Alkali volatilization of (Li,Na,K)NbO$_3$-based piezoceramics and large-field electrical and mechanical properties

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While lead-free (Li,Na,K)NbO$_3$ piezoceramics have a high Curie temperature ($T_C > 400$ °C) and an excellent piezoelectric constant ($d_{33} > 200 \text{ pC/N}$), the process window regarding the sintering temperature and dwelling time is narrow. Inappropriate sintering conditions can induce the volatilization of A-site alkali ions, leading to a deterioration in dielectric and piezoelectric properties. However, the effect of alkali volatilization on the ferroelastic properties is still uncertain. In this study, (Li,Na,K)NbO$_3$ piezoceramics were deliberately exposed to the sintering temperature for varying times to compare the susceptibility of the effect of alkali volatilization on the ferroelastic properties and compared to changes in the ferroelectric and ferroelastic and dielectric properties.

Key-words : Lead-free piezoceramics, Alkali volatilization, Electrical hardening, Ferroelasticity

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1. Introduction

Piezoelectric materials are an integral part of modern-day electronics because of their ability to transduce an electrical signal into a mechanical strain, and vice versa. Lead-free piezoceramics have been widely investigated over the past decades because of their sustainability in regards to the environment compared to lead-based piezoceramics.\(^1\)\(^-\)\(^3\) A promising alternative to lead-based piezoceramics was found in lead-free (Na,K)NbO$_3$ (NKN) ferroelectrics, which exhibit a high Curie temperature ($T_C > 400$ °C) and excellent piezoelectric constants. These notable piezoelectric properties of NKN-based materials seem to be caused by a phase coexistence. For instance, Na$_{0.3}$K$_{0.7}$NbO$_3$ has a polymorphic phase transition (PPT) between the orthorhombic ($Amn2$) and tetragonal ($P4mm$) phases at approximately $200$ °C. At this temperature, the piezoelectric properties are enhanced due to the coexistence of two phases. As such phase has different polarization directions, higher field-induced-polarization is enabled as more directions are available to be oriented.\(^4\)

As such, by shifting the PPT to lower temperatures via Li doping, this enhancement could be achieved at room temperature.\(^5\) The crystal structure of Li$_x$(Na$_{0.3}$K$_{0.7}$)$_{1-x}$NbO$_3$ (LNKN) is orthorhombic ($Amn2$) for $x < 0.05$,\(^5\) while a coexistence of monoclinic ($Pm$) and tetragonal ($P4mm$) phases appears for $0.05 \leq x \leq 0.07$ as determined by Raman spectroscopy\(^6\) and X-ray diffraction.\(^7\)\(^-\)\(^9\)

Meanwhile, most LNKN piezoceramics are synthesized within a narrow processing window regarding the sintering temperature and dwelling time. Slightly higher temperature or prolonged dwelling time at the sintering step can easily leads to significant volatilization of the alkali metal elements and abnormal grain growth, which results in inferior piezoelectric properties.\(^10\)\(^-\)\(^13\) For example, the piezoelectric $d_{33}$ constant of LNKN piezoceramics sintered at $1060$ °C showed the highest value of $314 \text{ pC/N}$, while LNKN piezoceramics sintered at $1080$ °C showed only $220 \text{ pC/N}$.\(^10\) Therefore, sintering studies have been conducted to find the critical sintering temperatures and dwelling times. Wang and Li reported that while a high temperature above $1050$ °C was needed to obtain well-densified LNKN piezoceramics, alkali volatilization (especially Na) proceeded at approximately $1000$ °C and above.\(^11\) On the other hand, Wang et al. reported that (Li$_{0.04}$Na$_{0.48}$K$_{0.48}$)-(Nb$_{0.80}$Ta$_{0.20}$)O$_3$ piezoceramics sintered at $1145$ °C for 1 h showed significant volatilization (particularly of K) when the ceramics were annealed without a protective atmosphere, which led to an increase of the PPT temperature.

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resulted in lithium disilicate (Li₂Si₂O₅), which is a eutectic.

In this study, the alkali volatilization effect on ferroelectric and ferroelastic properties was investigated. Regarding the defect structure of NKN, interactions of defect complexes and domain walls in B-site acceptor doped NKN have been reported, but the interaction of A-site vacancy caused by the alkali volatilization to ferroelectric and ferroelastic properties is still unclear. Hence, the alkali volatilization effect on ferroelectric and ferroelastic properties was investigated.

