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Dielectric properties of composite $LaMnO_3$ nanofiber by electrospinning technique

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Abstract: Electrospinning technique has been extensively developed as a simple and vasatile method for drawing nanofibers from polymer solutions. Lanthanum Maganite La_{1-x} Mn_xO_3 (x = 0.02 mol) nanofibers were obtained by calcinations of PVA/$LaMnO_3$ composite at different temperatures with electrospinning utilizing sol-gel precursors. Novel polycrystalline $LaMnO_3$ nanofibers were yielded at 500 °C, 600 °C and 700 °C for 2 h as the final products. Field Emission Scanning Electron Microscopy (FESEM) was employed to study the fiber diameter of samples. The average diameter of the $LaMnO_3$ nanofibers was found to be in the range of 85 nm to 150 nm at different temperatures. The dielectric properties of $LaMnO_3$ nanofibers were identified by C-f, ε_r-f, tan δ - f and σ_{ac}-f characteristics. The results obtained from this research will lead to enable new levels of electronic applications, biomedical applications and protective clothing.

Keywords: $Lamno_3$ Nanofibers, Electrospinning Technique, FESEM, Dielectric Characteristics

1. Introduction

Nanotechnology is the field of science concerning with the synthesis, characterization and application of nanoscale materials. Especially when the size become smaller than 100 nm, quantum effects can begin to dominate the behavior of matter at the nanoscale affecting the optical, electrical and magnet behavior of materials [1-5]. Fiber reinforcement using particles is well known technique in textile and composite industries not only for enhancing the properties but also for synthesizing new materials having unique properties [6-9]. Polymer nanofibers are used in a wide variety of applications from a gel state. [10-12]. Nanofiber composites are concerned with carbon nanofibers or nanotubes reinforcements. These nanofibers or nanotubes are generally not obtained through electrospinning. Several comprehensive reviews have summarized the researches done until very recently on these composites. [13-15]. The sol-gel process is a versatile solution process for making nanofibers, ceramic and glass materials. Nanofibers can be made of Lanthanum Manganite ($LaMnO_3$) by electrospinning technique. One-dimentional nanostructure materials such as nanofibers, have received great interest due to their specific electrical and catalytic properties. [16]. The rapid development of electronic technology has brought about a demand for materials with good physical and mechanical properties. Functional polymer composites have the ability to meet these needs and the use of various conductive fillers, such as metallic, ceramic, carbon black particles, thin films and nanofibers have been extensively explored and shown to improve the conductivity and dielectric properties.

2. Experimental Procedure

2.1. Electrospinning Technique for $LaMnO_3$ Nanofibers

Electrospinning is a technique that can be used to produce nanofibers under the influence of an high electric field. With the production of nanofibers, nanotechnology extends its application to a vast area. Nanofibers can be made of Lanthanum Manganite by electrospinning technique. Local-made electrospinning set-up was firstly constructed. The high voltage applied in this work was 10 kV~30kV. The local-made electrospinning apparatus contained a needle or spinneret, high voltage power supply and a grounded collector. It was known by using high voltage probe that produces maximum voltage 30kV but the operating voltage was 27 kV. A syringe holder and a collector were kept in the cylindrical shape of glass tube, length of 36.3 cm, outside diameter was 9 cm and inside diameter was 8.45 cm. DC voltage generator of positive terminal was connected with hypodermic needle (0.55 mm x 25 mm) and the circular shape of Al collector which was connected by negative terminal of power supply as system ground. In order to create an electric field the system must contain along with the charged needle, a grounded plate. This conductive plate completes a circuit and allows a strong electric field to be created between the needle and the plates. This grounded plate also serves as the collector for the completed nanofiber web that is fabricated during the electrospinning process. The schematic diagram of electrospinning set-up was shown in figure 1.

