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Fabrication of $LaMnO_3$ Nanofibres by Electrospinning Technique and its Characterization

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Abstract— **$LaMnO_3$ nanofibre has attracted much interest recently due to their specific electrical and catalytic properties. A few methods on the preparation of $LaMnO_3$ nanocrystalline materials were reported. This research work was concerned with the preparation of nano-sized $LaMnO_3$ powder by pyrolysis method. Polycrystalline perovskite structure of Lanthanum manganite ($LaMnO_3$) nanofibres were obtained by calcination of the PVA/[$LaCl_3$+$MnCl_2$+ $(NH_4)_2$ CO_3] composite nanofibres at 700°C for 2 h. $LaMnO_3$ nanofibres were fabricated onto Al-substrate by using locally constructed set-up electrospinning technique. The crystal structure and phase formation were characterized by using X-ray Diffraction (XRD) and found that the crystal structure of the prepared $LaMnO_3$ nanofibres was in its orthorhombic symmetry. The diameter of the $LaMnO_3$ nanofibres were examined by Field Emission Scanning Electron Microscopy (FESEM) and the results were found to be 45 nm, 50 nm and 62 nm for different spinning time at constant temperature of 700°C.**

Keywords- ***Electrospinning machine, $LaMnO_3$ nanofibres, pyrolsis , FESEM***

I. INTRODUCTION

The term nanotechnology describes a range of technologies performed on a nanometer scale with widespread applications as an enabling technology in various industries. Nanotechnology encompasses the production and application of physical, chemical and biological system at scales ranging from individual atoms or molecules to around 100 nanometers, as well as the integration of the resulting nanostructures into larger systems [1]. Nanofibres are an exciting new class of material used for several value added applications such as medical, filtration, barrier, personal care, composite, garments, insulation and energy storage [2]. Electrospinning makes it relatively easy to spin continuous nanofibres from many different polymers. The electospinning process is driven by the electrical forces on free charges on the surface or inside a polymeric liquid [3]. Mechanical formation of polymer crystals often produces fibres which are observed in electron micrographs of feature surfaces, for example such fibres typically have diameters of a few tens of nanometers and lengths up to a few micrometers [4].Many images of polymer nanofibres exist in the literature that deals with polymer morphology, but in almost all cases the nanofibres were observed incidentally to other features of the polymer [5]. The sol-gel process is a versatile solution process for making nanofibres, ceramic and glass materials. Nanofibres can be made of Lanthanum Manganite ($LaMaO_3$) by electrospinning technique. One-dimentional nanostructure materials, such as nanofibres, have received great interest due to high surface area to mass or volume ratio [6]. Jinxian,W,et al got that the diameter of fibre was 150 nm by using starting chemicals of PVA /[La $(NO_3)_3$+Mn ($CH_3COO)_2$]. In this paper, the diameter of fibre was manual to be about 62 nm from the starting chemicals of PVA/[$LaCl_3$+$MnCl_2$+ $(NH_4)_2$ CO_3].

II. EXPERIMENTAL PROCEDURE

2.1 *Sample Preparation of $LaMnO_3$ nano fibre*

The experimental procedure and operational parameter are as describe in Figure 1 and Table 1. 0.82 g of $LaMnO_3$ powder was dissolved in 10 g of Poly Vinyl Alcohol (PVA). The mixtures were dissolved in distilled water and stirred by magnetic stirrer for 3 hour to form a homogeneous solution. The viscosity of the solution was measured and placed at room temperature for 24 hour to get a uniform solution. Finally the solution was expected to be viscous enough for electrospinning. Horizontal experimental set-up was chosen for electrospinning process. The $LaMnO_3$ solution was taken in a syringe with hypodermic needle. The hypodermis syringe needle was connected to the positive terminal of a high DC voltage generator that produce maximum voltage 26 kV and the negative terminal of the power supply was connected to the collector (Aluminium foil) opposite to the syringe needle with a distance about 9 cm. Al-substrate was then stuck 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 high electrical potential overcomes the surface tension of the solution in the syringe needle and the jet of charged precursor solution was ejected out from the collector. The precursor solution was deposited on Al substrate by electrospinning method with various running time intervals (15 min, 20 min, 25 min). And then the samples with different spinning time were heat-treated at 700°C for 2 h. The crystal structure was examined by XRD and the morphology and diameter of the fibres were observed with Field Emission Scanning Electron Microscopy [FESEM].