2. Experimental methods

Li₀.₀₆Na₀.₅₂K₀.₄₂NbO₃ containing 0.65 mol % Li₂CO₃, 1.3 mol % SiO₂, 0.2 mol % MnCO₃, and 0.5 mol % SrZrO₃ (hereafter, LNKN6-a) piezoceramics were synthesized by a conventional sintering method as described elsewhere. According to the report, the microstructure of LNKN6-a piezoceramics was sputtered with gold and analyzed in the SEM-EDS. The particle diameters on the ceramic surfaces were measured according to their Krumbein diameter (300 grains were counted for each sample). EDS spot measurements were performed using an acceleration voltage of 20 kV. The spot size of the EDS measurement was about 7 μm, and it was taken care of that the measurements were made by targeting the center of the particle. Quantitative elemental analysis on Na, K, and Nb on the sintered samples was performed using an inductively coupled plasma optical emission spectrometer (ICP-OES; SPECTRO GENESIS, SPECTRO Analytical Instruments).

Polarization—electric field (P–E) and strain—electric field (S–E) hysteresis loops were determined with a ferroelectric test system (TF Analyzer 2000, aixACCT). The post-annealed LNKN6-a piezoceramics before and after poling treatment were characterized for the hysteresis measurements. Three-continuous bipolar waves were applied to the samples to obtain information of switchable domains from a closed hysteresis loop, and then the polarization change of the second bipolar wave was extracted. At the time as-poled samples were measured bipolar P–E loop, samples were set in the parallel and the antiparallel to the poling direction to compare the internal bias field E_b. The hysteresis measurements were conducted at room temperature with bipolar waves of 5 kV/mm at 1 Hz. In contrast, stress-strain hysteresis loops were characterized in an original setup: a screw-type loading frame equipped with a differential dilatometer and a heating furnace.

3. Results and discussion

3.1 Microstructure and chemical analysis

Figure 1 shows the X-ray powder diffraction patterns of post-annealed LNKN6-a piezoceramics. According to previous reports, this material shows a phase coexistence between monoclinic (Pm) and tetragonal phase (P4mm).
All samples showed perovskite-type reflections, indexed with a pseudo cubic parent cell and denoted with a subscript pc. No secondary phases were detected even for the post-annealed samples. Besides the phase composition, changes in the microstructure can influence the electrical and mechanical properties of piezoceramics. For that reason, microstructural images and grain size distributions of LNKN6-a are shown in Fig. 2. With increasing the annealing time up to 8 h, the median diameter $d_{50}$ increased from 4.34 to 6.38 $\mu$m [Figs. 2(d) and 2(f)]. Note that the distribution shape of as-sintered was unimodal, while the distribution of the annealed sample for 8 h was bimodal due to the abnormal grain growth during the post-annealing process.\(^{12}\)

ICP-OES and EDS point analysis were conducted to investigate the chemical composition of the post-annealed samples. Figure 3 shows the results of ICP-OES analysis on Na, K, and Nb of the as-sintered and the annealed samples. Here, Nb was considered a non-volatile ion, so we calculated $(\text{Na} + \text{K})/\text{Nb}$ ratio. The atomic number ratio of Na/Nb and K/Nb in the as-sintered state was almost the same to the nominal composition,\(^{23}\) while Na/Nb and K/Nb slightly decreased by 0.12 and 0.08 in the annealed sample for 8 h. As a result, the atomic number of $(\text{Na} + \text{K})/\text{Nb}$ in the annealed sample for 8 h was lower than 0.02 from the nominal composition. However, the annealed sample for 2 h showed the opposite behavior. In the ICP-OES analysis, crushed powders from the cylinder-type samples were used. Therefore, on the assumption that alkali volatilization proceeded from the sample surfaces, the volatilization effect on the ICP results were maybe weakened. Figure 4 shows the EDS point analysis results of LNKN6-a piezoceramics. The atomic number ratio of $(\text{Na} + \text{K})/\text{Nb}$ was lower than its nominal composition by about 0.15 for each condition. Note that the detection Fig. 2. Microstructure of LNKN6-a piezoceramics. Backscattering images of (a) as-sintered, (b) annealed for 2 h, and (c) annealed for 8 h were shown. The grain size distribution of (d) as-sintered, (e) annealed for 2 h, and (f) annealed for 8 h were shown. 300 grains were measured for each sample by Krumbein method.\(^{19}\)

Fig. 3. ICP analysis results of (a) Na/Nb, (b) K/Nb, and (Na + K)/Nb on as-sintered, annealed for 2 h, and annealed for 8 h LNKN6-a piezoceramics. The nominal composition was indicated as a red dotted line.
sensitivity of sodium deteriorates when the excitation voltage is as high as 20 kV. Therefore, we could not conclude that the gap from the nominal composition of (Na + K)/Nb was due to alkali volatilization. On the other hand, the atomic number ratio of O/Nb gradually decreased from 2.70 for the as-sintered state to 2.36 for the annealed sample for 8 h. This decrease suggests that the alkali volatilization mainly proceeded on the sample’s surface during the post-annealing process leaving oxygen vacancies that were generated due to the volatilization of sodium and potassium as Na2O and K2O, respectively. The results of ICP-OES and EDS point analysis showed that the alkali volatilization proceeded from the sample’s surface and did not proceed to the deep interior of the sample, as other literature reported.