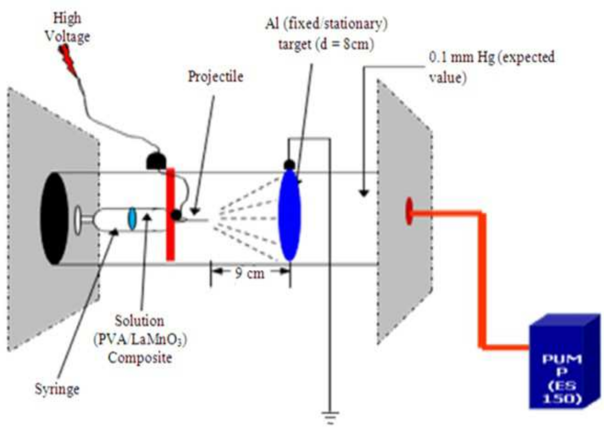

Figure 1. *Schematic diagram of electrospinning Set-up*

2.2. Sample Preparation of $LaMnO_3$ Nanofibers

In this study, Lanthanum Manganite ($LaMnO_3$) and poly vinyl alcohol (PVA) and distilled water were chosen as the starting chemicals and solvent. $LaMnO_3$ nanofibers were formed by calcinating preparation of composite fibers; sol-gel process was used. 0.75 g of $LaMnO_3$ was mixed in 15 g of alcohol (PVA). These mixtures were dissolved in 50 cc of distilled water at room temperature. Then these solutions were stirred vigorously to get the homogeneous precursor sol-gel by magnetic stirrer for 3 h at room temperature. After stirring, the temporary solutions were kept at room temperature for 24 h, a viscous get of PVA/$LaMnO_3$ composite was obtained, and that sol-gel was determined by measuring its viscosity. The result of $LaMnO_3$ sol-gel was to be 1200 cP. The solution gel was expected to be viscous enough for electorspinning. Before setting the vacuum glass-ambient, the experimental set-up was prepared. The glass wall of the vacuum tube was cleaned alcohol (CH_3CH_2OH). PVA/$LaMnO_3$ precursor solution about 20 cc was put into the syringe. The hypodermis syringe needle was connected to the positive terminal of a high DC voltage generator that produce maximum voltage 27 kV and the negative terminal of the power supply was connected to the collector (Al foil) opposite to the syringe needle with a distance about 9 cm. Al-substrate was then struck on the collector. Before supplying the power, glass tube was created as vacuum condition by using vacuum pump and also tested by vacuum tester.

The electrospinning process was taken place in a cylindrical shape glass tube. There are basically three components such as high voltage supplier, capillary tube and a metal collecting screen. In the electrospinning process, a high voltage was applied to create an electrically charged jet of colloidal solution which solidified to leave a fiber. One electrode was placed into the spinning solution and the other attached to a colloidal solution. This induced a charge on the surface of the liquid. In this way a charged jet of liquid was ejected from the tip of the capillary tube. A voltage was applied to the polymer solution which causes a jet of the solution to be drawn toward a grounded collector. The fiber jets dry to form polymeric fibers, which can be collected on a web. The electrospinning process has been documented using a variety of $LaMnO_3$ nanofibers forming polymers. And then, it was heat-treated at 500 °C, 600 °C and 700 °C for 2 h respectively.

3. Results and Discussion

3.1. FESEM Analysis of $LaMnO_3$ Nanofibers

The PVA/$LaMnO_3$ composite fibers on aluminium foil were carried out to examine by FESEM images. In order to study the morphology and nano structural properties of fabricated $LaMnO_3$ nanofibers with different temperature for 20 min was depicted in figure 2 (a-c). These figures showed the FESEM micro-graphs with the respective diameter histograms of the as-prepared and calcined PVA/ $LaMnO_3$ composite nanofibers. The as-spun composite nanofibers appeared quite smooth and each individual nanofiber was quite uniform in cross section. These nanofibers were found in the formation of aligned structure. The diameter of nanofibers calcined at 500 °C was about to be 85 nm in figure 2 (a). After calcination at above 500 °C, the nanofibers remained as continuous structure and their diameter appeared to be increased with increasing calcination temperatures at 600 °C and 700 °C. In contrast, the image of the nanofibers calcined at 600 °C and 700 °C showed in figures 2(b) and 2(c) that each fiber contains small $LaMnO_3$ particles of 120 nm and 150 nm in diameter. According these results, the morphology and size of the fibers were varied strongly with increased of calcination temperatures.

