Sol-gel synthesis and structural characterization of Fe doped barium titanate nanoceramics

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Abstract

Fe-doped barium titanate (BFe_xT) nanoceramics were successfully prepared by a simple sol-gel process, these materials are very interesting and could be a candidate for many optoelectronic applications. X-ray diffraction (XRD) patterns of the obtained samples, heat-treated at a quite low temperature (800 °C/3h) revealed that BFe_xT nanoceramics crystallized into a tetragonal phase perovskite structure. The occupation of the Ba and Ti sites by Fe in the $BaTiO_3$ lattice and the evolution of the different parameters (crystallite size, lattice parameters and strain) as functions of the Fe doping have been discussed in details using several characterization techniques including XRD and Fourier Transformation Infrared (FT-IR).

Keywords: Sol-gel, Barium titanate nanoceramics, Fe-doped barium titanate nanoceramics, X-ray Diffraction

Kulcsszavak: Szol-gél, bárium-titanát, nanokerámia, Fe-adalékolt bárium-titanát, röntgendiffrakció

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1. Introduction

The popularity of the research in the area of the high-tech ceramics is largely grown lately [1-31], since the discovery of the ferroelectricity phenomenon, barium titanate $BaTiO_3$ (BT) has been a fundamental member of the ferroelectric perovskite family. This compound has been used for a long time in many industrial sectors including full swing multilayer ceramic capacitors (MLCCs) [32], the realization of the ferroelectric random access memories (FRAMs) [33], thermistors manufacturing [34] and detection of polluting gases such as CO [35]. However, multilayer capacitors have long been at the forefront of $BaTiO_3$ applications. The $BaTiO_3$ nanoceramics can be synthesized by various methods such as solid-state reaction [36], hydrothermal synthesis [37], sol-gel [38] and co-precipitation method [39], compared to other methods, sol-gel is considered as a simple and easy method which can allows production of a high purity $BaTiO_3$ with very fine particles of controllable size, moreover, it is possible to easily modify the physical properties of BT since these are very sensitive to doping on the A-site (Ba_DTiO_3), B-site ($BaTi_DO_3$) and substitution on both sites (co-doping) $Ba_DTi_{D'}O_3$ which mainly depends on their ionic radii [40]. Recently, the doping of $BaTiO_3$ nanoceramics by 3d transition metals (Fe, Co, Mn, Ni...) have attracted much attention, 3d transition elements are more likely to be considered as dopants due to their electric and high magnetic properties. To date, a number of research works have reported the effect of the 3d transition metals on the structural and physicochemical properties of $BaTiO_3$ nanoceramics. Rani et al [41] have studied the effect of Fe doping on structural, magnetic and magnetoelectric properties of $BaTiO_3$ prepared via solid-state reaction route. Khirade et al [42] have reported the effect of Fe doping on the structural, optical and electrical properties of $BaTiO_3$ nanoceramics synthesis by sol-gel process. Maikhuri et al [43] have demonstrated the influence of A- and B-site substitution on the structural and magnetic properties of $BaTiO_3$. In the present work, Fe doped $BaTiO_3$ nanoceramics have been prepared using sol-gel method, the prepared samples were characterized using X-Ray Diffraction and FT-IR. The analysis of X-ray patterns was used to calculate the crystallite size, lattice parameters and lattice strain. The aim of this investigation is to study the effect of Fe dopant on the structural properties of $BaTiO_3$, In particular, the occupation of Ba and/or Ti sites by Fe in the $BaTiO_3$ structure.

2. Method of elaboration

Pure and Fe-doped $BaTiO_3$ were synthesized using sol-gel method, barium acetate trihydrate ($Ba(CH_3CO_2)_2.3H_2O$), Iron acetate $Fe(C_2H_3O_2)_2$ and titanium alkoxide $Ti[OCH(CH_3)_2]_4$ were used as precursors, lactic acid (CH_3CH (OH)COOH) was used as peptizing agent, acetic acid was added to dissolve Iron acetate, and distilled water as solvent. As shown in *Fig. 1*, the first step is to prepare a colloidal solution of TiO_2, to do this; the

titanium alkoxide was added to an aqueous solution of lactic acid and H_2O with continuous stirring at 70 °C. After 24 hours of reaction, the white precipitate obtained is changed into a clear homogeneous solution. In a second step, the colloidal solution produced was added in stoichiometric quantities to iron and barium acetates. The obtained transparent sol was converted to a translucent gel after stirring at 90 °C. Fine powders were prepared from the gel after drying at 90 °C and grinding. Finally, the nanopowders produced were calcined in air at a temperature of 800 °C for 3 h in a programmable oven. Phase identification of the prepared BFexT was performed using X-ray diffraction (Cu Kα radiation, λ = 1.5405980 Å) and Fourier transform infrared.

