

Full Length Research Paper

Effect of production parameters on the structure and morphology of aluminum titanate nanofibers produced using electrospinning technique

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Accepted 11 November, 2010

Al₂O₃.TiO₂ nanofibers have been successfully produced by electrospinning method using a solution containing alumina sol, titania sol and poly [vinyl pyrrolidone] [PVP]. The effect of applied voltage, flow rate and distance between needle and collector were investigated. Aluminum titanate fiber was produced by using electrospinning technique for the first time. The effect of some parameters on the electrospinning process and morphology of the fibers obtained were investigated. Results of observations made by scanning electron microscopy and X-ray diffraction were interpreted and compared with the literature data available on the electrospinning behavior of other materials.

Key words: Nanomaterials, fibre technology, electrospinning, ceramics, aluminum titanate, sol-gel preparation.

INTRODUCTION

Today, ceramic, polymer, composite nanofibers and nanotubes are the most charming materials for nanotechnology. Due to their small characteristic dimension, high surface area, and micro structural features, they provide unique mechanical, optical, electronic, magnetic, and chemical properties for a wide variety of materials and applications (Tanriverdi, 2006).

Electrospinning is a term used to describe a class of fiber forming processes during which electrostatic forces are employed to control the production of fibers (Rutledge and Fridrikh, 2007). Electrospinning is a novel and efficient fabrication process that can be utilized to assemble fibrous polymer mats composed of fiber diameters ranging from several microns down to fibers with diameter lower than 100 nm (Tekmen et al., 2008).

Nanofibers are the ultra-fine solid fibers notable for their minute diameters, their large surface areas per unit mass and small pore sizes. Due to the inherent properties of electrospinning process, which can control

the deposition of polymer fibers onto a target substrate, nanofibers with complex and seamless three-dimensional shapes can be obtained (Frenot and Chonakis, 2003). The morphologies and properties of nanofibers depend on the properties of the materials and the electrospinning parameters, including the solution concentration, viscosity, applied electric field strength, and tip-to-collector distance (Lee et al., 2007).

Sol-gel processing

Sol-gel is a low temperature, chemical solution method that can be used to produce ceramics and glass with better purity and homogeneity than those obtained by using the high temperature conventional process (Ergin, 1997). Sol-gel processes have long been used for the powderless processing of glasses and ceramics. This process uses inorganic metal salts or organo-metallic precursors such as metal alkoxides. The most commonly preferred precursors are metal alkoxides [M(OR)_n], where M is a metal, R is an alkyl group such as methyl (CH₃), ethyl (C₂H₅) or propyl (C₃H₇), and n = the valence of the metal atom. The transition from sol to gel requires two

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Figure 1. Experimental setup.

reaction steps: Hydrolysis and condensation of metal alkoxides (Tanrıverdi, 2006).

In this study, aluminum titanate/PVP solution was prepared using the sol-gel method. Poly vinyl pyrrolidone (PVP) was added into this solution to increase the viscosity for electrospinning. Afterwards aluminum titanate/PVP solution was electrospun into fibers. The products were characterized by scanning electron microscopy (SEM) and X-ray diffractometry (XRD) techniques.

MATERIALS AND METHOD

The Al_2TiO_5 nanofibers were prepared as follows. Based on conventional sol-gel process, aluminum tri sec-butoxide and titanium isopropoxide were used as starting materials. To achieve stoichiometric Al_2TiO_5 , 1:1 molar ratio was maintained. First, $\text{C}_{12}\text{H}_{27}\text{AlO}_3$ (Merck) was dissolved in hot water and acetic acid and was stirred for 1 h. Prior to this, $[(\text{CH}_3)_2\text{CHO}]_4\text{Ti}$ (Merck) was dissolved in ethanol and acetic acid and was stirred for 1 h. Following the dissolution of all the compounds, the precursor solutions were mixed. Afterwards, a solution consisting of poly vinyl pyrrolidone (PVP, $M_w=1,300,000$) dissolved in ethanol was added into the solution. After being stirred at room temperature for 2 h, the mixture was loaded into a plastic syringe.

A positive terminal was connected to the syringe needle tip while aluminum foil covered collector worked as a counter electrode (Figure 1). Al_2TiO_5 nanofibers were synthesized by applying between 5 to 18 kV to the solution through the needle tip.

The distance between syringe needle tip and collector varied between 4 to 10 cm. Aluminum titanate/PVP nanofibers were dried for 6 h at 25°C , then sintered in high-temperature furnace for about 2 h at 1200°C under atmospheric conditions. After heat treatment, volatile and organic groups were removed and pure aluminum titanate nanofibers obtained.

The morphology and average fiber diameter of nanofibers was characterized by scanning electron microscope (LEO 1430 VP). The X-ray diffraction (XRD) measurements were performed for crystal phase identification (XRD 6000-SHIMADZU) with $\text{CuK}\alpha$ radiation.

