공학석사학위논문

전하량 균형을 통한 전기 변색 소자의 가속 수명 시험

Accelerated Life Test of Electrochromic Devices Using Charge Balance Control

2018년 8월

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지도교수 안 성 훈

이 논문을 공학석사 학위논문으로 제출함

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Abstract

Accelerated Life Test of Electrochromic Devices Using Charge Balance Control

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We fabricated 10mm × 10 mm WO_3 electrochromic devices (ECDs) using nanoparticle deposition system (NPDS), a type of coating method using kinetic spray. NPDS has the advantage of mass production due to relatively its low pressure and low temperature, while it forms porous surface having a bad effect on the durability. In order to verify durability of ECDs, we developed an optical transmittance measurement and voltage control system that enables dynamic control. We then characterized WO_3 ECDs focusing on the relationship between transmittance and current under well-known static operating conditions. On the basis of above result, a new algorithm for an accelerated life test was proposed considering charge balance, and then we conducted the accelerated life test during 1,000,000 cycles. The result showed that optical performance maintained until the test ends. After the accelerated life test, the sample persevered its transmittance performance as 45.4% transmittance difference even after the sample underwent long time operation. It was verified that the durability of ECDs could be enhanced by adopting dynamic control, not using an expensive fabricating method.

Keyword： Electrochromic devices, Tungsten oxide Nano particle deposition system, Accelerated life test, durability test

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Chapter 1. Introduction

1.1. Electrochromic devices (ECDs)

As a sort of environmental-friendly nanotechnology and energy conserving method of building construction, electrochromic window is receiving a lot of attention recently [1-2]. Electrochromic window can be used to automatically control temperature inside of building and conserve energy consumption by using sunlight penetrability [3]. Prospective applications of ECDs are glass for automobile, spacecraft, airplanes and optical instrument such as sunglasses, displays [4].

Electrochromism is defined as a phenomenon of optical transmittance change which is induced by an applied voltage. Figure 1.1 shows the principle of ECDs. Electrochromic effect was discovered 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 with electrochromic properties including various color changes [5]. Electrochromism on Tungsten Trioxide, WO_3, was first studied in the 1970s and then optical and electrochemical properties of WO_3 using various fabrication methods have been investigated [6]. Previously, electrochromic window was fabricated by chemical vapor deposition (CVD), radio frequency (RF) sputtering, cathodic electrodeposition, the sol-gel process, spray pyrolysis, DC magnetron sputtering and Nano-particle Deposition System (NPDS) [7-12].

The fabrication process, however, still requiring high temperature or pressure, is not easily accessible by researchers and industries. This also means that the durability of the ECDs fabricated by various manufacturing methods are not sufficiently verified. Herein, relatively low-cost fabrication method and necessity for durability are discussed.

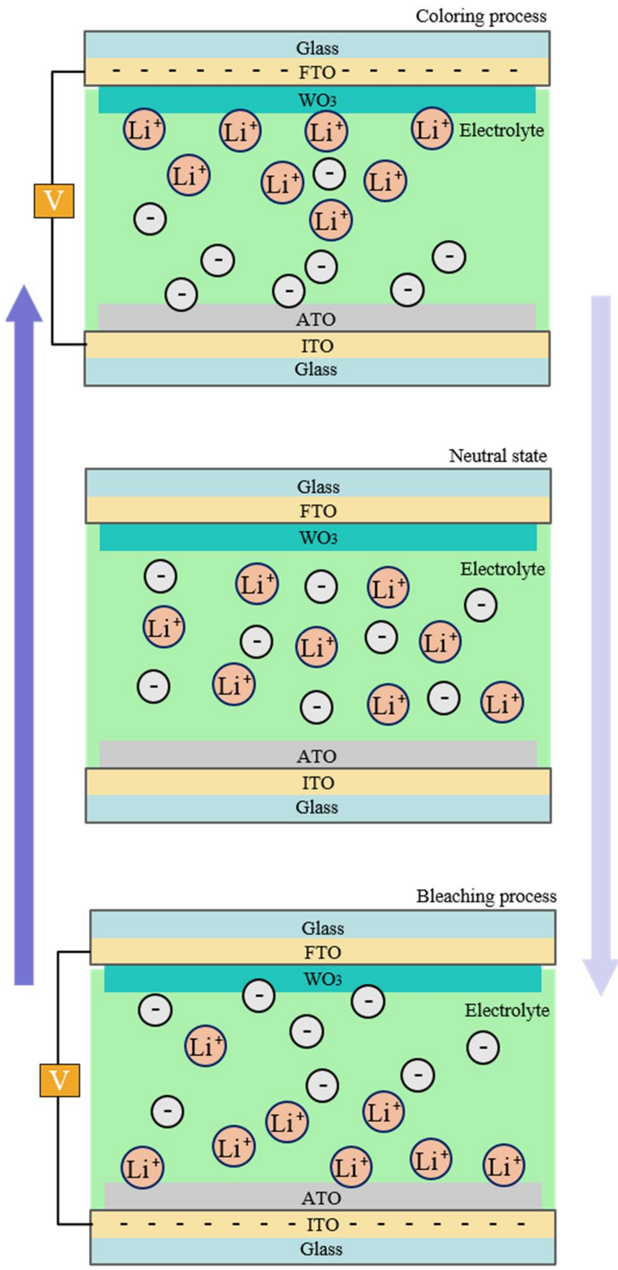

Figure 1.1 Schematic diagram of coloring, bleaching process

1.2. Nanoparticle deposition system (NPDS)

NPDS, a type of a kinetic spray, is firstly developed in 2008 and several metal oxide materials were deposited onto different types of substrates until now [13-16]. As applications of this process, Dye-Sensitized Solar Cell (DSSC) and WO_3 based electrochromic window were fabricated [12,17]. NPDS is capable of fabricating thin film in low-temperature, low-vacuum condition with no post-processes. Therefore, it has high productivity for depositing WO_3 than other processes.

Figure 1.2 shows a diagram of NPDS. It consists of air jet parts, stage and controller for substrates and slit nozzle. As solid WO_3 powder is mixed with 0.3 MPa air, it passes through the subsonic nozzle and then the particle is deposited onto a substrate amorphously. Figure 1.3 shows the picture of the large area nanoparticle deposition system. A 300×300 mm^2 class electrochromic window was fabricated by large area NPDS, shown in figure 1.4.

