

Chapter 7

Conducting Polymers

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Abstract Soft actuators based on conducting polymers are discussed in terms of strain, stress and stability taking the mechanism into consideration. The actuation is generated by the insertion of anions from the electrolyte solution, which is triggered by electrochemical redox reactions. Characteristics of the actuation in polypyrrole, polyaniline, polythiophene, and poly(3,4-ethylenedioxythiophene) (PEDOT) are described. The maximum strain and stress are reported to be 39.9 % and 22 MPa, respectively, in polypyrrole actuator. However, the strain is usually less than 10 %. The stress (contraction force) originates from the elasticity of conducting polymers or Young's modulus. Creeping under tensile loads, which is intimate issue in soft actuators, is discussed in terms of conformation change of polymer chains and shape memory effect. The actuation generated by sorption and desorption of moisture controlled with electrical heating is also introduced with the mechanism and characteristics.

Keywords Artificial muscle • Conducting polymer • Creeping • Electrochemomechanical deformation • Shape memory • Soft actuator • Stress–strain

7.1 Introduction

Artificial muscle is a future device to realize human friendly welfare robots mimicking living body. The artificial muscle, which is defined as a transducer of chemical energy into the contraction force of elastic materials, is too complicated to fabricate. Therefore, presently soft actuators with simple structure have being developed using synthetic polymers. The soft actuators are characterized by self-deformation with stimuli, softness, light-weight and silent movement, contrasting to piezoelectric actuators and shape-memory alloys.

Table 7.1 shows performance in various soft actuators [1–7] driven by electrical stimulus as well as the skeletal muscle for comparison. Conducting polymers have been interested in, because the relatively large strain and stress are generated by the application of low voltages [8–11]. The form of deformation in conducting polymer

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Table 7.1 Performance of various soft actuators

Materials	Form of deformation	Voltage (V)	Strain (%)	Stress (MPa)	Response time (s)	Cycle stability	Environment and remarks
Skeletal muscle	Expansion and contraction	–	20	0.1–0.4	0.1	⊙	Wet [1]
Ionic polymer and metal composites	Bending	2–3	>3	30	0.1	○	Wet [2]
Conducting polymers	Expansion and contraction	1–2	39	22	1	△	Wet [3]
Dielectric elastomers	Expansion and contraction	5,000	100	8	0.2–1	⊙	Dry [4]
Polymer gels	Bending	800	1.3	0.015	0.1	⊙	Dry (Organic Solvent) [5]
Hydro gels	Bending	2–3	–	–	–	○	Wet [6]
Carbon nanotubes	Bending	3–4	0.9	0.1	5	○	Wet (Ionic Liquid) [7]

is expansion and contraction, being similar to that of skeletal muscle. The other polymers except for dielectric elastomers exhibit bending motion. The bending motion is magnified by the bimorph structure and fun for the demonstration, however, is hard to utilize for the practical application.

In this chapter, mechanism, measurement, characteristics, performance and some other interesting features in conducting polymer soft actuators are mentioned.

7.2 Mechanism of Actuation

Conducting polymers, polypyrrole (PPy), polythiophene (PT), polyaniline (PANi) and poly(3,4-ethylenedioxythiophene) (PEDOT) are shown in Fig. 7.1, which have been well studied for soft actuators. These are π -conjugated linear polymers and semiconductor (or insulator) at the neutral state. Upon either electrochemical or chemical oxidation the conducting polymers turn highly conductive with the conductivity larger than several S/cm (metal like). The electrochemical oxidation takes place by removing π -electron via an external circuit and generating of positive charges (polaron or bipolaron) [12]. The schematic picture of polaron in PPy is shown in Fig. 7.1e. The polaron (or bipolaron) is delocalized along the polymer chain by about three unit rings, and is drifted by electric field as the charge carrier.

