Journal of Alloys and Compounds 712 (2017) 147–151

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Hydroxyapatite based nanocomposite ceramics

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ARTICLE INFO

Article history:
Received 22 August 2016
Received in revised form
29 March 2017
Accepted 2 April 2017
Available online 4 April 2017

Keywords:
Ceramics
Optical properties
Sol gel method

ABSTRACT

Lanthanum-doped hydroxyapatite ceramics were synthesized by the sol gel method and their chemical composition analyzed by FTIR and EDS. SEM observations showed all samples to exhibit a spherical nanoparticles nanostructure. The electrical properties of La doped HAp ceramics were investigated by dielectric and polarization -voltage measurements. The dielectric constant, ε_r increased gradually with the amount of lanthanum doping. A non-Debye type dielectric relaxation mechanism was observed as well as a ferroelectric behavior. Results indicate that La doping improves the electrical and ferroelectric properties of hydroxyapatite ceramics.

1. Introduction

Physical and chemical properties of apatites based boronphosphates have been investigated for various applications [1–4]. One of calcium phosphate (CaP) ceramics is hydroxyapatite (HAp) with the chemical formula, $Ca_{10}(PO_4)_6(OH)_2$ [5,6]. In medical applications such as repairing of bone defects and bone augmentation, orthopedics, odontology, coating of metallic implants, it has been used [7,8]. Hydroxyapatite (HAp) needs to improve its electrical properties for potential biomedical sensing applications [9–11]. Dielectric properties of hydroxyapatite material are related to electrical polarization. It is well known that the biological response can be improved by polarized surface and therefore, electrical polarization provides an excellent ability to manipulate biomedical tissues [12–15]. It is evaluated that the electrical and dielectric properties of hydroxyapatite material can be controlled using La content for biomedical applications.

In the present study, La-doped hydroxyapatite ceramics were synthesized by the sol gel method. The structural, dielectric and polarization properties of La-doped hydroxyapatite ceramics were investigated for various La contents.

2. Materials and method

Lanthanum doped hydroxyapatite ceramics were synthesized for various La contents (x = 0.1, 0.2, 0.3, 0.4 and 0.5) by the sol gel method. The ceramics were synthesized using calcium nitrate tetrahydrate ($Ca(NO_3)_2{\cdot}4H_2O$) and diammonium hydrogen phosphate ($(NH_4)_2HPO_4$) and lanthanum acetate precursors. Firstly, nominal amount of ($Ca(NO_3)_2{\cdot}4H_2O$) was dissolved in deionized water and stirred for 10 min pH of solution was adjusted to 8 by adding ammonia and then, ($(NH_4)_2HPO_4$) was added to the solution and stirred at 80 °C for 2 h. Lanthanum doped hydroxyapatite solutions were prepared for various La contents and solutions dried at 100 °C for 12 h to get the powders. Powders of La doped HaP ceramics were calcinated at 800 °C for 2 h. Dielectric properties of La doped HaP ceramics were performed using an HIOKI LCR meter. FTIR spectra of La doped HaP ceramics were analyzed using a Nicolet spectrophotometer. Ferroelectric measurements of La doped HAp ceramics were performed using Radiant ferroelectric tester.

3. Results and discussion

3.1. Structural properties of lanthanum doped hydroxyapatite ceramics

The chemical composition of lanthanum doped hydroxyapatite ceramics was analyzed using FTIR spectra. FTIR spectra of the ceramics are shown in Fig. 1. The bands observed at 960–963 cm^{-1} and 561–602 cm^{-1} belong to the phosphate groups and OH groups of hydroxyapatite. A change in peak intensity of FTIR spectra was observed with La doping. The formation of HAp and α-TCP structures was confirmed with the presence of OH bands at 632-635 cm^{-1} and PO_4 bands at 1091-962 cm^{-1}. The band at 1020–1100 cm^{-1} is the most intense peak of phosphate group. This

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http://dx.doi.org/10.1016/j.jallcom.2017.04.021
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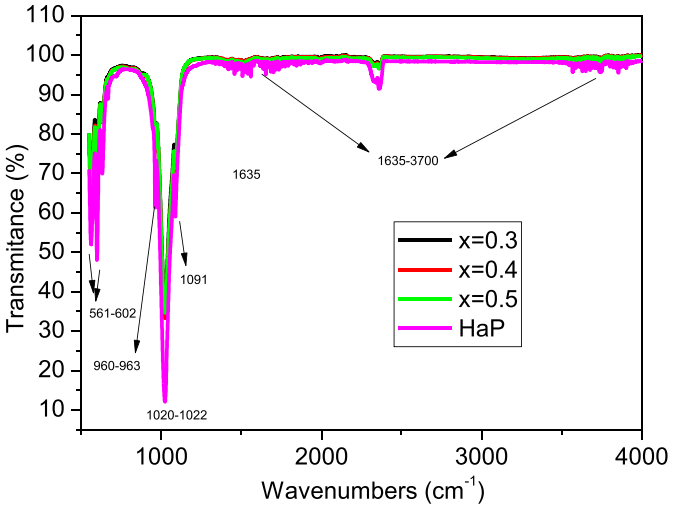

Fig. 1. FTIR spectra of La doped HAp ceramics.

