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Structural and Thermogravimetric Analysis of Piezoelectric KNbO3

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ABSTRACT

Piezoelectric ceramic $KNbO_3$ powder was prepared by hydrothermal reaction using Nb_2O_5 in K_2CO_3 solution. A single phase of $KNbO_3$ was obtained when the molar ratio of K_2CO_3/Nb_2O_5 was above 1.2. It shows the orthorhombic structure at room temperature from XRD analysis. The morphology of the prepared ceramic is done using SEM analysis. Two dips in DTA at 70°C and 1080°C indicate the absorption of energy. At temperature up to 100°C the crystal shows endothermic behavior, while above 100°C, the behavior is exothermic up to 1000°C. After 1000°C the crystal shows the endothermic behavior again with a valley at near 1080°C.

Keywords : Piezoelectric, Ceramic, XRD, $KNbO_3$, Endothermic.

I. INTRODUCTION

Lead-based piezoelectric ceramics (PZT) have good piezoelectric properties and therefore frequently used in many applications[1-2]. however since they contain large concentration of PbO, unfavourable to environment, search for lead-free or low lead materials has been initiated. As far as PZT are concerned, considerable attention has been paid to (K, Na)NbO_3(KNN)-based ceramics because of their excellent piezoelectric properties, high Curie temperature [3-4], and environmental friendliness [5-7]. KNN with perovskite structure is known for its better nonlinear optical and electro-optic properties.

Although recent researches reported many KNN-based lead-free ceramics with improved piezoelectric properties, it still has a long way to run for lead-free piezoelectric materials to substitute commercial PZT. Similar to the more extensively studied isomorphous analog, $BaTiO_3$, There are two phase transition temperatures above room temperature for $KNbO_3$: structural phase transformations on cooling from high temperature with the crystallographic symmetry being reduced from cubic - tetragonal -orthorhombic - rhombohedral. The mechanisms for the phase transformations (i.e., their displacive or order-disorder character) are still under discussion. Unlike tetragonal $BaTiO_3$, $KNbO_3$ is orthorhombic at room temperature. In PZT perovskite structure, the acceptor (hard) dopants such as K^+, Rb^+, Na^+ (occupy A-site, i.e., Pb-site) and Sc^{3+}, Mg^{2+}, Fe^{3+}, Fe^{2+}, Co^{2+}, Co^{3+}, Mn^{2+}, Mn^{3+}, Ni^{2+}, Ga^{3+}, In^{3+}, Al^{3+}, Cr^{3+} (occupy B-site, i.e. Ti-site) in the perovskite structure[8-10]. The properties of hard doping are

lower dielectric constant, lower dielectric loss, higher coercive field, low k_p and high Q_m where different acceptor doping ions affect different properties. The effect of dopant Al^{3+} on the crystals growth mechanism and the crystal textures is studied in this work.

II. EXPERIMENTATION

2.1. Synthesis

$KNbO_3$ ceramics were prepared by modified conventional ceramic fabrication process. The growing large size single crystals are somewhat difficult. The present method is used to grow the large size single crystals of $KNbO_3$. The K_2CO_3 and Nb_2O_5 were taken in the molar ratio of 1.2:1. The dopant Al_2O_3 was added to the mixture of K_2CO_3 and Nb_2O_5 . This mixture was mixed thoroughly by grounding them together in the mortar for a sufficiently long time (4 to 5 hrs) and packed in a 50 ml flat bottom platinum crucible covered with a platinum lid. The crucible was heated till 900°C at the rate of 50°C/h. After a temperature of 900°C, the rate of heating was reduced to 20°C/h, till a temperature of 1100°C was reached. The crucible was maintained at this temperature for a period of 24 h. After soaking for 24 h, the homogeneous mixture was then cooled to 840°C at the rate of 14 to 17°C /h to allow for crystallization. The material was reheated to 1000°C, maintained at this temperature for 18 h and slowly cooled to room temperature at the rate of 20°C /h for the complete crystal growth. The crystal blocks separated from flux were found at the top by a thick crystalline layer projecting from the walls of the crucible. A thin layer of small crystals was also found near the thick layer. Crystal plates could also obtain on the bed of flux. The crystal plates found on the bed of the flux are mostly white. Some crystal plates are colourless. The size of the colourless plates range from 2 mm to 50 mm on edge length. The thickness is about 0.4mm. The crystals plates usually show quite simple twinning and are very good for dielectric as well as domain studies. The white crystal plates are larger in area and smaller in thickness (of the order of 0.1mm.), very fragile and highly twinned. The large size single crystals is of greenish colour and transparent. Also the crystal could be easily doped with Al_2O_3. The crystals doped with Al_2O_3 produced some interesting effects on the domain structures which will be discussed later in a different paper. The aluminum doped single crystals of $KNbO_3$ were characterized using X-ray diffractometer. The thermal properties using DTA and DSC were also carried out to observe phase transitions.

