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Synthesis of Ferroelectric $Na_{0.5}Bi_{0.5}TiO_3$ by MSS (Molten Salt Synthesis) Method

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1. Introduction

Environmental destruction has been a serious problem worldwide. One problem is the release of harmful materials (e.g., Cd, Hg, Pb) from electrical industries. Thus, the restriction on hazardous substance (RoHS) will be enforced soon to prevent the release of harmful waste (Hiruma et al., 2007; Panda, 2009). Lead – based ferroelectric ceramics represented by Pb $(Zr,Ti)O_3$ (PZT), have been widely used for piezoelectric transducers, sensors and actuators due to their excellent piezoelectric properties. However, the evaporation of harmful lead oxide during preparation causes a crucial environment problem. Therefore, it is necessary to develop environment – friendly lead – free piezoelectric ceramics to replace the PZT – based ceramics, which has become one of the main trends in present development of piezoelectric materials. Sodium bismuth titanate, $Na_{0.5}Bi_{0.5}TiO_3$ (abbreviate as NBT), discovered in 1960 (Smolenskii et al., 1960), is considered to be a promising candidate of lead – free piezoelectric ceramics (Pookmaneea et al., 2001; Isupov, 2005; Panda, 2009; Zhou et al., 2010).

NBT is a relaxor ferroelectric material with the general formula $A'_xA''_{1-x}BO_3$. The ferroelectricity in NBT ceramic is attributed to $(Bi_{1/2}Na_{1/2})^{2+}$ ions, especially Bi^{3+} ions at the ,,A" site of the perovskite structure (ABO_3) and due to rhombohedral symmetry at room temperature. It has high Curie temperature (T_c = 320°C), and shows diffuse phase transition (Suchanicz et al., 2000; Suchanicz et al., 2001; Raghavender et al., 2006). However, the piezoelectric properties of NBT ceramics are not good enough for most practical uses. In order to enhance the properties and meet the requirements for practical uses, it is necessary to develop new NBT – based ceramics (Raghavender et al., 2006; Zhou et al., 2010). Researches have investigated many dopants into NBT ceramics (Panda, 2009). Also, it is desirable to fabricate ceramics with a textured microstructure in order to improve the properties (Hao et al., 2007).

The ferroelectric ceramic powders are synthesized trough conventional solid – state method which needs high calcination temperature and repeated grindings (Lu et al., 2010). In order to eliminate these defects, the wet chemical synthesis techniques have been developed, for instance hydrothermal method (Cho et al., 2006; Wang et al., 2009), sol – gel method (Xu et al., 2006; Mercadelli et al., 2008), and molten salt method (Zeng et al., 2007; Li et al., 2009). But the hydrothermal and sol – gel synthesis are usually long and complex processes, use hazardous solvents such as 2-methoxyethanol, and result in agglomerated particles (Bortolani & Dorey, 2010). Moreover, in the sol –gel method the cost of starting materials is high (Li et al., 2009).

Molten salt synthesis (MSS) is a process that yields large amounts of ceramic powders in a relatively short period of time. Moreover, it is a suitable method for preparation of complex oxide compounds with anisotropic particle morphologies. In this technique starting materials are mixed together with a salt (usually alkaline chloride and sulphate) and then heat treated at a temperature higher than the melting point of the salt. The melting temperature of the salt system can be reduced by using a eutectic mixture of salts, e.g. the use of NaCl – KCl instead of pure NaCl reduces the melting point from 801 to 657°C. A reaction between the precursors takes place in the molten salt (the flux) and the solid product obtained is separated by washing of the final mixture with hot deionised water. The typical starting materials are oxides, but carbonates, oxalates and nitrates can also be used. There are several requirements for the selection of salt to be used for MSS. First, the melting point of the salt should be relatively low and appropriate for synthesizing of the required phase. Second, the salt should possess sufficient aqueous solubility in order to eliminate it easily after synthesis by washing. Finally, the salt should not react with the starting materials or the product (Bortolani & Dorey, 2010; Hao et al., 2007). MSS has been used to form various ceramic powders such as niobates relaxors (Yoon et al., 1998), $Bi_4Ti_3O_{12}$ (Kan et al., 2003), $ZnTiO_3$ (Xing et al., 2006), $BaTiO_3$ (Zhabrev et al., 2008) and $Pb(Zr, Ti)O_3$ (Cai et al., 2008; Bortolani & Dorey, 2010).

It was found that ternary compound $Na_{0.5}Bi_{0.5}TiO_3$ was formed in the solid – state process through the intermediate binary compound, i.e. bismuth titanate – $Bi_4Ti_3O_{12}$ (Zaremba, 2008). $Bi_4Ti_3O_{12}$ (abbreviate as BIT) belongs to the Aurivillius family with a general formula $(Bi_2O_2)[A_{m-1}(B)_mO_{3m+1}]$, which consists of $(Bi_2O_2)^{2+}$ sheets alternating with $(Bi_2Ti_3O_{10})^{2-}$ perovskite – like – layers (Aurivillius, 1949, as cited in Stojanović et al., 2008). In general formula *m* represents the number of octahedra stacked along the direction perpendicular to the sheets, and A and B are the 12- and 6- fold coordination sites of perovskite slab, respectively. This kind of structure promotes plate – like morphology (Dorrian et al., 1971, as cited in Stojanović et al., 2008).

In this paper, $Na_{0.5}Bi_{0.5}TiO_3$ powders were prepared by molten salt synthesis in the presence pure NaCl or NaCl - KCl as fluxes. The first stage of the study related to direct synthesis of NBT via MSS from Na_2CO_3, Bi_2O_3 and TiO_2. For comparison, the synthesis of NBT by the conventional method (CMO – conventional mixed oxides) was investigated. The second stage included obtaining intermediate binary compound BIT via MSS from oxide precursors, i.e. Bi_2O_3 and TiO_2, and then synthesis of NBT via MSS using BIT, Na_2CO_3 and TiO_2 as starting materials.

The details pertaining to studies of synthesis of NBT and an Aurivillius – structured BIT precursor are reported in the following sections.

2. Synthesis of ferroelectric $Na_{0.5}Bi_{0.5}TiO_3$

Chemically pure powders of Bi_2O_3, TiO_2 (rutile) and Na_2CO_3 were used as starting materials. The $Na_{0.5}Bi_{0.5}TiO_3$ (NBT) was prepared by the following two routes:

$$Na_2CO_3 + Bi_2O_3 + 4TiO_2 \rightarrow 4Na_{0.5}Bi_{0.5}TiO_3 + CO_2 \quad (1)$$

$$Bi_4Ti_3O_{12} + 5TiO_2 + 2Na_2CO_3 \rightarrow 8Na_{0.5}Bi_{0.5}TiO_3 + 2CO_2 \quad (2)$$

