

# Structural and Dielectric Properties of Microwave Processed PZT-NZF Composite

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**Abstract:** For present work, we are reporting a study on structural and dielectric properties of lead zirconate titanate and nickel zinc ferrite (PZT-NZF) composite having compositional formula  $0.9\text{PbZr}_{0.65}\text{Ti}_{0.35}\text{O}_3 - 0.1\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$  synthesized by solid state reaction route using microwave sintering. The composite sample was sintered at  $1150^\circ\text{C}$  for 10 minutes. The presence of two phases (PZT and NZF) was confirmed by using x-ray diffraction technique. Experimental density of sintered pellet was calculated using Archimedes principle. Dielectric properties were studied as a function of temperature ( $35-500^\circ\text{C}$ ) at 1, 10 & 100 kHz frequency and function of frequency (100 Hz- 1 MHz) at room temperature.

**Keywords:** Dielectric; Diffraction; Ferroelectric; Ferrite; microwave.

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## 1. INTRODUCTION

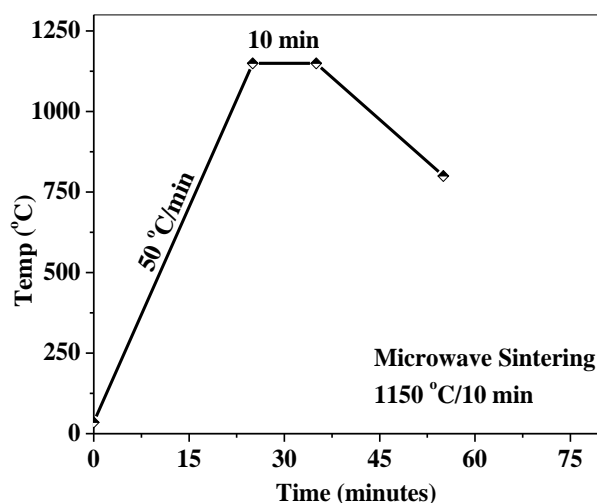
The growing interest in microwave sintering over conventional sintering is due to the fact that in case of microwave sintering, energy is delivered directly to materials through molecular interaction with the electromagnetic field. This absorption of microwave energy is followed by heating of the material as a whole by the conversion of electromagnetic energy into thermal energy [1], [2], [3], [4]. It results in many attractive advantages such as volumetric and rapid heating of material, less thermal energy losses, fine microstructure and reduced processing time. Especially for Lead zirconate titanate (PZT) ceramics, there is a problem of lead loss due to lengthy sintering soaking time during conventional sintering and this lead loss can be minimized by microwave technique.

Lead zirconate titanate ( $\text{PbZr}_{1-x}\text{Ti}_x\text{O}_3$ , PZT) with varying Zr/Ti ratio, exhibits interesting behavior in piezoelectric, ferroelectric and dielectric properties and used in number of applications such as non-volatile memories, smart sensors, actuators, electro-optic devices and high energy capacitors [5], [6]. Some polycrystalline materials like nickel zinc ferrites show ferromagnetic behavior and have been used in many applications such as microwave devices and power transformers [7]. There are many multifunctional devices like sensors for magnetic field measurements, transducers for magnetoelectric conversion and magnetoelectric memory devices etc. require presence of both ferroelectric and ferromagnetic properties simultaneously that show magnetoelectric effect [8]. Thus for present work, ferroelectric-ferrite based composite was synthesized.  $\text{PbZr}_{0.65}\text{Ti}_{0.35}\text{O}_3$  (PZT) composition for ferroelectric phase was selected because of high piezoelectric coefficient, high electromechanical coupling constant and low dielectric loss [9], [10] and  $\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$  (NZF) having high resistivity, high piezomagnetic coefficient and low eddy current losses was selected as a ferrite phase [11].  $0.9\text{PbZr}_{0.65}\text{Ti}_{0.35}\text{O}_3 - 0.1\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$  composite was sintered using microwave technique in order to minimize PbO loss and detailed structural and dielectric properties are presented here.

## 2. EXPERIMENTAL

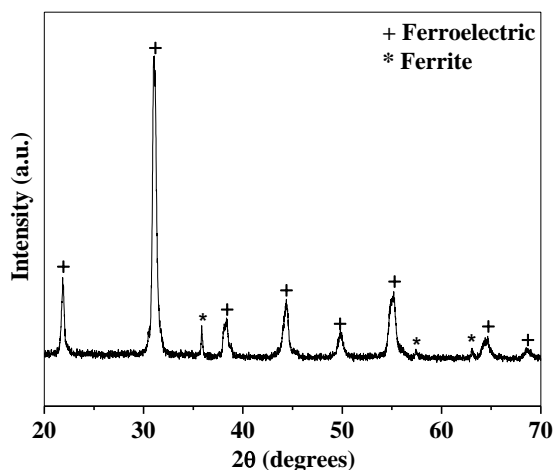
Individual phases ( $\text{PbZr}_{0.65}\text{Ti}_{0.35}\text{O}_3$ /PZT and  $\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ /NZF) were synthesized by conventional solid state reaction route. AR grade PbO,  $\text{ZrO}_2$  and  $\text{TiO}_2$  for PZT were weighed in the required molar proportions. An excess of 2% of PbO was added to compensate for lead loss during sintering. The mixing process was carried out by ball milling in distilled water using zirconia balls as the milling media. The dried powder was calcined at  $800^\circ\text{C}$  for 4 hours in a conventional furnace.

