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Synthesis and study of dielectric properties of $Ba_5SmZr_3V_7O_{30}$ ceramics

B.K.Giri[1], B. B. Mohanty[2]*, P. S. Sahoo[3], R. N. P. Choudhary[4]

[1]Department of Physics,Barbil Colllege, Barbil, Keonjhar, Odisha India
[2]Department of Physics, Betnoti College Betnoti, Mayurbhanj, Odisha India
[3]Department of physics, North Orissa University, Baripada ;Mayurbhanj, Odisha India
[4]Department of Physics, ITER, Bhubaneswar, India. Odisha India

Abstract

Ferroelectric materials have procured extensive importance due to their wide spread applications as they possess high dielectric constant, non-linear spontaneous polarization, negative temperature coefficient of temperature behavior etc.. Ba5SmZr3V7O30 is a polycrystalline tungsten-bronze structured ceramic sample prepared by high-temperature solid-state reaction route. Preliminary room temperature structural (XRD) analysis of the compound confirms the formation of single-phase orthorhombic structures. Detailed studies of dielectric properties as a function of temperature (330C-5000C) and frequencies (102-106Hz) were done using a Hioki LCR meter which shows no dielectric anomaly in the experimental temperature range. The nature of the variation of conductivity and value of activation energy in different regions, calculated from the temperature dependence of ac conductivity (dielectric data) suggest that the conduction process is of mixed type (i.e., ionic–polaronic and space charge generated from the oxygen ion vacancies).

Key words: Ceramics, XRD, electrical properties , dielectrics.

1. Introduction

Materials of tungsten-bronze (TB) structure belong to dielectric materials dominant the field of materials research as it displays interesting ferroelectric, pyroelectric, piezoelectric, and nonlinear optical properties for various devices applications such as transducers, actuators, capacitors, and ferroelectric random access memory [1-3]. A number of Ba based TB compounds with high dielectric constant have attracted much more attention because of their importance in the miniaturization of microelectronic devices and extensive applications in microwave telecommunications, satellite broadcasting and other related technologies. It has been found that different ionic size substitutions at the A, B and C sites of TB compounds having a general formula $(A_1)_2(A_2)_4(C)_4(B_1)_2(B_2)_8O_{30}$ can play an important role in tailoring their physical properties for device applications. The present work reports on the study of ferroelectric phase transition of new single-phase polycrystalline Ba5SmZr3V7O30 compound.

2. Experimental details

A suitable stoichiometric ratio of precursors; $BaCO_3$, Sm_2O_3, TiO_2, V_2O_5, ZrO_2 of high purity (>99.9%) were weighed, and mixed mechanically in an agate mortar first in air and then in wet methanol condition for 3 h each. The compound was then calcined in an alumina crucible at an optimized temperature and time (9500C, 12h). Cylindrical pellets of diameter ~ 10 mm and thickness 1-2 mm were made from the calcined powder using a hydraulic press at a pressure of ~ 4N/m2. The pellets were then sintered in an air atmosphere at an optimized temperature and time (9500C, 12 h. The faces were polished with fine emery paper followed by coating the flat surfaces with high purity conductive silver paint, and was dried at 1500C for 2h before carrying out electrical measurements. X-ray diffraction (XRD) data (pattern) of the material was obtained in a wide range of Bragg angle 2θ (200≤ 2θ ≤800) at a scanning speed of 30 min-1 by an X-ray diffractometer (Rigaku, Miniflex) with CuKα radiation (λ= 1.5405Å) at room temperature. Scanning electron micrograph of the material was recorded with a high-resolution scanning electron microscope (SEM: JOEL-JSM model: 5800F) to study the surface morphology of

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the sample (pellet). The impedance studies were carried out in the temperature range of 320 -5000C and frequency range of 1 kHz to 1MHz, using a computer-controlled Hioki LCR meter.margin on a separate line.

3. Results and discussion

3.1. Structural analysis

The XRD pattern of the Ba5SmZr3V7O30 (BSZV) sampleis shown in fig.1.

