[101]-Oriented (Li,Na,K) NbO₃ ceramics prepared by magnetic field-assisted forming, sintering, and electric poling

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Crystal orientation is a strategy for the improvement of piezoelectric properties of lead-free piezoelectric materials. [101]-Oriented Li-modified (Na,K) NbO₃ ceramics (LNKN) were fabricated using a colloidal method under a strong magnetic field, sintering, and subsequent electrical poling. For the Li₀.₀₆Na₀.₅₂K₀.₄₂NbO₃ powder compact, which has a monoclinic phase at room temperature, the (-101) planes were oriented perpendicular to the magnetic field. Although the monoclinic phase transitioned to tetragonal and cubic phases above the Curie temperature during sintering, the crystal-oriented structure was maintained and developed even after phase transitions and grain growth during sintering. Furthermore, crystal reorientation was enhanced by domain switching under an electric field. Thus, [101]-oriented LNKN ceramics with a tetragonal crystal system were obtained. The piezoelectric d₃₃ constant was improved to 208 pC/N from 112 pC/N due to the effects of electromechanical coupling coefficient and specific dielectric coefficient by the texturing process with magnetic field assisted shaping, sintering, and electrical poling.

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1. Introduction

Alkaline niobate-based perovskite ceramics have been investigated as candidate materials for lead-free piezoelectric ceramics.¹–¹⁷ The materials have a perovskite structure, A²⁺B⁵⁺O₃, where the A-site cations are Li⁺, Na⁺, and K⁺ and the B-site cations are Nb⁵⁺, Ta⁵⁺, and Sb⁵⁺. Among these, (Li,Na,K) NbO₃ (LNKN) ceramics have received significant attention owing to their high performance and high Curie temperature.⁶–¹² Guo et al. first demonstrated that Li₀.₀₆Na₀.₅₂K₀.₄₂NbO₃ ceramics in which the A-site is substituted by Li⁺ exhibit an excellent piezoelectric d₃₃ constant of ~240 pC/N.⁹ This enhanced value resulted from the composition of the polymorphic phase boundary between the orthorhombic and tetragonal phases. Saito et al. first reported a piezoelectric d₃₃ constant of 300 pC/N for (K₀.₄₄Na₀.₅₂Li₀.₄₄)(Nb₀.₈₆Ta₀.₁₄-Sb₀.₄₄)O₃ ceramics due to the substitution of Ta and Sb ions and polymorphic phase boundary.⁷ Furthermore, they enhanced the piezoelectric d₃₃ constant of 416 pC/N by crystal orientation. This value was comparable to that of Pb(Zr,Ti)O₃-based piezoelectric ceramics. However, this material contained toxic elements, and the crystal-oriented microstructure was fabricated by reaction template growth, which was a complicated process involving the synthesis of anisotropic template particles using alkali molten salt, orientation of the particles, and topochemical reaction of the templated particles during sintering.

To fabricate the crystal-oriented microstructures, colloidal forming technique under a magnetic field is a convenient method that can orient equiaxial particles without contact.¹⁸–²⁰ Using this method, a weak magnetic material can be oriented by a strong magnetic field. Previously, this method had been applied to various lead-free piezoelectric material systems such as bismuth-titanate-layered oxide ceramics with an aurivillius structure,²¹–²³ and strontium calcium niobate with a tetragonal tungsten-bronze structure.²⁴–²⁵ Recently, this method was applied to sodium niobate-barium titanate with a perovskite structure.²⁶,²⁷ For these ceramics, crystal-oriented microstructures were developed not only by particle orientation and sintering but also by electrical poling. Although the c-axis of sodium niobate-barium titanate particles could be oriented...
to the magnetic field during forming, the c-axis orientation changed to both a- and c-axis orientations above the Curie temperature during sintering. The c-axis orientation was thereafter restored by electrical poling.

The objective of this study was to fabricate crystal-oriented LNKN ceramics using a colloidal method under a strong magnetic field and to evaluate their piezoelectric properties. The following points must be considered for the fabrication of crystal-oriented LNKN systems. First, the oriented crystal directions of the LNKN particles under a magnetic field must be examined, which should depend on the crystal phase of the raw particles. Second, the crystal-oriented microstructure must be maintained even after phase transitions during sintering. Finally, domain reorientation by poling treatment under an electric field should be considered.

