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Research Impact on Emerging Quantum Materials for Electrochromic Applications

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Abstract: Electrochromic devices (ECDs), which can dynamically vary optical properties under applied specific voltage, have been actively studied for use in applications requiring human comfort and energy efficiency (i.e. smart windows and electronic displays). In order to improve the electrochromic (EC) performances, many researchers have been using nanomaterials. In this respect, in the present study, we highlight some of valuable examples using WO_3 from unique nanostructures to quantum dots (QDs) and discuss the core relationship between nanostructuring and EC performances. In particular, the present study proposes major impact on the novel approach using QDs to upgrade technical value and application of ECDs.

Keywords: Electrochemistry · Electrochromism · Quantum dots · Nanostructures · Metal oxide

1. Introduction

Commercial success in smart windows for architectural buildings is fundamental for the development of electrochromic devices (ECDs) that can adjust solar radiation or incident visible light and thus improve human comfort and energy efficiency by changing transmittance levels according to the dynamic needs.^[1,2] Furthermore, owing to their unique ability to reversibly vary optical properties (color and reflection) depending on applied potential, ECDs have been extensively used in various applications such as electronic displays, military camouflage, and satellite thermal control.^[2-4] A typical ECD presents a multi-layered structure consisting of cathodic and anodic electrochromic (EC) layers, two transparent conducting layers, and an electrolyte layer, in which the EC reaction is generated when the ions are moved into and out of the EC layers by the electrochemical redox processes.^[5] Therefore, the performances of the ECDs, such as transmittance modulation, switching speed, coloration efficiency (CE), and cycling retention, are determined by the degree of reaction capacity and kinetics that occur in the EC layers. Among various EC materials reported thus far, oxide-based EC materials have received considerable attraction for their use in practical applications. This is so due to their unique advantages such as a good chemical and electrochemical stability and wide working temperature as compared to those of organic-based materials, which can be largely classified into "cathodic" coloring under ion insertion (WO₃, TiO₂, and Nb₂O₅) and "anodic" coloring under ion extraction (NiO, IrO₂, and FeO₂).^[6,7] Specially, oxide-based EC materials have a significant potential as a type of EC materials that satisfy the durability of the ECDs and can withstand repetitive damage in the full range of solar and climatic conditions to degrade lifetime of the windows.^[8] However, since oxide-based EC materials have low ion diffusion coefficient and electrical conductivity, these two factors can decrease the EC performances and restrict their actual application as EC materials in various industrial fields.^[1] Effective ways to overcome this obstacle include obtaining acceptable levels in specific EC performances to meet client requirements. There performances concern the number of electrical, optical, electronic, and electrochemical behaviors of the ECDs mainly determined by nanostructuring of EC materials. In particular, in the ECDs, the interfacial interactions of the EC materials with the electrolyte provide unobstructed ion channels, improve electron conduction, and shorten ion diffusion distance, all of which significantly affect the EC performances.^[3] Recently, several papers using quantum dots (QDs) as next-generation EC materials have been published.^[9] The EC behavior of ODs can be attributed to size-dependent optoelectronic properties to cause the collection of electrons in the conduction band and electron injection and optical transitions of surface energy states during the electrochemical reaction.^[9] It this respect, it should be noted that the overall understanding of nanostructuring of EC materials is essential for the development of the ECDs. Therefore, in the present study, we will discuss representative samples that include the content of highperformance ECDs using from various nanostructures to QDs.

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Figure 1. (a) Schematic illustration of the constitution and operation on the ECDs and spectra representing typical parameters of the EC performances ((b) transmittance modulation, switching speed, and cycling retention and (c) CE).