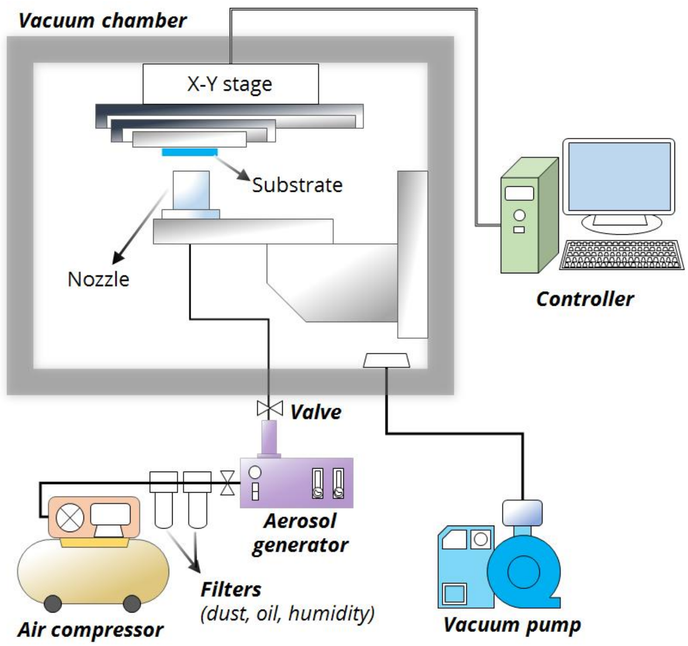

Figure 1.2 Schematic diagram of the nanoparticle deposition system (NPDS) (Park, Sung-Ik, et al. "A review on fabrication processes for electrochromic devices." *International Journal of Precision Engineering and Manufacturing-Green Technology* 3.4 (2016): 397-421.)

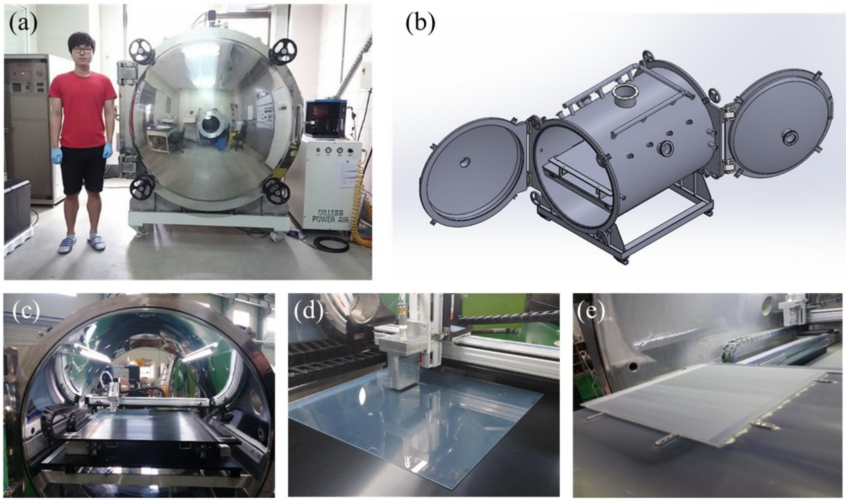

Figure 1.3 Real pictures and the large-area nanoparticle deposition system (NPDS): (a) exterior of equipment, (b) 3D model, (c) interior of equipment, (d) deposition process, (e) deposited substrate (Park, Sung-Ik. "Fabrication of Electrochromic Device Using Nano-particle Deposition System and Its Response Time Modeling for Large-area($1m^2$ Class) Application" Ph. D. thesis, Seoul National University, (2018): 64.)

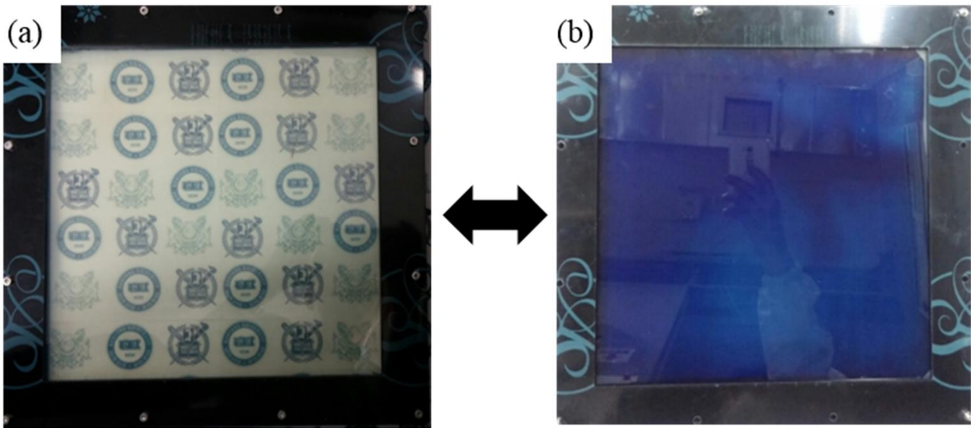

Figure 1.4 A 300 × 300 mm^2 class electrochromic window fabricated using large-area NPDS: (a) bleaching state (b) coloring state

1.3. Durability requirements of ECDs

As a performance factor of ECDs, active voltage, speed of coloration, optical transmittance, optical reflectance, solar heat efficiency, active temperature and lifetime were suggested [4]. Of these, the lifetime of ECDs requires about 25 years with a total 100,000 coloration and bleaching states [18]. National Renewable Energy Laboratory (NREL) studied about cyclic environmental testing on EC and published ASTM standards regarding accelerated durability test on EC, ASTM E2355, E2241, and E2141. Over 50,000 cycles and 5,000 hours includes metrology for irradiance, temperature and humidity.

There are 4 major defects due to an amorphous state of films that affect the optical transmittance performance of WO_3 electrochromic device: Stoichiometric tungsten oxides, oxygen vacancies, oxygen interstitials, and effect caused by the insertion of Li [19]. The effect caused by the insertion of Li was treated by the ion de-trapping method and conducted to the electrochromic window and recovered its original performance [20].

WO_3 film fabricated by NPDS is amorphous due to characteristics of NPDS. However, lifetime test on WO_3 film fabricated by NPDS has not been conducted sufficiently, durability test on the ECW is necessary. Figure 1.5 shows schematic model of accelerated lifetime test. An accelerated lifetime test defined as a test conducted under severe stress in order to shorten test time and clarify an aging failure mechanism. In this research, WO_3 based ECW was fabricated by NPDS and tested several samples to verify its optical performance. The durability test was conducted until the device had severe defects. Furthermore, novel life-conserving algorithm was designed and accelerated lifetime test during one million cycles was conducted in order to verify cyclic durability of ECW fabricated by NPDS.

