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Der Chemica Sinica, 2012, 3(4):891-895

Pelagia Research Library
ISSN: 0976-8505
CODEN (USA) CSHIA5

Preparation and characterization of perovskite $LaAlO_3$ nanocrystals

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ABSTRACT

The aim of said study was to obtain nanostructured $LaAlO_3$ through self combustion synthesis using lanthanum nitrate, aluminium nitrate as precursors and glycine as a fuel, without the subsequent heat treatments after synthesis. The temperature variation with respect to a sample with constant molar ratio was investigated. The crystallinity (phases present and crystallite size: estimated by single-line method) of the product obtained was determined by X-ray diffraction (XRD) measurement, thermogravimetric analysis(TG-DTA),scanning electron microscope(SEM),and transmission electron microscopy (TEM). The synthesis method facilitated the production of Perovskite $LaAlO_3$ with crystallite size between 40-70 nm.

Keywords: La_2ZnO_4 nanostructure, XRD, TG/DTA, EDX, SEM, TEM.

INTRODUCTION

Perovskite-type ABO_3 and related compounds have been reported to be of importance due to their wide uses in fuel cells[1] , catalysts[2,3] membranes in syngas production[4], sensors[5,6] and environmental monitoring applications [7]. Gas sensing application etc. Among the chemical sensors $LaCoO_3$, $BaTiO_3$, $LaFeO_3$, $LaMnO_3$ etc. are perovskite-type materials of general formula ABO_3 are extensively studied owing to their notable gas sensitivity for different poisonous gases in addition to their magnetic, catalytic and other physical properties. The perovskite-type metal oxide including the d-block and rare earth elements has attracted the attention of many researchers due to their homogeneity, interesting structural, catalytic and gas sensing properties. There is an increasing interest in finding new materials in order to develop high performance solid state gas sensors. Combustion synthesis is an easy and convenient method for the preparation of a variety of advanced ceramics, catalysts and nanomaterials [8]. In this technique, based on the principles of the propellant chemistry,[9] a thermally induced redox reaction takes place between an oxidant and a fuel. Many types of combustion synthesis exist which differ mainly in the physical state of the reactants or in the combustion modality [10,14].

Herein, we present a novel and facile synthesis of porous $LaAlO_3$ nanocrystals based on the self-combustion, were prepared from Al $(NO_3)_3 \cdot 9H_2O$, $La(NO_3)_3 \cdot 6H_2O$, and glycine source of the cation, but also the fuels. Homogeneity is achieved primarily because it is mixed on an ionic scale, which ensures the formation of nanopowders. To our knowledge, this is the first report of formation of perovskites through the self-combustion, which is a high yield, solvent less route and helps to develop the new field of $LaAlO_3$ in synthesis of inorganic materials.

MATERIALS AND METHODS

Powder preparation

Polycrystalline $LaAlO_3$ was synthesized by the combustion synthesis method[21-23] using glycine as fuel (organic fuel). All chemical reagents were analytical grade and used without further purification. Stoichiometric quantity of solid mixture of one mole reagents i.e. La $(NO_3)_3$ nH_2O (purity 98.5%), Al $(NO_3)_3$ $9H_2O$ (purity 98.5%) and two mole of glycine (purity 99.5%) were mixed together in a flat Pyrex disc. The solid were stirred for five minutes (clear solution was obtained). The solution formed was evaporated on hot plate in temperature range 80-90^{0}C which give thick gel. The gel was kept on a hot plate for auto combustion and heated in the temperature range of 160-180^{0}C.The nanocrystalline $LaAlO_3$ powder was formed within five minutes. This powder was grind in the mortal pestle and used for the characterization and white colour of synthesized $LaAlO_3$ powder [15,16].

Characterization Technique

The as –prepared samples were characterized by TG/DTA thermal analyzer (SDT Q600 V 20.9 Build 20), XRD Philips Analytic X-ray B.V. (PW-3710 Based Model diffraction analysis using Cu-K α radiation), scanning electron microscope (SEM, JEOL JED 2300) coupled with an energy dispersive spectrometer (EDS JEOL 6360 LA), A JEOL JEM–200 CX transmission electron microscope operating at 200 kV analysis.

