Volume : 1 | Issue : 4 | Sep 2012 • ISSN No 2277 - 8179

Research Paper

Synthesis Of Nano Bismuth Ferrite Multiferroics By Microcontroller Based Thermogravimetric Analyzer

Material Science

KEYWORDS : microcontroller, multiferroics, nanomaterials, thermogravimetric analysis

Prof. Ramani	Department of Physics, Bangalore University, Bangalore, India
Prof.M.C Radhakrishna	Department of Physics, Bangalore University, Bangalore, India
Dr. B Angadi	Department of Physics, Bangalore University, Bangalore, India
Dr. J.T Devaraju	Department of Electronic Science, Bangalore University, Bangalore, India.

ABSTRACT

Microcontroller based thermogravimetric analyzer (MTGA) is an home built technique used to study the thermal decomposition of inorganic solids, chemical properties of intermediate and final products, synthetic conditions and synthesis of nanomaterials. The MTGA has additional features like controlled gas delivery system suitable for synthesis of nanomaterials in various gas atmospheres. This paper reports characterization of nano bismuth ferrite-BifeO3 (BFO) synthesized by MTGA. The MTGA system has been calibrated for BFO precursors. The variation in mass of redox mixture (metal nitrates and gylcene) with respect to temperature (thermogram) reveal the ideal conditions for the preparation of nano BFO by different techniques. It was found that structural and electrical results of BiFeO3 synthesized by MTGA are in good agreement with that prepared by other techniques like solution combustion method, sol-gel method, Pechini method, sonochemical method, hydrothermal method etc. The microcontroller based thermogravimetric analyser is one of the good techniques for the preparation of nanomaterials in different gas atmaspheres.

Introduction: Microcontroller based thermogravimetric analyzer [1] (MTGA) is an home built technique and it consists of microcontroller assembly, thermobalance, furnace, personal computer and IR grating assembly. The MTGA technique can be used to study the thermal decomposition of inorganic solids, their kinetics reaction mechanism, structure of intermediate and final products, synthetic conditions and synthesis of nanomaterials. The MTGA has additional features like controlled gas delivery system which is suitable for synthesis of nanomaterials in various gas atmospheres. The multiferroic material BFO exhibit simultaneous presence of ferroelectricity, ferromagnetism and ferroelasticity in the same phase or in a certain range of temperature and possess potential applications for the future technology in information storage, sensors, multiple state memory elements, electric field controlled ferromagnetic resonance devices and transducers with magnetically modulated piezoelectricity. This paper reports thermal structural and electrical properties of nano bismuth ferrite-BifeO3 (BFO) synthesized by MTGA.

Experimental Details: The nano sized bismuth ferrite oxide with a nominal formula BiFeO3 were prepared by Microcontroller based Thermogravimetric analyzer [1] (MTGA) using respective metal nitrates as oxidiser and glycene as fuel. The compositions of metal nitrate [Fe (NO3)3.9H2O (Merck) Bi(NO3)3.6H2O (Merck)] and glycene [C2H5NO2 (Merck)] were ground and it was taken in the sample bucket of MTGA. The redox mixture (metal nitrates and glycene) was heated in air at a rate of 4°C/min from room temperature to 700°C. The contraction of spiral spring of MTGA is due to variation in mass of redox mixture and it was recorded for every 5°C rise of temperature by using cathetometer (Least Count=0.001cm). The voluminous foamy product was ground and obtained powder was characterized by X-ray diffraction (XRD), thermogravimetric analysis (TGA) transmission electron microscope (TEM). The pellet samples were prepared by isostatic press at a pressure of 250 MPa, and then were sintered at 8000 C for 2 hr for electrical property measurements.

Results and discussions: The variation in mass of redox mixture (metal nitrates and gylcene) with respect to temperature (thermogram) is as shown in the Fig-1. The thermal studies describe the existence of two decomposition stages.

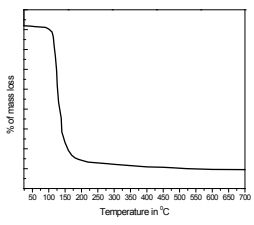

Fig-1 Thermogravimetric analysis (thermogram) of BFO Precursor.

