



A photo-sensitive piezoelectric composite material of poly(vinylidene fluoride-trifluoroethylene) and titanium oxide phthalocyanine



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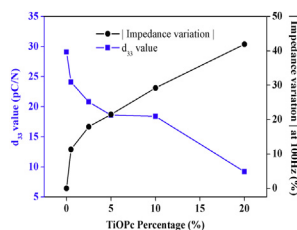
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HIGHLIGHTS

- Our light-sensitive piezoelectric material is a composite of P(VDF-TrFE) and TiOPc.
- The added TiOPc does not affect the P(VDF-TrFE) crystallization.
- Our composite material possesses a high broad sensitivity to visible light.
- The impedance of the composite decreases significantly with light illumination.
- An optimal 10% TiOPc gives the best visible spectrum and piezoelectric effect.

GRAPHICAL ABSTRACT



The optimal composition of the TiOPc to P(VDF-TrFE) is 10wt% for both good piezoelectric and photo-electric response.

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ABSTRACT

Photo-driven materials are an emerging research field due to its real-time definable capabilities and wide ranging potential applications. Previous research in the field has been limited to opto-electrical interactions. In this study, we investigated an opto-mechanical composite material of poly(vinylidene fluoride-trifluoroethylene) [P(VDF-TrFE)] and titanium oxide phthalocyanine (TiOPc). The characteristics of this developed composite material were examined using SEM, FTIR, and XRD analytical methods. Our results showed that a remnant polarization and coercive field can be increased with a proportional increase of TiOPc in the composite. Experimental results show that the optimal composition of the TiOPc to P(VDF-TrFE) was 10 wt% ($d_{33} = 18$ pC/N, 30% of impedance variation at 100 Hz). Our developed composite material has good potential as an opto-configurable mechanism for various applications.

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1. Introduction

In recent years, mechanical manipulation has become more widely developed in the bioengineering and microsystem fields to control cells and molecules [1]. The most powerful and common techniques include atomic force microscopy (AFM), magnetic tweezers and optical tweezers [2]. AFM probes can provide

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nanometer spatial resolution with a pN force resolution and has the ability to scan surfaces and measure the force curve of a target. However, the minimal force of 10 pN is too large, and it cannot provide 3D or long distance control [3]. A magnetic tweezer is a tool that can provide force from fN to mN. Its typical application is for cell manipulation and DNA topology [4,5]. Unfortunately, it is characterized with a lower time and lower spatial resolution for video-microscopy data collection [6]. In the 1970s, an optical tweezer was proposed for particle manipulation. It provided 3D manipulation with a millisecond response for particle transportation, ablation, and even controlling electron spin behavior [7–10]. Its major drawback was it generated heat and had the potential to cause sample damage from the high-power laser. Other indirect optical control techniques such as optothermal capillary and optoelectronic tweezers were also widely adopted for optofluidic manipulation [11,12].

Starting from the 1980s, beyond direct optical forces, an optothermal mechanism was found to provide a much larger force to allow for faster droplet manipulation (cm/s) [13]. However, the high-power pulse laser also generated undesirable heat on the specimen sample. In 2003, opto-electrowetting (OEW) and optical dielectrophoresis techniques were proposed to move liquid droplets and microparticles [11,14]. However, these conventional optoelectronic technologies were only available for manipulation at nano-to sub-micro Newton scales. To obtain a better manipulation efficiency for cells sorting in a micro-environment, a much larger force such as a piezoelectric force driven by lead zirconate titanate (PZT) vibration was developed [15,16]. An optical controlling piezoelectric vibration mechanism has recently been proposed which suggests a coupling of spiropyran-doped liquid crystal on PZT plates [17]. However, piezoelectric ceramic is difficult to integrate with a biocompatible microfluidic chip substrate. Furthermore, UV light will damage the biomolecules or cells in the biochip. Therefore, we set out to find an optical controlling piezoelectric material that can be extended to biological applications.

In our previous work, a visible-spectrum-sensitive piezoelectric polymer material was developed using a composite of poly(α -vinylidene fluoride-trifluoroethylene) [P(VDF-TrFE)] and titanium oxide phthalocyanine (TiOPc). We briefly examine the application and photosensitivity of this composite [18]. This material not only provides a biocompatible light response but also allows us to maintain the piezoelectric characteristics which can be adopted for microfluidic biochip applications. In our current work, we study in much more detail the material physical properties and their correlation. We also further advance the material composition to allow us to achieve a more efficient photoconductive effect on our composite when illuminated with white light.

