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Particle size analysis of gadolinium doped sodium bismuth titanate ceramics

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ABSTRACT

The gadolinium doped sodium bismuth titanate (NBGT) ceramics have been synthesized by solid state reaction method. NBGT is prepared in the ratio $Na_{0.5}Bi_{(0.5-x)}Gd_xTiO_3$ where x = 0.05, 0.1 and 0.15. The powder was calcined at 800 - 900°C for 2 hours. The sample was sintered at 1000 to 1100°C for 1 hr. The phase formation was confirmed by X-ray diffraction (XRD) studies and morphology by scanning electron microscope (SEM). The presences of functional groups were confirmed by Fourier transform infrared spectroscopy (FTIR). The particle size measurements were done for these samples using laser scattering particle size distribution analyzer.

Keywords: Titanates, X-ray diffraction, particle size.

INTRODUCTION

Piezoelectric lead zirconate titanate (PZT) compounds were most widely used ceramic material for few decades. But due to environmental pollution of its toxicity and its vapour pressure during sintering or growth processes, the lead free materials are becoming important. Sodium Bismuth titanate is considered to be one of the excellent candidates among lead free relaxor ferroelectric material. Bismuth sodium titanate ($Na_{0.5}$ $Bi_{0.5}$ TiO_3 - NBT) is a material which has general formula of $A^{I}_{x}A^{II}_{1-x}BO_3$. NBT ceramics have rhombohedral symmetry below 200^{0}C with curie temperature of T_c 320°C. The electrical properties of this material with various dopants including dysprosium and samarium have been reported early. Exclusively, bismuth layer structured ceramics were recently interested due to its good ferroelectric property. Smolensky et al., first reported the ferroelectric property in layered sodium bismuth titanate [1] which has general formula of $A^{I}_{x}A^{II}_{1-x}BO_3$ (ABO_3) [2] and has curie temperature (T_c) 320°C. And also, NBT related solid solutions like $(Na_{1-x}K_x)_{0.5}Bi_{0.5}TiO_3$, $(Na_{0.5}Bi_{0.5})_{1-x}Pb_xTiO_3$ and $(Na_{0.5}Bi_{0.5})_{1-x}Sr_xTiO_3$ are attracted interesting materials because of its piezoelectric and ferroelectric properties [2,3]. The various NBT-based solid solutions have been developed already [4–7]. NBT doped dysprosium, samarium and other elements were also been reported with interest in enhancement of electrical properties.[8-10]. In this study, the Gd-doped NBT ceramics were synthesized by solid state reaction method and subjected to various instrumental techniques like x-ray diffraction (XRD), scanning electron microscope (SEM), fourier transform infrared analysis (FTIR) and particle size measurements.

MATERIALS AND METHODS

The chemical reagents with 99% purity of Bi_2O_3 (Sigma Aldrich), Na_2CO_3 (Sigma Aldrich), Gd_2O_3 (Sigma Aldrich) and TiO_2 (Sigma Aldrich) were used as raw materials. The stoichiometric amount of chemicals were taken as per the chemical formula $Na_{0.5}Bi_{(0.5-x)}Gd_xTiO_3$ where x = 0.05, 0.10 and 0.15 (NBGT-1, NBGT-2 and NBGT-3, respectively). The powder mixtures were grounded and calcined at 800 - 900°C for 2 hours. The calcined powders grounded again and sintered at 1000 to1100°C for 1 hour in the air atmosphere. After sintering the ceramic specimens were analyzed for phase purity using powder x-ray diffractometer (Siemens D500). The morphology and microstructure of these specimens were investigated by a Jeol JSM-5610LV scanning electron microscope (SEM). The FT-IR spectrums of the specimens were measured by a Jasco FT-IR-300E spectrometer using KBr method for the wave number range of 400–4000 cm^{-1}. Particle size measurements were carried out using laser scattering particle size distribution analyzer (Horiba LA-910).

