



Dielectric, mechanical, and microstructural characterization of HA–BST composites

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Abstract

Hydroxyapatite (HA) is an excellent inorganic biomaterial and has various applications because its structure is similar to the inorganic matrix of human bone and dental enamel. In this present study, the HA–(Ba_{0.7}Sr_{0.3})TiO₃; HA–BST composites were synthesized by a solid state reaction technique. The phase, densification, microstructure, mechanical and dielectric properties of HA–BST composites sintered at 1350 °C were investigated. The HA–BST composite showed an increase in densification, Vickers hardness, Young's modulus and fracture toughness with maximum value as 3.42 g/cm³, 5.01 GPa, 161.70 GPa, and 1.48 MPa m^{1/2}, respectively. The average grain size was decreased from 12.09 μm for a reference HA sample to 1.14 μm for the 30 wt% BST in HA–BST composites. The dielectric constant as a function of temperature and frequency tend to increase with increasing BST contents.

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1. Introduction

Many intensive studies have investigated the properties of calcium phosphate with the objective of using in orthopedic and dental applications in the future. The investigation revealed several interesting properties such as good bioactivity, biocompatibility, high wear resistance, osteoconductivity and the mineral component similarity with nature mineral in bone and teeth [1–6]. However, calcium phosphate has many forms and phase such as hydroxyapatite (HA), biphasic calcium phosphate (BCP), beta tricalcium phosphate (β-TCP), alpha tricalcium phosphate (α-TCP), and unsintered apatite (AP), which are dependent on fabrication process conditions [7]. Recently, electrical properties such as dielectric, ferroelectric and conductivity of HA have been of interest because these properties are linked to bioactivity property [3,8–11]. These relations are interesting for many researches. Since Yamashita et al. [9] reported that the induced surface charge of polarized HA can

significantly accelerate or decelerate bone-like crystal growth on the surface deposition in simulated body fluid (SBF), depending on electric polarization conditions, types and volume of surface charge which are connected with dielectric properties. For improving electrical and mechanical properties, the modified HA was focused to study by doping and synthesizing composites with HA such as Fe-doping [12], Cd-doping [7] and HA–ZrO₂ [13], HA–Sr [14], HA–BaTiO₃ [15], HA CaTiO₃ [16]. Especially, HA–BaTiO₃ and HA–CaTiO₃ have been presented as a good substrate for apatite growth with high osseointegration and osteoconction [15,17]. Therefore, the properties of hydroxyapatite–perovskite composites, only non-toxic chemical compound, are highly potential for HA application due to their high polarizability and reasonable biocompatibility.

BSTs (Ba_xSr_{1-x}TiO₃, 0 < x < 1) are ferroelectric materials that have high dielectric, low dielectric loss and spontaneous polarization. The temperature of changing ferroelectric phase to paraelectric phase was determined by the substitution of Ba²⁺ by Sr²⁺ in the A-site of perovskite structure (ABO₃). Among BSTs, Ba_{0.7}Sr_{0.3}TiO₃ has a phase transition temperature of about 40 °C,

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which is a high dielectric constant ($\epsilon_r \sim 13,000$) [18–20]. Therefore, this material has high dielectric and still ferroelectric phase at close to normal human body temperature.

Detailed literature survey reveals the new bone formation has been found to be significantly improved on polarized surface. For this significant context, many electroactive biomaterials have been developed [15,17]. To our literature reviews, a composite of HA–Ba_{0.7}Sr_{0.3}TiO₃; HA–BST has not yet been synthesized and their properties have not been reported. Consequently, the effects of BST in HA–BST composites were investigated and discussed compared with other previous reports in this work.