RESULTS AND DISCUSSION

TG and DTA measurements were performed to study the thermal behavior of sol Figure 1. At the beginning synthesized powder was stable up to the temperature 450^{0}C. This indicates that no loss of any material but beyond that temperature there is slight weight gained and this is due to the adsorption of nitrogen gas supplied by the instrument. The slight weight gained was occur up to the temperature 750^{0}C.After this temperature the weight loss take place due to the desorption of nitrogen gas. At 850^{0}C temperature the powder is stable. This weight loss and weight gained is very negligible. This weight change is in the range of 0.002 % only. This indicates that the synthesized powder was almost stable from the beginning.

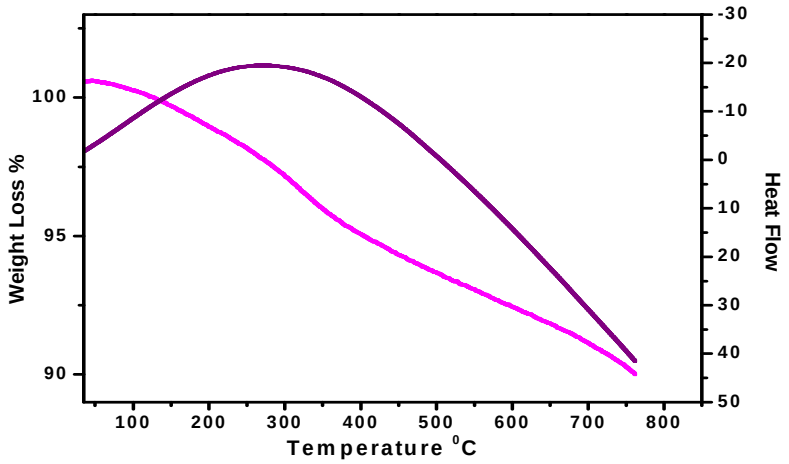

Fig. 1. TG-DTA curve of mixed precursor $LaAlO_3$

X-ray diffraction XRD measurements were performed on Philips Analytic X-ray B.V. (PW-3710 Based Model) Advanced X-ray diffraction using Cu Kα 1.54056 radiation. The XRD pattern shows that the product is pure perovskite oxide $LaAlO_3$ with an orthorhombic structure. The diffraction data is good agreement with JCPD card of $LaAlO_3$ (JCPDS No.33-0699). The average crystalline size of $LaAlO_3$ perovskite powder is determine with the help of Scherrer's equation $t=0.9\lambda/\beta\cos\theta$ [17] , where t is the average size of the particles, λ is wavelength of X-ray radiation, β the full width at half maximum of the diffracted peak and θ is the angle of diffraction. The average crystalline size obtained for nanocrystalline $LaAlO_3$ calcinated at 850^{0}C is found to be 45 nm. $LaAlO_3$ nanocrystals are more attractive in the field of catalytic application.

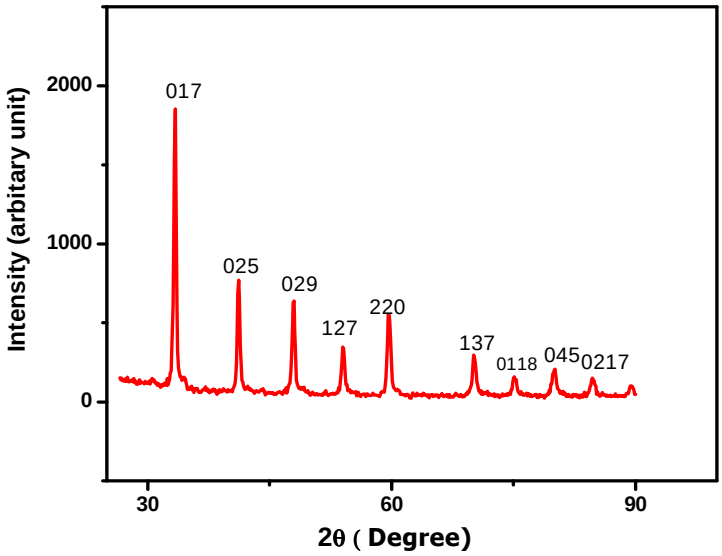

Fig. 2. XRD pattern of calcinied mixed precursor $LaAlO_3$ at 850^{0}C in air for 4 h.