The first one starts from 100-250°C being characterized by high mass loss (40%) [2]. This decomposition consists of initial weak endothermic effect representing water evaporation, followed by a fast intensive exothermic process consisting simultaneous evolution of NO3- and glycene. The second stage decomposition (340-400°C) accompanied by a medium exothermic effect and very low mass loss represents the burning up of remaining carbaneous matter. This result indicates that the ignition temperature for the synthesis of nano size BFO is in between 100-400°C. The MTGA system has been calibrated for BFO precursors the thermogram results obtained in Fig-1 are in good agreement with the results which are published earlier for the same sample [2-5].

The XRD pattern (Fig- 3) of nano BFO synthesized by MTGA reveals that, BFO obtained at low temperature belongs to rhombohedral distorted perovskite structure according to JCPDS. The pattern also shows the presence of secondary phase, as evident by a peak at 28°. The same type of graphs is obtained for BFO synthesized by combustion technique at different temperatures [2,6]. The crystallite size were determined from FWHM of the (010) diffraction peak at 2θ=22.5° for all samples using Scherrer's equation. The crystallite size was found to be nano metric range, i.e., 20-30nm.

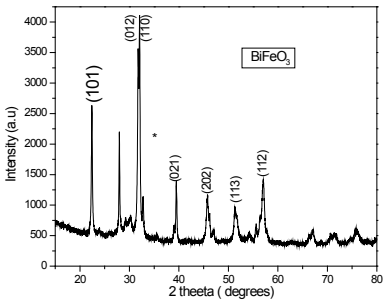

Fig-2 XRD pattern of nano BFO synthesized by MTGA.

The FTIR spectrum of nanopowder BFO is shown in Fig. 3. The strong absorption peaks at 400-600 cm-1 are attribute to the Fe-O stretching and bending vibrations, bring the characteristics of the octahedral characteristic FeO6 group in the per-

Volume : 1 | Issue : 4 | Sep 2012 • ISSN No 2277 - 8179

oskite compounds. The formation of peroskite structure can be confirmed by the presence of metal-oxygen bond. The band at around 1380 cm-1 arose due to the presence of trapped nitrates while band at 1630 cm-1 corresponds to the bending vibrations of H2O [7].

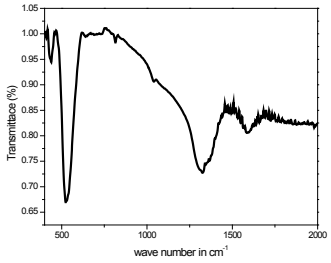

Fig-3 FTIR spectra of nano BFO synthesized by MTGA.

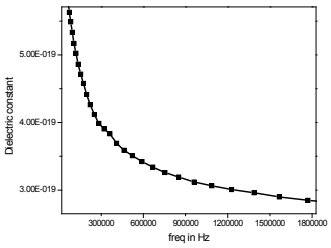

Fig-4 Frequency dependence of dielectric constant at room temperature of nano BFO synthesized by MTGA.

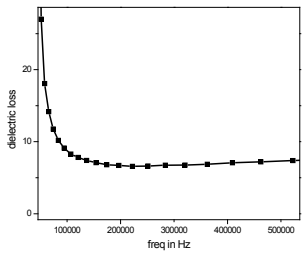

Fig-5 Frequency dependence of dielectric loss at room temperature of nano BFO synthesized by MTGA.

The Figures 4 and 5 shows the frequency (f) dependence of dielectric constant (ε) and dielectric loss (tanδ) for BFO synthesized by MTGA. The ε and tanδ decreases slowly when f is increased from 100 Hz to 20MHz [8-10]. At sufficiently low frequencies vacancies (VO2- and VBi3-) are able to follow f, resulting in increased ε values. On the other hand weak dependence of ε and tanδ on f implies that electron/domains rather than dipoles of the charged defects (VO2- and VBi3- vacancies) mainly contribute to the characteristics of ε and tanδ above 100Hz higher frequency [11,12].

The Fig-6 shows typical TEM image of the sample BFO prepared by MTGA. The BFO powder consists of nano particles with an average size of about 28nm. This is consistent with the average particle size obtained from XRD data. Such consistency implies that the formed nano particles are a single phase. Although it is difficult to define a shape, morphology of the particles seemed to be approximately spherical [13].

