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Fabrication of Barium Titanate/Acrylonitrile-Butadiene Styrene/Poly(methyl methacrylate) Nanocomposite Films for Hybrid Ferroelectric Capacitors

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A new organic–inorganic ferroelectric hybrid capacitor designed by uniformly incorporating surface modified monodisperse 15 nm ferroelectric BaTiO₃ nanocubes into non-polar polymer blends of Poly(methyl methacrylate) (PMMA) polymer and acrylonitrile-butadiene-styrene (ABS) terpolymer is described. The investigation of spatial distribution of nanofillers via a non-distractive thermal pulse method illustrates that the surface functionalization of nanocubes plays a key role in uniform distribution of charge polarization within polymer matrix. The discharged energy density of the nanocomposite with 30 vol. % BaTiO₃ nanocubes is ~44×10⁻³ J/cm³, which is almost six times higher than that of the neat polymer. The facile processing, along with the superior mechanical and electrical properties of the BaTiO₃/PMMA-ABS nanocomposites make them suitable for implementation into capacitive electrical energy storage devices.

Introduction

Inorganic ferroelectric materials with high dielectric constant and low dielectric loss values have attracted a considerable attention for the design of high energy density and multi-layer ceramic capacitors (MLCCs) for use in advanced technologies, such as portable electronic devices, electrical power systems, and hybrid electric vehicles.¹⁻² The large intrinsic dielectric polarization in ceramic ferroelectrics, such as barium titanate $BaTiO_3$ (BTO) was found to substantially improve the storage capacity (*i.e.* the released energy density) in capacitors. Although inorganic ferroelectrics demonstrate very high values of the dielectric constant (ε_r >1000), their use has been limited by the low breakdown strength, difficult processability and oftentimes incompatibility with flexible substrates.¹⁻⁴ At the other end of the spectrum, organic polymer-based insulators have low cost and present excellent mechanical properties for implementation into flexible electronics, but they possess relatively low dielectric permittivity values (ε =2-10) despite of characteristic high breakdown electric field values.⁵⁻⁶ One

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potential strategy for designing high performance flexible materials with high dielectric constant, high dielectric strength, and easy processability is the so-called organic/inorganic hybrid approach. This technique became popular in the past few years and consists of encasing ferroelectric ceramic fillers into a polymeric matrix.^{1,2,7-9} As such, several methods have recently developed for the fabrication of ferroelectric nanoparticles-based polymer composites. In most cases, the addition of nonfunctionalized inorganic nanoparticles to polymers is accompanied by the clustering and aggregation of the nanoparticles, phase separation and the formation of voids, which are detrimental to the dielectric properties of the nanocomposites as a result of the increase of dielectric loss and current leakage.^{8,9} The incompatibility between non-functionalized inorganic nanoparticles and the organic polymer can be primarily ascribed to the intrinsic high surface energy of the nanoparticles, which weakens drastically the adhesion in filler/polymer interface.⁹⁻¹¹ Two different approaches have proven to be promising to improve the strength of interfacial adhesion in biphasic polymer nanocomposites without deteriorating the macroscopic dielectric properties. These are: (i) preparing core-shell structures by connecting polymer chains to the surface of the nanoparticles¹⁰⁻¹³, and (ii) functionalizing the surface of the nanoparticles with capping agent molecules to render them compatible with the polarity of the polymer host matrix.^{1,2,14-17} Encapsulation of ferroelectric nanoparticles with organic molecules in core-shell architectures can improve the interfacial contact/interaction between the nanoparticles and the polymer. However, the relatively low free volume of the polymer matrix in such structures decreases the interspaces between individual nanoparticles, thereby leading to a strong cooperative coupling between the electrical dipoles of individual ferroelectric nanoparticles and the enhancement of the hysteresis loss upon charging the capacitor.^{2,11-13} Recently, several groups reported an in-situ polymerization method to design core-shell structured nanocomposites using 100 nm BaTiO₃ nanoparticles with poly(methyl methacrylate) (PMMA) and hyperbranched polymers (HBPs). In all cases, nanocomposites were prepared by compacting a mixture of core-shell nanoparticles and the polymer matrix into disk films at high temperature, which yielded densely packed interfacial regions at nanoparticles.^{10,13}

