1. Introduction

Demand for devices that can control electrical energy is increasing as parts become electronic and miniaturized. A dielectric capacitor, which is one of the devices for storing electrical energy, has a very high-power density along with a very fast charge–discharge rate through changes of the polarization caused by an external electric field. These high-power dielectric capacitors are used in many pulse-discharge and power-controlled electronic devices, such as electric vehicles, medical equipment, energy systems, and military supplies [1–5]. However, as compared with batteries, dielectric capacitors have extremely low energy densities, which limit their applications. To improve these capacitors, the rate of polarization change or the amount of energy stored must be increased.

Because an increase in energy storage capacity is generally accompanied by an increase in the volume or weight of a dielectric, the energy storage density ($W_{rec}$), that is, the amount of energy stored per unit weight or volume, and the charge and discharge efficiency ($\eta$) should be increased to enable the miniaturization of electronic devices. In the dielectric, $W_{rec}$ and $\eta$ are expressed as:

$$W_{rec} = \frac{\int P_{acc} EdP}{P_{oc}}$$

(1)

$$\eta = \frac{W_{rec}}{W_{rec} + W_{loss}} \times 100 \text{ (\%)}$$

(2)

As shown in Equation (1) and (2), the dielectric should have high saturation polarization ($P_{max}$), low remnant polarization ($P_r$), and high dielectric breakdown voltage ($E_D$) to obtain high $W_{rec}$ and $\eta$ values. Bismuth-based Bi$_{10.5}$Na$_{0.5}$TiO$_3$ (BNT) is a perovskite dielectric with a high $P_{max}$ of 43 $\mu$C/cm$^2$ but low $W_{rec}$ and $\eta$ values because of its high $P_r$ (38 $\mu$C/cm$^2$) [2,6]. Several studies have investigated the combinations of BNT and other dielectrics to increase the $W_{rec}$ values. These studies focused on the composition modification to obtain the behavior of relaxor ferroelectrics, which have thin polarization–electric field (P-E) hysteresis loops and a high $W_{rec}$ [7–9].

Sr$_{0.5}$Bi$_{10.2}$TiO$_3$ (SBT) is a relaxor ferroelectric with a perovskite structure [10,11]. Relaxor ferroelectrics have smaller $P_{max}$ values than ferroelectrics but better energy storage properties due to lower $P_r$ values [12,13]. Meanwhile, in addition to the energy storage properties, thermal stability is another important requirement of dielectric capacitors. Because the dielectric constants ($\varepsilon_r$) of capacitors are temperature-dependent and rapidly decrease above the Curie temperature ($T_c$), these capacitors are effective only if they have constant $\varepsilon_r$ values over a wide temperature range below $T_c$ [14]. BNT-SBT has excellent thermal stabilities with constant $\varepsilon_r$ values from 300 to 600 K. Therefore, it was suggested that the combination of BNT-SBT might exhibit improvements in energy storage properties and thermal stabilities simultaneously [15].

Aerosol deposition (AD) is a deposition method that can be used to fabricate high-density thick films at room temperature. The greatest advantage of the AD...
2. Experimental procedure

6BNT-4SBT powder was prepared using Bi₂O₃, SrCO₃, Na₂CO₃, and TiO₂ (99.9% Sigma-Aldrich, USA) through a solid-state reaction. First, four powders were mixed in the stoichiometric ratio and wet ball milled for 24 hours. The slurry was dried at 120°C for 24 hours. The powder obtained was sieved and calcined at 950°C for 4 hours. The calcined powder was treated to a suitable size for AD by secondary wet ball milling. Then, AD process was performed. The process conditions are provided in Table 1.

6BNT-4SBT thick films of thickness 4 μm were fabricated by AD process. As-deposited AD films (as-dep AD) were post-annealed at 550°C for 2 hours at a heating rate of 7°C/min to control crystallinity and internal stress. High-resolution X-ray diffraction (HR-XRD; Philips X’pert pro MRD Diffractometer; Philips, Netherland) was performed to assess crystallinity changes in the 6BNT-4SBT powder, as-dep AD, and post-annealed films (6BNT-4SBT550). Field emission scanning electron microscope (FE-SEM; S-4300SE, Hitachi, Japan) was used to observe the microstructure of the thick films. To measure the electrical properties of the thick films, these were fabricated on a Pt-coated Pt/Ti/SiO₂/Si substrate. After making thick films on the substrate, upper Pt electrodes (0.5 mm in diameter) were produced by a DC sputtering system (Crossington108 auto sputter coater). The εₑ and dielectric loss of the thick films were measured using an impedance analyzer (Agilent technologies 4194a, Santa Clara, CA), and P-E hysteresis loops were measured using a ferroelectric test system (Precision multiferroic and ferroelectric test system, P-PMF-K; Radiant Technologies, Albuquerque, USA). Then, Wₑₑ and η values were calculated from measured P-E hysteresis loops. To measure thermal stabilities with additional SBT, 6BNT-4SBT550 was heated to 140°C and P-E hysteresis loops for each temperature were measured and compared with room temperature values.

