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Letter

Enhanced piezoelectric properties of Ta substituted-$(K_{0.5}Na_{0.5})NbO_3$ films: A candidate for lead-free piezoelectric thin films

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ABSTRACT

Piezoelectric $(K_{0.5}Na_{0.5})NbO_3$ (KNN) and $(K_{0.5}Na_{0.5})(Nb_{0.7}Ta_{0.3})O_3$ (KNNT) thin films were prepared via chemical solution deposition. An analysis of X-ray diffraction revealed that Ta^{5+} diffuses into the KNN to form a single perovskite structure. Compared to KNN films, KNNT films exhibited a low leakage current density due to their fine-grain nonporous structures. The partial substitution of Ta^{5+} for the B-site ion Nb^{5+} in the KNNT films decreased the Curie temperature (T_C). This in turn led to the existence of a polymorphic phase transition near room temperature and further improvement in the piezoelectric properties. Lead-free KNNT films exhibited a well-saturated piezoelectric hysteresis loop with a effective piezoelectric coefficient ($d_{33,eff}$) value of 61 pm/V, comparable to that of PZT thin films.

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1. Introduction

$Pb(Zr,Ti)O_3$-based electroceramics have been widely studied for use in various sensors and memory applications, such as microelectromechanical systems, transducers, and piezoelectric transformers, due to their desirable ferroelectric and piezoelectric behaviors [1]. However, environmental considerations dictate that lead-free thin films be used in the new generation of functional devices. For this reason, considerable attention has been focused on $(K_{0.5}Na_{0.5})NbO_3$ (KNN)-based electroceramic materials. While these materials exhibit a relatively high Curie temperature, their electrical behavior is still not as satisfactory as those of some of their lead-containing counterparts [2,3]. Recently, a large number of studies were carried out in order to improve the piezoelectric properties of KNN ceramics through the partial substitution of the A and/or B-site atoms, such as in $(K,Na,Li)NbO_3$ [4], $(K,Na)(Nb,Ta)O_3$ [5,6], $(K,Na,Li)(Nb,Ta)O_3$ [7], and $(K,Na,Li)(Nb,Ta,Sb)O_3$ [8] ceramics.

Lead-free ferroelectric and piezoelectric materials have primarily been studied in their bulk ceramic forms due to the difficulty of forming thin films with these materials. Lead-free KNN thin films with well-saturated polarization-electric field (*P*-*E*) hysteresis loops have been deposited using the chemical solution deposition (CSD) method. However, their piezoelectric properties are generally inferior to those of bulk ceramics because of the difficulty in obtaining an ideal thin film [9]. Recently, Wang et al. [10] demonstrated that Mn-doped KNN films (1.3 μm in thickness) exhibited a low leakage current density and a well-saturated *P*-*E* hysteresis loop with a remanent polarization (P_r) of ~7.2 μC/cm^2; the piezoelectric properties were not reported. Abazari et al. [11] demonstrated that Mn-doped $(K,Na,Li)(Nb,Ta,Sb)O_3$ thin film exhibited a effective piezoelectric coefficient, $d_{33,eff}$ of ~53 pm/V. Also, Mn-doped $(K,Na)(Nb,Ta)O_3$ thin film was reported improved electrical and piezoelectric properties with $d_{33,eff}$ of ~58 pm/V [12]. In this work, Ta-modified $(K_{0.5}Na_{0.5})(Nb_{0.7}Ta_{0.3})O_3$ thin films were deposited onto $Pt/TiO_2/SiO_2/Si$ substrates. Such structures have been shown to exhibit low leakage current behavior and piezoelectric properties that are superior to those of KNN thin films. Ta-modified $(K_{0.5}Na_{0.5})(Nb_{0.7}Ta_{0.3})O_3$ thin films also display rather variable transition temperatures, such as in the orthorhombic to tetragonal transition, and Curie temperatures. In this work, a possible explanation as to why Ta substitution leads to enhanced piezoelectric properties in KNN films is suggested.

