



Study on material performances of lead zirconate titanate/shape memory polyurethane composites combining shape memory and piezoelectric effect



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ABSTRACT

A class of functional composites are compounded by piezoelectric particles and shape memory polymers, and the resultant composites perform both shape memory effect and piezoelectric effect. Although many kinds of piezoelectric composites have been developed, few papers report the effect of interfacial bonding between fillers and matrices on piezoelectric performances. Here, two groups of samples are investigated to clarify the role of interfaces in piezoelectric performances and permittivity properties. The experimental results testify that the samples with poor interfaces exhibit lower relative permittivity and are unable to show usable piezoelectric effect. The piezoelectric effect critically depends on interfacial bonding. Compared with pristine shape memory polymers, interfaces efficiently improve thermodynamics but influence the glass transition temperature very little. Moreover, shape recovery rates all reach at least 95% in the third cycle, even though decrease with the addition of fillers because of increasing interfaces.

1. Introduction

Up to now, composites have been applied in a number of fields such as aerospace [1], automotive industry [2], biomedicine [3] and energy harvesting [4] due to the attractive material performances benefit from the combinations of fillers and matrices. Composites are able to inherit material performances from fillers and matrices, and even create some new kinds of material properties. Therefore, various fillers and matrices are employed to reinforce materials performances, overcome the material shortcomings and produce new material function. Polymers (polyurethane [5] and polyvinylidene fluoride [6,7]) are used to prepare flexible lead zirconate titanate (PZT) composites with the purpose of surmounting the brittleness of PZT ceramics. Conductive fillers, carbon nanotubes (CNTs) [8–10] and nanographite [11] are added into PZT polymeric composites for the enhancement of permittivity and conductivity. Silica particles are brought in shape memory composites to improve the mechanical performance [12], carbon nanofibers (CNFs) are employed to lessen the time of shape recovery [13], and silk fibroins are utilized to increase the recovery rates [14]. Shape memory composites could be transformed from thermal actuation to electric actuation by conductive fillers (carbon black [15], CNFs [16] and carbon nanopaper [17]). CNTs and graphene are capable of endowing shape memory composites with not only electric actuation [18–21] but also

remote actuation [22,23]. Besides, shape memory composites containing magnetic particles could be actuated by electromagnetic field [24,25]. So far, researchers have done a lot of work on PZT composites and shape memory composites and some literatures has reported the PZT/shape memory alloy (SMA) laminated composites [26,27], but a kind of composite synthesized by PZT particles and shape memory polymer is hardly reported. Although a lot of literatures have been reported on the factors (PZT content, polarization conditions and permittivity) of PZT composites and different models [28,29] for calculating permittivity and the piezoelectric constant, whereas there are very few studies on material performances of the composites with poor interfaces. As for PZT/SMA laminated composites, deformations and flexibility of SMAs are much less than shape memory polymers.

Here, we propose a class of smart composites consisted of PZT particles (average diameter is 400 nm) and shape memory polyurethane (SMPU). Three kinds of PZT/SMPU composites, PZT 60% (wt%), PZT 70% (wt%) and PZT 80% (wt%), are prepared with both shape memory and piezoelectric effect. The direct current (DC) relative permittivity and nanoscale displacements of PZT/SMPU composites are studied to clarify the effect of interfaces on piezoelectric effect and dielectric property. The results of dynamic mechanical analysis (DMA) reveal the improvement in storage moduli and very little variation in glass transition temperature (T_g).

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2. Materials and methods

2.1. Materials

PZT/SMPU composites were synthesized by PZT particles (DPZ-LQ2-P6, Dai Nippon Toryo Co., Ltd., Japan) and a kind of commercial shape memory polyurethane (MM6520, SMPU Technologies Inc., Japan). First, PZT particles with an average diameter of 400 nm were poured into tetrahydrofuran (THF) and the PZT/THF mixture was dispersed for 15 min at the level 3 by an ultrasonic device (Sonifier 250, Branson Ultrasonics Corp., U.S.A.). Then, shape memory polyurethane pellets (the weight was one seventh of THF) with the glass transition of 65 °C were dissolved in the PZT/THF mixture and a magnetic stirrer was employed to blend for 48 h at a speed of 400 r/min. Next, THF evaporated naturally when the mixture was still stirred until the weight ratio of shape memory polyurethane and THF became 1:5. After the mixture was put statically for 48 h to remove air bubbles, it was casted on a Teflon film with an auto film applicator (PI-1210, Tester Sangyo Co., Ltd., Japan). The resultant films cured after evaporating THF again for 3 days in nature, and then were further dried at 65 °C for 3 days. Finally, the resultant films were divided into two groups. The first group members were pressed for 8 h under the pressure of 6 MPa at 80 °C by a hot-press machine (SA-303, Tester Sangyo Co., Ltd., Japan), while the second group members were not hot-pressed.

2.2. Polarization

P_b-P_t alloy electrodes were sputtered (E-1030, Hitachi Co., Ltd, Japan) on the surfaces of PZT/SMPU composite films. Then the films were polarized at 80 °C for 2 h in a silicone oil bath under the electric field of 10 kV/mm supplied by a high voltage power supply (ES30, Gamma High Voltage Research Inc., U.S.A.).

