in water (pH = 3.5) was used. The polymer solution was prepared by dissolving poly(ethylene glycol):poly(ethylene oxide) (1:2 weight ratio) in a water-tetrahydrofuran (10:1 by volume). Two to 30 wt% of the zirconia suspension was added to the polymer solution.

The sol-gel process is a technique which is applicable for forming ceramic materials. In this process, liquid precursor materials are reacted to form a sol which then polymerizes into an inorganic polymeric gel. Advantages of this process over other techniques of ceramics are: compositional and microstructure control, wide variety in shape fabrication, and low processing temperatures. Furthermore, the metal alkoxide precursors used in the sol-gel process give homogeneity and high purity of metal oxide products although they are greatly

moisture sensitive and more expensive. Unfortunately, the sol-gel technique traditionally applies metal alkoxide and organic solvent as raw materials and requires the careful addition of water to prevent uncontrolled hydrolysis of metal alkoxide. Chelating agents, such as acetylacetone, are sometimes added to control hydrolysis during the gelation process, and the reactions often are carried out in nitrogen atmosphere. The synthesis of novel precursor is necessary for the development of metal alkoxide chemistry and the investigation in sol-gel field (Charoenpinijkarn et al., 2001; Ksapabutr et al., 2004a; Ksapabutr et al., 2004b; Ksapabutr et al., 2005c). Generally, the atrane complexes are performed by means of trans-esterification reaction, using alkoxide derivatives in non-aqueous dried solvent under an inert atmosphere. The procedure is quite complicated. Furthermore, alkoxide starting materials used for the synthesis of atrane complexes are commercially expensive (Menge et al., 1991; Nugent et al., 1994). Anodic dissolution of the metal can provide an easy and straightforward way to scale up synthesis of alkoxides for many metals. Reactions between oxides or hydroxides and aminoalcohols can also be cost-effective routes to new metal alkoxides of some elements. This route is termed as the “oxide one-pot synthesis” (OOPS) process. The oxide one-pot synthesis process is a cheap, simple and straightforward process for preparing highly moisture stable and pure metal alkoxide precursors using inexpensive and readily available starting materials (Charoenpinijkarn et al., 2001; Opornsawad et al., 2001; Ksapabutr et al., 2004c; Panapoy & Ksapabutr, 2008c). Interestingly, metal derivatives of triethanolamine are comparatively more hydrolytic resistant than their alkoxide analogues. The chelating nature of the triethanolamine and coordinative saturation achieved by the central atoms in the final products appear to be the main factor for their hydrolytic stability to retard the hydrolysis and condensation reaction rates in order to obtain homogeneous gels rather than precipitates. Zirconatran, which is one of atrane complexes, is an aminoalkoxide derivative of zirconium metal. Its structure possesses a remarkable stability property, leading to more controllable chemistry and minimizing special handling requirement. This advantage has made zirconatran a promising candidate for use in ceramic and composite processing. Therefore, the use of another zirconia source that possesses higher stability towards air and moisture could overcome such obstacles. The present study is aimed at exploring the use of zirconatran as the zirconia source for the fabrication of one-dimensional zirconia fibers via a combination of sol-gel technique and electrospinning method with subsequent calcination.

2. Synthesis of zirconatran precursor

Zirconatran used as a precursor for preparing zirconia nanofibers was synthesized directly from inexpensive and abundant raw materials, zirconium hydroxide and triethanolamine, via the oxide one-pot synthesis (Panapoy & Ksapabutr, 2008c). A mixture of 0.11 mol of zirconium hydroxide, 0.25 mol of triethanolamine, 0.15 mol of sodium hydroxide, and 20 mL of ethylene glycol was heated at 200°C to distill off ethylene glycol along with the removal of by-product water under nitrogen atmosphere. The reaction was completed within 5 h, and the resulting solution was slowly cooled down to room temperature. Dried methanol and acetonitrile used as precipitants were added into the solution and a white solid precipitated. After filtration, the white solid was washed with dried acetonitrile to remove unreacted ethylene glycol and triethanolamine and dried under vacuum at room temperature. The precursor obtained was identified using Fourier-transform infrared spectroscopy (FTIR), ^1H and ^{13}C nuclear magnetic resonance (NMR) spectroscopy, elemental

analysis (EA), mass spectrometry (MS). Fourier-transform infrared spectroscopic analysis was conducted using a Bruker Optik Vertex70 spectrometer with a spectral resolution of 4 cm^{-1} using transparent KBr pellets. ^1H and ^{13}C -nuclear magnetic resonance spectra were obtained on a Bruker AV300 spectrometer using deuterated dimethyl sulfoxide (DMSO-d_6) as solvent and reference for chemical shift measurements at room temperature. Elemental analysis was carried out on a CHNS/O analyzer (Perkin Elmer, PE-2400 series II). Matrix assisted laser desorption ionization time of flight mass spectrometry (MALDI-TOF MS) was performed on a ReflexIV instrument (Bruker Daltonics, Germany).

The reaction between a metal oxide or hydroxide and triethanolamine (or ethylene glycol) is a condensation reaction in which water is produced as a by-product of the reaction (Piboonchaisit et al., 1999; Opornsawad et al., 2001; Ksapabutr et al., 2004c). Therefore, the reaction was carried out at the boiling point of ethylene glycol under a nitrogen atmosphere in order to remove water and drive the reaction in the direction of the products. The reaction sequence in the synthesis of zirconatrane complex is illustrated in Fig. 1.

