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Dielectric and Characterization Study of $BaNb_2O_6$ Nanoparticles Synthesized by Sol-Gel Method

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ABSTRACT

The BaNb2O6 (BN) is synthesis by sol-gel method in Nanoscale. The Particle sized was rod like structure with diameter ~ 100nm. The X-ray diffraction patterns of $BaNb_2O_6$ powder is used for the identification of tetragonal Phase by indexing of the peak in predominant tetragonal phase using list square fit method and Bragg's relation. The dielectric property of material has studied with various temperature and frequencies.

In the present work the $BaNb_2O_6$ (BN) in nano scale was synthesis by sol-gel method. The XRD characterisation was used for the identification of the polymorphic tetragonal phase. The particle sized was observed by Transmission electron microscopy (TEM), dielectrics behaviour with temperature and frequency has been also studied.

Keywords : Tetragonal Phase, Nanoparticles, Dielectrics

I. INTRODUCTION

BaNb2O6 (BN) is an important member of alkaline earth niobates group. Its orthorhombic phase is studied for dielectric properties [1]. However, Tetragonal tungsten bronze (TTB) structure of this material is highly interesting and applicable due its ferroelectric and dielectric properties. Tetragonal tungsten bronze (TTB) family with a general formula (A1)2(A2)4C4 (B1)2(B2)8O30. The point group of this family is 4 mm and space group is P4bm. The ferroelectric behavior in these materials is mainly due to noncentrosymmetry of the structure. This structure is characterized by a network of BO6 octahedra [2,3]. The literature survey shows single Tetragonal Phase of BaNb2O6 (BN) is the not yet to be investigated, only two alkaline earth niobates compounds belonging to this class are studied. The first one is $Sr_xBa_{1-x}Nb_2O_6$ (SBN) with $0.25<x<0.75$ and the second is newly reported $Ca_xBa_{1-x}Nb_2O_6$ with $0.20< x<0.40$ (CBN). SBN, other than ferroelectric behavior, is widely studied for the optical, pyroelectric, piezoelectric and dielectric properties [4-6]. Various properties of CBN are being investigated [7-9]. Now there is an increasing interest of the researchers on synthesis and investigation of dielectric and ferroelectrics properties of BaNb2O6 (BN) in single Tetragonal Phase. This material is very useful for microwave dielectric properties, Photocatalytic activity and $Sr_xBa_{1-x}Nb_2O_6$ (SBN), $Ca_xBa_{1-x}Nb_2O_6$(CBN) synthesis [10-12].

II. EXPERIMENTAL

BaNb2O6 nanoparticles in tetragonal phase were prepared by the sol-gel method. The Detail sol-gel method has been described in our previous paper[13] in brief First, the Nb2O5 was dissolved in minimum amount of HF after heating in a water bath to get transparent NbF5 solution. Another solution was obtained by dissolving $BaCl_2.2H_2O$ in distilled water. Two solutions were mixed together by using a magnetic stirrer for 4-5 hours. While stirring, an excess quantity of concentrated HCl was added to dissolve the barium fluoride formed by the mixing of NbF_5 and $BaCl_2.2H_2O$. An appropriate amount of citric acid was added to the above mixture. This mixture solution was then kept in a water bath, which was maintained at 100^{0}C to evaporate water slowly from the mixture. After evaporation of water, a yellowish gel was formed which was dried in air and decomposed at 600^{0}C for ~15 h.

III. RESULTS AND DISCUSSION

X-Ray Diffraction:

The X-ray diffraction patterns of $BaNb_2O_6$ powder is shown in Fig. 1. The indexing of the peak in predominant tetragonal phase was done by list square fit method and Bragg's relation,

$$2d_{hkl} \sin\theta = n\lambda$$

where λ is the wavelength of CuKα (1.5406) x-ray radiation, d_{hkl} is interplaner spacing and θ is the Bragg's angle. Since, it was seen that the reflecting angles of XRD pattern of BN resemble to the JCPDS file no. 73-0126 of SBN, we prefer tetragonal structure of which d_{hkl} is given by,

$$\frac{1}{d^2_{hkl}} = \frac{h^2 + k^2}{a^2} + \frac{1^2}{c^2}$$

Indexing of all planes and the difference between observed and calculated d are listed in Table-1. A maximum intensity peak is obtained as (410) corresponding to d_{obs} = 3.019Å. Two other major peaks are observed corresponding to d_{obs} values 2.80 Å and 3.976 Å as (311) and (001) respectively. The lattice parameters in tetragonal phase are found to be **a** = **b** = 12.445Å, and **c** = 3.975 Å.