2.2 Characterization

The structural properties of $KNbO_3$ were identified by X-ray powder diffraction (XRD) with a X'Pert PRO advanced diffractometer using Cu (K_α) radiation (λ=1.5406 Å). The morphology of selected synthesized $KNbO_3$ films was examined using scanning electron microscopy (SEM, model S-4500, Hitachi, Japan) at 5-kV accelerating voltage. The thermal properties using DTA and DSC were carried out to observe phase transitions.

III. RESULT AND DISCUSSION

3.1 structural characterization

The XRD studies of the sample were carried out to determine the lattice parameters. The XRD pattern of $KNbO_3$ is shown in the Fig. 1 and the data is shown in table 1. From the XRD studies the lattice parameters of doped $KNbO_3$ single crystals are found as a=5.6632 Å, b=3.9745 Å, c=5.7218 Å. This is like an orthorhombic structure at room temperature.

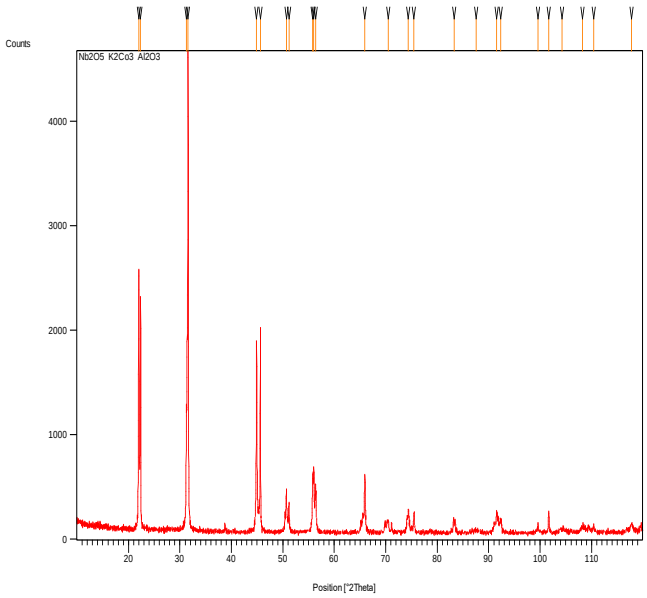

Fig. 1. The XRD Spectra of $KNbO_3$ sample.

Sr. No.	2θ	d_{obs}	d_{cal}	hkl	I/I0 (%)
1	22.3448	3.97549	3.9745	010	47.66
2	31.5705	2.83164	2.8316	200	100
3	44.8906	2.01754	2.01252	202	38.67
4	45.6177	1.98706	1.9872	020	41.86
5	50.6702	1.80014	1.8075	103	8.40
6	51.2021	1.78268	1.79546	212	6.05
7	55.8036	1.64609	1.64535	113	11.12
8	56.3745	1.63076	1.63413	311	8.79
9	65.9354	1.41556	1.4158	400	12
10	70.4516	1.33547	1.33110	321	2.33
11	74.4055	1.27399	1.27120	313	4.59
12	75.4907	1.25834	1.234646	322	4
13	83.3304	1.15873	1.14436	005	2.34
14	91.5603	1.07483	1.07357	413	3.91
15	92.3886	1.06735	1.0609907	205	2.63
16	99.5828	1.00864	1.006261	404	1.37
17	101.6695	0.99351	0.9936	040	4.16
18	108.2186	0.95083	0.950211	315	1.16
19	110.4443	0.93782	0.935948	225	1.09
20	117.7966	0.89962	0.8935	422	1.59

Table: 1: X-ray diffraction data of $KNbO_3$

3.2 Morphological Charecherization

The SEM of the different samples was carried out to determine the grain size and it is found in the range of 1.2 to 2.5µm.

