High Discharge Energy Density at Low Electric Field Using an Aligned Titanium Dioxide/Lead Zirconate Titanate Nanowire Array

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Polymer-based capacitors with high energy density have attracted significant attention in recent years due to their wide range of potential applications in electronic devices. However, the obtained high energy density is predominantly dependent on high applied electric field, e.g., 400–600 kV mm⁻¹, which may bring more challenges relating to the failure probability. Here, a simple two-step method for synthesizing titanium dioxide/lead zirconate titanate nanowire arrays is exploited and a demonstration of their ability to achieve high discharge energy density capacitors for low operating voltage applications is provided. A high discharge energy density of 6.9 J cm⁻³ is achieved at low electric fields, i.e., 143 kV mm⁻¹, which is attributed to the high relative permittivity of 218.9 at 1 kHz and high polarization of 23.35 µC cm⁻² at this electric field. The discharge energy density obtained in this work is the highest known for a ceramic/polymer nanocomposite at such a low electric field. The novel nanowire arrays used in this work are applicable to a wide range of fields, such as energy harvesting, energy storage, and photocatalysis.

Energy storage materials play an indispensable role in modern electronic devices and electric power systems. The development of high-energy-storage-density devices is of critical importance to meet the ever-increasing demand for these applications.¹² Dielectric materials, which possess high charge–discharge capability to store and release electrical energy through dielectric polarization and depolarization, have attracted immense interest for potential applications in energy storage devices, such as capacitors.¹³ The discharge energy density (Uₘₐ) of a dielectric material is given by

\[
U_{\text{dis}} = \int E \, dD
\]

where \( E \) is the applied electric field and \( D \) is the electric displacement

\[
D = \varepsilon_r \varepsilon_0 E
\]

where \( \varepsilon_0 \) and \( \varepsilon_r \) are vacuum permittivity and the relative permittivity of the dielectric, respectively. Therefore, relative permittivity and breakdown strength are key factors in determining the energy density.

Among numerous dielectric materials, polymer nanocomposites are receiving growing attention because of their advantage in combining the merits of ceramics (e.g., high permittivity) with those of polymers (e.g., high breakdown strength).²⁵ A number of studies have reported ceramic/polymer nanocomposites with high energy density.⁶,⁷ For example a discharge energy density of 20 J cm⁻³ at 646 kV mm⁻¹ was reported for BaTiO₃@TiO₂ core–shell fibers in a polyvinylidene fluoride polymer matrix (denoted as BaTiO₃@TiO₂/PVDF, where @ denotes a core–shell structure).⁸ This was subsequently improved to 31.2 J cm⁻³ at 797.7 kV mm⁻¹ for nanocomposites with large aspect ratio fibers as a result of their preferred orientation directions perpendicular to the external electric field.⁹ However, these nanocomposites still possess relatively low energy density at a lower electric field, e.g., an energy density of ≈2.8 J cm⁻³ at the electric field of 250 kV mm⁻¹. Use of these materials exclusively at high applied electric field is likely to present challenges relating to the failure probability. More recently, it was shown by Xie et al. that a high discharge energy density of 10.8 J cm⁻³ at a relatively low electric field of 240 kV mm⁻¹ could be achieved in BaTiO₃/PVDF nanocomposites with aligned BaTiO₃ nanowires.¹⁰

Therefore, polymers incorporated with aligned ceramic nanowires represent an effective design for high discharge energy density systems.¹⁰,¹¹ However, the preparation of aligned ceramic nanowires is challenging, especially for aligned ferroelectric ceramic nanowire arrays, such as lead zirconate titanate (PZT) and BaTiO₃ nanowire arrays due to their randomly oriented grains.¹² A simple method to prepare high-performance capacitors utilizing anatase TiO₂ nanowire arrays as the fillers has recently been reported.¹³,¹⁴ However, TiO₂ is not a ferroelectric and has relatively low permittivity and low saturation polarization, which lead to limited energy density.