From this viewpoint, the treatment of core-shell ferroelectric nanocomposites under both high pressure and temperature will not only decrease the efficiency of the capacitor by increasing the remnant polarization but will also weaken the mechanical performance of the resulting flexible nanocomposite films. Surface functionalization with coupling agents is an alternate route to prevent the aggregation of nanoparticles in the polymer host matrix. However, most studies have been performed on stabilizing dielectric nanoparticles in polar organic matrices (*e.g.* fluoro-polymers), which are characterized by high remanent polarization values despite the fact that this can limit the released energy density of the capacitors.^{1,4,9,14-16} So far, little effort has been focused on designing a viable surface modification strategy to render ferroelectric nanoparticles highly dispersible in non-polar polymers. Herein we report on a solution-processable approach to fabricate hybrid ferroelectric capacitors using surface-modified cuboidal BaTiO₃ nanocrystals which have been uniformly dispersed into a non-polar polymeric matrix. Furthermore, we report for the first time a non-distructive technique using thermal pulses for mapping the polarization distribution of inorganic nanofillers incorporated into the hybrid ferroelectric capacitors. Our findings in this work strongly suggested that the superior dielectric performance of these capacitors is associated with the uniform charge polarization due to the existence of BTO nanocubes as capacitive building blocks in host polymer.

Experimental

Oleic acid-capped BaTiO₃ nanocubes have been prepared following a previously reported low temperature method under hydrothermal conditions.¹⁸ The as-prepared nanocrystals were subsequently dispersed in toluene and the PMMA-ABS polymer was dissolved into the toluene solution of BTO nanocubes (10:1 by weight). The resulting mixture was directly spin cast onto various substrates thereby yielding BTO /PMMA-ABS nanocomposites films after drying in air atmosphere. The morphology and surface structure of BTO nanocubes and nanocomposite films were investigated by transmission electron microscopy (TEM) with a JEOL-2010 microscope, scanning electron microscopy (SEM) with a JEOL JSM-7500F, and Atomic Force Microscope (AFM) with an Asylum Research MFP-3D (AC240TS-R3 cantilever). Fourier-transform infrared (FTIR) spectroscopy experiments were performed at room temperature with a Thermo Scientific Nicolet 380 spectrometer. The mechanical properties of nanocomposite films were studied using Dynamic Mechanical Analysis (DMA) with a TA Q800 instrument. Two-terminal ferroelectric capacitors were fabricated in parallel-plate geometry by using 20 µm-thick nanocomposite films followed by thermal deposition of opaque AI electrodes (thickness: 50 nm). The ferroelectric and dielectric properties were measured using a Premier II tester (Radiant Technologies, Inc.), and an Agilent 4294A impedance analyser.

Results and Discussion

Figure 1 illustrates a schematic of the chemical structures of the nanocomposite and the design of the hybrid ferroelectric capacitor. The as-synthesized BaTiO₃ nanocubes (BTO NCs) are stable in non-polar media due to the retention of oleic acid (OA) molecules on their surface, which renders the colloidal solution of BTO nanocubes miscible with polymers having a similar polarity.



Fig. 1 Schematic of BTO NCs/PMMA-ABS nanocomposite capacitor.

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As it can be observed in the TEM micrograph of BTO nanocubes (Figure 2a), 15 nm cuboidal shape nanoparticles are separated with a distinctive interparticle distance, indicating the retention of carboxylic acid molecules on the surface of colloidal nanocrystals. A high degree of monodispersity and proper surface functionalization of the nanocubes provide strong spatial interaction at the filler/polymer interface, thereby enhancing the homogenous dispersion of BTO nanocubes into the continuous non-polar matrix. It is important to use a polymer matrix with superior mechanical properties and low energy losses to produce practical nanocomposite capacitors, which are scalable for flexible and lightweight electronic applications. To address this need, polymer blends of Poly(methyl methacrylate) (PMMA) polymer and acrylonitrile-butadiene-styrene (ABS) terpolymer have been used as host polymer matrix. The composition of ABS comprising styrene-acrylonitrile copolymer (SAN) matrix and a dispersion of rubbery butadiene render this thermoplastic as an excellent toughening agent for fabrication of highly flexible BTO NCs/PMMA-ABS hybrid films¹⁹.