Figure 2(a). *FESEM image of $LaMnO_3$ nanofibers at 500 °C*

The diameter of the fibers increasing and the surface changes rough gradually after sintered at 600 °C and 700 °C for 2 h respectively. The average diameter of the precursor's $LaMnO_3$ nanofibers was found between 85-150 nm. With the calcination temperatures increased gradually fiber diameter was thicker and fiber surface was extremely rough. This is due to that fiber composition at the beginning was composed of small grains and with the rise of temperature small grain grew up to a large grain and grew again toward surround. When the temperature reached 700 °C which observed that the long-grain completely grew together and the diameter of the fibers were increased ~ 150 nm.

Figure 2(b). *FESEM image of $LaMnO_3$ nanofibers at 600 °C*

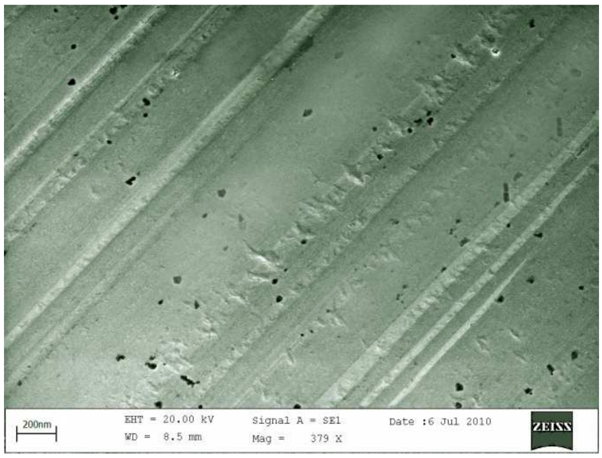

Figure 2(c). *FESEM image of $LaMnO_3$ nanofibers at 700°C*

3.2. Dielectric Characteristics of $LaMnO_3$ Nanofibers

For most applications of electronic materials, the dielectric constant, dielectric loss and AC conductivity are important practical parameters, so studies of their properties dielectric are basically electric insulators which ordinarily do not contain any free charge carriers for conduction. The change in capacitance, dielectric constant, dielectric loss and AC conductivity as a function of applied frequency modes (1 kHz – 100 kHz) at zero bias voltage of $LaMnO_3$ nanofibers by using copper electrodes were investigated. The variation of capacitances, dielectric constant, dielectric loss factors and AC conductivity with frequencies are identified for all different temperatures of $LaMnO_3$ nanofibers.

