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Synthesis and characterization of PB($ZR_{0.52}$, $TI_{0.48}$)O_3 nanofibers by electrospinning, and dielectric properties of PZT-Resin composite

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ABSTRACT

Lead zirconate titanate (PZT) / Poly vinil pyrolidone (PVP) nanofibers were spun from spinnable solution by electrospinning method. Zirconium n-propoxide, Titanium isopropoxide and Lead acetate trihydrate were solved in 2-Metoxy ethanol to obtain a precursor solution with a suitable viscosity. The obtained mats were calcined at 550, 600, 650, 700 and 800 °C for 2 h. The morphology and crystalline phase of PZT nanofibers were characterized using X-ray diffraction (XRD) and Scanning electron microscopy (SEM). XRD spectra indicated the formation of perovskite Pb($Zr_{0.52}$, $Ti_{0.48}$)O_3 phase at 600 °C. Fibers had diameters between 50 to 500 nm by controlling of the process parameters. Aging time and PVP percentage were found to have a significant influence on the nanofibers morphology. Increasing PVP and aging lead to increased viscosity and a correlated increase in green fiber diameter. The dielectric constant of PZT nanofibers were measured at 1 kHz.

1. Introduction

Lead Zirconate Titanate (PZT) is widely used as ferroelectric materials in sensor, actuators, structural systems, non-volatile ferroelectric memory devices, micro-electromechanical systems (MEMS), because of their ability to transform energy from electrical to mechanical and vice versa [1–3]. Fibrous PZT has potential for utilization in high performance hydrophones and ultrasonic transducer applications [4]. Thin-film PZT and powders have been synthesis through metallo-organic decomposition (MOD) [5,6], coprecipitation [7], sol–gel [8], hydrothermal reaction [9], reactive calcinations [10], ball milling [11] and others [12,13]. A few methods including hydrothermal synthesis [14], sol electrophoretic deposition [15] and the metallo-organic decomposition (MOD) based on electrospinning method [16–18] have been developed. The electrospinning MOD can produce continuous long fibers with diameter ranging from ten to hundred nanometers [18].

This study aims to synthesize perovskite lead zirconate titanate fibres with ultra-fine dimensions between 50 to 500 nm and to investigate influence aging time and PVP percentage on their viscosity and then fibers morphology (to be uniform and bead-free) and diameter.

2. Experimental

The preparation of PZT sol was carried out as reported earlier [19]. Lead (II) acetate trihydrate ($Pb(CH_3CO_2)_2.3H_2O$, Merck), zirconium

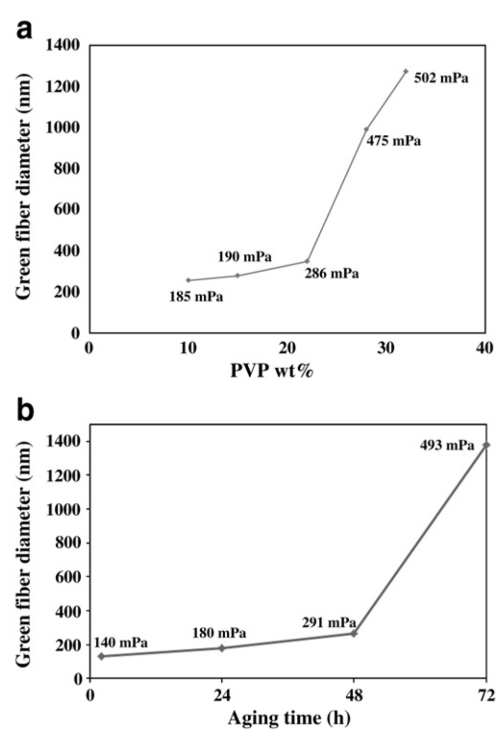

Fig. 1. Variation of the Green fiber diameter and (a) PVP % (b) Aging period, with their viscosities.

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(IV) n-propoxide (70 wt.% solution in 1-propanol, $Zr[OCH(CH_3)_2]_4$, Merck) and titanium (IV) isopropoxide ($Ti[OCH(CH_3)_2]_4$, Merck) were used as starting materials. 2-Methoxy ethanol ($CH_3OCH_2CH_2OH$, Merck) and Acetic acid (CH_3COOH, Merck) was used as a solvent and chelating agent, respectively [19,20]. Pb(II) acetate trihydrate (1.10 mol) was dissolved in 2-methoxy ethanol by heating and stirring at 70 °C. Then, this solution was refluxed for 1 h and then cooled down to 70 °C. The Zr/ Ti solution was prepared by dissolving 0.52 mol of Zr-n-propoxide and 0.48 mol of Ti-isopropoxide in 2-methoxy ethanol and acetic acid. The acetic acid to alkoxide molar ratio was kept at 1:1. Pb and Zr/Ti precursor solutions were then mixed and refluxed at 80 °C for 3 h. Then, water (water/alkoxide = 1) was added and the final refluxing has been done at 80 °C for 6 h.

