

# Electrochromic Characteristics of Polyaniline and Poly(3-Methylthiophene) Thin Films for Display

Jung-Chuan Chou, *Member, IEEE*, Chai-Yu Liu, Cheng-Jung Yang, Yi-Hung Liao, *Associate Member, IEEE*, Meng-Wei Su, and Chien-Cheng Chen, *Member, IEEE*

**Abstract**—In this study, the polyaniline (PANI) and the poly(3-methylthiophene) (PMeT) of the conductive polymer electrochromic thin films had been deposited on the indium tin oxide/glass (ITO/Glass) substrate by electrochemical polymerization, respectively. The cycling stability of the electrochromic thin film prepared with the variety of the different charge densities was investigated. The electrochromic behaviors of the PMeT/ITO/Glass and PANI/ITO/Glass were performed in the electrolytes of 0.1 M lithium perchlorate ( $\text{LiClO}_4$ )/propylene carbonate (PC) and 0.1 M  $\text{LiClO}_4$  + 0.001 M perchloric acid ( $\text{HClO}_4$ )/PC, respectively. The colors of the PMeT thin film exhibited blue and red in oxidation and reduction states, respectively. And the colors of the PANI thin film exhibited green and yellow in oxidation and reduction states, respectively. Furthermore, the absorbance and transmittance (T%) of the electrochromic thin film were measured by ultraviolet-visible (UV-Visible) spectrometer in this study. Besides, in order to find the optimal manufacture conditions of the electrochromic thin film, the potentials of redox reaction and cycles were investigated by the cyclic voltammetry (CV) measurement system to analyze the cycling stability of the electrochromic thin film with the variety of the different charge densities. Finally, the reflectance (R%) of the optimal parameters of the electrochromic thin film was measured by chroma meter in this study. And the variational colors of the electrochromic thin film were observed by the chromaticity coordinates of the Commission Internationale de l'Éclairage (CIE), so that the electrochromic thin film can be applied to the filed of display.

**Index Terms**—Conductive polymer, electrochemical polymerization, electrochromism, poly(3-methylthiophene), polyaniline.

## I. INTRODUCTION

THE conjugated polymers are organic macromolecules, which consist of one or more backbone chains of alternating double and single bonds. Conjugated polymers have been widely used in variety of applications, such as polymer

conductors [1], electronic components [2], [3], light-emitting diodes [4], batteries [5], and polymer electrochromic devices (PECDs) [6], because they exhibit several advantages, such as thermal stability, low cost and easy preparation.

Various methods have been proposed to deposit the conjugated polymers thin film, such as chemical and electrochemical polymerizations. The conjugated polymers prepared by chemical or electrochemical polymerization have received significant attention due to the wide range of electrical, electrochemical, and optical properties [7], [8]. In this study, the thin film of the conjugated polymers was deposited on the indium tin oxide/glass (ITO/Glass) substrate by electrochemical polymerization. The advantages of electrochemical polymerization comparing with other methods include rapidity, simplicity, generation of the polymer directly on the electrode in the doped or undoped states, and easy controlled synthesis of these compounds [9]. According to these results, the preparation, characterization and application of electrochemically active and electronically conjugated polymeric systems are still investigated in electrochemistry [10].

Polyaniline (PANI) shows yellow color in the reduction state and green color in the oxidation state [11]–[17]. It can be applied to electrochromic displays [18]. According to the mentioned above, the electrochromic PANI thin film can be applied in display for green pixel.

In this study, the PANI thin film has been electrodeposited on the indium tin oxide/glass (ITO/Glass) substrate with the various deposition charges to optimize electrochromic property of the PANI thin film. Furthermore, the electrochromic property of the PANI thin film has been studied in a 0.1 M lithium perchlorate ( $\text{LiClO}_4$ )/propylene carbonate (PC) electrolyte solution, and color of the PANI thin film was switched between green [1.0 V (PANI versus Platinum (Pt))] and yellow [−0.5 V (PANI versus Pt)].

