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# Actuation behaviour of polyaniline films and tubes prepared by phase inversion technique

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## ABSTRACT

The phase inversion technique was used to produce polyaniline (PAn) actuators with different geometries that cannot be obtained by PAn cast from *N*-methyl-2-pyrrolidinone (NMP) solution in a conventional way. PAn was cast and coagulated in a water bath forming films and tubes with or without a platinum (Pt) wire helix as an interconnect. PAn was doped with hydrochloric solution (HCl, 1 M) (PAn/HCl) or methanesulfonic acid (MSA, 1 M) (PAn/MSA). In nitric acid (HNO<sub>3</sub>, 1 M) aqueous electrolyte, the actuation strain of PAn/HCl was 0.9 % which increased to 2.0 % and 2.7 % for the tubes without and with the Pt helix, respectively. The Pt helix helped prevent the IR drop along the actuator. Comparing with NaNO<sub>3</sub> (1 M) aqueous electrolyte, the use of HNO<sub>3</sub> aqueous electrolyte gave better actuation stability where at least 100 cycles were observed and the final actuation strain was determined by the size of dopant. Change of coagulation bath from water to NMP (30 % w/w)/water resulted in subtle difference in the Young's modulus of PAn/MSA in oxidized and reduced states. PAn prepared by phase inversion technique is porous by nature, consequently it is brittle and exhibits a low actuation stress (0.3 – 0.4 MPa).

**Keywords:** conducting polymer, polyaniline, actuators, phase inversion technique

## 1. INTRODUCTION

Electromechanical actuators based on conducting polymers have attracted considerable attention for a number of years due to the light weight, low operational voltage and ease of production [1]. Of the known conducting polymers, polyaniline (PAn) is one of the most attractive materials because PAn emeraldine base (PAn EB) can dissolve in a number of solvents forming a viscous solution for processing. Also, recent work on the interactions between PAn and carbon nanotubes [2] indicate that these two components can form interesting composites and presents a good potential for new actuator materials.

The actuation behaviour of PAn and its derivatives in aqueous solutions has been investigated in detail notably by Kaneto's group [3, 4] and Lu and Mattes [5]. The latter have examined different doping acids to optimize the actuation of PAn in non-aqueous electrolytes. The samples of these studies were PAn free standing films obtained by casting PAn EB from *N*-methyl-2-pyrrolidinone (NMP) solution onto a glass substrate with the residual NMP removed by heating. Although this conventional method is convenient for making films, it is almost impossible to obtain PAn with desirable microstructures or in different configurations such as fibres and tubes. A phase-inversion technique developed by Yang et al [6], where PAn was cast from a solution of PAn EB in *N,N*-dimethyl propylene urea (DMPU) and coagulated in a water or non-solvent bath, provides an alternative to fabricate PAn into desirable shapes and microstructures. It was also reported [7, 8] that DMPU as a solvent gave better solution stability than NMP as the gel easily occurred when PAn was dissolved in NMP even at concentrations as low as 5 % w/w. The stability of solution was greatly improved when it was dissolved in DMPU. It was found that a 10 % w/w concentration solution was stable for 1500 minutes with minimal change in solution viscosity [7]. The phase-inversion technique has been used to generate a gradient porosity across the

film thickness that controlled the bending of PAn actuators by absorbing/desorbing chemical vapours [9] and by electrochemically potential stimulation [10].

Polypyrrole (PPy) actuators with both a tubular geometry and a helical platinum wire interconnect have been developed in our research group [11, 12]. As a first step to construct PAn tubular actuators with platinum helix to prevent the IR drop along the length, PAn or PAn/CNT in a NMP solution was coated onto PPy helix tube which was used as a substrate [13]. It has been demonstrated that the actuation strain and the stiffness were enhanced by the platinum helix coil.

In the present work, neat PAn actuators are prepared by casting 10 % w/w of high molecular weight EB (MW: 300,000) in DMPU solution and a phase inversion technique was employed to form free standing PAn films and tubes with and without a platinum helix. The effect of actuator geometry and conductivity on actuation behaviour was investigated.