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PZT is the most widely used piezoelectric ceramic and shows advantages in energy storage applications attributed to the high permittivity, low remnant polarization, and low cost. Combining the electrical properties of PZT with TiO₂ to readily form nanowires represents an attractive strategy for the fabrication of a high discharge energy density nanocomposite. Here we present a simple two-step method for synthesizing titanium dioxide/lead zirconate titanate (TiO₂@PZT) nanowire arrays; the PZT was designed as a shell layer coated on the surface of TiO₂ nanowires via spin coating and subsequent annealing. The morphologies of the synthesized TiO₂@PZT nanowire arrays were tuned by modulating the concentration of the Ti source. Poly(vinylidene fluoride-trifluoroethylene-chlorotrifluoroethylene) (P(VDF-TrFE-CTFE)) was selected as the polymer matrix to combine with the synthesized TiO₂@PZT nanowire arrays, since it is a relaxor ferroelectric polymer with high permittivity and low remnant polarization. Due to the high interfacial polarization, nanocomposites with high permittivity and high discharge energy density at low electric field were achieved. This work provides a novel route to prepare ceramic nanowire arrays and high energy density polymer based capacitors capable of operating at low voltage.

Figure 1 shows the X-ray diffraction (XRD) patterns of the TiO₂ nanowire array, Pb(Zr₀.₅₂Ti₀.₄₈)O₃ powders, and the TiO₂@PZT nanowire arrays. As illustrated in Figure 1a, in addition to the peaks of the fluorine doped tin oxide (FTO) substrate, three peaks located at 2θ values of 36.30°, 54.69°, and 63.11° are observed, which match well with the (101), (211), and (002) planes of rutile TiO₂ (JCPDS No. 01-88-1172). The narrow peak width indicates that TiO₂ nanowires possess relatively good crystallinity. The XRD pattern of the PZT power derived from the sol confirms PZT to be in the perovskite tetragonal phase with lattice constants of a = 0.4056 nm and c = 0.4110 nm, matching well with the literature values of a = 0.4036 nm and c = 0.4146 nm (JCPDS No. 33-0784). The XRD pattern of TiO₂@PZT nanowire arrays can be observed in Figure 1b. In addition to the strong peaks associated with TiO₂, there are weaker peaks at 30.92°, 31.36°, and 44.88° 2θ degrees, corresponding to the (101), (110), (200) planes of PZT.

Figure 2a,b illustrates the cross-sectional and the surface scanning electron microscope (SEM) images of the TiO₂ nanowire array grown on FTO, respectively. It can be observed that the TiO₂ nanowire array is uniformly distributed on the FTO substrate and had preferred orientation in the [001] direction. It is believed that FTO substrate plays an important role in generating the 1D structure by promotion of epitaxial nucleation and growth in the [001] direction due to the small lattice mismatch between the rutile structures of FTO and TiO₂.[16] The morphologies of the synthesized TiO₂@PZT nanowire arrays were tuned by modulating the concentration of Ti source from 0.03 to 0.06 mol L⁻¹, as shown in Figure S1 in the Supporting Information. After the spin-coating and annealing process, the PZT phase was coated on the surface of TiO₂ nanowire array. The resulting TiO₂@PZT nanowire arrays were relatively denser than the parent TiO₂ nanowire array, as shown in Figure 2d, and maintained the perpendicular orientation to the substrate.