2.2 *Electrospinning Set-up*

The locally constucted electrospinning machine contained a needle or spinneret, high voltage power supply and a grounded collector as shown in Figure 2. Horizontal experimental set-up was chosen for electrospinning process. High voltage power supply which was transferred from 21" TV fly pad (219 × 6M, Toshiba) indirectly. To obtain the capable of producing the high voltage in the range of 10 kV ~ 30 kV, 60 of 10 M Ω resistors were used in series connection. A syringe holder and a collector were kept in the cylindrical shpae of glass tube, length of 36.5 cm and inside diameter was 8 cm. DC voltage generator of positive terminal was connected with hypodermic needle (0.55 × 25 mm) and a circular shape of Al-substrate collector which was connected by negative terminal of power supply as system ground. The polymer solution was to be spun through a syringe pump. When the high voltage was applied to the polymer solution, it was directed toward the collector, which collect the charged fibres. This grounded plate also served as the collector for the completed nanofibres web that was fabricated during the electrospinning.

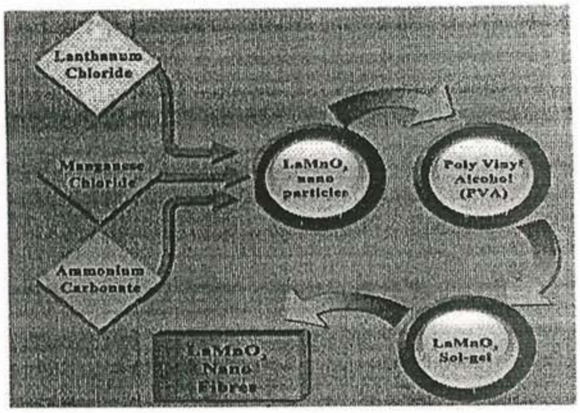

Fig 1 The flow chart of experimental procedure of $LaMnO_3$ nano fibres

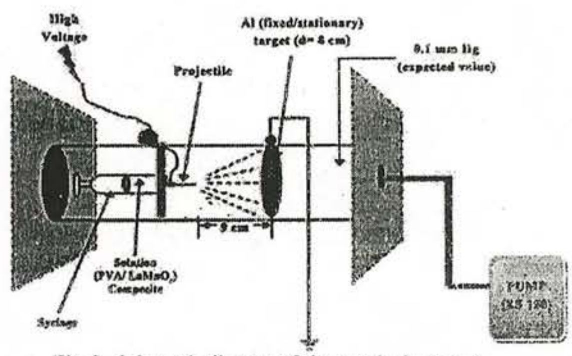

Fig 2 Schematic diagram of electrospinning set-up

TABLE.I OPERATING PARAMETER OF $LaMnO_3$

Polymer Solution	$LaMnO_3$
Syringe capacity	20cc
Electrodes spacing	9cm
Capillary Diameter	0.65mm
High voltage power supply (DC)	~30kV
Working high voltage power supply (DC)	~26kV
Running time	15min, 20min, 25min
Cooling time	3h
Annealing temperature	700 °C
Annealing time	2h

III. RESULTS AND DISCUSSION

3.1 *X-ray Diffraction Analysis*

Lanthanum Manganite nano powders was firstly characterized by XRD to make sure its structure. The X-ray diffraction pattern showed that all peaks of $LaMnO_3$ are consistent with that $LaMnO_3$ standard (JCPDS) powder as shown in Figure 3. It was found that the orthorhombic phase of LMO fine powder was obtained at this temperature range of 800 °C to 1200°C. The PVA/ composite fibres obtained were characterized by XRD as indicated in Figure 4. The d (spacing between crystallographic planes) values and relative intensities of The $LaMnO_3$ nano fibres calcined at 700 °C were consistent with those of JCPDS standard card and the crystal structure of the prepared $LaMnO_3$ nanofibres was found to have orthorhombic symmetry.

3.2 *FESEM Analysis*

The FESEM images of $LaMnO_3$ samples were shown in Figure 5 and 6 (a-c). Figure (5) showed the FESEM image of LMO powder sintered at 1000°C exhibit grained nanostructure with small crystallite size of 38 nm. In order to study the morphology and size of the synthesized fibres, the prepared fibres were examined by FESEM, as shown in Figure 6 (a-c). Figure 6 showd that, the morphology and size of the fibres varied strongly with calcination temperature. From FESEM results, the diameter of the $LaMnO_3$ fibres are found to be 45nm, 50nm, 62 nm, at 700°C with different spinning time of 15 min, 20 min, 25 min respectively.

