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REGULAR PAPER

One Million Cycle Durability Test of Electrochromic Devices Using Charge Balance Control

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Abstract

We fabricated 10 mm × 10 mm WO_3 electrochromic devices (ECDs) using a nanoparticle deposition system (NPDS), which is a coating method that uses kinetic spray. NPDS has the advantage of mass production due to its relatively low pressure and low temperature working conditions but can result in porous surfaces, which has a negative effect on device durability. We developed an optical transmittance measurement and voltage control system that allows dynamic control. Transmittance was measured by Optical Power Meter, 2936-R Newport, while voltage was dynamically applied by NI9477, National Instrument. Current was measured by high resolution current, National Instrument. This system enables ECDs to be feedback controlled depending on the measured electronic properties. We then characterized the WO_3 ECDs, focusing on the relationship between transmittance and current under well-known static operating conditions. Finally, we proposed a new algorithm to extend durability performance by adopting charge balance control and conducted an accelerated life test over 1,000,000 cycles. According to our results, the optical performance was maintained until the test ended, and there was a 45.4% difference in the maximum and minimum transmittance of the sample after the accelerated life test. Therefore, the charge balance control enhanced the lifetime of the ECDs by avoiding negative charge accumulation, verifying that the durability of ECDs could be enhanced by adopting dynamic control.

Keywords Electrochromic devices · Tungsten oxide · Nanoparticle deposition system · Accelerated life test · Durability test

1 Introduction

Electrochromic windows have recently received significant attention as environmentally friendly nanotechnology and energy conservation method for building construction [1, 2]. Electrochromic windows can be used to automatically control the temperature inside a building and conserve energy consumption by harnessing the penetrability of sunlight [3]. Prospective applications of electrochromic devices (ECDs)

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include glass for automobiles, spacecraft, airplanes and optical instruments, such as sunglasses and displays [4].

Electrochromism is defined as a change in optical transmittance induced by an applied voltage. Electrochromic effects have been observed in a range of compounds, from transition oxide materials to organic materials and polymers. CoO, In_2O_3, MoO_3, NiO, WO_3, CeO_2 and MnO_2 are studied frequently as metal oxide films that exhibit electrochromic properties, including various color changes [5]. The electrochromism of tungsten trioxide, WO_3, was first studied in the 1970s; subsequently, the optical and electrochemical properties of WO_3 obtained using various fabrication methods have been investigated [6, 7]. The electrochromic device changes its optical properties by insertion or extraction of metal ions, such as Li^+ ions, within the electrolyte from the surface of WO_3 thin film, according to the redox reaction as follows [8];

$$WO_3 + x(Li^+ + e^-) \leftrightarrow Li_xWO_3 \tag{1}$$

where x is the mole fraction of metal ions. Previously, electrochromic windows have been fabricated by chemical vapor deposition (CVD), radio frequency (RF) sputtering, cathodic electrodeposition, the sol–gel process, spray pyrolysis, DC magnetron sputtering and nanoparticle deposition systems (NPDS) [9–14].

The fabrication process, however, still requires high temperature or pressure, so is not easily accessible to researchers and industry [15]. Accordingly, the durability of the ECDs fabricated by various manufacturing methods has not been sufficiently verified. In this paper, we discuss a relatively low-cost fabrication method and the resulting durability. The performance of the electrochromic device (ECD) can be indicated by the transmittance differences between bleach and coloring states. The coloration efficiency (η) of ECD was formulated by equation below [16];

$$\eta = \frac{\Delta A}{Q} = \frac{\log\left(\frac{T_b}{T_c}\right)}{Q} \tag{2}$$

If ions were trapped on the electrochromic layer, the performance of electrochromic device can be decreased. Therefore, charge balance could be an important indicator for controlling the durability of ECD.

