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Preparation and Study of Dielectric and Electrical Conductivity of $Ba_5NdTi_3V_7O_{30}$ Ceramics

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Abstract

$Ba_5NdTi_3V_7O_{30}$ is a tungsten bronze structured ceramic sample, prepared by Solid State reaction route at high temperature (950˚C). The room temperature XRD analysis confirms orthorhombic crystal structure of the compound. Dielectric peak is observed at ~460˚C showing the transition of the compound from ferroelectric to paraelectric phase. Appearance of hysteresis loop confirms the existence of ferroelectric properties in the materials. Different values of activation energy in different temperature regions of the ac conductivity versus inverse absolute temperature graph exhibit mixed type of conduction process in the compound (*i.e.*, ionic-polaronic and space charge generated from the oxygen ion vacancies).

Subject Areas

Electrochemistry

Keywords

Solid-State Reaction, X-Ray Diffraction, Dielectric Properties, Tungsten Bronze Structure

1. Introduction

Materials with various structural forms have always hypnotized the Materials researchers for their massive implementation in Scientific and industrial research. The development of nanoscience & technology enable a step forward to

intensify the physical properties suitable for a broad field of challenging applications.

Materials with tungsten-bronze (TB) structure belong to family of dielectric materials, which exposes many exciting properties like ferroelectric, pyroelectric, piezoelectric, and nonlinear optical properties for various devices applications, such as transducers, actuators, capacitors, and ferroelectric random access memory [1]-[6].

The TB structure compose of a framework of distorted BO_6 octahedral sharing corners with three different types of interstices (A, B and C) available for different array of cations filling in a general formula $(A_1)_2(A_2)_4(C)_4(B_1)_2(B_2)_8O_{30}$. The substitution of different ionic size at the above-mentioned sites plays significant role in tailoring various physical properties of the materials. This paper reports on the preparation and study of dielectric and electrical conductivity of $Ba_5NdTi_3V_7O_{30}$.

2. Experimental Details

2.1. Material Preparation

The polycrystalline compound $Ba_5NdTi_3V_7O_{30}$ (BNTV) was prepared using high purity (>99.9%) ingredients; $BaCO_3$, Nd_2O_3, TiO_2, V_2O_5 by Mixed Oxide Method. These materials were mixed in appropriate amount satisfying the stoichiometry of $Ba_3Sr_2NdTi_3V_7O_{30}$, with formula;

$$5BaCO_3 + 1/2Nd_2O_3 + 3TiO_2 + 7/2V_2O_5 = Ba_5NdTi_3V_7O_{30} + 5CO_2$$

The ingredients were mixed in an agate mortar in air atmosphere for 3 h, followed by wet (methanol) condition to get homogeneous mixture. The mixture was then calcined in an alumina crucible starting from 700˚C in steps of 50˚C and was found to be calcined at an optimized temperature and time (950˚C, 12 h). This process was repeated till the formation of single-phase compound was confirmed by X-ray diffraction technique. After mixing the calcined powder with polyvinyl alcohol (PVA) as binder, cylindrical pellets of 10 mm diameter and 1 - 2 mm in thickness were made by a hydraulic press at a pressure of ~ 5×10^6 N/m^2. The pellets were then sintered in an air atmosphere at an optimized temperature and time (950˚C, 12 h) The pellets were then polished to make their faces flat and parallel and finally coated with high purity conductive silver paint, and dried at 150˚C for 2 h before carrying out electrical measurements to make them moisture free.

2.2. Material Characterization

The structure of the material was studied from X-ray diffraction (XRD) and Scanning electron micrograph. The room temperature.XRD pattern of the material was obtained in a wide range of Bragg's angle 2θ ($20^\circ \leq 2\theta \leq 75^\circ$) at a scanning speed of 3° min^{-1} by an X-ray diffractometer (Rigaku, Miniflex) with CuKα radiation (λ = 1.5405 Å) Using high-resolution scanning electron microscope (SEM: JOEL-JSM model: 5800F), the surface morphology of the sample

was studied. The dielectric measurement was done in a wide range of temperature (33˚C - 500˚C) and frequency (100 Hz - 1 MHz), using a computer-controlled impedance analyzer (PSM 1735, model: N 4L). Then (P~E) hysteresis loop was recorded on the poled sample at room temperature using a high precision workstation (M/S-Radiant Technologies, Inc. USA).

