

# Pyrochlore structure and dielectric properties of bismuth zinc niobate thin films prepared by RF sputtering

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## ABSTRACT

$\text{Bi}_{1.5}\text{Zn}_{1.0}\text{Nb}_{1.5}\text{O}_7$  (BZN) thin films with thickness from 60 nm to 200 nm were prepared by radio-frequency magnetron sputtering and post-annealed from 550 °C to 650 °C. The x-ray diffraction results indicated that the BZN thin films possessed a cubic pyrochlore phase. The BZN thin films exhibited thickness-independent dielectric properties with dielectric constant of ~180 and low loss tangent less than 1% at 10 kHz as the film thickness decreased to 60 nm. The BZN thin films with thickness of 200 nm and post-annealed at 650 °C had a tunability of 32.7% at a DC bias field of 1.5 MV/cm. The results suggest that the BZN thin films have promising applications on the embedded capacitors, tunable devices and energy storage devices.

## 1. Introduction

The increasing in functionality, computing speed and portability in consumer electronics has put a lot of pressure on designers and manufacturers to integrate more circuitry into smaller chips while maintaining or even improving the overall performance of the product. Under these circumstances, miniaturized devices with improved performance are of urgent need. Capacitors, especially the multilayered ceramic capacitors (MLCCs) are widely used, and they are the major components in term of size and numbers among passive devices. But one of the inevitable drawbacks of MLCCs is the relative large size, especially for the application in the printed circuit boards (PCB) [1]. Preparing multilayered capacitors with high performance dielectrics via thin-film techniques is one of the widely studied methods to miniaturize the size of the devices, and at the same time to maintain or even to increase the capacitance [2–6].  $\text{Bi}_{1.5}\text{Zn}_{1.0}\text{Nb}_{1.5}\text{O}_7$  (BZN) is a pyrochlore dielectric and has captured a lot of researchers' attention for the embedded capacitor application owing to its moderate dielectric constant of 150–220 and low dielectric loss. BZN has been extensively and detailed studied for capacitors, electric field tunable devices and microwave dielectric devices [7–19]. Besides, BZN thin films show very high breakdown strength, which makes it a promising candidate for energy storage dielectrics [9,20]. Various thin film deposition techniques can be applied for the fabrication of BZN thin films, like metal organic deposition, pulse laser deposition, RF magnetron sputtering and aerosol deposition [9–11,14,21–24]. Ren et al. reported the cubic BZN thin films deposited by metal organic deposition and annealed at

750 °C had a dielectric constant of 150 at 10 kHz and a negative temperature coefficient of capacitance of –400 ppm/°C [10]. Zhang and Ren reported BZN thin films post-annealed in  $\text{O}_2$  atmosphere exhibited an optimized dielectric constant of 202 and loss tangent of 0.0002 [25]. Liu et al. reported cubic BZN thin films deposited by RF magnetron sputtering with dielectric constant up to 220, the electric-field tunability of 55% at 2.4 MV/cm and very low dielectric loss tangent less than 0.0005 [23]. Cao et al. investigated the dielectric properties of BZN thin films with different thicknesses by PLD [26]. Most of reported research work focused on the BZN thin films with thickness of several hundred nanometers. But the structures and dielectric properties of BZN thin films within 100 nm were still lack of detailed research.

In this work,  $\text{Bi}_{1.5}\text{Zn}_{1.0}\text{Nb}_{1.5}\text{O}_7$  (BZN) pyrochlore thin films with thickness from 60 nm to 200 nm have been successfully deposited at 550 °C by radio frequency magnetron sputtering and post-annealed from 550 °C to 650 °C. The structures and dielectric properties of BZN thin films have been investigated.