## 2. EXPERIMENTAL

### 2.1 Reagents and Materials

N, N-dimethyl propylene urea, N-methyl-2-pyrrolidinone, phenylhydrazine, methanesulfonic acid (MSA) were obtained from Aldrich and used as received. PAn emeraldine base (EB) (MW: 300,000) was purchased from Santa Fe Science & Technology (USA). Nitric acid (HNO<sub>3</sub>) and hydrochloric acid (HCl) was obtained from Asia Pacific Specialty Chemicals Limited. Platinum wires in 250 and 50  $\mu$ m diameter were obtained Goodfellow Cambridge Limited.

### 2.2 Instrumentation

The pulse current during actuation test was applied using an EG and G Princeton Applied Research Mode 363 potentiostat/galvanostat. The actuation tests were conducted on an Aurora Scientific Dual Mode Lever System 300B. All the experiment data was processed and recorded by MacLab/4e AD Instruments and a computer.

### 2.3 Preparation of PAn Actuators

Reducing agent phenylhydrazine (3 % w/w of DMPU) was first added to DMPU to prevent polymer gelation. PAn EB (10 % w/w of DMPU) was gradually added to DMPU/ phenylhydrazine, the solution was constantly stirred for 24 hours with the temperature controlled at 0 °C. PAn EB was converted to PAn leucoemeraldine base (LEB) by the presence of phenylhydrazine.

The preparation of PAn actuators was carried out using the phase inversion technique pioneered by Loeb and Sourirajan [14]. As-prepared PAn LEB/DMPU solution was cast on a glass slide to form a thin layer of polymer solution. The glass slide with the polymer solution layer was immediately immersed in a water coagulation bath for 24 hours to ensure removal of the DMPU through a solvent/non-solvent exchange process. After removed from water, the film was peeled off and cut into strips of 20 mm long and 3mm wide. Similarly, another PAn film was coagulated in a NMP (30 % w/w)/water bath for 24 hours.

A PAn tube was prepared by dipping a 250  $\mu$ m platinum wire into the PAn EB/DMPU solution, allowing the solution to coat on the surface of the Pt wire. For a PAn tube with a Pt helix, a 250  $\mu$ m Pt wire wound with another 50  $\mu$ m Pt wire into a helix was used. The Pt wire (with or without the Pt helix) with polymer solution coating was then immersed in the water coagulation bath as described above. After removed from the water coagulation bath, the 250  $\mu$ m Pt wire was pulled out leaving a PAn hollow tube with or without the Pt helix. PAn films and tubes were doped in HCl (1 M) aqueous solution for 24 hours or in MSA (1 M) aqueous solution for 6 hours and then dried at room temperature for further tests. The doping process converted the PAn LEB to PAn emeraldine salt (ES) which is in conductive state.

### 2.4 Actuation test

The actuation performance of PAn actuators was tested on a Dual Mode Lever System 300B. Two-electrode configuration was used while pulse current was applied as electrochemical stimulation. The positive and negative current was applied for 30 s each alternatively from  $\pm 1$ mA to the current at which PAn actuators

produced a maximum actuation strain.  $\text{HNO}_3$  (1 M) and  $\text{NaNO}_3$  (1 M) aqueous solutions were employed as an electrolyte. Isotonic actuation tests were conducted at steadily increasing loads until samples were broken. The applied current for the isotonic actuation tests was close to the current where the maximum actuation strain was obtained.

## 2.5 Young's modulus measurements

Samples of the same dimension with ones for actuation tests were used for the stress/strain test to obtain the Young's modulus at both the contraction and expansion states using a Dual Mode Lever System 300B. Similar to the isotonic actuation testing conditions, a positive/negative current density was applied to the test sample for 30 s prior to the test to obtain the expansion/contraction (oxidized/reduced) state. The sample was gradually stretched to a desirable strain, while corresponding stress was recorded. The Young's modulus was calculated from the linear portion of the stress/strain curve with linear regression.