Poly vinyl pyrrolidone (PVP) polymer (10 to 32 wt.%) solution was prepared in 2-Methoxy ethanol. The viscosity of the PZT/PVP solution was measured using a Brookfield viscometer LVF NDJ4 at 20 °C.

The PZT/PVP solution was put in the 5 ml syringe. The distance between the tip of syringe and the collector, the feeding rate of PZT/PVP solution that was maintained by controlling the syringe pump and the voltage was optimized. These parameters were obtained 12 cm, 0.5 ml/h and 18 kV, respectively.

Green fibers were sintered with a heating rate of 1°C/min. Simultaneous Thermal Analysis (STA 1640) was used to identify the crucial steps in the pyrolysis and sintering processes, from room temperature to 900 C, with a heating rate of 5°C/min. The sintered fibers were analyzed by X-ray diffraction (XRD) using a Philips X-Pert X-

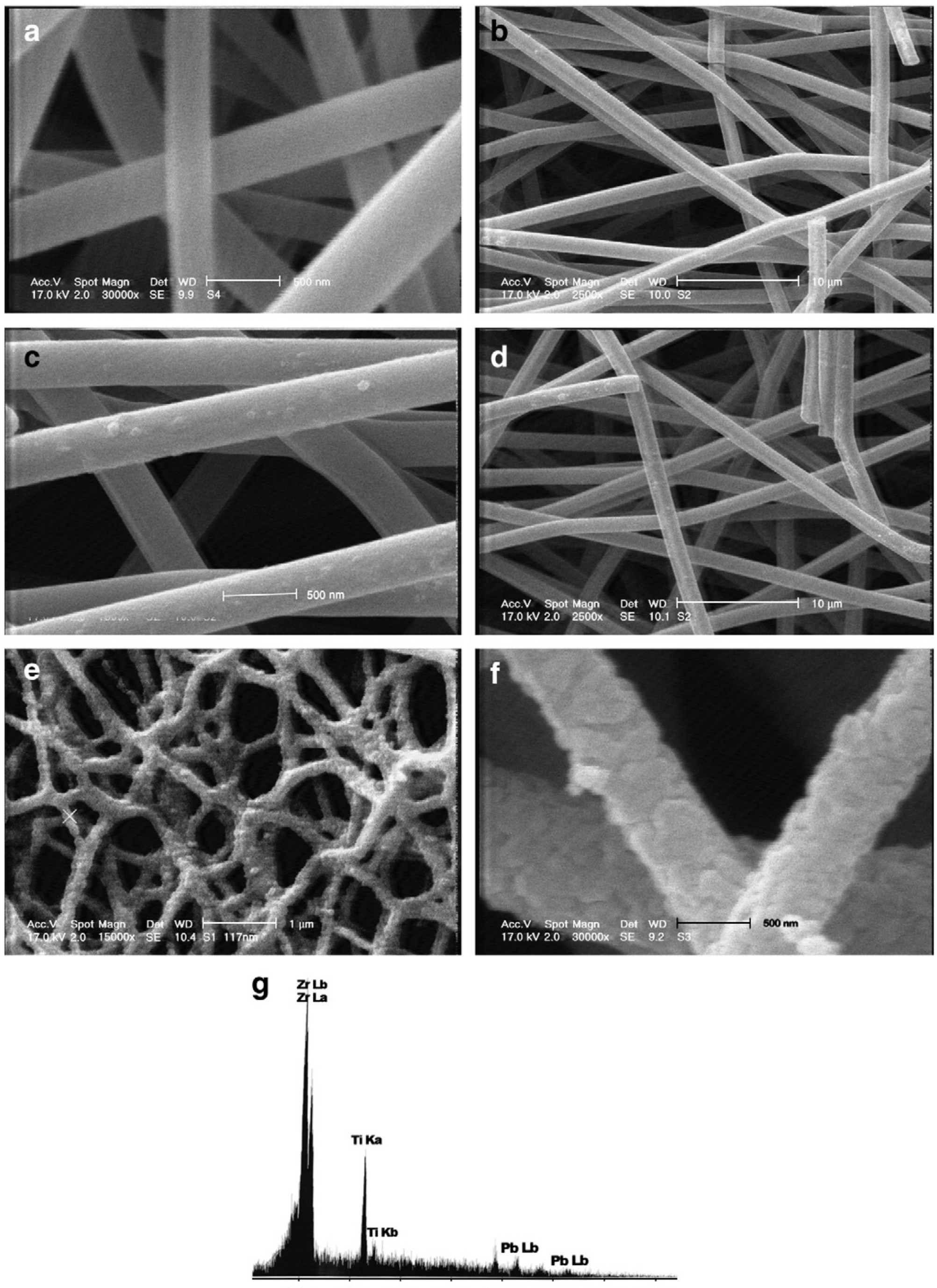

Fig. 2. SEM image (a) PZT green fibers calcined with 22 wt% PVP (b) PZT green fibers with 32 wt% PVP (c) PZT green fibers with 24 h aging time (d) PZT green fibers with 72 h aging time (e) PZT calcined fibers of fig. a. (f) PZT calcined fibers of fig. b. (g) EDS of fig. f.