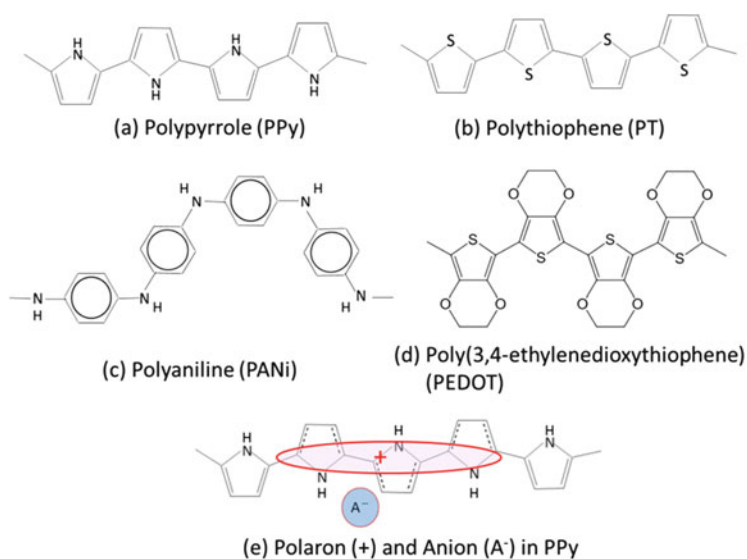


Fig. 7.1 Conducting polymers studied for soft actuator

7.2.1 Electrochemomechanical Actuation

The electrochemical oxidation is accompanied with insertion of anions from electrolyte solution, resulting in the expansion of conducting polymers by the volume of total inserted anions [1]. The degree of oxidation, namely, density of polaron (number of anion) can be controlled by the applied voltage to the conducting polymer (working electrode) against counter electrode. The considerably large swelling is demonstrated, when bulky anions are employed in electrolyte solution at high level of oxidization [3]. The deformation is named as electrochemomechanical deformation (ECMD). On the other hand, the chemical oxidation, which occurs spontaneously depending on the activity of oxidizing agent, is uncontrollable.

The mechanism of actuation in conducting polymers is shown in Fig. 7.2 for two modes of (a) anion drive and (b) cation drive actuations [13, 14]. Usually, conducting polymers are synthesized by either chemical or electrochemical oxidative polymerization of monomers; hence, the as-grown form is oxidized. Oxidized state is stiff compared with the reduced state, because of ionic crosslink at the polaron site with the presence of anion as shown in the right hand side of Fig. 7.2 and also delocalization of π -electron [14]. By the electrochemical reduction, the conducting polymers undergo two routes depending on the size of anions involved in polymerization process as shown in Fig. 7.2a, b. When small anion like ClO_4^- and bis-trifluoromethansulfonylimide (TSFI^-) is employed, the anion moves out from the bulk of conducting polymers into electrolyte solution during electrochemical reduction. In this route the reduced state is shrunk and the actuation is named as the anion drive shown by Fig. 7.2a. On the other hand, large anion like dodecylbenzenesulfonic (DBS^-) acid is used; the anion with long alkyl chain is entangled and immobilized in polymer network [13]. In this case, the conducting polymers are reduced by insertion of cation from electrolyte solution, hence swollen by the volume of additional cations as shown by cation drive in Fig. 7.2b.

