



Electrical conductivity and *ac* dielectric properties of $\text{La}_{0.8}\text{Ca}_{0.2-x}\text{Pb}_x\text{FeO}_3$ ($x = 0.05, 0.10$ and 0.15) perovskite compounds



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ABSTRACT

Complex perovskite lanthanum calcium lead iron oxide compounds with the composition $\text{La}_{0.8}\text{Ca}_{0.2-x}\text{Pb}_x\text{FeO}_3$ ($x = 0.05, 0.10$ and 0.15) were synthesized by the sol–gel method using the citric acid route. X-ray diffraction patterns showed that the nanosize crystalline powders present an orthorhombic symmetry with *Pnma* space group. Impedance spectroscopy was used to characterize the electrical behavior of these materials as a function of frequency (10^2 – 10^6 Hz) at various temperatures (30–400 °C). The imaginary part of the complex impedance (Z'') frequency dependence revealed one relaxation peak. The Cole–Cole plots of the impedance values exhibited a semi-circular arc that can be described by an $(R_1\text{CPE}_1)/(R_2C_1)$ electrical equivalent circuit. The frequency dependence of the conductivity has been investigated with the Jonscher universal power law. The *ac* electrical conduction was studied and associated to the non-overlapping small polaron tunneling (NSPT) model.

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1. Introduction

The perovskite oxides (ABO_3), especially the orthoferrites, are an interesting class of materials [1] due to the large variety of applications in which they can be employed. Examples of these applications include electrode materials for high temperature solid oxide fuel cells, catalysts for the complex oxidation of hydrocarbons and sensor material for the detection of humidity, alcohol and oxygen [2,3]. Among these perovskite oxides, the LaFeO_3 and the A and B-site substituted LaFeO_3 with the general formula of $\text{La}_{1-x}\text{A}_x\text{FeO}_3$ and $\text{LaFe}_{1-x}\text{B}_x\text{O}_3$ (where A represents a rare-earth elements and B a transition metal) have been studied thanks to their extensive usage in applications such as oxide fuel cells (SOFC) cathode [4–8], oxygen dense membrane [9–11] and oxygen sensor [12]. Compositions of $\text{La}_{1-x}\text{M}_x\text{FeO}_3$ are plausible cathodes for LAMOX, which is [13] since LSF ($\text{La}_{1-x}\text{Sr}_x\text{FeO}_3$) materials, have demonstrated superior electrochemical properties and adjustable thermal expansion coefficients [4]. The solid solutions of $\text{La}_{1-x}\text{Ca}_x\text{FeO}_3$ (LCF), compositions related to LSF, have been studied as catalysts for methane combustion [14] and oxygen sensors [15].

In this paper, $\text{La}_{0.8}\text{Ca}_{0.2-x}\text{Pb}_x\text{FeO}_3$ (LCPFO) compounds in

nanocrystalline form were synthesized to investigate the effect of Pb-doping on the structural and dielectric properties, using the alternating current (*ac*) impedance at different temperatures and frequencies. To the best of our knowledge, this is one of the first reports about the dielectric properties of lead doped $\text{La}_{0.8}\text{Ca}_{0.2-x}\text{Pb}_x\text{FeO}_3$ compounds.

2. Experimental

The nanocrystalline $\text{La}_{0.8}\text{Ca}_{0.2-x}\text{Pb}_x\text{FeO}_3$ (LCPFO) powders ($x = 0.00, 0.05, 0.10, 0.15$ and 0.20) were prepared by the sol–gel method [16,17] using lanthanum nitrate, lead nitrate, calcium nitrate, ferric nitrate and citrate acid (all analytically pure) as raw materials. In a first step, all nitrates were separately dissolved in ion free water at 343 K. Afterward, citrate acid, as a metal complex agent, was added to the mixture of these nitrates using the molar ratio of $n(\text{La}^{3+} + \text{Ca}^{2+} + \text{Pb}^{2+} + \text{Fe}^{3+}):n(\text{citric}) = 1:2$, under constant stirring for 1 h. The molar excess of citric acid is to make sure that all metal cations are complexed. The next step was the addition of polyethylene glycol (PEG molecular weight 20,000) to the citric acid solution, to promote the polymerization and consequent formation of the gel. This stage took 5 h under constant stirring at room temperature. After this, the gel was dried at 443 K and the obtained powder was carefully crushed to form a fine powder. For subsequent annealing, the samples were placed in an electric oven