The surface functionalization of the BTO nanoparticles with the oleic acid ligand was evidenced by FTIR spectroscopy and the results are shown in Figure 2b. The retention of oleic acid molecules to the surface of BTO nanoparticles in both the colloidal solution (BTO nanocubes) and the polymer matrix (BTO NCs/PMMA-ABS) is confirmed by the shift of the band at 1710 cm⁻¹, associated with the C=O stretching mode of the free carboxylic group, to 1560 cm⁻¹. Moreover, the band observed around 1730 cm⁻¹ ascribed to the C=O vibration in PMMA molecules and which is detectable in the spectrum of neat PMMA-ABS films was also observed in the case of BTO NCs/PMMA-ABS nanocomposite films.



Fig. 2 (a) TEM image of the 15 nm BTO nanocubes; (b) FTIR spectrum of pure oleic acid, pure PMMA, oleic acid-capped BTO nanoparticles, and 30% volume fraction of oleic acid-capped BTO nanocubes dispersed in a PMMA-ABS matrix.

The surface SEM image collected from a polymer-ceramic nanocomposite film containing 30% volume fraction of BTO nanocubes shows that the fillers are uniformly distributed within the PMMA-ABS host matrix (Figure 3a). The optical image of a typical bent BTO NCs/PMMA-ABS hybrid film shown in Figure 3a (inset) indicates that films are uniform over their entire surface and present a good mechanical flexibility. The effects of the addition of ABS polymer on mechanical properties of nanocomposite films were investigated and typical stress–strain responses under a constant loading rate of 0.22 Mpa/s can be seen in Figure 3b. It is clearly revealed that the Young's modulus decreases from 1060 Mpa and 1520 Mpa for PMMA and BTO NCs/PMMA films to 590 Mpa and 890 Mpa for PMMA-ABS and BTO NCs/PMMA-ABS films, respectively, thereby confirming the increased flexibility of the films containing the ABS elastomer. Anotherkey requirement for composite

materials is the attainment of toughness; that is the total energy needed to fracture the material. This parameter depends on both the strength and the failure strain of the material. Our experiments revealed that the measured toughness increases significantly upon adding ABS to PMMA based films and the highest measured toughness value corresponds to BTO NCs/PMMA-ABS films (187.82 × 10⁴ Jm⁻³), 62% higher than that of the BTO NCs/PMMA films (69.8× 10⁴ Jm⁻³). The topography of BTO NCs/PMMA-ABS nanocomposite was studied with atomic force microscopy and the corresponding AFM image is shown in Figure 3c. The results indicate the surface of the nanocomposite film is smooth with a root-mean-square roughness (R_{rms}) value of ~5 nm for a scanned area of the sample of 1 μ m².



Fig. 3 (a) Top view SEM image of a NCS/PMMA-ABS nanocomposite film with 30% filler nanoparticles (inset: photograph of the flexible nanocomposite film); (b) Tensile stress–strain curves of PMMA, PMMA-ABS, 30% BTO NCs/PMMA, and 30% BTO NCs/PMMA-ABS films; (c) 3D view AFM of 30% BTO NCs/PMMA-ABS films.