NPDS, a type of a kinetic spray, was first developed in 2008 and several metal oxide materials have been deposited onto different types of a substrate [17–20]. As applications of this process, dye-sensitized solar cells (DSSCs) and WO_3-based electrochromic windows have been fabricated [14, 21, 22]. NPDS is capable of fabricating thin film under low-temperature, low-vacuum conditions without the need for post-processing. This kinetic spray is a facile method to deposit ceramic, as well as metallic powders on a substrate by accelerating particles to subsonic speed. NPDS is composed of a stage, controller for substrate, a slit nozzle, and the air compressor with an aerosol generator which blows the air to float target particles. The floated particles are accelerated to subsonic speed through the nozzle in the low vacuum chamber. Finally, the particles are deposited on the substrate with maximum velocity at a distance which is defined as stand-off distance (SoD) between the nozzle and the substrate. Solid WO_3 powder is mixed with 0.3 MPa air and passes through the subsonic nozzle; the particles are highly shattered and amorphously deposited onto the substrate. Therefore, it has higher WO_3 deposition efficacy compared to other conventional processes.

The active voltage, speed of coloration, optical transmittance, optical reflectance, solar heat efficiency, active temperature and lifetime have been suggested as performance factors for ECDs [4]. The lifetime of ECDs should be approximately 25 years, with a total of 100,000 coloration and bleaching states [23]. The National Renewable Energy Laboratory (NREL) performed cyclic environmental tests on EC and published ASTM standards for accelerated durability tests in EC, ASTM E2355, E2241, and E2141. Testing is required over 50,000 cycles and 5000 h, including metrology tests pertaining to irradiance, temperature, and humidity.

Four major types of defects can arise due to the amorphous states of films, and these can affect the optical transmittance performance of WO_3 ECDs: stoichiometric tungsten oxides, oxygen vacancies, oxygen interstitials, and negative effects of Li insertion [24]. The negative effects of the insertion of Li can be combatted by an ion de-trapping method, which recovers the original performance of the device [25]. A WO_3 film fabricated by NPDS at different temperatures was recently characterized, and we achieved a transmittance change greater than 40% at 150 °C [26]. However, there have been no long-term durability tests on WO_3 film fabricated by NPDS. Therefore, durability tests on ECWs are necessary. In this research, dual-phase WO_3 particles which are composed of crystalline core and amorphous shell structure, were deposited by NPDS to fabricate ECD. According to the previous studies done by Kim et al., amorphous phase in the amorphous/crystalline heterostructure of the film is more loosely packed with a randomly ordered atomic structure so that diffusion of Li ions in the heterostructure was found to be more favorable and faster than that in the crystalline structure only [19]. The electrochromic device using this WO_3 film showed its transmittance change of more than 45% in this study. Furthermore, tests were conducted to verify their optical performance as well as durability. Therefore, a novel life-conserving algorithm was designed, and an accelerated lifetime test over one million cycles was carried out to verify the cyclic durability of ECWs fabricated by NPDS.

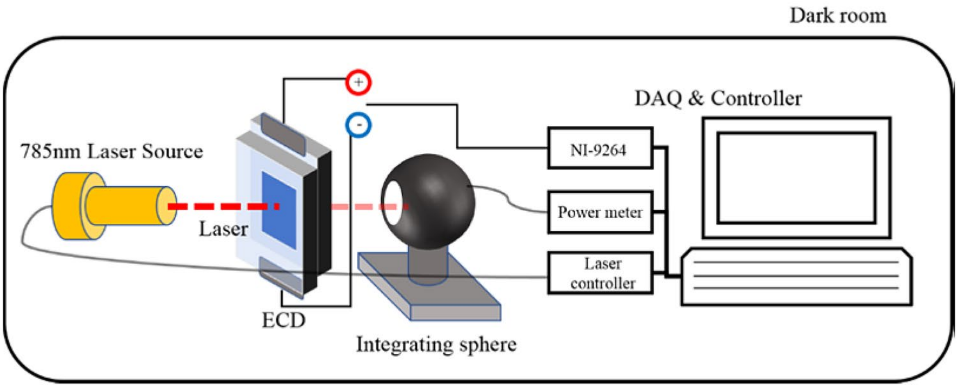


Fig. 1 Schematic of the cyclic test system for assessing the durability of electrochromic windows (ECWs)