In route (1), the starting materials were weighed in the proportion to yield NBT and mixed in isopropyl alcohol employing an agate mortar and pestle for 1 h. Using MSS method, the dry mixture of the precursors in the stoichiometric composition was mixed with an aqual

weight of salt. Salts used in this experiment were NaCl and eutectic mixture of 0.5NaCl – 0.5KCl, i.e. 43.94% NaCl – 56.06% KCl (by weight). The mixture of the precursors and flux was dried at 120°C for 2 h for complete removal of isopropyl alcohol, placed in a Pt crucible and heated in a sealed alumina crucible (to prevent salt evaporation) at temperatures between 800°C and 1100°C for a different time period. After thermal treatment the chlorides were removed from the products by washing with hot deionized water several times until the filtrates gave no reaction with silver nitrate solution. The powders were finally dried at 100°C for 2 h. NBT powders were also prepared by a conventional mixed oxide method (CMO) for comparison. All the syntheses were carried out in a conventional electric furnace.
Platelike $Bi_4Ti_3O_{12}$ (BIT) particles were obtained by MSS method in 0.5NaCl – 0.5KCl flux (abbreviate as NaCl – KCl) in the same way as described above. Temperature of thermal treatment ranging from 700°C to 1100°C for a different time period.
In route (2), BIT crystals produced earlier were subjected to second molten salt synthesis. Na_2CO_3 and TiO_2 were added to give the total composition of NBT. Again, pure NaCl or NaCl – KCl mixture was added (weight ratio of precursors to flux = 1:1).
Finally, the phase composition of the synthesized samples was analyzed by the powder X-ray diffraction (XRD; model 3003 TT, Seifert) using Ni – filtered Cu K_α radiation. The microstructure was observed by a scanning electron microscope (SEM; model BS 340, Tesla). The samples were coated by a gold layer by using a metal – coating plant under a vacuum. X–ray energy dispersive spectra (EDS) were measured using a Hitachi S-3400 N scanning electron microscope with an EDS system Thermo Noran.

2.1 Synthesis of $Na_{0.5}Bi_{0.5}TiO_3$ from Bi_2O_3, TiO_2 and Na_2CO_3

Fig. 1 represents the XRD patterns of the selected powders synthesized through route (1), i.e. directly from Bi_2O_3, TiO_2 and Na_2CO_3 via MSS (NaCl flux) and CMO. Similar trends were also observed for NBT produced using NaCl- KCl flux. The particle morphology of the starting materials and synthesized powders is compared in Figs 2 – 4.
NBT perovskite phase was observed in all the prepared samples. A comparison of interplanar spacings determined from XRD patterns of the samples prepared by a conventional solid state reaction and via MSS shows that agreement is quite satisfactory. Analysis of XRD patterns of NBT samples obtained via MSS has not shown displacement of maxima of diffraction peaks as the NaCl-KCl flux was used.
Isometric particles are found to exist in the samples of NBT. Typical micrograph of the NBT powder prepared by CMO is shown in Fig. 3a. There is high degree of agglomeration in this powder. The NBT particles prepared directly by CMO and MSS (NaCl flux) are very small (about 1 μm). The size of the particles increased with increasing temperature, especially, as NaCl-KCl flux was used. Probably, this is mainly due to the different melting points for each salt used. NaCl and 0.5 NaCl – 0.5 KCl have melting points of about 800°C and 650°C, respectively.
According to (Cai et al., 2007, as cited in Bortolani & Dorey, 2010) the solubility of the starting materials in the molten salt plays an important role in the synthesis as it has an influence on the final product morphology. For a simple two reactant system, two different cases can be distinguished: either both reactants are equally soluble in the molten salt or one oxide is more soluble than the other (Li et al., 2007, as cited in Bortolani & Dorey, 2010). In the first case (dissolution – precipitation mechanism) both reactants fully dissolve, react in the molten salt and the final product precipitates from the molten salt after formation. The shape of the product has no connection with the shape of the starting materials. In the second case, the more soluble precursor dissolves in the salt and diffuses to the less soluble

one. Here, at the surface, it reacts to final product. This template formation mechanism would result in a product morphology that is similar to that of the less soluble reactant which has acted as a template.

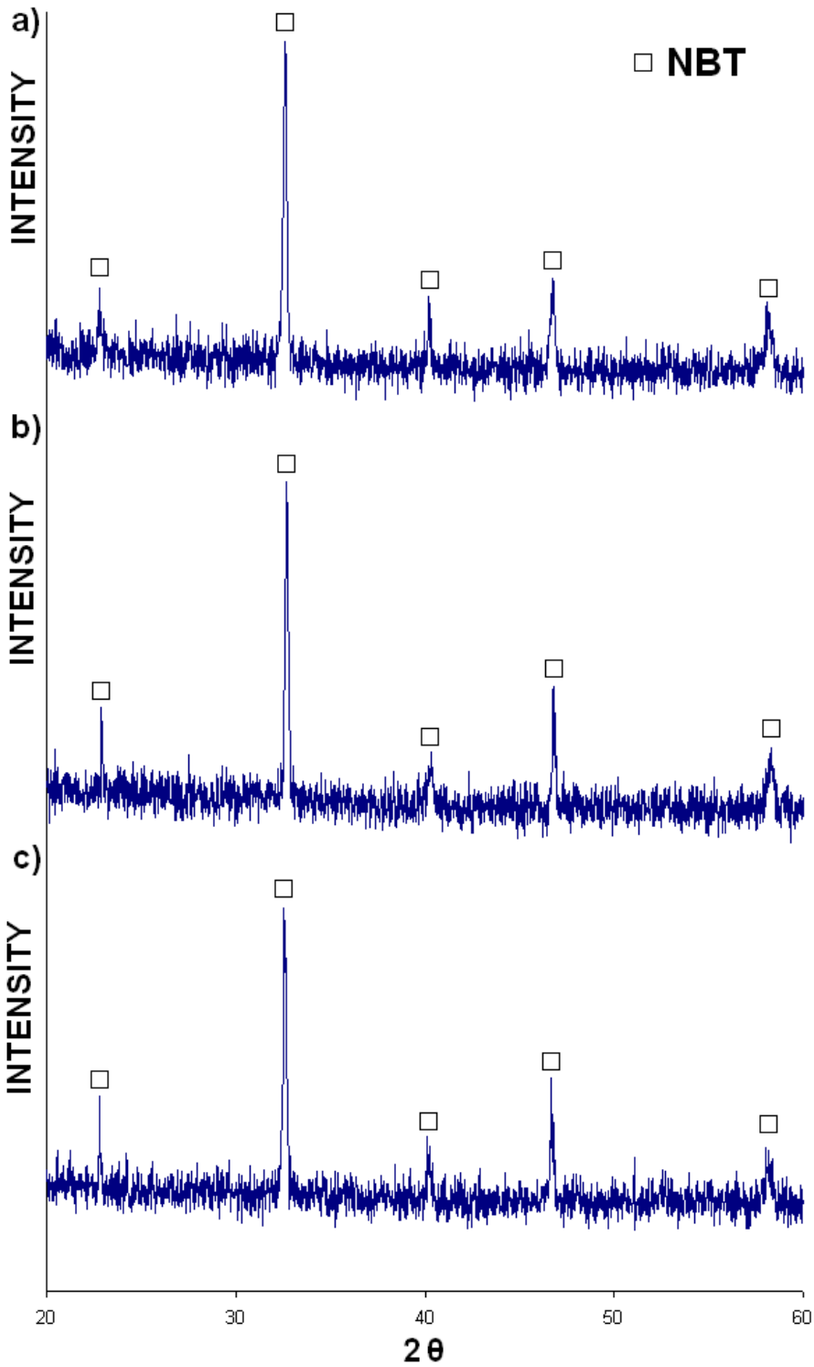
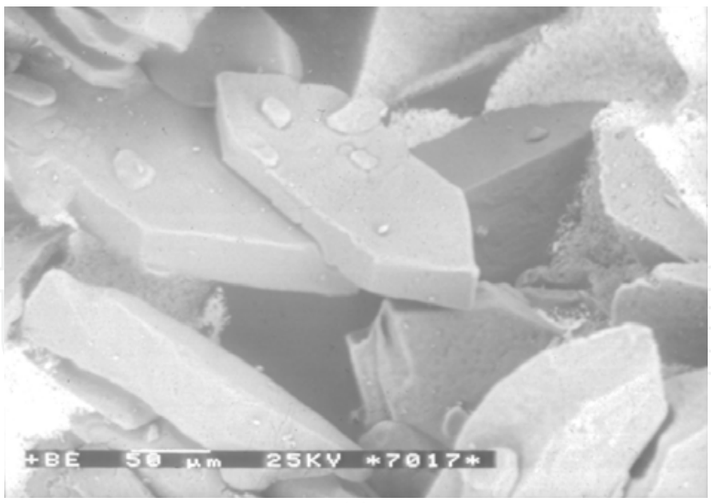
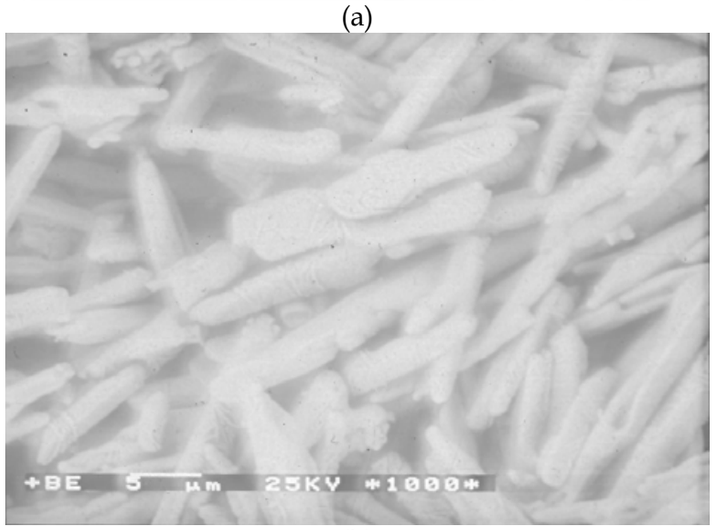