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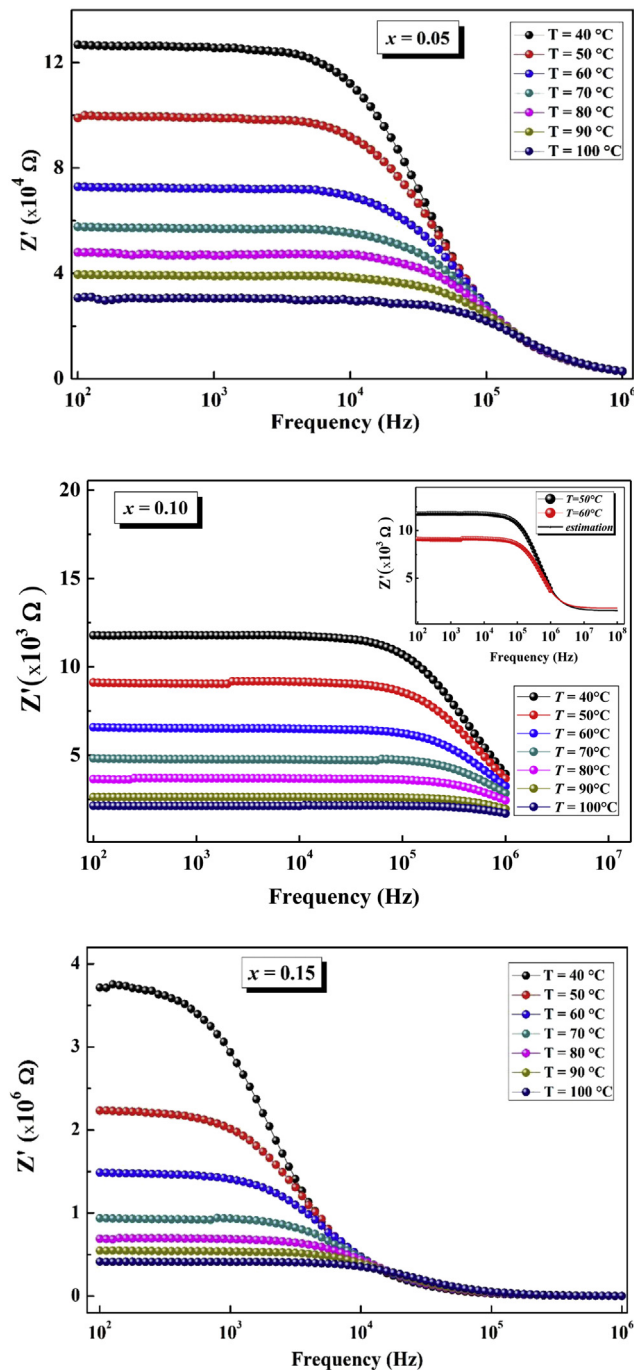


Fig. 1. The frequency dependence of the real (Z') part of the complex electrical impedance at several measurement temperatures.

at 527 K, for 12 h. Next, the powders were gridded again, and using a uniaxial pressure system, pellets of approximately 10 mm in diameter and 1.5 mm in thickness, approximately, were made and submitted to heat treatments at different temperatures (673, 773, 873 and 973 K), for 24 h. The resultant powders were characterized by X-ray diffraction (XRD) with Cu-K α radiation to determine phase compositions.

For the electrical measurements, a system formed by a vertical electric furnace and a dry rotary pump, in a closed cycle setup, connected to the extremes of the furnace was used [18]. This non-inductive electrical furnace operates from room temperature up to

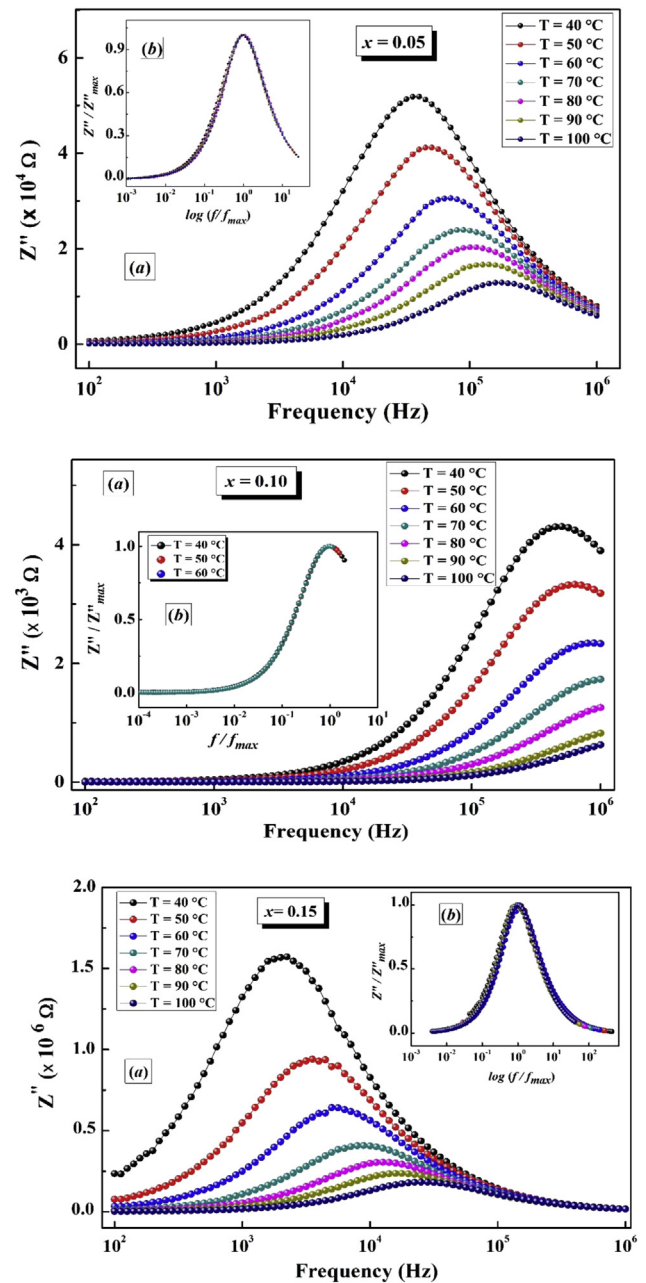


Fig. 2. The frequency dependence of the imaginary (Z'') part of the complex electrical impedance at several measurement temperatures.

1473 K. In each measurement, the sample was mechanically pressed between two parallel platinum plates, forming the electrodes. The *ac* impedance of the samples was measured between room temperature and 673 K, and in the frequency range of 100 Hz up to 1 MHz, using an Agilent 4294A Precision Impedance Analyzer.

3. Results and discussion

3.1. Complex impedance spectroscopy

In order to understand the dynamics of the mobile ions (relaxation mechanism) in LCPFO compounds, we have represented the angular frequency dependence of the real (Z') (Fig. 1) and imaginary (Z'') (Fig. 2) parts of the complex electrical impedance at several

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