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Synthesis and characterization of $Na_{0.5}Bi_{4.5}Ti_4O_{15}$ powders by stearic acid gel method

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ABSTRACT

The Aurivillius family compound of sodium bismuth titanate, $Na_{0.5}Bi_{4.5}Ti_4O_{15}$ (NBT) was prepared for the first time by stearic acid gel method. The NBT powders were sintered at 1200 °C for 1 hr. The phase confirmation of compound was done by using XRD. Microstructure of sintered sample was analyzed using scanning electron microscope (SEM). Fourier transform infrared spectroscopy (FTIR) showed compound confirmation peaks of sodium bismuth titanate sample. From FTIR the band at 661.29 cm^{-1} shows formation of presence of large amount of NBT compound. Finally, thermal analysis of NBT was done by thermo gravimetric analysis, differential thermal analysis and differential scanning calorimetry. The temperature of sol gel reaction and the ending process of crystallization were analyzed by differential thermal analysis (DTA). The combustion, decomposition and weight loss temperature ranges were identified by using thermo gravimetric analysis (TGA). From the differential scanning calorimetry (DSC) the heat flow ranges has been observed.

Key words: Ceramics; X-ray diffraction; thermal analysis.
PACS:- 82.45.Yz; 61.05.cp; 81.70.Pg

INTRODUCTION

In early days the piezoelectric lead based lead titanate ceramics ($Pb(Zr,Ti)O_3$, abbreviated PZT) were developed and widely used for transducers, piezoelectric actuators, surface acoustic wave (SAW) filters and sensors because of their excellent electrical properties. It also required increased process optimization and high cost. However, high volatilization of lead oxide led to search for alternative lead free material during firing process, and its toxicity created contamination to environment and affected human health [1,2]. The lead free ceramics such as sodium bismuth titanate ($Na_{0.5}Bi_{0.5}TiO_3$), layer structure oxides of bismuth and tungsten bronze type oxides are attractive due to their non-toxicity. Further, due to the lower processing cost and

ability to produce desirable electrical properties the sodium bismuth titanate based ceramics are widely studied. These ceramics find high power applications such as in transformers, actuators and ultrasonic motors [3,4]. Sodium bismuth titanate based ceramics are considered to be one of the low lead piezoelectric ceramics [5-7].

This study reports the preparation of $Na_{0.5}Bi_{4.5}Ti_4O_{15}$ by stearic acid gel method for the first time. The ferroelectric layered bismuth compound $Na_{0.5}Bi_{4.5}Ti_4O_{15}$ (NBT) is potential material for many applications like piezoelectric devices and ferroelectric random access memories. This compound was prepared by various conventional ceramic processing techniques so far [8-13]. In comparison with other conventionl methods, the stearic acid gel method shows uniform mixing after removing organic substance by combustion. In this method the mixing process was done in molten state so that the metal ions were well dispersed. The main advantage of this method is the carboxylic group and long carbon chains in stearic acid show dispersing ability in metal precursors.

MATERIALS AND METHODS

2.1 Stearic acid gel method

By the stearic acid gel method, the fine single phase NBT powders were prepared by using stearic acid [$CH_3(CH2)_{16}COOH$)] , 99% pure bismuth nitrate [$Bi(NO_3)_3$], sodium acetate [CH_3COONa], and tetrabutyl titanate Ti(OC_4H_9) as chemical constituents. First by heating the appropriate amount of stearic acid was melted at 80 °C. Then, in molten stearic acid the stoichiometric amount of CH_3COONa, $Bi(NO_3)_3$ and Ti(OC_4H_9) were added corresponding to the formula $Na_{0.5}Bi_{4.5}Ti_4O_{15}$. Subsequent to this process, the solution was heated at 80-100°C for 2 hrs. After this, the solution was allowed to cool at room temperature to form white gel. Finally this gel precursor was decomposed at a temperature of 1200°C for 1 hr in air to obtain NBT powders in crystalline form.