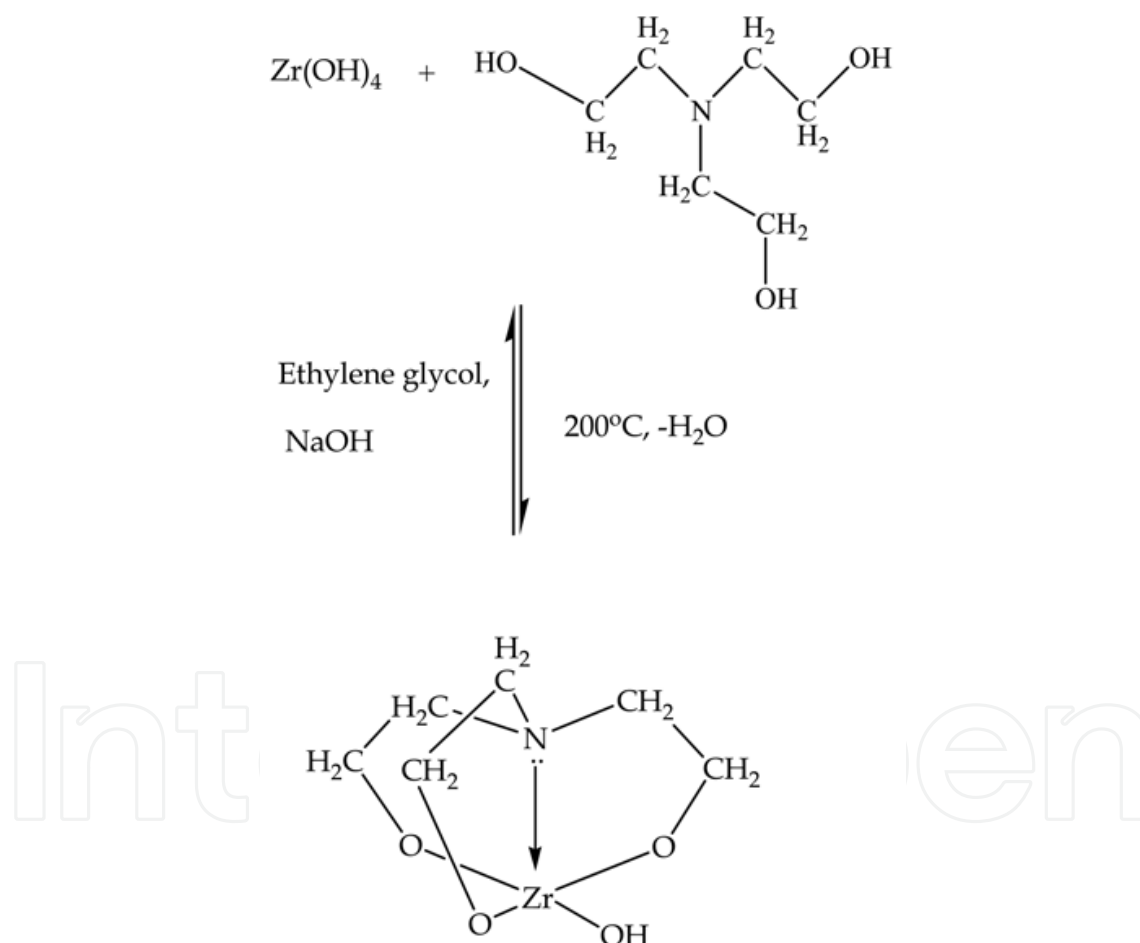


Fig. 1. Diagram of the reaction sequence in the synthesis of zirconatrane (adapted with permission from (Panapoy & Ksapabutr, 2008c))

The Fourier-transform infrared spectroscopic result showed the following peaks: 3000–3680 cm^{-1} (O-H), 2800–2976 cm^{-1} (C-H), 1240–1275 cm^{-1} (C-N), 1120 cm^{-1} (C-O) and 1000–1090 cm^{-1} (Zr-O-C). The ^1H -nuclear magnetic resonance spectra of product recorded in deuterated dimethyl sulfoxide solvent: the resonances at 2.3–2.7 and 3.0–3.5 ppm were assigned to N-

CH₂ and CH₂-O, respectively. The result of ¹³C- nuclear magnetic resonance showed peaks at 57.6 and 60.1 ppm, corresponding to N-CH₂ and CH₂-O, respectively. Both elemental analysis and mass spectrometry were also carried out. The results are shown in Tables 1 and 2, respectively. For the elemental analysis, it was found that the obtained percentages of carbon, hydrogen and nitrogen are very close to those theoretically calculated (Table 1). The proposed structures and fragmentation patterns present in Table 2 clearly indicate the tetramer of zirconatrane (m/z 1019). According to the obtained fragmentation patterns, the expected products were successfully synthesized.

Element (%)	Experimental	Theoretical
C	28.49	28.32
H	5.47	5.11
N	5.25	5.50

Table 1. Percentages of carbon, hydrogen and nitrogen present in the synthesized zirconatrane

m/z	Intensity	Proposed structure
1019	63	[Zr ₄ C ₂₄ H ₅₂ O ₁₆ N ₄]+2H ⁺
931	25	[Zr ₄ C ₂₀ H ₄₄ O ₁₄ N ₄]+2H ⁺
887	20	[Zr ₄ C ₁₈ H ₄₀ O ₁₃ N ₄]+2H ⁺
764	100	[Zr ₃ C ₁₈ H ₃₉ O ₁₂ N ₃]+H ⁺

Table 2. Proposed structures and fragmentation patterns of zirconatrane (reproduced with permission from (Panapoy & Ksapabutr, 2008c))