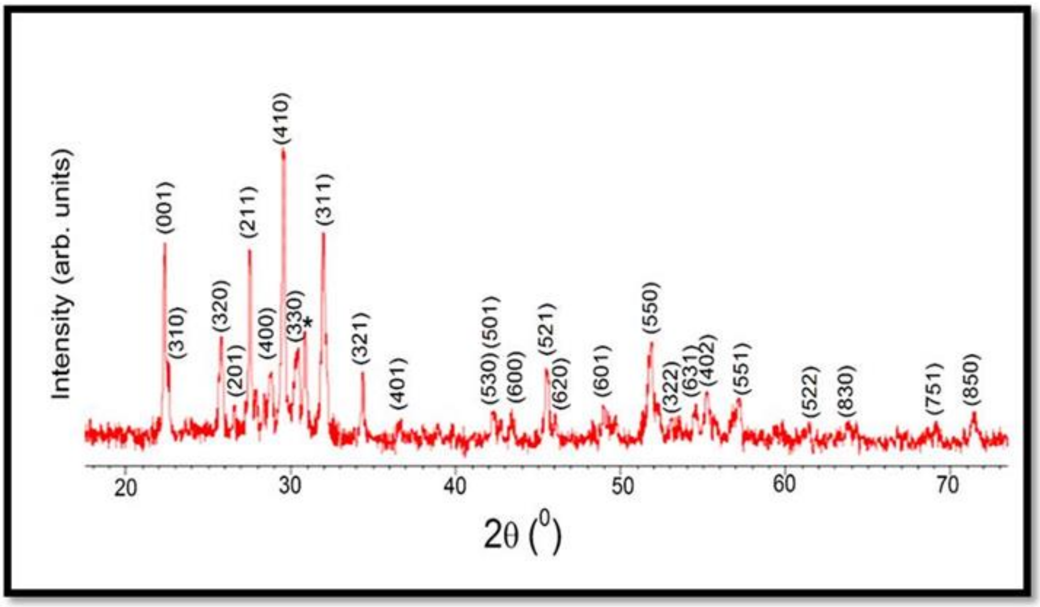

Fig. 1 : XRD pattern of as synthesized $BaNb_2O_6$.

It is observed from the Table1 that reflection angles 2θ =28.35^0 and 30.9^0 do not fit in this pattern. The reflection at 28.35^0 is recognized as a characteristic peak of orthorhombic BN. However, its relative intensity is small; therefore, we can expect that the TTB cell structure get disturbed negligibly due to this trace.

Table 1 : Indexing of XRD diffraction pattern of $BaNb_2O_6$ nanoparticles in (TTB) structure

S.N.	2θ	% Intensity	dobs	dcal	(hkl)	Δd(dcal~dobs)
1	22.34	69	3.9763	3.9756	(001)	0.0007
2	22.6	22	3.9311	3.9355	(310)	0.0044
3	25.78	40	3.4530	3.4516	(320)	0.0014
4	26.57	12	3.3521	3.3502	(201)	0.0019
5	27.54	65	3.2362	3.2350	(211)	0.0012
6	28.35 #	16	3.1455	-	-	-
7	28.82	28	3.0953	3.1113	(400)	0.0160
8	29.56	100	3.0195	3.0184	(410)	0.0011
9	30.46	34	2.9323	2.9333	(330)	0.0010
10	30.9 *	37	2.8915	-	-	-
11	31.93	73	2.8005	2.7969	(311)	0.0036
12	34.35	23	2.6086	2.6064	(321)	0.0022
13	36.6	6	2.4532	2.4502	(401)	0.0030
14	42.24	11	2.1378	2.1343	(530)	0.0035
15	42.74	5	2.1139	2.1096	(501)	0.0043
16	43.37	8	2.0846	2.0742	(600)	0.0104
17	45.52	30	1.9910	1.9979	(521)	0.0069
18	46.07	7	1.9686	1.9677	(620)	0.0009
19	49	14	1.8575	1.8389	(601)	0.0186
20	51.87	32	1.7698	1.7600	(550)	0.0098
21	53.06	8	1.7245	1.7226	(322)	0.0019
22	54.53	11	1.6814	1.6811	(631)	0.0003
23	55.2	21	1.6626	1.6751	(402)	0.0125
24	57.17	17	1.6099	1.6093	(551)	0.0006
25	61.23	3	1.5116	1.5070	(522)	0.0046
26	63.78	6	1.4581	1.4566	(830)	0.0015
27	69.14	4	1.3575	1.3595	(751)	0.0020
28	71.41	9	1.3198	1.3191	(850)	0.0007

Orthorhombic BN,* no reflection without twinning

IV. MORPHOLOGY

The morphology of BaNb2O6 particles is shown in Fig. 2(a). The particles are rod-like shaped with diameter ~ 100nm and slightly agglomerated. The representative HRTEM image of the crystallite and SAED pattern are shown in Fig. 2(b) and 2(c), respectively. Fig. 2(b) shows, the inter-planer distance is about 0.275nm which corresponds to (311) plane of the tetragonal BaNb2O6. The SAED pattern shows many white dots on the rings, which confirms the well crystallization of the material. The diffraction rings corresponding to planes in SAED are also identified (Fig.2c). Interestingly, the plane (311) appeared in all the three investigations namely XRD, HRTEM and SAED, indicating that it is one of the characteristic peak of TTB structured BN.