The space charge and spatial distribution of the electrical polarization with depth resolution in BTO NCs/PMMA-ABS and pristine PMMA-ABS capacitors have been investigated by using a thermal-pulse setup²⁰ displayed, as shown in Figure 4a. The electric field (*E*), was varied from 0 to 20 MV/m, being applied to the samples via a current-limiting 10 M Ω resistor and a 3.3 nF capacitor in order to isolate the current amplifier in the detection circuit form the high voltage.²¹ A light pulse from a Nd:YAG laser (duration 5 ns, λ = 532 nm) is absorbed by the opaque front electrode, thereby leading to the non-uniform heating of the sample. As the thermal pulse diffuses through the ferroelectric material, it generates a transient pyroelectric current from which the electrical polarization can be calculated.²² The measured transient current was Fourier-transformed into the frequency domain. The difference between the real and the imaginary components of the frequency-domain current is proportional to electrical polarization at depth *z*, described by the following relationship:²³

 $P(z) \propto [\operatorname{Re}(I(f)) - \operatorname{Im}(I(f))]$ (1)

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where the frequency *f* is related to the depth via $z = (D/\pi f)^{0.5}$ and *D* stands for the thermal diffusivity of the sample, which can be deduced form the thermal conductivity (*k*), density (ρ) and specific heat (*c*), by using Equation (2):²³

 $D=k(c\rho) \tag{2}$

The effective thermal diffusivity of BTO NCs/PMMA-ABS nanocomposite and pristine PMMA-ABS was estimated to be to the values of 4.3×10^{-7} m²/s and 9.8×10^{-8} m²/s, respectively by using the rule of mixture²⁴. As shown in Figure 4 b, the homogeneous distribution of the polarization charge across the large part of the BTO NCs/PMMA-ABS nanocomposite confirms the excellent dispersion of the ferroelectric nanodomains associated with the BaTiO₃ nanoparticles into the polymer matrix. However, at a depth of 1 µm in the nanocomposite, the polarization maps reveal a substantial edge depolarization, which could be result of decreased concentration of BTO nanocubes near the top surface. Furthermore, compared with the polarization depth profiles of the PMMA-ABS matrix, the nanocomposites suggests the existence of an polarization, which is enhanced progressively upon applying the applied electric field. These results indicate the presence of a strong charge injection in the composites due to the existence of the high capacitance single-domain ferroelectric BTO nanocubes across the polymer phase.



Fig. 4 (a) Experimental setup for thermal-pulse measurements with a supplemental applied electric field circuit; (b) Polarization depth profile of 30% BTO NCs/PMMA-ABS and pristine PMMA-ABS capacitors as a function of the electric field.

The ferroelectric response, energy density values and dielectric properties of the BTO NCs/PMMA-ABS nanocomposite films, in which the volume fraction of the BTO nanocubes was varied from 0 to 30%, were analysed in order to investigate

systematically the performance of the ferroelectric hybrid capacitors. Figure 5a shows the ferroelectric polarization versus the electric field (*P vs. E*) loops of each sample. This experimental finding clearly demonstrates that the polarization of the PMMA-ABS polymer can be substantially improved upon dispersing BTO nanocubes into the polymer matrix, thereby making the BTO NCs/PMMA-ABS nanocomposite films suitable for implementation in high energy density capacitors. For instance, the 30% BTO NCs/PMMA-ABS nanocomposite capacitor presents an almost six-fold increase of the maximum polarization $(4.8 \times 10^{-3} \text{ C/m}^2)$ compared to that measured for the neat PMMA-ABS capacitor $(8 \times 10^{-4} \text{ C/m}^2)$ at the electric field of 20 MV/m.



Fig. 5 (a) P-E characteristics for 0-30% BTO NCs/PMMA-ABS nanocomposite capacitors; (b) Variation of the released energy density of the nanocomposite capacitors as a function of volume fraction of BTO nanocubes.