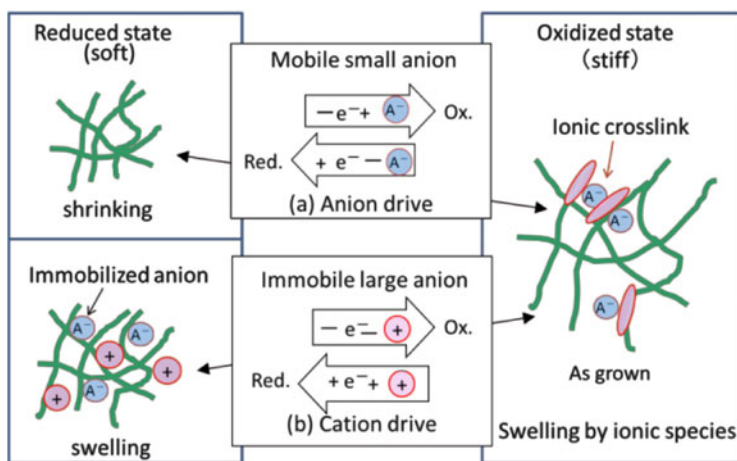


Fig. 7.2 Mechanism of ECMD in conducting polymers

7.2.2 Water Vapor Sorption Based Actuation

Besides the ECMD mechanism, conducting polymer actuators have been fabricated using water vapor induced swelling phenomenon [15–17]. Oxidized conducting polymer is salt form with the ionic bond between positive charges (polaron and bipolaron) and anions. The salt form is favorable to absorb water vapor, resulting in swelling. A film of oxidized conducting polymer is electrically conductive, and can be heated by passing a current with Joule heating. The heating evaporates the absorbed water, then the film shrinks. By turning off the current, the film cools down and absorbs moisture, resulting in swell again. Based on this mechanism, characteristics of conducting polymer actuators are studied for PPy and PEDOT in detail [16, 17].

7.3 Measurement of Actuation

By electrochemical reaction the conducting polymers change the dimension, which can be measured using a handmade apparatus shown in Fig. 7.3 [9, 13, 14]. The conducting polymer films with the approximate dimension of length of 10 mm, width of 2 mm and thickness of 10 μm was hung with a wire connected to working electrode in a glass cell with a pinhole at the bottom. The other end of film was connected through the pinhole to a plate of reflector, on which a weight of load was put to give an isotonic tension to the film. The change of film length was measured by a laser displacement meter under the reflector. Rectangular or triangle voltages are

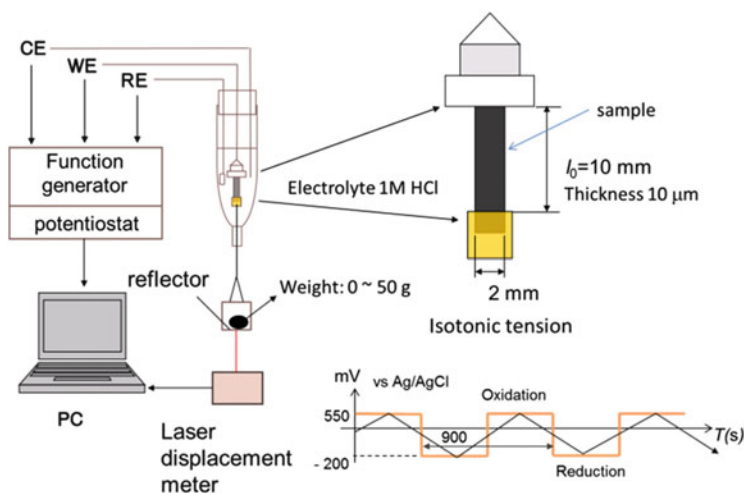
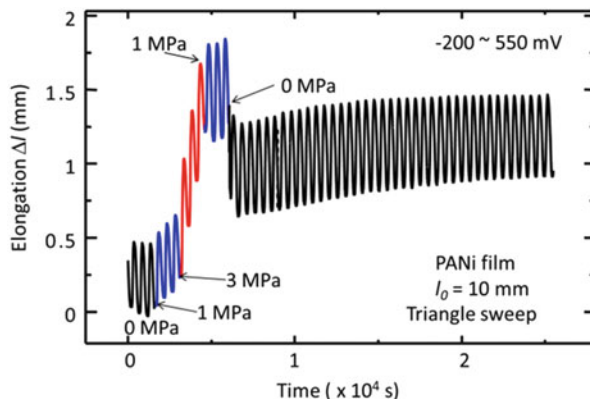


Fig. 7.3 Experimental setup for the measurement of ECMD in conducting polymer films, the typical waveforms of applied voltage are shown at the bottom

Fig. 7.4 Typical time dependence of electrochemomechanical elongation in PANi film under various load stresses



were applied to the film by a computer control system. Using this apparatus, the change of film length can be directly measured at various tensile loads.