2 Experimental

2.1 Material Preparation and Fabrication of ECDs

An electrochromic layer was formed by depositing nano-size WO_3 (100 nm; HKKSOLUTION Co., Ltd.) powder on fluorine-doped SnO_2 (FTO) glass (Pilkington) by NPDS. NPDS was also used to form an ion storage layer to deposit ATO powder (30 nm; JL Chem) onto indium-doped SnO_2 (ITO) glass. The deposition process was carried out at room temperature and the injection pressure was 0.3 MPa. A new gel-type electrolyte with superior durability was also developed. Then, 2 g of poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) pellets (Mn ~ 130,000; Sigma Aldrich) were dissolved in 20 ml of acetone at 60 °C under stirring. An ultraviolet (UV) agent was used to blend poly(ethylene glycol) methyl ether dimethacrylate (PEGDMA, Mn 550) as diacrylate monomer and poly(ethyleneglycol) methyl ether methacrylate (PEGMA, Mn 950) as monoacrylate monomer. This UV agent was blended with a mixing ratio of diacrylate monomer to monoacrylate monomer of 3:7 wt%, and we then fabricated the UV agent by adding a photoinitiator (Irgacure 184; BASF) of 2 wt% to the blended solution. A 1 M $LiClO_4$ + PC:EC (1:1; propylene carbonate:ethylene carbonate; Sigma Aldrich) liquid electrolyte was mixed in a 1:1 ratio, while the blended solution contained 70 wt% dissolved PVDF-HFP solution and 30 wt% UV agent. Finally, we attempted to fabricate a free-standing membrane from the gel-type electrolyte by mixing a solution of 6 ml, which was applied to a petri-dish and cured by UV treatment (Raynics Inc.) for 12 min. After UV treatment, a free-standing electrolyte was fabricated by cooling at room temperature and its thickness was measured to be 100 μm.

2.2 Measurement and Control System

For the cyclic test, a programmable voltage was generated by NI-9264 and an 80 mW 785 nm laser (785-80; STRADUS) was used as the light source. An optical power meter (2936-R; Newport) and integrating sphere detector (918D-SL-2CAL2; Newport) were used to measure the transmitted optical power (Fig. 1). The cyclic test device was installed onto an anti-vibration optical table and covered by a black acrylic box, to avoid disturbance due to external light exposure. Data acquisition and control software was developed using LabVIEW. The optical transmittance and current were measured every 50 ms and the applied voltage could be controlled spontaneously.

3 Results and Discussion

3.1 Optical and Electronic Properties of ECDs

The optical and electrochemical properties of the ECDs were characterized by analyzing the optical transmittance and current. Under widely used electric potential (2, 2.5, and 3 V) conditions with fixed coloring and bleaching times (40 and 100 s, respectively), the transmittance at 785 nm and current were measured every 50 ms for 24 h. Figure 2a shows the variation in transmittance under these conditions. Within each cycle, the transmittance decreases during coloring and increases during bleaching. This indicates an overall increasing tendency and decrease in the width of the transmittance. The difference in the transmittance is the contrast in the transmittance of each cycle (Fig. 2b). A steep drop in the transmittance difference was observed in the early stages, followed by a gradual decrease until the end of the test. Transmittance difference ratio is related to the applied voltage. As shown in Fig. 2b, the first cycle of the test has reached to maximum transmittance difference ratio. However, high voltage like 3.0 V has decomposed electrolyte so that its transmittance difference ratio has rapidly decreased. As a result, we have confirmed that the high voltage of 3.0 V is not suitable for the electrochromic device since the applied high potential induced the degradation of the device. These results indicate that, even though the ratio between the coloring time