2.3. Nanoscale displacements

The displacements were measured by a laser interferometer (SI-F01, Keyence Corp., Japan) with a moving average filter (the mean of 256 points). The applied step voltage signals were generated by a high voltage power supply (HJPQ-30P1, Matsusada Precision Inc., Japan).

2.4. Shape recovery rates

The shape recovery rates of PZT/SMPU composites were all measured by a thermo-mechanical analyzer (TMA/SS6100, Hitachi High-Tech Science Corp., Japan). Rectangle samples (the width of 2 mm, length of 10 mm and thickness between 127 and 150 μm) were tested for 3 cycles in the controlled force mode. Before the cyclic tests, the samples were heated from ambient temperature up to 71 °C with a stress of 0.025 MPa to protect samples from buckling. One experimental cycle was that firstly the stress was increased to 3.5 MPa at a speed of 0.139 MPa/min, then a fan was employed to quickly cool the samples down with the stress of 3.5 MPa, next the stress was unloaded from 3.5 to 0.025 MPa at a speed of - 0.695 MPa/min and finally the samples were heated up to 71 °C again while the stress was kept at 0.025 MPa. The strain of every sample was the quotient of the continuous displacement recorded by TMA device and its length.

2.5. Microscopic morphology

The microscopic images of cross-sections of the PZT/SMPU composites were taken by a field emission scanning electron microscopy (FE-SEM) (S-5000, HITACHI CO., Ltd., Japan). The samples were all broken off in liquid nitrogen, and FE-SEM images showed the cryo-fractured surfaces.

The DMA of pristine SMPU and hot-pressed PZT/SMPU composites was performed on a DVA-225 instrument (IT Keisoku Seigy Co., Ltd.,

Japan). The rectangular samples with the width of 2 mm, length of 10 mm and thickness from 100 to 175 μm were tested under force track 200%, 10 Hz, and 5 °C/min (from 30 °C to 100 °C).

2.6. Relative permittivity

Circular samples with the diameter of 10 mm were measured by a SI 1260 Impedance/Gain-Phase Analyzer (AMETEK Inc., U.S.A.) and 1296A Dielectric Interface System (AMETEK Inc., U.S.A.). The DC voltages (averagely divided into 30 points) from 100 to 500 V were supplied by a high voltage power supply system (High Voltage Interface HVI-1000, TOYO Corp., Japan and Bipolar Operational Power Supply/Amplifier Model BOP 1000M, TOYO Corp., Japan).

3. Results and discussion

3.1. Microscopic morphology of PZT/SMPU composites

The interfaces (the interfacial interactions between fillers and matrices) play a very important role in the material performances of composites [30]. Fig. 1 shows the interfaces between PZT particles and SMPU matrices in visual. Fig. 1a–c illustrates the microstructures of composites in the first group whose group members (PZT 60%-HP, PZT 70%-HP and PZT 80%-HP) are all hot-pressed. The PZT particles are surrounded tightly by the SMPU matrices, and no void or free space is found. Fig. 1d–f proves the poor interfaces of composites in the second group whose group members (PZT 60%-N, PZT 70%-N and PZT 80%-N) are all not hot-pressed. Free spaces exist between the PZT particles and SMPU matrices. According to the mechanisms of polymeric film formation, polymer chains would interpenetrate, go through a gel state and then form a film as the solvent evaporates [31]. However, with the evaporation of solvent, the polymer chains in composites would aggregate together and form the polymer phase by the polymer chain interpenetration, while the spaces of solvent molecules left by evaporation would be transferred to the regions between the fillers and matrices. In pristine polymeric films, all polymer chains would get together to form a film by the polymer chain interpenetration and the spaces left by solvent molecules would be removed to the outside of polymer chains. It is similar in polymeric composites, but the difference is that the outside of polymer chains represents the regions between fillers and polymer matrices. That is the main reason for the free spaces.

Whereas, these free spaces can be eliminated by hot-pressing, because SMPU matrices are viscous fluids at the temperature above T_g . If the applied pressure is enough, SMPU matrices would fill in the free spaces due to the high mobility at the temperature above T_g and embrace PZT particles tightly.

3.2. Thermo dynamics

Fig. 2 is the results of DMA performed on pristine SMPU, PZT 60%-HP, PZT 70%-HP and PZT 80%-HP. Fig. 2a shows that the storage moduli increase with the amount of PZT particles in PZT/SMPU composites, and the increment of storage moduli becomes stronger when the temperature goes up. This improvement in storage moduli, especially the impressive increase at the temperature above T_g , is another evidence for good interfaces between PZT particles and SMPU matrices. Because the molecular chains of SMPU would deform freely at the temperature above T_g (like viscous fluid), storage moduli must result from strong interfacial bonding. A similar enhancement is also discovered in the loss moduli, because interfaces have effect on both elastic part and viscous part. The storage moduli and loss moduli respectively represent elastic part (stored energy) and viscous part (energy lost as heat) [32]. Besides, the data of DMA also exhibit the small variations of T_g . Usually, the temperature of $\tan \delta_{\max}$ (the maximum value of the quotient of storage moduli by loss moduli) and the maximum value of loss modulus is defined as T_g [33,34], although the glass

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