Figure 2e shows an image of the TiO₂@PZT nanowire array-2 polymer nanocomposite, which was typical and was ≈7 µm thick. The upper P(VDF-TrFE-CTFE) layer had a thickness of ≈4 µm, and the TiO₂@PZT nanowire array can be seen sandwiched between the polymer layer and the FTO substrate. No obvious defects were found in the nanocomposites, which indicated that the TiO₂/PZT nanowire array was tightly encapsulated by the polymer matrix. Energy-dispersive X-ray spectroscopy (EDS) elemental mapping and transmission electron microscopy (TEM) results in Figure 2f–k were used to prove the PZT layer was coated on the surface of TiO₂ nanowires. Ti and O are seen to be uniformly distributed in the TiO₂ nanowire array (Figure 2f), while in the TiO₂@PZT nanowire array shown in Figure 2g Pb and Zr were additionally detected in the EDX map. Figure 2h shows a TEM image of the as-prepared TiO₂ nanowires illustrating a clear nanowire shape with a diameter of 10–80 nm. The high-resolution transmission electron microscopy (HRTEM) image of the TiO₂ nanowire is depicted in Figure 2i, with a lattice spacing of 0.324 nm, which matches well with the (110) diffraction plane of rutile TiO₂. Figure 2j shows the TEM image of a TiO₂@PZT nanowire, indicating the existence of a thin layer on the fringe of TiO₂ nanowire. Figure 2k shows an HRTEM image of this thin layer, which shows a lattice spacing of 0.291 nm attributable to the (101) plane of the tetragonal phase of PZT, consistent with the XRD results. These results confirm the successful preparation of TiO₂@PZT nanowire arrays.

Figure 3a,b shows the variation of the permittivity and dielectric loss of P(VDF-TrFE-CTFE) and TiO₂@PZT nanowire array/P(VDF-TrFE-CTFE) nanocomposites with different...
concentrations of the Ti source as a function of frequency at room temperature. As illustrated in Figure 3a, the permittivity of the nanocomposites decreases with increasing frequency for all samples, and increases with increasing Ti source concentration over the whole frequency range. The permittivity of the TiO$_2$@PZT nanowire array-1 nanocomposite reached 168.7 at 1 kHz, while that of the pure P(VDF-TrFE-CTFE) sample was 33.5 at the same frequency. The permittivities of the other two samples were 198.3 and 218.9 at 1 kHz, respectively. All nanocomposites showed much higher relative permittivity than the pure polymer. This can be explained by considering the various contributions to the overall polarization made by the different components of the composite and the interfaces between them.

First, the aligned TiO$_2$ nanowire array fillers play an important role in contributing to the polarization. Yao et al. reported that nanocomposites with orientated TiO$_2$ nanowire array fillers showed significantly enhanced permittivity.$^{[13]}$ In addition the PZT has a high relative permittivity, with values around 1800,$^{[17]}$ while the terpolymer P(VDF-TrFE-CTFE) itself also has a relatively high permittivity, which is demonstrated by the test in Figure S2 (Supporting Information). Second, the system comprises two large interfacial regions, i.e., the TiO$_2$-PZT and PZT-P(VDF-TrFE-CTFE) interfaces. The interfacial polarizations will also contribute to the enhanced permittivity of the system.

The breakdown strengths of the nanocomposites analyzed with a two-parameter Weibull distribution function are shown in Figure 3c. As can be seen, the characteristic breakdown strengths are relatively lower than many reported results. There are a number of reasons why breakdown strengths are lower than in other nanocomposite systems; these include (i) defects at the interfaces between components of the composites can lead to premature failure; (ii) the electric field is mainly contained in the thin polymer layer above the nanowire array due to the big permittivity contrast between the nanowire array and polymer matrix; and (iii) the relatively high conductivity and leakage current shown in Figures S3 and S4 in the Supporting Information can also decrease the breakdown strength of the composites due to the increased charge motion.$^{[18]}$ Typical electrical polarization–electric field ($P$–$E$) loops of the nanocomposites measured at a frequency of 10 Hz at room temperature are shown in Figure 3d. The maximum polarization ($P_{\text{max}}$) values for the TiO$_2$@PZT nanowire array-1, TiO$_2$@PZT nanowire array-2, and TiO$_2$@PZT nanowire array-3 nanocomposites were 14.22, 18.38, and 23.35 $\mu$C cm$^{-2}$, respectively (Figure 3e), while that for the pure terpolymer was 5.2 $\mu$C cm$^{-2}$.$^{[19]}$ The increase of $P_{\text{max}}$ with increasing Ti concentration is in agreement with our previous work,$^{[14]}$ which also indicated that the density and orientation of TiO$_2$ nanowire array play an important role in...
the enhancement of dielectric displacement for the nanocomposites. The PZT phase introduced to the nanocomposites provides more interfacial areas; therefore, the enhanced interfacial polarization leads to a high total polarization value. The relation between electric displacement ($D$) and polarization ($P$) is given by