2. Experimental procedure

For the preparation of the KNNT thin films, sodium acetate (CH_3COONa), potassium acetate (CH_3COOK), niobium pentaethoxide ($Nb(OCH_2CH_3)_5$), and tantalum pentaethoxide ($Ta(OCH_2CH_3)_5$) were used as the starting chemicals, and 2-methoxyethanol ($CH_3OCH_2CH_2OH$) and acetylacetone ($CH_3COCH_2COCH_3$) were employed as the starting solvent and chelating agent, respectively. To compensate

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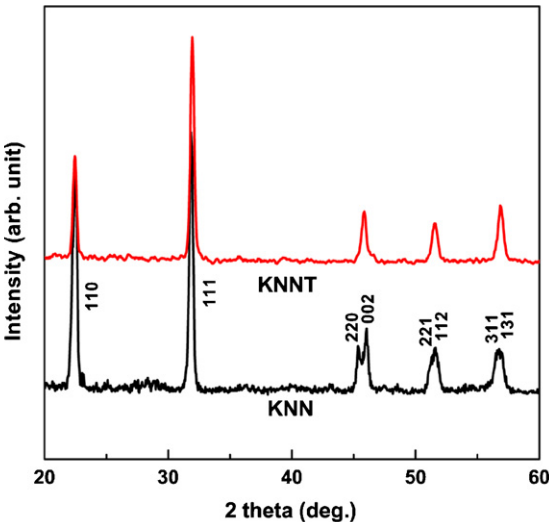

Fig. 1. XRD patterns of the KNN and KNNT thin films.

for the loss of alkaline metals, excesses of 10 mol% of K and Na were added to the precursor solutions. To enhance densification, polyvinylpyrrolidone ($(C_6H_9NO)_n$) was added to the KNNT precursors, and the concentrations of the final solutions were adjusted to 0.2 mol/L. To obtain thin films, the precursor solutions were spin-coated onto Pt(111)(150 nm)/TiO_2(20 nm)/SiO_2(300 nm)/Si(100)(525 μm) substrates at a rate of 3000 rpm for 30 s. The wet films were dried at 200 °C for 5 min on a hot plate. The dried gel films were calcined at 400 °C for 5 min and then heated at 550 °C for 5 min in an oxygen atmosphere using a tube furnace by direct insertion. The aforementioned steps were repeated several times in order to obtain thin films with the desired thickness. These films were then annealed at 700 °C for 30 min in an oxygen ambient. KNNT thin films with thicknesses of 500 nm were ultimately obtained.

The phase and structure of the fabricated thin films were identified through X-ray diffraction (XRD; Philips XPERT PW1710) scans obtained with a thin film diffractometer operated at 40 kV and 30 mA with Cu Kα radiation ($\lambda = 1.54056$ Å). The XRD data were attained at a scanning speed of 0.05°/s in increments of 0.05°. The compositions of the films, as determined with an electron probe micro analyzer (EPMA; JEOL JXA-8900R), were $(K_{0.50}Na_{0.50})NbO_3$ and $(K_{0.50}Na_{0.50})(Nb_{0.69}Ta_{0.31})O_3$ and were respectively denoted as KNN and KNNT. The morphologies of the films were confirmed via scanning electron microscopy (SEM; Hitachi S-4200) and atomic force microscopy (AFM; Digital Instrument Multimode). For electrical contacts, Pt top electrode 2.5×10^{-4} cm^2 in area were deposited on the film through a metal shadow mask by dc sputtering. The *P-E* hysteresis and leakage current density were measured with a ferroelectric tester (Radiant Technologies RT66a Radiant) and an electrometer (Keithley 237), respectively, and the dielectric constants of the films were measured as a function of temperature using an impedance analyzer (HIOKI 3522-50 LCR). The piezoelectric responses of the films were examined through piezoresponse force microscopy (PFM; Seiko Instruments SPH-300HV) with a lock-in amplifier (Stanford Research SR830), which was used to measure the first harmonic of a cantilever defection signal. Pt-coated Si cantilever tips (NT-MDT NSG01-PT) with an estimated spring constant of 5.5 N/m and a tip diameter of approximately 30 nm were used for the measurements. For the calibration of the piezoelectric response signal, we used an *x*-cut quartz single crystal for which the relevant piezoelectric constant, d_{11}, was known to be 2.3 pm/V.