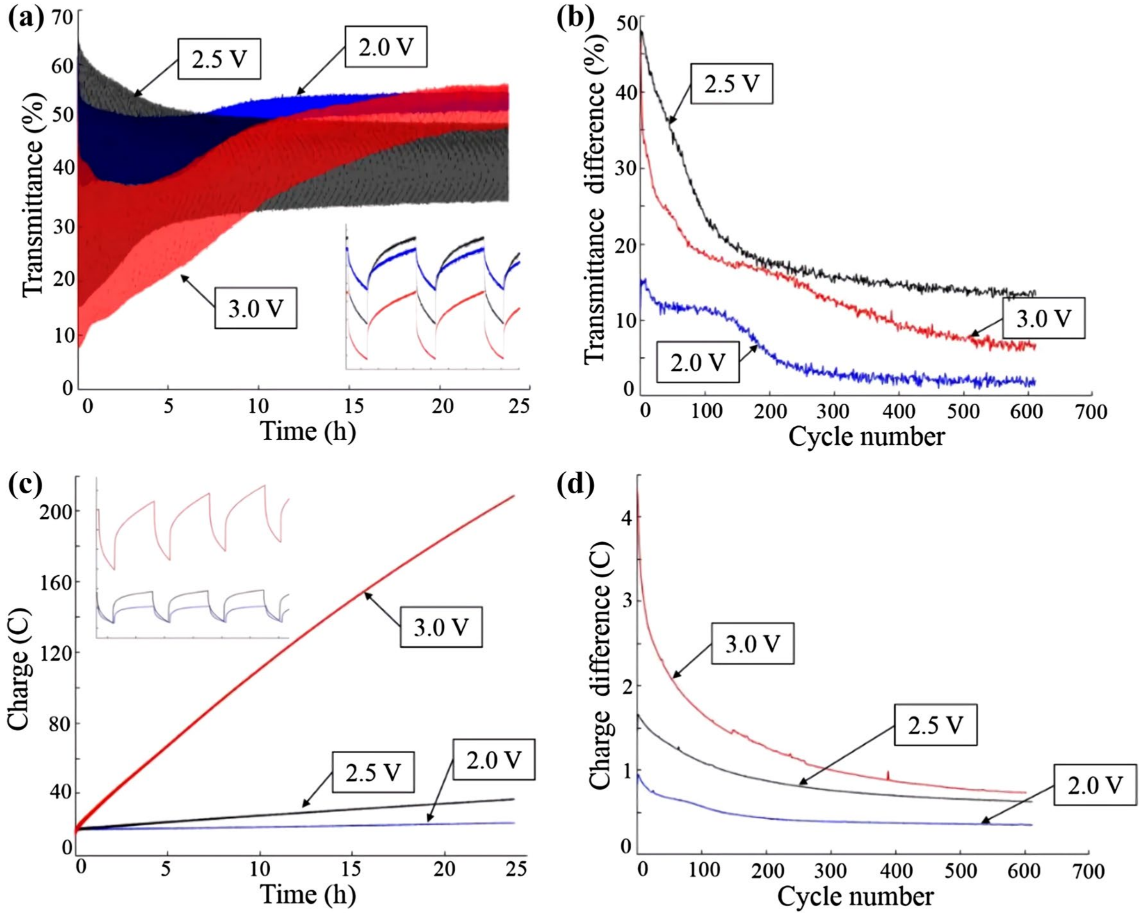


Fig. 2 Measured transmittance and charge data of electrochromic devices (ECDs) under 2.0, 2.5, 3.0 V fixed time conditions: **a** variation in transmittance, **b** transmittance difference, **c** variation in charge, **d** charge difference