2. Electrochromic Devices (ECDs)

Figure 1a shows a typical laminate structure of the ECDs with five functional layers placed between two substrates.^[1,10] A commonly used substrate is glass, but polymer materials, such as polyethylene terephthalate, are an alternative to allow flexibility of the devices for expanding application of the ECDs. The central part of the ECDs is a pure ion conductor named as the electrolyte, which serves as an insulator for electrons and which separates cathodic and anodic EC materials. The ions in the electrolyte should have small ionic radius, such as lithium ions (Li⁺) and protons (H⁺), to be easily moved by an applied electrical voltage. One side in contact with the electrolyte consists of EC materials that can provide characteristics of both ionic and electronic conductiv-



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ities, of which WO₃ is well known as the premier example. For the other side, the EC materials with ion storage property complementary to that of the opposite side are used. The three-layer structure of cathodic EC material/electrolyte/anodic EC material is covered by two transparent conducting oxides (TCO), such as F-doped SnO₂, Sn-doped In₂O₃, and Sb-doped SnO₂, which could be electrically conductive and optically transparent.^[11–13] The operation of the ECDs is possible when ions are inserted or extracted in the EC materials via an electrolyte by applied voltage to transparent conducting layers. At this time, the electrons balance on charge of ion-containing EC materials; hence, they are strongly responsible for the performances of ECDs.^[6]

In previous research, quantifiable parameters to compare between different materials or devices have been used as a criterion of EC performance. In what follows, we discuss the essentially-used parameters, including switching speed (τ) , transmittance modulation (ΔT), CE, and cycling retention. Proceeding of the coloration process needs to apply specific potential so that charge migration is generated in the constituent EC layers of the ECDs. The potential level related to electronic and ionic behavior in the component of the ECDs is generally reduced by realizing fast electrical conductivity and high ionic diffusion rate of the EC materials.^[14] The phenomenon is significant in terms of increasing the converting velocity between bleached and colored states of the ECDs. This is called to switching speeds defined as the time to reach 90% of the entire transmittance level (see Figure 1b), which improves energy efficiency and visual comfort for practical applications.^[8] For example, the smart windows function to save energy of the building by controlling intensity of illumination. Therefore, for an efficient energy control without visible irritation, it is important to quickly switch between the bleached state and the colored state of the ECDs. Furthermore, with an increase of the size of the devices, the switching speed gradually decreases, and even a faster value (from seconds to milliseconds) is required in display-related applications. Therefore, attention to upgrade this value constitutes a promising challenge for optoelectronic engineering fields.^[15]



Figure 2. (a) The SEM images depending on pore sizes of threedimensional ordered porous WO₃ films and illustration of bicontinuous electron/ion conducting and lights transmission on the films and (b) the formation mechanism of the mesoporous WO₃ films fabricated with the camphene-assisted sol-gel method and W 4*f* XPS spectra showing the existence of increased oxygen vacancy defects on the films. Copyright 2012 Elsevier. Copyright 2017 The Royal Society of Chemistry.

In addition, the ECDs need to quantify the varied degree of optical properties, which is transmittance modulation to be defined by difference in transmittance values of the bleached and the colored states measured at a specific wavelength. An essential prerequisite of a successful commercialization of the ECDs is being able to perform coloring-bleaching processes without degradation of the performances.^[8] As for conventional smart windows, the expected lifetime of the ECDs is 20-30 years, and the number of average working is 3-5 per a day. Therefore, sustainable cycling life of the commercial ECDs should operate in the range of 25,000-50,000 cycles. As another parameter, the CE (see Figure 1c) defined as the change in the optical density (Δ OD) to charge (O) intercalated per unit area (A) of EC layers indicates the standard to synthetically decide whether or not EC performance is good. It can be calculated using Eq. (1)–(2):^[1]

$$CE = \Delta OD / (Q/A) \tag{1}$$

$$\Delta OD = \log(T_{\rm b}/T_{\rm c}) \tag{2}$$

where T_b and T_c are transmittance values in the bleached state and the colored state, respectively. That is, a higher value of the CE indicates an efficient EC layer that has a large transmittance modulation at a small intercalated charge density. Therefore, in an effort to improve the EC performance, many researchers have tried nanostructruing of the EC materials, such as porous nanostructures, doped nanostructures, and composite nanostructures (see Section 3 for further discussion).