## 2.6 Conductivity measurement

Conductivity of PAn samples was measured by using a 4-point probe. A constant current was passed across the two outer electrodes and the voltage between the two inner electrodes was measured by a voltmeter. Each sample was measured three times by applying three different amount of current. The average value was used to determine the material conductivity.

## 2.7 Cyclic voltammetry study

Cyclic voltammetry (CV) study was conducted in a three-electrode electrochemical cell. PAn EB in DMPU solution was coated on a platinum working electrode and immersed in a water coagulation bath following the same approach as actuator preparation that described above. A reference electrode ( $\text{Ag}/\text{Ag}^+$ ) and a platinum mesh auxiliary electrode were used when CV study was conducted in  $\text{HNO}_3$  (1 M) or  $\text{NaNO}_3$  (1 M) electrolyte solution.

# 3. RESULTS AND DISCUSSION

Conductivity of PAn films, tubes with or without the Pt helix are shown in Table 1. Conductivity is increased 4 times by changing the geometric shape of the actuator from film to tube. When casting films, the polymer solution was spread out on the surface of a glass slide, after coagulation the solidified film had no direction. On the other hand, in the process of making a tube the polymer solution was coated on the Pt wire, the Pt wire was then drawn out from the solution very rapidly. As a result, the polymer layer around the Pt wire may be aligned along the length of the wire. The molecular alignment would contribute to the observed increased conductivity. This enhancement in conductivity was clearly observed in spun PAn fibres with sufficiently high conductivity (90 S/cm) resulted from the axial alignments [15]. Incorporation of a Pt helix into the PAn tube increased the conductivity of actuator by 180 times compared with that of the tube without the helix (Table 1).

Table 1 Conductivity and Actuation Strain of different PAn sample

Sample	Conductivity (S/cm)	Actuation Strain (%)
PAn film*	0.34	0.9
PAn tube*	1.34	2.0
PAn tube with helix*	244	2.7
PAn film**	1.21	0.65
PAn film***	2.72	0.71

\* coagulated in water bath and doped with  $\text{HCl}$  (1 M) for 24 hours.

\*\* coagulated in water bath doped with  $\text{MSA}$  (1 M) for 6 hours.

\*\*\* coagulated in  $\text{NMP}$  (30 %)/water bath doped with  $\text{MSA}$  (1 M) for 6 hours.

Figure 1 shows the actuation strain of PAn film and tubes with or without the Pt helix actuated at increasing current density when tested in a  $\text{HNO}_3$  (1 M) aqueous solution. These samples were doped in  $\text{HCl}$  (1 M) solution. The strain increases to a maximum value before declining. In each case, the decline is associated with irreversible degradation of the polymer. The PAn film gives the maximum actuation strain of 0.9 % whereas the

PAn tube with the Pt helix produced 2.7 % strain which is the highest among the three tested samples. Similar results were found in polypyrrole (PPy) actuators [16] when comparing the actuation strains produced by PPy film, PPy tube and PPy tube with the Pt helix. The improvement of the actuation behaviour of the tube over the film is due to the higher conductivity of the tube. Furthermore, the Pt wire inside the PAn tube significantly prevents the IR drop along the actuator and improves the current going through from one end to the other end. The electrochemical efficiency of the actuator is thus increased and resulted in a higher actuation strain.

While the conductivity of the PAn tube with Pt helix (244 S/cm) is substantially higher than that of the PAn tube (1.34 S/cm), the actuation strain can only increase to 2.7 % from the 2.0% strain of the PAn tube (Table 1). Thus, the value of 2.7 % can be considered as the maximum isotonic actuation strain for the PAn doped with HCl operated in  $\text{HNO}_3$  aqueous solution under the given test condition. This value is much higher than the strain (1 %) of the PAn film sample tested in  $\text{H}_2\text{SO}_4$  (0.5 M) [17]. Kaneko and Kaneto [3] reported a strain of 2.5 % for PAn samples cast from NMP solution at the potential scan rate of 5 mV/s in HCl aqueous solutions. In the present study, the actuation test is operated under current controlled condition that gives an average potential changing rate of 100 mV/s. It is expected that the maximum strain of 2.7 % for the PAn tube with the Pt helix can be increased by decreasing potential scan rate because the transport of the dopant will be more efficient and in phase with electrical stimulus at a slower scan rate.