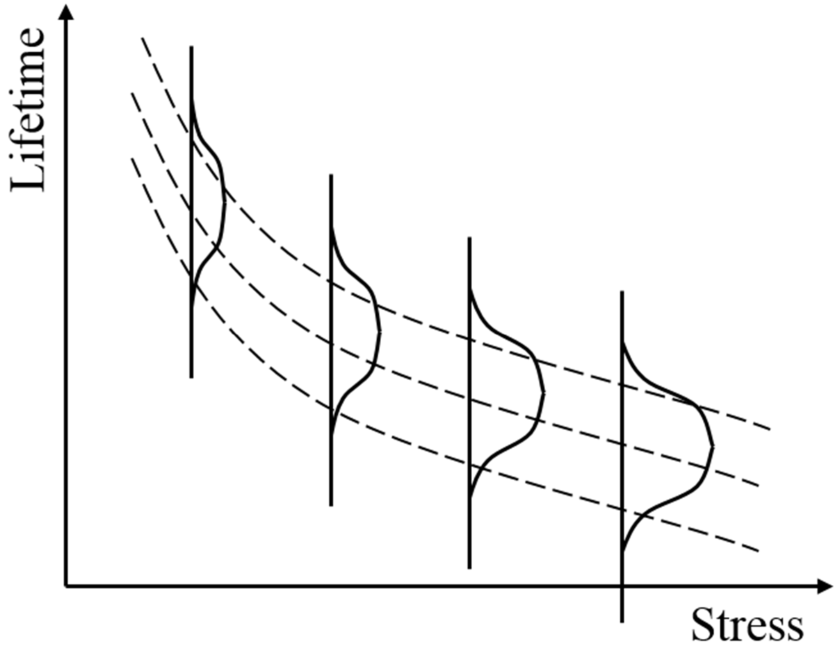

Figure 1.5 Schematic model of accelerated lifetime test.

Chapter 2. Materials and Experiments

2.1. Materials

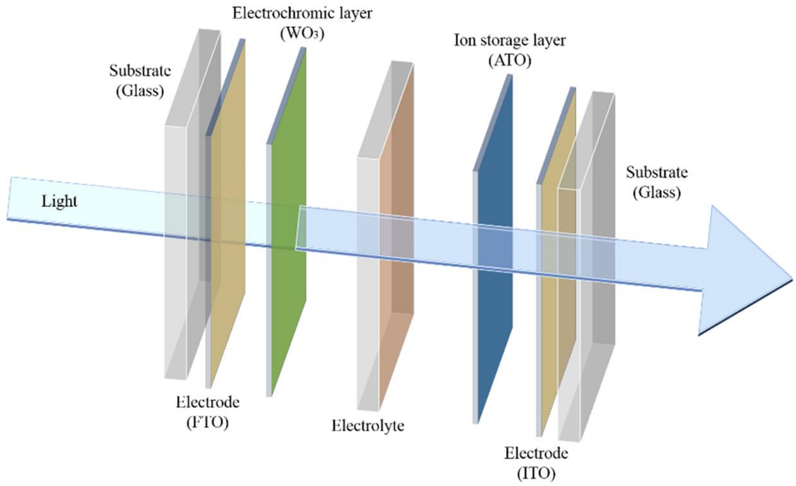

Figure 2.1 Schematic diagram of an ECD

Figure 2.1 shows the structure of ECDs. Electrochromic layer was formed by depositing nano-size WO_3 (100 nm, HKKSOLUTION CO. , LTD.) powder on fluorine-doped SnO2 (FTO) glass (Pilkington) by the NPDS. The NPDS was also used to form ion storage layer in order to deposit ATO (30 nm, JL Chem.) powder onto Indium-doped SnO2 (ITO) glass. The depositing process was carried out at room temperature and injection pressure was 0.3Mpa. A new gel-type electrolyte superior in durability was also developed. Poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP, Sigma Aldrich, Mn ~130,000) pellet of 2 g was dissolved in Acetone of 20 ml at 60^{o}C with stirring condition. 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 : monoacrylate monomer = 3 : 7 wt.%, and then it was fabricated the UV agent by adding photoinitiator (Irgacure 184, BASF) of 2 wt.% into the blended solution. A 1 M $LiClO_4$ + PC:EC (1:1) (Sigma Aldrich, propylene carbonate (PC), ethylene carbonate (EC)) as liquid electrolyte was mixed in 1:1 with blending solution between dissolved PVDF-HFP solution of 70 wt.% and UV agent of 30 wt.%. Finally, we tried to make the gel-type electrolyte as a free standing membrane through the mixed solution of 6 ml, which was applied onto a petri-dish and cured by UV treatment (Raynics Inc., Korea) for 12 minutes. After UV treatment, a free standing electrolyte was fabricated by cooling at room temperature and its thickness was measured to be 100um. Figure 2.2 shows transmittance spectra and Figure 2.3 shows Cyclic Voltammetry (CV) curve of ECD fabricated by NPDS