3. Results and discussion

Fig. 1 shows the XRD patterns of the KNN and KNNT thin films in the 2θ range of 20–60°. In agreement with the previously reported studies [5,6], KNN thin film without the substitution of Ta possess a single phase perovskite structure with orthorhombic symmetry, as evidenced by the splitting of (2 2 0) and (0 0 2) at a 2θ of around 46°. However, it can be seen from Fig. 1, when 30 mol% Ta was substituted for Nb in the $(K_{0.5}Na_{0.5})(Nb_{0.7}Ta_{0.3})O_3$ thin film, the split (2 2 0) and (0 0 2) peaks of orthorhombic phase merged into a single peak, suggesting that the crystal structure of the KNNT thin film evolves from the orthorhombic to a pseudo-cubic symmetry [5,13].

Fig. 2(a) and (b) shows AFM micrographs of the KNN and KNNT thin films, and cross-sectional SEM images of the KNN and KNNT films are shown in Fig. 2(c) and (d), respectively. In the cross-sectional image of the KNN film, many pores are shown to exist

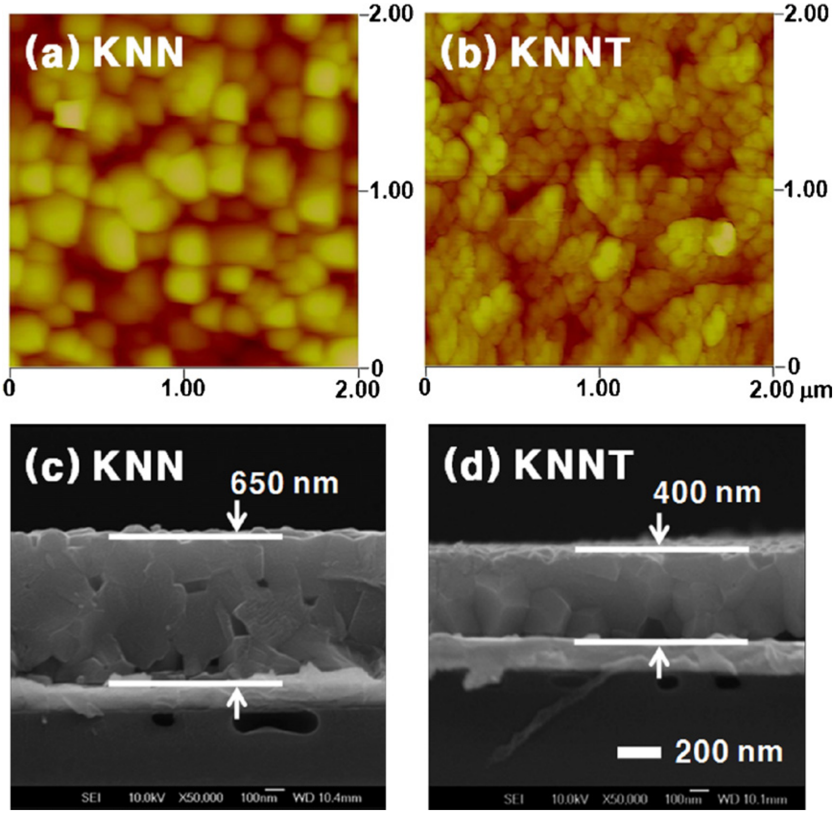

Fig. 2. (a) and (b) AFM micrographs and (c) and (d) cross-sectional SEM images of the KNN and KNNT thin films.