RESULT AND DISCUSSION

During electrospinning, some problems such as high fiber diameter, discontinuity of the spinning process, bead formation on the fibers, and surface defects were encountered. Moreover, beaded thick fibers, and some interesting branched structures were observed.

First, Aluminum titanate/PVP composites were firstly prepared at 5 kV electric voltages, with varying tip-to-target distances. From their SEM images, fibers could not be formed efficiently. Moreover, beaded thick fibers, and spherical particles were observed. At this voltage, it was found that there is a greater tendency for bead formation.

A crucial element in electrospinning is the application of a high voltage to the solution. The high voltage will induce the necessary charges on the solution and together with the external electric field, will initiate the electrospinning process when the electrostatic force in the solution overcomes the surface tension of the solution. When the applied voltage was increased, the slow formation of fibers can be easily observed.

Figure 2 gives the XRD patterns of Al_2TiO_5 powders heat treated at 1200°C . The crystalline peaks indicate the crystal structure of Al_2TiO_5 .

During experimental studies, it was difficult to maintain stable solution properties and electrospinning conditions.

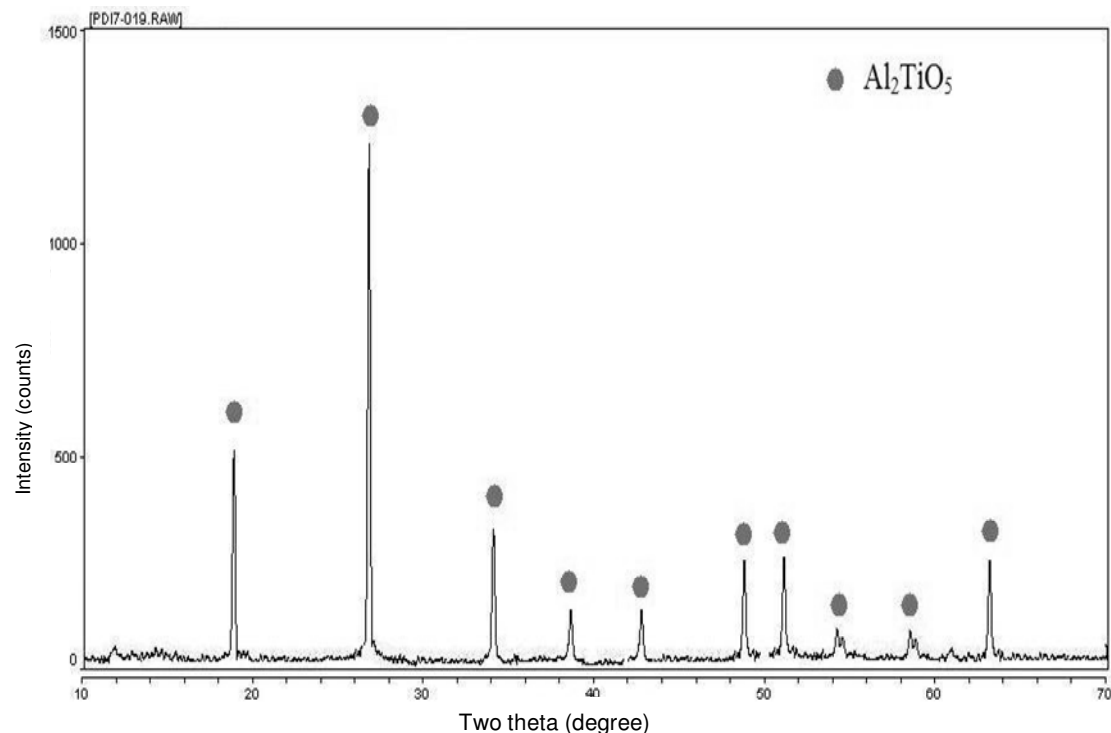


Figure 2. XRD results of the prepared Al₂TiO₅ powders.

Thus, some parametric considerations were applied to obtain favorable nanofibers. These were (1) flow rate, (2) varying applied electric voltage, and (3) tip-to-collector distance.

While voltage was kept constant (16 kV) and distance between syringe needle tip and collector was kept constant (4 cm) as the flow rate was increased from 0.4 to 0.8 ml/h, average diameters of fibers increased from 106.4 to 235.2 nm. The most appropriate result of 0.5 ml/h for flow rate was achieved. Further studies were carried out using this value for flow rate.

At the flow rate (0.5 ml/h) and voltage (16 kV), average diameters of fibers increased from 91.7 to 438.2 nm as distance between syringe needle tip and collector was increased from 4 to 10 cm.

At constant flow rate (0.5 ml/h) and constant syringe needle tip and collector distance (10 cm), the average diameters of fibers decreased from 1208.9 to 468.3 nm as shown Figure 3 as voltage increased from 12 to 18 kV. These results were in accordance with the reference mentioned as "Greater columbic forces in the jet as well as stronger electric field. These have the effect of

reducing the diameter of the fibers (Lee et al., 2004).