## 2. Experimental procedure

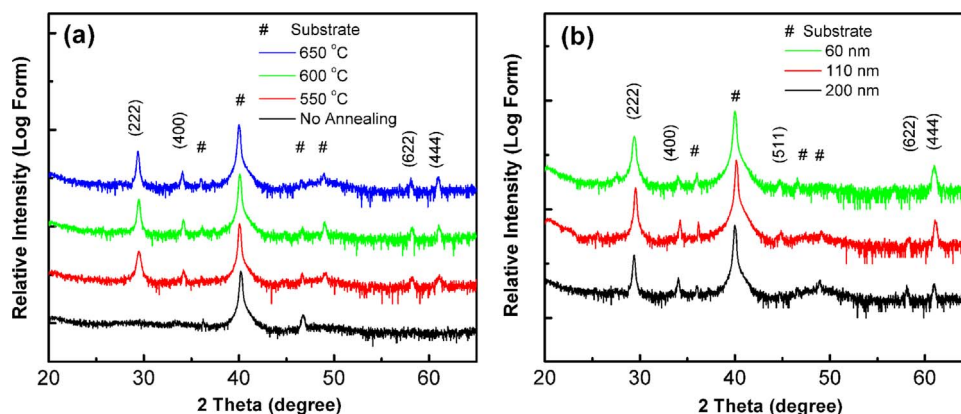
BZN thin films were deposited on Pt/TiO<sub>2</sub>/SiO<sub>2</sub>/Si(100) substrate at 550 °C from a cubic  $\text{Bi}_{1.5}\text{Zn}_{1.0}\text{Nb}_{1.5}\text{O}_7$  (BZN) ceramic target by RF magnetron sputtering. The BZN ceramic target was made via a conventional solid-state reaction method. According to the stoichiometric ratio of  $\text{Bi}_{1.5}\text{Zn}_{1.0}\text{Nb}_{1.5}\text{O}_7$ , the raw material of Bi<sub>2</sub>O<sub>3</sub>, Nb<sub>2</sub>O<sub>5</sub> and ZnO were weighted and mixed. The mixed powder was calcined at

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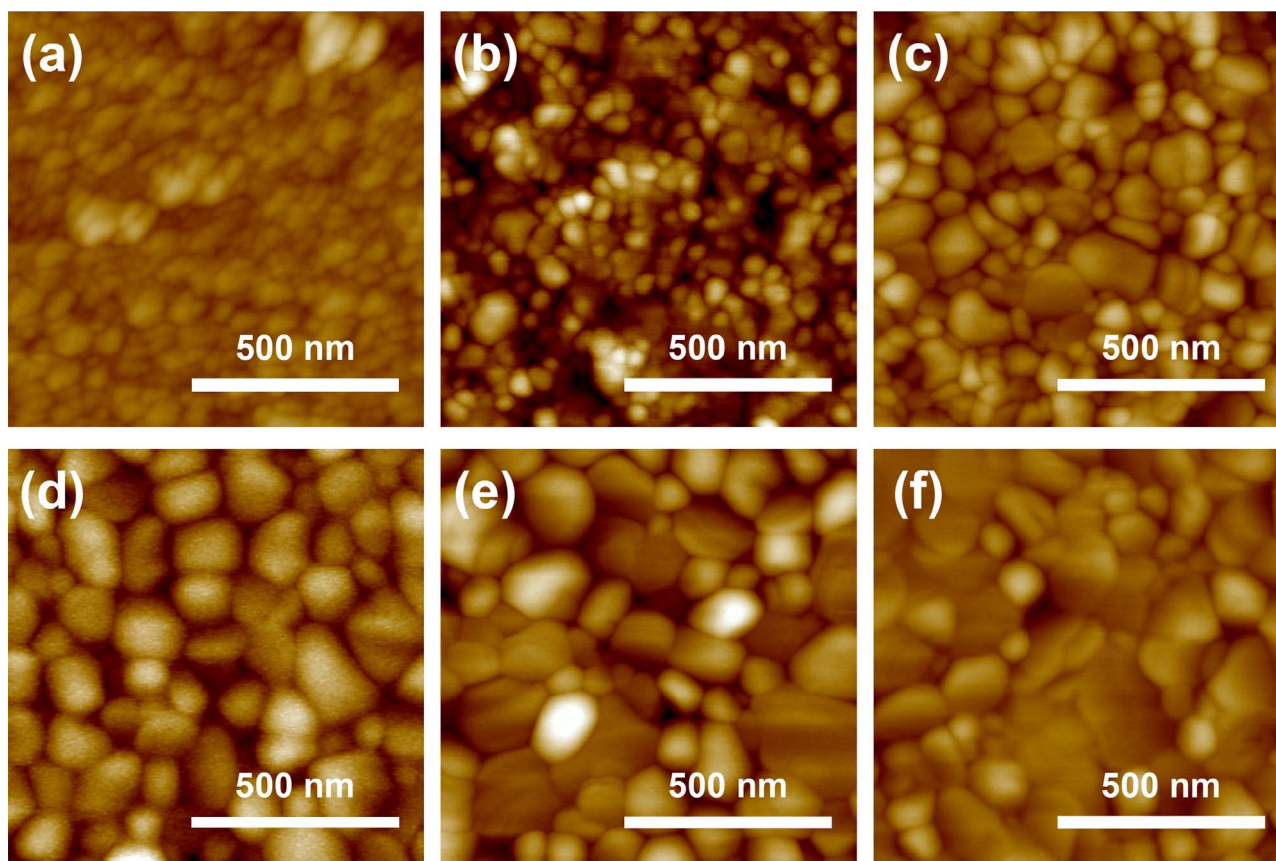
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**Fig. 1.** XRD patterns of BZN thin films, (a) post-annealed at different temperatures and with thickness of 200 nm, and (b) with different thicknesses and post-annealed at 650 °C.