3. Zirconia nanofibers

3.1 Fabrication of zirconia nanofibers

The configuration of the electrospinning setup used for the fabrication of zirconia nanofibers is schematically demonstrated in Fig. 2. The main equipment consists of a high-voltage power supply unit, a starting solution feeding unit, and a temperature control unit. The starting solution was pumped towards a nozzle by means of a syringe pump. When high positive voltage was applied to the starting solution, an electrostatic field was established between the metal capillary nozzle and the grounded collector. A small pendant drop is formed on the nozzle tip and the resulting electrostatic field is sufficient to overcome the surface tension of the starting solution and create a Taylor cone shape at the nozzle tip, whereupon a cone-jet of highly charged liquid particles is formed. During jet travel, the solvent evaporates and the remaining solid fibers are deposited on the grounded collector. Nanofibers were accumulated on the collector as long as the nozzle tip was continually supplied with the starting solution. Finally, an interconnected web of small filaments or a nonwoven fiber was created on the surface of the grounded collector. Zirconia nanofibers were prepared according to an electrospinning in combination with sol-gel process and subsequently calcination (Panapoy & Ksapabutr, 2008c) with a slight modification. The spinning solutions were first prepared by mixing 12 mL of zirconatrane in crude form and 3 g of poly(vinyl pyrrolidone) with ethyl alcohol. The amount of ethyl

alcohol was varied from 12 to 24 mL and the resulting mixture was continuously stirred for 30 min. The flow rate of the spinning solution was 1.0 mL/h, controlled by syringe pump. The stainless steel capillary tube (inner diameter: 0.394 mm, outer diameter: 0.711 mm) with a tilted angle of 15° at the end was used for the capillary nozzle. The spinning solution was electrospun under ambient air atmosphere at 300°C , as measured by a thermocouple placed on the heating plate. In all of the experiments, the applied voltage was maintained at 20 kV. A grounded metal screen covered by an aluminum foil was used as the counter electrode and was placed 15 cm from the tip of the capillary. As the jet accelerated towards the collector, the solvent evaporated, leaving only ultrathin fibers on the collector. Continuous fibers were deposited for 2 h and collected in the form of fibrous nonwoven mats. Subsequently, calcinations at 500° , 600° and 700°C for 2 h were also carried out for each sample after electrospinning.