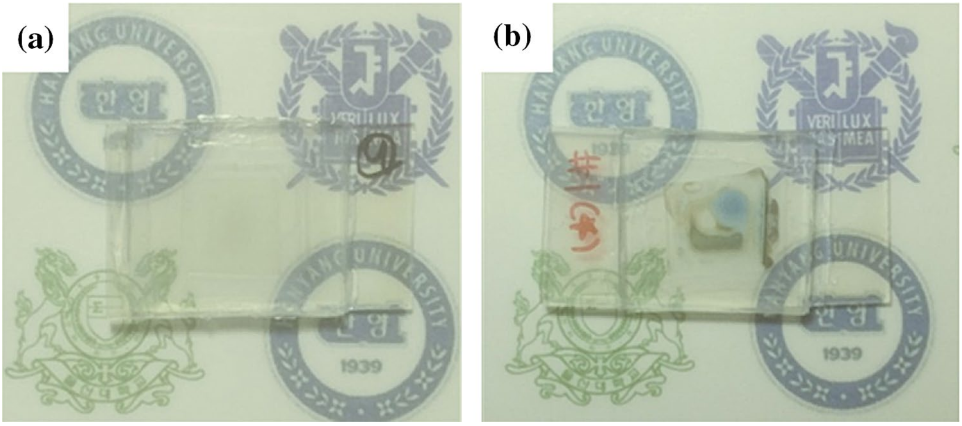


Fig. 3 The ECDs after the durability test in a bleaching state **a** 2.0 V, **b** 3.0 V

and bleaching time is the same, a transmittance bias can arise, depending on the electric potential. Figure 2c shows the variation in charge accumulation with respect to the current applied to the ECDs. This also showed a tendency to increase during the bleaching state, even though the ratio of coloring time to bleaching time is the same under all conditions. Similar to the difference in transmittance, the charge difference is calculated by subtracting the coloring charge from the bleaching charge of each cycle (Fig. 2d). Likewise, a sharp decrease in the charge difference was observed under all conditions, indicating a bias in the charge value that caused a critical loss of charge difference.

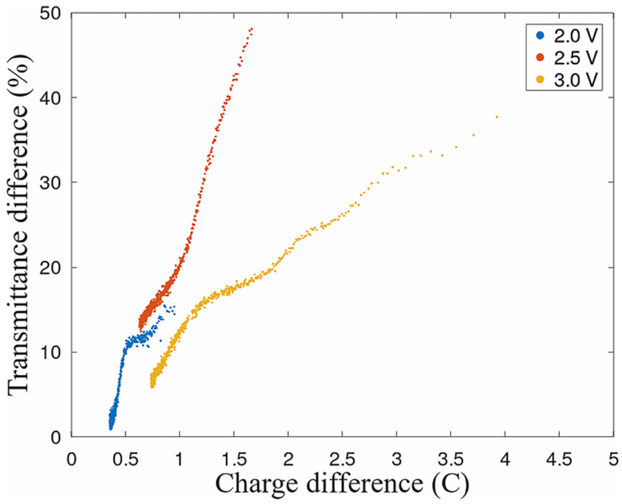


Fig. 4 Relationship between the charge difference and transmittance difference

Figure 3 shows a picture of ECDs after tests conducted at 2.0 V (Fig. 3a) and 3.0 V (Fig. 3b) in the bleaching state. Compared to the other samples, permanent damage to the sample can clearly be seen with the naked eye with the 3 V test.

Figure 4 shows the relationship between the charge difference and transmittance difference. There is an almost linear relationship between the parameters. Under the same conditions, a large difference in transmittance can be obtained by inducing a high charge difference. The gradient of the graph is the transmittance difference induced per unit charge difference. Despite the results obtained at 3 V, i.e., the highest energy case, we cannot infer that there is a high rate of exchange between charge difference and transmittance difference. In other words, electric potential conditions do not ensure good durability or transmittance performance. Maintaining the transmittance difference to obtain sufficient durability requires induction of a high charge difference. Under the above conditions, the charge bias had an adverse effect with respect to both inducing chemical reactions and obtaining a valid charge difference. To overcome this problem and extend the life of ECDs, it is necessary to design a dynamic algorithm to evaluate the charge balance in real time.