$$D = \varepsilon_0 E + P$$

The maximum in electric displacement, $D_{\text{max}}$, is approximately equal to $P_{\text{max}}$ due to the low value of the vacuum permittivity. The difference between $P_{\text{max}}$ and residual polarization ($P_r$) for the TiO$_2$@PZT nanowire array-1 nanocomposite reached 11.42 $\mu$C cm$^{-2}$ at 100 kV mm$^{-1}$, while that for the TiO$_2$@PZT nanowire array-2 nanocomposite was 15.62 $\mu$C cm$^{-2}$ at 130 kV mm$^{-1}$, the highest value of 16.93 $\mu$C cm$^{-2}$ achieved for the TiO$_2$@PZT nanowire array-3 nanocomposite at an electric field of 143 kV mm$^{-1}$. Indeed, the $P_{\text{max}}$ and $P_{\text{max}} - P_r$ values for the TiO$_2$@PZT nanowire array-3 nanocomposite are the highest recorded at such a low electric field for a polymer-based dielectric. It is worth noting that the enhanced $P_{\text{max}}$ and $P_{\text{max}} - P_r$ values at a low electric field are critical for obtaining a high-endurance high-discharge energy density. High fatigue endurance is an important factor during the long-term charge–discharge cycling process of dielectrics. Charge–discharge cycling was carried out for up to $10^6$ cycles, with pulse field amplitudes of 40 kV mm$^{-1}$, four points per decade of 4, and a pulse frequency of 1 kHz. The residual polarization ($P_r$) of the nanocomposites as a function of the cycle number is shown in Figure 3f. It can be seen that the nanocomposites hold high fatigue endurance, e.g., the $P_r$ of the TiO$_2$@PZT nanowire array-3 nanocomposite only increased by 6.8% after $10^6$ cycles.

Figure 4a shows a typical $P$–$E$ loop for a nonlinear dielectric material, in which the blue colored region represents the discharge energy density, the red region is the energy loss, and the stored energy density includes the discharge energy density and energy loss. The discharged energy density and efficiencies for the TiO$_2$@PZT nanowire array nanocomposites were determined from their $P$–$E$ loops and are plotted as a function of electric field in Figure 4b–d. The efficiency, $\eta$, was calculated as follows

$$\eta = U_{\text{dis}} / U_{\text{max}}$$

where $U_{\text{max}}$ is the energy storage density. All of the nanocomposites exhibit a large discharge energy density and a relatively high $\eta$ value. The maximum discharge energy density of TiO$_2$@PZT nanowire array nanocomposites was 4.0 J cm$^{-3}$ ($\eta = 58.1\%$) at 100 kV mm$^{-1}$ and 6.0 J cm$^{-3}$ ($\eta = 59.7\%$) at 130 kV mm$^{-1}$, arrays 1 and 2, respectively. Most notably, the TiO$_2$@PZT nanowire array-3 nanocomposite achieved the highest discharged energy density of 6.9 J cm$^{-3}$ with a relatively high $\eta$ of 50.0% at an electric field at 143 kV mm$^{-1}$. To the best of our knowledge, this is the highest discharge energy density obtained at such a low electric field for a polymer-based nanocomposite. Table 1 summarizes the performance of some typical polymer-based dielectric composites with the electric field. The large energy density at such a low electric field in these nanocomposites can be attributed to (i) the dual phase nature of the TiO$_2$@PZT nanowire array, which results in large interfacial regions and hence large interfacial polarization; (ii) the highly oriented nanowire also plays an important role in increasing the polarization and electromechanical coupling of the nanocomposites; and (iii) the obtained high
permittivity of the nanocomposites yields high saturation polarization (Equation (3)), and the nanocomposites possess narrow hysteresis loops with low values of $P_r$ as shown in Figures S5–S7 (Supporting Information), which lead to largely enhanced $P_{\text{max}} - P_r$ values.