RESULTS AND DISCUSSION

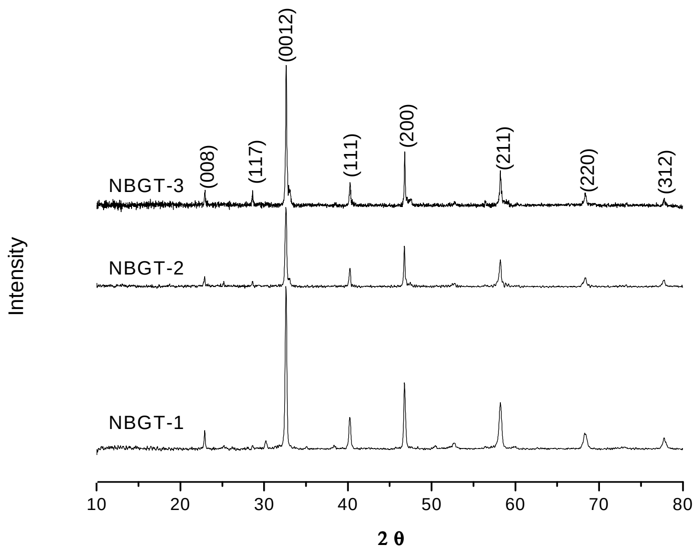

Figure 1. XRD patterns of a. NBGT-1, b. NBGT-2 and c. NBGT-3.

Figure 1 shows the powder XRD patterns of NBGT-1, NBGT-2 and NBGT-3. The pattern confirms the formation of single phase with rhombohedral structure. The shift of the peak at (117) confirms the presence of gadolinium in NBT.

