

# Relaxor Fluorinated Polymers: novel applications and recent developments

François Bauer

Piezotech S.A. 9, rue de Colmar 68220 Héisingue, France  
Fax: 0033389675043, e-mail: [francois.bauer@piezotech.fr](mailto:francois.bauer@piezotech.fr)

## ABSTRACT

It has been found that by introducing defects into the P(VDF-TrFE) copolymers, it is possible to convert the polymer from a normal ferroelectric to a relaxor ferroelectric. A new class of ferroelectric polymers, i.e., the terpolymers of P(VDF-TrFE-CFE) or of P(VDF-TrFE-CTFE), was developed from the normal ferroelectric PVDF-TrFE polymer by employing proper defect modifications which eliminate detrimental effects associated with a normal first order F-P transition while maintaining high material responses. Relevant studies show that this class of electroactive polymers offers unique properties in comparison with other ferroelectric polymers. The syntheses of these relaxor ferroelectric polymers have been done by a combination of the suspension polymerization process and an oxygen-activated initiator at a temperature of 40 °C. Films from cast solution can be made in different length and thicknesses. Stretching of these films increases the performance as well as the mechanical properties. These relaxor-ferroelectric terpolymers P(VDF-TrFE-CFE), P(VDF-TrFE-CTFE) are multifunctional i.e. electrostrictive material, dielectric for electric energy storage. The terpolymer exhibits high electrostrictive strain (>7%) with relatively high modulus (>0.4GPa) and high electrocalorific effect. Dielectric polymers with high dipole density have the potential to achieve very high energy density. Examples of devices applications using unimorph systems are presented. Micropump and Optical device concerning a liquid-filled varifocal lens on a chip are described.

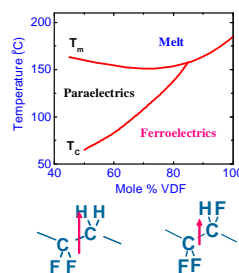
Index Terms — Relaxor-ferroelectric terpolymer, P(VDF-TrFE-CFE) and P(VDF-TrFE-CTFE), Terpolymer Relaxor properties, Harvesting energy, Micro pump, Electro calorific coefficient, Optical device, Energy storage.

## 1 INTRODUCTION

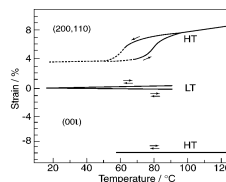
Ferroelectric materials like Poly(vinylidene fluoride) (PVDF) and its copolymer with trifluoroethylene P(VDF-TrFE) are intrinsically multifunctional. In the copolymer with TrFE these ferroelectric polymers will exhibit a Curie transition, where a phase transition between the ferroelectric and paraelectric phases occurs with increasing temperature; figure 1. In the poled ferroelectric phase, these polymers exhibit relatively high piezoelectric response and have found a broad range of applications [1, 2]. However, the low responses in the traditional ferroelectric materials such as low strain level (~0.1% strain) and low sensitivity to external stimulus limit the performance of the devices and systems. As has been observed in many ferroelectric materials, by operating near the instability regions such as ferroelectric-paraelectric (F-P) transitions, many of these responses can be significantly enhanced [3]. On the other hand, phase transition occurs over a narrow temperature range and, in most cases, involves large hysteresis which

prevents these enhanced responses from practical and general applications, figure 1.

P(VDF-TrFE) Phase Diagram



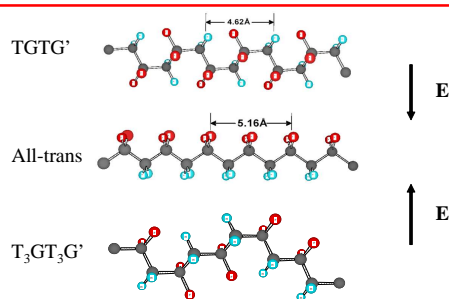
Large strain in PVDF-TrFE associated with structure change between polar and non-polar phases