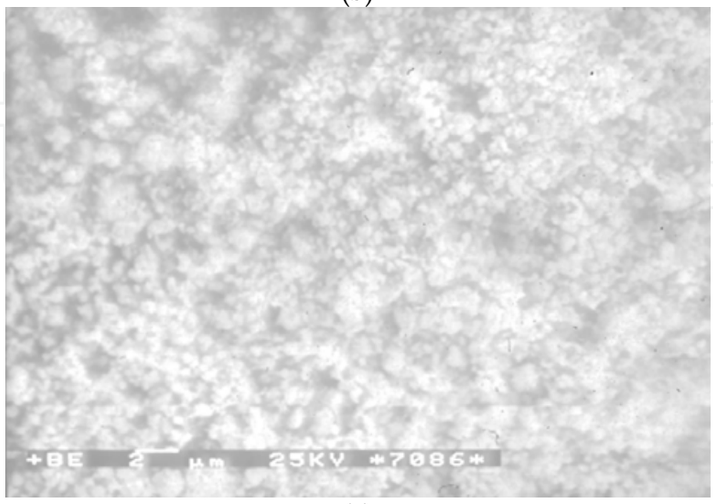
Fig. 1. XRD patterns of $Na_{0.5}Bi_{0.5}TiO_3$ powders fabricated through route (1): (a) via MSS (NaCl flux) at 950°C for 1.5 h; (b) via MSS (NaCl flux) at 1000°C for 4 h; (c) via CMO at 1000°C for 4 h



(a)

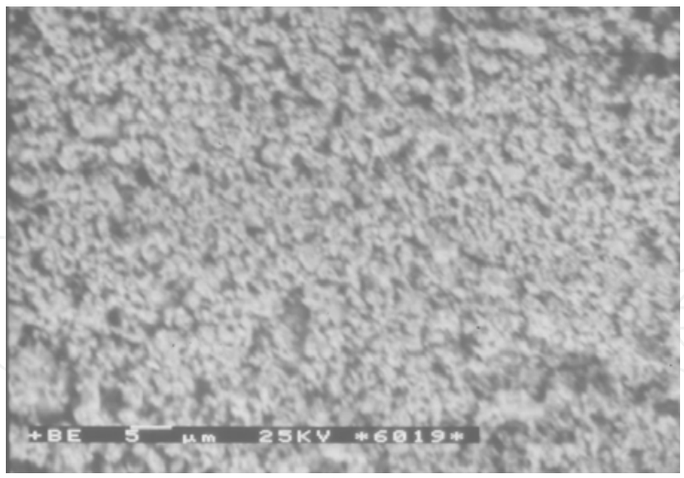


(b)

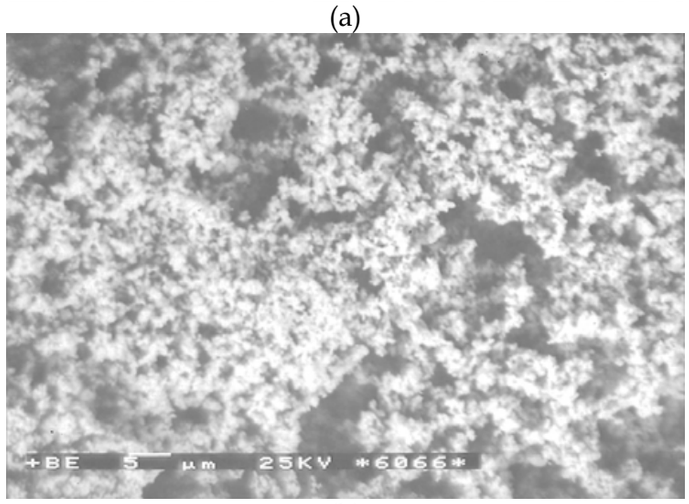


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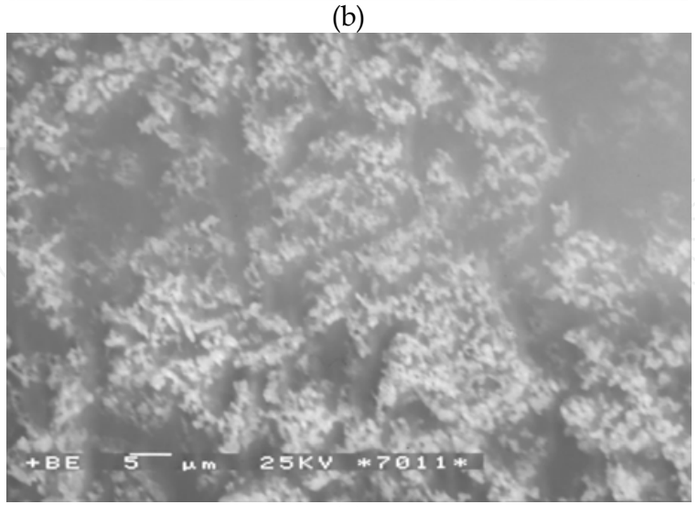
Fig. 2. SEM micrographs of the starting materials: (a) Na_2CO_3; (b) Bi_2O_3; (c) TiO_2 - rutile