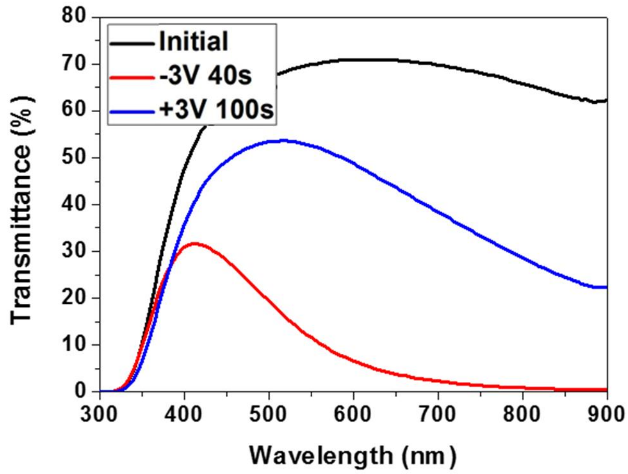

Figure 2.2 Transmittance spectra of ECD fabricated by NPDS

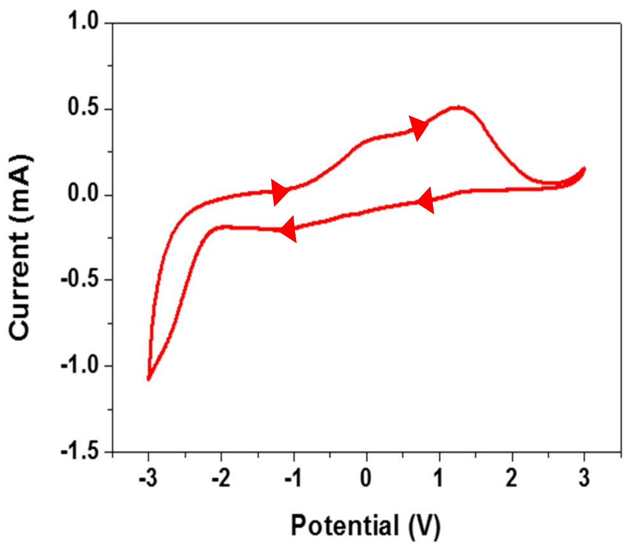

Figure 2.3 Cyclic Voltammetry (CV) curve of ECD fabricated by NPDS

2.2. Experiment system configurations

For cyclic test device, as shown in Figure 2.5, programmable voltage was generated by NI-9264, STRADUS 785-80 80mW 785nm laser was used as light source and Newport optical power meter and Integrating sphere detector (918D-SL-2CAL2) measured transmitted optical power. Figure 2.6 shows schematic diagram of integrating sphere. Transmittance is defined as a ratio of incident light to transmitted light, and integrating sphere collects scattered light from ECDs and helps to get precise value of transmittance. The 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 with LabVIEW. Data acquisition interval was 50ms and applied voltage was controlled spontaneously.

Electrochromic cycle test environment, which includes not only hardware system however also software system, were well developed. Given specimen was tested over 1,000,000 cycles and it spent about a month.

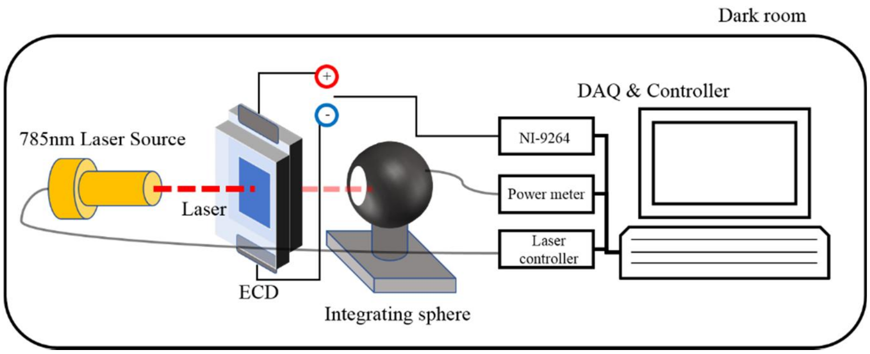

Figure 2.4 Schematic diagram of performance measurement and control system.

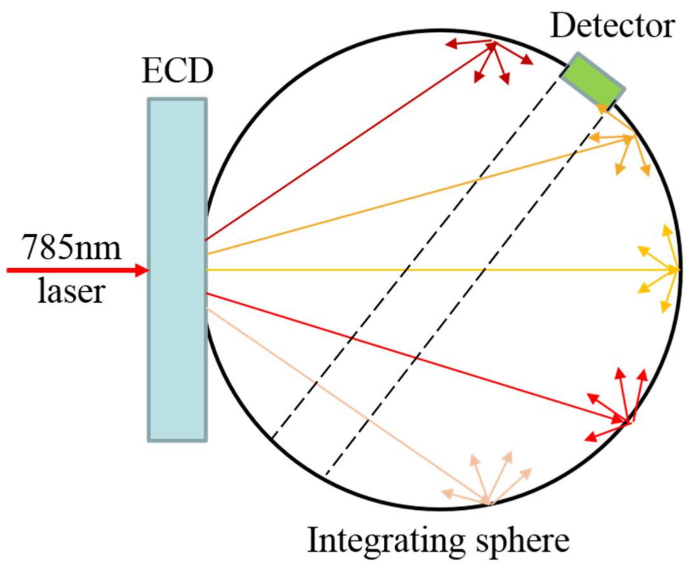

Figure 2.5 Schematic diagram of integrating sphere

Chapter 3. Characteristics test of ECDs

3.1. Experimental conditions

In order to design proper algorithm for 1,000,000 cycles test, preceding experiments were conducted under well-known conditions to know optical and electrochemical characteristics of the ECW fabricated by NPDS. Table 3.1 shows the voltage and time conditions of the specimen.

Table 3.1 Experimental conditions for characteristics test for ECDs.

Parameter	Voltage		Time	
	Reduction (Coloring)	Oxidation (Bleaching)	Reduction (Coloring)	Oxidation (Bleaching)
Condition 1	2.0 V	2.0 V	40 s	100 s
Condition 2	2.5 V	2.5 V		
Condition 3	3.0 V	3.0 V		

2.0 V, 2.5 V and 3.0 V were selected for coloration voltage because these are well-known conditions for operating WO_3 based film ECD. Time conditions were selected experimentally when the specimen shows high optical transmittance performance at the initial state. These conditions were not optimized for long time durability test.

The tests under the above conditions were conducted during 24 hours, about 600 cycles. In the meantime, optical transmittance and current induced on the specimen were measured in order to discover optical, electronical, chemical characteristics of the ECD fabricated by NPDS.

3.2. Optical and electrochemical properties

Figure 3.1 and 3.2 show the optical transmittance and transmittance difference of the ECDs under proposed 3 different voltage conditions. Optical transmittance of the samples was measured every 50 ms during 24 hours. Transmittance difference is the contrast in transmittance of each cycle. Biasing of transmittance and progressive diminution of transmittance difference were observed at all conditions. A steep drop of transmittance difference was found in its early stages until stabilized, and still gradual decrease was found until the test ends. These results indicate that coloration (or discoloration) time is dominant factor to long time test and the fixed time condition is not proper to verify the cyclic durability of ECDs.