**Figure 1.** P(VDF-TrFE) phase diagram. Structure change between polar and non-polar phases.

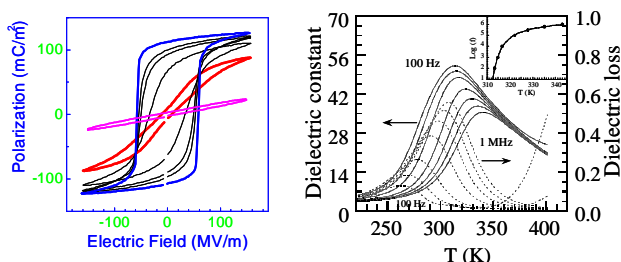
A reversible molecular change between the non-polar to polar forms can generate large polarization change, large strain, and large change in other properties, figure 2. In

### Molecular Conformations of P(VDF-TrFE) Copolymer



**Figure 2.** The conformations of P(VDF-TrFE) copolymer.

traditional ferroelectric PVDF and copolymers, no such a change can be realized. It has been found that by introducing defects via high electron irradiation of the P(VDF-TrFE) copolymers, the copolymer is converted from



**Figure 3.** Defect modifications converting normal ferroelectric P(VDF-TrFE) copolymer into a ferroelectric Relaxor (Qiming Zhang [4]).

a normal ferroelectric to a relaxor ferroelectric, figure 3, [4]. The polarization is nearly eliminated and the dielectric response shows typical ferroelectric relaxor behavior. It is the change in conformation from the disordered TG and T<sub>3</sub>G to the all trans conformation that leads to the observed large electrostrictive strain. This high electrostrictive strain coupled with relatively high modulus (>0.3GPa) makes the polymers suitable as a structure component in addition to the electroactive functions (actuators and sensors). A new class of ferroelectric polymers, i.e., the terpolymers of P(VDF-TrFE-CFE) and P(VDF-TrFE-CTFE), were synthesized via suspension process at Piézotech from the normal ferroelectric PVDF polymer by employing proper defect modifications which eliminate detrimental effects associated with a normal first order F-P transition while maintaining high material responses [5-7]. The introduction of the third monomer into the polymer chain serves to interrupt the ferroelectric domains, thereby reducing their size [7]. Random defect introduction, as in the irradiated copolymer samples [3], broadens the ferroelectric transition and reduces the ferroelectric-paraelectric transition temperature. The random incorporation of the bulky third monomer into the polymer chains forces a conformation change from the all-trans (T<sub>m</sub>≥4) or conformation to the transgauche (TG) and T<sub>3</sub>G conformations [8,9]. Addition of defects in the form of chemical monomers, in this case 1, 1-chlorofluoroethylene (CFE) or chlorotrifluoroethylene

(CTFE) which are copolymerised with the VDF-TrFE, eventually favor the TG conformation and eliminate the normal ferroelectric phase, leading to a relaxor ferroelectric with electromechanical strain greater than 7% and an elastic energy density of 0.7 J/cm<sup>3</sup> under electrical field up to 150 MV/m [11]. It is the change in conformation from the disordered TG and T<sub>3</sub>G to the all trans conformation that leads to the observed large electrostrictive strain. This high electrostrictive strain coupled with relatively high modulus (>0.3GPa) makes the polymers suitable as a structure component in addition to the electroactive functions (actuators and sensors). These relaxor-ferroelectric terpolymers P(VDF-TrFE-CFE) and P(VDF-TrFE-CTFE) exhibit a room temperature dielectric constant greater than 50 [5,7]. To our knowledge, these values are among the highest ones reported in the literature [12]. It should be borne in mind that the amount of CFE or CTFE added to the P(VDF-TrFE) strongly affects the strain response and the polarization hysteresis by changing the spontaneous polarization, the crystallinity, the Young's modulus, the dielectric properties, and the structural conformations [8].

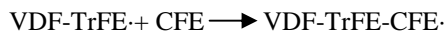
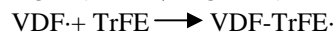
In this paper, we will recall the relaxor properties as well as some novel applications and developments. We will present the capacity of these terpolymers for the storage of electric electric energy.