Polythiophene derivatives are the most challenging class of conjugated polymers [19], and the Poly(3-methylthiophene) (PMeT) is one of the Polythiophene derivatives, as well as, the color of the PMeT thin film was determined by methyl groups which introduced in polythiophene backbone. The colors of the PMeT thin film includes pale blue, blue and violet in the oxidation state, and purple, yellow, red and orange in reduction state, respectively. The different colors of the PMeT thin film relates with changes of effective conjugation length of the polymer chain [20]. In this study, the PMeT has been electrodeposited on ITO/Glass. The PMeT thin film shows bright red color in the reduction state and turns to light blue in the oxidation state [21]. The PMeT electrodeposited in boron fluoride ethyl ether

Manuscript received July 21, 2013; revised May 16, 2014; accepted February 16, 2015. Date of publication March 02, 2015; date of current version April 24, 2015. This work was supported by the National Science Council, Republic of China, under Contract NSC 100-2221-E-224-017, Contract NSC 101-2221-E-265-001, and Contract NSC 102-2221-E-224-075.

J.-C. Chou, C.-Y. Liu, and C.-C. Chen are with the Graduate School of Electronic and Optoelectronic Engineering, National Yunlin University of Science and Technology, Douliou, 64002, Taiwan (e-mail: choujc@yuntech.edu.tw; M10013303@yuntech.edu.tw; g9710803@yuntech.edu.tw).

C.-J. Yang and M.-W. Su are with the Graduate School of Electronic and Optoelectronic Engineering, National Yunlin University of Science and Technology, Douliou, 64002, Taiwan 64002 (e-mail: m10113321@yuntech.edu.tw; g9918737@yuntech.edu.tw).

Y.-H. Liao is with the Department of Information Management, TransWorld University, Yunlin, 64063, Taiwan (e-mail: liaoh@twu.edu.tw).

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Digital Object Identifier 10.1109/JDT.2015.2407911

(BFEE) was reported by Alkan *et al.* [22]. They had proposed that PMeT film was electrodeposited in BFEE solution, and had better electroactivity and cycling stability [21].

In this study, the PMeT thin film has been electrodeposited on the ITO/Glass substrate with the various deposition charges to optimize electrochromic property of the PMeT thin film. Furthermore, the electrochromic property of the PMeT thin film has been studied in the 0.1 M lithium perchlorate ( $\text{LiClO}_4$ )/propylene carbonate (PC) electrolyte solutions, and color of the PMeT thin film was switched between light blue [ $+0.8$  V [PMeT versus Platinum (Pt)]] and red [ $0.4$  V (PMeT versus Pt)]. According to the results, the electrochromic PMeT thin film can be applied in display.

## II. EXPERIMENTAL

### A. Materials

Indium tin oxide/glass (ITO/Glass) substrate was manufactured by Sinonar Corp., Taiwan, and its sheet resistivity is  $7 \Omega/\square$ . Aniline (ANI) solution, hydrogen chloride, (HCl) solution, 3-methylthiophene (MeT) solution, boron fluoride ethyl ether (BFEE) solution, lithium perchlorate ( $\text{LiClO}_4$ ) powders and propylene carbonate (PC) solution were all purchased from Acros Organics Corp., USA. Cu tape was purchased from Centenary Materials Corp., Taiwan.

### B. Instrumentation

BioLogic potentiostat/Galvanostat (model SP-150, France) was used to electrodeposit the PANI thin film on the ITO/Glass and to perform cyclic voltammetry (CV) measurements, respectively. OTO Photonic Spectrometer with D65 light source (model SD1200-LS-HA, Taiwan) was used to measure chromaticity coordinates of the Commission Internationale de l'Eclairage (CIE) and transmittance of electrochromic thin film.

### C. Preparation of the PANI Thin Film

The PANI thin film was deposited on the ITO/Glass by potentiostatic polymerization in 1 M ANI and 2 M HCl with deionized (D. I.) water, and the potential was set at 1.2 V. Furthermore, the charge densities were controlled at 10, 20, 30, 40, 50, 60, and 70  $\text{mC}/\text{cm}^2$ . The purchased ITO/Glass substrate was cut into  $1.4 \text{ cm} \times 5 \text{ cm}$ , and the active area was  $1 \text{ cm} \times 4.6 \text{ cm}$ . The manufacturing conditions of the PANI thin film are listed in Table I. The PANI thin film electrode was removed from the monomer/electrolyte solution after electrochemical polymerization and rinsed with 0.1 M HCl to produce cleaned surface without monomer. The PMeT/ITO/Glass (or PANI/ITO/Glass) and ITO/Glass counter electrode were sandwiched together by utilizing the 3 M tape (thickness =  $30 \mu\text{m}$ ) as shown in Fig. 1.

