Structural, Microstructural and Analysis of Lanthanum Manganite (x=0.01mol) Powder

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Abstract

An improved method is proposed for the preparation of lanthanum manganite ($LaMnO_3$) powder by the calcinatins of a composite carbonate of the respective metallic elements formed by mixing an aqueous solution of a water-soluble ammonium carbonate and an aqueous solution of inorganic salts, e.g, chlorides, of the respective metallic elements. The surface morphology of the $LaMnO_3$ has been studied by scanning electron microscopy (SEM) and the wavelengths of the fluorescent X-rays (XRF) can be measured by an X-ray spectrometer as means of chemical analysis. Structural properties is characterized by XRD. Chemical and physical reaction of $LaMnO_3$ powder is investigated by TGA-DTA. The perovskite-structured $La_{1-x}Mn_xO_3$($x=0.01,_1x=0.99$) can accommodate at the A-site deficiency.

Keywords: *Lanthanum Manganite, surface morphology, XRF, XRD, TGA, perovskite*

Introduction

$LaMnO_3$ powder have been prepared by using pyrolysis methods. The present invention relates to a material of various kinds of catalysts, electrodes and the like mainly in the form of a sintered body. A method for the preparation of a lanthanum manganite powder has problems in several respects. For examples, it is almost unavoidable that a considerable portion of the starting powder remains in the product obtained by calcination and the amount of the unreacted starting materials can hardly be undetectably small even by conducting the process of calcination at a higher temperature for a longer length of time than usually undertaken. [AJAYAN P.M., SCHADLER, L.S. & BRAUN, P.V. 2003]

When the lanthanum manganite powder is to be used in the form of a powder as such as in the application for thermal spraying and the like because growth of particles is unavoidable in the powder blend to give a semi-sintered mass of coarse particles which must be disintegrated and finely pulverized. The product powder is poor in respect of the flowability behavior so that the

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powder is not suitable for use, for instance, in thermal spraying in which good flowability of the refractory powder is essential and in order not to cause bridging of the powder in the feeder hopper. [Greenwood.N.N, Earnshaw. A 1997]

Experimental Procedure of Lanthanum Manganite Powder

(1) $LaCl_3$, $MnCl_2$ and $(NH_4)_2\ CO_3$ were weigh by balance 37.137g, 19.790g and 9.6g respectively.

(2) Each compound was obtained with mixing distilled water of 100CC.

(3) The 0.99 mole of $LaCl_3$ and 0.01 mole of $MnCL_2$ were kept in a conical flask.

(4) It was stirred with magnetic stir about 10 minutes.

(5) After that $(NH_4)CO_3$ solution was added in it drop by drop.

(6) The mixture in the flask was to become precipitation for about 6 hours.

(7) By using filter paper, the cream of compound was left on the paper.

(8) The cream of composite was annealed at 100°C for 30 minutes.

(9) The dry composite was then heated 2 times of 1000°C for 30 minutes each.

(10) Finally, $LaMnO_3$ powder was obtained.

(11) The morphology of the powder was investigated by Scanning Electron Microscopy (SEM)

(12) The $LaMnO_3$ composition of chemical analysis can be measured by the fluorescent X-rays (XRF).

(13) X-ray diffraction are used determination of the structure of crystalline materials such as; crystal axes, size and shape of the unit cell, positions of the atoms in the unit cell.

(14) Thermo-gravimetric Analysis (TGA) is a type of testing that is performed on samples to determine changes in weight in relation to change in temperature.

(15) In this study, $LaMnO_3$ ferrites corresponding to the non-stoichiometry $La_{1-x}Mn_xO_3$ ($x = 0.01$, $1-x = 0.99$) were synthesized from the inorganic

agent and compared with respect to morphology, chemical composition and flowability.