3. Characterization

3.1 Powder X-ray diffraction

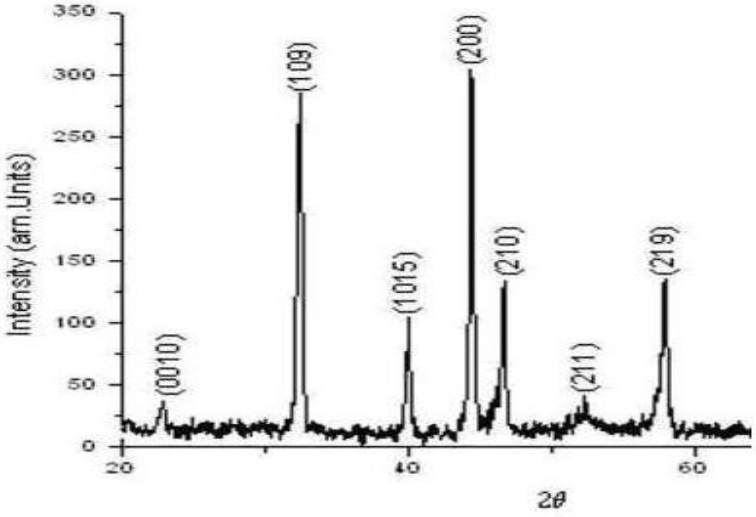

Figure 1. Powder XRD of NBT powder sintered at 1200°C for 1 hr

The powder X-ray diffraction (XRD) pattern was taken using D/MAX-III X-ray diffractometer with Cu Kα radiation of wavelength around 1.5318 Å. The data were recorded for 2Θ between 20 and 80° with step size of 0.02°. The powder X-ray diffraction data of the compound which was synthesized by stearic acid gel method was compared with the JCPDS data (JCPDS number-74-1316) of the same compound. The peaks were found to match, which confirms the formation of tetragonal crystal system of the NBT compound. Also, the X-ray diffraction data of stearic acid gel method compound shows exact matching of peaks with the solid state reaction compound which is reported already [13]. Figure 1 confirms the structure and phase formation of the compound.

3.2 Scanning Electron Micrograph (SEM)

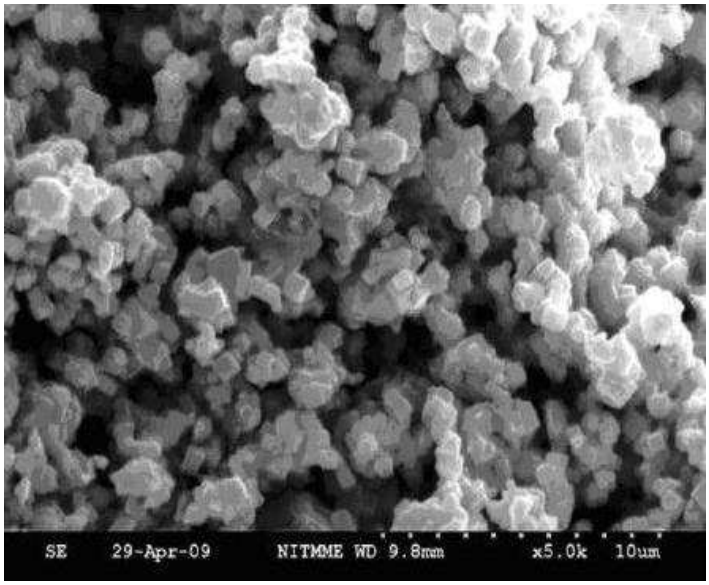

Figure 2. SEM image of NBT powder sintered at 1200°C for 1 hr stearic acid gel Method.

The morphology and microstructure of samples were studied by using a Jeol JSM-5610LV scanning electron microscope (SEM). Figure 2 shows the SEM photograph of the sintered sample which is taken at a magnification of 5000. The photograph shows the homogeneous microstructure and fine grain size.