(a)

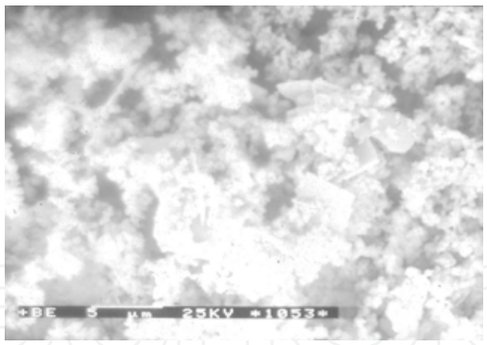


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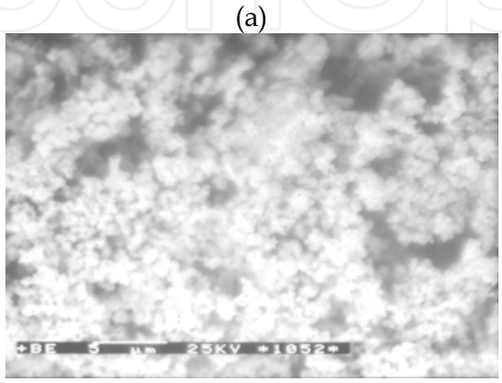


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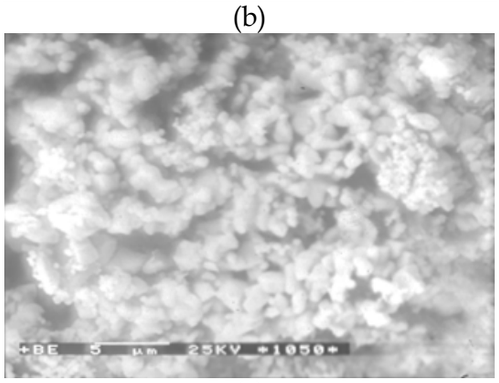
Fig. 3. SEM micrographs of $Na_{0.5}Bi_{0.5}TiO_3$ powders obtained through route (1): (a) via CMO at 950°C for 1.5 h.; (b) via MSS (NaCl flux) at 950°C for 1.5 h; (c) via MSS (NaCl flux) at 1000°C for 4 h



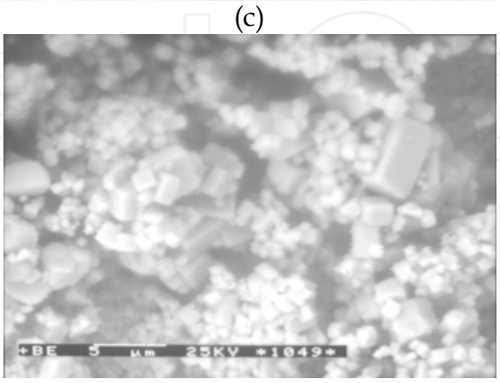
(a)



(b)



(c)



(d)

Fig. 4. SEM micrographs of $Na_{0.5}Bi_{0.5}TiO_3$ powders obtained through route (1) via MSS (NaCl - KCl flux) at different temperatures for 4 h: (a) 800°C; (b) 900°C; (c) 1000°C; (d) 1100°C

In the case of NBT the mechanism is further complicated due to the presence of (at least) 3 reactants. According to (Cai et al, 2007, as cited in Bortolani & Dorey, 2010) TiO_2 is not soluble in molten alkali chlorides. The final NBT morphology should be similar to the morphology of TiO_2.

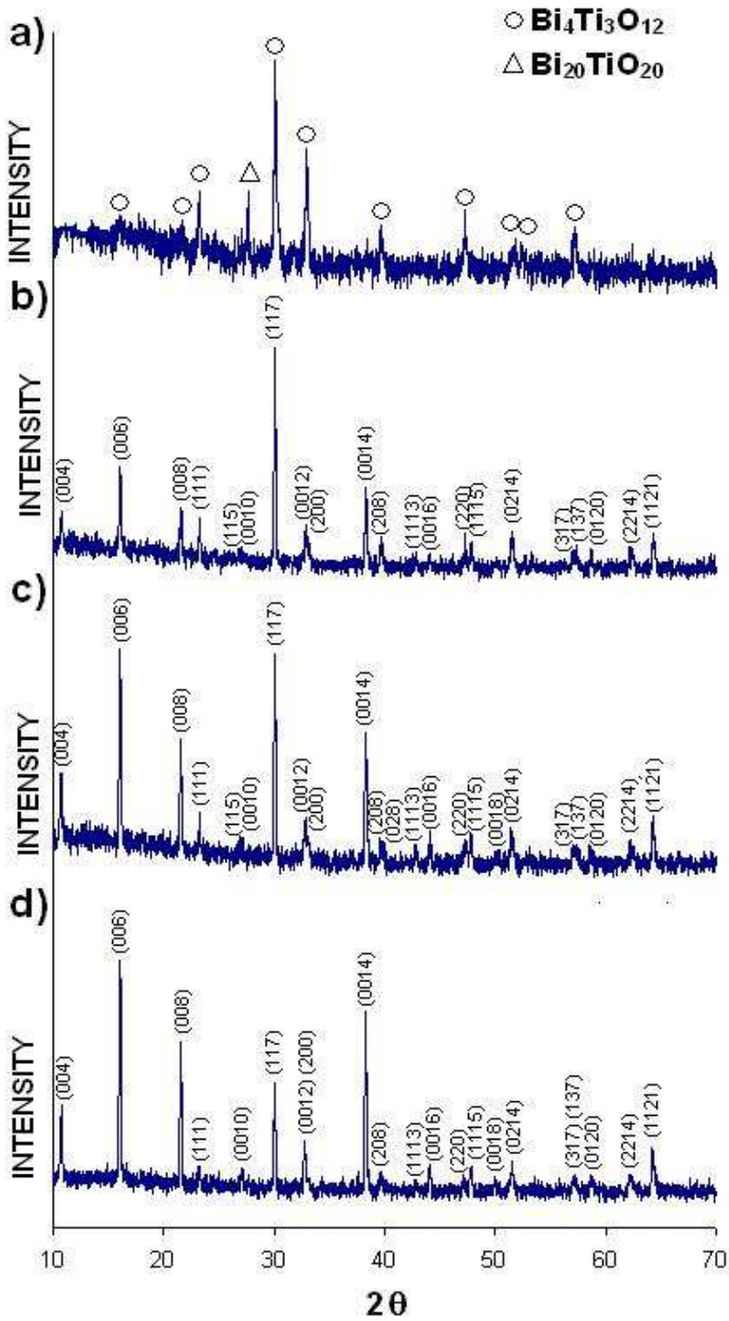
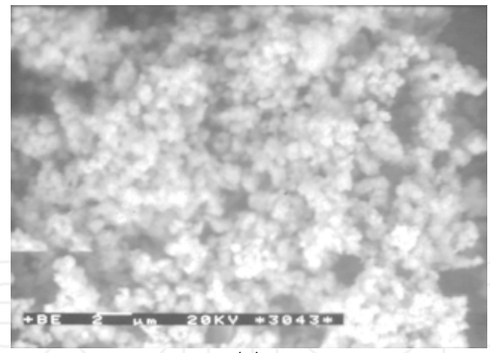
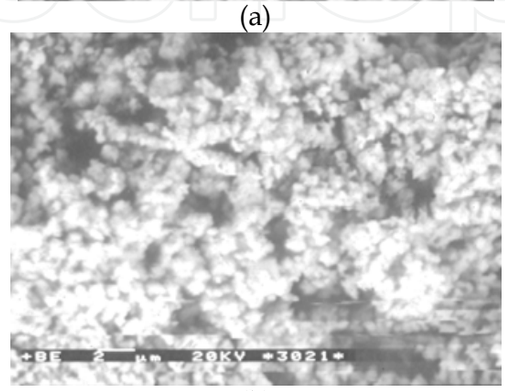