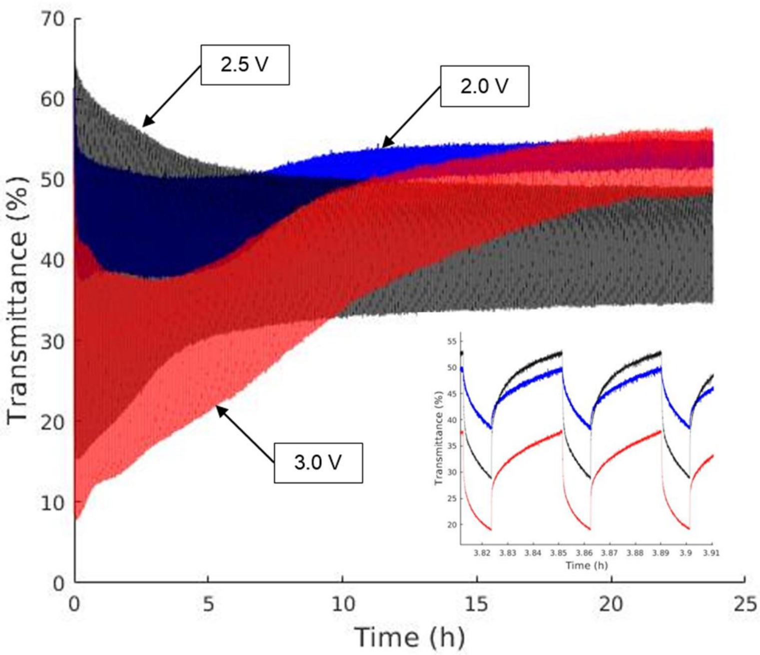

Figure 3.1 Measured optical transmittance data of ECDs during characteristics test

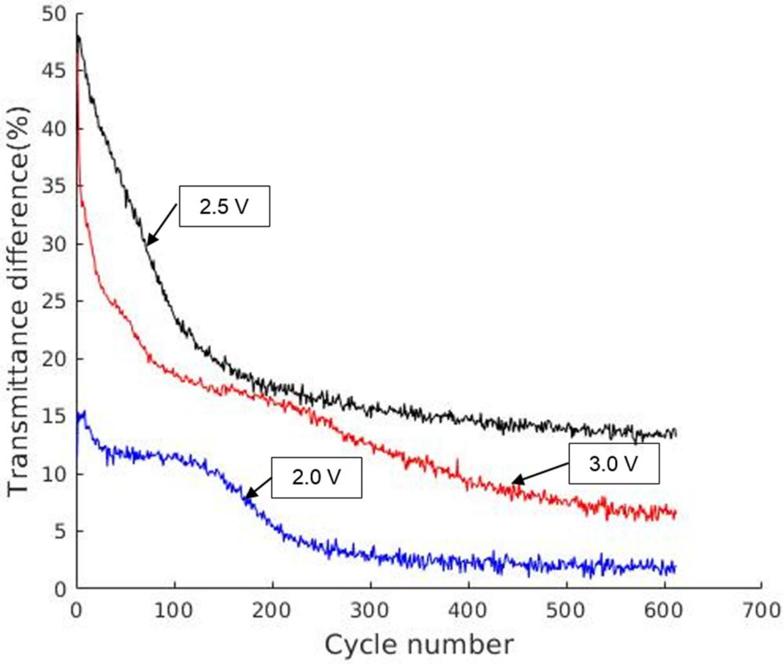

Figure 3.2 Transmittance difference data of ECDs during characteristics test

Figure 3.3 and 3.4 show charge amount and charge difference of the samples of 3 different conditions. The current induced on the samples was measured every 5 ms during the entire test time. Charge value was calculated by summation of current, and charge difference also drien by subtract charge value of coloration from charge value of discoloration. On the suggested fixed time condition, charge has steady bias from the beginning to end of the test. Charge difference decreased gradually similar to behavior of transmittance difference. The suggested condition provoked bias of charge value resulting in critical loss of charge difference.

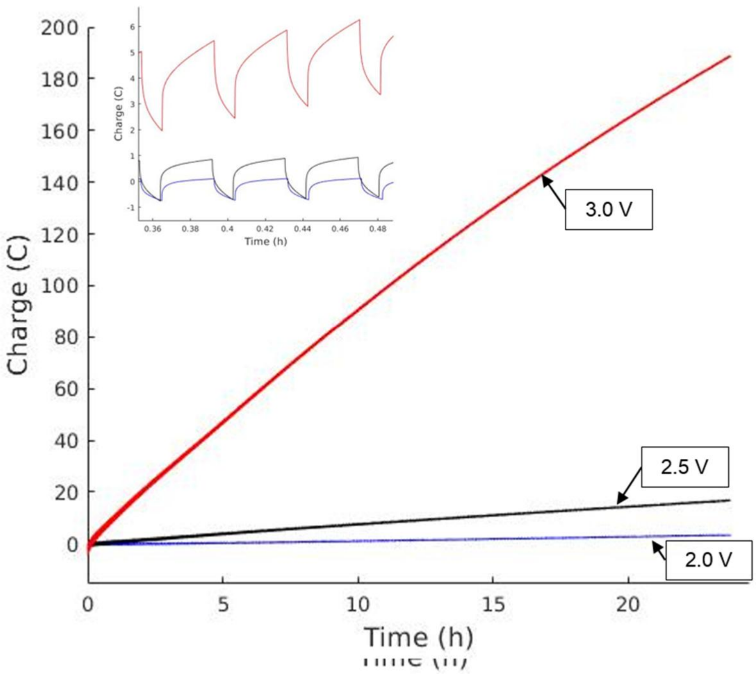

Figure 3.3 Measured charge quantity of ECDs during characteristics test

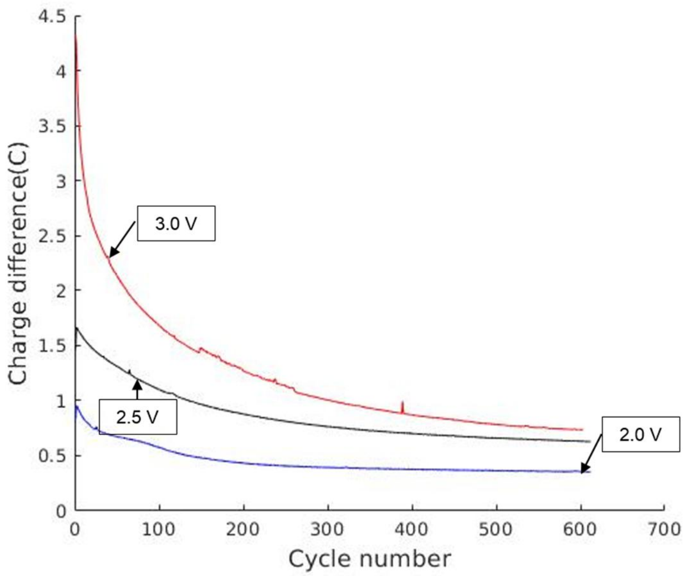

Figure 3.4 Charge difference of ECDs during characteristics test

Figure 3.5 shows that the picture of sample under 3 V condition before and after the test. Compared to other other samples, permanent damage clearly would be seen with the unaided eye from 3 V sample. The irreversible damage resulted from chemical reaction from the repeated operation at the high voltage.