In this study, \( \text{Li}_{0.06}\text{Na}_{0.52}\text{K}_{0.42}\text{NbO}_3 \) particles with a monoclinic phase were used to examine their magnetic field orientation. This material shows multiple piezoelectric properties along the [001], [100], and [101] directions, and the monoclinic phase undergoes a transition during sintering to a cubic phase via a tetragonal phase.\(^{13}\) To enhance piezoelectric properties, it is important that the [001], [100], or [101] crystal-oriented structure is achieved via colloidal forming under a magnetic field and its structure must be maintained by phase transition during sintering and by electrical poling.

### 2. Experimental procedure

\( \text{Li}_{0.06}\text{Na}_{0.52}\text{K}_{0.42}\text{NbO}_3 \) with a monoclinic phase system was synthesized by a solid-state reaction method.\(^{14}\) Reagent-grade \( \text{Li}_2\text{CO}_3, \text{Na}_2\text{CO}_3, \text{K}_2\text{CO}_3, \) and \( \text{Nb}_2\text{O}_5 \) (>99.5% purity) were weighed in accordance with the formula of \( \text{Li}_{0.06}\text{Na}_{0.52}\text{K}_{0.42}\text{NbO}_3 \) and mixed by ball milling for 24 h. After drying, the entire mixture was calcined at 900°C for 3 h, and the calcined powder was crushed by ball milling for 24 h using ethanol.\(^{14}\) The crystal orientation of the powder was confirmed by X-ray diffraction (XRD) patterns (Ultima IV, Cu-K\(\alpha \), Rigaku Co. Ltd., Tokyo Japan) with Cu-K\(\alpha \) radiation. The specific surface area of the LNKN powder was measured with a Brunauer–Emmett–Teller specific surface meter (Flowsorb2010, Micromeritics Instrument Co., Norcross, GA, USA) using nitrogen as an adsorbing gas. The powder was dispersed into isopropyl alcohol with polyethylene imine (MW 10000, Nacalai Tesque, Kyoto, Japan) as a dispersant, where the solid content in the slurry was 35 vol %.

The slurry was poured into a cylindrical container that was 25 mm in diameter and 20 mm in depth for the preparation of a green compact. To form a green compact in a magnetic field, the container was placed in the static vertical magnetic field (10 T) of a superconducting magnet (TM10VH10, Toshiba, Tokyo, Japan) at room temperature until the slurry was fully dried. Another portion of the slurry was dried in the absence of a magnetic field to prepare a reference sample. The green compacts were subjected to cold isostatic pressing at 200 MPa for 2 min, and were then heat-treated for binder burnout in air for 1 h at 450°C. The samples were then sintered in a magnesia container at 1050°C for 1 h under an oxygen atmosphere.

The crystal orientations of the powder compact and sintered ceramics were characterized by XRD over the 2\(\theta \) range of 20–70°, and the relative densities of the samples were measured by the Archimedes’ method. The sample microstructures were observed with scanning electron microscopy (SEM, JEOL JSM5310LV, Tokyo, Japan). For SEM sample preparation, the polished samples were thermally etched at 900°C for 20 min in air.

The AC electric field was in the range of −3.0 to 3.0 kV/mm. The piezoelectric \( d_{33} \) constants were measured with a quasi-static piezo \( d_{33} \) meter (ZJ-6B, Institute of Acoustics, Chinese Academy of Sciences). Prior to the measurement, the polarizations of the samples were conducted in silicone oil at 25°C. The AC electric field was in the range of −3.0 to 3.0 kV/mm. The piezoelectric \( d_{33} \) constants were measured with a quasi-static piezo \( d_{33} \) meter (ZJ-6B, Institute of Acoustics, Chinese Academy of Sciences).

The electromechanical coupling coefficients \( k_{33} \), elastic compliance constants, \( s_{ij}^D \) and \( s_{ij}^E \), and piezoelectric \( d_{33} \) constants of samples were evaluated based on the ANSI/IEEE 176-1987 standards\(^{23}\) via the resonance-antiresonance method using an impedance analyzer (4294A, Agilent Technologies). Mechanical coupling coefficient \( k_{33} \) was calculated by following equation:

\[
(k_{33})^2 = \frac{\pi f_s}{2 f_p} \tan \frac{\pi f_p - f_s}{2 f_p}
\]

where, \( f_s \) and \( f_p \) are resonance frequency and antiresonance frequency, respectively. The sample was a square plate...
with a side length of 0.5 mm and thickness of 1.5 mm. Gold electrodes were sputtered onto both surfaces of the samples. The measured frequencies were in the range of 100 Hz to 1 MHz, and the relative permittivity $\varepsilon_{33}/\varepsilon_0$ was measured at 1 kHz and 25°C.