Typical response of ECMD in PANi film is shown in Fig. 7.4 under tensile load stresses [18]. Short cycle response is due to elongation and contraction of the film by ECMD. The shift of elongation originates from creeping of the film under load stresses, and is enhanced by electrochemical cycles. It should be noted that the elongation of short cycle Δl after the application of tensile load of 3 MPa is increased, compared with that of the initial cycle at 0 MPa. This phenomenon is a training effect of actuators [18], and is discussed later.

7.4 Characteristics and Performance

7.4.1 Basic Characteristics in Conducting Polymer Actuators

Figure 7.5 depicts stress (σ)-strain (ϵ) curves in DBS doped PPy (PPy/DBS) film for the oxidized and reduced states [13]. Most conducting polymers behave similar in the magnitudes. The σ and ϵ are related with $\sigma = \epsilon E$, where E is Young's modulus. The result indicates that the oxidized state is stiffer than that of the reduced state by 2–3 times. The linear portion of the curves is the region of Hooke's law or elasticity. This elasticity indicates, for example, that 10 MPa stress approximately induces 5 % strain balancing to contraction force of the elastic body.

Load stress (f) and actuation strain ($\Delta l/l_0$) curves in PPy and PANi films are shown in Fig. 7.6. Δl is the incremental length at f and l_0 is the original length of the film. The linear dependence between $\Delta l/l_0$ and f gives an empirical relation [19] of $\Delta l/l_0 = \Delta l_0/l_0 - f/E'$, where Δl_0 is the elongation at $f = 0$ MPa and E' is a constant related to Young's modulus. The f_0 in Fig. 7.4 is so called blocking force or the maximum contraction force of actuators. The result of Fig. 7.6 indicates that actuations in PPy and PANi are similar in the magnitudes of strain and stress

Fig. 7.5 Stress–strain curves in PPy/DBS film

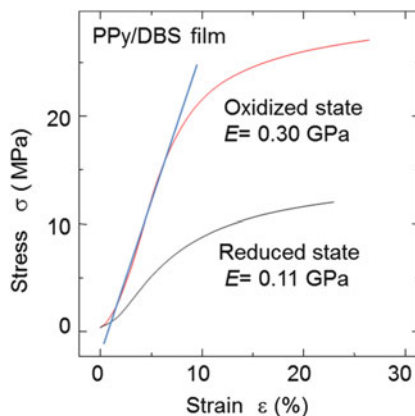
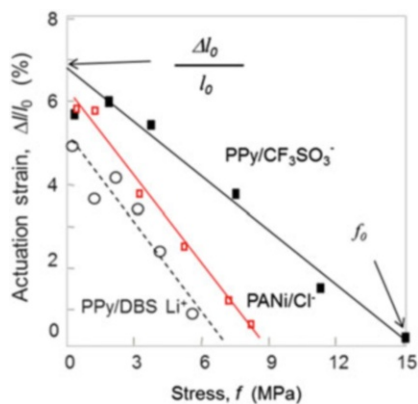


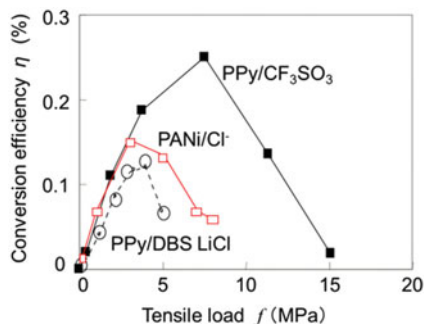
Fig. 7.6 Stress–strain curves in actuation of conducting polymers, PPy and PANi



curves, also being consistent with the static stress and strain curves shown in Fig. 7.5. For example, E' of PPy/DBS from Fig. 7.6 is obtained to be 0.14 GPa using the relation of $\Delta l_0/l_0 - f_0/E' = 0$, and corresponds to the middle of E .

Figure 7.7 shows energy conversion efficiencies in PPy and PANi actuators [20], that is, the ratio of mechanical output energy of $W_{\text{Mout}} = fS\Delta l_0$ to electrical input energy of $W_{\text{Ein}} = \int V i dt$, where S is cross section of the film, V is applied voltage and i is current for the actuator. The efficiency is small, because the most of W_{Ein} is stored in film as the secondary battery, which can be recovered during reduction or elongation. If the electrical energy is harvested during reduction, the efficiency should be increased. An interesting result is that the maximum efficiency was obtained at the half of blocking stress, indicating the W_{Ein} is nearly constant.