(a)

(b)

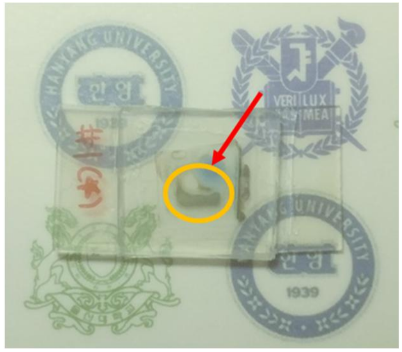

Figure 3.5 Image of ECDs under 3 V condition (a) before characteristics test (b) after characteristics test

3.3. Linearity between optical and electronical properties

Figure 3.6 shows the linear relationship between the charge difference and transmittance difference. If given same condition, high transmittance difference could be derived by inducing high charge difference. The slope of the graph means a transmittance difference induced per unit charge difference. Despite of 3 V, most high energy case, it does not mean high exchanging rate between charge difference and transmittance difference.

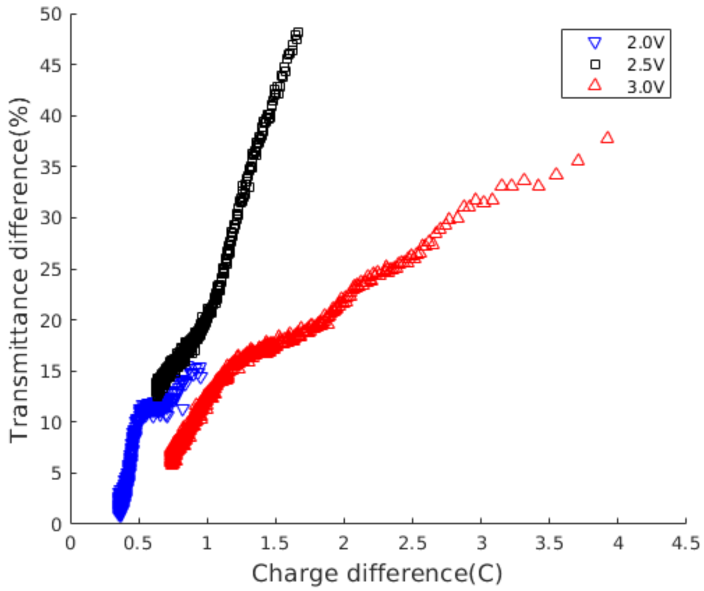

Figure 3.6 Relationship between charge difference and transmittance difference

Under the suggested condition, the optical transmittance decreased seriously, from 48% to 12% (2.5 V case), because charge balance was not considered quantitatively. In order to overcome the problem and extend life of the ECDs, it is necessary to design proper algorithm considering charge balance.

Chapter 4. Accelerated life test of ECDs

4.1. Algorithm design

In order to optimize conditions for extending lifetime of the ECDs, we have to consider charge balance in real time. The total charge amount induced on the ECDs per one cycle could be analyzed as 3 parts: charge reduced by coloring, charge increased by bleaching and charge of error, depending on the state of ECDs.

$$Q_{total} = Q_{coloing} + Q_{bleacing} + Q_{error} \tag{4.1}$$

Charge of bleaching and coloring can be expressed by time domain including a current term induced on ECDs. Chemical reaction and measurement error caused Q_{error}.

$$Q_{total} = \int_0^{t_{coloring}} i_{coloring} dt + \int_{t_{coloring}}^{t_{bleaching}} i_{bleaching} dt + Q_{error} \tag{4.2}$$

A new approach to extend lifetime is to automatically decide bleaching time ($t_{bleaching}$) only until charge amount of coloring ($Q_{coloing}$) reaches charge amount of bleaching ($Q_{bleacing}$). By adopting this new approach, we minimized charge bias due to unbalance between coloring and bleaching and therefore charge difference will be preserved.

$$\int_0^{t_{coloring}} \left| i_{colroing} \right| dt = \int_{t_{coloring}}^{t_{bleaching}} \left| i_{bleaching} \right| dt \tag{4.3}$$

In practice however, we cannot separate only current for coloring (including bleaching) from actually measured current, because error current due to chemical reaction and sampling occurs simultaneously. Therefore, total charge quantity includes only error charge under charge balance condition, we should consider the error when designing the algorithm for accelerated life test.

$$Q_{total} = \int_0^{t_{bleaching}} i_{measure} dt = \int_0^{t_{bleaching}} i_{error} dt = Q_{error} \tag{4.4}$$

A simple way to cancel out the charge error (Q_{error}) is periodically initializing charge quantity to zero after the lapse of certain time. The charge quantity at the moment (when initialized) will be regarded as permanent error. Figure 4.1 shows the final design of the algorithm for accelerated life test of ECDs. 2.0 V coloring happens during 1s. After that, 2.5 V oxidation starts until charge quantity reaches zero thereby bleaching end time will be decided automatically. During the cycle test, charge quantity will be initialized to zero at every 1000s interval and this cycle is repeated until cycle number reaches one million.