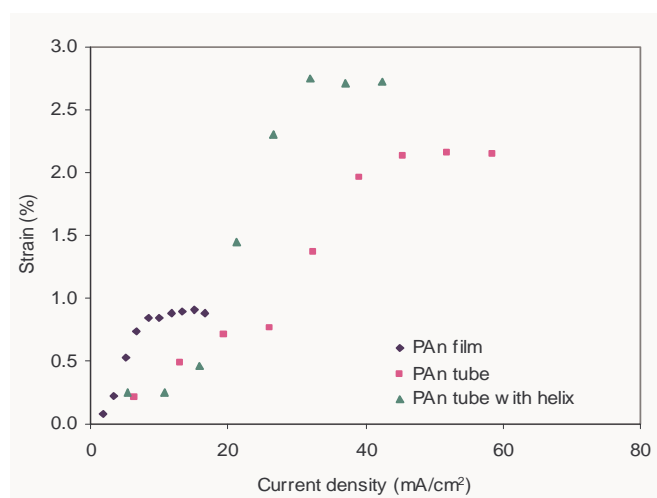
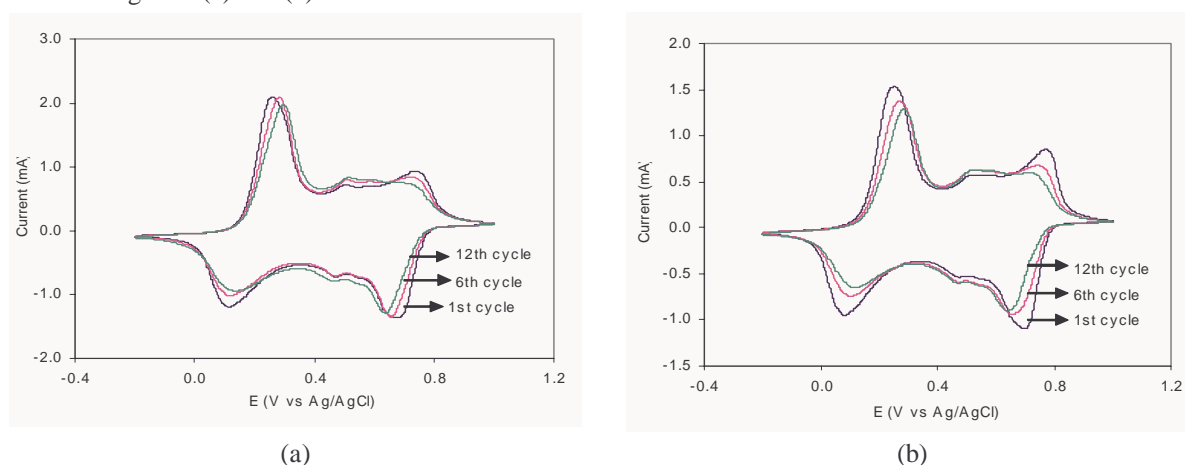


Fig. 1: Actuation strain of PAn/HCl film, tube and tube with Pt helix in  $\text{HNO}_3$  (1 M) aqueous solution under increasing current density

Cyclic voltammograms of PAn films doped with HCl (PAn/HCl) and MSA (PAn/MSA) in  $\text{HNO}_3$  (1 M) are shown in Figure 2 (a) and (b) below.