3.2 Charge Balance Algorithm

To optimize the conditions for extending the lifetimes of ECDs, it is necessary to consider the charge balance in real time. The total charge induced on the ECDs per cycle can be analyzed based on three terms: charge reduction by coloring, charge increase by bleaching, and error charge, which depend on the state of the ECD:

$$Q_{total} = Q_{coloring} + Q_{bleacing} + Q_{error} \tag{2}$$

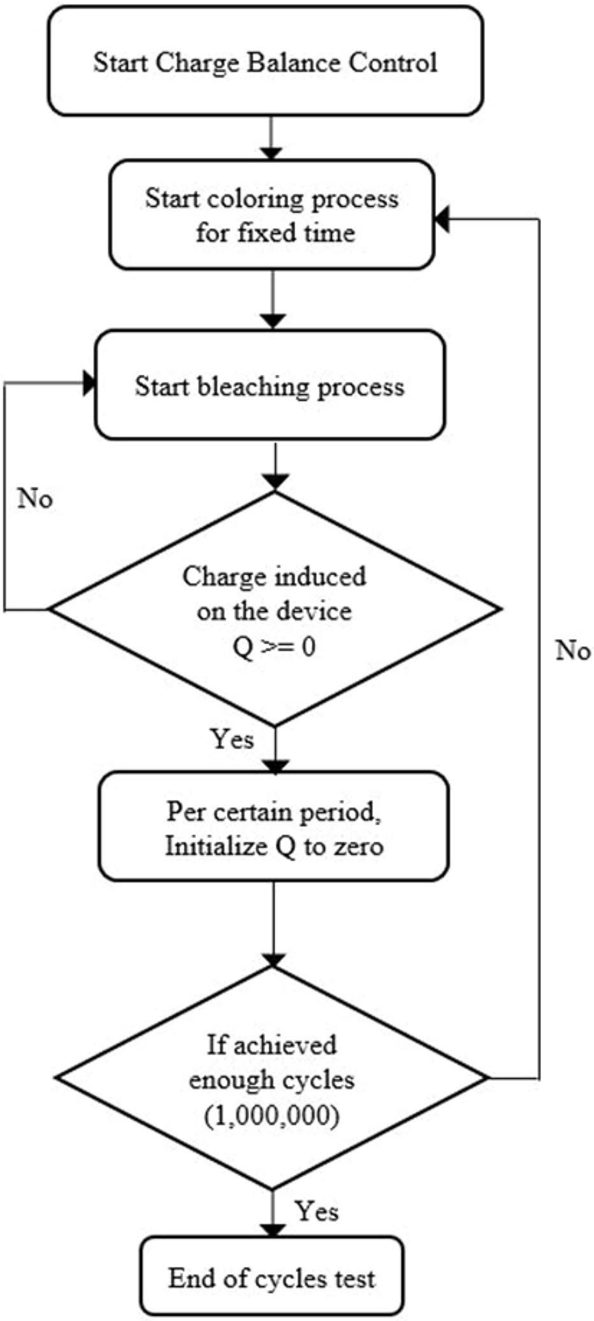


Fig. 5 Charge balance algorithm for accelerated life test of ECDs

The bleaching and coloring charges can be expressed in the time domain, including a term for the current induced on ECDs. Chemical reactions and measurement error cause Q_{error}:

$$Q_{total} = \int_0^{t_{coloring}} i_{coloring} dt + \int_{t_{coloring}}^{t_{bleaching}} i_{bleaching} dt + Q_{error} \tag{3}$$

A new approach to extending the lifetime of the device is to automatically select the bleaching time ($t_{bleaching}$) until