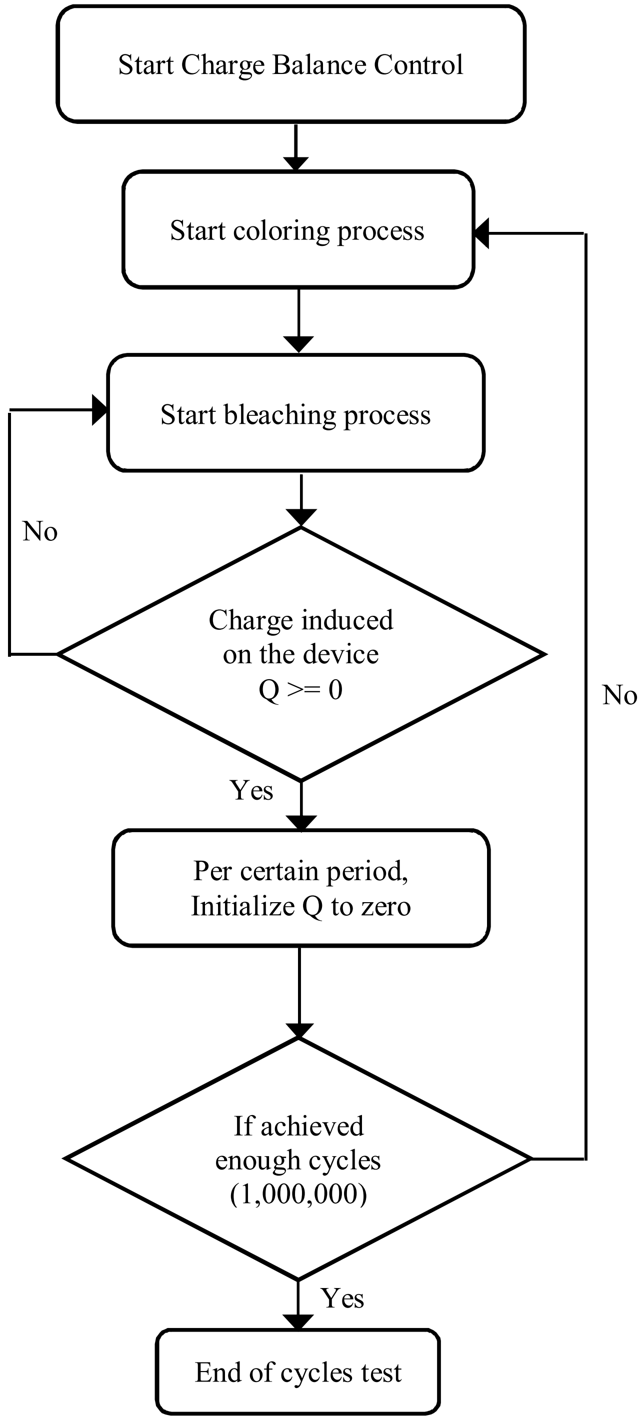

Figure 4.1 Charge balance algorithm for accelerated life test

4.2. One million cycles test of ECDs

Optical transmittance was measured during one million cycles under suggested condition considering charge balance. Bleaching period changes fluidly depending on the charge quantity. As shown in Figure 4.2, the accelerated life test was conducted during 380 hour, one million cycles. Transmittance difference maintained from initial state to the end of test. Transmittance bias was diminished compare to fixed time control.

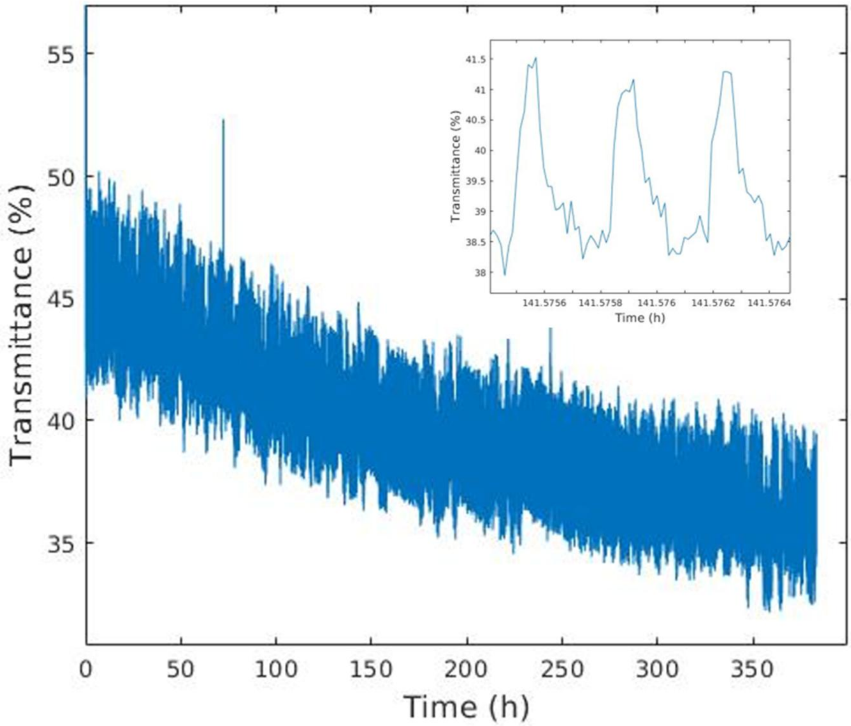

Figure 4.2 Measured transmittance of accelerated life test

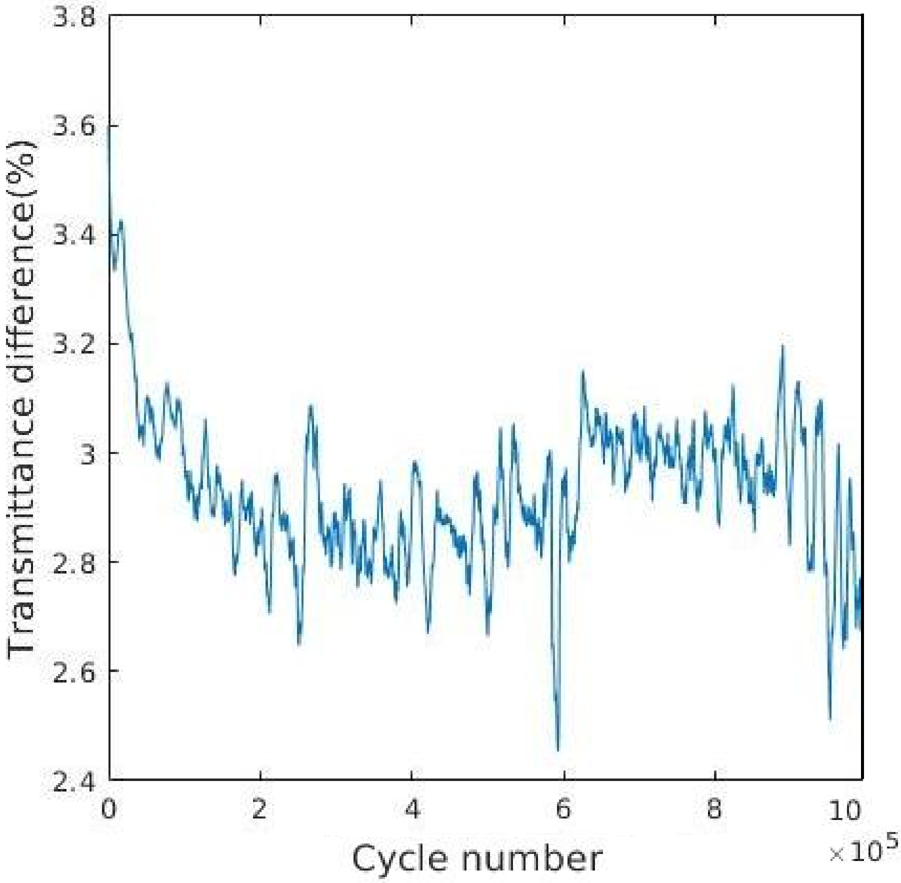

Figure 4.3 Transmittance difference of accelerated life test

4.3. Performance re-verification after one million cycles test

After the accelerated test was conducted, the sample underwent re-verifying process to confirm the optical performance had successfully conserved after the cycle test. Figure 4.4 shows optical transmittance results and figure 4.5 shows the real picture of the sample after the accelerated cycle test. 2.5 V coloring and 2.5 V bleaching were applied to the sample. The transmittance difference between maximum value and minimum value was 45.4%. The picture of sample indicates optical performance successfully conserved even after one million cycles.