2. Experimental

The nanocrystallinity of HA was performed by peroxide-based route, with control pH at 9. Calcium nitrate solution (Ca(NO₃)₂ · 4H₂O, Sigma-aldrich), ortho-phosphoric acid 85% (Merck) and hydrogen peroxide solution 30% (Qrec) were used as the starting reagent. During the chemical reaction, the solution pH of the control was at a constant value of 9 by adding ammonium hydroxide solution 30% (J.T. Baker). The obtained powders of HA are in the nano-range (~60 nm). The BST powders were prepared by conventional solid state processing. The reagent grades of BaCO₃ (99.9%, Sigma-aldrich), SrCO₃ (99.9%, Sigma-aldrich), and TiO₂ (99.9%, Sigma-aldrich) were used as starting material. The mixed powers were calcined at 1150 °C for 4 h. The obtained powers had perovskite structure and particle size about 0.9 μm. For the preparation of the composite, the HA–xBST composites were prepared by adding various amounts of BST contents with $x=0, 5, 10, 15, 20, 25,$ and 30 wt% in HA. The mixed powers were ball milled in isopropanol for 24 h. This slurry was dried and sieved to form homogeneous powders. These mixed powders were then granulated using polyvinyl alcohol (PVA) 3 wt% organic binder and formed into discs, typically 10 mm in diameter and 2 mm in thickness by cold uniaxial pressing. The resulting discs were sintered in air at a temperature of 1350 °C in a step of 100 °C/hour (with soaking time of 3 h). The polish of these ceramics was examined via an X-ray diffractometer with Cu K α radiation to check the phase formation. Density of samples was measured using the Archimedes method. The surface morphology and elemental composition of the samples were then analyzed using a scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX). Average grain size was determined using the intercept method. Mechanical properties were studied on a polished surface using a micro-hardness tester. For dielectric measurements, the ceramics were coated with silver paste electrodes on both sides of the pellets and the dielectric property was measured using an impedance analyzer.

3. Results and discussion

Fig. 1 shows the XRD patterns of a reference HA, and HA–BST composites ceramics. All studied samples were analyzed by comparison of peak location to JCPDS database

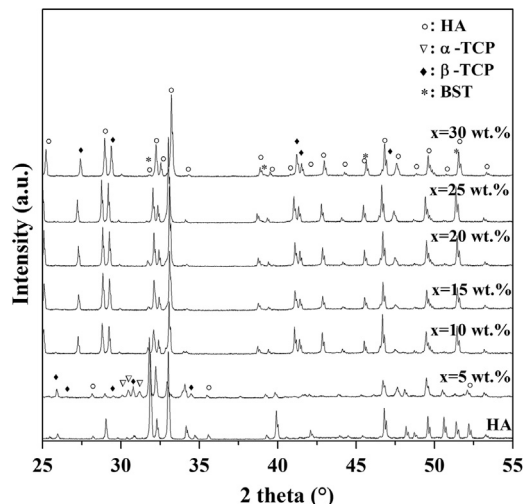
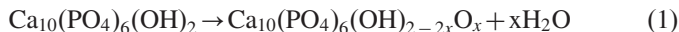
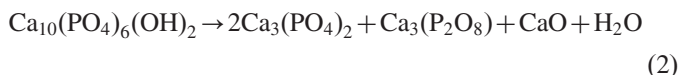


Fig. 1. XRD analysis of HA–BST composite as well as a reference HA.

reference pattern spectra. The HA, α -TCP, and β -TCP phases were detected in a reference HA and all HA–BST composites samples with reference numbers 09-0432, 09-0348, and 09-0169 for HA, α -TCP, and β -TCP phases, respectively. It should be noted that the decomposition of HA was observed in reference HA and HA–BST composites. This result is in agreement with previous reports [21,22]. In general, HA phase transformation during thermal treatment consists of two processes, which are dehydroxylation and decomposition. Dehydroxylation to oxyhydroxyapatite proceeds at temperature about 850–900 °C through the following equation [21]:



For above 900 °C, the decomposition process of HA can be found according to the reaction in the following equation [22]:



However, the perovskite structure of BST in HA–BST composites was shown at $x > 5$ wt%, according to JCPDS reference no. 01-089-0274. The intensity of BST structure peaks is increased associated with BST contents in HA–BST composites. However, phases of BST with $x \leq 5$ wt% cannot be observed due to the infinitesimal of BST contents were nearly a limit of this XRD instrument. The morphology and composition of BST were analyzed by SEM and EDX as show in Fig. 3. The morphology form of SEM showed clearly two phases in the matrix of the HA–BST surface, i.e., HA phase and BST phase. The phase of HA and BST was determined by EDX analysis. The spectra corresponding to HA showed a peak corresponding to that of phosphorus and calcium as shown in $x=15$ wt% BST. The EDX pattern of small grain size in a matrix of HA–BST composites at $x=10$ wt% showed a peak corresponding to the major elements present in composites (Ba, Sr, and Ti) as expected from BST grain.

In this work, reference HA and HA–BST composites samples were sintered with different sintering temperatures for optimum sintering temperature condition. All samples were

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