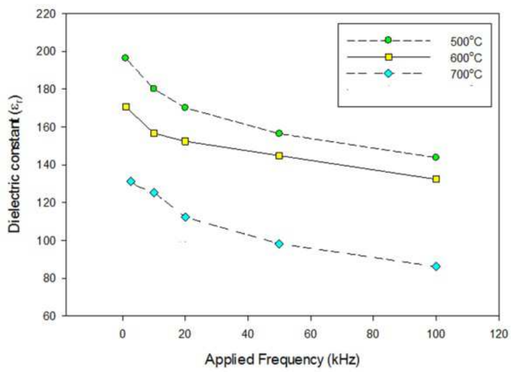

Figure 3(a). *C-f of $LaMnO_3$ nanofibers at different temperatures*

The dielectric constants were calculated from the capacitances measured without a bias voltage it was clearly observed that step-like capacitance decay is crushed over applied frequency. Decreasing dielectric constant is observed with increasing in frequency for Cu electrode. The decrease in dielectric constant arises from the fact that polarization does not occur instantaneously with the application of the electric field as charges have inertia. Dissipation factor (D factor) is a measure of the losses of the capacitor under AC operation. At higher frequencies, tan δ decreases with increasing frequency because of the active component of the current is practically independent of frequency and the relative component increases in proportion to the frequency. But the other fact of getting lower dielectric and higher lose current is that AC conductivity is rising with frequency. From the graphs, it were seen that tan δ value was minimum the fiber at 500 °C and maximum the fiber at 700 °C. The largest value of dielectric constant for $LaMnO_3$ nanofibers was found at 500 °C and the smallest was at 700 °C. $LaMnO_3$ nanofibers were measured C-f, ε_r-f, tan δ-f and σ_{ac}-f variations with different temperatures by LCR analyzer as shown in figure 3 (a-d).

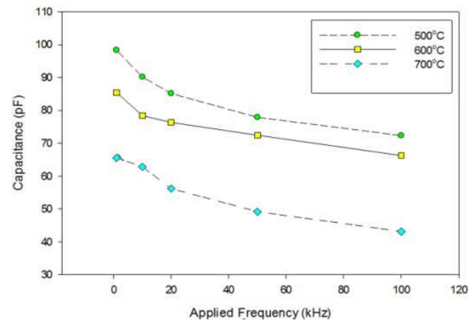

Figure 3(b). *tanδ - f factor of $LaMnO_3$ nanofibers at different temperatures*

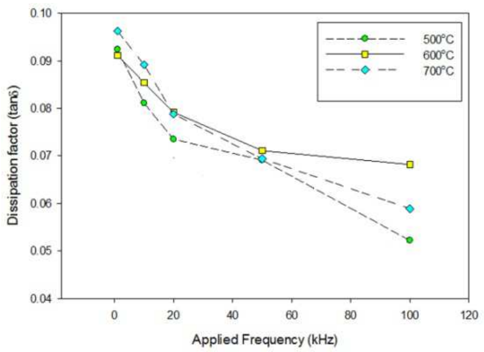

Figure 3(c). *ε_r-f of $LaMnO_3$ nanofibers at different temperatures*

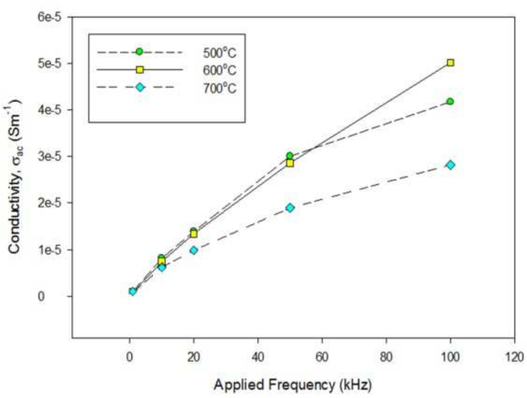

Figure 3(d). *σ_{ac} - f of $LaMnO_3$ nanofibers at different temperatures*

4. Conclusions

Nanofibers of $LaMnO_3$ have been successfully fabricated by using an electrospinning and sol-gel technique. The electrospinning process was taken place in the cylindrical shape of glass tube that had very limited exposure to external conditions to the tube. Polycrystalline $LaMnO_3$ nanofibers (diameter of 85-150 nm) as confirmed by FESEM were formed after calcinations of the as-spun PVA/$LaMnO_3$ composite nanofibers in at 500 °C, 600 °C and 700 °C for 2 h respectively. According to FESEM images, with the temperatures of calcination increased gradually, fiber diameter was thicker and fiber surface was extremely rough. Therefore the crystal structure and morphology of the nanofibers were influenced by the calcination temperatures. The variation of capacitance, dielectric constant, dielectric loss and AC conductivity as a function of applied frequency for $LaMnO_3$ nanofibers were successfully investigated at zero bias conditions. According to the results obtained, it was seen that dielectric loss values were minimum for the fiber at 500 °C while the maximum value of the fiber was occurred at 700 °C. The convenience of producing nanofibers should be useful new functional opportunities with the advantage of low cost, biomedical applications and nano-composites.

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