3.2 Electrical and mechanical properties

Changes in the microstructure were apparent, and thus alternations in the electrical and mechanical properties are to be expected. Figure 5 shows P–E and S–E hysteresis loops of post-annealed LNKN6-a piezoceramics. With increasing the annealing time from 0 to 8 h, the remanent polarization 2Pp decreased from 48.5 to 35.6 µC/cm², and the coercive field 2Ec increased from 3.6 to 3.8 kV/mm, respectively [Figs. 5(a) and 5(b)]. In addition to these electrical hardening behaviors, note that there were two switching currents in the annealed samples. One peak was seen at 1.4 kV/mm in as-sintered and annealed samples, while the other peak was seen over 3.5 kV/mm in the annealed samples but not seen in the as-sintered sample. Bipolar S–E loops of post-annealed LNKN6-a piezoceramics were
shown in Fig. 5(c). The butterfly curve of the as-sintered sample was relatively sharp compared to the annealed samples which curves were more like sprout shape seen in relaxor materials.3) To characterize this shape differences, two parameters were characterized as the peak to peak strain $S_{pp}$ and the crossing field at zero strain $E_{x0}$. The electromechanical hardening behavior was obtained in the decrease of $S_{pp}$ from 0.260 to 0.211 % with increasing the annealing time from 0 to 8 h, and $E_{x0}$ increased from 2.38 kV/mm in the as-sintered sample to 2.83 kV/mm in 8 h annealed sample with changing the shape from butterfly to sprout.

Why did the polarization reversal current peak separated, and $S-E$ loops changed from butterfly to sprout-like shape in LNKN6-a piezoceramics? There are three possibilities for these hardening behaviors. First, the effect of the microstructural change in the post-annealed LNKN6-a piezoceramics. The average grain size of as-sintered LNKN6-a piezoceramics was optimized to 3 $\mu$m16) according to the report that the piezoelectric $d_{33}$ constant of LNKN piezoceramics showed the maximum with the grain size of 3 $\mu$m.26),27) While it has been reported that grains larger than this optimum size result in a smaller coercive field $E_{C}$,26),28) the opposite was true in post-annealed samples; $E_{C}$ increased with larger grains. Therefore, the microstructural effect on the hardening behavior was considered as a minor impact in this case. Secondly, the sintering additive of MnCO$_3$ could perform as an acceptor substituting B-site Nb$^{5+}$ for Mn$^{2+}$ and then performed as an acceptor ion Mn$^{2+}$ in Nb$^{5+}$. If it was formed, dimetric $\langle V_0/C_1/C_1, C_0/C_1/O \rangle$ or trimetric $\langle V_0/C_1/C_1, O/C_0/V_0 \rangle$ defect association, that could have performed as hardener. However, scanning transmission electron microscopy (STEM) analysis result showed that MnO segregated on the grain boundary and hardly reacted with LNKN at 1000 °C.16) Therefore, this also not to be considered as dominant factor in hardening behavior.

The last and most promising theory is the change of the phase ratio around the PPT region caused by the alkali volatilization. Figure 6 depicted the temperature dependent permittivity change of the post-annealed LNKN6-a piezoceramics. According to the linear extrapolations in Fig. 6(b), $T_{PPT}$ of the as-sintered sample was 49.4 °C. This material was designed to have $T_{PPT}$ around 50 °C;9),16),17) however, the temperature decreased to 36.6 °C with the inappropriate thermal treatment. Thus, the phase-coexistent region approached room temperature and seemed to result in increasing the tetragonal phase ratio. Regarding to the phase-coexistence, Zhao et al.29) reported the $E_C$ of Er$^{3+}$-doped Li$_x$(Na$_{0.48}$K$_{0.52}$)$_{1-x}$NbO$_3$ piezoceramics in the vicinity of the
phase-coexistent composition: the $E_C$ of orthorhombic phase was 1.43 kV/mm ($x = 0$), while that of tetragonal phase was 2.12 kV/mm ($x = 0.06$). In particular, the $E_C$ of phase coexistent composition ($x = 0.06$) showed the highest value at 2.35 kV/mm. It is suggested that the increase in the $E_C$ and the split of the polarization reversal current seen in the post-annealed LNKN6-a piezoceramics was due to such a phase coexistence region.

Another evidence of the compositional fluctuation on the annealed LNKN6-a was observed in the permittivity change. The dielectric anomalies in the heating process from tetragonal to cubic phase were shown in Fig. 6(c). The temperature at the maximum permittivity from tetragonal to cubic phase were shown in Fig. 6(c). In the heating process, the annealed LNKN6-a was observed in the permittivity due to such a phase coexistence region.

