



Design of high energy-storage properties in eco-friendly AgNbO₃-based ceramics via two-step sintering method and tuning phase boundary

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Received: 28 August 2022

Accepted: 10 November 2022

Published online:
20 November 2022

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ABSTRACT

Ceramic samples AgNb_{0.85}Ta_{0.15}O₃ (ANTO15) and Ag_{0.85}Bi_{0.05}NbO₃ (ABNO5) were obtained by two-step sintering method. Dielectric spectra revealed that the M1–M2 and M2–M3 phase boundaries were adjusted to room temperature for ANTO15 and ABNO5, respectively. The ABNO5 sample exhibits pure perovskite phase structure with small grain size, dense, and uniform microstructure. Most importantly, superior comprehensive energy-storage performances of a large recoverable energy-storage density value $\sim 3.53 \text{ J/cm}^3$, high energy-storage efficiency $\sim 86\%$, high power density $\sim 73.57 \text{ MW/cm}^3$, as well as good energy-storage stabilities were obtained in the ABNO5 ceramic. Our results indicate that the combinative utilization of tuning phase boundary and two-step sintering method gives a feasible method to prepare high energy-storage properties AgNbO₃-based eco-friendly ceramic capacitors.

Introduction

Dielectric energy-storage ceramic capacitors characterized by ultrafast charge–discharge speed, long lifetime, and high power density have received global attentions in recent years [1]. But the low energy-storage density greatly limits their application in real life and production [2]. In general, for dielectric energy-storage materials, the recoverable energy-storage density (W_{rec}) and energy-storage efficiency

(η) are obtained by the polarization–electric field (P – E) hysteresis loop as following [3]:

$$W_{rec} = \int_{Pr}^{P_{max}} EdP \quad (1)$$

$$\eta = W_{rec} / (W_{rec} + W_{loss}) \quad (2)$$

where E presents the electric field, P_{max} is the saturation polarization, and P_r means the remnant polarization as well. In addition, the W_{loss} equals the area surrounded by the hysteresis loop. Antiferroelectric (AFE) ceramics have been considered to hold tremendous promise for energy-storage application

Handling Editor: David Cann.

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due to their double P - E loops [4]. However, most AFE ceramics with high energy-storage density contain lead element [5, 6], which is harmful for human health and environment. Thus, there is an ongoing need for exploring novel lead-free AFE energy-storage ceramics. In recent years, Zhao et al. reported that AgNbO_3 lead-free ceramic showed the typical anti-ferroelectric behavior of double P - E loops and high energy-storage density [7]. However, pure AgNbO_3 ceramic still exhibits the shortcomings of low values of breakdown electric field (E_B) and η . In order to solve the problems, the optimizing preparation technologies have been performed in an effort to improve the E_B value of pure AgNbO_3 ceramic [8, 9]. In our previous work, we have confirmed that pure AgNbO_3 ceramic with fine-grained and dense microstructure can be prepared by a simple two-step sintering method. This structure feature gives rise to enhanced breakdown field strength. Nevertheless, the two-step sintering method has no obvious influence on the phase transition temperature and energy-storage efficiency of pure AgNbO_3 ceramic [9]. Currently, the common method for adjusting phase transition temperature and improving energy-storage efficiency of the pure AgNbO_3 ceramic is element-doping at the A- or B-sites [10, 11].

Based on these backgrounds, it is believed that combinative utilization of tuning phase boundary and two-step sintering method would contribute to superior comprehensive energy-storage properties of AgNbO_3 ceramics. Therefore, element -doping at A- or B-sites was used aiming at tuning the phase boundaries of AgNbO_3 ceramics. Two groups AgNbO_3 -based ceramic samples, namely $\text{AgNb}_{0.85}\text{Ta}_{0.15}\text{O}_3$ (abbreviated as ANTO15) and $\text{Ag}_{0.85}\text{Bi}_{0.05}\text{NbO}_3$ (abbreviated as ABNO5), were designed. According to Refs. 10 and 11, ANTO15 and ABNO5 should show M1–M2 and M2–M3 phase transitions at room temperature, respectively. Meanwhile, the two groups of samples were prepared by two-step sintering method with the purpose to achieve boosted E_B in these samples. A schematic diagram of this novel approach to obtain excellent energy-storage properties of AgNbO_3 -based lead-free ceramics is clearly displayed in Fig. 1.

Experimental

The experimental procedure for the synthesis of the ceramics and property measurements are described in the Supplementary files.

