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Study of Electrical Conductivity Properties of $Sr_5NdTi_3V_7O_{30}$ Compound

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Abstract: **According to modern researchers ferroelectric ceramics show good insulating behaviour and are widely used as base materials for composite film and high frequency resistors. So it requires a meticulous analysis of transport properties of these materials. $Sr_5NdTi_3V_7O_{30}$ is a lead free Tungsten Bronze ceramic prepared by high temperature solid state reaction route. Room temperature XRD study confirms single-phase orthorhombic structures The SEM study of the sample showed no structural variation, non-uniformly distributed grain. Dielectric study reveals that there is no phase transition within the observed experimental temperature range. But presence of hysteresis loop confirms the ferroelectric nature of the sample. Various conduction mechanisms are observed in ferroelectrics. In this paper emphasis is given on the study of dielectric and electrical conductivity. The frequency dependent a.c. conductivity obeys the Jonscher's power law. The nature of variation of dc conductivity with temperature suggests Arrhenius type of electrical conductivity.**

Keywords: **The Ceramics, X-ray diffraction, SEM, Electrical conductivity.**

1. INTRODUCTION

The tungsten–bronze structure consists of a complex array of distorted BO_6 octahedra sharing corners in such a way that three different types of interstices (A, B and C) are available for suitable cations and anions substitutions. The general formula of complex and disorder TB structure is normally written as $(A_1)_2(A_2)_4(C)_4(B_1)_2(B_2)_8O_{30}$, which is a derivative of the perovskite structure. As C is the smallest, this site is normally empty. A number of ferroelectric compounds and solid solutions of complex composition in the perovskite and tungsten bronze families exhibit dielectric dispersion, optical and thermal properties [1-3]. The study of the nature of electrical conductivity is an important aspect of the physics of ferroelectrics for various device applications. In ferroelectrics, electrical conduction is due to the ordered motion of weakly bound charge particles under the influence of an external electric field. Detailed literature survey shows that some work in this direction has been done on perovskite compounds, but not much has been done on TB structured compounds. In view of the importance of the eco-friendly (lead-free) materials of the above family, we have carried out the systematic study on structural and electrical properties of the $Sr_5NdTi_3V_7O_{30}$ compound which is reported here.

2. EXPERIMENTAL DETAILS

2.1 Material preparation and characterization:

The polycrystalline sample of $Sr_5NdTi_3V_7O_{30}$ (SNTV) was prepared using the high-purity (>99.9%) precursors; $SrCO_3$, Nd_2O_3, TiO_2, V_2O_5 by a mixed-oxide method. The stoichiometric mixtures of the high purity(99.9 %) powders of $SrCO_3$

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,TiO_2,Nd_2O_3 (All from M/s Sarabhai M. Chemicals Pvt. Ltd., India), and V_2O_5 (M/s. Koch Light Ltd, England) were weighed and thoroughly grinded in an agate mortar to obtain homogeneous mixtures and calcined at an optimum temperature (900^0C for 12 hrs). The calcined powders were grinded and dried, followed by mixing with organic binder polyvinyl alcohol (PVA) to prepare cylindrical pellets of 10 mm diameter and 1-2 mm thickness at a pressure of 4×10^6 N/m^2. The pellets were sintered in air at 950^0C for 12 h to yield dense ceramics. The binder was burnt out during high temperature sintering. The formation and quality of the compound was checked by an X-ray diffraction (XRD) technique. The X-ray diffraction pattern of the compounds was recorded at room temperature using an X-ray powder diffractometer (Rigaku, Miniflex) with CuKα radiation (λ=1.5405 Å) in a wide range of Bragg's angles 2θ (20^0> 2θ >80^0) with a scanning rate of 3^0/ minute. To study the surface morphology of the sintered pellets, both the surfaces were made flat and parallel. On the flat and clean surface gold coating was done by a sputtering method to increase the resolution of micrograph. Microstructures of sintered pellets were recorded by JEOL –JSM: 5800F model scanning electron microscope (SEM). The average grain size was determined by a linear intercept method. The electrical properties of the sintered pellets were studied with the data recorded by an impedance analyzer (PSM 1735, model: N 4L). over a wide frequency range (10^2–10^6 Hz) at different temperatures (31–500^0C).