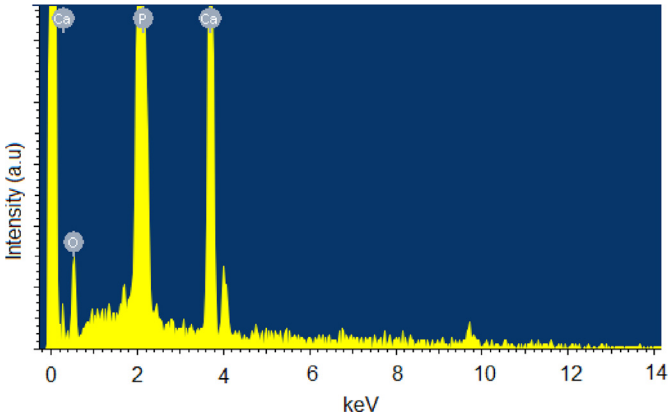

Fig. 3. EDS spectra of HAp ceramic.

peak was observed in all samples and it is due to P-O stretching vibration of phosphate group [16,17]. The bands at 1635 cm^{-1} and 2500 cm^{-1} extending to 3700 cm^{-1} correspond to the absorbed water. The obtained FTIR bands are in good agreement with the bands of HAp [18].

Fig. 2 shows SEM images of lanthanum doped hydroxyapatite ceramics. As seen in Fig. 2, all samples exhibited a spherical nanoparticles nanostructure. Lanthanum doped hydroxyapatite ceramics are nanomaterials, because their sizes are in nano regime. The distribution of nanoparticles is changed with La content. The chemical composition of the ceramics was analyzed by X-ray Energy dispersive spectroscopy (EDS). EDS spectrum was given only for HAp sample, as shown in Fig. 3. As seen in Fig. 3, HAp sample included only phosphorus, calcium, and oxygen elements. The ratio of Ca/P was found to be 1.67. The crystal structure of lanthanum doped hydroxyapatite ceramics was analyzed by X-ray diffraction technique. X-ray diffraction patterns of the ceramics are shown in Fig. 4. X-ray patterns of the samples were indexed to hexagonal phase with the space group of $P6_3/m$ and cell constants, a = b = 9.4240 Å, and c = 6.8790 Å (JCPDS: 74-0565). As seen in Fig. 4, XRD intensities of undoped Hap is higher than those incorporated with lanthanum and the peaks became wider with La. This indicates that La is incorporated in to HaP [19].

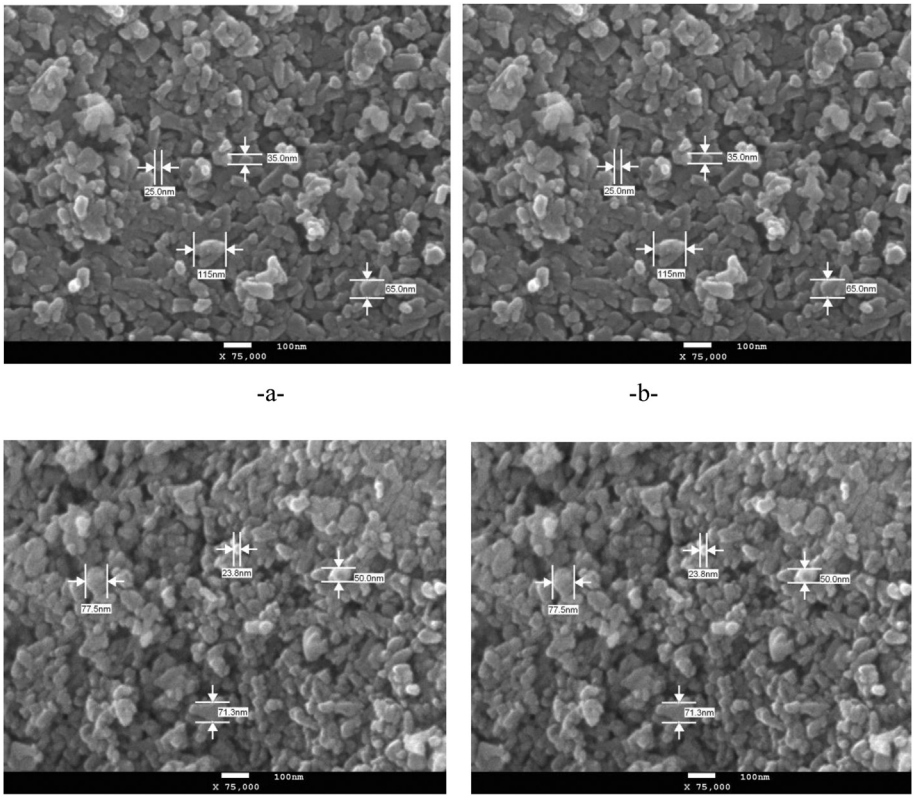

Fig. 2. SEM images of La doped HAp ceramics a) x = 0 b) x = 0.3 c) x = 04 d) x = 0.5.