3. Results and discussion

To confirm the crystal structure of the powders used in the AD process, the XRD results of the calcined 6BNT-4SBT, mixed 6BNT-4SBT, SBT, and BNT powders were compared as shown in Figure 1. BNT, SBT each powder was calcined to compare with calcined 6BNT-4SBT powder and 6BNT-4SBT mixture powder (Mixed 6BNT-4SBT). Figure 1(a) shows that the calcined 6BNT-4SBT powder had the same perovskite crystal structure as BNT and SBT. There was a peak around 30° formed by Bi-rich phase such as Bi₂Ti₂O₇, Bi₄Ti₃O₁₂ because of Na-deficiency or Bi-excess. When

| Table 1. Typical AD conditions. |
|-----------------------------|
| AD conditions               |
| Carrier gas                 | N₂                     |
| Size of the nozzle orifice  | 5 × 1 mm²              |
| Pressure in the deposition chamber | 0.060 torr          |
| Mass flow controller (MFC)  | 8–16 L/min             |
| Scanning speed of the substrate | 0.7–1 mm/sec          |
| Distance between the nozzle and substrate | 1–5 mm                |

![Figure 1. XRD patterns of BNT, SBT, and 6BNT-4SBT powders used for AD.](image-url)
calcination is conducted to synthesize. A site of ABO3 structure is partially empty due to the various charge valence of Sr$^{2+}$, Bi$^{3+}$, Ti$^{4+}$, O$^2-$. This defect makes few Bi-rich secondary phases formed easily and abnormal 30° peak in XRD results. After the calcination of 6BNT-4SBT, the 30° peak decreases by forming a solid solution [19,20]. An enlarged section from 2θ = 38 to 48° is shown in Figure 1(b), which shows the calcined 6BNT-4SBT peaks located between the BNT and SBT peaks, contrary to the mixed 6BNT-4SBT peaks involving both BNT and SBT peaks. This suggests that calcined 6BNT-4SBT, which is a solid solution, was produced by the reaction between BNT and SBT during calcination.

Through the AD process, films are formed by instantaneous chemical bonding induced by the mechanical energy release when the ceramic powder impacts the substrate. At particles smaller than 100 nm, powder particles do not have sufficient kinetic energy to form a dense film. On the other hand, when the particle sizes are >10 μm, the ceramic could damage the substrate. Thus, it is important to have appropriate particle sizes around 1–2 μm to form pore-free, dense films. Figure 2 presents an SEM image of a cross-section as-dep AD. The film was deposited uniformly on the substrate at a thickness of ~4 μm. It had a dense microstructure without visible pores and adhered well to the substrate. More details of the morphologies and grain sizes of AD films can be found in our previous studies [21–23].

In Figure 1, secondary phases remained after calcination of 6BNT-4SBT. However, referring to Figure 3, it could be seen that the 30° peak reduced due to few amounts of secondary phases and dispersion with scattering after the AD process. When a highly crystalline powder collides with a substrate to form a film, the crystallinity of the powder is reduced because of lattice distortion and large internal stresses generated by the release of kinetic energy during AD process. To improve crystallinity and remove residual stress, AD films were post-annealed at 550°C for 2 hours [22,23]. XRD results of 6BNT-4SBT powder, as-dep AD, and 6BNT-4SBT550 are shown in Figure 3. The peak intensity and the peak position of as-dep AD were reduced and left-shifted, respectively, as compared with 6BNT-4SBT powder. This can be suggested because of residual stress caused by the collision of the powder during the AD process. However, the peak position of the 6BNT-4SBT550 was shifted to an original position with increased diffraction intensity through annealing. It suggests that annealing eliminated the inner stress of AD film and recovered the crystallinity of the 6BNT-4SBT powder [24].

$$D = \frac{0.9\lambda}{\beta(\theta)\cos\theta}$$

Equation (3) is Scherrer’s equation, allowing crystal sizes to be calculated using XRD data. $\lambda$ (0.15406 nm) is the wavelength of Cu-Kα, D is the average crystal size, and $\beta$ is the full width at half maximum (FWHM) [25]. Equation (3) shows a greater full width at half-maximum, corresponding with a smaller crystal size. Compared to the 6BNT-4SBT powder, as-dep AD and 6BNT-4SBT550 had broader peaks because AD reduced crystal sizes. Calculated crystal sizes from the peaks in Figure 3 showed that the crystal sizes of the as-dep AD and 6BNT-4SBT550 were 24 and 29 nm, which confirmed AD reduced crystal size [22]. On the other hand, the X-ray patterns of as-dep AD and 6BNT-4SBT550 showed the presence of amorphous material,