The elemental analysis of the sample was carried out by using energy dispersive X-ray spectrometer (EDS) and is shown in Figure 3.The EDS result clearly shows that $LaAlO_3$ contains La, Al, and O without any impurity. Quantitative EDX analysis verified that doping with lanthanum is close to the expected concentration.

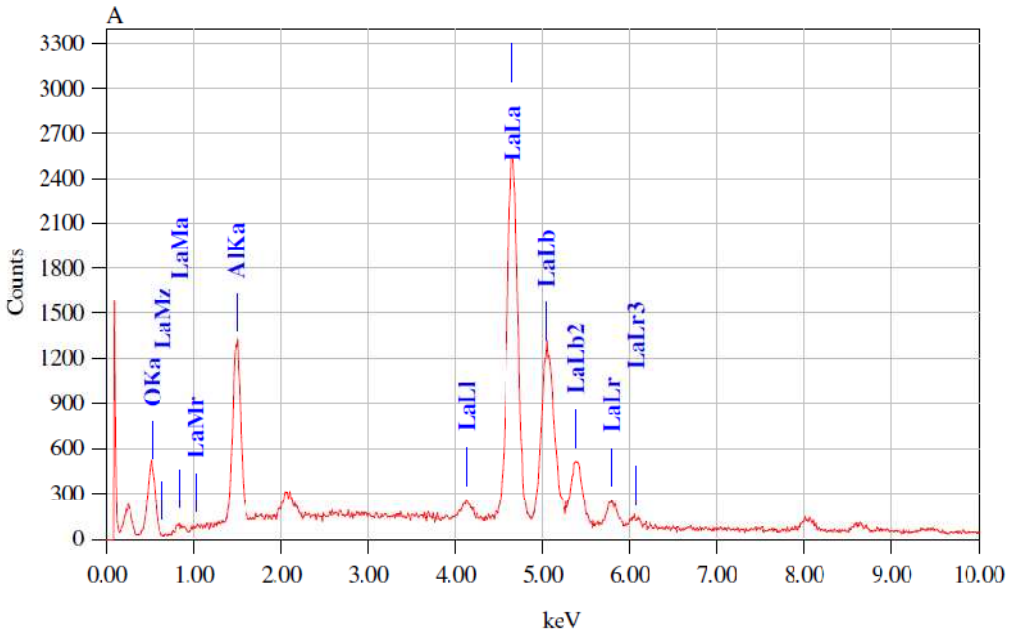

Fig. 3. EDX pattern of mixed precursor $LaAlO_3$at 850^{0}C in air for 4 h.

