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Short communication

Synthesis of $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powders through the molten salt method

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Abstract

The $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powder synthesis through molten salt method was investigated in the temperature range of 650–700 °C for 2–4 h. The XRD results indicated that the optimal synthesizing temperature for molten salt method was 700 °C, significantly lower than that for conventional processing route of solid state reaction method, where a calcining temperature of 850 °C was needed. The SEM results revealed better crystallization of the powders obtained through molten salt method, compared with those through the conventional processing route of solid state reaction method.

Keywords: $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powder; Molten salt method; Synthesis

1. Introduction

Lead oxide based ferroelectrics, represented by lead zirconate titanate (Pb(Zr, Ti)O_3, PZT) are widely used for piezoelectric actuators, sensors and transducers due to their excellent piezoelectric properties [1,2]. However, lead is a heavy metal and its toxicity is well known. Therefore, it is necessary to develop lead-free piezoelectric ceramics to replace PZT based ceramics. Sodium bismuth titanate ($Na_{0.5}Bi_{0.5}TiO_3$, NBT), discovered by Smolenskii et al. in 1960 [3], is considered to be one of candidates of lead-free piezoelectric ceramics. However, NBT has a drawback of high conductivity and high coercive field E_c which cause problems in polarizing process. Thus, solid solution of NBT with $BaTiO_3$ [4], $SrTiO_3$ [5], $K_{0.5}Bi_{0.5}TiO_3$ [6,7], $NaNbO_3$ [8], and $BiFeO_3$ [9] has been used to improve its piezoelectric properties. $Bi_{0.5}(Na_{1-x-y}K_xLi_y)_{0.5}TiO_3$, as one kind of lead-free NBT ceramics, has been reported to be a promising system [10–12].

In the present study the $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powder synthesis was investigated using molten salt method. In addition, the powder synthesis was also conducted through the conventional solid state reaction method for comparison. The effects of both the synthesizing temperature and reaction time were explored and the obtained results were discussed.

2. Experimental procedure

In the present study commercial ceramic powders of Bi_2O_3 (99.0 wt%), TiO_2 (98.0 wt%), Na_2CO_3 (99.8 wt%), K_2CO_3 (99.0 wt%), and Li_2CO_3 (97.0 wt%) were used as the raw materials. A powder mixture of these raw materials according to the stoichiometric ratio of $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ was prepared as a reactant. An inorganic salt mixture was prepared using KCl–NaCl (99.0 wt%) as a solvent. The prepared reactant and the solvent were mixed in a weight ratio of 1–3 and ball milled for 12 h in an ethanol medium. After ball milling, the obtained slurry was dried at 60 °C for 6 h to remove the ethanol. Heat treatment was then carried out in the temperature range of 650–700 °C for 2–4 h in a muffle furnace. After heat treatment, the products were washed using hot deionized water to remove the residual salt. The products were then dried and characterized using an X-ray diffractometer (XRD, Model D8 Advance, Bruker, Germany) for phase composition and a scanning electron microscopy (SEM, Model JSM-5800, JEOL, Japan) for particle morphology.

In order to compare these results with those of powders synthesized through the conventional solid state reaction

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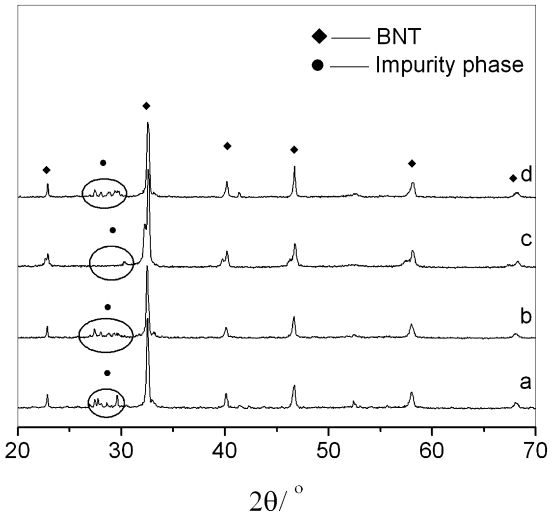

Fig. 1. XRD patterns of $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powders obtained through the conventional solid state reaction method at temperatures of 750 °C (a), 800 °C (b), 850 °C (c), and 900 °C (d) for 2 h.

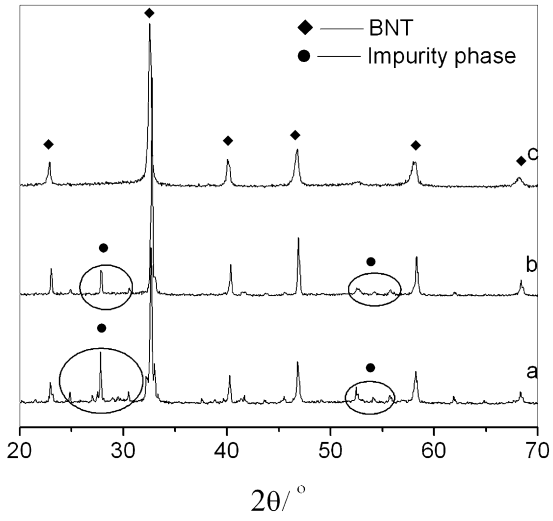

Fig. 3. XRD patterns of $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powders obtained through molten salt method at temperatures of 650 °C (a), 675 °C (b), and 700 °C (c) for 4 h.

method, the $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powders were also prepared by directly calcining the reactant at temperatures in the range of 750–900 °C for 2–4 h and the obtained product was characterized using the XRD and SEM.