**Fig. 2.** AFM images of BZN thin films, (a)–(d) with thickness of 200 nm and post-annealed at (a) without post-annealing, (b) at 550 °C, (c) at 600 °C, (d) at 650 °C, and (e) with thickness of 110 nm and post-annealed at 650 °C, (f) with thickness of 60 nm and post-annealed at 650 °C.

800 °C for 3 hrs. The calcined powder was shaped in to a disc of 120 mm in diameter by dry-pressing. The target green body was sintered at 1000 °C for 3 h. The target could be put into use after cleaning with alcohol in an ultrasonic bath. The sputtering chamber was evacuated to a base pressure of  $6.0 \times 10^{-4}$  Pa. The mixture gas of Ar and O<sub>2</sub> with Ar/O<sub>2</sub> flow was set at a ratio of 85/15 and the gas pressure was kept at 2.6 Pa for the deposition ambient. The RF power density of 1.4 W/cm<sup>2</sup> was applied on the target. The films' thickness was controlled by regulating the deposition time and was in a range from 60 nm to 200 nm. BZN thin films were post-annealed *ex-situ* in a rapid thermal process furnace, at various temperatures ranging from 550 °C to 650 °C in the air for 20 min after the deposition.

The x-ray diffractometer (XRD, D/Max-2400, Rigaku, Japan) was applied to examine the phase structure of BZN thin films. The cross-sectional morphologies of the thin films were characterized by a field-

emission scanning electron microscope (FE-SEM, Quanta 250, FEI, USA). The surface morphologies and roughness of the thin films were studied by an atomic force microscope (AFM, Dimension ICON, Bruker, USA). Pt top electrodes were deposited onto BZN thin films by RF magnetron sputtering at room temperature via a lithographic lift-off processing for the electric properties measurement. The dielectric properties were investigated by a precision LCR meter (E4980A, Agilent Technologies, USA). The leakage current behavior was obtained by a precision source/measure unit (B2901A, Keysight, USA). The elements ratio of BZN thin films was acquired by an electronic probe microanalysis (EPMA, JXA-8100, JEOL, Japan).

### 3. Results and discussion

Fig. 1 shows XRD patterns of BZN thin films on Pt-coated silicon

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