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ARTICLE

Ferro- and Piezo-electric Properties of Poly(vinyl fluoride) Film with High Ferro- to Para-electric Phase Transition Temperature

Received 00th January 20xx,
Accepted 00th January 20xx

DOI: 10.1039/x0xx00000x

www.rsc.org/

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Excellent ferro- and piezo-electric performance is proposed for the first time in poly(vinyl fluoride) (PVF) in beta crystal phase of hexagonal plane and high crystallinity. A Currie phase transition from ferro- to para-electric is found at 168°C, which is the highest among the reported polymeric ferroelectrics. At room temperature, a coercive electric field as high as 120 MV/m and a remnant displacement of 5 $\mu\text{C}/\text{cm}^2$ are obtained on D-E loops. After polarized at 300 MV/m, a relatively large piezoelectric coefficient of -11 pC/N is detected. Regarding its high depolarization temperature and facile films processing procedure, PVF ferroelectric films may attract interests and find particular applications at elevated temperature.

Introduction

Poly (vinylidene fluoride) (PVDF) was firstly reported to exhibit excellent ferroelectric and piezoelectric properties in 1969 by Kawai [1], and has attracted a great of interests both in academy and industry for their high mechanical elasticity, high electrical breakdown strength, ease of film fabrication, low cost, and self-healing ability. It has been well accepted that semicrystalline PVDF possesses at least four distinct crystal phases including non-polar α phase (TGTG'), polar β phase (TTTT), γ phase (TTTGTGTG') and δ phase (TGTG') (T, G and G' are referring to *trans*, *gauche* and *anti-gauche* conformation, respectively) depending on the processing conditions [2-4]. Among them, obtained by either mechanical stretching or electric poling of α phase, β phase is the most thermodynamically stable form and has the highest polarity [5-7]. After polarized under high electric field, β -PVDF shows excellent ferroelectric properties with a remnant polarization (P_r) as high as 8 $\mu\text{C}/\text{cm}^2$, a maximum polarization (P_m) of 12 $\mu\text{C}/\text{cm}^2$, a coercive electric field (E_c) of 80-100 MV/m, and high piezoelectric properties with a piezoelectric coefficient (d_{33}) of -25 pC/N [8-10]. That allows them to be utilized as nonvolatile memory devices [11], integrated circuits [12], miniature capacitors for telecommunication [13], and stationary power generations [14]. By chemically introducing trifluoroethylene (TrFE) or tetrafluoroethylene (TFE) with certain contents into PVDF polymer chain, the crystal phase of resultant

copolymers could be readily maintained in high polar β phase. The difference of steric bulk between VDF and TrFE (or TFE) units is found to block the formation of kinetically stable TGTG' conformation and favor all-trans chain conformation. It is confirmed that at least 20 mol% of TrFE is required to achieve β phase in P(VDF-TrFE) [15-16] independent on the fabrication conditions. After polarized under high electric field at elevated temperature, P_r , P_m , and E_c could be observed as 10 $\mu\text{C}/\text{cm}^2$, 13 $\mu\text{C}/\text{cm}^2$ and 50 MV/m, respectively, depending on the VDF/TrFE molar ratios as well as the fabrication conditions. The piezoelectric coefficient d_{33} of polarized P(VDF-TrFE) was obtained as -21 to -25 pC/N and showed slight composition dependence. The major advantages of piezoelectric films made of P(VDF-TrFE) is the facile fabrication process although it is badly limited by the poor resource and high cost of TrFE comonomer [17].

More recently, a facile P(VDF-TrFE) synthesis method involving the full hydrogenation of P(VDF-CTFE) has been developed [18-19], which could solve the poor resource and high cost problems of TrFE. The VDF and TrFE units in the resultant copolymer was found to be connected mostly in head-to-head or tail-to-tail way (-CF₂CH₂-CFHCF₂- or -CH₂CF₂-CF₂CFH-), which is different from the head-to-tail connection in major of copolymer prepared directly from the copolymerization of VDF and TrFE monomers. That has also been recognized as the major reason for the difference observed between the copolymers prepared from different process even their chemical composition is consistent [20]. The optimized composition of P(VDF-TrFE) from hydrogenation process was found to be VDF/TrFE=80/20 mol% and the maximum P_r , P_m and E_c was obtained as 8 $\mu\text{C}/\text{cm}^2$, 12 $\mu\text{C}/\text{cm}^2$ and 50-70 MV/m [21]. After polarized, its d_{33} was observed as high as -23 pC/N.