Fig. 7.7 Typical energy conversion efficiencies in PPy and PANi actuators derived from Fig. 7.6



7.4.2 Polypyrrole Actuator

Characteristics and performances of typical conducting polymer actuators are tabulated in Table 7.2. The maximum strain is the value at the load stress free. PPy has been widely studied among conducting polymers, because of the easy preparation by electrochemical deposition, stable actuation and large strain. The largest ECMD was reported for gel like porous PPy film, demonstrating the strain of 39.9 % [39]. Generally, in the anion drive actuators, the larger the anion the larger the strain. In additional, the large strain has been observed by the effect of solvation, in which ion is surrounded by solvent and forms cluster. It is also noted that even in cation drive mode the PPy film obtained by electrodeposition in DBS acid is strongly anisotropic, and the expansion of the film thickness direction is reported to be 35 % [25]. PPy prepared by chemical method has not been studied for the actuation, since it has been hard to obtain the high quality material, so far.

The PPy actuator based on water vapor absorption mechanism [15, 16] is also shown in Table 7.2. The strain depends on the ambient humidity and power of heating. For instance, PPy film with the conductivity of 94 S/cm, length 35 mm, width 4 mm and thickness 38 μm was heated by applied voltage of 3 V and current of 70 mA, resulted in 1.5 % strain at relative humidity (RH) of 50 %. In PEDOT/PSS film [17] with the conductivity of 3 S/cm, the applied voltage of 35 V gave the strain of 3.3 %. The response times for contraction and expansion are several tens second depending on the applied voltage.

7.4.3 Polyaniline Actuator

The merits of PANi are the stable operation in aqueous electrolytes, and the film can be chemically prepared by mass production using the cheap raw material [40]. The demerits of PANi actuator are that the actuator has to be operated in a strong acid (HCl) of pH < 1 with rather small strain of 3.2 %. By using high salt (3 M NaCl) concentration as the supporting electrolyte, the pH was increased to 3 and a larger strain of 6.7 % was obtained [29].

Table 7.2 Characteristics and performances of typical ECMD in various conducting polymers

Conducting polymers	Electrolyte solution	Strain (%)	Stress (MPa)	Preparation	Remarks and reference	
PPy	TBATFSI/H ₂ O	26.5	Blocking 7.6	ECD TBATFSI/ MB	Anion drive. [21]	
	NaPF ₆ /H ₂ O	12.4	Blocking 22	ECD BACF ₃ SO ₃ / MB	Anion drive. [22]	
	LiCl/H ₂ O	4.9	Blocking 5	ECD DBS/H ₂ O	Cation Drive. [23]	
	TBAPF ₆ /PC	2	At 5.0	ECD TBAPF ₆ /PC	Anion Drive. [24]	
	NaDBS/H ₂ O	thickness change 35	–	ECD NaDBS/H ₂ O	Cation Drive [25]	
	NaDEHS/CH ₃ CN/ H ₂ O	30	–	ECD NaDEHS/ H ₂ O	Anion Drive [26]	
	Ionic liquid: BMPTFSI	5	–	ECD LiTFSI/ MB/DP	Cation drive [27]	
	Ionic liquid: EMITFSA	2.5–3.0	0–6.5	ECD TBAPF ₆ /PC	Cation Drive [28]	
	Joule heating	1.5	–	ECD TBAClO ₄ / PC	Water Vapor, RH46–52 % [16]	
PANi	HCl/H ₂ O	6.7	Blocking 9	Chemical HCl	Anion drive. [29]	
	HCl/H ₂ O	0.92	Blocking 34	Chemical AMPS	Anion drive [30]	
	Ionic Liquid: BMIMBF ₄	0.28	–	Chemical	Cation drive [31]	
PAT	PHT	TBABF ₄ /CH ₃ CN	3.5	–	Chemical	Anion drive [32] 347 (2007)523
	PMT	TBAPF ₆ /PC	2.1	–	ECD. TBAPF ₆ /PC	Anion drive [33]
PEDOT	TBAPF ₆ /PC	2	–	ECD. TBAPF ₆ /PC	Anion and Cation drive [34]	
	TBACF ₃ SO ₃ /PC	2–3	At 1.35	ECD. TBACF ₃ SO ₃ / PC	Cation drive. [35]	
	KCl/H ₂ O	1.4	At 2	ECD. TBACF ₃ SO ₃ / MB	Cation drive [36]	
	Ionic liquid EMITFSI/PEO: PTHF IPN	0.75	–	Chemical FeCl ₃	Bilayer [37]	