The liquid electrolyte solution was injected into the cell, and then it was sealed using cover plastics. The electrolyte was injected into the vacant space between two electrodes, which meant that the quantity of electrolyte in ECD was controlled by thickness of 3 M tape.

### D. Preparation of the PMeT Thin Film

ITO/Glass was cleaned by acetone, alcohol and deionized water for 10 minutes, respectively, and then ITO/Glass was dried by nitrogen. The PMeT thin film was deposited on

TABLE I  
MANUFACTURING CONDITIONS OF PANI THIN FILM

Parameters	Conditions
Molar volume concentration of PANI (M)	1
Molar volume concentration of HCl (M)	2
Solvent (mL)	50
Constant Voltage (V)	1.2
Charge density ( $\text{mC}/\text{cm}^2$ )	10~70
Temperature ( $^\circ\text{C}$ )	25

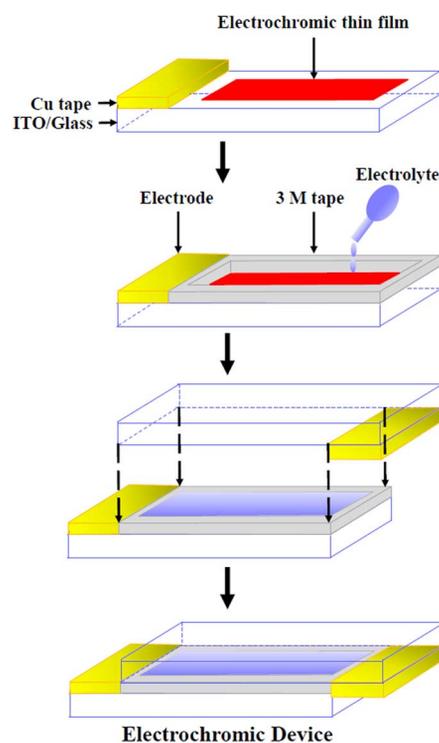


Fig. 1. Sketch of assembling ECD.

TABLE II  
MANUFACTURING CONDITIONS OF PMeT THIN FILM

Parameters	Conditions
Molar volume concentration of PMeT (M)	0.01
Solvent (mL)	50
Constant Voltage (V)	1.39
Charge density ( $\text{mC}/\text{cm}^2$ )	10~70
Temperature ( $^\circ\text{C}$ )	25

the ITO/Glass by potentiostatic polymerization in 0.01 M MeT/BFEE electrolyte, and the potential was set at  $+1.39$  V. Furthermore, the charge densities were controlled at 10, 20, 30, 40, 50, 60 and 70  $\text{mC}/\text{cm}^2$ . The manufacturing conditions of the PMeT thin film are listed in Table II. The PMeT thin film electrodes were removed from the monomer/electrolyte solution after electrochemical polymerization and rinsed with acetone to produce clean surfaces without monomer. The PMeT/ITO/Glass (or PANI/ITO/Glass) and ITO/Glass counter electrode were sandwiched together by utilizing the 3 M tape (thickness =  $30 \mu\text{m}$ ). and the entities of assembling pixel of tertiary colors as shown in Fig. 2.

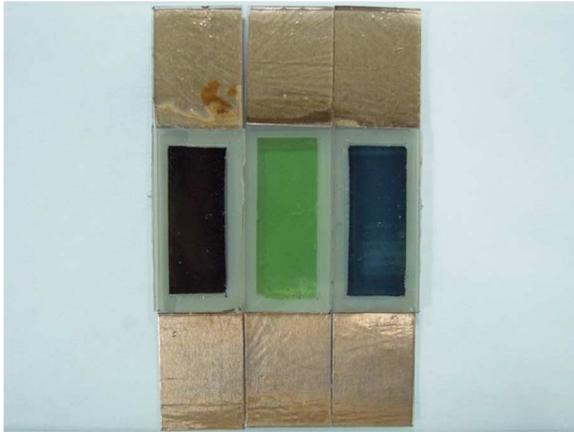


Fig. 2. Entities of assembling pixel of tertiary colors.