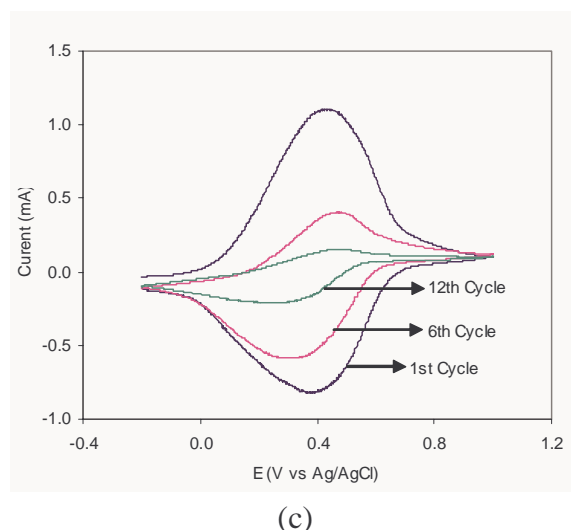


Fig.2: Cyclic voltammograms of PAn recorded at 50mV/s sweep rate  
(a): PAn/HCl in 1M HNO<sub>3</sub>, (b): PAn/MSA in 1M HNO<sub>3</sub>, (c): PAn/HCl in 1M NaNO<sub>3</sub>

Potential was swept between  $-0.2$  V to  $1.0$  V at  $50$  mV/s sweep rate for 12 cycles. Three redox pairs were observed with oxidation peaks at  $0.2$  V,  $0.5$  V and  $0.7$  V. The first peak ( $0.2$  V) corresponds to the first step of oxidation of neutral PAn, from polyleucoemeraldine base to polyemeraldine salt. The third peak ( $0.7$  V) corresponds to the further oxidation of PAn from polyemeraldine salt to polypyrnigraniline base. The middle pair is from the presence of ortho-coupled polymer or the degradation of PAn. These results correspond well with results by Pruneanu et al [18] when PAn was tested in HClO<sub>4</sub> ( $1$  M) solution. In contrast, a featureless CV was observed in NaNO<sub>3</sub> ( $1$  M) reflecting low electro-activity. Figure 2 (c) shows a cyclic voltammogram of PAn/HCl in NaNO<sub>3</sub> ( $1$  M) solution. Only one pair of redox peaks was observed with oxidation peak at  $0.5$  V. Compared to CVs of PAn/HCl and PAn/MSA in acid aqueous solution (Figures 2a and b), the current decreased quickly from the first cycle to the sixth cycle and again to the twelfth cycle, indicating poor electrochemical property and stability of PAn in NaNO<sub>3</sub>. The poor electroactivity of PAn in nitrate is due to the non-acidic pH that results in the conversion of the polyemeraldine salt to the non-conductivity polyemeraldine base.

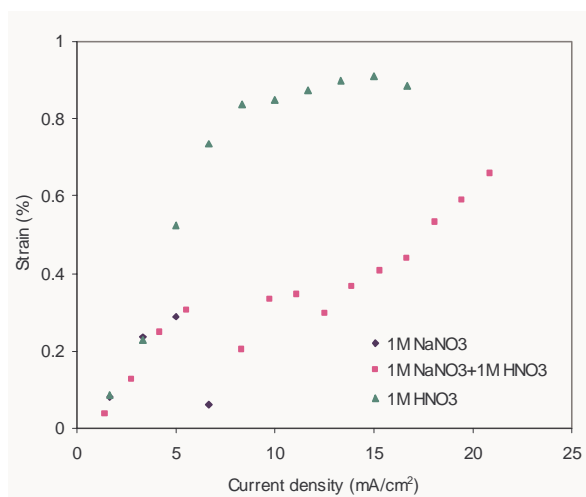


Fig. 3: PAn/HCl film strain under increasing current density in different electrolytes

Acid aqueous solution and salt aqueous solution as electrolytes are compared for actuation in Figure 3. The PAn films doped in HCl (PAn/HCl) were tested in  $\text{HNO}_3$  (1 M),  $\text{NaNO}_3$  (1 M) and mixture of  $\text{HNO}_3$  (1 M) and  $\text{NaNO}_3$  (1 M) aqueous solutions. In  $\text{HNO}_3$ , PAn film gives the highest strain (0.9 %) but in  $\text{NaNO}_3$  the strain is the lowest (0.3 %). When tested in the mixture of  $\text{HNO}_3$  and  $\text{NaNO}_3$ , the electrolyte pH is half of 1M  $\text{HNO}_3$ , the actuation strain is thus between the best and worst performance (0.65 %). This comparison confirms that acid electrolyte can maintain PAn doping level during the actuation test. In contrast, in neutral  $\text{NaNO}_3$  electrolyte, PAn gradually becomes deprotonated and non-conductive. Similar results were observed in the PAn doped in MSA. This is different from our previous work [13] that PPy/PAn composite actuators perform very good stability in  $\text{NaNO}_3$  as electrolyte where PPy dominates the actuation behaviour.