3. Results and Discussion

3.1. Structural Analysis

The XRD pattern of the sample shown in **Figure 1** confirms the formation of a single-phase new compound. The reflection peaks were indexed and the lattice parameters were determined in various crystal systems and with cell configurations using computer software "POWDMULT" [7]. A suitable orthorhombic unit cell with lattice parameters: a = 23.0868(26) Å, b = 4.2284(26) Å, c = 6.3899(26) Å were chosen. The crystallite size of the sample was evaluated from the broadening of the peaks ($\beta_{1/2}$) using Scherrer's equation [8]; $P = K\lambda/\beta_{1/2}\cos\theta_{hkl}$, where K = constant = 0.89, k = 1.5405 Å and $\beta_{1/2}$ = peak width of the reflection at half height. The crystallite size of the compound was observed to be 8 nm.

The room temperature scanning electron micrograph of the compound shown in **Figure 1** (inset) resembles polycrystalline arrangement of the material with grains of irregular in shape and size distributed non-uniformly and densely over the entire surface of the sample. A similar microstructure is also observed in some of our materials of this family [9] [10]. The grain size of the sample from histogram (**Figure 1** (right)) was measured to be in the range of 220 nm.

3.2. Dielectric Analysis

The temperature dependent relative dielectric constant (ε_r) and loss tangent (tan δ) (inset) of $Ba_5NdTi_3V_7O_{30}$ compoundis shown in **Figure 2** at some selected frequencies. **Figure 2** describes the decreasing trend of both ε_r and tan δ (inset) with increase in frequency which is exhibited by polar dielectrics. The compound

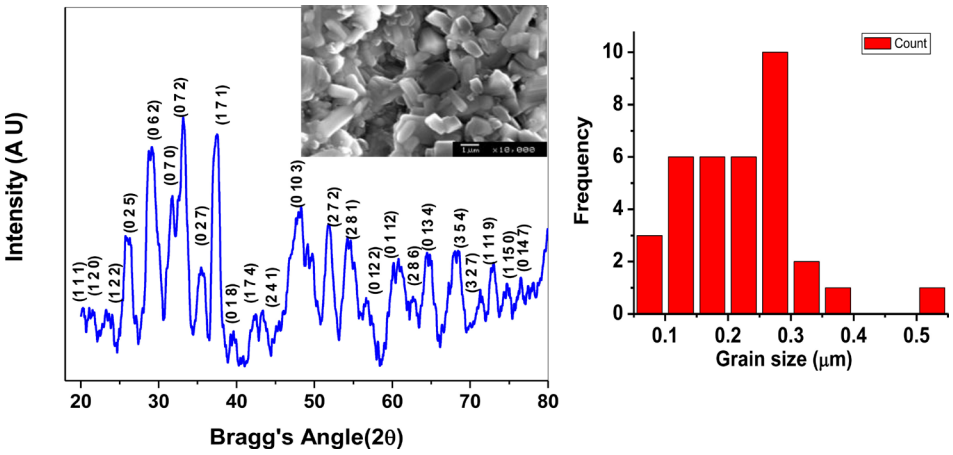

Figure 1. Room temperature XRD pattern, SEM (inset) and histogram of $Ba_5NdTi_3V_7O_{30}$.

has a frequency independent dielectric anomaly at ~460˚C indicating the possible occurrence of ferroelectric-paraelectric phase transition. The value of ε_r is more at low frequencies confirms the presence of all types of polarization whereas at high frequencies it is mainly due to the contribution of electronic polarization [11].

The value of loss (tanδ) is observed to be low indicating good quality of the material. The value of tanδ is observed to be increased at higher temperature region (**Figure 2** inset) caused by the intensifying conductivity for setting up of space charge polarization, and also due to trimming in ferroelectric domain wall's contribution at high temperature [11].

3.3. Conductivity Study

The variation of a.c. conductivity (σ_{ac}) as a function of inverse temperature at two different frequencies (50 kHz and 500 kHz) shown in **Figure 3**.