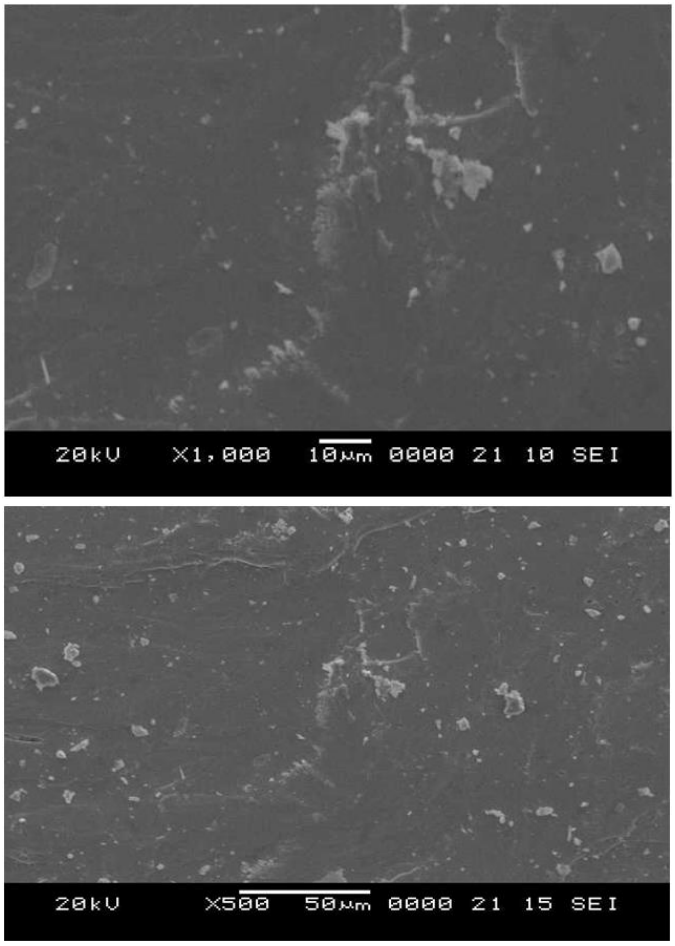

Fig. 2 SEM micrographs of $KNbO_3$ ceramics

3.3 Thermogravimetric charecterization

The thermal studies were carried out using TGA, DTA. The DTA curves in fig. 3 and fig. 4 shows two dips or valleys at temperatures of 70°C and 1080°C. These valleys indicate the absorption of energy. The reason for this is not clear here. But, at temperature up to 100°C the crystal shows endothermic behavior, while above 100°C, the behavior is exothermic up to 1000°C.

After 1000°C the crystal shows the endothermic behavior again with a valley at near 1080°C.

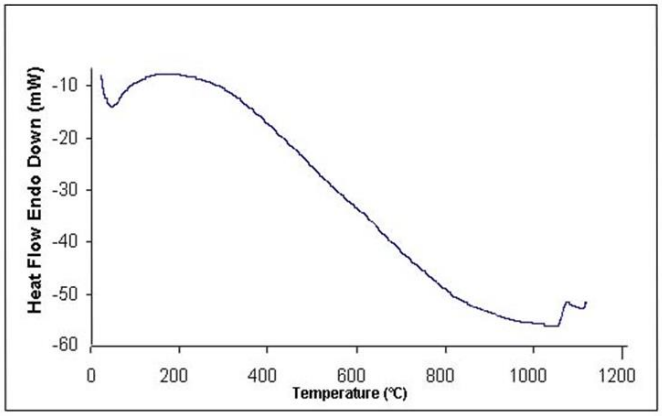

Fig.3: TGA - DTA curve for heat flow (mW) Vs temperature.

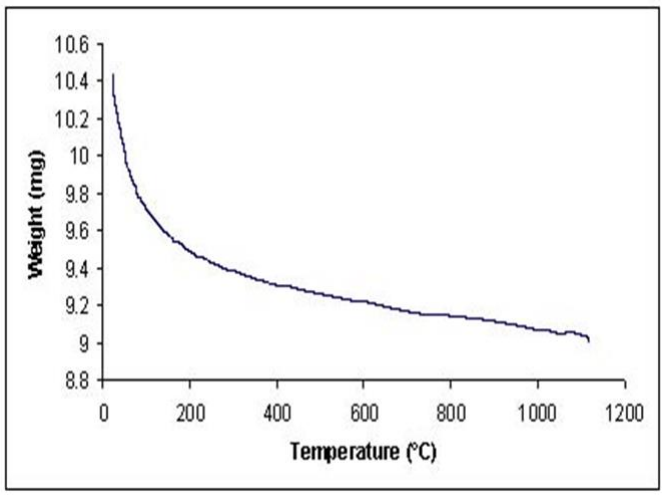

Fig.4: TGA - DTA curve for weight (mg) Vs temperature.

IV. CONCLUSION

A single phase of $KNbO_3$ with Al dopant was successfully synthesised by hydrothermal method. . From the XRD studies the lattice parameters of orthorhombic structure $KNbO_3$ single crystals are calculated as a=5.6632 Å, b=3.9745 Å, c=5.7218 Å. The crystals of average 1.2 to 2.5 μm size of $KNbO_3$ ceramics are measured from SEM. Two dips in DTA at 70°C and 1080°C indicated the absorption of energy. At temperature up to 100°C the crystal shows endothermic behavior, while above 100°C, the behavior is exothermic up to 1000°C. After 1000°C the crystal shows the endothermic behavior again with a valley at near 1080°C.

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