## 2 TERPOLYMER SYNTHESIS

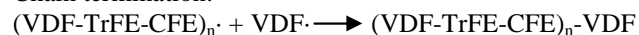
The synthesis of these relaxor ferroelectric polymers at different compositions [5] has been carried out at Piézotech S.A., France. The terpolymers were prepared by a combination of the suspension polymerization process and an oxygen-activated initiator at a temperature of 40 °C. The process of synthesis of P(VDF-TrFE-CFE) or P(VDF-TrFE-CTFE) terpolymer is a radical process using a suspension method. In a radical polymerization process there are three steps: Initiation, Chain propagation, Chain termination as shown in following:

Initiation: peroxide generated radical: RO-OR → 2 RO·

Chain propagation:



Chain termination:



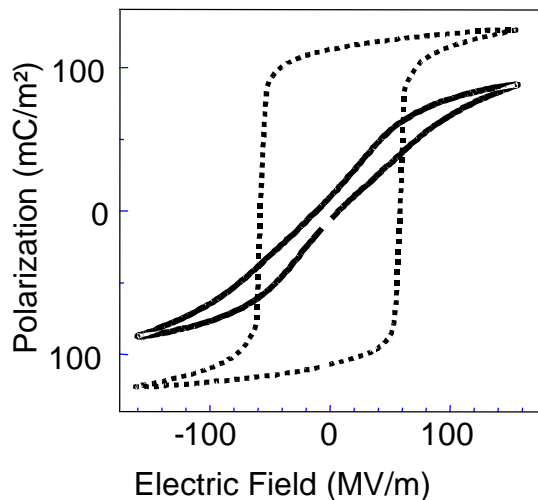
The resulting terpolymer powder (~ 1.5 kg) was recovered, washed with distilled water and dried.

The polymers were then dissolved in methylethylketone or in methylisobutylketone and cast as ~5 -20 μm films depending of the application. Polarization hysteresis measurements were made with our owned circuit, and the electromechanical strain was monitored by a photonic sensor [7]. The dielectric properties were measured in a temperature chamber by an impedance meter, model HP 4284.

## 3 POLARIZATION RESPONSE

It has been shown [5-7] that by co- polymerization of chlorofluoroethylene (CFE) or of chlorotrifluoroethylene (CTFE) with the P(VDF-TrFE), the polymer can be

converted to a relaxor ferroelectric which eliminated the polarization hysteresis (dielectric heating) at room temperature associated with the change of polarization, figure 4, [8].



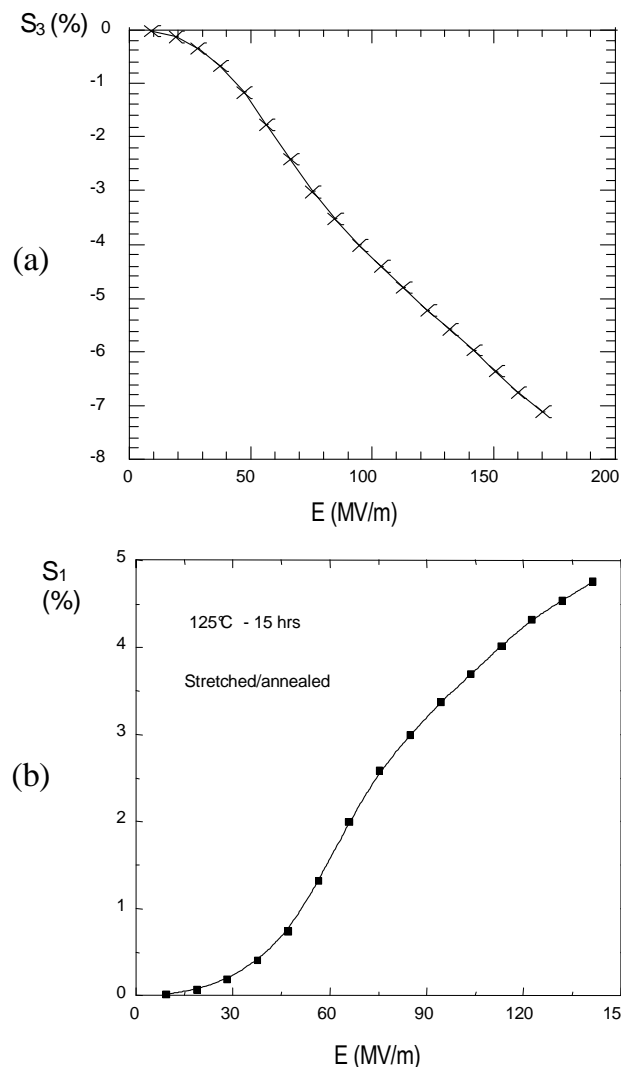
**Figure 4.** Comparison of the polarization hysteresis of the normal ferroelectric polymer (dashed curve, large hysteresis) and relaxor ferroelectric polymer (black curve) at room temperature.