Results and discussion

Figure 2A and b shows the temperature-dependent dielectric properties measured at various frequencies of the ANTO15 and ABNO5 ceramics. Consistent with our design goals, the M1–M2 and M2–M3 phase boundaries were adjusted to room temperature for the ANTO15 and ABNO5, respectively. The dielectric properties of ABNO5 also exhibit excellent temperature stability. This material would be a novel Class II dielectrics for multilayer ceramic capacitors (MLCC) applications. To corroborate this point, the dielectric constant variations of $\Delta\epsilon'/\epsilon'_{25^\circ\text{C}}$ @50, 100, and 500 kHz in the temperature range of -100 – 250 °C are calculated and presented in Fig. 3a. The variations in dielectric constant within 15% with low loss less than 0.01 (seen in Fig. 3b) can be achieved over the temperature range of -100 – 196 °C. This finding demonstrates that the ABNO5 ceramic meets the requirements for X8R capacitors.

Figure 4a and b presents P - E loops of both samples measured at room temperature. The ANTO15 sample shows double loops, characterizing the typical anti-ferroelectric feature. The W_{rec} and η values under the measured electric field of 28 kV/mm were calculated to be 4.2 J/cm³ and 72%, respectively, which are better than those of the sample prepared by one-step sintering method in other work [10]. As seen from Fig. 4b, the ABNO5 ceramic sample does not show a square-shaped P - E curve similar to that of AgNbO_3 -based ceramics reported in the past. It shows a slim P - E curve, which is similar to that of relaxor antiferroelectrics reported before [12]. As is well known, in AgNbO_3 -based ceramics, the M1 phase shows metastable AFE characteristic under applied electric field, leading to a large P_r value. On the other hand, the M2 and M3 phases are regarded as two disordered AFE phases. As a result, relaxor characteristic with slim P - E loop is expected if M2–M3 phase transition temperature moves downward to room temperature, which can decrease P_r value and enhance energy-storage properties. Under the tested electric field of 37 kV/mm, W_{rec} was deduced to be

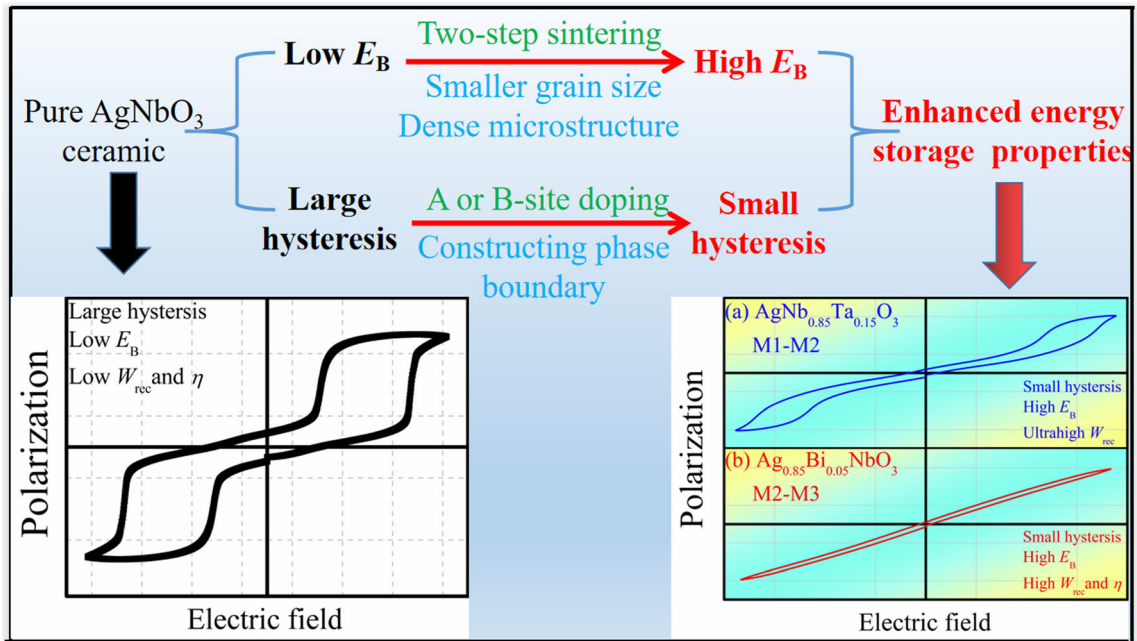


Figure 1 A schematic diagram of the approach to obtain excellent energy-storage properties of AgNbO_3 -based lead-free ceramics.