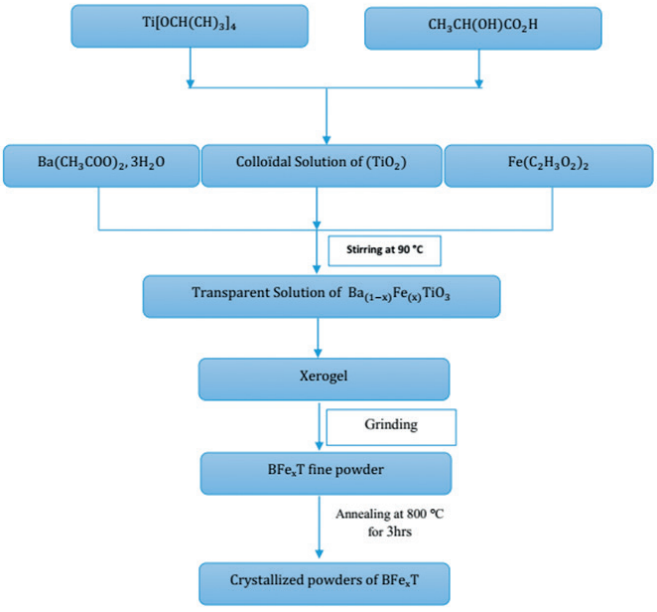

Fig. 1 *Flow chart of the preparation of Fe doped $BaTiO_3$ nanoceramics by sol-gel process*
1. ábra *A Fe-adalékolt $BaTiO_3$ nanokerámia szol-gél eljárással történő előállításának folyamatábrája*

3. Results and discussion

3.1 Structural studies

Fig. 2 shows the X-ray diffraction patterns of $Ba_{1-x}Fe_xTiO_3$ (x = 0, 1, 3%) samples prepared via sol-gel method. XRD patterns of pure and Fe-doped $BaTiO_3$ show a single tetragonal phase without any evidence of secondary phases. The XRD pattern of the samples can be indexed to the tetragonal perovskite structure with P4mm space group, which is in great concurrence with JCPDS No. 05-0626. An important fact revealed in *Fig. 2* is that the peak of (101) of $Ba_{1-x}Fe_xTiO_3$ (x=0.01) is shifted to higher 2θ angle compared to the pure $BaTiO_3$, while the peak of (101) of $Ba_{1-x}Fe_xTiO_3$ (x=0.03) is shifted to lower 2θ angle. The Peaks shifted to lower and higher angles indicate an increase and decrease in volume (V) of the unit cell respectively as shown in *Table 1*. Fe^{2+} has a smaller ionic radius (0.78 Å) compared to that of Ba^{2+} (1.35 Å). The slight shift of the peak positions in BFe1T with respect to $BaTiO_3$ is may be attributed to the Fe^{2+} ionic size difference, which could eventually replace the Ba^{2+} ions in the $BaTiO_3$ lattice [40] and leads to a reduction in the volume of the unit cell. In addition, the lower angle shift in the peaks of BFe3T can be assigned to the substitution of Fe^{2+} ion by Ba^{2+} and Ti^{4+} ions, but with a predominance of the Ti site occupancy in the $BaTiO_3$ lattice. In contrary, and because of the reason that the Fe^{2+} ionic radii is larger compared to Ti^{4+} (0.605 Å), it can be observed that the substitution of Ti-sites by Fe^{2+} create oxygen vacancies to neutralize the charge. Therefore, an increase in the unit cell volume is predicted as the unit cell parameters a and c have increased.

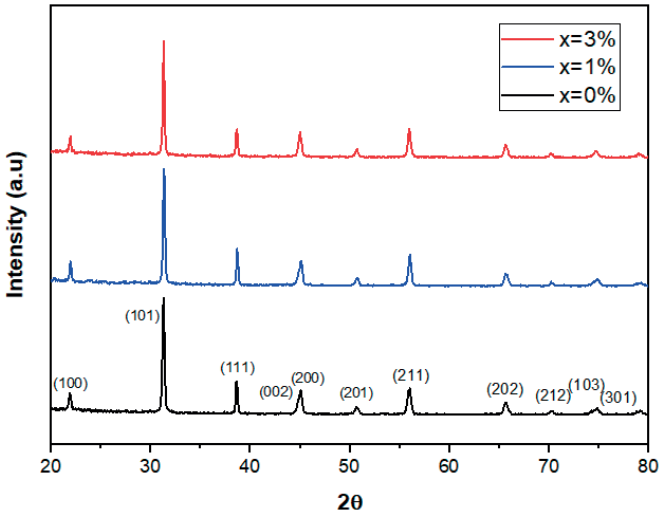

Fig. 2 *X-ray diffraction patterns of prepared Fe-doped barium titanate nanoceramics*
2. ábra *Az elkészített Fe-adalékolt bárium-titanát nanokerámiák röntgendiffraktogramja*

The crystallite size of the pure and Fe-doped $BaTiO_3$ was calculated from the X-ray diffraction patterns according to (101) peak using Debye-Scherrer formula [44]:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

Where D is the crystallite size, λ is x-ray wavelength, θ is the diffraction angle and β is full width at half maximum (FWHM) of the (101) peak. The average crystallite size was calculated by settling the highest intensity peak. As listed in *Table 1*.