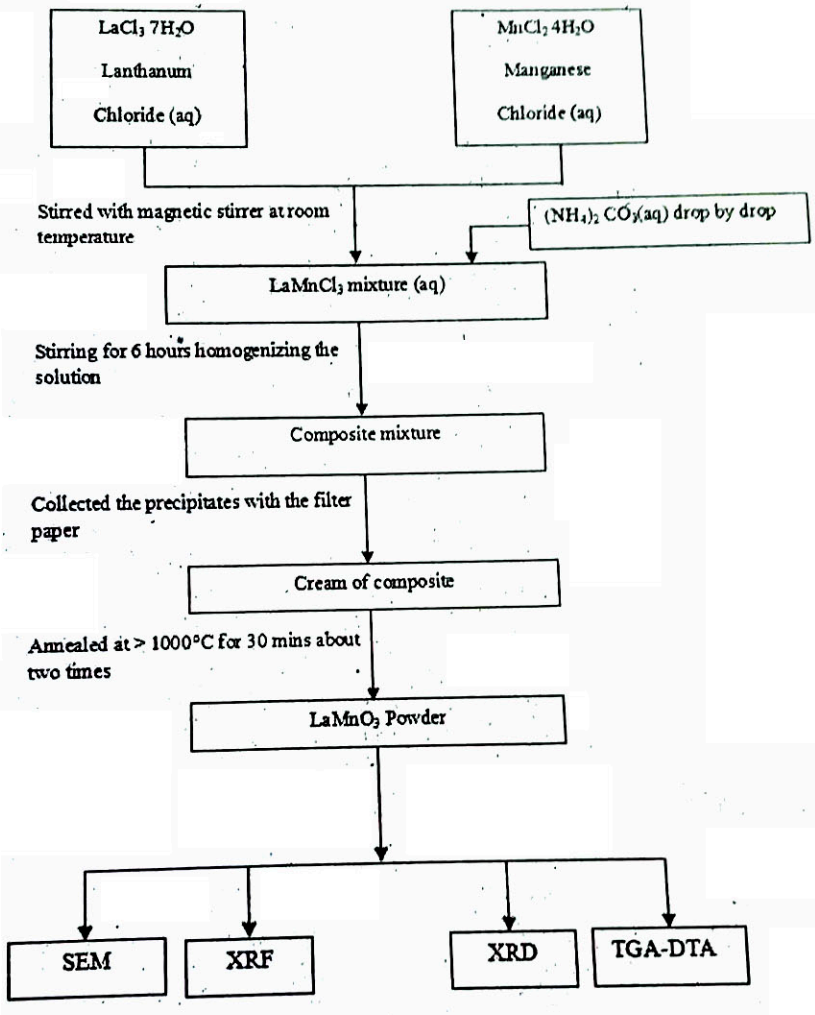


Fig. 1 The block diagram of preparation of $LaMnO_3$ Powder

Results and Discussion

The SEM is producing high resolution images of a sample surface. SEM images have a characteristic three – dimensional appearance and are useful for judging the surface structure of the sample. SEM analysis was performed to examine the particle size and microstructural properties of $LaMnO_3$ powder phase. The observed SEM microphotograph was shown in Fig (2).

This figure 2(a) showed the fairly dense image with fine grain size 10,000 photo magnification. The uniform grain distribution was observed on SEM image. The particle size was calculated to be 0.32μm. The grain – oriented was right size and SEM image was smooth. The resulting grain shape was found to be round.

This figure 2(b) showed the strongly dense image with fine grain size 10,000 photo magnification. The homogeneous grain distribution was observed on SEM image. The calculation of the particle size was to be 0.19μm. The orientation of grain was right size and SEM image are a little rough. The resulting grain shape was seen to be round cotton.