![Figure 2](image-url). Cross-sectional SEM image of as-dep AD.
which was caused by the decrease of crystallinity [22,26]. The recovery of crystallinity by annealing was confirmed by comparing the P-E hysteresis loops, $W_{\text{rec}}$ and $\eta$ values, $\varepsilon_r$, and $E_{\text{DBS}}$ values of as-dep AD and 6BNT-4SBT550.

Figure 4 shows the P-E hysteresis loops for films before and after annealing. Figure 4(a) shows a P-E hysteresis loops of an as-dep AD, which had a lower $P_{\text{max}}$ than 6BNT-4SBT550. After annealing, the $P_{\text{max}}$ of 6BNT-4SBT550 increased; its P-E hysteresis loops are shown in Figure 4(b). When the $P_{\text{max}}$ of 6BNT-4SBT550 and as-dep AD were compared at 900 kV/cm, annealing was found to increase $P_{\text{max}}$ by 263% from 13.7 to 36.1 µC/cm$^2$, which was attributed to the recovery of crystallinity [22,23,27,28]. In addition, the P-E hysteresis loops of 6BNT-4SBT550 were thin and resembled that of a relaxor ferroelectric [15]. Crystal size calculated by Equation (3) from the 6BNT-4SBT550 was 29 nm; that is, nano-sized grains were maintained after annealing.

$W_{\text{rec}}$ and $\eta$ values were calculated from the P-E hysteresis loops, as shown in Figure 5. Table 2 shows $W_{\text{rec}}$ and $\eta$ values of as-dep AD and 6BNT-4SBT550 at 900 kV/cm, which were increased 266% and 126%, respectively, in 6BNT-4SBT550, as compared with as-dep AD. For as-dep AD and 6BNT-4SBT550, $W_{\text{rec}}$ increased and $\eta$ decreased as the applied electric field was increased. From 100 to 900 kV/cm, decreased $\eta$ value of 6BNT-4SBT550 ($\Delta \eta = 20\%$) changed less than the as-dep AD ($\Delta \eta = 49\%$), which suggests 6BNT-4SBT550 had enhanced energy storage properties. Also, increased $E_{\text{DBS}}$ could be obtained with nano-sized grains. This effect exhibited a clearer difference when compared with the value in the bulk form (pellet) conducted in the previous study [15].

$$E_{\text{DBS}} \propto \frac{1}{\sqrt{G}} \quad (4)$$

Because the $E_{\text{DBS}}$ is inversely proportional to grain size ($G$), as shown by Equation (4), $E_{\text{DBS}}$ can be increased by nano-sized grains [29]. The average $E_{\text{DBS}}$ values of as-dep AD and 6BNT-4SBT550 were 1334.2 and 934.3 kV/cm, respectively, which represented increases of 830% and 580%, respectively, versus the pellet’s $E_{\text{DBS}}$ (160 kV/cm). Increases in $E_{\text{DBS}}$ lead to increases in $W_{\text{rec}}$, and as shown in Table 2, the $W_{\text{rec}}$ of 6BNT-4SBT550 was 10.4 J/cm$^3$, which is around 470% higher than the 2.2 J/cm$^3$ of pellet [15]. These results show energy storage properties were greatly improved with nano-sized grains by overcoming the disadvantages of conventionally formed ceramic pellet with large grains.

![Figure 3](image3.png)

**Figure 3.** XRD patterns of as-dep AD and 6BNT-4SBT550.

![Figure 4](image4.png)

**Figure 4.** P-E hysteresis loops of as-dep AD and 6BNT-4SBT550 at 100 Hz.

![Figure 5](image5.png)

**Figure 5.** Energy density ($W_{\text{rec}}$) and efficiency ($\eta$) versus electric field of the as-dep AD and 6BNT-4SBT550 at 100 Hz.