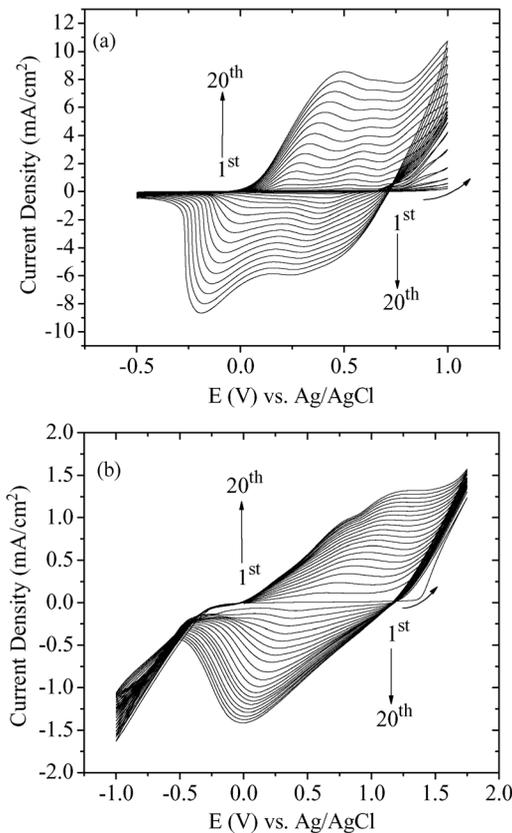


Fig. 3. (a) Cyclic voltammograms of PANI thin film during electrodeposition (scan rate = 100 mV/s); (b) Cyclic voltammograms of PMeT thin film during electrodeposition (scan rate = 100 mV/s).

### III. RESULTS AND DISCUSSION

#### A. Polymerization Potetial of PANI and PMeT Thin Film

The PANI and PMeT thin films were obtained by electrochemical polymerization at potential slightly higher than the monomer oxidation onset potential, and obtained a homogeneous thin films on the the ITO/Glass. The redox behavior of the ANI monomer was obtained in 1 M ANI and 2 M HCl with D. I. water by cyclic voltammetry, as shown in Fig. 3(a). An oxidation onset potential was obtained about 1.0 V. Because lower

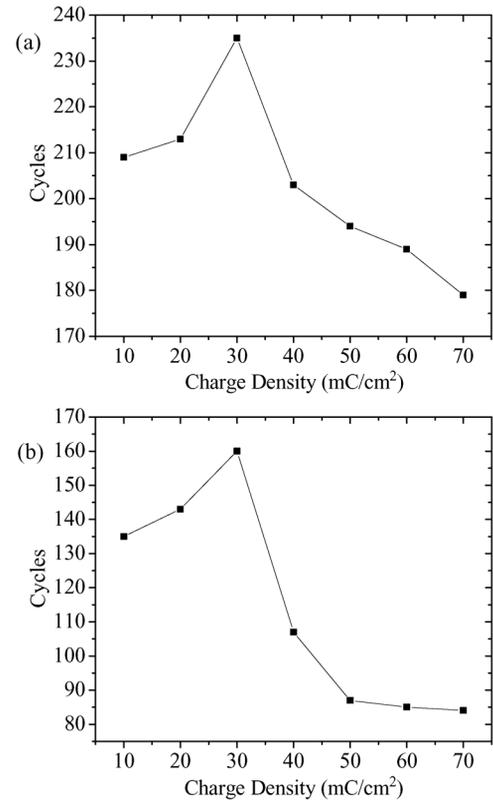


Fig. 4. (a) Cycles of PANI thin film was changed with the different charge densities. (b) Cycles of PMeT thin film was changed with the different charge densities.

potential could not synthesize the ANI, the potential of the polymerization of the PANI thin film was set at 1.2 V. The CV curves of the PANI thin film exhibited that the onset potential of oxidation of ANI was at +1.1 V in 1 M ANI and 2 M HCl with D. I. water, also, that the oxidation peak of the PANI thin film at +0.3 V and reduction peak of the PANI thin film was at +0.25 V during the first cycle. The redox behavior of the MeT monomer was obtained in MeT(0.01 M)/BFEE solution by cyclic voltammetry, as shown in Fig. 3 (b). An oxidation onset potential was obtained about +1.3 V. Since lower potential could not synthesize the MeT, the potential of the polymerization of the PMeT thin film was set at +1.39 V. The CV curves of the PMeT thin film exhibited that the onset potential of oxidation of MeT at +1.3 V in BFEE solution, as well as, the oxidation peak of the PMeT thin film at +0.7 V and reduction peak of the PMeT thin film at +0.3 V at the first cycle.