One of the most known disadvantages of PVDF based ferroelectric polymers facing is their relatively low depolarization temperature

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Electronic Supplementary Information (ESI) available: [details of any supplementary information available should be included here]. See DOI: 10.1039/x0xx00000x

(below 100 °C) comparing with ferroelectric ceramics [22]. For PVDF ferroelectric films, the stretching of the films has to be carried out at relatively low temperature (usually below 100 °C) to achieve high orientation of PVDF polymer chain along the drawing direction, therefore high polar β -phase. Apparently, the films are not allowed to be utilized above this processing temperature for the relaxing of the orientated polymer chain and thus the depolarization. For P(VDF-TrFE) films, the Curie temperature (T_c), where the ferroelectric-paraelectric (F-P) phase transition taking place, is the highest temperature for the films to maintain their ferroelectric properties. T_c of P(VDF-TrFE) ranging from 70 °C to 120 °C is mostly dominated by the molar content of TrFE introduced [23-25]. The higher content of TrFE introduced would lead to continuously reduced T_c value to about 70 °C where the TrFE content is increased to about 50 mol%. Accordingly, the E_c of P(VDF-TrFE) films would be reduced from about 70 MV m⁻¹ to about 40 MV m⁻¹. The relatively low depolarization temperature and small E_c of the polymers results into the seriously poor stability when they are applied as piezoelectric films for mechanical and electric energy exchange purpose. Therefore, finding some flexible ferroelectric polymers capable of working at over 100 °C is still a great challenge.

In this work, we would like to report a kind of poly(vinyl fluoride) ferroelectric film with E_c as high as 120 MV/m, which allows it to be worked at higher temperature (up to 140 °C) than current PVDF based ferroelectric films. Without incorporating any other comonomers chemically or stretching the films under the restrict conditions required in PVDF based ferroelectric films, the PVF film is favourable in high polar β -phase and shows a piezoelectric coefficient of -11 pC/N. With respect to its relatively high depolarization temperature and facile processing procedure, it would attract particular interest in the applications for the high stability at elevated temperature.

2 Experimental Section

2.1 Materials

PVF films with a thickness of ~30 μ m (TEDLAR TED014) were purchased from DuPont. The P(VDF-TrFE) copolymer were synthesized by hydrogenation of P(VDF-CTFE) (20 mol% CTFE) copolymer as reported in our previous work [19] for comparison. The P(VDF-TrFE) films with a thickness of ~20 μ m were prepared by a solution casting process from 4% (w/v) N,N-dimethylformamide (DMF) solution at 70 °C on glass slides followed by annealing at 130 °C in a vacuum oven for 4 h.

2.2 Characterization

X-ray diffraction (XRD, RIGAKU D/MAX-2400, Rigaku Industrial Corporation, Japan) was performed to characterize the crystal structures and crystallinity of the PVF films, and the wavelength of the X-ray was 1.542 Å (Cu K α radiation, 40 kV, 100 mA, and 10° min⁻¹). Fourier transform infrared (FTIR, 27 FTIR spectrometer, Bruker Corporation, Germany) was carried out to characterize the crystalline structure and chain conformation of PVF films.

Differential scanning calorimetric (DSC) curves was obtained by a Netzsch DSC 200 PC (Netzsch, Germany) in a nitrogen atmosphere at a heating rate of 10 °C min⁻¹ during the first heating circle. The dielectric response of PVF and P(VDF-TrFE) as a function of frequency and temperature were obtained on an Agilent 4284 LCR meter. TF Analyzer 2000 ferroelectric test system and the electric field with a triangular wave form at a frequency of 10 Hz was employed to detect the displacement against electric field of PVF films.

3 Results and Discussion

3.1 Crystal structures

Figure 1 presents the XRD result of PVF and P(VDF-TrFE). We observe a single peak at 20.8°, which is assigned to the reflection of β crystal plane at (110, 200) and corresponds to *trans* conformation of PVF[26-29]. The high diffraction peak intensity means that the crystal size of PVF is much larger than that of P(VDF-TrFE). Meanwhile, the (200) reflection of PVF calculated from Bragg equation is 4.23Å, which is smaller than that of P(VDF-TrFE) (c.a. 4.57Å) and agrees well with ref [29]. That means the polymer chain of PVF is more densely packed in the crystal cell than that of P(VDF-TrFE). In P(VDF-TrFE), certain content of TrFE units is responsible for turning the chain conformation into *all-trans* structure, which could be attributed to the steric hindrance difference between -CF₂- and -CFH- on TrFE units. In PVF, VF units possess the similar steric structure difference (-CFH- and -CH₂-) as that of TrFE. Apparently, this steric difference is favourable for the formation of *all-trans* chain conformation in PVF, which has been well investigated in P(VDF-TrFE) copolymers.