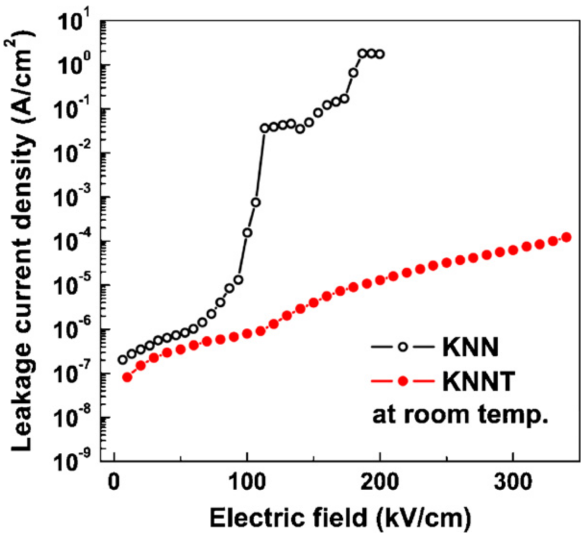

Fig. 3. The leakage current density–electric field (*J*–*E*) characteristics of the KNN and KNNT thin films at room temperature.

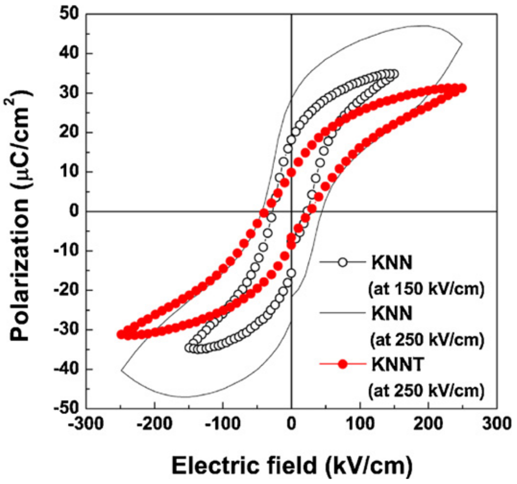

Fig. 4. The ferroelectric *P-E* hysteresis loops of the KNN and KNNT thin films at room temperature.

within the film. In general, such pores significantly influence the electrical properties of a thin film [14]. But, in the case of the KNNT film, the number of pores was relatively decreased. The KNN film includes larger grains. As Ta was substituted for Nb, the grain growth was inhibited and the average grain size was decreased. This led to the development of a homogeneous microstructure [13]. The reduction in the number of pores in KNNT film may be attributed to the inhibition in the grain growth. Also, it is well-known that a fine-grain homogeneous structure will lead to high mechanical strength for a piezoelectric film. With regard to the morphology changes observed in the KNNT film, a similar trend can be seen in the $(K_{0.5}Na_{0.5})(Nb_{1-x}Ta_x)O_3$ bulk ceramic system [5]. Lin et al. [5] reported that the average grain size decreased and the density increased with the substitution of Ta into the KNN ceramic system. Morphology changes in the KNNT ceramics thus lead to an improvement in the piezoelectric properties [15].

Fig. 3 shows the leakage current density (*J*)–electric field (*E*) characteristics of the KNN and KNNT thin films measured at room temperature. The leakage current density of the KNN thin film was about 3.5×10^{-7} A/cm^2 at a low electric field of 20 kV/cm. This value is similar to that reported by Cho et al. [16]. However, the leakage current density of the KNN film rapidly increased from 10^{-5} to 10^{-2} A/cm^2 around 90 kV/cm. In the case of the KNNT film, the leakage current density was almost the same as that of the KNN film at a low electric field up to 70 kV/cm. The leakage current density of the KNN film rapidly increased above an applied DC field of 90 kV/cm, while the KNNT film maintained a low leakage current density below 10^{-6} A/cm^2 up to an applied field of 150 kV/cm. When a high electric field is applied to a dielectric film with many pores, a current path is easily established through the pores [14]. As such, films with many pores generally exhibit a low dielectric strength. The substitution of 30 mol% Ta into the KNN film led to a significant improvement in the leakage current characteristics up to a higher electric field due to a decrease in the number of pores within the film.