Figure 3 obeys the Arrhenius relation: $\sigma_{ac} = \sigma_0 \exp(-E_a/k_BT)$, where the symbols have their usual meanings. The plot is divided into two distinct regions as I in high temperature and II in low temperature regions for both the frequencies. From calculation, the activation energy (Ea) are found to be 0.105 eV and 0.051

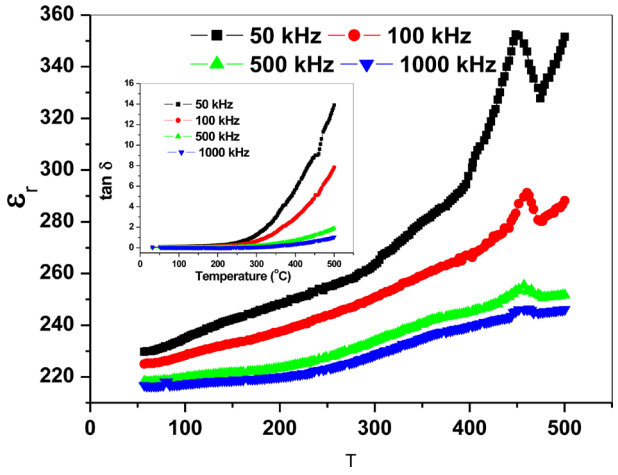

Figure 2. Variation of ε_r and tanδ (inset) with temperature of $Ba_5NdTi_3V_7O_{30}$ at some selected frequencies.

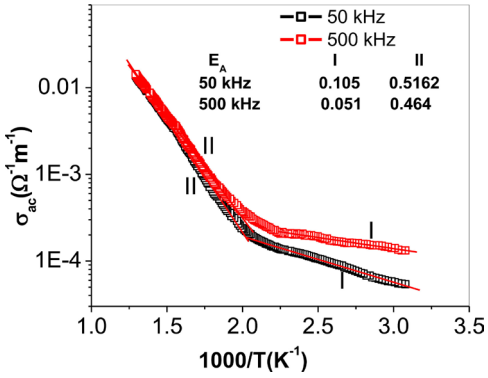

Figure 3. Variation of σ_{ac} with 1000/T of $Ba_5NdTi_3V_7O_{30}$, at two selected frequencies.

eV in region-I and 0.5162 eV and 0.464 eV in region-II which are low and different and informs the existence of different conduction mechanisms. The small values of activation energy implies that little amount of energy is sufficient to activate the carriers for electrical conduction. Frequency independent dc conduction is confirmed from merging of the two curves at high temperature.

3.4. Hysteresis Study

The ceramics of this family have dielectric breakdown at even low electric field, the saturation polarization could not be observed at the given field; hence we could not get a proper hysteresis loop. The dielectric anomaly in the studied compounds is assumed to be related to ferroelectric-paraelectric phase transition. This assumption was confirmed by appearance of hysteresis loop as seen in Figure 4. Even with smaller remnant polarization the existence of ferroelectric properties in the compounds can be concluded.

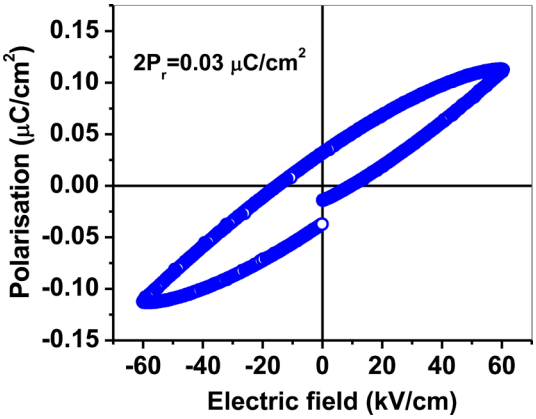

Figure 4. Room temperature hysteresis loop of $Ba_5NdTi_3V_7O_{30}$.

4. Conclusion

The polycrystalline sample of BGTV ceramics is synthesized by solid-state reaction root. The formation of the compound is verified from XRD to form the orthorhombic tungsten-bronze structure. The compound has dielectric anomaly of ferroelectric to paraelectric type at 460˚C. The dielectric constant of the ceramics decreases with increasing frequency. The comparatively low room temperature dielectric constant indicates that these materials may have attractive benefits in electro-optic and infrared pyroelectric detector applications when grown in bulk single crystal or thin-film form. The temperature dependence of ac conductivity was found to obey Arrhenius equation. The activation energy of the compound was found to be different in different region indicating presence of different conduction mechanism. The remanent polarization is found out to be very small.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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