3. Results and discussion

Fig. 1 shows the XRD patterns of the $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powders obtained through the conventional solid state reaction method at different temperatures for 2 h. Some unidentified impurities were found to be present in the powders at all temperatures. Their amounts, however, were different to some extent. The lowest peaks for these impurities in Fig. 1(c) indicated an optimal powder synthesis temperature of 850 °C. The XRD patterns of the powders synthesized through the molten salt method at different temperatures for 2 h are shown in Fig. 2. The lines in Fig. 2 were similar to those in Fig. 1. The synthesis temperatures in Fig. 2, however, were much lower than those in Fig. 1. As the reaction time increased to 4 h (see Fig. 3), the powders, however, showed much better

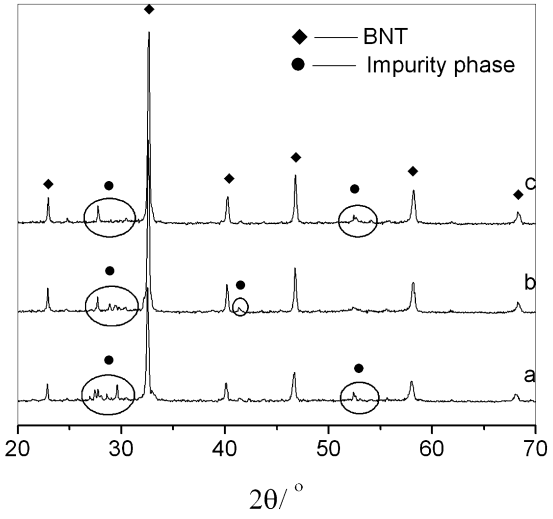

Fig. 2. XRD patterns of $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powders obtained through the molten salt method at temperatures of 650 °C (a), 675 °C (b), and 700 °C (c) for 2 h.

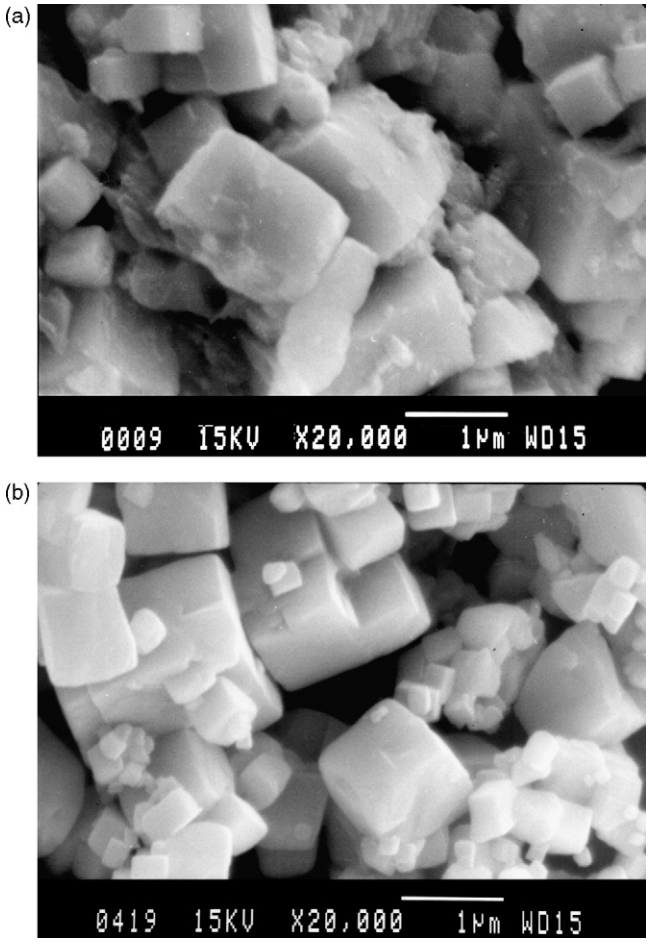

Fig. 4. SEM morphologies of the $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powders synthesized both through the conventional solid state reaction method at 850 °C for 2 h (a) and through the molten salt method at 700 °C for 4 h (b).

crystallization than those of powders for 2 h and the impurities disappeared at 700 °C (Fig. 3(c)), indicating a favorable synthesizing temperature of 700 °C and a reaction time of 4 h. Fig. 4 shows the SEM morphologies of the $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powders synthesized both through the conventional solid state reaction method at 850 °C for 2 h and through the molten salt method at 700 °C for 4 h. The $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ particles in Fig. 4(b) had better surface finish than those in Fig. 4(a), indicating better crystallization of the particles.

4. Conclusions

The $Bi_{0.5}(Na_{0.7}K_{0.2}Li_{0.1})_{0.5}TiO_3$ powder synthesis through molten salt method was investigated in the temperature range of 650–700 °C for 2–4 h. Powder synthesis was also conducted through the conventional processing route of solid state reaction method in the temperature range of 750–900 °C for 2–4 h for comparison. The XRD results indicated that the favorable synthesizing temperature for molten salt method was 700 °C. This was significantly lower than that for solid state reaction method, where a calcining temperature of 850 °C was needed. The SEM results revealed better crystallization of the powders obtained through the molten salt method, compared to the conventional solid state reaction method.

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