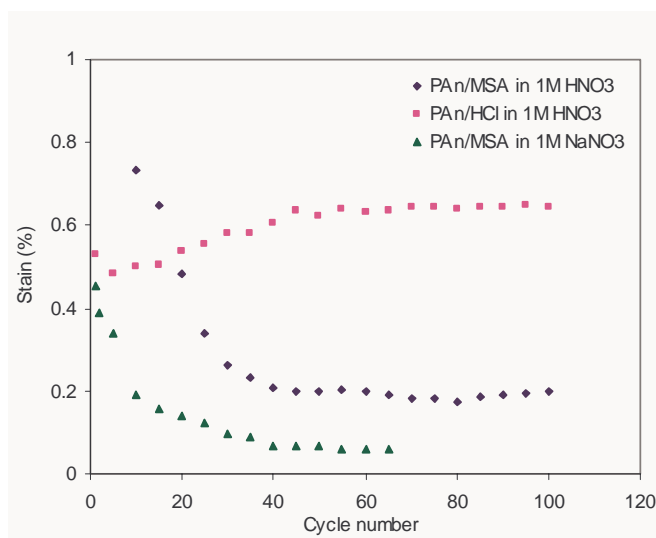


Fig. 4: Electrochemical stability of PAn samples in different electrolytes

As expected and shown in Figure 4, due to low electro-activity (Fig. 2), the actuation strain decreases rapidly by cycling at a fixed current density in  $\text{NaNO}_3$  aqueous solution while the stability of the PAn film actuator maintained for at least 100 cycles in  $\text{HNO}_3$  aqueous solution. For the PAn/HCl sample, a slight increase in the strain during the first 45 cycles is due to the replacement of  $\text{Cl}^-$  with a larger electrolyte dopant  $\text{NO}_3^-$  increases the expansion/contraction state to 0.6%. On the other hand, the replacement of  $\text{MSA}^-$  with a smaller  $\text{NO}_3^-$  molecule decreases the actuation strain of PAn/MSA to 0.2 % during the first 45 cycles then stays stable.

The microstructure of the films prepared by the phase inversion technique are best thought of as porous asymmetric membrane [19]. The top surface in Figure 6 corresponds to the side of the film in contact with the coagulation bath, and the bottom side corresponds to the side in contact with the substrate. Clearly, the size of the pores increases from the more dense top side to the more porous bottom side. The thickness of the dense top layer is larger for the pure water bath, than for the water/NMP bath. Similar observation was reported by Kim et al [20]. Similarly, the porosity of the top layer is larger for the water/NMP bath relative to the water bath due to a slower quenching, indeed some evidence of the deep macrovoid structure is already present (Figure 5). Figure 7 shows the effect of current density of the PAn/MSA films coagulated in a water bath or in a NMP (30 % w/w)/water bath. The maximum actuation strain is approximately 0.7 % for both samples prepared in different bath.

Weakness of the PAn/MSA films was observed with the maximum applied load is approximately 0.3 MPa before the samples failed (Fig. 7). The value for PAn/HCl films and tubes is slightly better at 0.4 MPa but it is still very low compared with PPy actuator (1 – 1.5 MPa) [20] and PPy/PAn composite actuator (11.8MPa) [13]. Table 2 listed the Young's modulus,  $E$ , of the PAn/MSA films. Comparing with other conducting polymers such as polypyrrole in propylene carbonate electrolyte of which  $E$  is around 80 MPa, the low values of  $E$  reflect the porous nature of the samples.



