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Research Article

Growth and Characterization of Electrospun $LaMnO_3$ Nanofibers by Electrospinning Technique

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Abstract

The polycrystalline perovskite structure of $LaMnO_3$ nanofibers was obtained by calcination of the PVA/[$LaCl_3$+$MnCl_2$+$(NH_4)_2CO_3$] composite at 600°C with electrospinning technique. The decomposition and crystalline behavior of sample were examined by Thermogravimetric and Simultaneous Differential Thermal Analysis (TG-DTA). The crystal structure and phase formation were characterized by X-ray Diffraction (XRD). Scanning Electron Microscopy (SEM) was performed and the diameters of the $LaMnO_3$ nanofibers were calculated to be 42 nm and 55 nm with different spinning intervals at 600°C. SEM analysis was also carried out to examine the fiber diameters and morphological properties.

Keywords: Electrospinning machine; $LaMnO_3$ nanofibers; TG-DTA; XRD; SEM

1. Introduction

Nanotechnology is the experimental process of manipulating matter on an incredibly minute scale in order to create new products and materials or delivery systems. Nanofibers are an exciting new class of material used for value-added applications such as medical, filtration, barrier, personal care, composite, garments, insulation and energy storage (Subbiaii, 2004). Electrospinning makes it relatively easy to spin continuous nanofibers from many different polymers. The electrospinning process is driven by electrical forces on free charges on the surface or inside a polymeric liquid (Li and Xia, 2004). Mechanical formation of polymer crystals often produces fibers that can be observed in electron micrographs of feature surfaces; for example, such fibers typically have diameters of a few tens of nanometers and lengths up to a few micrometers (Sautter, 2005). Many images of polymer nanofibers exist in the polymer morphology literature, but in almost all cases the nanofibers were observed incidentally to other features of the polymer (Geil, 1963; Reneker and Chun, 1996). The sol-gel process is a versatile solution process for making nanofibers, ceramic and

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glass materials. Nanofibers can be made of Lanthanum Manganite ($LaMnO_3$) using the electrospinning technique. One-dimensional nanostructure materials such as nanofibers have received great interest due to their high surface area to mass or volume ratios. $LaMnO_3$ has attracted much interest recently due to its specific electrical and catalytic properties (Jinxian et al., 2009; Dong et al., 1994). Jinxian et al, using starting chemicals of PVA, found that its fiber diameter was 150 nm by PVA/[$La(NO_3)_3$+$Mn(CH_3COO)_2$] (Jinxian et al., 2009). In this paper, the fiber diameter was measured to be about 42 nm and 55 nm from the starting chemicals of PVA/[$LaCl_3$+$MnCl_2$+ $(NH_4)_2CO_3$].

2. Experimental Procedure

2.1 Sample Preparation

The starting chemicals used in this study were lanthanum chloride, manganese chloride and ammonium carbonate of reagent grade. First of all, 0.82 g of $LaMnO_3$ was dissolved in 10 g of Polyvinyl Alcohol (PVA). The mixture was also dissolved in deionized water. Then it was stirred using a magnetic stirrer at constant speed for 3 h to form a homogeneous solution and aged for 24 h. Next, it was placed in a hypodermic syringe. The high voltage was set at 26 kV and the distance between syringe and Al-target was set to 9 cm. Fig 1 showed the block diagram of preparation for $LaMnO_3$ nanofibers.

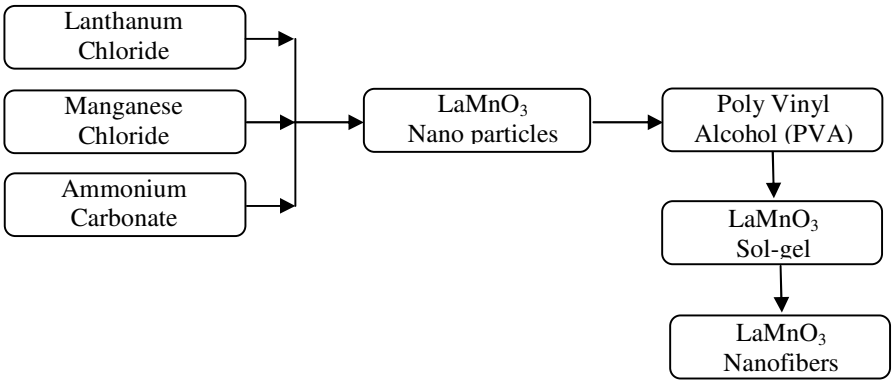

Fig 1. Block diagram of preparation for $LaMnO_3$ nanofibers

2.2 Electrospinning Setup

The home-made electrospinning device consisted of a spinneret, HV power supply and a ground collector. The HV supply was transferred from 21'' fly pad (219×6M, Toshiba) indirectly. The 60 resistors (10 MΩ) were connected in a series arrangement to produce a high voltage range from 10 kV to 30 kV. A spinneret (syringe) projectile holder and collector were kept in the glass tube of cylindrical shape. The positive terminal of the DC source was connected with a hypodermic needle (0.55 × 25 mm) while the Al-substrate (collector) (target) was connected with the negative terminal to ground it. The $LaMnO_3$ polymer solution was directed towards the Al-target when the HV was applied. The spinning or running time interval was set to 15 min and 20 min. In this way, a $LaMnO_3$ fiber web was formed on Al-substrate. After operating and annealing in oxygen, atmospheric ambient was performed at 600°C for 1 h. The schematic diagram of the