#### 4 ELECTROSTRICTIVE PROPERTIES OF P(VDF-TRFE-CFE)

A unique feature of this relaxor ferroelectric polymer is the large electrostrictive strain which can be induced by external electric field [7,8,11]. An example of the thickness strain as a function of electric field is shown in Fig. 5 (a), for a 65/35/8.6 VDF/TrFE/CFE sample. This is a typical thickness strain plot, which shows both the quadratic dependence of strain at low electric fields and the saturation in strain as the polarization approaches saturation at high fields. The electrostrictive strain reaches more than -7% along the thickness direction. For unstretched films, the transverse strain is 3% and for the uniaxially stretched films, the strain along the stretching direction is 5% under 150 V/μm, Fig. 5(b).

One objective of studying this class of terpolymer is to maximize the electromechanical properties as a function of VDF and CFE contents. Other compositions have been synthesized. Decreasing the CFE fraction below ~8% causes an undesirable rise in the fraction of normal ferroelectric phase in the crystalline regions, while increasing the CFE fraction above ~9% significantly decreases the crystallinity and the Young's modulus [8,11,13]. The electrostrictive strain for the P(VDF-TrFE-CFE) terpolymers has been found to be a maximum near 8.5% CFE, for the VDF-TrFE composition near 65/35. For these terpolymers, the observed range of the Young's modulus for unstretched samples is equal to a value ranging from 150 up to 450 MPa, depending of the CFE content [8,13]. In the study of the electromechanical response of the relaxor ferroelectric polymers, it has been observed that the

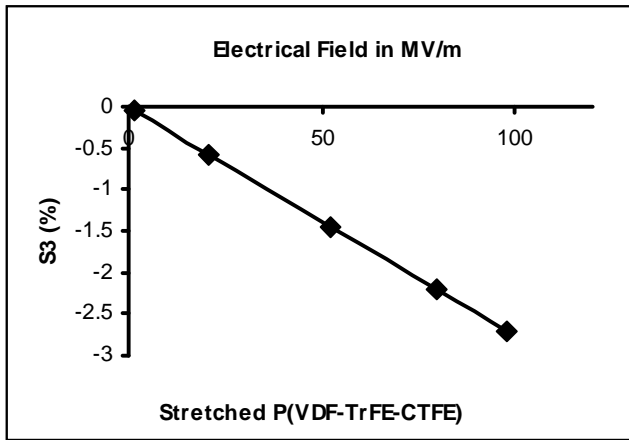
large electrostrictive strain is nearly constant in the temperature range from 20°C to 80 °C [11].



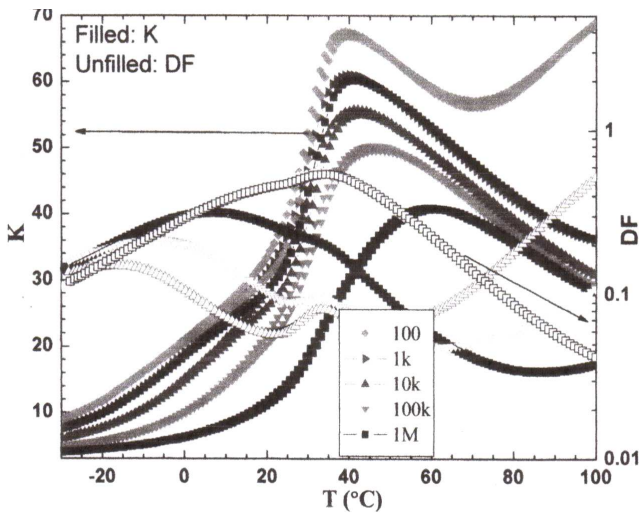
**Figure 5.** The electrostrictive strain of a relaxor ferroelectric polymer (P(VDF-TrFE-CFE) terpolymer): (a) Longitudinal strain, (b) transverse strains measured from stretched terpolymer films.

#### 4.1 ELECTROSTRICTIVE PROPERTIES OF P(VDF-TRFE-CTFE)

Large electrostrictive strain can be induced by external electric field in P(VDF-TrFE-CTFE) too, [7,8,11]. The nonlinear behavior in strain change on cast film solution which shows both the quadratic dependence of strain at low electric fields and the saturation in strain as the polarization approaches saturation at high fields has been observed and measured [8]. Figure 6, shows a typical thickness strain plot, for one dimensional stretched film, as a function of electric field for a 66/34/8.3 VDF/TrFE/CTFE sample. This typical thickness strain plot shows surprisingly the linear dependence of strain change at low electrical fields as well as at higher electrical fields. The Figure 7, presents the dielectric constant  $K$  and dielectric losses referred by "DF" as a function of temperature for the P(VDF-TrFE-CTFE) (66/34/8.3) terpolymer.