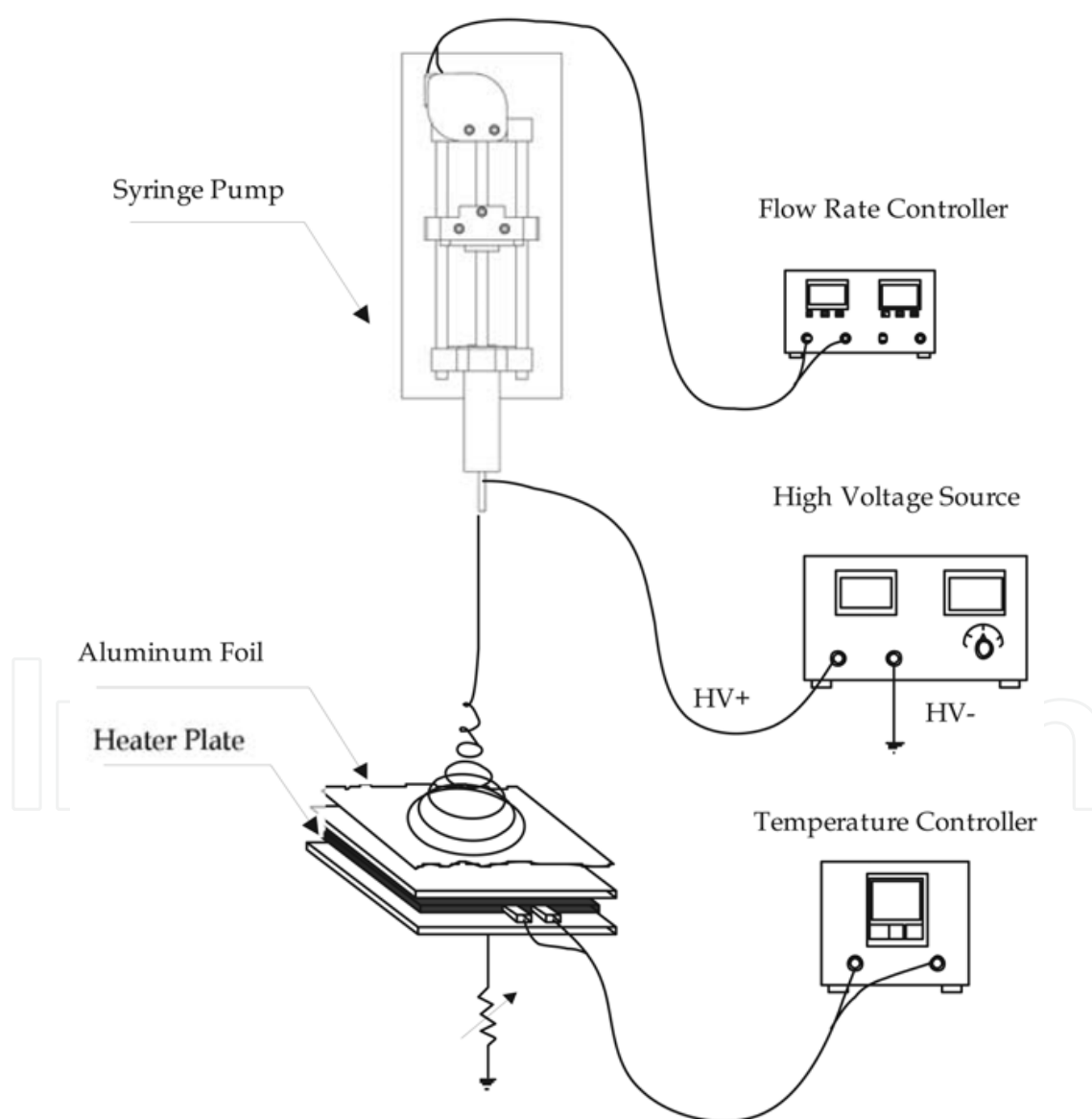


Fig. 2. Schematic illustrating the electrospinning setup used in this work

3.2 Properties of zirconia nanofibers

Morphology and size of the as-electrospun and calcined fibers were observed using a scanning electron microscopy (SEM, Model S3400N, Hitachi). Crystalline phase of the fibers was identified by an automated Rigaku D/Max 2000HV diffractometer with a CuK α radiation. Fourier-transform infrared analysis was measured on a Bruker Optik Vertex70 spectrophotometer with a resolution of 4 cm⁻¹. The solid samples were prepared by mixing 1% of samples with anhydrous potassium bromide, while the spinning solution was analyzed using a Zn-Se window cell. Characterization of the thermal behavior of the spinning solution was achieved by thermogravimetric analysis (Perkin Elmer, TGA7) with a ramp rate of 5°C/min in an air atmosphere. Fig. 3 shows thermogravimetric and derivative thermogravimetric (TG/DTG) curves of the spinning solution. The results revealed weight losses below 340°C, which corresponded to the evaporation of ethyl alcohol, ethylene glycol and some thermal decomposition of the poly(vinyl pyrrolidone) in the spinning solution. While the weight loss at approximately 550°C is probably due to decomposition of residual organic group such as 2-pyrrolidone group resulting from poly(vinyl pyrrolidone).

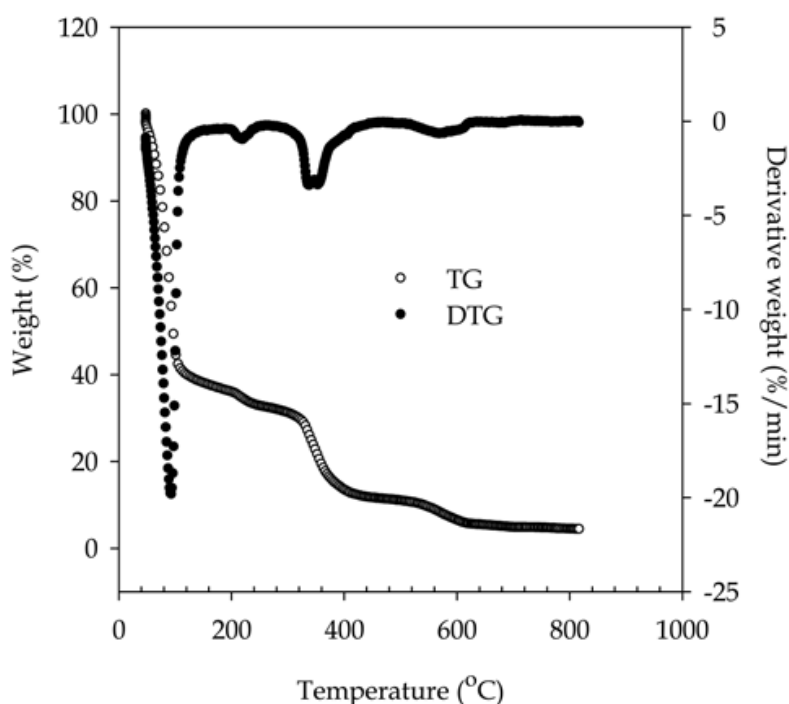


Fig. 3. Thermogravimetric and derivative thermogravimetric curves of the spinning solution

The sizes of as-spun and calcined fibers are compared in Table 3. The effect of ethyl alcohol content of the spinning solutions on morphology of the as-spun fibers is shown in Fig. 4. Obviously, ultrathin fibers with an average diameter of 359 nm were prepared from the spinning solution with an ethyl alcohol content of 24 mL. Increasing amount of ethyl alcohol led to a decrease in the fiber diameter. By viscosity measurement, it was found that the viscosity of the spinning solutions with the amount of ethyl alcohol of 12, 18 and 24 mL are 9.7, 7.4 and 4.3 mPa.s, respectively. The decrease in the fiber diameters with increasing ethyl alcohol content is likely a result of the decrease in the viscosity of the spinning solutions. A similar result was also reported by Jing et al. (2005) even though a different precursor, i.e., zirconium oxychloride, was used as the zirconia source.