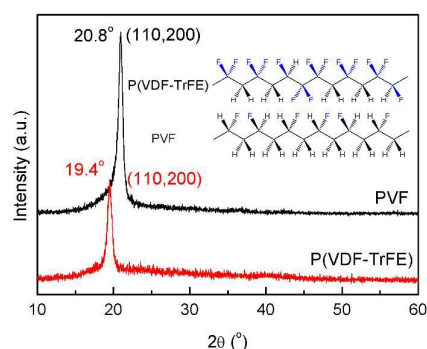


Figure 1. XRD and chain conformation of PVF and P(VDF-TrFE) (80/20 mol%).

XRD results could also be proved by FTIR absorption spectra, as shown in Figure 2. The absorption bands at 840, 506 and 468 cm⁻¹ are observed in the FTIR of P(VDF-TrFE), which are characteristics of PVDF in β phase with *all-trans* chain conformation. In PVF, the absorption bands at 1144, 1092, 1033, 888 and 461 cm⁻¹ related to C-F bonds could confirm the planar *zig-zag* chain with *all-trans* conformation. More detailed investigation shows that PVF

possesses *atactic* structure [26], which means the steric position of C-F and C-H bond on the same carbon atom is dispersed on the two directions randomly as indicated in Figure 1. The bands at 765 and 723 cm^{-1} are from the tail-tail connection of VF units. [28]

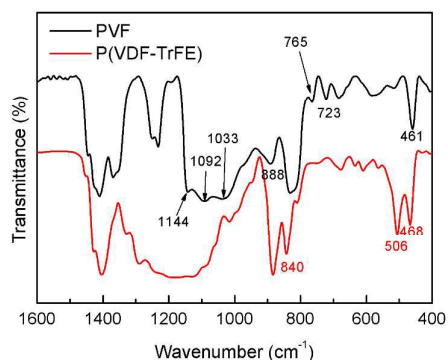


Figure 2. FTIR of PVF and P(VDF-TrFE) (80/20 mol%).

DSC curves of PVF and P(VDF-TrFE) copolymer were presented in Figure 3 for illustrating their phase transition temperature. Apparently, the single endothermic peak at 146 °C is corresponding to the melting temperature (T_m) of β phase P(VDF-TrFE), and the F-P transition temperature is observed at 97 °C. While, T_m of PVF film obtained at 190 °C is rather close to its degradation temperature and a broad endothermic peak at 165 °C corresponding to F-P transition temperature is detected. After annealed at 150 °C for 3h in an oil bath as shown in Figure 3, PVF film exhibits a rather sharp F-P transition peak at 168 °C together with significantly elevated phase transition enthalpy from 10.56 J/g to 16.98 J/g. That means F-P transition temperature of PVF film is even higher than T_m of P(VDF-TrFE) and close to T_m of β -PVDF. Different from high ratio oriented ferroelectric PVDF films at relatively low temperature to achieve all-*trans* conformation, both the high F-P transition temperature and large enthalpy allow PVF film to retain ferroelectricity at the elevated temperature.

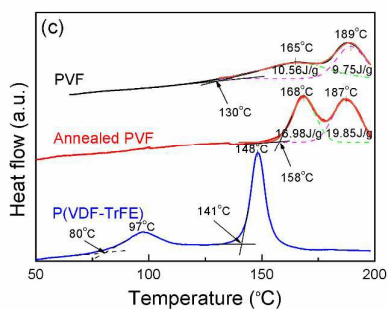


Figure 3. DSC of PVF and P(VDF-TrFE) (80/20 mol%).

3.2 Dielectric properties

Figure 4(a) shows the dielectric response of PVF and P(VDF-TrFE) as a function of frequency from 100Hz to 1 MHz at room temperature.