3.3 Fourier Transform Infrared Spectroscopy (FTIR)

By using Jasco FT-IR-300E spectrometer the fourier transform infrared spectroscopy (FTIR) data of the sample was measured in the range of wave numbers between 400– 4000 cm^{-1}. Figure 3 shows the bands at 1025.57, 1642.56, 2359.33, 3420.28 and 3683.2 cm^{-1} which indicates the presence of carbonate groups due to the usage of stearic acid gel as a precursor [14]. The band at 661.29 cm^{-1} shows formation of large amount of NBT compound [14,15].

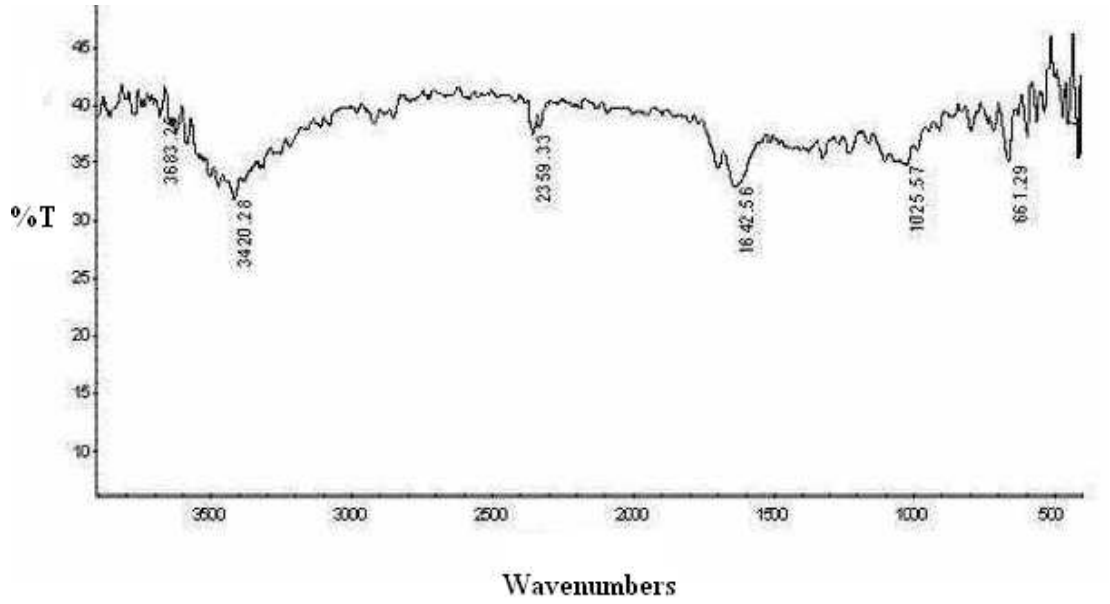

Figure 3. FTIR spectra of NBT powder sintered at 1200°C for 1 hr

3.4 Thermal analysis

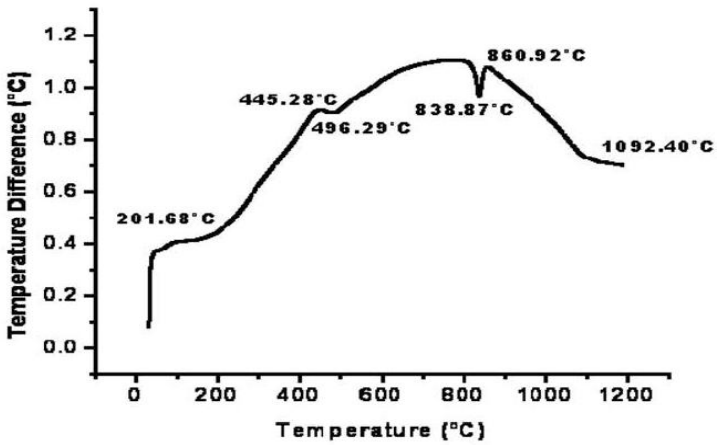

Figure 4. DTA curve of NBT

Figure 4 shows the DTA curve of NBT sample. The peak which appears at 201.68° C is due to evaporation of water and melting of gel. The peak at 496.29° C was caused by burning of organic substances and the peak at 838.87 to 860.92° C shows that the temperature of sol gel reaction [15]. The last peak which is at 1092.40° C shows the ending process of crystallization.