It has been demonstrated that TiO$_2$@PZT nanowire arrays can be prepared by a simple hydrothermal method via a spin-coating and annealing process. The TiO$_2$@PZT nanowire array polymer nanocomposites exhibit significantly enhanced permittivity, as well as electric displacement at low electric field, compared to pure polymers and most previously reported polymer nanocomposites. This is achieved due to high interfacial polarization and interfacial coupling effects. It has been shown that a TiO$_2$@PZT nanowire array nanocomposite can achieve a high energy density of 6.9 J cm$^{-3}$ at a low electric field of 143 kV mm$^{-1}$.

This work provides a promising strategy for high-energy density capacitors at a lower operating voltage.

**Experimental Section**

Materials: The terpolymer P(VDF-TrFE-CTFE) composition had a mole ratio of 63:29:8 and was provided by PolyK Technologies, LLC; FTO substrate (Pilkington, TEC7 coated, 1.6 mm thickness, 7 $\Omega$ sq$^{-1}$), tetrabutyl titanate ($C_{16}H_{36}O_4Ti$) (Guoyao from China, 98.0%), lead acetate trihydrate [$Pb(CH_3COO)_2.3H_2O$] (Guoyao from China, 99.5%), zirconium nitrate pentahydrate [$Zr(NO_3)_4.5H_2O$] (Guoyao from China), and tetrabutyl titanate ($C_{16}H_{36}O_4Ti$). All chemicals were used “as received,” unless indicated.

Synthesis of TiO$_2$ Nanowire Array: The TiO$_2$ nanowire arrays were synthesized through a hydrothermal method. A solution containing a mixture of 25 mL HCl (36–38%), 25 mL deionized (DI) water, and various volumes of $C_{16}H_{36}O_4Ti$ (0.5, 0.7, and 1.0 mL corresponding to 0.03, 0.04, and 0.06 mol L$^{-1}$, respectively) was stirred until it became transparent. Subsequently, the transparent solution was transferred into a 100 mL autoclave together with a piece of FTO substrate (10 mm × 15 mm), which was sonicated successively in acetone, ethanol, and DI water for 10 min. The autoclave was maintained at 180 °C for 3 h to synthesize the TiO$_2$ nanowire array on FTO substrate.

**Table 1.** Comparison of discharged energy density for dielectric composites with different nanofiber fillers at applied electric field.