The CV curves of the PANI and PMeT thin film indicated that the thickness of the PANI and PMeT thin film were increased with increasing current intensity, and the PANI and PMeT thin films were formed on the ITO/Glass [23]. The CV curves also exhibited that the broad anodic and cathodic peaks of the PANI and PMeT thin films corresponded to the oxidation and the reduction of the PANI and PMeT thin films, respectively. The currents of redox reaction of PANI and PMeT thin films were increased proportionally with increasing scanning cycles. Moreover, the oxidation potentials of the PANI and PMeT thin films were shifted to positive potential, and the reduction potentials of the PANI and PMeT thin films were shifted to negative potential [24]. This phenomenon exhibited that conductivity, counter-ion



TABLE III.  
COMPARISON OF THE COLORATION EFFICIENCY OF THE PANI/ITO/GLASS IN THIS STUDY AND PREVIOUS LITERATURES [26]–[28]  
FOR ELECTROCHROMIC DEVICES

Electrochromic Device	Method	Coloration Efficiency (cm <sup>2</sup> /C)	Wavelength (nm)	Transmittance Variation ( $\Delta T$ (%))	Time of Manufacture	Cost of Manufacture	Ref.
PANI/ITO/Glass	Electrochemistry	7.50	550	22	short	low	In this study
PANI/ITO/Glass	Chemistry	42.80	550	41	long	high	[26] (2009)
PANI/G/Glass	Chemistry	39.60	550	45	long	high	[26] (2009)
PANI/ITO/Glass	Screen-Printed	52.00	550	N/A	long	high	[27] (2009)
POSS-PANI/ITO/Glass	Screen-Printed	69.00	550	N/A	long	high	[27] (2009)
PANI/ITO/Glass	Electrochemistry	6.20	550	N/A	short	low	[28] (2009)

N/A: Not Applicable

TABLE IV.  
COMPARISON OF THE COLORATION EFFICIENCY OF THE PMeT/ITO/GLASS IN THIS STUDY AND PREVIOUS LITERATURES [21], [29]  
FOR ELECTROCHROMIC DEVICES

Electrochromic Device	Method	Coloration Efficiency (cm <sup>2</sup> /C)	Wavelength (nm)	Transmittance Variation ( $\Delta T$ (%))	Time of Manufacture	Cost of Manufacture	Ref.
PMeT/ITO/Glass	Potentiostatic Method	79.06	655	26	short	low	In this study
PMeT/ITO/Glass	Potentiostatic Method	135.00	655	N/A	short	high	[21] (2006)
PMeT/ITO/Glass	Potentiostatic Method	92.00	650	45	long	high	[29] (2010)

N/A: Not Applicable

### C. Colored and Bleached Transmittances of the PANI/ITO/Glass and PMeT/ITO/Glass

Fig. 6(a) shows the colored and bleached transmittances of the PANI/ITO/Glass, the PANI/ITO/Glass was performed in 0.1 M LiClO<sub>4</sub>/PC electrolyte solution. The potential was between  $-0.5$  V and  $+1.0$  V. The solid shapes and hollow shapes were coloring and bleaching, respectively. The PANI/ITO/Glass has the optical transmittance variation [ $\Delta T$ (%)] (3%) at 550 nm when the constant potential and the charge density of the PANI thin film were set at 1.2 V and 30 mC/cm<sup>2</sup>, respectively. Fig. 6(b) shows the colored and bleached transmittances of the PMeT/ITO/Glass, the PMeT/ITO/Glass was performed in 0.1 M LiClO<sub>4</sub>/PC electrolyte solution. The potential was between  $-0.4$  V and  $+0.8$  V. The solid shapes and hollow shapes were coloring and bleaching, respectively. The PMeT/ITO/Glass has the optical transmittance variation [ $\Delta T$ (%)] (26%) at 655 nm when the constant potential and the charge density of the PANI thin film were set at 1.39 V and 30 mC/cm<sup>2</sup>, respectively.