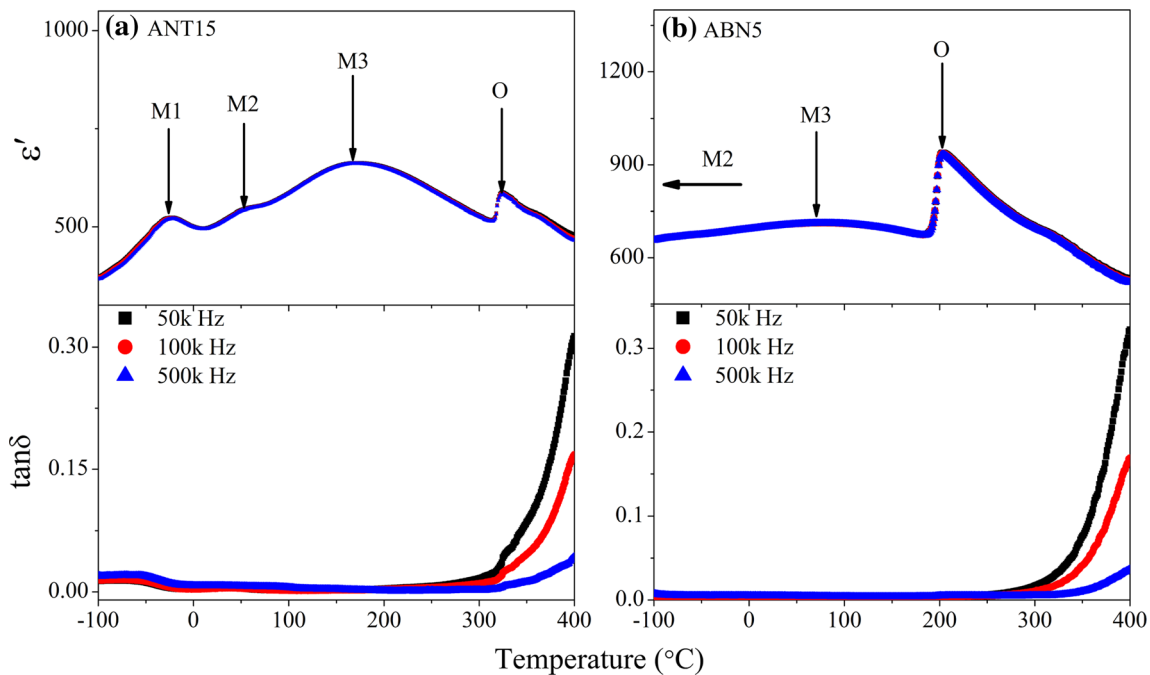


Figure 2 Temperature-dependent dielectric properties of **a** ANTO15 and **b** ABNO5.

3.53 J/cm^3 and $\eta = 86\%$. This fact demonstrates that high values of W_{rec} and η are simultaneously achieved in the ABNO5 ceramic. It can be seen from Figs. S1 and S2 that both samples in this work exhibit pure perovskite and dense microstructure. Compared with ANTO15 sample, the ABNO5 sample has

smaller average grain size ($2.48 \mu\text{m}$). It is well known that small grain size, homogeneous, and dense microstructure can significantly improve the dielectric breakdown strength [1]. For the present samples, the W_{rec} value of the ABNO5 ceramic is somewhat smaller than that of the ANTO15. But the η value of

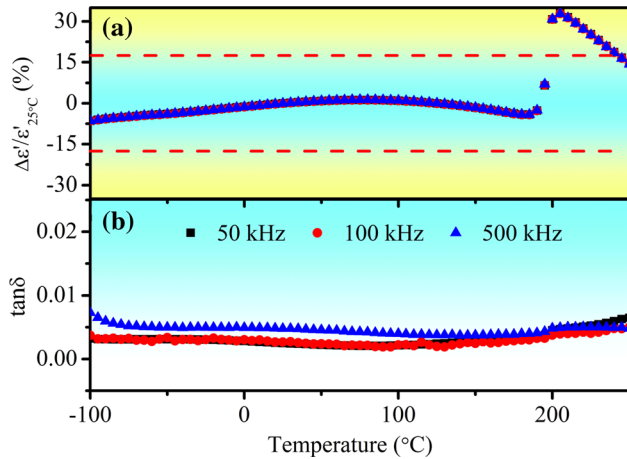


Figure 3 (a) Dielectric constant variation normalized to that at 25 °C and (b) corresponding loss tangent of ABNO5 over the temperature range of - 100–250 °C.

the ABNO5 ceramic is much higher than that of the ANTO15. Furthermore, Fig. 4c displays a comparison

of the energy-storage properties between the ABNO5 sample and a large number of other energy-storage ceramics reported recently in literature [11, 13–41]. We can clearly see that the energy-storage properties of ABNO5 sample are much better than most of them.