3. Nanostructured Approach of Electrochromic Materials

3.1 Porous Nanostructures

Porous nanostructures can effectively enhance EC performances to better penetrate the electrolyte into the EC framework and to shorten the diffusion distance of Li⁺ in the EC laver.^[16-18] Hence, numerous studies have reported various approaches and unique effects on the porous nanostructures for the ECDs. For instance, Yang et al. developed threedimensional ordered porous WO₃ films using the templateassisted sol-gel method and demonstrated the effect of pore sizes (325 nm, 410 nm, and 620 nm) on the EC performances.^[17] Compared to the nonporous film, the porous films revealed improved EC performances as the result of their unique structure features. As shown in Figure 2a, interconnected pores have an attractive impact of a continuous pathway that improves electron-ion conduction and increases the opportunities for a multiple light refraction in the interior layer. In addition, the ordered porous nanostructures enable Mie plasmon interaction in the layer surface, resulting in an improvement of the infrared (IR) optical modulation. In particular, the effect of small pore size on the ordered porous WO₃ films is much more favorable in terms of the improvement of EC performances with a faster switching speed (5.1 s for the coloration speed and 8.7 s for the bleaching speed) and a high CE value $(50.1 \text{ cm}^2/\text{C})$ due to the accelerated conduction of ions/electrons and increased transmission of the light. As a unique approach, in a previous study, we reported mesoporous WO₃ films with oxygen vacancy defects by using the camphene-assisted sol-gel method (see Figure 2b).^[18] This film is formed by the thermally decomposed effect of camphene to induce the Kirkendal effect of W^{6+} relative to the formation of a mesopore nanostructure and partial reduction of WO₃ relative to the formation of oxygen vacancy defects. As a result, the mesoporous WO3 films with oxygen vacancy defects fabricated by optimized camphene (10 wt%) exhibited outstanding multifunctional EC performances with fast switching speeds (5.8 s for the coloration speed and 1.0 s for the bleaching speed) and a high CE value (51.4 cm^2/C), which can be attributed to the synergistic effects of the mesoporous morphology with reduced Li⁺ diffusion pathway and effective electrostatic contact and oxygen vacancy defects with increased electrical conductivity and optical bandgap on the WO₃.

3.2 Doped Nanostructures

The general EC process involves a double insertion/extraction of the electron and cation (Li⁺) in the WO₃ layers: WO₃ (transparent) + $nLi^+ + ne^- \leftrightarrow Li_nWO_3$ (blue), which indicates that the diffusion rate of the cation in the WO₃ and the charge transfer at the interface between WO₃ and electrolyte are

related to the coloration and bleaching processes, respectively. Said differently, the electrical and optical properties that vary by carrier concentration and mobility of the WO₃ layers mainly determine the kinetics of the EC reactions. Therefore, many studies on the doped nanostructures have used suitable metal ions with a lower oxidizing capability or a higher electronegativity compared to those of host ions, such as Ti, Ni, Fe, and P.^[19–22] Interestingly, in addition to the modification of electrical and optical properties on the host EC materials, this approach can induce a variation of morphology or crystallinity, which is expected to benefit the EC performances of switching speed, CE, and cycling retention. As a major result, Cai *et al.* reported the hierarchical structure Ti-doped WO₃ films prepared using the hydrothermal method (see Figure 3a).^[19] The effect of Ti doping into WO₃ caused the



Figure 3. (a) The SEM images of the hierarchical structure of Tidoped WO₃ films and the results of reduced WO₃ crystallization and improved electrical conductivity by Ti doping into WO₃ and (b) the formation process for the tunneled P-doped WO₃ films by ignited red P and data showing accelerated P doping into WO₃ by ignited red P. Copyright 2013 and 2019 The Royal Society of Chemistry.