### D. Color Analysis of the PANI/ITO/Glass and PMeT/ITO/Glass

The variational colors of the electrochromic thin film were observed by the chromaticity coordinates of the Commission Internationale de l'Eclairage 1931 (CIE 1931). Fig. 7(a) shows the chromaticity coordinates of CIE of PANI/ITO/Glass (charge density of 30 mC/cm<sup>2</sup>) were performed in 0.1 M LiClO<sub>4</sub>/PC

electrolyte solution. The PANI/ITO/Glass in yellow color state (reduction state) was at  $-0.5$  V (PANI versus Pt), and the chromaticity coordinate of the PANI/ITO/Glass was (0.35, 0.47). Green color state (oxidation state) of PANI thin film was at  $+1.0$  V (PANI versus Pt), and the chromaticity coordinate of the PANI/ITO/Glass was (0.04, 0.55). Fig. 7(b) shows the chromaticity coordinates of CIE of PMeT/ITO/Glass (charge density of 30 mC/cm<sup>2</sup>) was performed in 0.1 M LiClO<sub>4</sub>/PC electrolyte solution. The PMeT/ITO/Glass in red color state (reduction state) was at  $-0.4$  V (PMeT vs. Pt), and the chromaticity coordinate of the PMeT/ITO/Glass was (0.53, 0.21). When blue color state (oxidation state) of PMeT thin film was at  $+0.8$  V (PMeT versus Pt), and the chromaticity coordinate of the PMeT/ITO/Glass was (0.35, 0.13). According to the experimental results, the electrochromic PANI and PMeT thin films can be applied in display.

### E. Coloration Efficiency of the PANI/ITO/Glass and PMeT/ITO/Glass

Compare with PANI/ITO/Glass and PMeT/ITO/Glass, the fabricated time of PANI/ITO/Glass and PMeT/ITO/Glass are short and the cost is low to have excellent coloration efficiency in this study. And the coloration efficiency of the PANI/ITO/Glass and PMeT/ITO/Glass were lower than other literatures [21], [26]–[29] as summarized in Tables III and IV. The previous literatures [21], [26], [27], [29] had large

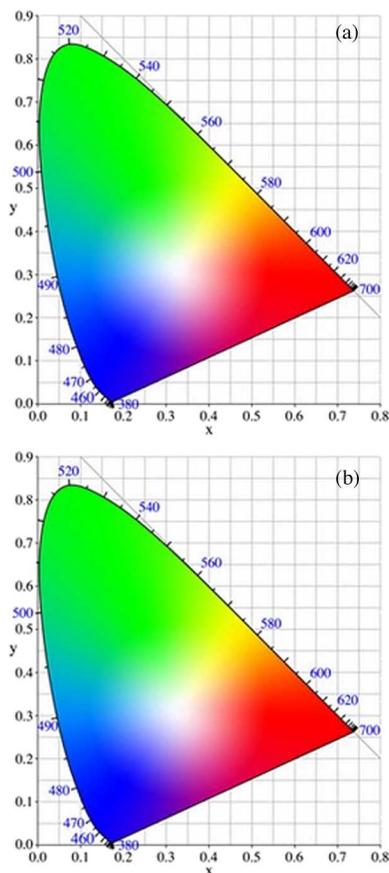


Fig. 7. (a) Chromaticity Coordinates of CIE 1931 of PANI/ITO/Glass. (b) Chromaticity Coordinates of CIE 1931 of PMeT/ITO/Glass.

coloration efficiency due to porosity of electrochromic thin films. The electrochromic thin films of previous literatures [21], [26], [27], [29] had the highest degree of porosity than this study and previous literature [28].

#### IV. CONCLUSION

In this study, the electrochromic PANI and PMeT thin films have been successfully deposited on ITO/Glass. The PANI/ITO/Glass has the best cycling stability (charge density of  $30 \text{ mC/cm}^2$ ) when the constant potential and the charge density of the PANI/ITO/Glass were set at 1.2 V and  $30 \text{ mC/cm}^2$ , respectively. The PMeT/ITO/Glass has the best cycling stability (charge density of  $30 \text{ mC/cm}^2$ ) when the constant potential and the charge density of the PMeT/ITO/Glass were set at 1.39 V and  $30 \text{ mC/cm}^2$ , respectively. The coloration efficiency of the PANI/ITO/Glass was  $7.50 \text{ cm}^2/\text{C}$ . The experimental results obtained the colors of the PANI/ITO/Glass was from green (oxidation state) to yellow (reduction state). The coloration efficiency of the PMeT/ITO/Glass was  $79.06 \text{ cm}^2/\text{C}$ . The experimental results obtained the colors of the PMeT/ITO/Glass were from blue (oxidation state) to red (reduction state). According to the experimental results, the electrochromic PANI and PMeT thin films can be applied in display for green pixel in the future.

