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Graphene oxide-tailored dispersion of hybrid barium titanate@polypyrrole particles and the dielectric composites

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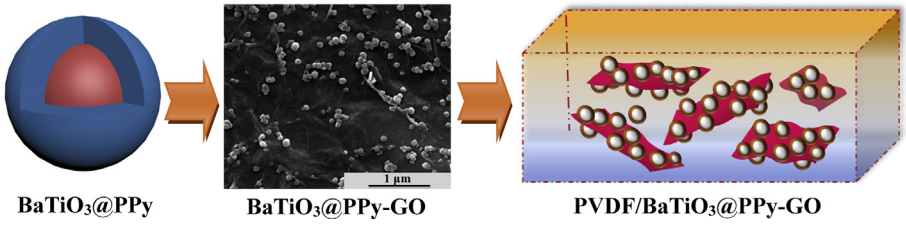
HIGHLIGHTS

- The 'core–shell'-like $BaTiO_3$@PPy particles were deposited on the GO platelets.
- GO-tailored $BaTiO_3$@PPy particles dispersion were achieved in the PVDF composites.
- PVDF/$BaTiO_3$@PPy-GO composites had low dielectric loss and high breakdown strength.
- The mechanism was attributed to the GO-induced aggregation of $BaTiO_3$@PPy particles.

GRAPHICAL ABSTRACT



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ABSTRACT

Dielectric composites with low dielectric loss recently attract increasing concerns of researchers due to the great potential applications in microelectronic devices. In this work, novel hybrid particles, which were composed of barium titanate ($BaTiO_3$), polypyrrole (PPy) and graphene oxide (GO), were prepared through the *in situ* polymerization of PPy on the surface of $BaTiO_3$ particles and the deposition of $BaTiO_3$@PPy particles on the surface of the GO platelets. Morphology and microstructure characterizations showed that the 'core–shell'-like $BaTiO_3$@PPy particles were prepared and these particles exhibited homogeneous dispersion on the GO platelets. Then, two kinds of poly(vinylidene fluoride) (PVDF)-based composites, including PVDF/$BaTiO_3$@PPy and PVDF/$BaTiO_3$@PPy-GO, were fabricated. Different from the relatively homogenous dispersion of $BaTiO_3$@PPy particles in the whole PVDF/$BaTiO_3$@PPy composites, the $BaTiO_3$@PPy-GO particles exhibited slight aggregation in the PVDF/$BaTiO_3$@PPy-GO composites due to the GO-induced local aggregation of $BaTiO_3$@PPy particles. The two kinds of particles exhibited good nucleation effect and mainly promoted the crystallization of α-form PVDF. Electrical conductivity and dielectric property measurements showed that the PVDF/$BaTiO_3$@PPy-GO composites had lower electrical conductivity, smaller dielectric loss but higher breakdown strength compared with the PVDF/$BaTiO_3$@PPy composites. The mechanism was mainly attributed to the presence of GO platelets, which restricted the formation of the conductive path in the composites.

1. Introduction

With the fast development of microelectronic devices and the tremendous requirement for green energy, theories and technologies relating to energy storage and comprehensive utilization attract increasing concerns of researchers either from academia or from industry. Dielectric materials which exhibit the ability to store and release energy under the electrical condition is one of the most important components

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in the energy devices. Dielectric constant, dielectric loss and dielectric breakdown strength are those of the most important parameters relating to the dielectric properties of the dielectric materials, which greatly determine the energy storage efficiency and the service behavior of the dielectric materials [1,2]. The ideal dielectric materials are expected to simultaneously have high dielectric constant, low dielectric loss and high dielectric breakdown strength.

Compared with the ceramic dielectric materials, polymeric dielectric materials have apparent advantages, such as light weight, easy processing, good comprehensive properties, etc. However, it is worth noting that most of polymers exhibit rather low dielectric constant, which is much smaller than that of the ceramic dielectric materials [3]. Incorporating other dielectric materials into polymers has been demonstrated an efficient strategy to improve the dielectric properties of polymers. Generally, polymeric composites containing ceramic materials, such as barium titanate ($BaTiO_3$) [4–8], α-carborundum (α-SiC) [9] and lead zirconate titanate (PZT) [10,11], etc., exhibit high dielectric constant and low dielectric loss. But it is worth noting that the acquirement of the high dielectric constant can be only achieved at relatively high filler content. Undoubtedly, high filler content not only results in the deterioration of the processing ability but also results in the deterioration of the comprehensive mechanical properties of the composites [9].

Incorporating conductive fillers, such as electrically conductive polymers [12–15], carbon nanotubes (CNTs) [16–18], graphene [19–21], etc. is the most efficient way to enhance the dielectric constant of polymeric composites at relatively low filler content. However, the shortage of the conductive filler-incorporated polymeric composites is also very apparent. Namely, these dielectric composites usually exhibit high dielectric loss due to the occurrence of the leakage current in the electric field, which is mainly attributed to the network structure of the conductive fillers formed at filler content that is higher than the percolation threshold of filler [22]. Reducing the content of conductive fillers as much as possible or preventing the formation of the conductive network structure in the composites may suppress the dielectric loss at a low level.