After ball milling, the powder mixture was re-calcined at 1000°C for 4 hours in a conventional furnace again. AR grade NiO, ZnO and Fe<sub>2</sub>O<sub>3</sub> were used as raw materials for synthesis of NZF. The powder mixture was ball milled and then calcined conventionally at 1000°C for 4 hours. A small amount of MnO<sub>2</sub> (0.5% by weight) was added to increase the resistivity of the ferrite phase. The powder mixture was ball milled again and then re-calcined at 1100°C for 4 hours (conventionally again). The composite with compositional formula 0.9PbZr<sub>0.65</sub>Ti<sub>0.35</sub>O<sub>3</sub> – 0.1Ni<sub>0.8</sub>Zn<sub>0.2</sub>Fe<sub>2</sub>O<sub>4</sub> was prepared by mixing the two phases in distilled water using ball mill. After drying, a small amount of diluted polyvinyl alcohol (2-3 drops) was added to the powder mixture as a binder which was pressed into circular discs of 1 mm thickness and 15 mm diameter. The pellets were finally sintered at 1150°C for 10 minutes with a heating rate of 50° C/minute in a programmable microwave furnace. The temperature-time graph for microwave sintering is shown in Figure 1.



**Figure 1: Temperature-time graph for Microwave Sintering**

The experimental density of sintered pellet was determined using Archimedes principle and found to be 7.0 g/cc. X-ray diffraction (XRD) patterns were recorded at room temperature using a Philips XPERT-PRO Diffractogram with a Cu  $k_{\alpha}$  ( $\lambda = 1.5406 \text{ \AA}$ ) in a range of Bragg angles  $2\theta$  ( $20^{\circ} \leq 2\theta \leq 70^{\circ}$ ). To measure the dielectric properties, the sintered pellet was ground and then electroded properly using silver paste on flat surfaces by heating at 400°C for 30 minutes. The dielectric properties as a function of temperature (35-500°C) at 1 kHz, 10 kHz and 100 kHz and function of frequency (100 Hz-1 MHz) at room temperature were measured using an Agilent 4284A LCR meter.



**Figure 2: XRD pattern for 0.9PbZr<sub>0.65</sub>Ti<sub>0.35</sub>O<sub>3</sub> – 0.1Ni<sub>0.8</sub>Zn<sub>0.2</sub>Fe<sub>2</sub>O<sub>4</sub> composite**

### 3. RESULTS AND DISCUSSION

Typical XRD pattern of  $0.9\text{PbZr}_{0.65}\text{Ti}_{0.35}\text{O}_3 - 0.1\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$  composite is shown in Figure 2 which shows well defined peaks. The occurrences of peaks that are characteristics of perovskite and spinel structure confirm the coexistence of two phases (PZT and NZF) in composite. No extra peak was found.

Figure 3 represents the variation of dielectric constant and loss tangent as a function of temperature at three different frequencies (1, 10 and 100 kHz) for microwave sintered composite sample. It is observed that as temperature increases, the value of dielectric constant increases and passes through a maximum (at Curie temperature,  $T_c$ ) and then decreases. The position of dielectric maximum does not change with change in frequency. The increase in loss tangent at higher temperatures is caused by a corresponding conductivity increase [12] due to the presence of ferrite phase (NZF). Frequency dependence of dielectric constant and loss at room temperature is shown in Figure 4. The value of dielectric constant and loss decreases with increase in frequency. The higher values at low frequencies are due to the presence of all types of polarization mechanisms.