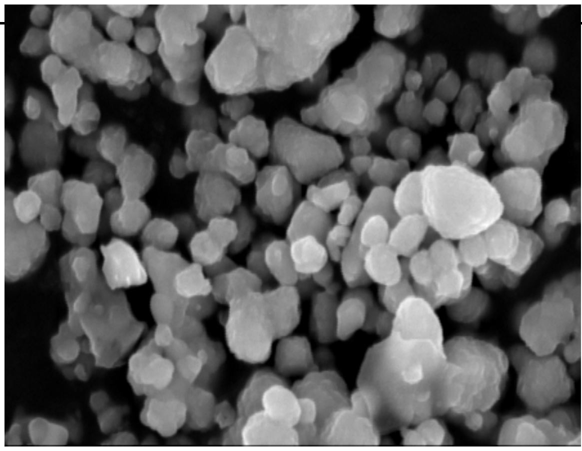

Figure 2(a). SEM image of NBGT-1

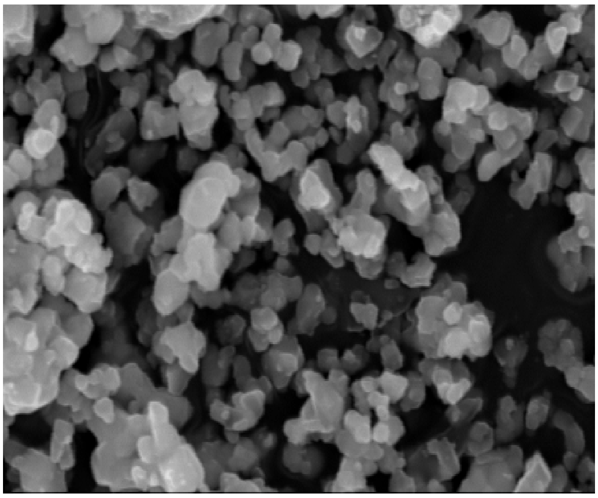

Figure 2(b). SEM image of NBGT-2

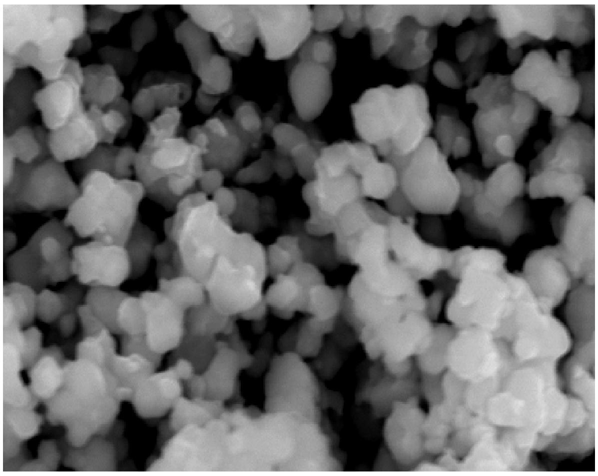

Figure 2(c). SEM image of NBGT-3

Figure 2(a), (b) and (c) show SEM micrographs of ceramic specimens with various doping of gadolinium oxide in NBT powders and reveal the cross-section morphological features of NBGT-1, NBGT-2 and NBGT-3 samples.

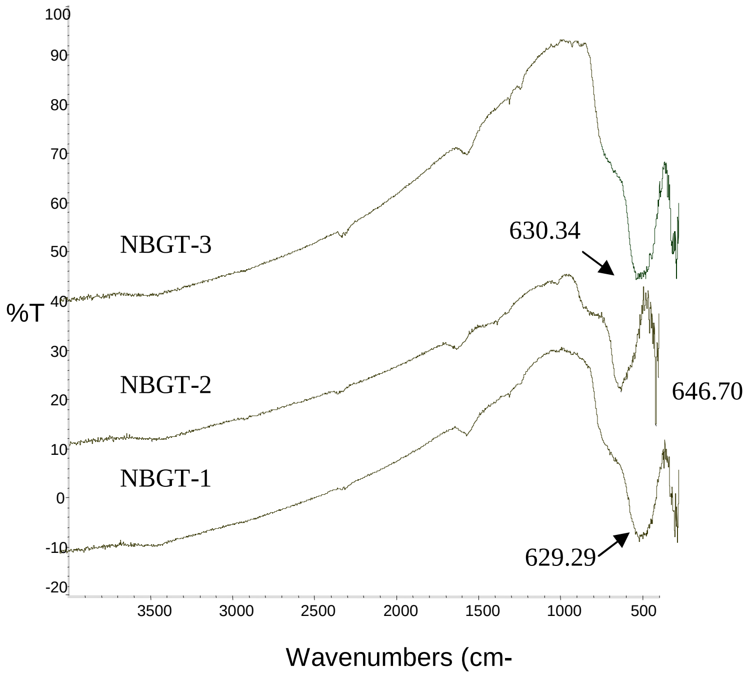

Figure 3. FTIR patterns of a. NBGT-1, b. NBGT-2 and c. NBGT-3

The FTIR spectra of NBGT-1, NBGT-2 and NBGT-3 are shown in Figure 3. All the three compounds show identical vibration of asymmetric bands at 629.29, 630.34 and 646.70 cm^{-1}. The asymmetric bands are assigned to the stretching vibration of octahedral BO_6 groups in perovskite structure and confirming B-O bonds along c-axis.