3. Results and discussion

Figure 2 shows the SEM images of Li$_{0.06}$Na$_{0.52}$K$_{0.42}$NbO$_3$ powder particles. The average particle diameter is approximately 0.5 μm, and the particles have a cubic-like shape with a specific surface area of 4.9 m$^2$/g. This size is appropriate for orientation in strong magnetic field because the particles are well-dispersed in the slurry and are oriented by sufficiently high magnetic torques. Figure 3 shows the XRD pattern of the powder used in this study. The crystal phase is confirmed to be monoclinic by comparison with International Centre for Diffraction Data (ICDD)-No.01-082-327.

Figure 4 shows the XRD patterns of the polished horizontal planes of the powder compacts. Figures 4(a) and 4(b) show the XRD patterns of the samples formed with and without the magnetic field, respectively, and the expanded XRD patterns in the ranges of 21–24° and 31–33° are shown in Figs. 4(c) and 4(d), respectively. For the compact powder subjected to the magnetic field, the peaks originating from the (-101), (101), (110), and (011) planes at 28–32° are intense, particularly the (-101) plane peak, as shown in Fig. 4(d). Here, let us consider a (-101) plane orientation of particles with a monoclinic crystal system in the magnetic field. For uniaxial crystal systems such as tetragonal and hexagonal, either the $a$- or $c$-axis is preferentially oriented to the magnetic field by the anisotropic magnetic susceptibility. For example, as the diamagnetic susceptibility of the $a$-axis of alumina is strong, the $c$-axis of a single crystalline particle orients parallel to the magnetic field. Contrarily, for a monoclinic crystal system, the symmetry must be considered to understand its oriented direction. Figure 5 shows the model of the monoclinic crystal system reported by Hatano et al. [13] in which the mirror plane is in the (-101) plane. From the viewpoint of magnetic anisotropy, either the (-101) or (101) plane perpendicular to the [-101] direction can align to the magnetic field. As a result, the (-101) plane aligns perpendicular to the magnetic field.

![Fig. 2. Particle morphology of LNKN powder.](image)

![Fig. 3. (a) XRD pattern of LNKN raw powder. ICDD patterns of (b) monoclinic (No.01-082-3273), and (c) tetragonal (No.01-082-3272) phases.](image)

![Fig. 4. XRD patterns of the polished horizontal planes for powder compact. (a) Sample formed in the magnetic field and (b) outside the magnetic field. Expanded XRD patterns in the ranges of (c) 21–24° and (d) 31–33°. Red line corresponds to forming in the magnetic field and blue line represents forming at normal conditions.](image)

![Fig. 5. Crystal lattice model with monoclinic crystal systems (a = 3.9825 Å, b = 3.9438 Å, c = 4.0217 Å, $\alpha = \gamma = 90^\circ$, $\beta = 90.29^\circ$), in which the mirror phase is in the (-101) plane.](image)
that is, the [101] direction has the strongest diamagnetic susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable direction for the piezoelectric property because of its susceptibility. The [-101] orientation is the preferable orientation of the sintered sample prepared with the magnetic field. As a result, the degree of orientation is determined to be 0.38.

**Figure 6** shows the XRD patterns of the polished horizontal planes for the sintered ceramics of powder compact formed with magnetic field to the (-101) and (101) planes is calculated by a simplified equation:

$$ F = \frac{P - P_0}{1 - P_0}, $$

where $P_0 = \Sigma[I_0(hk0) + I_0(101)]/\Sigma I(hk0)$, $P = \Sigma[I(hk0) + I(101)]/\Sigma I(hk0)$, and $I$ and $I_0$ are the intensities of the XRD diffraction peaks in the $2\theta$ range of 20–45° for the samples prepared with and without the magnetic field, respectively. As a result, the degree of orientation is determined to be 0.38.

**Figure 7** shows the SEM microstructures of the ceramic samples formed with and without the magnetic field and sintered at 1050°C. The relative density of both samples (oriented and non-oriented) is 93%. After sintering, the grains are ~15 μm in size and have an equiaxed shape. As indicated by Figs. 4 and 6, it is possible that the grains oriented to the magnetic field preferentially grow by incorporating other grains during sintering.

**Figure 8** exhibits the $P-E$ hysteresis loops of the oriented and non-oriented LNKN ceramics with an electric field of 3.0 kV/mm.
The samples can barely withstand the application of a large electric field. The results indicate that the polarization and inversion of domain structures is facilitated by the oriented microstructures. Herein, the samples can barely withstand the application of a large alternating electric field of 3.0 kV/mm by sintering under an oxygen atmosphere. When an electric field greater than 4.0 kV/mm is applied, the samples break down electrically and show the semi-conductivity of an unstable alkali metal element. At high temperatures, the volatilization or oxidation of alkali metals likely occurs, and these are easily lost. In addition, this material is polymorphic at room temperature. Therefore, the defects occur at the A site and oxygen.