The bipolar $P-E$ loops were characterized parallel to the poling direction shifted to the left side, while those characterized as antiparallel to the poling direction were shifted toward the opposite side. These shifts from the zero point were defined as internal bias field $E_{bias} = (1/2)(E_C^+ - |E_C^-|)$. Where $E_C^+$ and $E_C^-$ were the coercive fields on the positive and negative sides of the $P-E$ loops, respectively. With increasing the annealing time from 0 to 8 h, the value of $E_{bias}$ increased from $-0.73$ to $-0.91$ kV/mm in parallel direction and increased from 0.53 to 0.71 kV/mm in antiparallel direction, respectively. This anisotropic change of the $E_{bias}$ is due to the existence of defect associations between alkali deficiencies $V_M^-$ ($M = Li$, Na, K) and oxygen vacancies $V_O$. With increasing the post-annealing time, the amount of the associated defects of $V_M^-$ and $V_O$ could be increased, which contributed to impede transformation of domain structure. This hardening effect could be smaller than the case of B-site substitution because the associated defect of $V_M^-$ and $V_O$ were weakly bound. The increase of $E_{bias}$ in the annealed samples is considered to be due to the increase of the A-site defect associations based on the switchability of the $E_{bias}$.

**Figure 7** shows stress-strain hysteresis loops of as-sintered LNKN6-a piezoceramics in a solid line. The inset shows a schematic depiction of domain structures at labeled positions. The sequence symbols from (I) to (IV) represent the different domain states. The material started with a randomly oriented domain structure for an as-sintered sample (I). While compressing, the strain increased linearly at first. Further application of compressive stress changed the strain response to nonlinear behavior. This nonlinearity continued increasing with further
compression until all ferroelastic domains available for reorientation had been ferroelastically switched to in-plane direction. The inflection point during the loading process (II) was defined as coercive stress $\sigma_C$, which was analogous to the poling field $E_{pol}$ in $P-E$ hysteresis loops.\(^{37,38}\) On the point of $\sigma_C$, most domains were being switched during the application of stress. At the maximum stress level (III), no additional domains were reoriented, and the material response in the vicinity of the point was again linear elastic. Upon unloading, the initially linear elastic material towards ideal strain $S_i$ began to display non-linearity with decreasing compressive stress. This non-linearity in the unloading process was due to the release of constraint forces toward the in-plane direction and then tried to switch back to the initial state (I). Nevertheless, the sample did not reach its initial state (IV $\neq I$) because not all of the domains switch back to the initial state and resulted in a remanent strain $S_r$. Additionally, the second stress-strain loop was also shown in Fig. 8(a) in a dotted line for a reference. The second loop trajectory in the loading process was different from the first stress-strain loop (solid line) because most of the switchable domain had already mechanically poled to in-plane direction during the first loop. In contrast, the unloading process of the second stress-strain loop was matched to the first loop, which means that the ferroelastic domain had sufficiently oriented in the first loop, and then no further orientation had occurred in the second loop.

Figures 8(b) and 8(c) show the annealing time dependent stress-strain loops of LNKN6-a piezoceramics and the coercive stress $\sigma_C$ and domain backswitching $\eta_B$. The coercive stress $\sigma_C$ increased from $-123.1$ to $-139.5$ MPa and the domain backswitching $\eta_B$ increased from 48.8 to 57.6 $\%$, respectively. Although there seemed to be some hardening in the average values, the stress-strain hysteresis loops did not show any significant changes in these ferroelastic properties. This difference in susceptibility is most likely due to the difference in the volume ratio of the samples used in the ferroelectric and ferroelastic measurements. It seems that the cylindrical shape used for measuring ferroelectric properties was less affected by alkali volatilization, which occurs only on the surface, than the disk shape used for measuring ferroelectric properties.

4. Conclusion

In this study, the alkali volatilization effect on ferroelectric and ferroelastic properties of LNKN6-a piezoceramics was investigated. Alkali volatilization was introduced exposing to the sintering temperature for up to 8 h, which was inspected with chemical analyses of SEM-EDS and ICP-OES. The annealed samples showed a deterioration of
remnant polarization $2P_r$ and enhancement of coercive field $E_c$ and internal bias field $E_{bias}$, which suggested that the annealed sample showed an electrical hardening effect. The switchability of the polarity of $E_{bias}$ according to the poling direction suggested that there were some kinds of associated defects. On the other hand, stress-strain hysteresis revealed that coercive stress $\sigma_C$ and domain backswitching $\eta_{sw}$ were not changed so much, which suggested that mechanical properties did not show hardening effects. In light of the above, we concluded that alkali volatilization affected ferroelectric properties but not a significant effect on ferroelastic properties because the alkali volatilization proceeded from the surface of the samples.

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