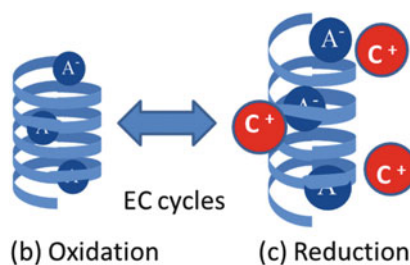
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Table 7.2 (continued)

Conducting polymers	Electrolyte solution	Strain (%)	Stress (MPa)	Preparation	Remarks and reference
	Ionic liquid BMITFSI etc./	Bending beam		Chemical	Bilayer Anion [38]
	Joule heating	3.3		Chemical	Water Vapor, RH20–90 % [17]

TBA tetrabutylammonium, *AMPS* 2-acrylamido-2-methyl-propane-1-sulfonic acid, *PC* propylene carbonate, *DEHS* di-(2-ethylhexyl) sulfosuccinate, *EMI* 1-ethyl-3-methylimidazolium, *MB* methyl benzoate, *DP* dimethyl phthalate, *BMP* 1-butyl-1-methylpyrrolidinium, *BMIM* 1-butyl-3-methyl imidazolium, *PTHF* poly(tetrahydrofurane), *IPN* Inter-Penetrating Network

Fig. 7.8 Schematic drawing of helical structure of PEDOT and EC cycles



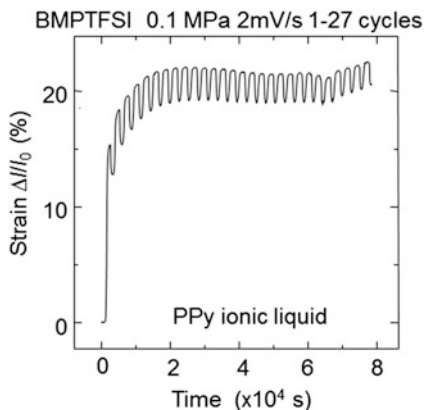
7.4.4 Polyalkylthiophene and PEDOT Actuators

Poly(alkylthiophene), PAT, is rather hard to prepare either chemically or electrochemically for tough film suitable to actuator. This is reason why few study in electrochemical devices and also because of small strain and unstable actuation [32, 41]. On the other hand for the instability of PAT actuators, PEDOT being polythiophene derivative is remarkably stable in air and electrochemical cycles. However, the ECMD strains reported are small as shown in Table 7.2, the ECMD is mostly cation drive mode due to the PEDOT structures. It is proposed that PEDOT prepared by electrochemical polymerization takes the helical structure, and the anions are thought to be immobilized inside helix [36] as shown in Fig. 7.8.

7.4.5 Ionic Liquids

Ionic liquids (ILs) have attracted much attention because of the large ion mobility, stability, inflammability and large electrochemical window. ECMDs using ILs have been studied for many soft actuators including conducting polymers, carbon nano-tube and IPMC as discussed in other chapters. ILs are consisting of large cations

Fig. 7.9 Initial creeping of PPy actuator in ionic liquid by EC reduction



and anions, therefore, the response of ECMD is usually slow. ECMD in conducting polymers and ILs mostly exhibit cation drive mode, even anions are smaller than cations [27, 28].