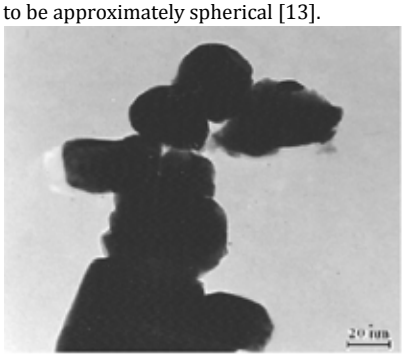

Fig-6 TEM image of the nano BFO synthesized by MTGA.

Conclusion: The nano BFO was synthesized by a home built microcontroller based thermogravimetric analyzer. The calibration curve (thermogram) of BFO reveal the ideal conditions for the preparations of nano sized BFO by other techniques. The structural and electrical results obtained in XRD graph, FTIR spectra, and frequency dependence of dielectric constant and dielectric loss of nano BFO synthesized by MTGA are good agreement with the results of nano BFO synthesized by other techniques and published. So microcontroller based thermogravimetric analyser (MTGA) is one of the simple sysnthesis techniques for the preparation of nano materials.

REFERENCE

[1] J.T. Devaraju, Suresha P. H, Ramani and Radhakrishna M. C, "Devolopment of microcontroller based thermogravimetric analyser", Measurement, 44, (10) , 2011, 2096-2103 . | [2] Suresha P.H, Ramani and Radhakrishna M. C, Basavaraj Angadi and J.T. Devaraju "Characterisation of BiFeO3 synthesised by microcontroller based thermogravimetric analyser" IJEMS, 19, 2012, 196-198. | [3] T.T Carvalho and P.B Tavares " synthesis and thermodynamic stability of multiferroic BiFeO3" Mater Lett, 62, 2008 3984-3986. | [4] Fruth V, Berger D, Matei C, Lanculescu A, Popa M, Tenae E and Zaharescu M, "Preparation and characterization of BiFeO3 nanopowders" J Phys IV, 128, 2005, 7-11. | [5] Monica Popa, daniel Crespo, and Jose M.Calderon-Moreno " Synthesis and structural characterisation of single-phase BiFeO3 powder from a polymeric precursor". J. Am Ceram. Soc, 90(9), 2005, 2723-2727. | [6] Shewtha Shetty, V R Palkar and R Pinto " Size effect study in magnetoelectric BiFeO3 system" Pramana: J Phys, 58 (5-6), 2002, 1027-1030. | [7] Chen Chao, Cheng Jinrong, Yu Shengwen, Che Lingjuan, Meng Zhongyan, " Hydrothermal synthesis of peroskite bismuth ferrite crystallites" J Cryst Growth, 291, 2006, 135-139. | [8] A. Gajovic, S. Sturn, B. Jancar, A. Santic, K .Zagar and M. Ceh "The synthesis of pure-phase bismuth ferrite in the Bi-Fe-O system under hydrothermal conditions without a mineralizer" J.Am.Ceram.Soc 93 (10), 2010, 3173-3179. | [9] Z. Dai, and Y. Akishige " Electrical properties of multiferroic BiFeO3 Ceramics synthesized by spark plasma sintering" J Phys.D; Appl Phys 43, 2010, 445403(5PP). | [10] Q. Jiang, C. Nan, Y. Wang, Y. Liu, Z. Shen "Synthesis and properties of multiferroic BiFeO3 ceramics" J Electroceram 10832, 2007, 9265-5. | [11] G.L Yuan, S.W Or, Y.P Wang, Z.U Liu, J.M Liu "preparation and multiproperties of insulated single-phase BiFeO3 ceramics " Solid state communications 138 , 2006,76-81. | [12] R Mazumder, D. Chakravarty, D. Bhattachary, A Sen " Spark plasma sintering of BiFeO3 " Materials Research Bulletin 44, 2009, 555-559. | [13] Y Wang, Gang Xu, Z. Ren, X. Wei, W. Weng, Piyi Du, Ge Shen and G. Han " Mineralizer-Assisted hydrothermal synthesis and characterization of BiFeO3 nanoparticles " J.Am.Ceram.Soc 90 (8) 2007, 2615-2617. | |