Figure 9 shows the XRD patterns of the sintered ceramic samples after poling. For poling, sintered ceramic samples in silicone oil at 25°C were poled for 30 min under a direct electric field of 3.0 kV/mm. Direct electric field was applied normal to the plate samples; for the sample prepared with the magnetic field, the poling directions were the [101]- and [110]-oriented directions. In the XRD pattern of the oriented sintered ceramic sample, the (101) plane peak increases in intensity after poling and the (110) plane peak decreases after poling. This indicates that poling treatment leads to reorientation, that is, the electric field induces a 90° domain switch from the [110] domain to the [101] domain. As a result, for the sample formed in the magnetic field, [101]-oriented LNKN ceramics were obtained by forming in a magnetic field, sintering, and poling treatment in a direct electric field. Contrarily, the (001) and (101) peaks increase in intensity by the application of direct electric field to the sample formed without a magnetic field, and domain switching also occurs. Here, the degree of orientation of [101]-oriented and poled LNKN ceramics is also estimated for the (101) planes by using Eq. (2) with $P_0 = [I_0(101) + I_0(202)]/\Sigma I_0(hkl)$, $P = [(I(101) + I(202))/(\Sigma I(hkl)]$, and $I$ and $I_0$ values in the 2θ range of 20–70°. For the [101]-oriented samples, the degree of orientation after poling is 0.31. The calculated degree of orientation is not very high because other peaks such as (001), (102), and (112) are also enhanced slightly by poling. In this way, for the sample formed in the magnetic field, [101]-oriented LNKN ceramics were obtained by forming in a magnetic field, sintering, and poling treatment in a direct electric field.

Table 1 shows the electrical properties of the samples measured by the impedance resonance-antiresonance method and the quasi static $d_{33}$ meter. Here, the measured parameters include the dielectric constant $\varepsilon_{33}$, elastic compliance $s_{33}$, relative dielectric constant $\varepsilon_{33}/\varepsilon_0$, and piezoelectric $d_{33}$ constant. Each parameter increases with an increase in [101] orientation. The piezoelectric $d_{33}$ constant of the [101]-oriented LNKN sample prepared in the magnetic field is 208 pC/N, which is higher than the value of 112 pC/N for the sample prepared without the magnetic field. The enhancement of the electro-mechanical coupling coefficient $k_{33}$ and relative dielectric constant $\varepsilon_{33}/\varepsilon_0$ by the [101]-crystal orientation contributes to an improvement in the piezoelectric $d_{33}$ constant. The piezoelectric $d_{33}$ constant of the [101]-oriented LNKN ceramic prepared in a magnetic field is also determined as 193 pC/N by the quasi static $d_{33}$ meter, whereas it is 109 pC/N for the sample prepared without the magnetic field. The $d_{33}$ values measured by the resonance-antiresonance method were confirmed by the results obtained by the quasi static $d_{33}$ meters.

4. Conclusion

The aim of this study was to fabricate particle-oriented LNKN ceramics as lead free piezoelectric materials via the colloidal method under a strong magnetic field and subsequent sintering and poling treatment. Herein, the orientation of the crystal phase of Li$_{0.06}$Na$_{0.52}$K$_{0.42}$NbO$_3$ ceramics with a monoclinic phase at room temperature was examined. The [-101] and [101] directions of the monoclinic-phase particles were oriented parallel to the magnetic field, and their crystal symmetry affected the magnetic orientation. The crystal-oriented structure was maintained and a [101]-oriented structure in the tetragonal phase developed even after phase transitions and grain growth during sintering. Furthermore, the [101] orientation was enhanced by electric field poling. The piezoelectric $d_{33}$ constant was improved to 208 pC/N from 112 pC/N by the crystal orientation.

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Table 1. The electrical properties of LNKN measured by the impedance resonance antiresonance method and quasi-static piezo $d_{33}$ meter

|  | $k_{33}$ (%) | $s_{33}^\prime$ ($10^{-12}$ m$^2$/N) | $s_{33}^\prime$ ($10^{-12}$ m$^2$/N) | $\varepsilon_{33}/\varepsilon_0$ | $d_{33}$(pC/N) | $d_{33}$(pC/N) |
|---|---|---|---|---|---|---|
| random | 41.8 | 11.4 | 13.9 | 583 | 112 | 109 |
| 101 oriented | 47.0 | 11.0 | 14.2 | 1567 | 208 | 193 |

![Figure 9](image-url)
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