Recently, the method of incorporating two kinds of fillers into one polymer and constructing the hybrid structure in the composites to enhance the dielectric constant and simultaneously suppress the dielectric loss of the composites attracts the great attention of researchers [23–25]. At least, there are two kinds of approaches that have been proposed. One approach is simultaneously incorporating ceramic filler and conductive filler into one polymer. In this scenario, it is expected that the presence of ceramic filler with an insulated feature prevents the formation of the conductive network structure of the conductive fillers in the whole composite [26]. For example, Guan et al. [27] introduced CNTs and $BaTiO_3$ into polydimethylsiloxane (PDMS) and they found that the hydrogen bonding interaction occurred between CNTs with carboxyl groups and hydroxyl groups of $BaTiO_3$ facilitated the formation of the hybrid structure and consequently, the composites exhibited high dielectric constant and low dielectric loss simultaneously. In our previous work [28], boron nitride (BN) and CNTs were simultaneously incorporated into poly(vinylidene fluoride) (PVDF) and the results showed that the presence of BN induced the formation of CNT network structure only in a local region rather than in the whole composite and consequently, the composite also exhibited high dielectric constant and low dielectric loss.

The other kind of approaches are preparing the polymeric composites containing the hybrid particles with a 'core–shell'-like structure, which are fabricated through coating/depositing the conductive polymers on the surface of the insulated ceramic fillers [29,30] or encapsulating the conductive polymers or conductive fillers (core) by insulating fillers or other polymers (shell) [12,13,23,31–35]. For the two scenarios, the largely enhanced dielectric constants are usually attributed to the construction of the multiple interfaces in the composites, which provide more probability for the interfacial polarization [30,36,37]. Obviously, from a viewpoint of suppressing the dielectric loss, the hybrid 'core–shell'-like structures with the insulated shell are more appropriate because these structures greatly reduce the probability of forming the electrically conductive path in the composites even if these fillers contact each other and form the percolated network structure [25,32]. But for the hybrid 'core-shell'-like particles with electrically conductive shell, the composites still exhibit high dielectric loss since the aggregation and/or contact of adjacent particles still provides the conductive path in the composites. Therefore, for this kind of hybrid particles, more work is still required to seek appropriate method that can suppress the formation of the conductive path in the composites.

Furthermore, recent researches have also demonstrated that depositing dielectric materials on the two-dimensional graphene oxide (GO) platelets is also an efficient strategy to fabricate dielectric composites with high dielectric constant and low dielectric loss, and the deposited dielectric materials are various, such as ceramic fillers [38], conductive fillers [39,40] or conductive polymers [41–43], etc. On the one hand, GO has many oxygen-containing groups on the edges of the platelets and it can exhibit relatively strong interfacial interaction with many polymers, which is favorable for the interfacial polarization between GO and polymer matrix [44–47]. On the other hand, GO may improve the dispersion of the dielectric materials in the polymeric composites [48], which is believed to be favorable for the suppression of the dielectric loss of the composites.

In this work, we attempt to fabricate hybrid particles with 'core–shell'-like structure, in which the conductive polypyrrole (PPy) is *in situ* synthesized on the surface of the $BaTiO_3$ particles. Then, the hybrid $BaTiO_3$@PPy particles are further deposited on the GO platelets. The composite particles are further incorporated into PVDF through solution compounding processing, and the microstructure-performance relations of the PVDF composites are systematically investigated. It is interesting to observe that compared with the composites containing $BaTiO_3$@PPy particles (PVDF/$BaTiO_3$@PPy), the PVDF/$BaTiO_3$@PPy-GO composites exhibit largely suppressed dielectric loss and simultaneously enhanced dielectric breakdown strength, which provides more probability for the application of such composites in the microelectronic devices relating to energy storage and release.

2. Experimental part

2.1. Materials

PVDF (droplets, Kynar 720) with a weight average molecular weight of 1.05×10^5 g/mol and a density of 1.78 g/cm^3 was obtained from Arkema (France). $BaTiO_3$ (white powder) with an average diameter smaller than 100 nm and a purity of 99.9% was obtained from Aladdin Reagent Co. (Shanghai, China). Natural graphite which was obtained from Qingdao Heilong Ltd. Co. (China) was used to prepare the GO through the modified Hummers method and the detailed preparation process can be seen in our previous work [49]. Pyrrole monomer with the purity above 98% and Ferric trichloride ($FeCl_3$) with the purity above 98% were obtained from Kelong Chemical reagent Co. Ltd. (Chengdu, China).

2.2. Preparation of $BaTiO_3$@PPy and $BaTiO_3$@PPy-GO particles

The preparation procedures of hybrid particles were schematically represented in Fig. 1. To prepare the hybrid $BaTiO_3$@PPy particles, 30 g $BaTiO_3$ particles and 20 mL pyrrole were first added into the solution of 480 mL distilled water and 31.1 mL HCl and the mixture was stirred for 30 min (Fig. 1a). After that, 36.23 g $FeCl_3$ solution with a concentration of 0.7246 g/mL was dropped into the pyrrole/$BaTiO_3$ solution and the polymerization of PPy was initiated accompanied with the continuous change of the solution color from white to black (Fig. 1b). After being continuously stirred for 5 h, the solution was filtered. The obtained

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