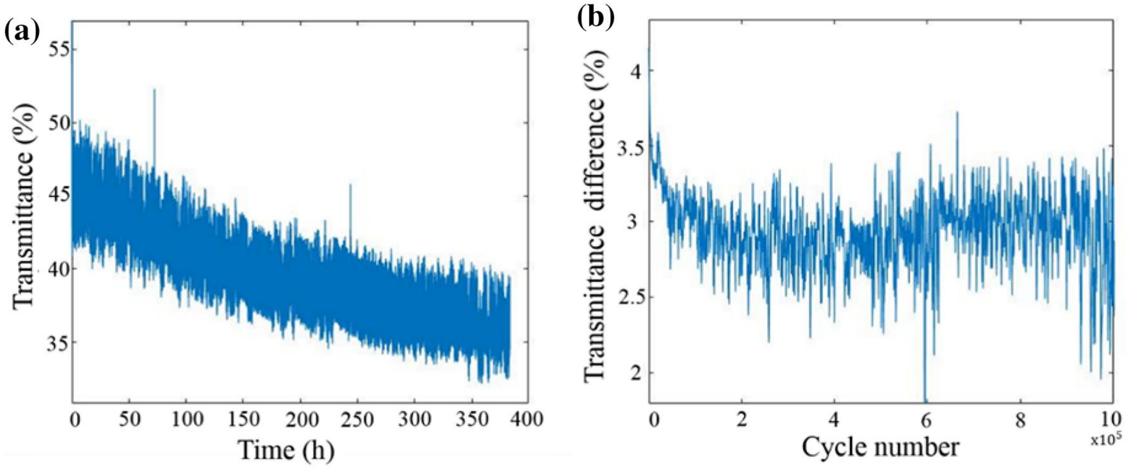


Fig. 6 Measured transmittance of ECDs adopting charge balance control during the one million cycle test

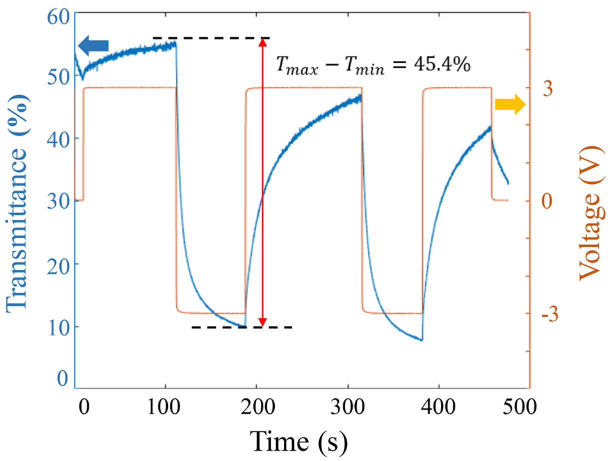


Fig. 7 Measured transmittance of ECDs after the accelerated life test

the coloring charge ($Q_{coloing}$) reaches the bleaching charge ($Q_{bleacing}$). By adopting this new approach, we minimized the charge bias due to imbalances between coloring and bleaching, therefore preserving the charge difference:

$$\int_{0}^{t_{coloring}} \left|i_{colroing}\right| dt = \int_{t_{coloring}}^{t_{bleaching}} \left|i_{bleaching}\right| dt \tag{4}$$

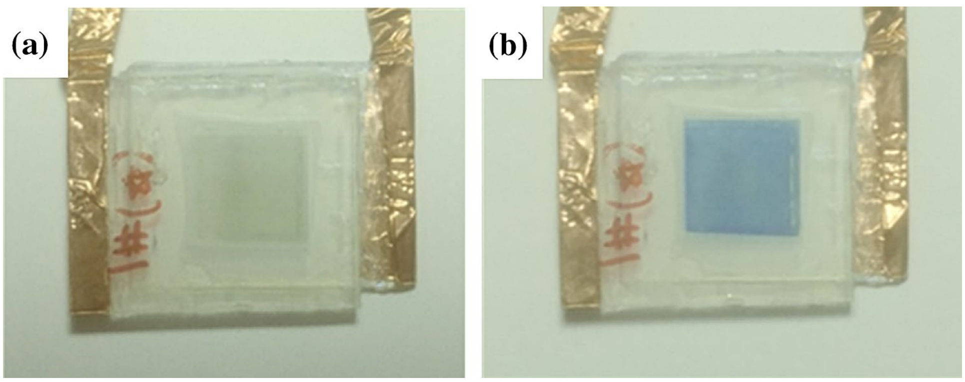
However, in practice, we cannot separate the current for coloring (including bleaching) from the actually measured current, because errors due to chemical reactions and the sampling rate occur simultaneously. Therefore, the total charge includes only the error in the charge under charge-balanced conditions, so it is necessary to consider the error when designing an algorithm for accelerated life tests:

$$Q_{total} = \int_{0}^{t_{bleaching}} i_{measure} dt = \int_{0}^{t_{bleaching}} i_{error} dt = Q_{error} \tag{5}$$

3.3 One Million Cycle Test of ECDs Under Charge Balance Control

A simple way to cancel out the charge error (Q_{error}) is to periodically reset the charge to zero. The charge quantity at the moment of initialization can be regarded as the permanent error. Figure 5 shows the final algorithm for accelerated life

Fig. 8 Picture of ECDs after the accelerated life test: **a** bleaching state, **b** coloring state



tests of ECDs. First, 2.0 V coloring occurs at 1 s. After that, 2.5 V oxidation continues until the charge reaches zero, so the bleaching end time will be decided automatically. During the cycle test, the charge will be reset to zero after every 1000 s, and this is repeated over one million cycles.

We turn the ECDs on and off one million cycles. We applied the charge balance control method to ECDs and measured the optical transmittance over one million cycles. The ECDs was feedback controlled, as the accumulated charge after every cycle, almost neutralized by the charge balance algorithm. Charge error occurred due to chemical reaction, was handled periodically during long-term test. The bleaching period varies fluidly depending on the charge quantity, as shown in Fig. 6a, We carried out an accelerated life test for 380 h (one million cycles). Figure 6b shows that the difference in transmittance was maintained at approximately 3%, from the initial state to the end of the test. The transmittance bias was diminished compared to the static time condition.

After carrying out the accelerated life test, we reverified the sample to confirm its optical performance. Figure 7 shows the transmittance results of ECDs after the accelerated cycle test. The 3.0 V coloring and 3.0 V bleaching conditions were applied to electrodes on the sample. The difference in transmittance between the maximum and minimum value was 45.4%. Figure 8 shows the sample in the bleaching state (Fig. 8a) and coloring state (Fig. 8b). In conclusion, the optical performance was successfully conserved, even after one million cycles.

4 Conclusion

We constructed ECDs with 10 mm × 10 mm WO_3 film fabricated by NPDS. However, durability issues arose due to the imbalance between ions and electrons caused by the porous surface of the electrodes. We then measured the transmittance at 785 nm, and the current, and confirmed the durability of the ECDs under well-controlled static conditions. The linearity of the transmittance difference with respect to the charge difference was confirmed. Permanent damage occurred under 3 V conditions. A new dynamic algorithm was implemented to improve the reliability of the ECDs, by balancing the coloring and bleaching charges. We carried out a durability test with one million cycles to confirm the operation of the ECDs controlled by the proposed algorithm. After the accelerated life test was completed, we carried out a confirmation test to verify the optical performance under controlled voltage conditions. We measured a 45.4% difference between the maximum and minimum transmittance of the sample after the accelerated life test. The proposed algorithm ensures the reliability of ECDs.

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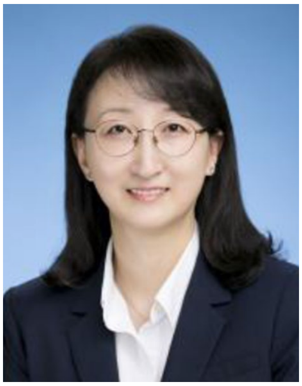

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