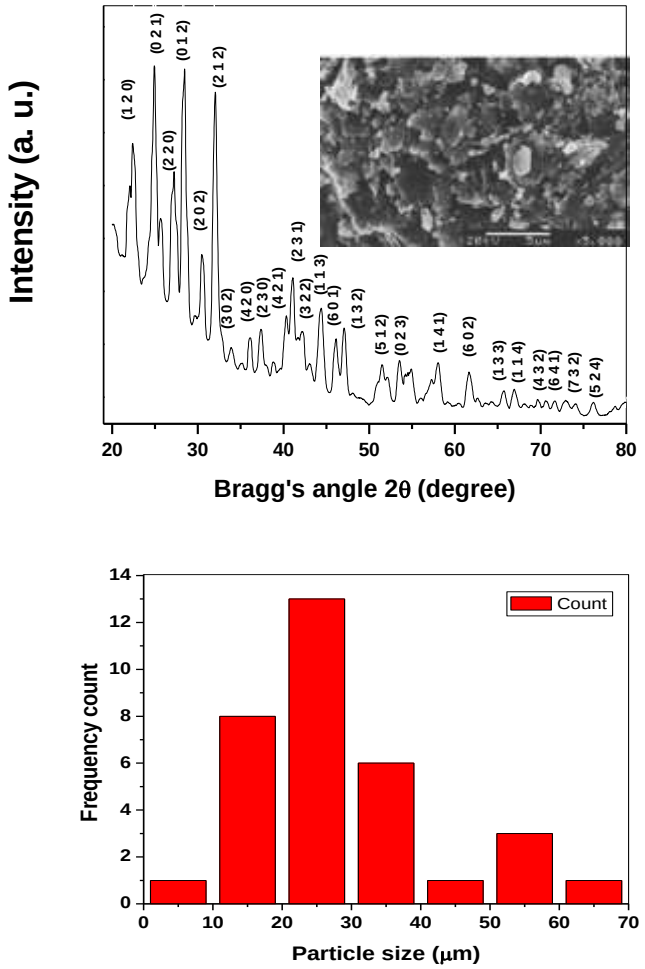

Fig. 1: Room temperature XRD pattern (above), SEM (inset) andhistogram (below) of $Ba_5SmTi_3V_7O_{30}$ ceramics

The sharp and single reflection peaks observed in the XRD patterns are different from those of the ingredients confirming the formation of new single-phase compound. Indexing of the peaks were done by taking their 2θ values using a computer program package, "POWDMULT" [4] in different crystal system and cell configuration. The unit cell of the compound was selected to be orthorhombic unit cell having lattice parameters: a = 12.3967(29) Á, b=5.8446(29), c= 7.5076(29) Á (estimated standard deviation in parenthesis) which are consistence with the reported ones. [5], which was done on the basis of the best agreement (based on least – squares refinement) between observed (obs) and calculated (cal) interplaner distance d (i.e., Σ (dobs – dcal) = minimum). The coherently scattered crystallite size (D) of the compound was measured to be ~14 nm using Scherrer's equation; P = 0.89λ / (β1/2cosθhkl), where λ = 1.5405 Å and β1/2 = peak width of the reflection at half maxima [6]. The contributions of strain, instrumental error and other unknown effects in the peak broadening have not been taken into account during the crystallite size calculation. The SEM micrograph of the compound with different magnifications at room temperature is shown in Fig.1 (inset). The avarage grain size evaluated from the histogram was 1.6 μm.

3.2 Dielectric study

Fig. 2 shows the temperature dependence of dielectric constant (ε_r) for BSZV.

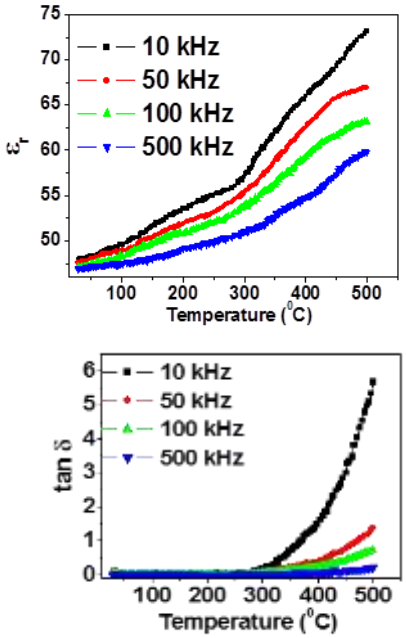

Fig. 2 Temperature variation of relative dielectric constant (ε_r) (above) and loss tangent (tan δ) (below) $Ba_5SmZr_3V_7O_{30}$ ceramics

The figure shows that ε_r increases with increase in temperature and does not show any dielectric anomaly in the measured temperature range. The rise in value of εr with rise in temperature

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exhibits temperature dependent nature in all temperature region. The linear rise in εr may be due to the creation of space charge polarization and defect in the material with rise in temperature. At low temperature (less than 300oC) the change in the values of tanδ of the compound is found to be negligibly small for all frequencies and temperature independent, but rises significantly for 10kHz above300oC temperatures. The low value of loss tangent implies good ferroelectric behavior in the material of the compound.