The ferroelectric *P-E* hysteresis loops of the KNN and KNNT thin films at room temperature are shown in Fig. 4. The KNNT film yielded a well-saturated *P-E* hysteresis loop with a P_r of 10.5 μC/cm^2. While the KNN film exhibited a leaky-shaped *P-E* hysteresis loop at an electric field of 250 kV/cm. This leaky *P-E* hysteresis loop caused an increase in the leakage current. The *P-E* hysteresis loop of the KNN film is square-shaped at a relatively low electric field of 150 kV/cm. However, for the KNNT film, the *P-E* hysteresis loop became flat and slanted, an indicator of weakening ferroelectric properties. With regard to the slanted loops attained for the KNNT film, a similar tendency can be seen in $(K_{0.5}Na_{0.5})(Nb_{1-x}Ta_x)O_3$ bulk ceramics [5]. Lin et al. [5] reported that a $(K_{0.5}Na_{0.5})(Nb_{1-x}Ta_x)O_3$ ceramic may transform from a normal ferroelectric to a relaxor ferroelectric, exhibiting a so-called slim loop. At sufficiently high electric fields, the nanodomains of a relaxor can be oriented with the field, leading to a large degree of polarization. However, most of these domains re-acquire their random orientations upon removal of the field, resulting in a small remanent polarization (P_r). A small P_r is evidence of the presence of some degree of cooperative freezing of dipolar (or nanodomain) orientations [17].

Fig. 5 shows the temperature dependence of the dielectric constants of the KNN and KNNT thin films measured at 100 kHz. The KNN film exhibited two transition peaks, in agreement with previously reported results for KNN ceramics [5,6]. The peak at $T_C = 324\,°C$ signified a phase transition from a tetragonal to cubic structure. The other peak at $T_{O-T} = 195\,°C$ is due to a phase transition from orthorhombic to tetragonal phase. Thus, it can be stated that the KNN film is orthorhombic at room temperature. After the substitution of Ta^{5+} for Nb^{5+} in the B-site, the KNNT film exhibited only one transition peak at $T_C = 112\,°C$ since the phase transition of T_{O-T} in the KNNT film was shifted to lower than room temperature. This suggests that, the KNNT thin film has a tetragonal symme-

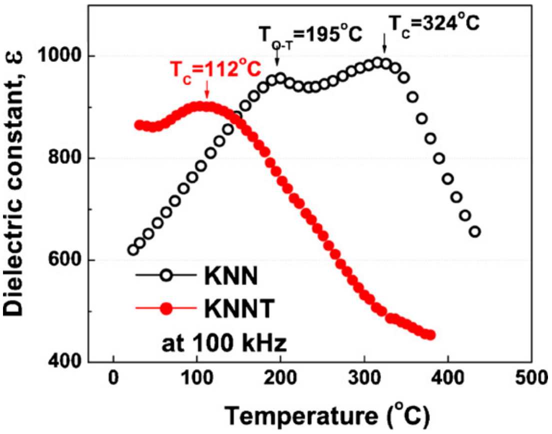

Fig. 5. Temperature dependence of the dielectric constants for KNN and KNNT thin films measured at a frequency of 100 kHz.