Table 2: Young's modulus,  $E$ , at expansion (oxidized) and contraction (reduced) states in  $\text{HNO}_3$  (1 M) aqueous solution

Sample	$E_{\text{expansion}}$ (MPa)	$E_{\text{contraction}}$ (MPa)
PAn film*	3.06	3.45
PAn film**	1.75	1.93

\* coagulated in water bath doped with MSA (1 M) for 6 hours.

\*\* coagulated in NMP (30%)/water bath doped with MSA (1 M) for 6 hours.

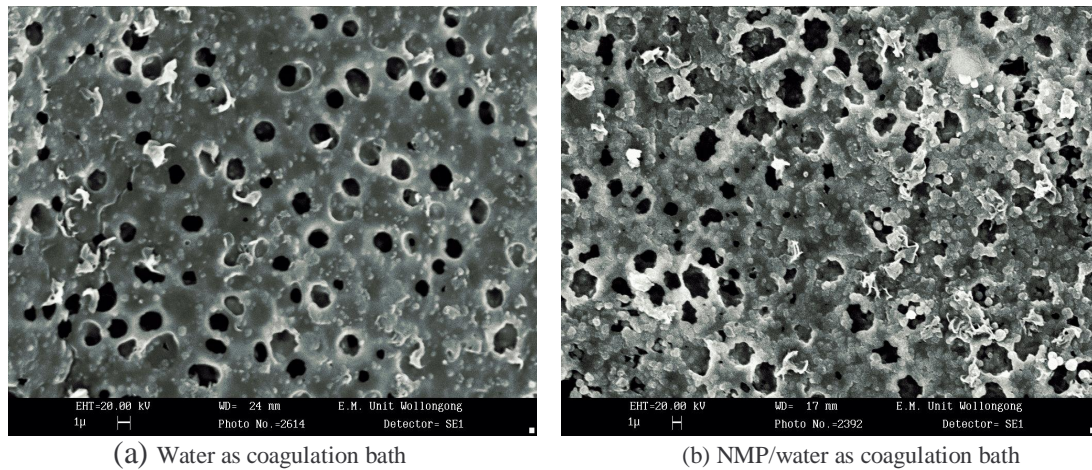


Fig. 5: SEM surface images of PAn EB films coagulated from (a) a water bath and (b) a NMP (30 %)/water bath.

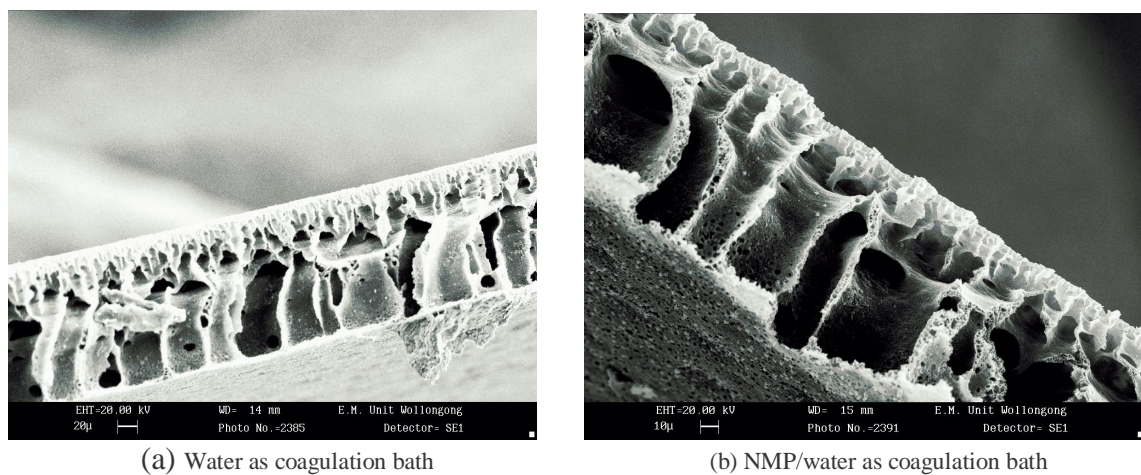


Fig. 6: SEM cross section images of PAn EB films coagulated from (a) a water bath and (b) a NMP (30 %)/water bath.