**Figure 6.** The electrostrictive transverse strain measured from stretched P(VDF-TrFE-CTFE) terpolymer film.



**Figure 7.** Temperature dependence of (a) dielectric constant and (b) dielectric losses temperature for the (66/34/8.3) P(VDF-TrFE-CTFE) terpolymer. The measuring frequencies are: (from the top to bottom for the dielectric constant and from the bottom to top for the dielectric loss) 0.1, 1, 10, 100, and 1000 kHz. Both data acquired during the heating and cooling runs are presented.

It can be seen that the dielectric constant values obtained during heating differ only very slightly from those obtained during cooling, and that the peak value (near ambient temperature) is greater than 50 (measured at 100 Hz). The dielectric constant of the terpolymer measured at different frequencies also exhibits typical ferroelectric relaxor behavior, i.e. the temperature of the dielectric peak shifts progressively toward higher temperature as the measuring frequency increases.

## 5 ELECTROCALORIFIC EFFECT IN RELAXOR TERPOLYMER

In the Sciences paper [17] it is well described by the authors, that applying an electrical field to a polar polymer may induce a large change in the dipolar ordering, and if

the associated entropy changes are large, they can be explored in cooling applications. With the use of the Maxwell relation between the pyroelectric coefficient and the electrocaloric effect (ECE), they have determined that a large ECE can be realized in the ferroelectric poly(vinylidene fluoride-trifluoroethylene) copolymer at temperatures above the ferroelectric-paraelectric transition (above 70°C), where an isothermal entropy change of more than 55 joules per kilogram per kelvin degree and adiabatic temperature change of more than 12°C were observed. They further showed that a similar level of ECE near room temperature can be achieved by working with the relaxor ferroelectric polymer of P(VDF-TrFE-CFE).

## 6 APPLICATION AND NOVEL DEVELOPMENTS

In Table 1 we summarize and compare the electromechanical properties of four different types of ferroelectric materials [4,6,14,15].  $S_M$  is the maximum strain and  $YS_M^2/2$  is the volumetric elastic energy density.  $Y$  is the elastic modulus along the actuation direction. These values for the terpolymer are of fundamental interest for actuators developments.

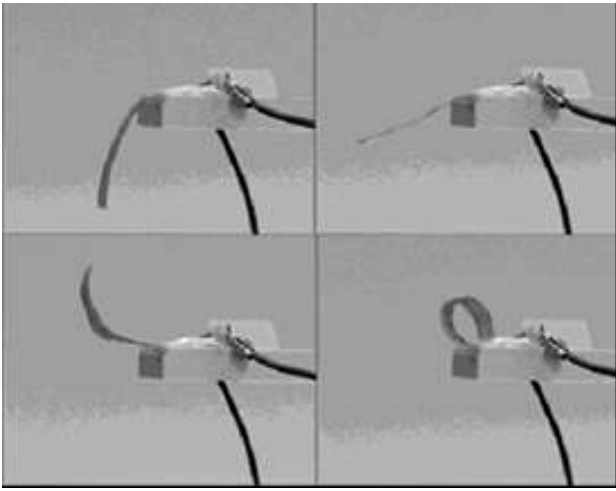
**TABLE 1**

Materials	Strain	Y (GPa)	$S_M$ (%)	Stress (MPa)	$YS_M^2/2$ (J/cm <sup>3</sup> )	$YS_M^2/2r$ (J/kg)	Coupling factor
Piezo-Ceramics (PZT-5)	$S_3$	54	<0.2	108	0.11	14.3	0.75
	$S_1$	61	<0.1	61	0.06	7.8	0.39
PZN-PT Single crystal	$S_3$	8	1.7	136	1.04	136	0.93
Irradiated P(VDFTrFE)	$S_3$	0.5	-5.0	25	0.625	337.8	0.3
	$S_1$	1.0	4.5	43	1.0	500.0	0.65
P(VDFTrFE-CFE) terpolymer	$S_3$	0.3	-7	21	0.73	365	
	$S_1$	0.4	5	20	0.5	250	