| Matrix       | Fillers          | $E_b$ [kV mm$^{-1}$] | $U_{\text{dis}}$ [J cm$^{-3}$] | $E$ [kV mm$^{-1}$] | $U_{\text{dis}}$ [J cm$^{-3}$] | Ref.     |
|--------------|------------------|----------------------|-------------------------------|-------------------|-------------------------------|---------|
| PVDF         | BaTiO$_3$        | 470                  | 18.8                          | 200               | ≈3.8                          | [6]     |
| PVDF         | BaTiO$_3$@TiO$_2$| 350                  | 12.5                          | 150               | ≈2.8                          | [20]    |
| P(VDF-HFP)   | BaTiO$_3$@TiO$_2$| 797.7                | 31.2                          | 200               | ≈2.4                          | [9]     |
| PVDF         | BaTiO$_3$        | 450                  | 10.0                          | 174               | ≈2.6                          | [21]    |
| PVDF         | BaTiO$_3$@Al$_2$O$_3$ | 420              | 10.6                          | 200               | ≈2.2                          | [22]    |
| PVDF         | Ba$_2$S$_3$TiO$_3$ | 450                  | 14.9                          | 150               | ≈2.0                          | [7]     |
| P(VDF-TrFE-CTFE) | BaTiO$_3$    | 300                  | 10.6                          | 150               | ≈4.0                          | [23]    |
| P(VDF-TrFE-CTFE) | TiO$_2$@PZT | –                   | –                             | –                 | 143                           | 6.9     | This work |
Preparation of the PZT Sol: A mixed solution with a Pb:Zr:Ti mole ratio of 1.1:0.52:0.48 was prepared from appropriate portions of lead acetate trihydrate [Pb(CH3COO)2·3H2O], zirconium nitrate pentahydrate [Zr(NO3)4·5H2O], and tetrabutyl titanate [C16H36O4Ti]. At first, the lead acetate and zirconium nitrate pentahydrate was dissolved in 2-methoxyethanol separately and then mixed together and stirred for 20 min. Due to the instability of tetrabutyl titanate, three to four drops of acetyl acetone were added to the solution as a stabilizer. Next, the solution was heated at 97 °C and stirred for 1 h. The lead zirconium titanium solution was aged for 20 min. Finally, the solution was heated at 97 °C and stirred for 1 h. The lead zirconium titanium solution was aged for 1 week, resulting in a light yellow clear solution.

Preparation of Nanocomposites: The TiO2 nanowire arrays on the FTO substrate were dipped into the Pb(Zr0.52Ti0.48)O3 (PZT, 0.2 mol L−1) sol, and treated by spin-coating, followed by heating at 200 °C for 5 min, 350 °C for 5 min, 400 °C for 5 min, and then thermal annealing at 600 °C for 500 s in air. The P(VDF-TrFE-CTFE)/N,N-dimethylformamide solution was coated on the surface of the TiO2@PZT nanowire arrays by spin-coating to obtain the TiO2@PZT/P(VDF-TrFE-CTFE) nanocomposite with a thickness of ~7 μm. The obtained samples were dried at 70 °C for 24 h. For electrical measurements the nanocomposites were sputtered with gold electrodes. Scheme 1 summarizes the preparation procedure for the TiO2@PZT/P(VDF-TrFE-CTFE) nanocomposites. The resulting nanocomposites were defined as TiO2@PZT nanowire array-1, TiO2@PZT nanowire array-2, and TiO2@PZT nanowire array-3, for the nanowire arrays prepared from 0.03, 0.04, and 0.06 mol L−1 Ti precursor solutions, respectively.

Characterization: The crystalline phases were characterized by XRD (Rigaku D-Max/2550VB+) utilizing Cu Kα radiation (λ = 1.5418 Å). The morphology and alignment of the nanowire arrays were observed by SEM (JSM-6390). TEM images of the samples were taken with a Titan 80-300, using an accelerating voltage of 300 kV. Gold electrodes with a thickness of ~20 nm were sputtered onto the nanocomposites (1.0 cm × 1.5 cm) using an eyelets mask with 2 mm diameter for electrical tests. The frequency dependence of permittivity and dielectric loss were performed using an Agilent 4294A LCR meter with a frequency range from 40 Hz to 10 MHz at room temperature. The permittivity was calculated from the measured capacitance using the relation \( \varepsilon_r = \frac{C \cdot A}{d \cdot \varepsilon_0} \), where \( \varepsilon_r \) is the permittivity of the capacitor, \( C \) is the capacitance (Farads), \( d \) is the thickness (m) of the samples, \( \varepsilon_0 \) is the permittivity of free space \( (8.854 \times 10^{-12} \text{ F m}^{-1}) \), and \( A \) is the surface area of the capacitor’s electrode (m²). The P–E loops of the nanocomposite were measured at 10 Hz at room temperature using a TF analyzer 2000 ferroelectric polarization tester (aixACT, Germany) and a Delta 9023 furnace in a silicone oil bath to avoid electrical discharges that would occur in air.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

ceramics, composites, dielectric materials, discharge energy density, nanowire arrays

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