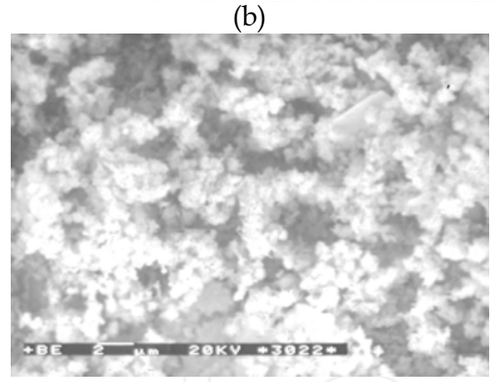
Fig. 5. XRD patterns of $Bi_4Ti_3O_{12}$ powders obtained via MSS at: (a) 700°C for 15 min; (b) 900°C for 30 min; (c) 900°C for 240 min; (d) 1000°C for 15 min (indexed peaks are those of $Bi_4Ti_3O_{12}$)



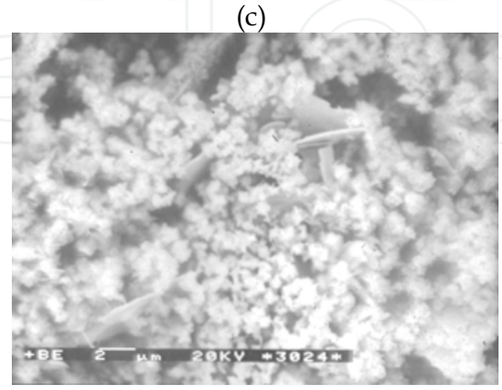
(a)



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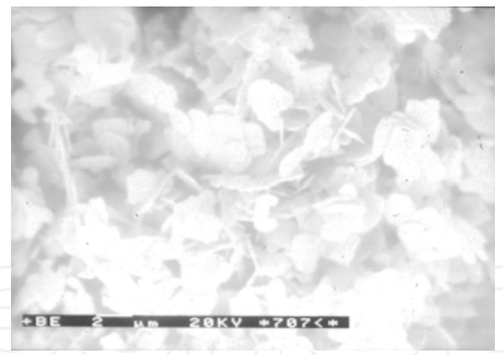


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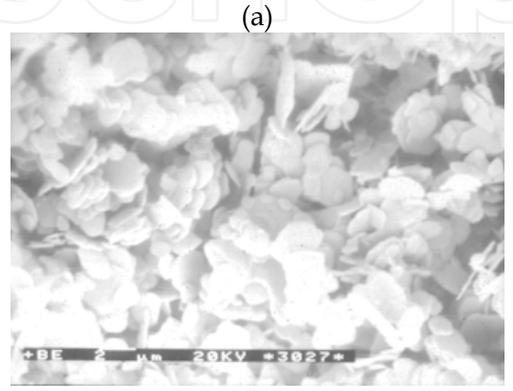


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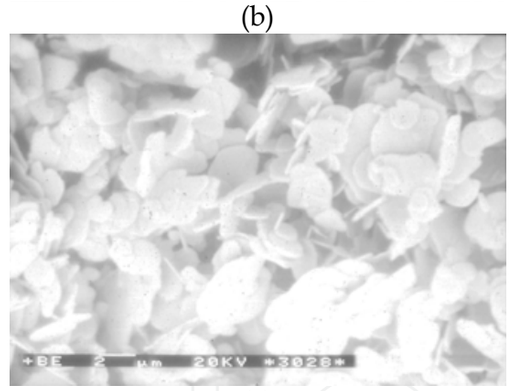
Fig. 6. SEM micrographs of $Bi_4Ti_3O_{12}$ powders obtained via MSS at 700°C for: (a) 15 min; (b) 30 min; (c) 60 min; (d) 120 min



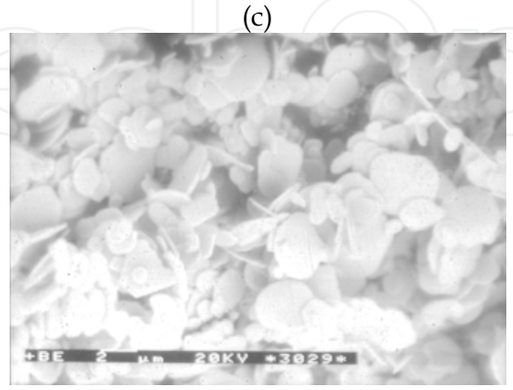
(a)



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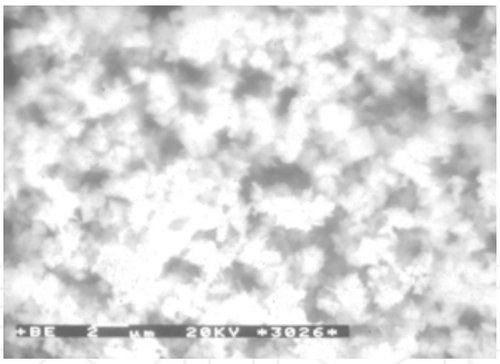


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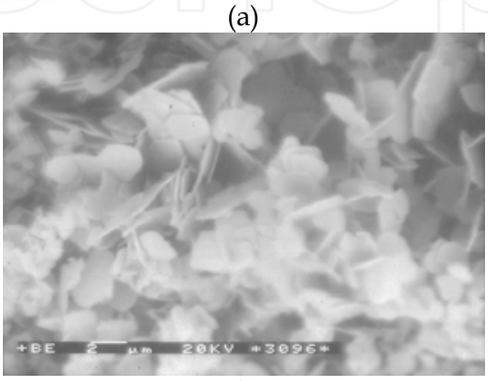


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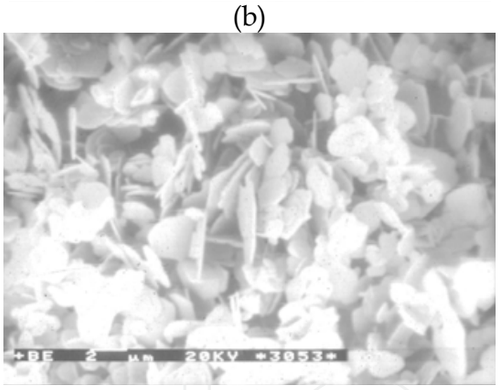
Fig. 7. SEM micrographs of $Bi_4Ti_3O_{12}$ powders obtained via MSS at 1000°C for: (a) 15 min; (b) 30 min; (c) 60 min; (d) 120 min