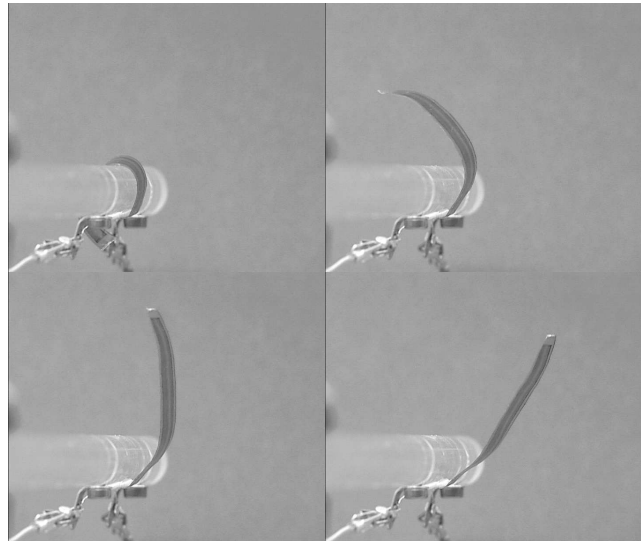
### 6.1 ACTUATORS

Many examples of unimorph actuators using ceramics or electrostrictive polymers have been realized. Nevertheless the high strain change and the high elastic energy density of the terpolymer (Table 1) can lead to high motion of a based terpolymer unimorph. Two layers of terpolymer (45 mm in length, 10 mm in width and 40 μm in thickness) have been bonded together. When the active layer is subjected to the action of an electric field the electrostrictive layer extends in length and the free extremity of the unimorph will follow a curved trajectory as we can see on the Fig.8.

Like other polymers, these terpolymers can be processed to a cylindrical shape. Fig. 9 shows the motion of cylindrical unimorph under the action of an electric field. At the maximum field applied (50 V/μm) the unimorph is practically in a straight position.



**Figure 8.** Motions of the unimorph when the applied electrical field is increasing from 0 up to 60 V/μm. The electric field is equal to 0 V/μm for the upper view on the left side. Then the views from the left to the right

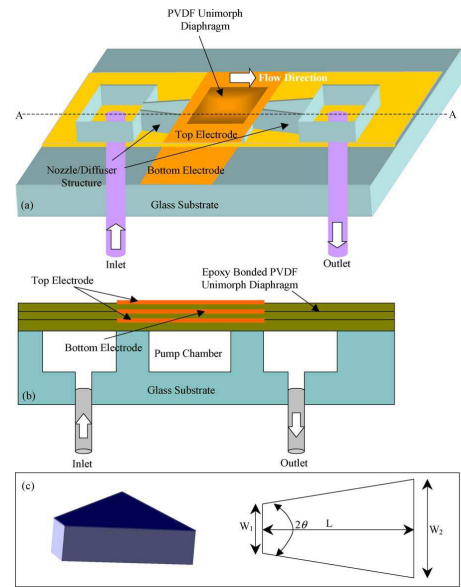


show the motion when the field is increasing to the maximum value applied.

**Figure 9.** From the left to the right and from the top to the bottom, the motions of the unimorph are displayed when the applied electrical field is increasing to a maximum value of 50V/μm and decreasing to 0 V/μm.

## 6.2 MICROFLUIDIC PUMPS

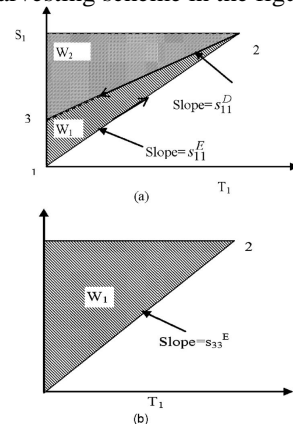
Unimorph electrostrictive polymer has been used for developing new microfluidic pumps by Qiming Zhang et al. [18]. A unimorph type diaphragm actuator consisting of electrostrictive polymer which was uniaxially stretched and high-energy electron irradiated was used as the actuator material. Figure 10 shows the schematic drawing of this micro pump with diffuser and nozzle (valveless pump), which makes use of non-symmetric flow characteristics of the diffuser and nozzle. Performance of such microfluidic pump is given in details in the published paper of Feng Xia et al [18]. The flow rate can reach 25 μL/m (at 60 Hz) for a 2 mm size pump. The flow rate increases linearly with frequency ( $f < f_m$ ). At higher frequencies (above the resonance frequency  $f_m$ ), the flow rate decreases with frequency. The fluid used was methanol.