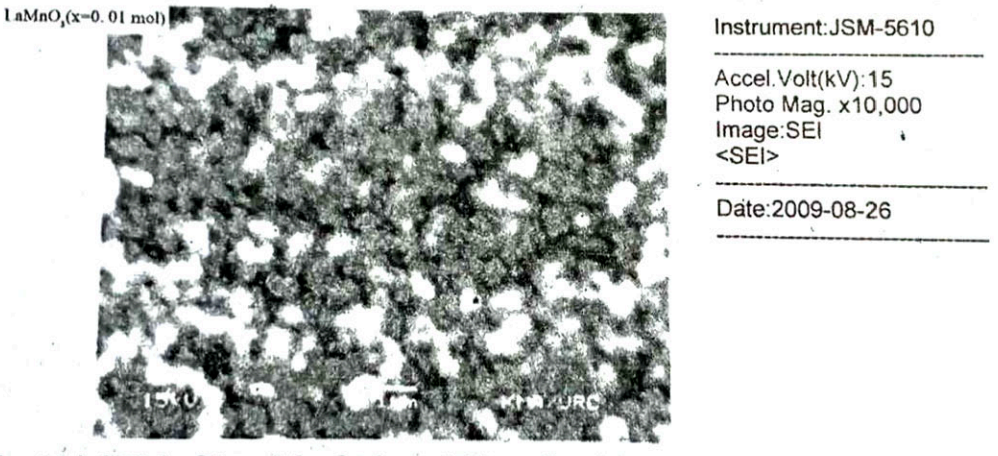


Fig .2(a) SEM of $La_{1-x}Mn_xO_3$ (x = 0.01 mol) powder

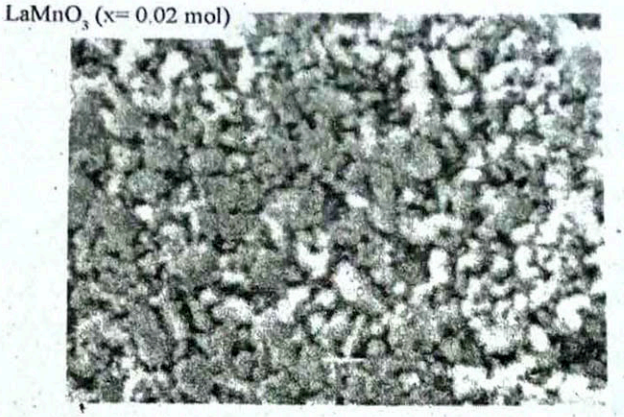


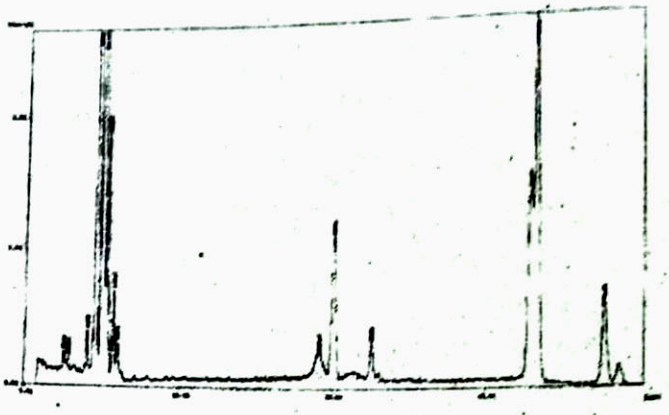
Fig .2(b) SEM of $La_{1-x}Mn_xO_3$ (x = 0.02 mol) powder

The measurement of XRF wavelength was advancement with reported values. In Fig. 3(a),

According to the XRF graph, it was found that the concentration of lanthanum (98.194%) and manganese (1.806%).

In Fig. 3(b),

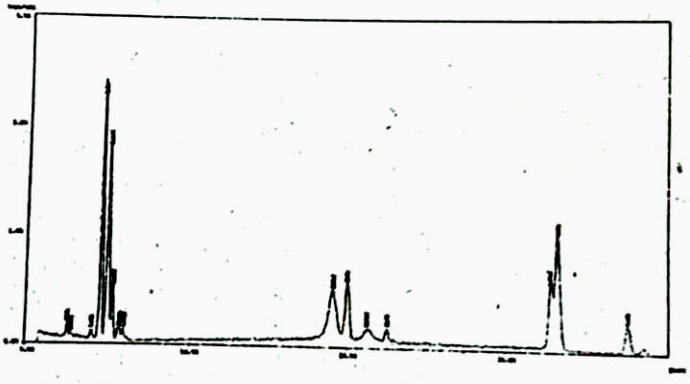
It was comprised that the concentration of lanthanum (82.051%), manganese (3.461%). Unfortunately, (14.488%) of chlorine was included in it. Because of time delay about 5 days. Therefore, lanthanum manganite powder was successfully formed by calcinations of the composite carbonate.



Quantitative Result

Analyte	Result
La	98.194%
Mn	1.806%

Fig. 3 (a) XRF of $La_{1-x}Mn_xO_3$ (x = 0.01 mol) powder.



Quantitative Result

Analyte	Result
La	82.051%
Cl	14.488%
Mn	3.461%

Fig. 3(b) XRF of $La_{1-x}Mn_xO_3$ (x = 0.02 mol) powder.

X-ray diffraction is one of the most important tools of solid-state chemistry, since it constitutes a powerful and readily available method for determining atomic arrangement in matter. X-ray diffraction methods depend upon the fact that X-ray wavelengths of the order of 1 nano-metre are readily available and that this is the order of magnitude of atomic dimensions.

The XRD spectra of LMO solid phase was shown in Fig 4. From the spectra, (111) identified peak was found that this peak was consistent with standard peak. The lattice constants were a = 5.4505Å and b = 4.9834, c = 6.9251Å.

The some powder structural parameters were collected in Table (1).

$La_{1-x}Mn_xO_3$ (x = 0.01 mol%)	Structural Parameters
Lattice constants, a (Å)	5.4505
Lattice constants, b (Å)	4.9834
Lattice constants, c (Å)	6.9251
FWHM (Å)	0.431
Crystallite size (nm)	18.9757
u-parameter (Å)	0.4565
bond – length (Å)	3.1612