Additionally, Fig. 5a presents the *P*-*E* loops of the ABNO5 ceramic measured at 25 kV/mm and 10 Hz in a wide temperature range of 20–100°C. Slim *P*-*E* loops can be observed in the measured temperature range. The calculated W_{rec} values are shown in Fig. 5b, which reveals that the W_{rec} values are fluctuating between 1.67 and 1.69 J/cm³. The *P*-*E* loops of the ABNO5 ceramic measured at 25 kV/mm and room temperature in the frequency range of 1–200 Hz are also shown in Fig. 5c. Correspondingly, the calculated W_{rec} (1.67~1.7 J/cm³) and η (90~96%) also maintain stable values in the frequency range as shown in Fig. 5d, which means that the ABNO5 sample also possesses excellent temperature and frequency stabilities for energy-storage

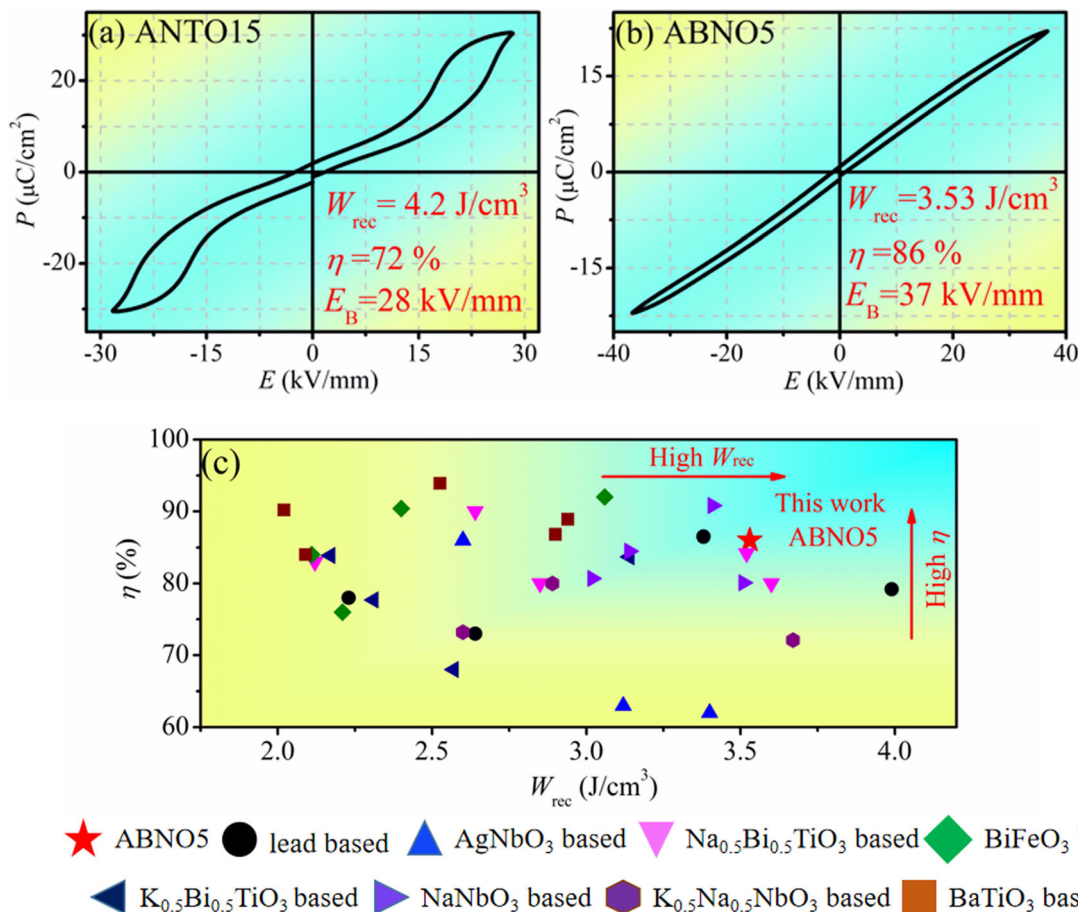


Figure 4 *P*-*E* loops of a ANTO15, b ABNO5, and c comparison of energy-storage properties between the ABNO5 sample and some other dielectric ceramics.