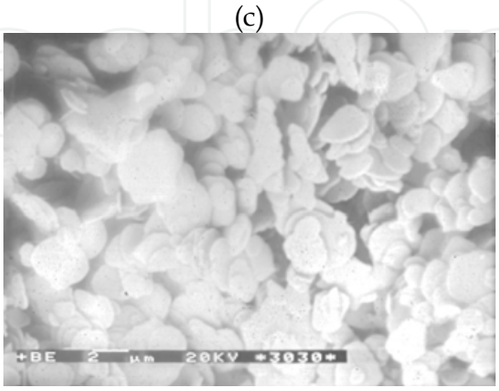
(a)



(b)



(c)



(d)

Fig. 8. SEM micrographs of $Bi_4Ti_3O_{12}$ powders obtained via MSS at different temperatures for 4 h: (a) 700°C; (b) 800°C; (c) 900°C; (d) 1000°C

2.2 Synthesis of $Bi_4Ti_3O_{12}$

The XRD patterns of selected samples BIT prepared from the mixture of Bi_2O_3 and TiO_2 via MSS (NaCl-KCl flux) are presented in Fig. 5. At 700°C, the phase $Bi_{12}TiO_{20}$ co-existed with BIT. Pure crystalline BIT was obtained after thermal treatment at 800°C for 15 min.. Increasing the temperature to 1100°C, the intensities of the (00*l*) diffraction lines were increased. These results indicate that during sample preparation for X-ray diffraction characterization, $Bi_4Ti_3O_{12}$ crystals with platelet morphology may align with (00*l*) planes parallel to the flat specimen holder.

Figs 6 – 8 show morphology and size of BIT crystals prepared at different temperatures. The synthesizing temperature could significantly influence the growth rate and crystallization habit of the BIT particles. Between 700°C and 800°C aggregate particles were formed. The powder synthesized at 700°C was composed of fine particles. The size of the primary particles increased and their shape changed from lumpy to plate-like with increasing temperature. Above 800°C discrete plate-like particles with increased particle size were formed. On the other hand, the effect of heating time on morphology and particle size is smaller. The degree of aggregation decreased with increasing prepare temperatures.

According to (Kimura & Yamaguchi, 1987) $Bi_4Ti_3O_{12}$ is formed via MSS by mechanism, when two reactants have comparable dissolution rates in molten salt. If this mechanism dominates during the formation process, the complex oxide powder with a characteristic or lumpy shape is formed, depending on the degree of interaction between the complex oxide and salt.

2.3 Synthesis of $Na_{0.5}Bi_{0.5}TiO_3$ from $Bi_4Ti_3O_{12}$, TiO_2 and Na_2CO_3

Fig. 9 shows the XRD patterns of the selected powders synthesized through route (2), i.e., from $Bi_4Ti_3O_{12}$ (BIT), TiO_2 and Na_2CO_3 via MSS (NaCl-KCl flux) at different temperatures. The diffraction lines were indexed based on the pseudocubic unit cell because of a small rhombohedral distortion of $Na_{0.5}Bi_{0.5}TiO_3$.

BIT has a BLSF (bismuth layer-structured ferroelectric) structure that is highly anisotropic, with the grain growth rate along the *c*-axis much lower than that along the *a (b)*-axis. So it is easy for them to form a plate-like morphology. Similar to the BIT particles, most of the NBT particles laid down with the *c*-axis aligning along the vertical direction during the sample preparation for the XRD analysis. So, they exhibit strong (100) and (200) diffraction peaks, especially, for higher temperatures of synthesis.

Fig. 10 shows the SEM micrographs of the NBT samples prepared from the BIT particles at different temperatures in the presence NaCl-KCl flux. Similar to the BIT particles, most of the NBT particles are large and of plate-like shape. Much larger crystals were grown at 1100°C, but the XRD pattern (Fig. 9d) shows that NBT co-existed with other crystalline phase (phases), probably it was related to the beginning of thermal decomposition of NBT.

It has also been found that the salt has a significant effect on the size of the synthesized particles. Fig. 11 shows the SEM micrographs of the ceramic powders synthesized in different fluxes at 1100°C. The particles for the powder synthesized in NaCl-KCl flux are larger than those synthesized in NaCl flux. NaCl-KCl flux at eutectic has a low melting point (650°C), so ions have a high diffusion rate at the synthesing temperature 1100°C. In order to determine the composition of the prepared samples, energy dispersive X-ray spectroscopy (EDS) data (Fig. 12) performed on a samples synthesized in the presence NaCl-KCl and NaCl flux show that the chemical components of the samples are the elements Na, Bi, Ti and O (without K from KCl for the sample obtained in the presence NaCl-KCl).

As a member of the BLSFs, BIT consist of the $(Bi_2Ti_2O_{10})^{2-}$ (pseudo-) perovskite layers interleaved by $(Bi_2O_2)^{2+}$ fluorite layers. After the reaction with the complementary reactants (Na_2CO_3 and TiO_2), the layer-structured BIT particles were transformed to the perovskite NBT. Although there are works reporting the transformation as a process from a lamellar phase to a perovskite phase (Schaak & Mallouk, 2000), the process involving the $(Bi_2O_2)^{2+}$ layers changing to the perovskite structure is still unclear.