Both the dielectric constant (ϵ_r) and loss ($\tan\delta$) of PVF were lower than that of P(VDF-TrFE). It was well confirmed that ϵ_r is the contribution of dipoles both in crystalline and amorphous phases of the polymer. Each VDF ($-\text{CH}_2\text{-CF}_2-$) unit possesses about twice dipole moment (7×10^{-30} C m) of each VF ($-\text{CH}_2\text{-CFH}-$) (4×10^{-30} C m), which is responsible for the lower dielectric constant of PVF (~ 8.2) at 100 Hz (Figure. 4(a)). Meanwhile, ϵ_r of PVF is decreasing much slower than P(VDF-TrFE) as frequency increases, especially in 10kHz to 1MHz frequency range. Correspondingly, $\tan\delta$ of PVF is smaller than that of P(VDF-TrFE) especially at frequency from 100kHz to 1MHz, where the quickly improved dielectric loss is attributed to the β -relaxation of dipole orientation in large scale. After annealed at 150 °C, ϵ_r of PVF is slightly increased and the dielectric loss at low frequency is reduced but no visible change is observed at frequency over 30kHz, which might be attributed to the improved film quality and reduced ion conduction at low frequency [30]. Therefore, the smaller ϵ_r and $\tan\delta$ of PVF than P(VDF-TrFE) are mostly attributed to its lower overall dipole moments and much higher content of crystal phase.

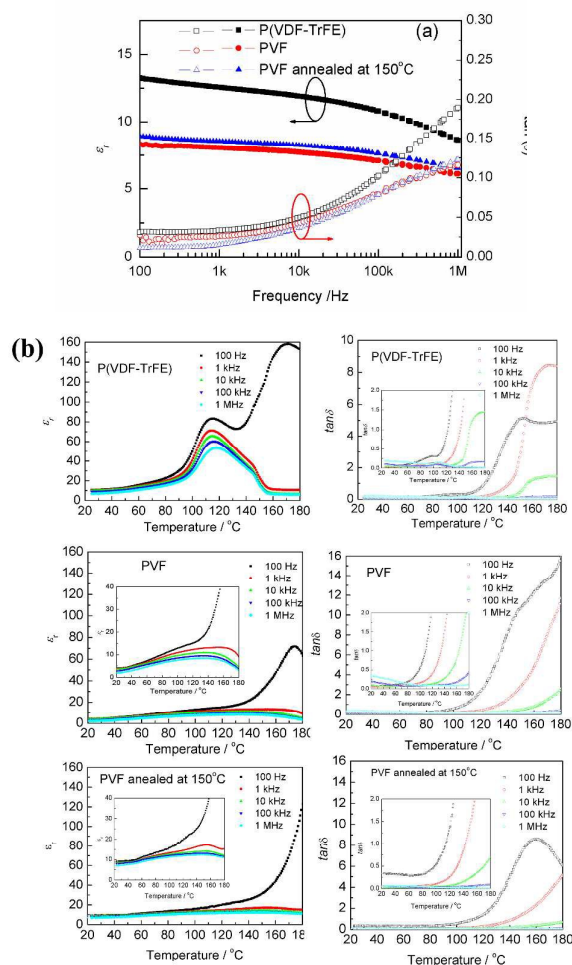


Figure 4. Dielectric constant and loss of P(VDF-TrFE) and PVF as a function of frequency (a) and temperature (b).