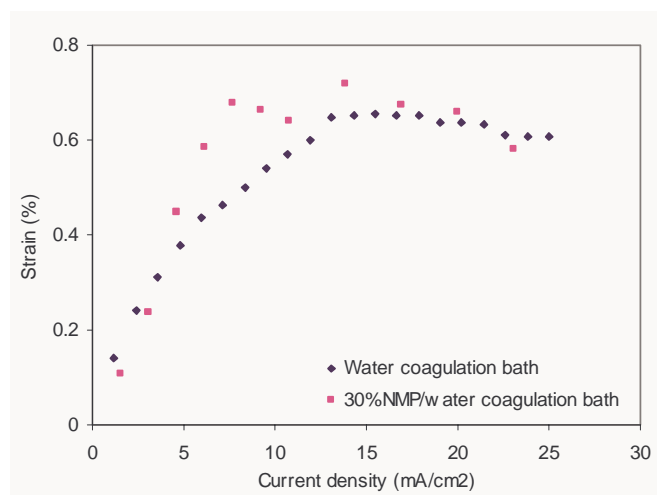


Fig. 7: Actuation strain of PAN/MSA films coagulated in water or NMP (30 %)/water bath in  $\text{HNO}_3$  (1 M) aqueous solution under increasing current density

The higher value of  $E$  at the contraction state than that at the expansion state predicts the actuation strain will increase with the applied load [2, 20]. As shown in Fig. 8, an initial increase of the strain with increasing applied load was observed up to 0.05 MPa. The ensuing decline of the strain may be due to appearance of microcracks resulting from the brittle nature of the sample.

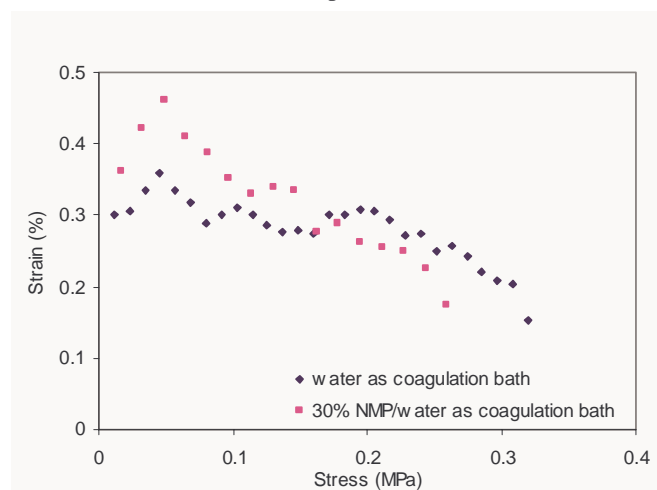


Fig. 8: Effect of applied load on the actuation strain of PAN/MSA films coagulated in water or NMP (30 %)/water bath in  $\text{HNO}_3$  (1 M) aqueous solution

### 3. CONCLUSIONS

The phase inversion technique is a flexible method for fabricating PAN into different geometrical configurations that cannot be obtained by the PAN cast from NMP solution in a conventional way. The conductivity can be increased by casting into a tube and by incorporating a Pt helix as an interconnector. The increased conductivity leads to a higher actuation strain.  $\text{HNO}_3$  aqueous electrolyte, in which PAN keeps doping level, provides a stable actuation environment.

Brittle and porous nature of the PAN samples prepared by the phase inversion method is a serious setback for practical applications. It is expected that high molecular weight (MW = 300,000) of the PAN EB would render

reasonable mechanical properties, but fabricating processes and dopants seem to be the dominant variables in determining the strength of the PAn materials. Spinning process to make PAn fibres where advantages of high molecular weight can be fully exploited is one of the methods to obtain a tough material by molecular alignments. The actuation behaviour of spun PAn fibres therefore merits further studies.

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