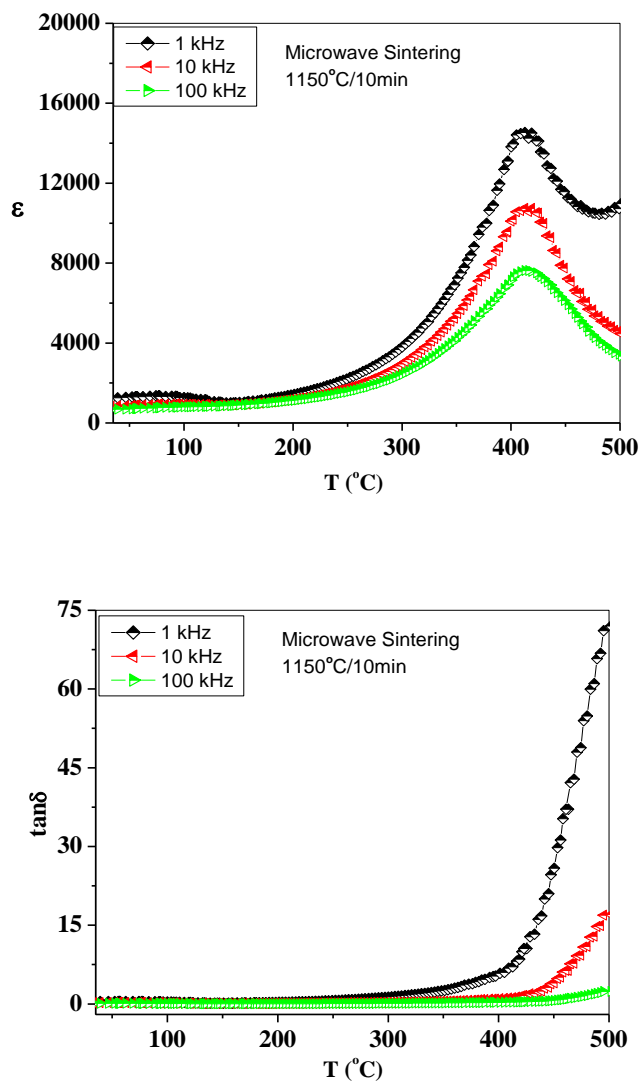
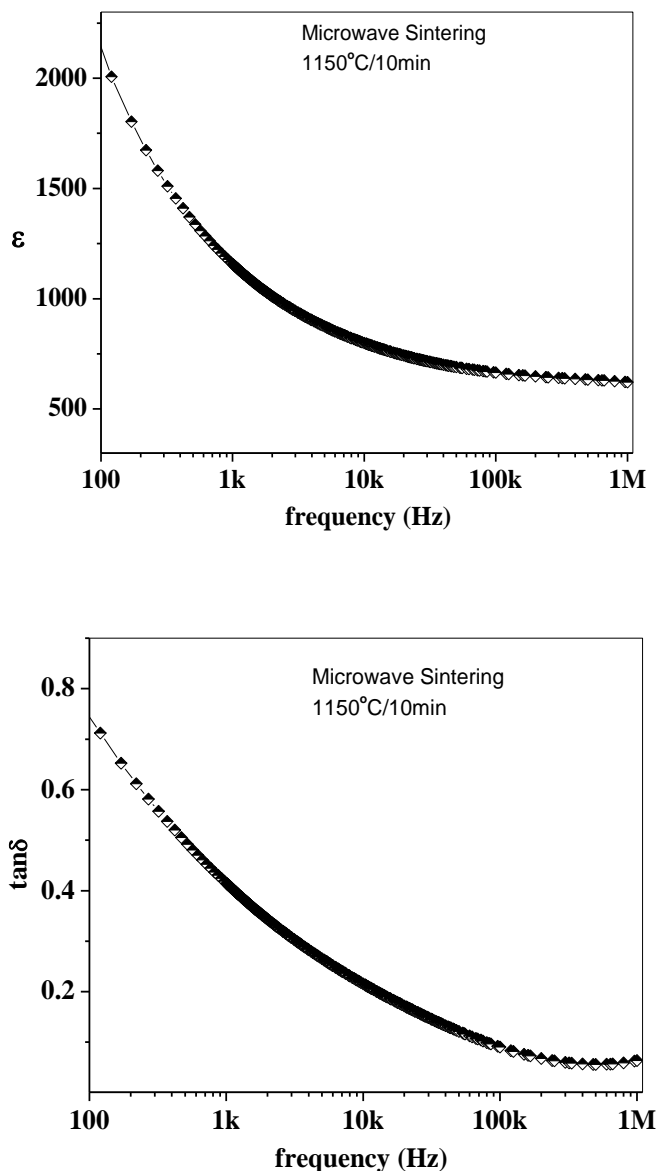


Figure 3: Temperature dependence of dielectric constant and loss for  $0.9\text{PbZr}_{0.65}\text{Ti}_{0.35}\text{O}_3 - 0.1\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$  composite



**Figure 4: Frequency dependence of dielectric constant and loss for  $0.9\text{PbZr}_{0.65}\text{Ti}_{0.35}\text{O}_3 - 0.1\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$  composite**

#### 4. CONCLUSION

$0.9\text{PbZr}_{0.65}\text{Ti}_{0.35}\text{O}_3 - 0.1\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$  composite was prepared by conventional solid state reaction route using microwave sintering. Structural and dielectric properties of the sample have been studied. Presence of individual phases was confirmed by XRD analysis.

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