3.3 Conductivity study:

The plot of frequency versus ac conductivity (σac) (right) of BSZV at various temperatures is as shown Fig. 3. The plot shows low frequencies and high temperatures, plateaus (i.e. frequency independent values of conductivity) which corresponds to the dc conductivity. The observed frequency dependent conductivity found to obey Jonscher's universal power law: σ (ω) = σdc + Aωn where n is the frequency exponent with 0< n < 1 and A is the temperature dependent pre-exponential factor [7].

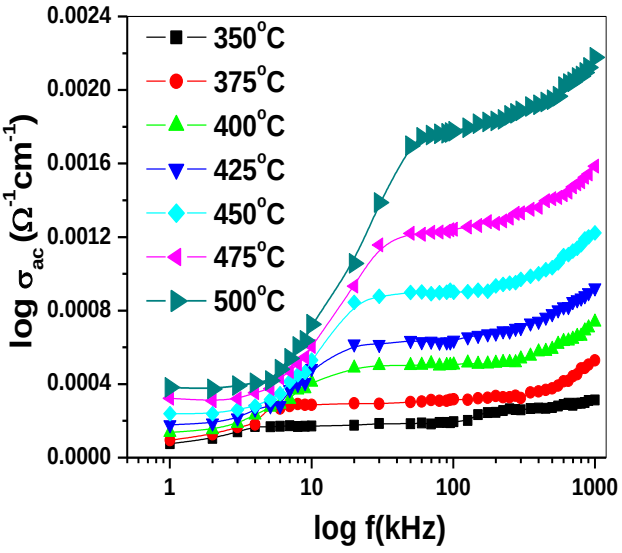

Fig 3. Variation of σ_{ac} with frequency of $Ba_5SmZr_3V_7O_{30}$ at some selected temperature.

According to Funke, the exponent n has a physical meaning and is related to the interaction of charges [8]. A unit value of n implies a pure Debye type conduction mechanism, where the interaction between the neighboring dipoles is almost negligible, and the only conductive element is the dc resistance. The values of n from the graph are found to be less than 1. In pure and stoichiometric state of the compounds, the V ions are in the 5+ valence states and should behave as an insulator with very low dielectric losses. But, oxygen vacancies and charge carriers (electrons and holes) are generated during the calcinations and sintering processes, thus contributing to a complex system of conduction mechanisms.

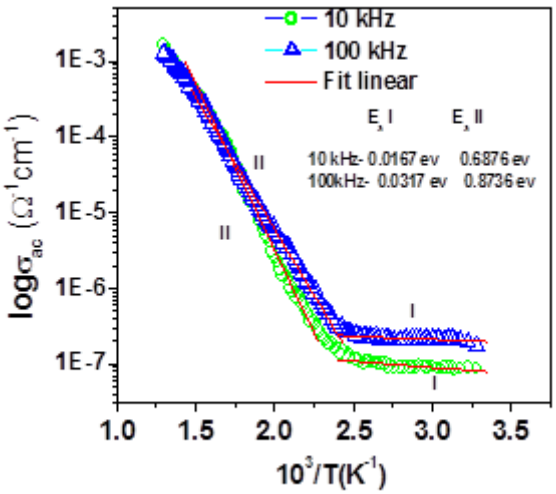

Fig 4. Variation of σ_{ac} with inverse temperature of $Ba_5SmZr_3V_7O_{30}$ at some selected temperature.

Fig 4 shows the variation of σac with inverse of absolute temperature (10^3/T) of BSGTV at different frequencies. The nature of the variation is almost linear over a wide temperature region obeying the Arrhenius relation: $\sigma_{ac} = \sigma_0 \exp(-Ea/k_BT)$ [8] where the symbols have their usual meanings. The activation energy calculated from fitted data is mentioned in Fig 4.

4. Conclusions

From the XRD pattern, formation of single-phase orthorhombic crystal structure is observed at room temperature. The compound has no a dielectric anomaly The different activation energy of the compound observed in different region indicates the presence of different conduction mechanism.

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