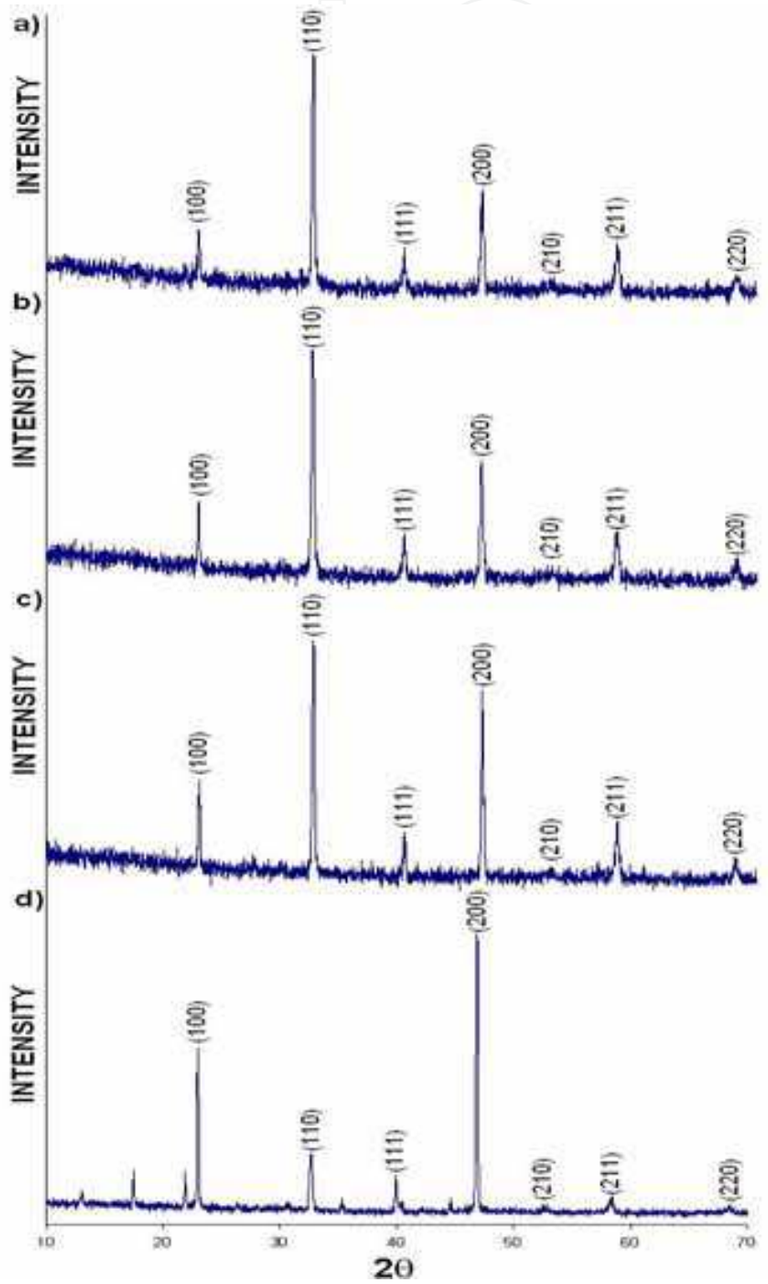
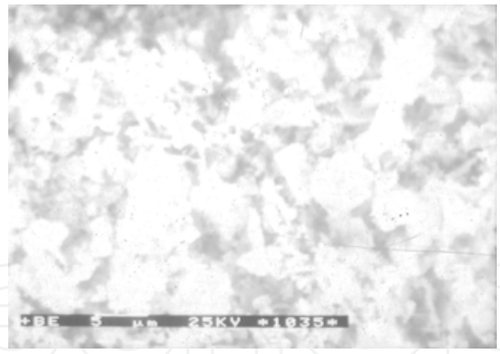
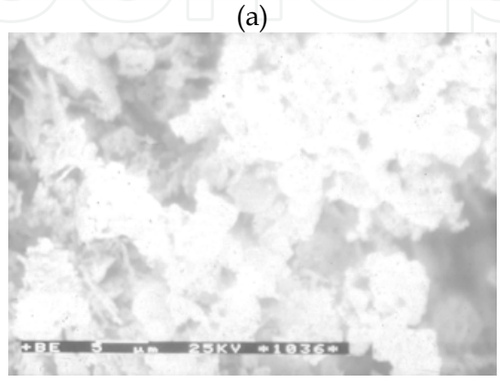


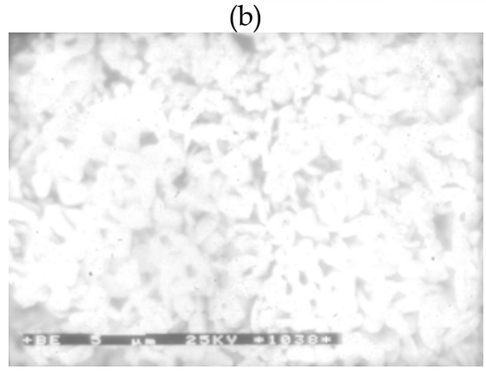
Fig. 9. XRD patterns of $Na_{0.5}Bi_{0.5}TiO_3$ powders fabricated through route (2) via MSS (NaCl - KCl flux) at different temperatures for 4 h: (a) 800°C; (b) 900°C; (c) 1000°C; (d) 1100°C(indexed peaks are those of $Na_{0.5}Bi_{0.5}TiO_3$)



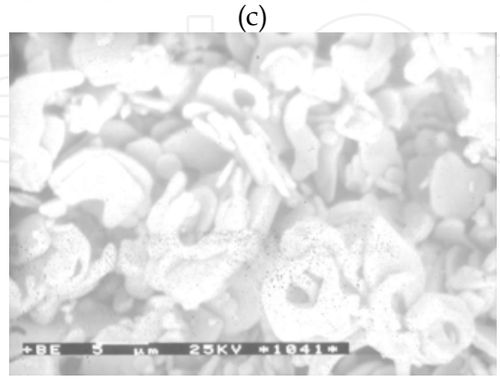
(a)



(b)

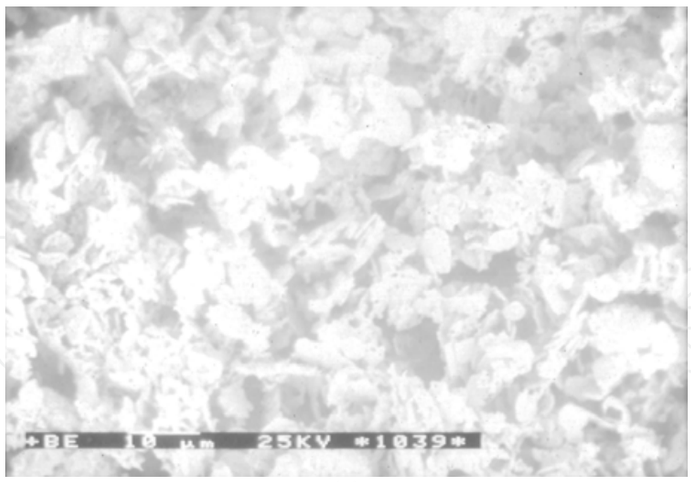


(c)

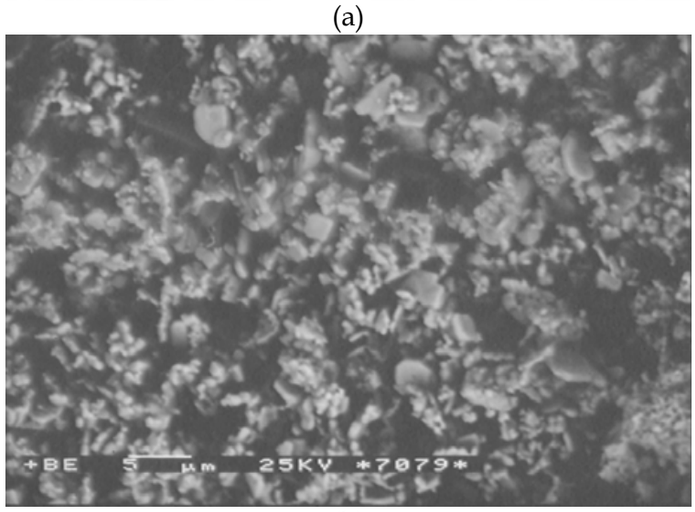


(d)