electrospinning setup is shown in Fig 2(a). Fig 2(b) shows the illustration of the electrospinning setup.

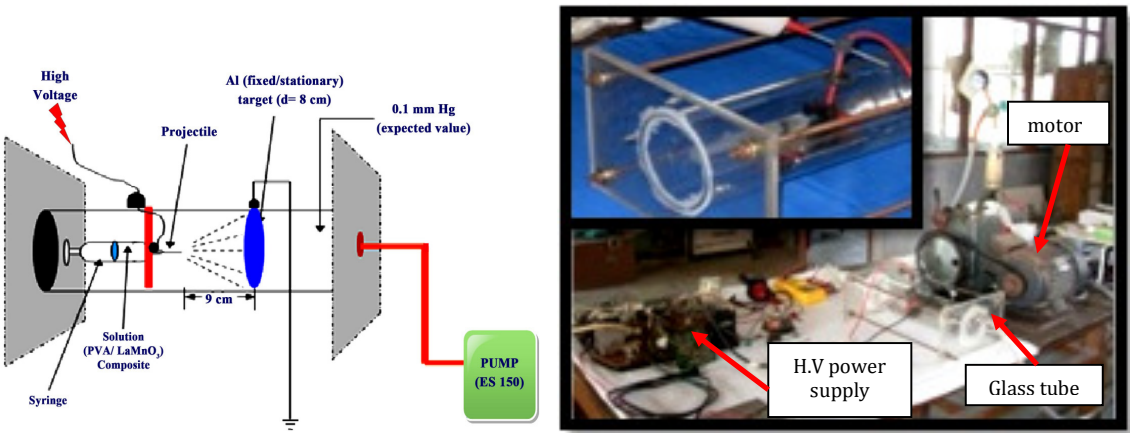

Fig 2 (a) Schematic diagram of electrospinning setup **(b)** home-made electrospinning setup

Table 1 Operating parameters of $LaMnO_3$ sample

Polymer solution	$LaMnO_3$	Working voltage power supply (DC)	~26 kV
Syringe capacity	20 cc	Running time	15 min, 20 min
Electrode spacing	9 cm	Cooling time	3 h
Capillary diameter	0.65 mm	Annealing temperature	600°C
High voltage power supply (DC)	~30 kV	Annealing time	1 h

3. Results and Discussion

3.1 TG-DTA Analysis

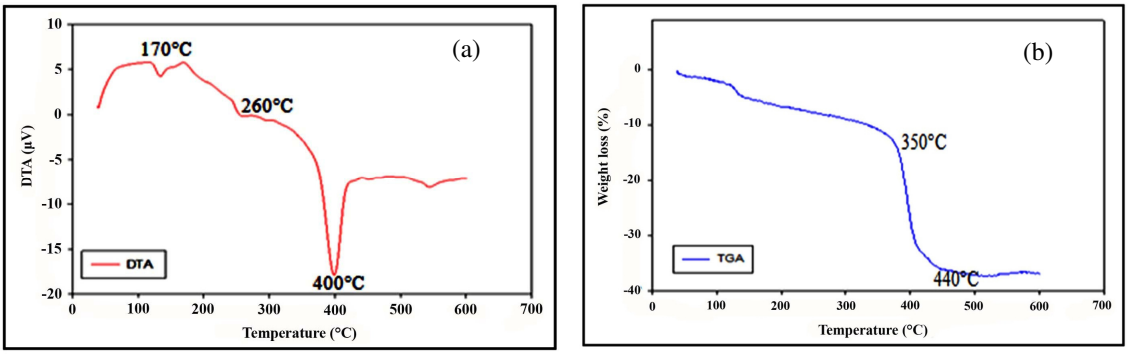

Fig 3(a). DTA analysis **(b)** TGA analysis of $LaMnO_3$ composite

TGA and DTA curves of $LaMnO_3$ sample are displayed in Fig 3 (a-b). From the Figure, it was observed that the DTA curve shows four distinct endothermic peaks. The first peak at 170°C shows

the loss of moisture from the $LaMnO_3$ composite. From the TGA curve, it was significant that three different states were formed. The final weight loss was completed at 400°C. Thus, the exothermic DTA peaks were associated with the TGA weight loss and indicated organic combustion steps. The exo peak was also formed from the crystalline process. The combined TGA and DTA data indicated that slow heating resulted in transformation of the colloidal solution to crystalline $LaMnO_3$ at about 400°C.