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**Jung-Chuan Chou** (GSM'09–M'11) was born in Tainan, Taiwan, R.O.C., in 1954. He received the B.S. degree in physics from Kaohsiung Normal College, Kaohsiung, Taiwan, R.O.C., in 1976; the M.S. degree in applied physics from Chung Yuan Christian University, Chung-Li, Taiwan, R.O.C., in 1979; and the Ph.D degree in electronics from National Chiao Tung University, Hsinchu, Taiwan, R.O.C., in 1988.

He taught at Chung Yuan Christian University from 1979 to 1991. Since 1991, he worked as an associate professor in the Department of Electronic Engineering at the National Yunlin University of Science and Technology. From 1997 to 2002, he was Dean, Office of Technology Cooperation at the National Yunlin University of Science and Technology. From 2002 to 2009, he was Chief Secretary at the National Yunlin University of Science and Technology; and from 2009 to 2010 he was Director of library at the National Yunlin University of Science and Technology. Since 2010, he has been a professor with the Department of Electronic Engineering, National Yunlin University of Science and Technology. From 2010 to 2011, he was Director of Office of Research and Development at the National Yunlin University of Science and Technology, and since 2011 he has worked as distinguished professor in the Department of Electronic Engineering at the National Yunlin University of Science and Technology, Douliou, Taiwan. His research interests are in the areas of sensor material and device, biosensor and system, microelectronic engineering, optoelectronic engineering, solar cell and solid state electronics.



**Chai-Yu Liu** was born in Tainan, Taiwan, in 1988. He received the B.S. degree from the Department of Electronic Engineering, National National Yunlin University of Science and Technology, Yunlin, Taiwan, in 2011, and the M.S. degree in microelectronic and optoelectronic engineering from the Graduate School of Electronic and Optoelectronic Engineering, National Yunlin University of Science and Technology, Douliou, Taiwan, in 2013.

His research interests are in electrochromism, conductive polymer and display.



**Cheng-Jung Yang** was born in Hualien, Taiwan, in 1989. He received the B.S. degree from the Department of Electrical Engineering, Southern Taiwan University of Science and Technology, Tainan, Taiwan, in 2012, and the master degree from the Graduate School of Electronic and Optoelectronic Engineering, National Yunlin University of Science and Technology, Yunlin, Taiwan, in 2014.

His research interests are in electrochromism, conductive polymer and ZnO nanowires.



**Yi-Hung Liao** (A'12) was born in Yunlin, Taiwan, R.O.C., in 1963. He received the B.S. degree in electronic engineering from National Taiwan Institute of Technology, Taipei, Taiwan, in 1990; the M.S. degree in electronic engineering from National Yunlin Institute of Technology, Yunlin, Taiwan, in 1997, and the Ph.D degree from the Graduate School of Engineering Science and Technology, National Yunlin University of Science and Technology, Yunlin, Taiwan, in 2010.

His research interests include the chemical sensors and its applications, the array sensors and multi-sensors for biosensing, the characterization of biosensors, and implement home care system with PIC microprocessor.



**Meng-Wei Su** was born in Yunlin, Taiwan, in 1988. He received the B.S. degree from the Department of Electro-Optical Engineering, National Formosa University, Huwei, Taiwan, in 2010, and the M.S. degree in microelectronic and optoelectronic engineering from the Graduate School of Electronic and Optoelectronic Engineering, National Yunlin University of Science and Technology, Douliou, Taiwan, in 2012.

His research interests are in electrochromism and chlorine sensor.



**Chien-Cheng Chen** (GSM'09–M'11) was born in Taichung, Taiwan, R.O.C., on August 20, 1984. He received the B.S. degree in the Department of Electronic Engineering at the National Yunlin University of Science and Technology, Yunlin, Taiwan, in 2006, the Master's degree from the Graduate School of Optoelectronics at the National Yunlin University of Science and Technology, in 2008, and the Ph.D degree from the Graduate School of Engineering Science and Technology, National Yunlin University of Science and Technology, Yunlin, Taiwan, in 2012.

His research interests are semiconductor devices, biosensors and their applications.