In order to detect the phase transition property of PVF, the dielectric response of PVF and P(VDF-TrFE) at various temperatures was measured on an Agilent 4284 LCR meter as presented in Figure. 4(b). As temperature increases, the dielectric constant of P(VDF-TrFE) exhibits a mono-peak at about 110 °C, which has been well expressed corresponding to F-P phase transition. At this temperature, the chain conformation of P(VDF-TrFE) is transferred from TTTT into TTTG structure, where C-C bonds on TrFE units start to rotate freely and the long sequence high polar P(VDF-TrFE) chains are separated into very short TTT segments with moderate polarity by TrFE in G or G' conformation. As a result, the alignment of these short chain segments with the assistance of elevated temperature even under low electric field may contribute huge polarity characterized with significantly elevated dielectric constant at Curie temperature. When the temperature is over T_c , the increased temperature leads to quickly improved disordering mobility of ferroelectric domain induced, thus the sharply dropped dielectric constant and quickly increased dielectric loss. The peak at 170 °C observed in P(VDF-TrFE) at 100Hz both on dielectric constant and dielectric loss curves refers to the pre-melting points caused by impurity ions and conduction loss at low frequency [30-31]. Nevertheless, the contributions of impurity ions and conduction loss will be depressed at high frequency. The dielectric constant of the PVF is reducing slightly as the frequency increases at the same temperature as shown in Figure. 4(b). However, the narrow peak at 175 °C observed at 100Hz both on ϵ_r and $\tan\delta$ curves of PVF refers to the F-P transition, which could be confirmed by DSC results. Because of its severe molecular thermal motion, $\tan\delta$ is apparently increased as the temperature is over 120 °C at low frequency corresponding to the F-P relaxation. Different from the sharp peak observed at about 110 °C in P(VDF-TrFE), as-casted PVF exhibits a broad peak between 50 °C and 180 °C, which agrees well with DSC results in Figure. 3 and might be attributed to the small size ferroelectric phase formed in the films. That means the uniformity of ferroelectric phase domains in as-casted PVF film is rather poor limited by the processing procedure. As temperature increases, the chain conformation of ferroelectric domains would be transferred from *all-trans* into TTTG or TTTG' due to the σ -bond flipping of VF units. With the increase of the refolding chain length in ferroelectric domains, the F-P transition would take place at elevated temperature continuously. The films are preceded by melt extrusion followed by stretching and cooling quickly, which leaves the ferroelectric domains with rather widely dispersed repeat chain length. Therefore, the F-P transition of the extruded films would be observed in a rather wide temperature range. After annealed at 150 °C, the ferroelectric phase in larger scale would be finely formed as indicated in DSC results. As a result, the dielectric constant of the annealed PVF film is increased in the whole testing temperature range as indicated in Figure 4(b). The onset temperature of F-P transition is improved from about 135 °C at 100Hz in PVF film to about 150°C in the annealed PVF, which is rather close to the annealing temperature and confirm the DSC results obtained. The peak of dielectric loss has been shifted to higher temperature with an increment about 15°C as well. That indicates the polarized PVF piezoelectric films at elevated

temperature has no risk of depolarizing at temperature up to 140 °C as that happened in P(VDF-TrFE)s at their Curie temperature. Annealing PVF films at elevated temperature may further improve not only their dielectric performance but also the temperature stability.

3.3 Ferroelectric and Piezoelectric properties

In order to compare their ferroelectric property, we measured the displacement against electric field of PVF and P(VDF-TrFE) films as presented in Figure.5.. At the same applied electric field of 300 MV/m, PVF shows smaller saturated ($P_s \sim 10 \mu\text{C}/\text{cm}^2$) and remnant electric displacement ($P_r \sim 5 \mu\text{C}/\text{cm}^2$) than that of P(VDF-TrFE) film ($12 \mu\text{C}/\text{cm}^2$ and $8 \mu\text{C}/\text{cm}^2$, respectively), which may be attributed to its lower dipole moments as indicated in dielectric section. Interestingly, PVF film possesses much larger coercive electric field (E_c) of 120 MV/m than that of P(VDF-TrFE) (~ 50 MV/m). Meanwhile, at least 250MV/m electric field is required for the orientation of PVF ferroelectric phase at room temperature as indicated in the D-E loops embedded in Fig.5. It has been well reported that E_c is mainly related to the size of ferroelectric domains and the coupling strength among these domains, referring to the ability of depolarization of a polymer. Much larger E_c of PVF indicates that its much longer *all-trans* chain conformation and the smaller distance between polymer chains in crystal cell as indicated in XRD results than P(VDF-TrFE) are favourable for the polarization and ferroelectricity retaining. Consequently, P_s is still increasing as a function of electric field, and the D-E loop is unsaturated even under 300 MV/m field, which is much higher than the saturation electric field of P(VDF-TrFE) (c.a. 200-250MV/m). After annealed at 150 °C, the D-E loop of PVF film shows insignificantly improved P_r , P_m and reduced E_c . Although annealing is able to combine the smaller ferroelectric domains together to form larger one characterized with elevated T_c and elevated dielectric constant under low electric field, but the overall ferroelectric phase content is barely increased. Therefore, the contribution to the overall displacement under high electric field is rather close.

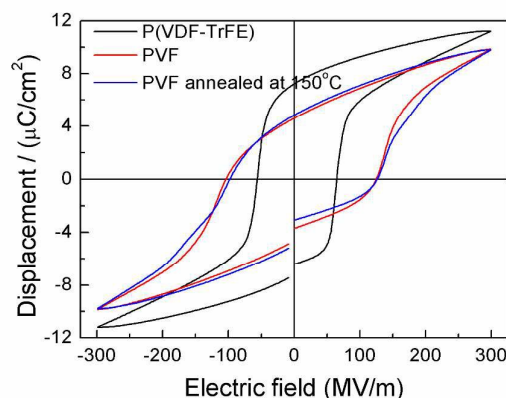


Figure 5. D-E hysteresis loops of PVF and P(VDF-TrFE) (80/20 mol%) under 300 MV/m.