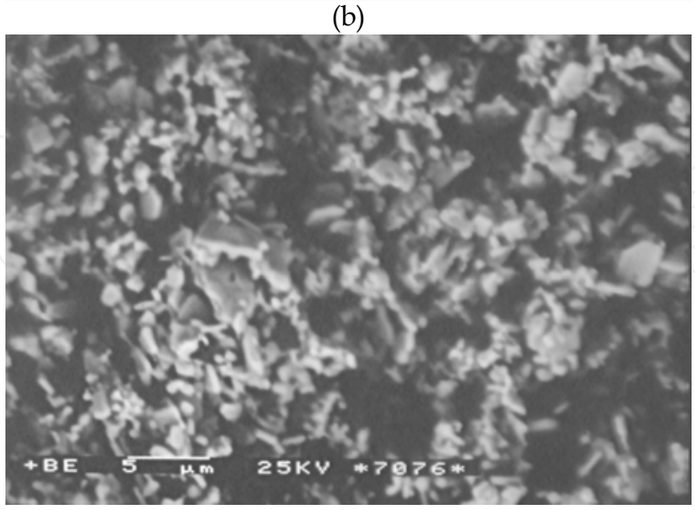
Fig. 10. SEM micrographs of $Na_{0.5}Bi_{0.5}TiO_3$ powders obtained through route (2) via MSS (NaCl - KCl flux) at different temperatures for 4 h: (a) 800°C; (b) 900°C; (c) 1000°C; (d) 1100°C



(a)



(b)



(c)

Fig. 11. SEM micrographs of $Na_{0.5}Bi_{0.5}TiO_3$ powders obtained through route (2) via MSS at 1100°C for 4 h: (a) NaCl - KCl flux; (b, c) NaCl flux

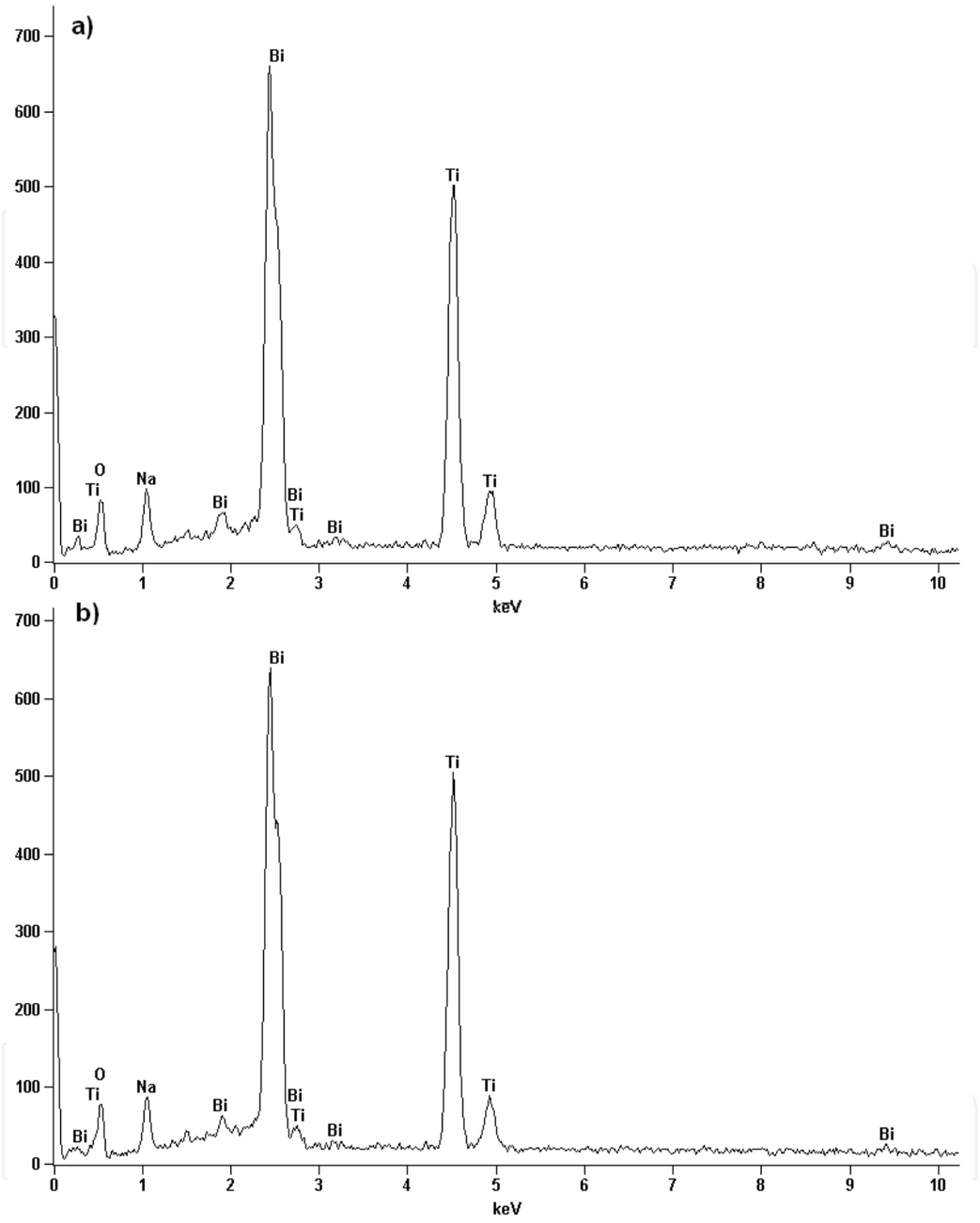


Fig. 12. EDS spectra of of $Na_{0.5}Bi_{0.5}TiO_3$ powders obtained through route (2) via MSS: (a) NaCl- KCl flux; (b) NaCl flux

3. Conclusion

NBT has a perovskite structure with high symmetry, therefore it is difficult to obtain large anisotropic NBT particles by methods as conventional solid-state reaction process or molten salt synthesis. Using Bi_2O_3, TiO_2 and Na_2CO_3 as starting materials, the equiaxed particles NBT

were obtained. NBT anisotropic particles with grain orientation were synthesized by conversion of BIT crystals with layered structure. Owing to its highly anisotropic structure, plate-like BIT was firstly fabricated in the NaCl-KCl flux. The plate-like BIT was reacted with the complementary Na_2CO_3 and TiO_2 in the presence of chloride flux, finally transformed to the perovskite NBT and maintained its morphology nearly unchanged. NBT particles show preferred orientation with the (*h*00) plane. The powder synthesized in 0.5 NaCl – 0.5 KCl flux has the larger particles than those synthesized in pure NaCl. The increase of temperature and soaking time of synthesis can make the plate-like grains of NBT more distinct and discrete. The NBT particles prepared in this experiment can be used to prepare ceramics with more uniform grain orientation, i.e., textured ceramics for improving piezoelectric properties.

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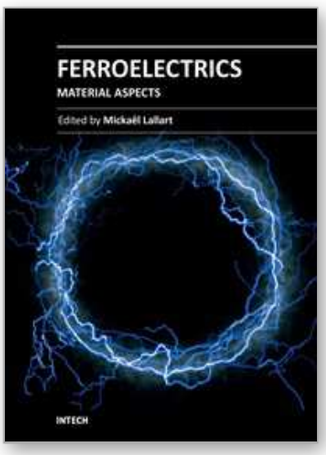
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Ferroelectric materials have been and still are widely used in many applications, that have moved from sonar towards breakthrough technologies such as memories or optical devices. This book is a part of a four volume collection (covering material aspects, physical effects, characterization and modeling, and applications) and focuses on ways to obtain high-quality materials exhibiting large ferroelectric activity. The book covers the aspect of material synthesis and growth, doping and composites, lead-free devices, and thin film synthesis. The aim of this book is to provide an up-to-date review of recent scientific findings and recent advances in the field of ferroelectric materials, allowing a deep understanding of the material aspects of ferroelectricity.

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