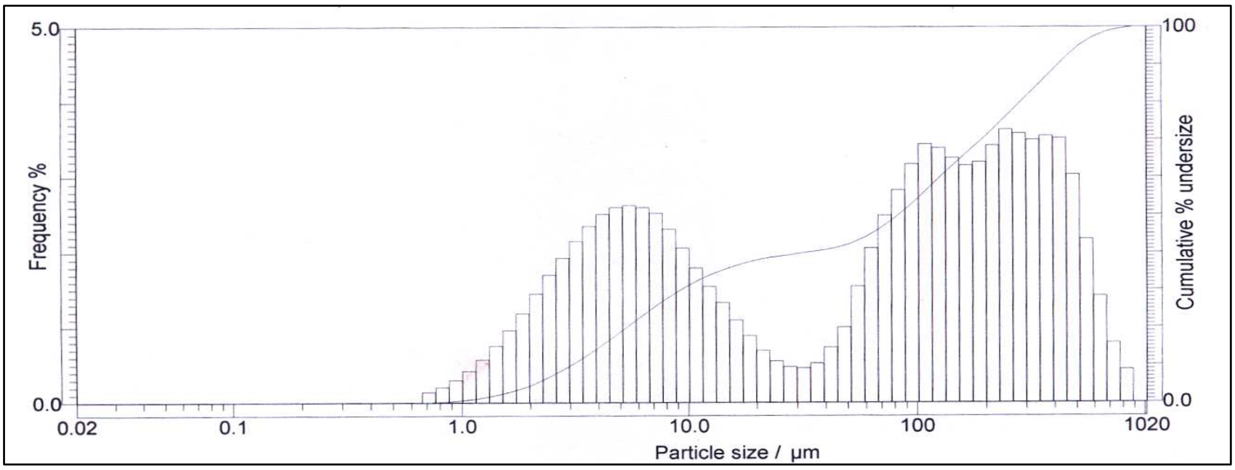

Figure 4. Particle size graph of NBGT-3 powder

Figure 4 shows particle size versus frequency graph and particle size with cumulative % of

NBGT-3 powders. The graph shows particle sizes of the NBGT-3 compound around 0.8 to 1020 µm.

Table. 1. Datas of particle sizes with different frequency levels

Size(µm)	Frequency (%)	Und(%)
1019.5	0.00	100.00
890.1	0.44	100.00
777.1	0.79	99.56
678.5	1.42	98.77
592.4	2.18	97.35
517.2	3.03	95.17
451.6	3.52	92.14
394.2	3.55	88.62
344.2	3.49	85.07
300.5	3.58	81.58
262.4	3.63	78.00
229.1	3.42	74.36
200.0	3.20	70.95
174.6	3.15	67.75
152.5	3.25	67.75
133.1	3.39	61.34
116.2	3.44	57.95
101.5	3.17	54.51
88.58	2.83	51.34
77.34	2.49	48.51
67.52	2.06	46.02
58.95	1.55	43.96
51.47	1.00	42.41
44.94	0.73	41.41
39.23	0.52	40.68
29.91	0.48	39.70
26.11	0.55	39.23
22.80	0.69	38.68
19.90	0.89	37.98
17.38	1.09	37.10
15.17	1.33	36.00
13.25	1.54	34.67
11.56	1.79	33.13
10.10	2.06	31.34
8.816	2.31	29.27
7.697	2.52	26.96
6.720	2.60	24.44
5.867	2.63	21.85
5.122	2.60	19.22
4.472	2.51	16.62
3.905	2.35	14.11
3.409	2.15	11.76
2.976	1.93	9.61
2.599	1.70	7.68
2.269	1.45	5.98
1.981	1.18	4.53
1.729	0.96	3.35
1.510	0.75	2.39

1.005	0.30	0.65
0.877	0.21	0.35
0.766	0.14	0.14
0.669	0.00	0.00
0.445	0.00	0.00
0.058	0.00	0.00
0.022	0.00	0.00

Table.1 shows datas of graph with particle sizes with different frequency levels. At maximum frequency level 3.63% the particle size was found around 262.4µm.

CONCLUSION

Single phasic $Na_{0.5}Bi_{(0.5-x)}Gd_xTiO_3$(NBGT) has been prepared by solid state reaction method. The doping of gadolinium in BNT ceramics did not show any major changes in XRD patterns due to its small amount. But the peak at (117) confirms the substitution of gadolinium in NBT ceramics. The SEM result shows the sintered samples of gadolinium doped BNT ceramic compounds. The complementary use of FTIR has allowed proposing the existence of compounds present in the material. The particle size analysis of gadolinium oxide doped with NBT compound was done.

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