**Figure 10.** (a) Schematic drawing of the microfluidic pump using P(VDF-TrFE) as the active polymer material. The planar pump operation is based on the rectifying action realized using the two nozzle/diffuser structures. (b) Cross-sectional view of the nozzle–diffuser pump along the line A–A shown in (a) The unimorph structure is electroded only in the pump chamber area with two 20 μm thick PVDF active layers bonded onto a 40 μm thick inactive layer. (c) 3D view of the rectangular nozzle/diffuser elements with labels of the various parameters characterizing the nozzle/diffuser structures after [18].

## 6.3 ACTIVE HARVESTING ENERGY

The high elastic energy density of the electrostrictive PVDF makes it attractive for energy harvesting. Kailang Ren et al [19] have investigated the energy harvesting with an electrostrictive polymer, possessing high electromechanical response and elastic energy density, which make it possible to generate high electric energy density and attractive for the active energy harvesting scheme in the figure 11.



**Figure 11.** From reference [19]: (a) A typical energy harvesting cycle for a piezoelectric material, where  $W_1$  is the harvested electric energy density,  $W_1 + W_2$  is the total input elastic energy density, and  $W_2$  is the elastic energy density delivered from the piezomaterial to the external mechanical environment. Here, the transverse strain and stress  $S_1$  and  $T_1$  are used.  $s_{11}^E$  and  $s_{11}^D$  are the elastic compliances under constant field (short circuit) and constant charge (open circuit) electric conditions. (b) One ideal energy harvesting cycle for a piezoelectric material in which by modifying electric boundary condition of the piezomaterial (imposing an electric field to oppose the strain in the path from 2 to 3 as the stress is reduced),  $W_2$  becomes zero and a higher harvested electric energy density can be obtained.

They have shown that combining the active energy harvesting scheme and high electromechanical response of the electrostrictive terpolymer yields a harvested electric energy density of  $\sim 40 \text{ mJ/cm}^3$  with a 10% efficiency at very low frequency [19].

### 6.4 LIQUID-FILLED VARIFOCAL LENS ON A CHIP ADAPTATIVE OPTICAL LENS

S. Choi et al. [20] have developed a liquid-filled varifocal lens on a chip, figure 12. The principle is as following: the EAP (Electro Active Polymer) actuators push the optical fluid in the chambers into the lens part, which produces a bending deformation of the elastomer membrane corresponding to the change of focal length.

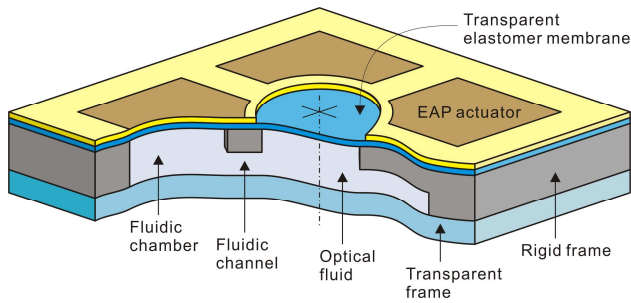


Figure 12. Scheme of the liquid-filled varifocal lens.

The voltage displacement result is depicted in figure 13. The data obtained are also reported below and are showing this new capability of the use of terpolymer in such application.

Important features are following:

- ❖ Wafer-level processes
- ❖ Thickness < 1 mm
- ❖ Dimension: 9.0 mm X 9.4 mm
- ❖ Lens aperture = 2.7 mm
- ❖ Response time < 20 ms
- ❖ Driving voltage < 40 V
- ❖ Dioptric power > 50 diopter

### Voltage-Displacement Characteristics

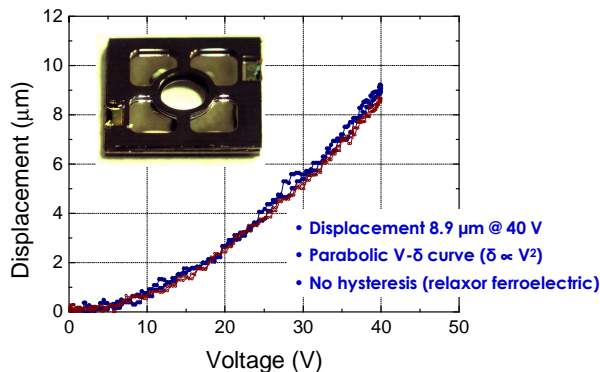


Figure 13. Voltage-Displacement characteristics of the liquid-filled varifocal lens.