3. RESULTS AND DISCUSSIONS

3.1. Structural analysis:

Fig. 1 shows the XRD pattern of the calcined powder of $BaSr_4LaTi_3V_7O_{30}$. As most of the materials of tungsten bronze structural family crystallize either in tetragonal or orthorhombic crystal system, we attempted to index all the peaks of XRD pattern in these systems but different unit cell configurations using computer software "POWDMULT" [4]. On the basis of the best agreement between observed (obs) and calculated (cal) inter-planer distance *d* (i.e., Σ (d_{obs} −d_{cal}) = minimum), a suitable unit cell (orthorhombic system) with lattice parameters : a= 9.5014(46) Å ,b= 9.3837(46) Å, c=11.8800(46)Å (estimated standard deviations in parentheses) were finally selected. The coherently scattered crystallite size of the sample was estimated from the broadening ($\beta_{1/2}$) of few peaks using Scherrer's equation [5]; $P= K\lambda/\beta_{1/2}\cos\theta_{hkl}$, where K = constant= 0.89, λ = 1.5405 Å and $\beta_{1/2}$ = peak width of the reflection at half height. The average crystallite size of the compound was found to be ~12 nm. The contributions of strain, instrumental and other unknown effects in the broadening have been ignored in the calculations since powder sample was used for XRD pattern.

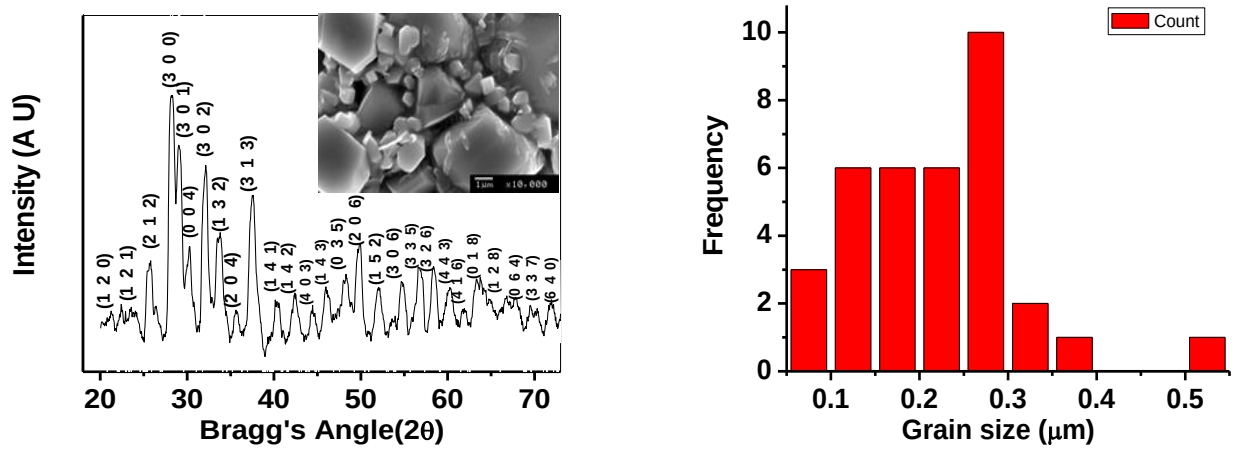

Fig 1: Room temperature XRD pattern, SEM (inset) and histogram (Right) of $Sr_5NdTi_3V_7O_{30}$

3.2. Micro structural analysis:

Fig.1 (inset), shows the room temperature scanning electron micrograph of the sample describing their surface property and microstructure. The micrographs the granules are observed to be spherical and columnar. Some plates like irregular shaped grains are also observed. The porosity seems to be decreased with the growth of grains. The average grain size of the pellet sample was found to be 0.22 μm obtained from the histogram Fig 1(Right).