Moreover, the lattice strain (ε) of all samples was estimated using the following equation [45]:

$$\varepsilon = \frac{\beta}{4\tan\theta} \quad (2)$$

The estimated lattice parameters a and c, unit cell volume (V), position of the peak (101), lattice strain and crystallite size are listed in *Table 1*. It was found that the average crystallite size (D) increased with Fe concentration x. However, the lattice constant (c) and lattice strain have been decreased after doping. The increase in crystallite size is due to the big difference between the ionic radius of Fe^{2+}, Ba^{2+} and Ti^{4+} [31, 42]. The shift in the position of (101) in the XRD patterns and the change in the volume of unit cell after doping reveal that Fe is totally soluble into $BaTiO_3$ lattice.

3.2 FT-IR analysis

The Fourier transform infrared spectroscopy (FT-IR) spectrums of $BaFe_xTiO_3$ (x=0, 1, 3%) are shown in *Fig. 3*. The corresponding spectra show absorption bands in the wave number ranging from 4000 – 450 cm^{-1}. The presence of a prominent peak at about 500 cm^{-1} in the pure $BaTiO_3$ spectrum corresponds to the vibration of Ti-O bond in the crystal lattice [46]. All the samples show a fingerprint of Ti-O and Ti-O-Ti bonds between 500 cm^{-1} and 750 cm^{-1} which is a molecular fingerprint of $BaTiO_3$. The wavenumber of the absorption peak of Ti–O bond increases after doping, the peak is shifted from 481.12 cm^{-1} in pure $BaTiO_3$ to 491.23 cm^{-1} in 3% Fe doped $BaTiO_3$ which indicates the change of the unit cell size. Moreover, the bands observed in the region from 1425 cm^{-1} to 1449 cm^{-1} could be attributed to $BaCO_3$ phase present in the prepared samples [47].

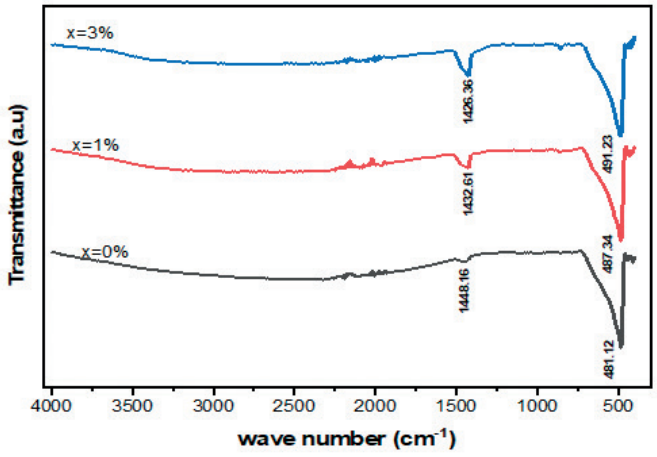

Fig. 3 FT-IR spectrums of pure and Fe-doped barium titanate at different Concentrations (x=0, 0.01 and 0.03)
3. ábra A tiszta és a Fe-adalékolt bárium-titanát FT-IR spektrumai különböző koncentráció esetén (x=0; 0,01 és 0,03)

x	a (Å)	c (Å)	V (Å³)	Position 2Θ of (101)	Crystallite size (nm)	Lattice strain
0	3.9943	4.0191	64.12245	31.31533	34.56	0.003883
0.01	3.9949	4.0164	64.01643	31.36792	36.38	0.003584
0.03	4.0013	4.0167	64.30898	31.33112	42.46	0.003159

Table 1 The position of the peak (101), lattice parameters, Volume of the unit cell, lattice strain and crystallite size of $BaFe_xTiO_3$ (x=0, 1 and 3%) nanoceramics
1. táblázat A csúcs helyzete (101), a rácsparaméterek, az egységcellák térfogata, a rácsos törzs és a $BaFe_xTiO_3$ (x=0; 1 és 3%) nanokerámia kristálymérete

4. Conclusion

To summarize, pure and Fe doped barium titanate nanoceramics were synthesized using sol-gel method, the obtained powders were calcined at 800 °C for 3h, the calcined samples were characterized using XRD and FT-IR spectroscopy. The XRD results confirm the presence of single tetragonal phase for both pure and Fe doped materials. This measurement reveals that the Fe^{2+} ion may replace Ba^{2+} for x=1% or the both Ba^{2+} and Ti^{4+} but with a predominance for the Ti-site in the case of x=3%. FT-IR measurements showed that the elaborated samples have perovskite structure, which is in a good agreement with XRD results. The modification in the structural properties after doping indicates that Fe has successfully doped into $BaTiO_3$ lattice.

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