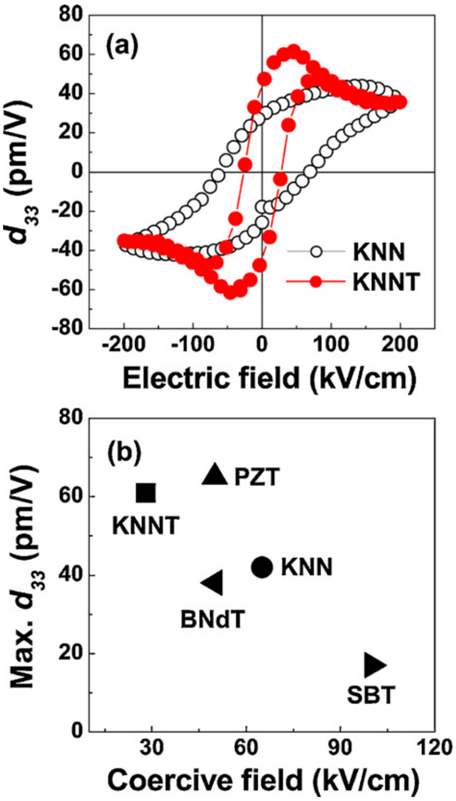

Fig. 6. (a) Longitudinal piezoelectric coefficient ($d_{33,eff}$) of KNN and KNNT films and (b) reported values of $d_{33,eff}$ for various lead-free and lead-based piezoelectric thin films.

try, although their (room temperature) diffraction peaks (2 2 0) and (0 0 2) merge into one (Fig. 1).

Fig. 6(a) shows the piezoelectric behaviors of the KNN and KNNT thin films as measured by the in-field hysteresis method [18]. This method utilizes a probing AC voltage with a peak-to-peak value of 0.7 V and a frequency of 17 kHz superimposed onto an electric field that is varied from zero to E_{max} (200 kV/cm). The electric field is then decreased to E_{min} (−200 kV) and increased again to zero in order to simultaneously measure the piezoelectric coefficient as a function of the applied field. As expected, the characteristic piezoelectric hysteresis loops for the KNN and KNNT films are quite different. The loops are characterized in terms of the remanent and maximum piezoelectric coefficient, as well as the coercive field (E_C). In terms of actuation, the $d_{33,eff}$ parameter is extremely important [19]. The KNN film exhibited a remanent $d_{33,eff}$ value of 28 pm/V, whereas the KNNT film displayed a higher remanent $d_{33,eff}$ value of 45 pm/V. The E_C of the KNNT film was found to be around 28 kV/cm, lower than the 65 kV/cm attained for the KNN film. The piezoelectric behaviors of the KNN and KNNT films are summarized in Table 1.

The KNNT film was found to have a higher $d_{33,eff}$ value compared to that of the KNN film. However, as the applied field increases, the $d_{33,eff}$ value decreases approximately linearly before reaching a "minimum" value. Questions regarding such behavior have been addressed in previous research. According to Kholkin et al. [20], the shapes of piezoelectric hysteresis curves can be explained by the competing influence of the polarization and dielectric constant ε versus the applied field on the piezoelectric coefficient via the linearization electrostriction expression. This expression may be written as $d_{33,eff} = 2Q_{11}P_3\varepsilon_{33}$, where $d_{33,eff}$ is the piezoelectric coefficient, Q_{11} is the longitudinal electrostrictive coefficient, P_3 is the polarization, and ε_{33} is the dielectric permittivity.

Table 1
Piezoelectric characteristics of the KNN and KNNT films deposited onto Pt/TiO$_2$/SiO$_2$/Si substrates.

	Remanent $d_{33,eff}$ (pm/V)	Maximum $d_{33,eff}$ (pm/V)	Average E_C (kV/cm)
KNN	28	42	65
KNNT	45	61	28

Recently, Sucwattana et al. [21] investigated the local structure and harmonic level dynamics of pure and Ta-substituted KNN using first principles calculations. They find that the structural effect of Ta is very short ranged consisting of a suppression of the off-centering on the Ta site, with some impact on the neighboring Nb. Furthermore, they reported that the dynamics of the Nb *B*-site ions is suppressed by Ta addition, and the Ta shows stiffer force constants even though it is closer to the center of its O cage. This connects the lower T_C and resulting higher dielectric constant of the Ta-substituted material with a very local tendency for the Ta to off-center more weakly than Nb in this material. Matsubara et al. [13] reported that the electric field-induced strain of Ta-substituted KNN ceramics was enhanced due to their electrostrictive natures and high dielectric constants (as shown in the expression $d_{33} = 2Q_{11}P_3\varepsilon_{33}$).