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3.3. Conductivity analysis:

i) ac electrical conductivity: The a.c. electrical conductivity is discussed as follows-.

a) Variation of ac conductivity with inverse absolute temperature at some selected frequencies:

The a.c. electrical conductivity (σ_{ac}) of the materials under study is calculated from the conductivity relation, $\sigma_{ac} = \omega\varepsilon\varepsilon_0\tan\delta$, where ε_0 is the vacuum dielectric constant, ε is the dielectric constant of the material, ω is the angular frequency and $\tan\delta$ is the dielectric loss of the materials.

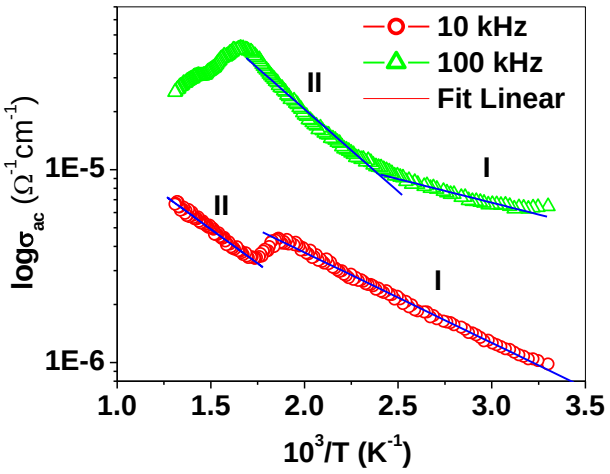

Fig 2: Variation of σ_{ac} with 1000/T of $Ba_{5-x}Sr_xNdTi_3V_7O_{30}$, (x=0-5) at two selected frequencies

The temperature dependent ac electrical conductivity (σac) obeys Arrhenius equation [6]:

$$\sigma_{ac} = \sigma_0 \exp(-E_a/k_BT)$$

Where E_a represents the activation energy for intrinsic conduction process of the materials. The value of activation energy corresponds to the energy required for the polaron to jump over the grain boundaries. Fig. 3 shows σ_{ac} versus inverse of absolute temperature(10^3/T) graph at two selected frequencies. The graphs are splitted in two different regions for both the frequency. Each region is characterized by different slopes showing different activation energy. The variation of ac conductivity is smaller at low temperature (up to~200°C) for the frequency 100 kHz than that of 10 kHz for same temperature range. But for higher temperature the slope is steep for the frequency. The compound follows Arrhenius behavior and shows the negative temperature coefficient of resistance (NTCR) behavior. The activation energy for different region at different frequency are tabulated in table -1. It is clear from the table that ac conductivity (σ_{ac}) in the $Sr_5NdTi_3V_7O_{30}$ compound is governed by the polaron hoping mechanism and the conductivity is influenced by both frequency and temperature.

Table 1: Comparison of activation energy E_a(eV) of $Sr_5NdTi_3V_7O_{30}$vanadates at two different frequencies, from σ_{ac}vs.1/Tgraphs

	E_a (eV)	
f (kHz)	I	II
10	**0.8914**	**0.8427**
100	**0.8802**	**0.5909**

b) Variation of ac conductivity with frequency at different temperatures:

As per Jonscher Power law the origin of the frequency dependence of conductivity lies in the relaxation phenomena arising due to mobile charge carriers. Fig.3. shows the frequency dependence of σ_{ac} for $Sr_5NdTi_3V_7O_{30}$ at various temperatures. The plot indicates the increase in σ_{ac} with an increase in frequency. In low frequency-high temperature region, the conductivity response flattens due to transition from long range hoping to short range ion motion and conductivity relaxation phenomenon occurs [7]. The increase in conductivity with increase in temperature and frequency shows the existence of negative temperature coefficient of resistance (NTCR) behaviour.