Ethyl alcohol content in spinning solution (mL)	Average diameter of fiber			
	As-spun at 300°C (nm)	Calcination at 500°C (nm)	Calcination at 600°C (nm)	Calcination at 700°C (nm)
12	687 ± 124	234 ± 35	230 ± 25	222 ± 39
18	378 ± 131	195 ± 42	192 ± 19	189 ± 26
24	359 ± 110	124 ± 23	122 ± 15	121 ± 26

Table 3. Average diameter of fibers as a function of alcohol content and processing condition

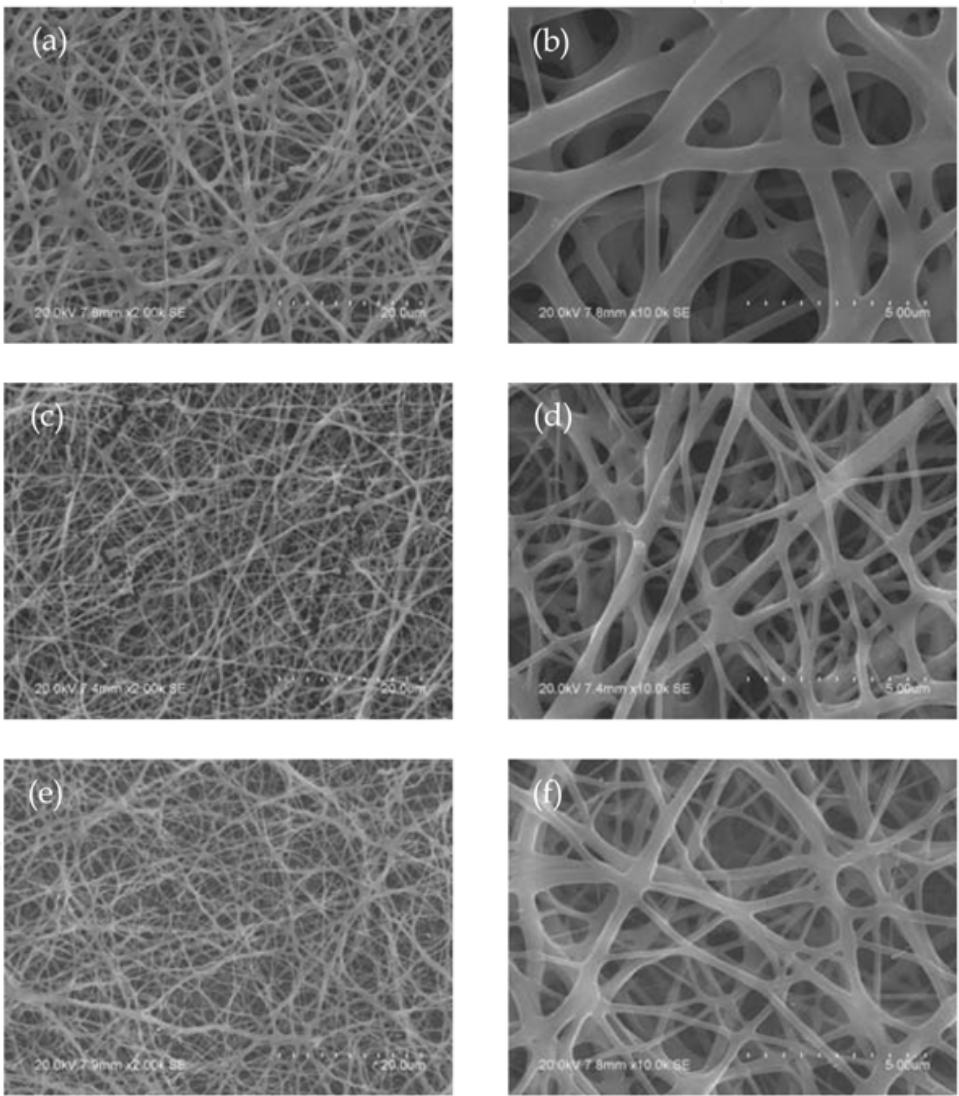


Fig. 4. Scanning electron microscopic images of as-spun zirconia fibers prepared using different amounts of ethyl alcohol (a,b) 12 mL; (c,d) 18 mL; (e,f) 24 mL

Considering the heat treatment process, it was found that the dimension and morphology of the calcined fibers at 500°, 600° and 700°C are different from those of the as-spun fibers at 300°C for all alcohol contents (see Figs. 5, 6 and 7). Moreover, the fiber shrinkage takes place by an increased calcination temperature. The shrinkage is mainly due to the removal of

poly(vinyl pyrrolidone) from as-spun fibers during the calcination process. These results were supported by the thermogravimetric and Fourier-transform infrared analyses. Fig. 8 shows the Fourier-transform infrared spectra of the spinning solution and the prepared fibers. The band in the range of $950\text{--}2000\text{ cm}^{-1}$ is attributed to the bending and stretching vibrations of poly(vinyl pyrrolidone). Accompanying with the disappearance of these adsorptions, the vibration of Zr-O-Zr at 600 cm^{-1} gradually increased with calcination temperature. Furthermore, the scanning electron microscopic images (Fig. 5) reveal that nanothorns protrude from the nanofibers after heat treatment of as-spun fibers at 500°C , in particular as the amount of ethyl alcohol decreases. The formation of zirconia nuclei may be a driving force for the formation of thorn-like nanocrystals on the zirconia nanofibers upon heat treatment process. With increasing calcination temperature from 500° to 700°C , it was noted that the thorn-like nanostructure on the nanofibers converted to flower-like architecture, probably due to the nucleation and growth of thorn-like nanocrystals.