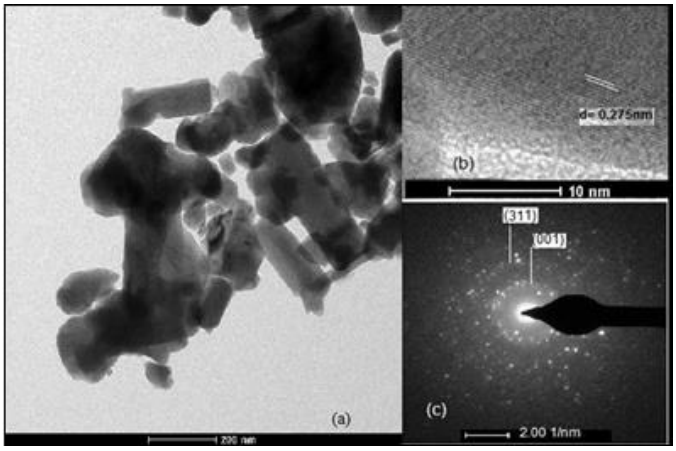

Fig. 2: (a) TEM image, (b) HRTEM images and (c) SEAD pattern of BaNb2O6

Dielectric study

Dielectric permittivity variation against temperature measured at 100Hz of the synthesized BN powder is shown in Fig 3(a). With the rise in temperature, the permittivity increases suddenly to 4000 at 40^{0}C then remain constant up to 110^{0}C. It is then falls sharply to 180^{0}C and remains constant thereafter. The nature of peak indicates a 'relaxor' type behaviour of the material. Since the material shows ferroelectric behaviour, the ferroelectric- paraelectric phase transition is diffused type. This may be a special feature of TTB type ferroelectrics.

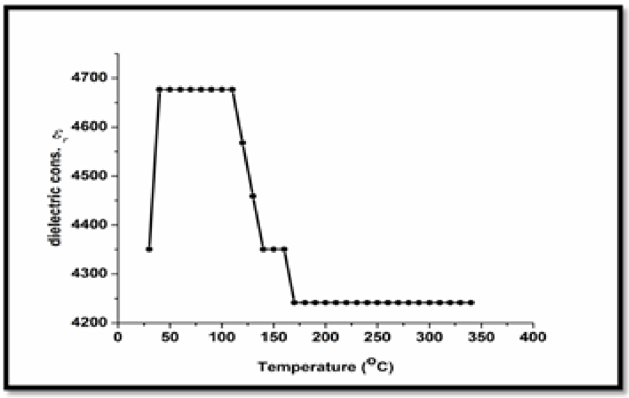

Fig. 3(a): Dielectric constant variation against temperature

The variation of dielectric constant with frequency as shown in the fig3(b). The polarization factor affects on the dielectric behaviour with frequency. AT low frequencies side the surface charge polarization and dipole polarization are predominant and hence the dielectric constant has higher values. From the frequency response plot it is clearly shows that dielectric constant has higher values at lower frequencies side and it sharply decreases as the frequencies increases to higher value. It nearly remains constant above 10KHz frequency as electronic and ionic polarization are predominant at the higher frequencies sides.

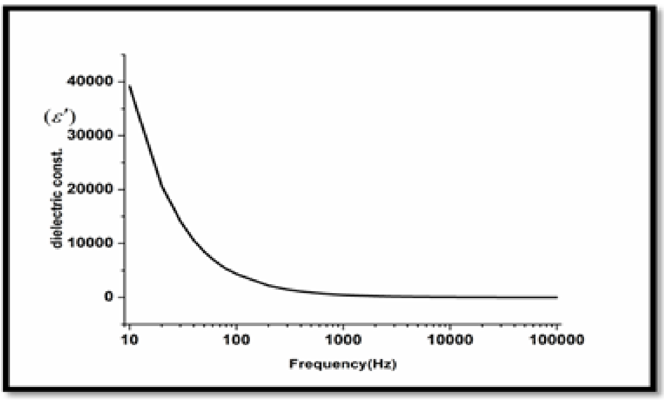

Fig. 3(b) : The variation of dielectric const. with frequency

V. CONCLUSION

The nanoparticles of $BaNb_2O_6$ were successfully obtained by the sole-gel technique by increasing the reaction period. Indexing and structural analysis of the XRD pattern used to conform the tetragonal Phase of material successfully. The dielectric study shows that it is a 'relaxor' type of ferroelectrics. Material has very high value of dielectric constant at low frequency. Dielectric constant is temperature and frequency dependent.

VI. REFERENCES

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