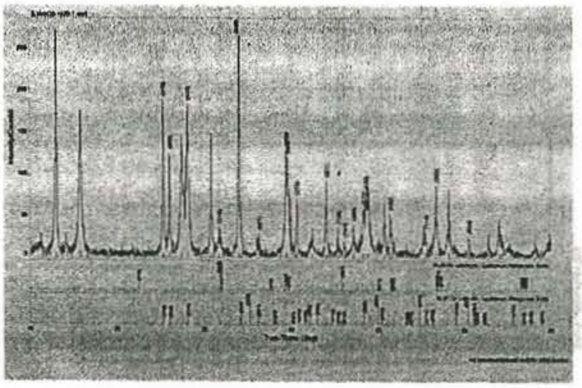

Fig 3 XRD patterns of $LaMnO_3$ powders, annealing temperature at 1000°C

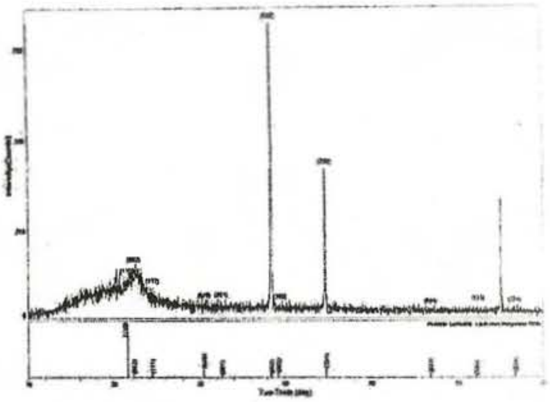

Fig 4 XRD Patterns of $LaMnO_3$ fibres, annealing temperature at 700°C

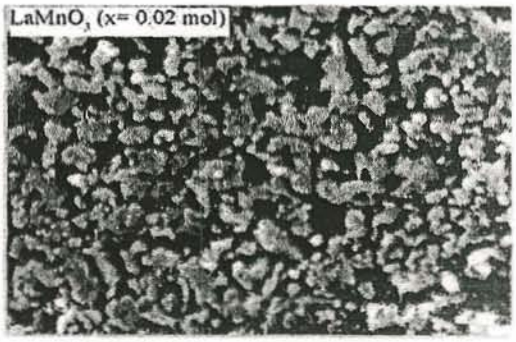

Fig 5 FESEM image of $LaMnO_3$ powder at 1000°C

Fig 6(a) FESEM image of $LaMnO_3$ fibre at 700°C (Spinning time-15min)

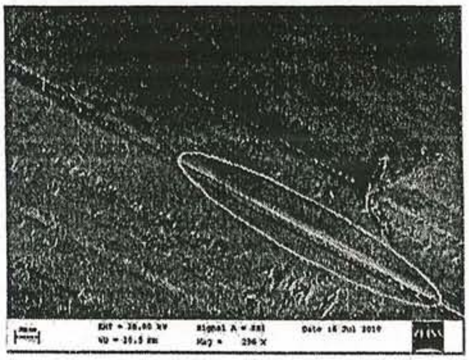

Fig 6(b) FESEM image of $LaMnO_3$ fibre at 700°C (Spinning time-20min)

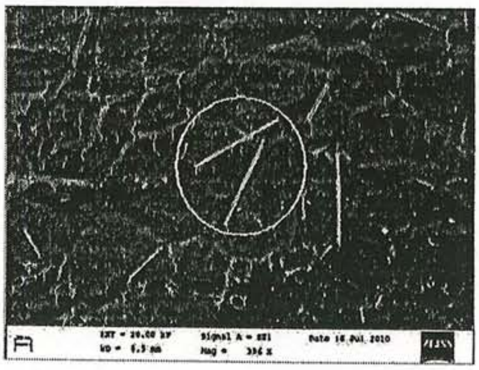

Fig 6(a) FESEM image of $LaMnO_3$ fibre at 700°C (Spinning time-25min)

IV. CONCLUSION

conclusion, PVA/ $LaMnO_3$ composite nanofibres were nthesized by calcining the relevant composite fibres at)0°C by electrospinning technique. FESEM images indicated that the surface of the prepared composite fibres were smooth and the diameters of the composite nanofibers were about 45 nm, 50 nm and 62 nm, with various spinning time at 700°C. The nanofibres can be useful in some potential application of electronic devices, separation membrane, biomedical applications and structural elements in artificial organs, nano- composites and protective clothing.

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