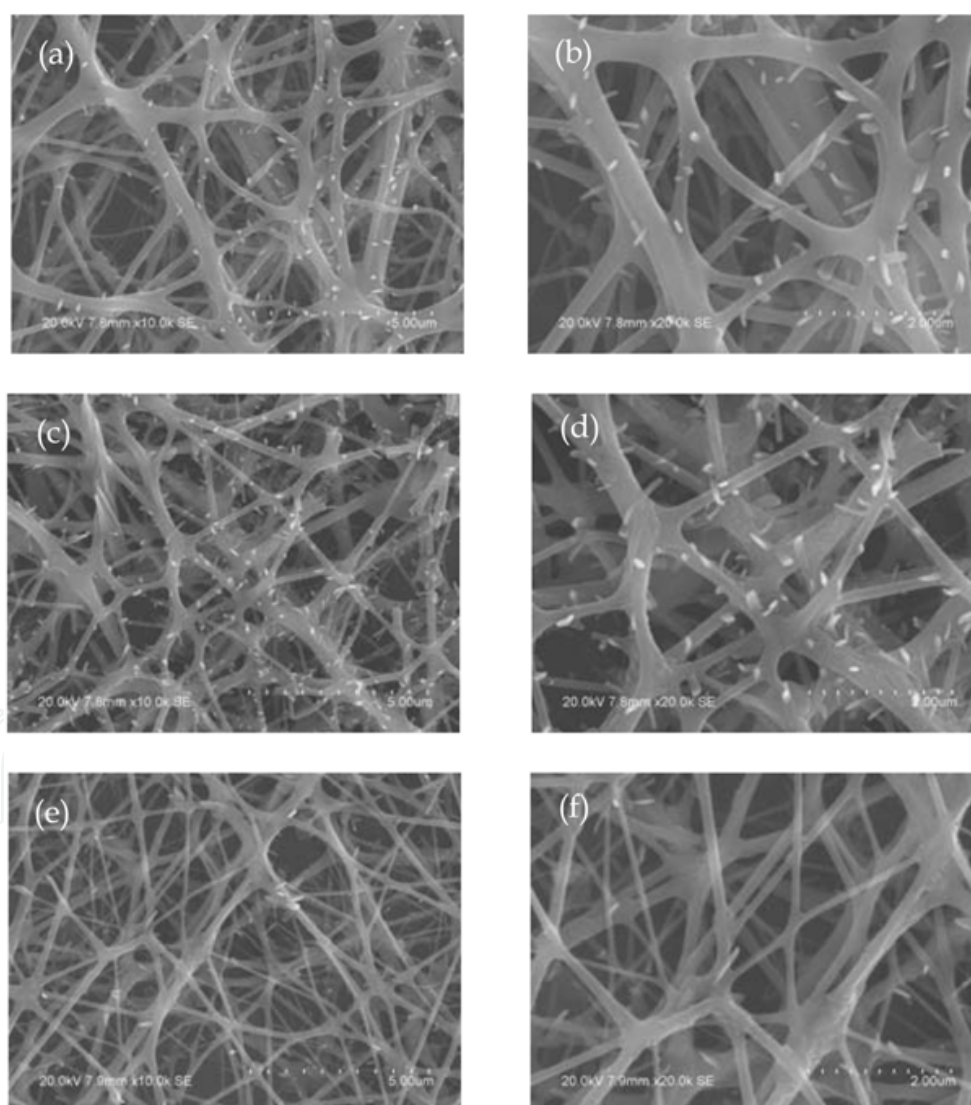


Fig. 5. Scanning electron microscopic images of zirconia fibers calcined at 500°C using different amounts of ethyl alcohol (a,b) 12 mL; (c,d) 18 mL; (e,f) 24 mL