**Table 2.** Energy density and efficiency ($\eta$) versus electric field of 6BNT-4SBT.

|                  | Energy density ($\mu$J/cm$^2$) | $\eta$ (%) |
|------------------|---------------------------------|------------|
| As-dep AD        | 3.9                             | 51.1       |
| 6BNT-4SBT550     | 10.4                            | 64.5       |
| Bulk (Pellet)    | 2.2                             | 75         |
As shown in Figure 6, the reliability of the $E_{\text{DBS}}$ average values was calculated using the Weibull static distribution according to the Equation (5–7).

$$P(E) = 1 - \exp \left\{ - \left( \frac{E}{\sigma} \right)^\beta \right\} \quad (5)$$

$$x_i = \ln(E_i) \quad (6)$$

$$Y_i = \ln \left\{ \ln \frac{1}{1 - c_i} \right\} \quad (7)$$

The Weibull static distribution method provides a measure of the reliability of $E_{\text{DBS}}$, and the shape factor ($\beta$) is proportional to the reliability of $E_{\text{DBS}}$ [12,18,22]. The average $E_{\text{DBS}}$ of as-dep AD was greater than that of 6BNT-4SBT550, but the $E_{\text{DBS}}$ value was widely distributed around the x-axis, meaning $\beta$ was smaller. Conversely, the average $E_{\text{DBS}}$ of 6BNT-4SBT550 was smaller, but its $\beta$ value was larger, meaning greater reliability. These results indicate that the reliability of $E_{\text{DBS}}$ was higher because post-annealing relieved residual stresses.

 Generally, because the value of polarization is proportional to $\varepsilon_r$, higher $\varepsilon_r$ values lead to better energy storage properties for ceramic capacitors. The $\varepsilon_r$ values and dielectric loss tangents (tan$\delta$) of as-dep AD and 6BNT-4SBT550 were measured in the frequency range 1 to 100 kHz, as shown in Figure 7. Table 3 shows $\varepsilon_r$ and tan$\delta$ at 1 and 100 kHz, and that $\varepsilon_r$ was increased by 832% due to the greater crystallinity of 6BNT-4SBT550. Measured tan$\delta$ was slightly increased with increasing frequency in both cases, though slightly more for 6BNT-4SBT550 at high frequency; in both cases, all values showed dielectric loss of <0.1 tan$\delta$ [29–31].

Electronic components must have constant properties over a wide or specified temperature range. Therefore, the unipolar P-E hysteresis loop of 6BNT-4SBT550 in the temperature range 20–140°C was measured in an electric field of 500 kV/cm at a frequency of 100 Hz. Figure 8 shows the temperature dependence of normalized $W_{\text{rec}}$ and $\eta$ values for 6BNT-4SBT550. As the temperature increased from 20 to 140°C, P-E loops become slightly thicker in the x-axis direction with increased remnant polarization [32]. Comparing $W_{\text{rec}}$ and $\eta$ values of 6BNT-4SBT550 at 20 and 140°C, $W_{\text{rec}}$ decreased from 3.75 to 3.40 J/cm$^3$ ($\Delta W_{\text{rec}} \sim -9\%$) and $\eta$ decreased from 86.12% to 60.08% ($\Delta \eta < -30\%$), indicating no significant change from room temperature. These results are consistent with previous studies that reported the addition of SBT to BNT improves thermal stability [13]. Also, as the temperature was increased, the $P_{\text{max}}$ of the 6BNT-4SBT550 increased from 18.5 to 21.1 $\mu$C/cm$^2$ between 20 and 140°C, which suggested $\varepsilon_r$ increased with temperature. These results show that the 6BNT-4SBT550 thick film formed by the AD process had constant energy storage properties in the temperature range of 20–140°C, with excellent thermal stability.

4. Conclusion

In this study, a dense 6BNT-4SBT thick film with a thickness of 4 $\mu$m was formed by AD to produce a dielectric capacitor with improved energy storage properties and thermal stability. By adding the relaxor ferroelectric SBT to ferroelectric BNT, the large hysteresis loss of BNT was reduced and high thermal stability was obtained. The 6BNT-4SBT thick film formed by AD exhibited high energy storage characteristics by lowering $P_r$ and increasing $E_{\text{DBS}}$. After heat treatment,
nano-sized grains and improved crystallinity in 6BNT-4SBT550 were confirmed by XRD. As compared with conventionally formed pellet, 6BNT-4SBT550 showed a 470% increase in energy storage characteristics with $W_{\text{rec}}$ of $10.4 \text{ J/cm}^3$ and $\eta$ of 64.5% at 900 kV/cm. $W_{\text{rec}}$ and $\eta$ were calculated by measuring P-E hysteresis loops at temperatures from 20 to 140°C. At 140°C, $W_{\text{rec}}$ was 91% ($\Delta W_{\text{rec}} < -9\%$) and $\eta$ was 70% ($\Delta \eta < -30\%$) of the room temperature values, indicating that 6BNT-4SBT had outstanding thermal stability. These results show the post-annealing recovered the crystallinity of 6BNT-4SBT thick film after AD and that this film has the potential use in electrical energy storage systems requiring thermal stability.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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