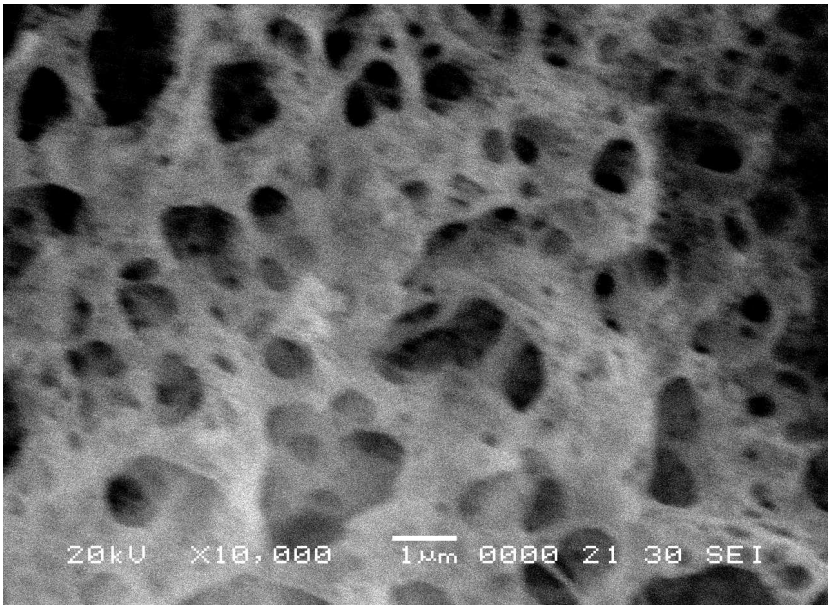

Fig.4 SEM images of $LaAlO_3$ powder.

The surface morphology is studied by using scanning electron microscopy (SEM) Model No. JEOL,JED-2300 analysis station. The SEM-EDX image of $LaAlO_3$ is shown in Figure 4. The SEM picture of pure $LaAlO_3$ shows that the size of $LaAlO_3$ particles are not uniform, agglomeration (particle-particle interactions) is observed. SEM images reveal the low density product, loose and porous material that is favourable to a gas sensing application.

The TEM specimens were prepared by placing microdrops of colloid solutions on a carbon film supported by a copper grid. The TEM images of the nanocrystalline $LaAlO_3$ calcinated at 850^0C in air for 5 h are shown in Figure 5 . It indicates the presence of $LaAlO_3$ nanoparticles with 40-70 nm size which form spherical type of oriental aggregation, agglomeration and polymeric linkage throughout the region. The pores are well developed spherical shape with diameter from 40-70 nm size. The results are correlated with the XRD.

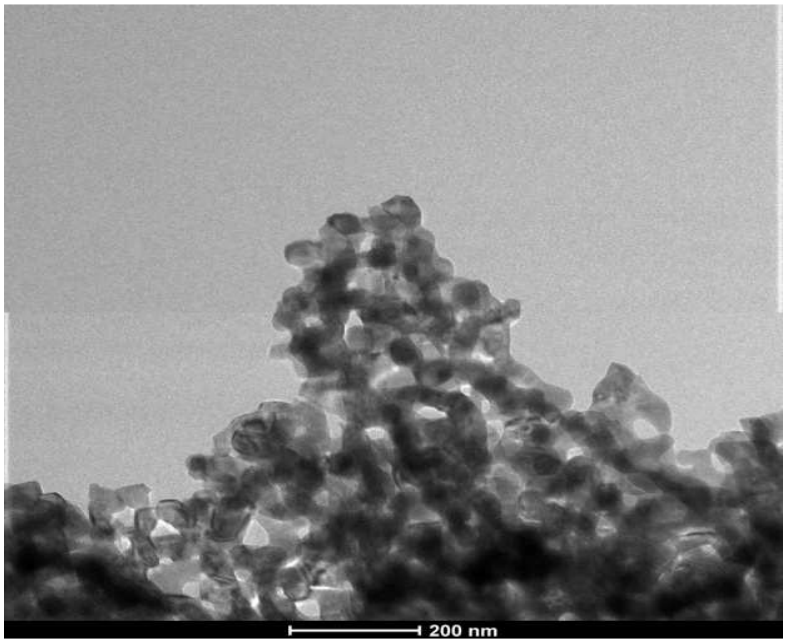

Fig. 5 Transmission electron microscopy image of $LaAlO_3$ calcinated at 850^0C

CONCLUSION

A novel combustion method was developed for preparation of pervoskite –type oxygen permeable materials. The method offer the advantages of facilitating the crystallization of pervoskite phase structure and easy handling of product. The phase formation of the $LaAlO_3$ is investigated by TG-DTA and XRD techniques. The synthesized product shows single phase of inverse Perovskite structure with an average diameter 30-70 nm.

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