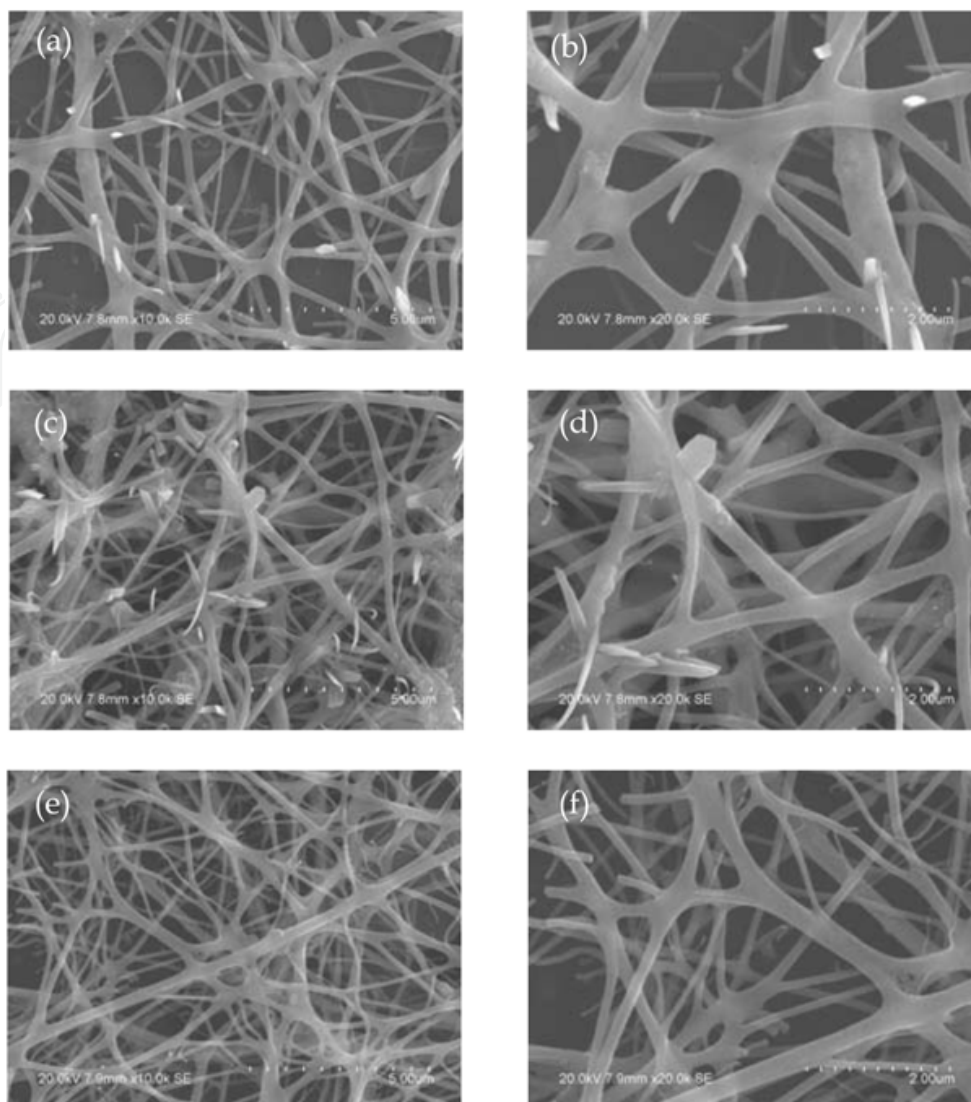


Fig. 6. Scanning electron microscopic images of zirconia fibers calcined at 600°C using different amounts of ethyl alcohol (a,b) 12 mL; (c,d) 18 mL; (e,f) 24 mL

This was supported by the X-ray diffraction analysis (see Fig. 9) that samples have a tendency to gradually crystallize upon further heat treatment. The crystallinity increased with increasing calcination temperature. Meanwhile, it can be seen that the average crystallite size of the calcined fibers at 500°C increased from 3.4 nm to 8.9 and 15.6 nm after heat treatment at 600° and 700°C, respectively. The major peaks and their intensities corresponding to the reflection lines of the tetragonal phase of zirconia are well matched with database in JCPDS (ICDD Card No. 17-923).

From knowledge of oxide one-pot synthesis process, zirconatranne can function as a zirconium alkoxide compound due to its higher stability and increased steric hindrance. Moreover, zirconatranne can be used successfully for the fabrication of one-dimensional zirconia nanofibers with controllable nanostructures. To have a clear understanding of the detailed mechanism of the thorn and flower-like structures, more in-depth studies are in progress. Moreover, the synthetic system providing an appropriate crystal growth environment for the formation of such a novel architecture will be studied and reported in detail later.

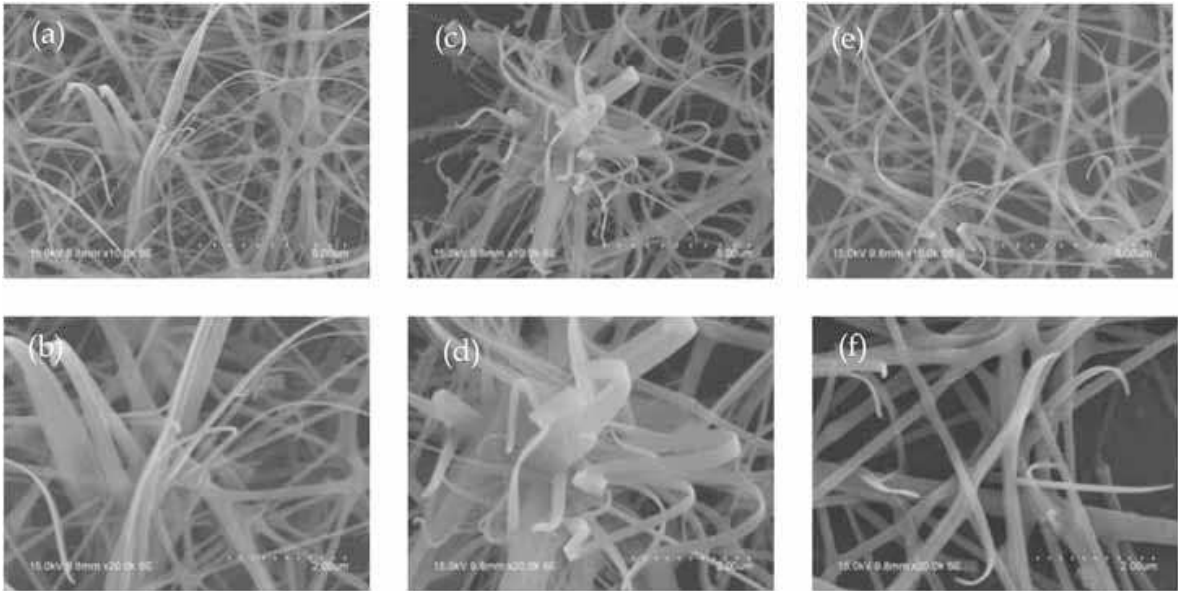


Fig. 7. Scanning electron microscopic images of zirconia fibers calcined at 700°C using different amounts of ethyl alcohol (a,b) 12 mL; (c,d) 18 mL; (e,f) 24 mL