A similar increment (almost six-fold) of maximum polarization was observed in thermal-pulse experiment for PMMA-ABS films after adding 30% volume fraction of BTO nanocubes, which is in excellent agreement with the results obtained from both polarization depth profile and ferroelectric loops. In addition, all nanocomposite capacitors present demonstrate narrow *P* vs. *E* loops (*i.e.*, the area enclosed by hysteresis loops), thereby indicating a low ferroelectric hysteresis loss. Such ferroelectric characteristics of the capacitors originate from the nonlinear dielectric response and polarization switching within monodomain BaTiO₃ nanocubes, which are uniformly encased into the PMMA-ABS polymer matrix. The energy density recoverable from a dielectric material can be described by the integral $U_e = \int EdD$, where *D* stands for the electric displacement and is defined by $D = \varepsilon_0 E + P$, where $\varepsilon_0 = 8.85 \times 10^{-12}$ F/m represents the vacuum permittivity.⁹ The discharged energy values of the capacitors were calculated using the ferroelectric loops and the results can be seen in Figure 5b as a function of BTO nanocubes. Therefore, the nanocomposite with 30% volume ratio of BTO nanocubes discharges an U_e of ~44×10⁻³ J/cm³ at 20 MV/m, representing a 5.5× enhancement of the released energy density of the PMMA-ABS film, whose value is ~8×10⁻³ J/cm³ at 20 MV/m.

Electrical impedance spectroscopy (EIS) measurements were performed at frequencies from 100 Hz to 1 MHz at 1 V_{rms}. The real and imaginary components of the impedance (Z=Z'-iZ'') were collected and the Nyquist plot (Z'' vs. Z') is shown in Figure 6a. The vertical straight trend illustrates the ideal capacitive behavior of ferroelectric capacitors presenting different concentrations of the BTO nanocubes; thus, a resistor–capacitor circuit (RC circuit) can be employed as a model system to analyse the dielectric properties of the BTO NCs/PMMA-ABS hybrid capacitors. The complex capacitance (C = C' - iC'') is given by the following relationship²⁵:

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$$C=(i2\pi fZ)^{-1}$$

From the real component of complex capacitance (*C'*), the permittivity of the dielectric material with a thickness *I* in parallel plate capacitor geometry with an electrode area *A* is obtained from the relationship: $\varepsilon_r = C' / \varepsilon_0 A$. The dielectric loss tangent (tan δ) provides further insight into the dielectric characteristics illustrates the dissipated electrical energy in capacitor. The imaginary part of the capacitance (*C'*) can be used to quantify the dielectric loss according the following relationship²⁵:

$$\tan \delta = C''/C' \tag{4}$$

As seen in Figure 6b, the dielectric permittivity values in the broadband frequency from 1 kHz to 1 MHz increase with increasing the concentration of BTO nanoparticles filler.



Fig. 6 (a) Nyquist plot, (b)-(c) frequency dependence of the dielectric constant and the loss tangent; (d) dielectric permittivity and loss tangent at 1 kHz as a function of BTO volume fraction.

The dielectric loss tan δ of the BTO NCs/PMMA-ABS hybrid capacitors (figure 6c) increases by adding BTO NC, which is presumably due to the existence of an ohmic current leakage through the nanofillers. At the fixed frequency of 1 kHz, both ε_r and tan δ reach their highest values (ε_r =28, tan δ =0.03) for 30% BTO NC content, thereby suggesting that such organic/inorganic hybrid films can be used in the design of ferroelectric capacitors for various applications in energy conversion/storage.

Conclusions

In summary, ferroelectric capacitors have been designed by a simple method, which uses surface modified barium titanate nanocrystals dispersed into a non-polar PMMA-ABS polymer matrix. A thermal-pulse setup for mapping the polarization distribution with depth resolution was used to study the electrical characteristics of the polymer-ceramic nanocomposite films and the results confirm that nanocrystals function as capacitive building blocks in BTO NCs/PMMA-ABS capacitors. The nanocomposite film capacitor containing 30% volume fraction of BTO nanocubes shows the enhanced energy density of ~44×10⁻³ J/cm³ at 20 MV/m compared to that measured for neat polymer film along with a relatively low hysteresis and dielectric losses. The proper electrical and mechanical properties of such capacitors due to the existence of OA capped-BTO nanocubes and ABS elastomer, respectively, highlight the great potential of employing highly dispersed surface modified nanocrystals into anon-polar polymer matrix for applications in high energy density and the design of low loss ferroelectric capacitors.

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