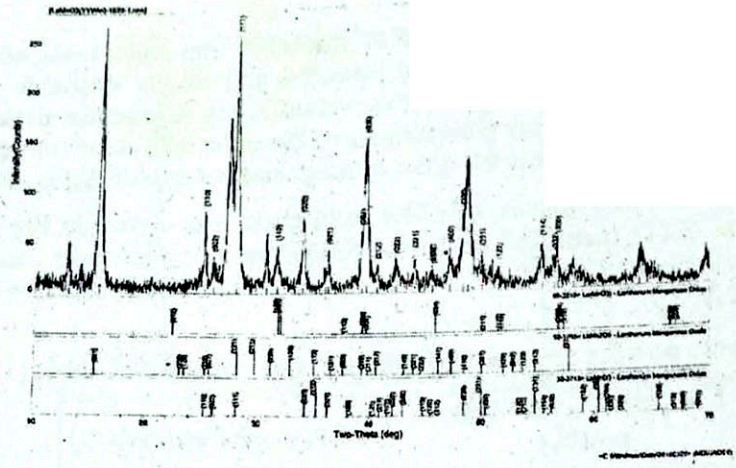


Fig. (4) XRD pattern of $LaMnO_3$ powder annealing temperature at 1000°C for 30 minutes.

The thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were shown in Fig 5 (a) and 5 (b) showed that the $LaMnO_3$ powder has no crystalline water and no phase transition before decomposition.

Complex degradation of $LaMnO_3$ compound take place above 230°C. The decomposition, point of $LaMnO_3$ powder was at 230°C and 400°C.

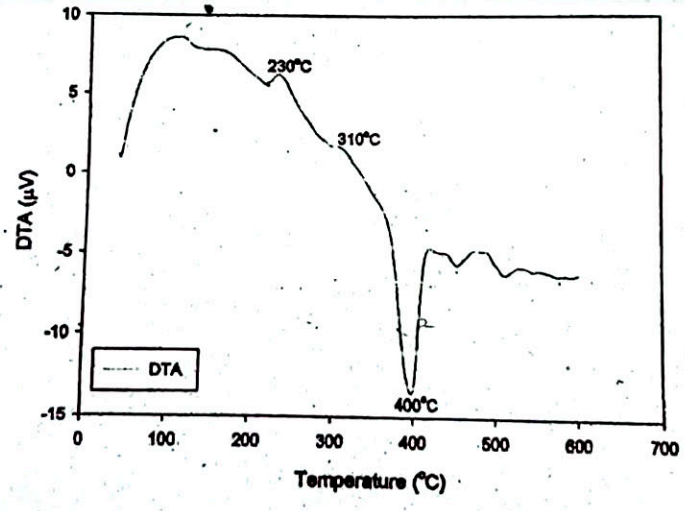


Fig5(a) DTA thermograms of $LaMnO_3$ powder

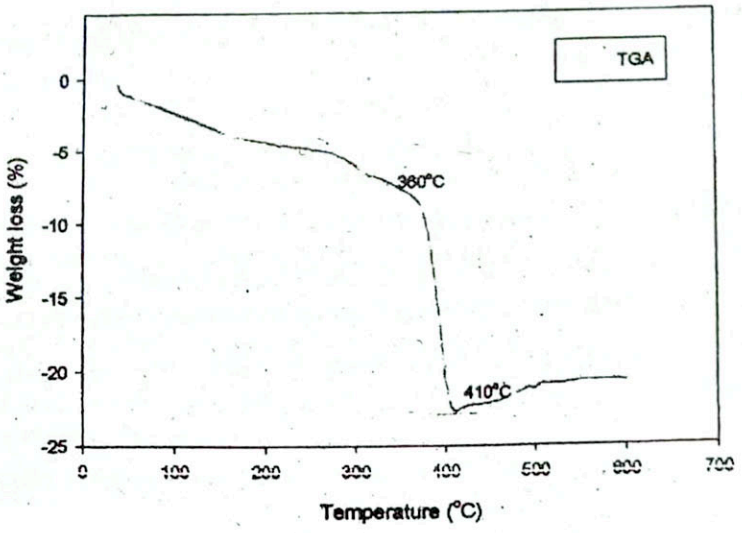


Fig5(b) TGA thermograms for weight loss of $LaMnO_3$ powder

Conclusion

Preparation of $LaMnO_3$ powder has been observed from these observations some remarkable conclusion could be established. The particle size of the $LaMnO_3$ powder has been increased (x=0.01 mol). The surface morphology Scanning Electron Image (SEI) are uniform and smooth. From X-rays Fluorescence (XRF) graph, the main chemical takes part in the final reaction. According to these results, thus, lanthanum manganite powder was successfully formed by calcinations of the composite carbonate. The lattice parameters and crystallite size evaluated by XRD technique. The lattice parameters and crystallite size of sample have been evaluated by XRD technique. The weight loss and thermal behaviours of the sample were analyzed by the TGA-DTA study. The $LaMnO_3$ sample was found to be thermally stable up to 400°C.

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