As shown in Fig. 7.9 the electrochemical cycle of PPy in IL starts by reduction and swelling with cation doping, and never begins by oxidation [42]. This is explained as follows. Conducting polymer films as-synthesized and oxidized form repel ILs and do not wet with ILs. The fact indicates that inside film is filled with dopant anions, then, cations and ILs initiate to move into the film to attain the concentration equilibrium of IL inside and outside of film. The initial large creeping is due to insertion of IL into the film. The mechanism is reasonable and favorable, contrary to that anions move out by reduction, due to high concentration of the ionic liquid in electrolyte. The strain of ECMD is small and approximately 1–2 %, since the cation of ionic liquid moves solely in the conducting polymer without making solvation. In another word, large strain may not be expected in conducting polymer and ionic liquid actuators.

7.5 Creep and Related Phenomena

Creeping is observed under tensile stress, when a load stress goes over the elastic or Hook's law region. In soft actuators the creeping is literally serious issues for the precise positioning [14, 18, 24]. Creeping results from (1) slipping of polymer chains, (2) breaking and yielding of chains and (3) conformation change of polymer alignment. Creeping is significantly enhanced during EC cycles [18] as shown in Fig. 7.10a, which is compared with the mechanical creeping of Fig. 7.10b without EC cycles. The reason is that ions move dynamically in the polymer network during electrochemical cycles generating and degenerating ionic crosslinks. This is named as electrochemical (EC)-creeping and a few studies [18, 24] are reported. The creeping due to (1) and (2) is the permanent deformation and that by (3) is recovered by removing the tensile load and EC cycles [14]. As shown in

Fig. 7.10 (a) EC creep and (b) mechanical creep in PANi film

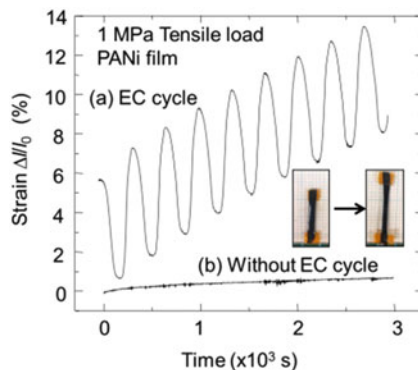


Fig. 7.11 Recovery of creep

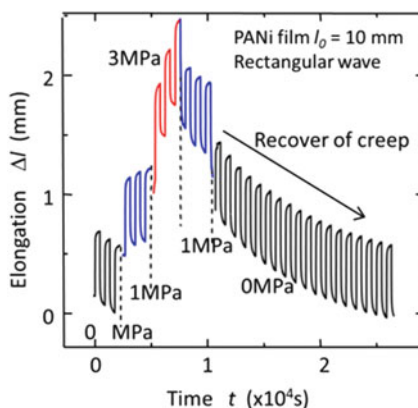


Fig. 7.11 the recovery of EC creep is clearly observed in PANi film, when operated by a rectangular wave forms, somehow different from the case of triangular sweep (Fig. 7.4).

The training, memory effects and recovery of creep are resulted from the conformation change of polymer chains [14, 18]. The mechanism of training effect is that the tensile stress stretches polymer chains to be anisotropic and enhances the electrochemical activity of the film. The stretched conformation is frozen or fixed with ionic crosslink, resulting in the shape memory. By removing the tensile stresses and EC cycles, stretched polymer chains come back to original shape by the elasticity of polymer chains (thermal relaxation), resulting in recovery of creeping.

7.6 Conclusion

Various approaches have been attempted to enhance the strain and stress to the level of demands for practical applications. The largest strain of 39 % and stress of 22 MPa are better than those of a typical skeletal muscle of 20 % and 0.1 MPa,

respectively. Conducting polymer actuators have some superiority compared with the other soft actuators, however, the total performance is still below the commercial level, so far. The conducting polymer actuators are suffering from issues of cycle life, response time. The cycle life has been improved by the operation of moderate potential spans and employing ionic liquids with wide potential window. In use of ionic liquid, the cycle life has been elongated to some extent; however, the strain is not satisfactory. The response time in typical conducting polymers are around 1 s, which is not quick enough for the application. We believe that using conformation change of polymer structure could be a possible mechanism for the innovative soft actuators.

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