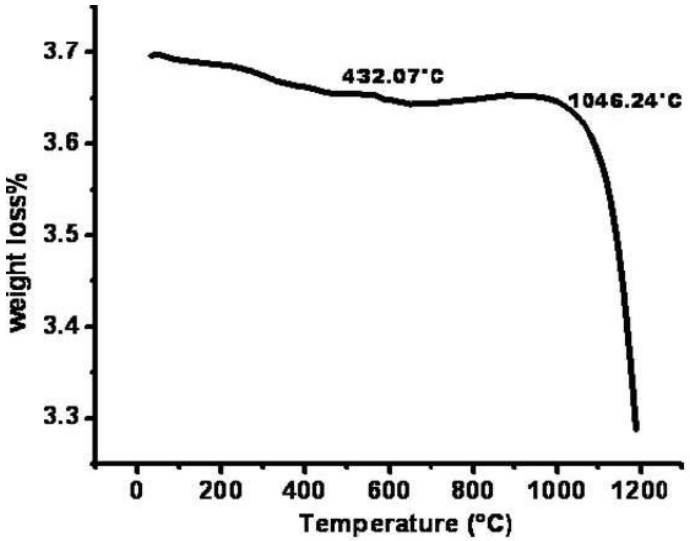

Figure 5. TG curve of NBT

Figure 5 shows the TGA curve of NBT sample. The curve shows the combustion and decomposition of NBT. The temperature range at 432.07°C to 1046.24° C shows the weight loss of the sample [15].

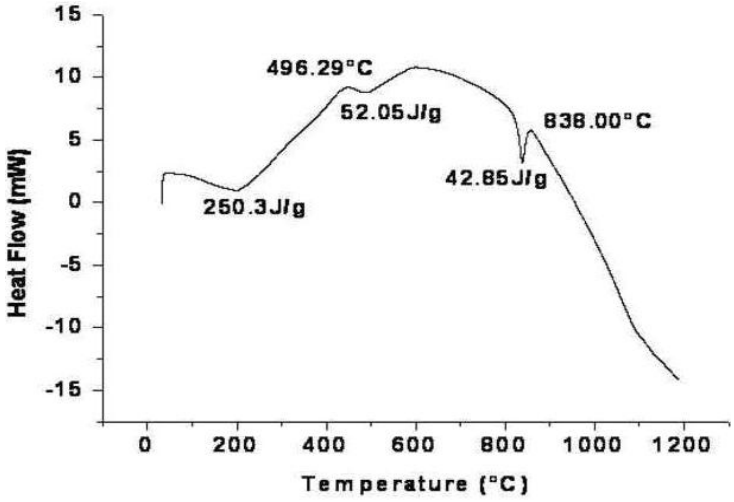

Figure 6. DSC curve of NBT

The DSC curve of NBT sample is shown in Figure 6. The heat flow ranges can be observed from the peaks of 496.29° C and 838° C.

CONCLUSION

The stearic acid gel method is one of the effective routes to synthesize crystalline NBT powders. By this gel method the single phase NBT compound was obtained. In this work we reported the NBT compound which was synthesized by stearic acid gel method for the first time. The phase formation was confirmed by XRD. The morphology and grain size were observed by using SEM analysis. The FTIR analysis confirms the formation of the NBT compound. The thermal properties were also analysed for this compound.

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