In this study, KNN film without the substitution of Ta revealed high T_C and T_{O-T} of 324 °C and 195 °C respectively. After 30 mol% Ta substitution into the KNN film, the T_C was drastically decreased to 112 °C, and T_{O-T} was shifted downward to room temperature. As a result, the dielectric constant was increased from ($\varepsilon_{KNN} \sim 600$) to ($\varepsilon_{KNNT} \sim 880$) and the piezoelectric coefficient $d_{33,eff}$ was significantly enhanced to 61 pm/V. In connection with the reported studies [13,21], this large piezoelectric coefficient in KNNT film can be attributed to the high dielectric constant ($\varepsilon_{KNNT} \sim 880$) at room temperature, and the occurrence of polymorphic phase transition (PPT) below the room temperature.

The $d_{33,eff}$ of the KNN film was about 42 pm/V at the maximum applied field. The maximum $d_{33,eff}$ of a well-saturated piezoelectric hysteresis loop from the KNNT thin film was 61 pm/V. This value of $d_{33,eff}$ in KNNT film may be significantly smaller than the d_{33} of the bulk material (∼110 pC/N) due to the clamping influence of the substrate [5]. It is well known that the so-called intrinsic response (response of an individual domain) in thin films is strongly affected by the misfit between the film and the substrate [22–24].

The 61 pm/V $d_{33,eff}$ value is comparable to those of lead-based counterparts [25,26] and among the highest values for a lead-free thin film. As demonstrated in Fig. 6(b), in comparison with other lead-free thin films materials, the 61 pm/V value is greater than 17 pm/V for $SrBi_2TaO_9$ films [25] and 38 pm/V for $Bi_{3.5}Nd_{0.5}Ti_3O_{12}$ films [26], and 45 pm/V for $K_{0.5}Na_{0.5}NbO_3$ film [9,27]. PZT thin films with submicron thicknesses typically have piezoelectric coefficients of 30–65 pm/V, while epitaxial or highly oriented PZT thin films have piezoelectric coefficients of 40–180 pm/V [18,28–30]. Thus, the piezoelectric coefficient of the KNNT film in this study is comparable to that of PZT thin films.

4. Conclusions

Ferroelectric KNN and KNNT thin films were prepared by chemical solution deposition in order to study the effect of Ta substitution on piezoelectricity. The KNNT films exhibited a low leakage current density due to their fine-grain homogeneous (pore-free) structures. Well-saturated piezoelectric hysteresis loops were attained with the KNNT films due to their low leakage properties. In addition, the $d_{33,eff}$ value of the KNNT film was 61 pm/V, higher than the 42 pm/V obtained with the KNN film. Therefore, the substitution of 30 mol% Ta into a KNN film led to enhanced piezoelectric properties. Two possible reasons may be given for the enhanced piezoelectric properties of the KNNT film compared to that of the KNN film:

1. The piezoelectric hysteresis loop of the KNNT film was well-saturated due to the low leakage current density of the film. This is in contrast to the leaky-shaped loop attained with the KNN film.
2. The KNNT film had a higher $d_{33,eff}$ value compared to that of the KNN film because the substitution of Ta led to reduced the phase transition temperatures (T_C and T_{O-T}). Instead, the dielectric constant was increased near the room temperature.

In this work, a well-saturated piezoelectric hysteresis loop and a $d_{33,eff}$ value of 61 pm/V were attained for a KNNT film. These results suggest that KNNT films may be employed in eco-friendly applications and may also be used as an alternative for PZT due to their good piezoelectric properties.

Acknowledgments

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