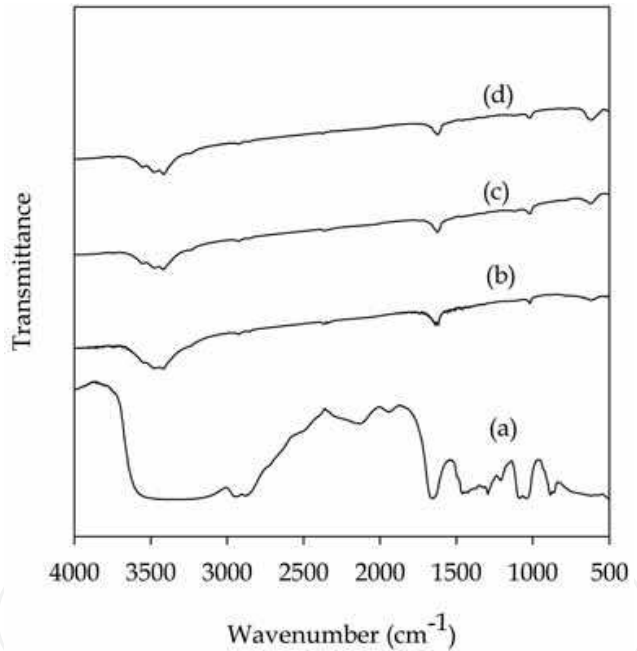


Fig. 8. Fourier-transform infrared spectra of (a) spinning solution, and zirconia fibers calcined at (b) 500°C; (c) 600°C; (d) 700°C using the ethyl alcohol content of 18 mL

4. Conclusion

Morphologically controlled synthesis of zirconia nanofibers prepared using an inexpensive and moisture-stable zirconatran compound, was achieved by a combined sol-gel processing and electrospinning technique with subsequent heat treatment process. The diameters of the zirconia fibers decreased with an increase in the ethyl alcohol content of the spinning solution. The average diameter of theas-spun fibers (based on the amount of ethyl alcohol of 24 mL) decreased from 359 nm at a spinning temperature of 300°C to 121 nm at a

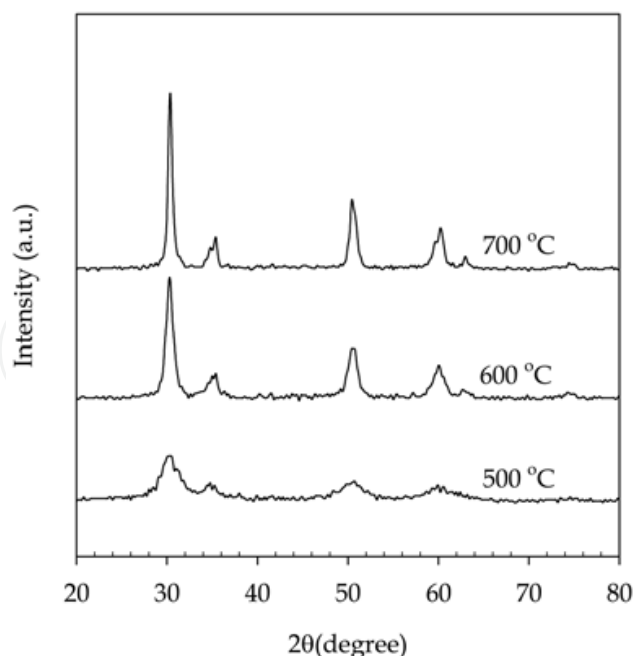


Fig. 9. X-ray diffraction patterns of zirconia fibers calcined at various calcination temperatures using the ethyl alcohol content of 18 mL

calcination temperature of 700°C. A morphological transition from thorn to flower-like architecture on zirconia nanofibers was observed upon increasing calcination temperature from 500° to 700°C. Thorn-like nanostructure on nanofibers prefers to form for lower heat-treated temperature and a smaller amount of ethyl alcohol, whereas flower-like nanostructure prefers to form for higher heat-treated temperature and a smaller amount of ethyl alcohol. The obvious crystal growth appears from 500° to 700°C, and the average size of the crystals in final fibers ranges between 3.4 and 15.6 nm. This present study provides a simple process to fabricate zirconia nanofibers with controllable architecture features, and could be applied to the fabrication of other metal oxide nanofibers.

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Nanofibers

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“There's Plenty of Room at the Bottom” this was the title of the lecture Prof. Richard Feynman delivered at California Institute of Technology on December 29, 1959 at the American Physical Society meeting. He considered the possibility to manipulate matter on an atomic scale. Indeed, the design and controllable synthesis of nanomaterials have attracted much attention because of their distinctive geometries and novel physical and chemical properties. For the last two decades nano-scaled materials in the form of nanofibers, nanoparticles, nanotubes, nanoclays, nanorods, nanodisks, nanoribbons, nanowhiskers etc. have been investigated with increased interest due to their enormous advantages, such as large surface area and active surface sites. Among all nanostructures, nanofibers have attracted tremendous interest in nanotechnology and biomedical engineering owing to the ease of controllable production processes, low pore size and superior mechanical properties for a range of applications in diverse areas such as catalysis, sensors, medicine, pharmacy, drug delivery, tissue engineering, filtration, textile, adhesive, aerospace, capacitors, transistors, battery separators, energy storage, fuel cells, information technology, photonic structures and flat panel displays, just to mention a few. Nanofibers are continuous filaments of generally less than about 1000 nm diameters. Nanofibers of a variety of cellulose and non-cellulose based materials can be produced by a variety of techniques such as phase separation, self assembly, drawing, melt fibrillation, template synthesis, electro-spinning, and solution spinning. They reduce the handling problems mostly associated with the nanoparticles. Nanoparticles can agglomerate and form clusters, whereas nanofibers form a mesh that stays intact even after regeneration. The present book is a result of contributions of experts from international scientific community working in different areas and types of nanofibers. The book thoroughly covers latest topics on different varieties of nanofibers. It provides an up-to-date insightful coverage to the synthesis, characterization, functional properties and potential device applications of nanofibers in specialized areas. We hope that this book will prove to be timely and thought provoking and will serve as a valuable reference for researchers working in different areas of nanofibers. Special thanks goes to the authors for their valuable contributions.

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