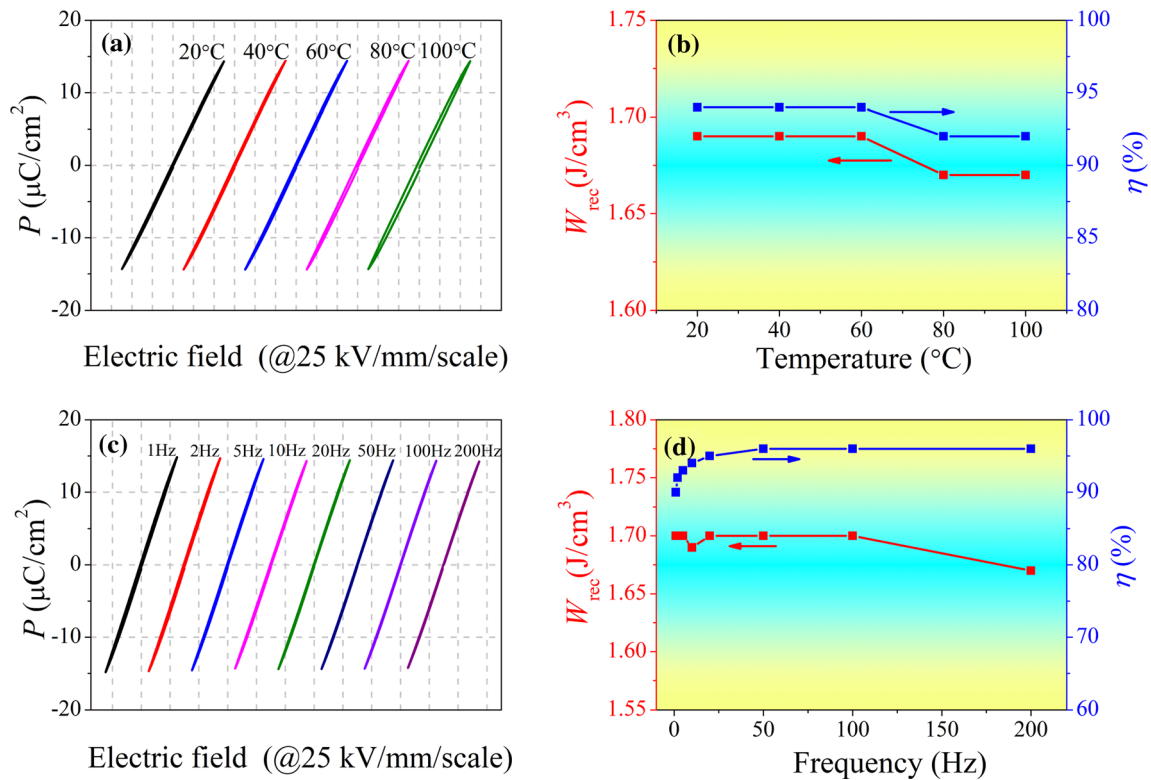


Figure 5 **a** P - E loops of the ABNO5 ceramic measured at 25 kV/mm and 10 Hz in the temperature range of 20–100 °C, **b** W_{rec} and η values as a function of temperature, **c** P - E loops of the ABNO5

ceramic measured at 25 kV/mm and room temperature in the frequency range of 1–200 Hz, and **d** W_{rec} and η values as a function of frequency.

performances. All the features underscore that the ABNO5 sample possesses superior comprehensive energy-storage properties suitable for device application.

Figure 6a shows the undamped pulsed current curves of the ABNO5 ceramic under various electric fields measured at room temperature. The current peak increases as the electric field increases. Under an applied electric field of 20 kV/mm, the maximum

current value of 23.1 A can be obtained. The current density (C_D) and power density (P_D) can be calculated as follows [42, 43]:

$$C_D = I_{\text{max}}/S \quad (3)$$

$$P_D = EI_{\text{max}}/2S \quad (4)$$

where E and S represent the electric field and electrode area, respectively. According to the above