3.2 X-ray Diffraction Analysis

The XRD spectrum of $LaMnO_3$ fiber at 600°C with spinning times of 15 min and 20 min were displayed in Fig. 4. The upper site of the XRD spectrum indicated the observed profile while the lower site gave the standard (reference) $LaMnO_3$ (JCPDS library file # 87-20-12>$LaMnO_3$). Some peaks of observed spectrum were well-matched with those of standard $LaMnO_3$ while some extra peaks were unidentified. Thus, the $LaMnO_3$ fiber was partially formed on Al-substrate at 600°C.

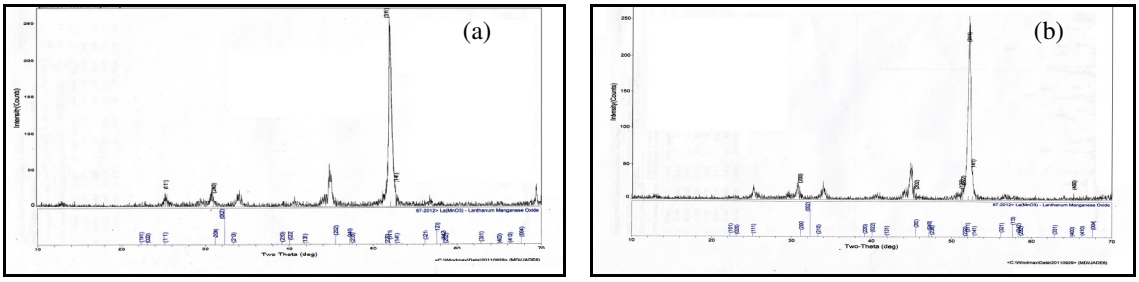

Fig 4. XRD Patterns of $LaMnO_3$ fibers at 600°C (a) 15 min (b) 20 min

3.3 SEM Analysis

FESEM investigation was carried out to examine the morphology and size of electrospun $LaMnO_3$ fibers at 600°C for different time-intervals. They were displayed at Fig 5 (a-b). The fiber diameters were calculated to be 42 nm and 55 nm for spinning time-intervals of 15 min and 20 min.

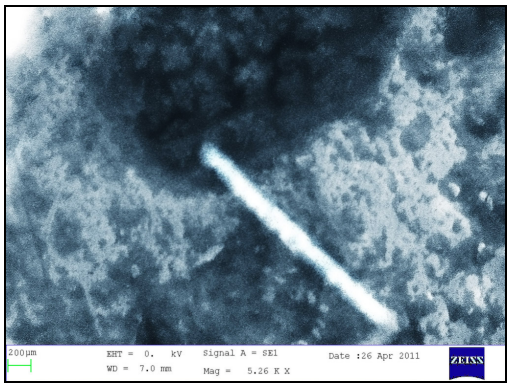

Fig 5(a). SEM image of $LaMnO_3$ fiber at 600°C (Spinning time 15 min)

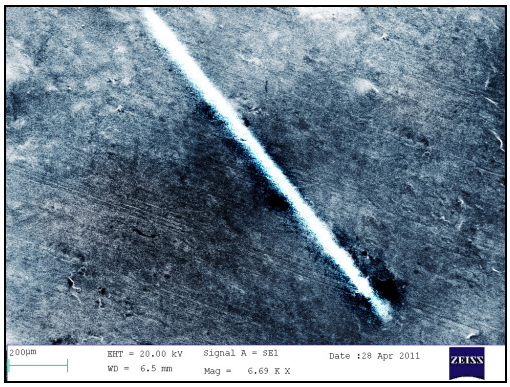

Fig 5(b). SEM image of $LaMnO_3$ fiber at 600°C (Spinning time 20 min)

4. Conclusion

Preparation and characterization of PVA/ $LaMnO_3$ composite nanofibers have been successfully investigated. As a result of XRD, it was found that the $LaMnO_3$ fibers were normally formed on Al substrate at 600°C. Some extra peaks were formed on XRD profile and might be due to the high voltage and vacuum system of the electrospinning device. From the SEM results, it was obvious that the altering solution chemistry was quite suitable for composite sol-gel preparation. This home-made setup was in fact a low-cost device compared to others, with estimates of cost 1/10 as much; however, it certainly attained the accepted value for nanofibers along with somewhat likely to require necessity. The experimental data resulted from this study indicated that the home-made device compiled and growth chemistry were technically simple and easily adaptable.

Acknowledgement

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