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Synthesis of $La_{1-x}Sr_xMnO_3$ powders by polymerizable complex method: Evaluation of structural, morphological and electrical properties

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Abstract

Lanthanum strontium manganite ($La_{1-x}Sr_xMnO_3$, LSM) powders were synthesized by polymerizable complex method, based on complexation of metal ions (MI) with citric acid (CA) and polyesterification between CA and ethylene glycol (EG). Firstly, the effect of the molar ratio of CA:MI (=1–3) was investigated on the synthesis of $La_{0.7}Sr_{0.3}MnO_3$ powders, which were characterized by thermal analysis (TGA/DTA), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The results indicated that the molar ratio CA:MI = 3 is adequate for a good crystallization of pure perovskite phase after calcination, with nanometric crystallite sizes and porous microstructure. For the $La_{0.7}Sr_{0.3}MnO_3$ sample synthesized with CA:MI ratio of 3, it was investigated the effect of calcination temperature, showing that the perovskite structure is better crystallized at 900 °C, without secondary phase formation. Using this same CA:MI ratio and calcination temperature, powders with different Sr content (x = 0.2–0.4) were synthesized, with surface areas of 4–10 $m^2\ g^{-1}$. These powders were sintered at 1100 °C to produce porous pellets. The porosity of the sintered pellets and the electrical conductivity, measured by two-probe technique, increased with increasing Sr content.

Keywords: A. Powders: chemical preparation; B. X-ray methods; C. Electrical conductivity; D. Perovskites; Lanthanum strontium manganite

1. Introduction

Solid oxide fuel cells (SOFCs) have become of great interest as a potential economical, clean and efficient means of producing electricity in a variety of commercial and industrial applications [1]. Lanthanum strontium manganite (LSM) is known to be a potential cathode material for SOFCs based on stabilized zirconia electrolyte because of its high electrical conductivity, chemical and thermal stability, high catalytic activity for the oxygen reduction and its compatibility with zirconia electrolyte [1,2]. The cathode material should have porosity between 20 and 40% and an electrical conductivity higher than 100 S cm^{-1} (measured by four-probe method) [3]. The crystalline structure of LSM depends on Sr-doping degree, in general it is rhombohedral, although some authors observed that LSM structure became cubic for $x > 0.3$, as shown by Gaudon et al. [4].

Recently, much attention has been given on the preparation method as the actual composition and properties of the materials depend on the synthesis procedure. The homogeneous starting powder is a prerequisite for the manufacturing of high performance electrodes, since the microstructure and properties of ceramics are significantly influenced by the characteristics of preliminary powder. Thus, it is fundamental to prepare high-quality powders with controlled stoichiometry and microstructure.

Several synthesis methods have been developed for preparation of LSM powders, such as solid-state reaction [5], combustion method [6,7] and some solution chemistry methods, for example, sol–gel process [4], co-precipitation technique [8] and citrate process [9–11]. Particularly, solution chemistry methods are used to prepare materials of high purity, with small grain size and at lower temperature compared to the conventional solid-state method [12].

The polymerizable complex method, developed by Pechini [13], uses citric acid (CA) to chelate metal ions (MI) and ethylene glycol (EG) as a solvent for the process of polymerization to form an intermediate of polyester-type resin. According to Popa and Kakihana [14] the process consists of

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