Piezoelectric coefficient (d_{33}) is another important factor to evaluate the piezoelectric performance of ferroelectric polymer, and large P_r is favourable for high piezoelectric property. As shown in Figure.6, d_{33} of PVF is increasing as a function of polarization field and a relatively large value of -11 pC/N is obtained in the film polarized under 300 MV/m field. Interestingly, a sudden increasing of P_r is observed in PVF film at 250-300 MV/m, which is attributed to the ferroelectric domains switching. That means the ferroelectric domains in PVF films was mostly oriented along the applied electric field under this field at room temperature. That strongly indicates that PVF film is rather difficult to be polarized under low electric field and on the other hand the piezoelectricity of the polarized films would be extremely stable. For the limitation of breakdown strength of the PVF film (~ 350 MV/m), the films are polarized at elevated temperature under increasing electric field as shown in Figure.6. As expected, d_{33} is improved significantly as a function of polarizing electric field, and the largest d_{33} is observed as -11 pC/N, which is about half of PVDF and P(VDF-TrFE) (~ 23 pC/N). Interestingly, d_{33} is increased from -3 pC/N to -11 pC/N abruptly, which is rather different from the linearly increasing d_{33} against polarization field observed in PVDF-based piezoelectric films. That means a rather high electric field or energy gap is required to polarize PVF films, and their depolarization would only take place at high temperature on the other hand. Annealing at 150°C shows invisible improvement on d_{33} of PVF film, which could be attributed to the insignificant improved ferroelectric phase as discussed above in D-E loops results. With respect to its high piezoelectric stability, PVF piezoelectric films may find particular applications at elevated temperature.

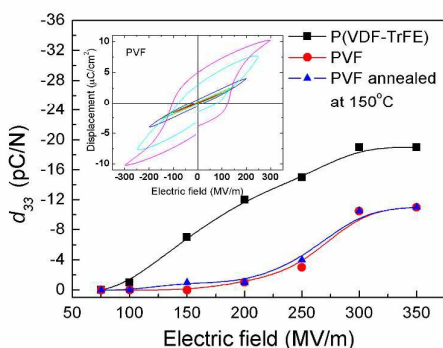


Figure 6 d_{33} of P(VDF-TrFE) (80/20 mol%) and PVF polarized under varied polarization electric field at elevated temperature.

4 Conclusions

In conclusion, PVF films with excellent ferro- and piezo-electric property was reported in this work. Comparing to traditional P(VDF-TrFE), the PVF film shows a unique crystal phase of hexagonal plane and a large crystallinity. Although the low dipole moments are not favorable for their high dielectric properties, the Currie phase transition observed at about 170 °C is the highest among

ferroelectric polymers so far. After poled under 300 MV/m field, a high remnant displacement of 5 $\mu\text{C}/\text{cm}^2$ and a large piezoelectric coefficient of -11 pC/N were obtained accordingly. Regarding its high depolarization temperature and facile processing procedure, it may attract particular attention in academic and practical area.

Acknowledgement

This work was financially supported by the National Nature Science Foundation of China-NSFC (No. 10976022, 51103115, 50903065), Fundamental Research Funds for the Central Universities (No.XJJ2013075, cxt2015003), International Science & Technology Cooperation Program of China (2013DFR50470), Natural Science Basic Research Plan in Shaanxi Province of China (No. 2013JZ003), and National Basic Research Program of China 973 (No. 6132620101).

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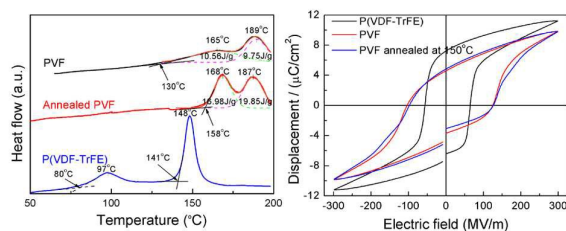
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Ferro- and Piezo-electric Properties of Poly(vinyl fluoride) Film with High Ferro- to Para-electric Phase Transition Temperature

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Excellent ferro- and piezo-electric performance is proposed for the first time in poly(vinyl fluoride) with a ferro- to para-electric transition temperature of 168°C, a coercive electric field of 120 MV/m and a relatively large piezoelectric coefficient of -11 pC/N after polarized under 300 MV/m electric field.