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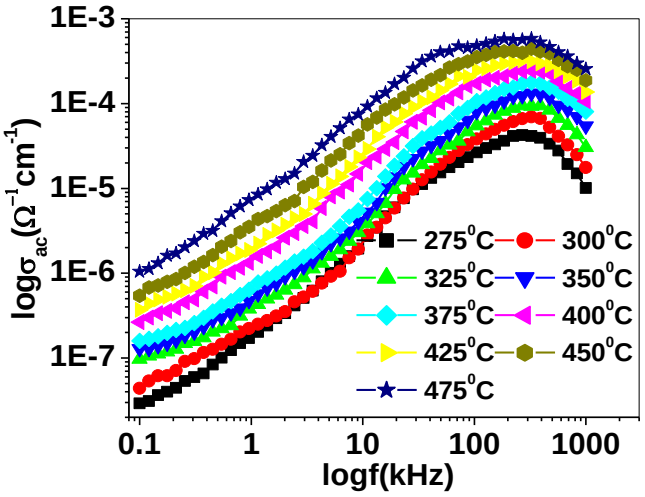

Fig 3: Variation of σ_{ac} with frequency at some selected temperature

c) dc Electrical Conductivity

The bulk electrical conductivity is calculated using impedance spectrum by the relation;

$$\sigma_{dc} = \frac{t}{R_b A},$$

Where R_b is the bulk resistance obtained from the low frequency intercept of the fitted data on the real (Z') axis in the complex impedance plot, t is the thickness and A is the area of the electrode deposited on the sample. The variation of σ_{dc} with $10^3/T$ for $Sr_5NdTi_3V_7O_{30}$ compound is as shown in Fig.3. From the plots it is observed that there is an increase in dc conductivity with the increase in temperature for the observed sample. Due to the addition of thermal energy, the electrons clouds could be set free from O^{-2} ions. The activation energy (due to bulk effect) of these materials in the high temperature region is 0.846 eV. The actual value of conductivity is the value of the bulk conductivity (σ_b). The bulk conductivity can be measured from the complex impedance plots small thickness of the grain boundary layer in the sample. It is observed that the dc conductivity also follows the Arrhenius law.

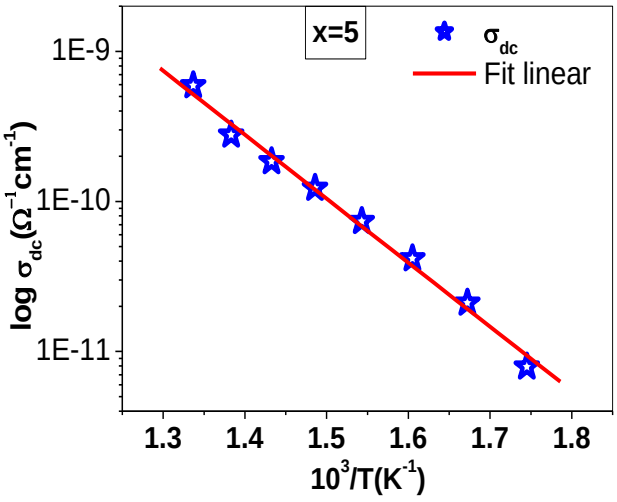

Fig 4: Variation of σ_{dc} with inverse of absolute temperature ($10^3/T$) $Sr_5NdTi_3V_7O_{30}$

4. CONCLUSIONS

The compound $Sr_5NdTi_3V_7O_{30}$ is prepared by high temperature solid state reaction route. The XRD study confirms the formation of single-phase orthorhombic crystal structure. The frequency dependent a.c. conductivity obeys the Jonscher's power law. The nature of variation of dc conductivity with temperature suggests Arrhenius type of electrical conductivity. The different activation energy of the compound observed in different regions indicates the presence of different conduction mechanisms.

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