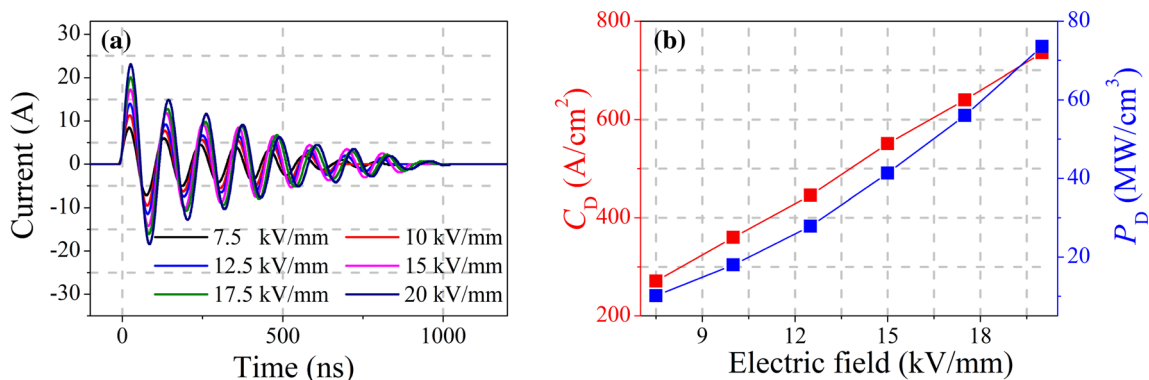


Figure 6 **a** Undamped pulsed current curves under various electric fields, and **b** C_D and P_D as a function of electric field of the ABNO5 ceramic.

Table 1 A comparison between the P_D values of ABNO5 ceramic and other dielectric energy-storage ceramics published in literature [9, 44–49]

Samples	P_D (MW/cm ³)	Ref
Ag _{0.85} Bi _{0.05} NbO ₃	73.57	This work
AgNbO ₃	25.7	[9]
Sr ₂ Ag _{0.2} Na _{0.8} Nb _{4.7} Ta _{0.3} O ₁₅	70.21	[44]
0.84Bi _{0.52} Na _{0.48} TiO ₃ -0.16KNbO ₃	66	[45]
0.9(Sr _{0.7} Bi _{0.2})TiO ₃ -0.1Bi(Mg _{0.5} Zr _{0.5})O ₃	62.6	[46]
0.9(0.76Bi _{0.5} Na _{0.5} TiO ₃ -0.24SrTiO ₃)-0.1Bi(Ni _{2/3} Nb _{1/3})O ₃	49.8	[47]
0.9(0.75BaTiO ₃ -0.25Na _{0.5} Bi _{0.5} TiO ₃)-0.1Bi(Zn _{0.2} Mg _{0.2} Al _{0.2} Sn _{0.2} Zr _{0.2})O ₃	34.76	[48]
Ag _{0.97} Nd _{0.01} NbO ₃	54	[49]

formulas, the C_D and P_D values obtained under various electric fields are shown in Fig. 6b. The ABNO5 ceramic has a high power density value $P_D = 73.57 \text{ MW/cm}^3$ at 20 kV/mm. Furthermore, it can be found in Table 1 that the P_D value of the ABNO5 ceramic is far greater than to those of other dielectric energy-storage ceramics [9, 44–49]. The high W_{rec} combined with high P_D in the ABNO5 ceramic suggests its promising application potential in pulsed power system.

Conclusions

In this work, ANTO15 and ABNO5 ceramics were successfully prepared via two-step sintering method. Both samples show high W_{rec} values. Excitingly, superior comprehensive energy-storage properties of large W_{rec} (3.53 J/cm^3), high η (86%), and ultrahigh power density (73.57 MW/cm^3) were achieved in ABNO5 sample. Furthermore, ABNO5 sample also has excellent dielectric temperature stability meeting the requirements for X8R capacitors. All above results indicate that the ABNO5 ceramic has great potential for the applications of pulsed power systems.

Acknowledgements

The authors acknowledge financial support from the National Natural Science Foundation of China (Grant Nos. 51872001), Scientific Research Starting Foundation of Anhui Polytechnic University of China (2021YQQ031), the Natural Science Foundation of Anhui Polytechnic University (Grant No. Xjky2022029), and the Open Research Fund Program

of the Key Laboratory of Functional Materials and Devices for Informatics of Anhui Higher Education Institutes, Fuyang Normal University (Grant No. FDMI202105).

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

Supplementary Information: The online version contains supplementary material available at <http://doi.org/10.1007/s10853-022-07964-5>.

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