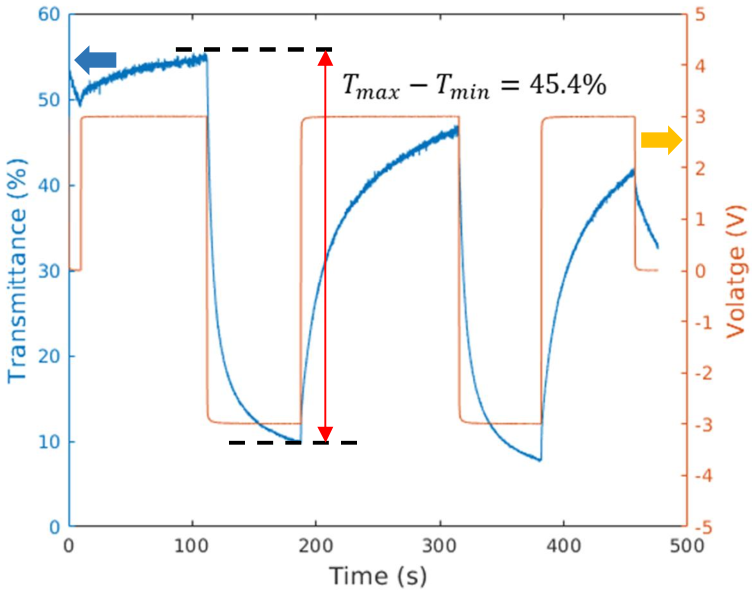

Figure 4.4 Transmittance performance of ECDs after accelerated test

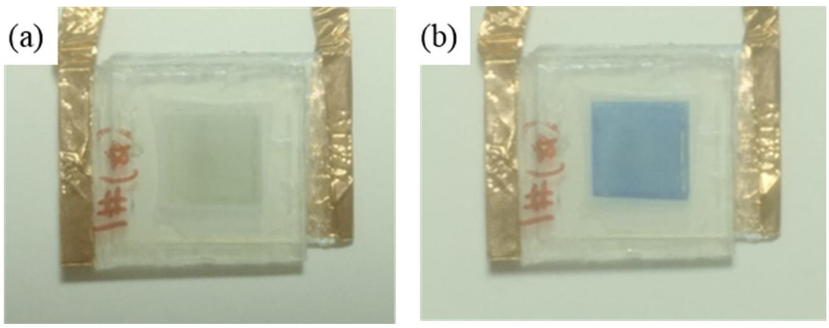

Figure 4.5 Image of ECDs after accelerated test (a) before coloration (b) after coloration

Chapter 5. Conclusion

Previous researches on ECDs were mainly focused on fabricating methods and verifying optical performance in order to show feasibility. In this study, ECDs with 10mm x 10mm WO_3 film dimension were fabricated by NPDS. NPDS has an advantage of efficiency in mass production, however has demerits due to it forms porous surface. Durability performance of amorphous WO_3 film based ECDs have not been verified sufficiently.

In order to optimize proper condition and design algorithm for durability, we measured optical transmittance and electronical characteristics by conducting cyclic tests under well-known conditions. It was confirmed that 3 V condition causes permanent damages on the sample and linearity between charge difference and transmittance.

We then proposed a new dynamic algorithm considering charge bias and conducted accelerated life test with 1,000,000 cycles. During the accelerated life test, we confirmed that transmittance difference maintained from start to end of the test. After the accelerated life test, an additional test was conducted in order to verify optical performance under well-known condition. As a result, 45.4% transmittance difference was confirmed without any visual damage after the accelerated life test. We verified the performance of ECDs fabricated by the low-cost coating method, NPDS, and overcame its low-quality surface problems by adopting charge balance control.

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초록

나노입자 적층 시스템 (NPDS)을 이용하여 투명전극 (FTO) 위에 산화텅스텐 (WO_3) 을 10 mm x 10 mm 면적만큼 적층하여 전기 변색 소자를 제작하였다. 나노입자 적층시스템은 입자를 고압에서 진공으로 에어로졸을 통해 분사하여 적층 하는 방식으로, 상대적으로 저 압력과 상온에서 사용 가능한 공정이므로 대량생산에 유리한 장점이 있다. 하지만 나노입자 적층 시스템은 무정형의 표면을 형성하기 때문에 제작된 소자의 내구성에 악영향을 끼치게 된다.

제작된 소자의 내구성 및 장기 수명 성능을 검증하기 위해서, 광 투과도 측정 시스템 및 소자에 가해지는 전압을 실시간으로 조절 가능한 시스템을 개발하였고, 해당 시스템을 사용하여 기존의 산화텅스텐 기반 변색소자의 작동 조건에서 광 투과도 특성 및 전기화학적 특성을 확인하였다.

위의 실험에서 확인된 특성을 바탕으로 전하량 균형을 고려함으로써 효과적으로 수명을 연장시키는 알고리즘을 제안하였고, 100만회 동안의 반복 시험을 수행함으로써 가속수명 시험 시작부터 종료될 때까지 광 투과도 성능이 유지됨을 확인하였다. 가속수명시험을 수행한 이후의 시편에서 투과도 성능을 측정한 결과 45.4%으로 오랜 시간의 작동 이후에도 성능이 보존됨을 확인하였다. 결론적으로 적층 공정의 개선이 아닌 동적인 제어를 통해 전기변색 소자의 수명을 효과적으로 향상시킬 수 있음을 증명하였다.

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주요어: 전기 변색, 나노 입자 적층 시스템, 가속 수명 시험, 광 투과도 반복 시험

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