In conclusion, aluminum titanate/PVP fibers were prepared by the sol-gel method and electrospinning technique for the first time in this study. This experiment showed that solution flow rate, supplied voltage, and distance had significant effects on fiber diameter and fiber uniformity. It had been shown that all of these parameters can be controlled by changing the electrospinning conditions. In order to achieve the optimum average fiber diameter (91 nm), 4 cm distance, 16 kv

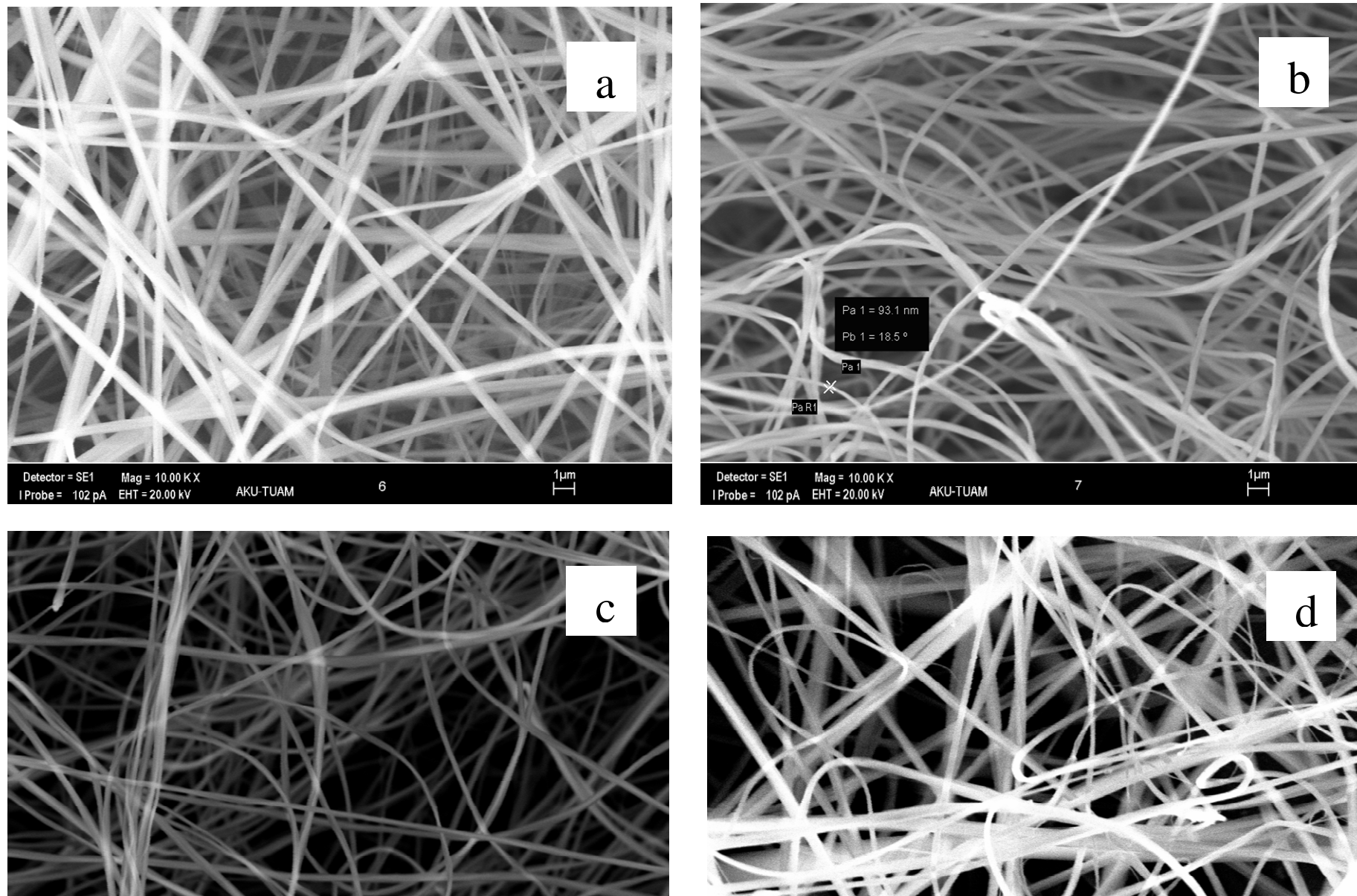


Figure 3. SEM photos showing the effect of voltage on the average diameters of fibers (flow rate: (0.5 ml/h), distance: (10 cm), a : 12 kV; b : 14 kV; c : 16 kV; d : 18 kV.

applied voltage and 0.5 ml/h flow rate were the required conditions.

ACKNOWLEDGEMENT

This work was supported by the Afyon Kocatepe University in the scope of the Scientific Research Projects (08.MUH.10-2008).

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