P(VDF-TrFE) is a commercial piezoelectric polymer that possesses an excellent piezoelectric coefficient. In addition, it also possesses a β -phase conformation just from annealing, unlike typical PVDF which must be both stretched and annealed to obtain a piezoelectric β -phase [19–23]. The photoconductivity of TiOPc as a charge generation material (CGM) can be mixed with P(VDF-TrFE) to respond to light over the entire visible wavelength range [24,25].

In this study, we show in our composite material that the piezoelectric characteristics and photo-sensitivity characteristics are dependent on the weight percentage of the TiOPc in the composite. In addition, the optimal TiOPc amount in the composite was also found to give the best response to both visible spectrum and piezoelectric properties. Combining the features of piezoelectric forces and optical tweezers, this composite can provide a quick deformation response with enough force for particle trapping. In addition, the long distance light illumination can eliminate photo damage and harmful heat on the specimen sample. Furthermore, considering the biocompatibility of P(VDF-TrFE) and TiOPc [26–31],

our photo-sensitive piezoelectric material has the potential for use in microfluidic biochip applications.

2. Material and methods

2.1. Material and film preparation

The powder copolymer P(VDF-TrFE) (70/30) was purchased from PiezoTech (France) and the dimethylformamide (DMF) from Acros Organics Co. Titanyl phthalocyanine (TiOPc, $C_{32}H_{16}N_8OTi$) powder was obtained from Sigma–Aldrich Co.

The P(VDF-TrFE) powder was dissolved in a DMF solvent at a concentration of 10 wt%. The solution underwent vigorous stirring at 70° C for more than 24 h after which the TiOPc powder was then added to the solution. The resulting mixture then underwent an ultrasonic process. The P(VDF-TrFE) and the composite material of P(VDF-TrFE) and TiOPc, which is represented as P(VDF-TrFE) + TiOPc, was coated uniformly onto the ITO glass and the solvent volatilized at 60° C on a hotplate for 1.5 h to form the 40 μ m thin film.

The film was then annealed two times at 135° C for 2 h and at 70° C for 1 h in a vacuum oven to increase their crystallinity, and then cooled to room temperature. The film was polarized by corona charging with a distance of 2.5 cm for 2 h at 90° C and then slowly cooled to room temperature.

To measure the hysteresis loop (P–E loop), an Au top electrode of 0.196 mm² was deposited onto the film using a sputtering process. For measuring the piezoelectric constant (d_{33}), the demolded film was deposited onto the Au electrode at both the top and bottom. For measuring the photo-electric response under white light illumination (Apex Fiber Illuminator 70 531, 200 W Hg, Newport), an Au top electrode of 1 cm² size was deposited onto the film. All measurements were performed at room temperature.

2.2. Characterization methods

2.2.1. Copolymer morphology and crystallization analysis

The surface morphology of the P(VDF-TrFE) and P(VDF-TrFE) + TiOPc film was recorded using a scanning electron microscope (SEM) (S-4800, Hitachi). The chemical bonds of the P(VDF-TrFE) + TiOPc composite material were confirmed by Fourier transformed infrared spectroscopy (FTIR) (Nexus 470, Thermo Nicolet) with a DTGS detector. The FTIR transmission was collected using 32 scans at a resolution of 2 cm⁻¹. The crystalline phases were analyzed using a θ/θ X-ray diffractometer (XRD) (X'Pert PRO Diffractometer, Panalytical) from 10 to 50°.

2.2.2. Piezoelectric characterization

The hysteresis loops (P–E loops) were measured using a ferroelectric analyzer (aixACCT, TF2000). The maximum electric field was triggered at 115 MV/m, 1 mA current and 100 Hz frequency. The piezoelectric constant (d_{33}) of the film was obtained from the d_{33} m (KCF Technologies, d_{33} Piezoelectric Meter) with 0.25 N at 110 Hz. The d_{33} value was calculated as $[+d_{33} - (-d_{33})]/2$, where $+d_{33}$ and $-d_{33}$ were the parallel and opposite parallel measurements to the poling direction.

2.2.3. Photo-sensitive experiment

The absorbance spectra of the P(VDF-TrFE) and P(VDF-TrFE) + TiOPc film was taken using an UV–Vis spectroscope (Lambda 900 spectrometer, PerkinElmer) from 300 nm to 900 nm. The photo-electric response was studied by measuring impedance, capacitance and resistance values before and after 5 min of light illumination. The light power on the sample was about 112 klux with a localized temperature at 30° C. The impedance was

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