## 7 TERPOLYMER WITH HIGH DIELECTRIC PERMITTIVITY: APPLICATION TO ELECTRIC ENERGY STORAGE

Let us recall here some published application to the application of storage of electrical energy [21]. It should be borne in mind that for a non-linear dielectric polymer, the stored and discharged electric energy density should be directly calculated from  $U_e = \int E dD$ , where  $D$  is the surface charge density (electric displacement which is the same as the polarization for the polymers investigated here). Using the polarization hysteresis loop measured under unipolar condition, the stored and discharged energy density can be obtained as schematically shown in figure 14, [21]. This figure 14, is a record for the terpolymer measured at 10 Hz

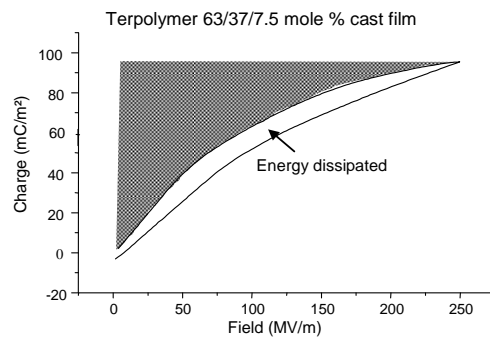


Figure 14. Determination of the discharged (released) energy density (shaded area) and dissipated energy density (open area).

and the shaded area is the discharged energy density. The enclosed and unshaded area represents the energy dissipated. The total stored energy density is the summation of the discharged energy density plus the dissipated energy density, from which the efficiency of the dielectric film can be determined [21]. If there is no conduction loss, the charge density at  $E=0$  at the end of discharging cycle should be the same as that at the beginning of the charging cycle. Figure 15 presents the discharged energy density for the terpolymer 68/32/9 mol% which can reach  $10 \text{ J/cm}^3$  under  $350 \text{ MV/m}$ .

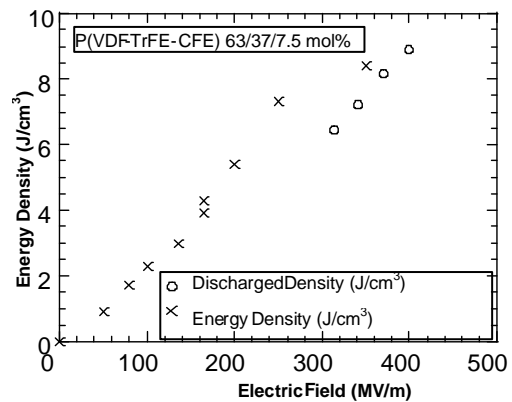


Figure 15. Energy density and discharged energy density for 63/37/7.5 mol% versus maximum electric field levels.

High room temperature dielectric constant ( $\sim 50$ , to our knowledge, this is the highest among the known polymers), high induced polarization ( $\sim 0.1 \text{ C/m}^2$ ), and high electric breakdown field ( $>400 \text{ MV/m}$ ) lead to very high electric energy storage density.

## 8 CONCLUSIONS

The class of electroactive polymers P(VDF-TrFE-CFE) and P(VDF-TrFE-CTFE) offers unique properties in comparison with other polymers. These relaxor-ferroelectric terpolymers are multifunctional i.e. electrostrictive material, dielectric for electric energy storage. High electrostrictive strain leads to large actuation capability. Novel applications have been recalled and presented. The high electrocaloric coefficient, measured on the terpolymer, can be lead to new cooling systems. Original applications in micro fluidic systems as well as in liquid-filled varifocal lens on a chip have been recalled. Active harvesting of energy with terpolymer seems promising. High room temperature dielectric constant, high induced polarization ( $\sim 0.1 \text{ C/m}^2$ ) and high electric breakdown field ( $>400 \text{ MV/m}$ ) lead to very high electric energy density for the electric energy storage capacitors.

## ACKNOWLEDGMENTS

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