formation of a star-like surface structure and a reduction of the WO₃ crystallization, which have an important effect on obtaining the outstanding EC performances of wide transmittance modulation (49.1% at 750 nm), fast switching speeds (1.6 s for coloration speed and 1.7 s for bleaching speed) and a high CE value (68.0 cm^2/C) as the result of low charge transfer and ion diffusion resistances, together with superb electrical conductivity of Ti-doped WO₃. In a previous study, we developed a novel approach of tunneled phosphorus (P)-doped WO₃ films using the ignition reaction of the red P.^[22] The ignited red P generated the critical exothermic reaction on the WO₃ surface, which resulted in a partial surface deformation and vaporization of red P (see Figure 3b). Therefore, the optimized tunneled P-doped WO₃ films provided good EC performances with fast switching speeds (6.1 s for the coloration speed and 2.5 s for the bleaching speed) and a good CE value (55.9 cm^2/C), which can mainly be attributed to the enhancement of Li⁺ diffusion by the tunneled structure and electrical conductivity by P-doping WO₃ and improved electrochemical activity by the tunneled structure, respectively. In addition, due to the combined effects of the efficient charge transfer and the structural alleviation by the tunneled structure, this structure has a highly stable cycling retention (91.5%) after 1,000 cycles.

3.3 Composite Nanostructures

Compared to pure oxide, composite nanostructures based on WO₃ can have unique properties. One example is WO₃-TiO₂ composite, where the TiO₂ leads to the improvement of conductivity or durability during the EC reaction. To maximize these characteristics, much effort has been invested into the effective design of the composite nanostructure, such as WO₃-TiO₂ composite nanowires, WO₃-TiO₂ core-shell nanorods, and WO_3 -on-TiO₂ nanotubes.^[23-25] For WO_3 -on-TiO₂ nanotubes (named TiO₂/WO₃), it was noted that ion storage capacity is higher than that of pure WO₃ and TiO₂ NT due to the increased electrical conductivity by the composite effect of TiO₂ NTs and WO₃ external layer, causing a wide transmittance modulation with a high EC cycling stability (see Figure 4).^[25] Another approach has also been reported on WO₃ having well-dispersed metal nanoparticles that can generate plasmon-induced optical absorption of the visible light (Ag, Pt, and Au) and conductive nanomaterials of carbon nanotubes and indium tin oxide.^[1,26] In addition, several polymer materials (poly(3,4-ethylenedioxythiophene): poly(styrene sulfonate) (PEDOT: PSS), polyaniline, and prussian blue) have been recently used for composite with WO₃, which is useful to the complementary aspect between EC performance and stability as well as for realizing the flexible devices by using them as efficient surfactant and binder for the WO₃ adhesion with transparent conducting layers.^[27-29]

4. Recent Advances of EC Quantum Materials

Owing to their unique size-dependent absorption characteristics, as well as photovoltaic conversion ability and photo-



Figure 4. The SEM images of TiO₂ NT and WO₃-on-TiO₂ nanotubes (TiO₂/WO₃) and spectra of the improved transmittance modulation with the EC cycling retention on the TiO₂/WO₃. Copyright 2015 American Chemical Society.

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Review stability, the QDs have recently received much interest as nanomaterials. In addition, the specific function of the QDs can be controlled by their surface states modified with adjusting the functional group or combining other materials. Among these materials, commonly-used CdSe QDs, owing to their tunable light emission, are attractive candidate sources of light-induced coloration materials. Therefore, Liu *et al.* have recently suggested using novel composite CdSe QDs-conjugated polymer films for the ECDs (see Figure 5).^[30] A typical



Figure 5. The absorption spectra (blue) and PL intensity (red) of CdSe QDs solution; The inset: CdSe QDs solution at the sunlight (left) and at the irradiation of 365 nm (right); the TEM images of CdSe QDs, Optical transmittance change of samples fabricated with CdSe QDs, and the mechanism of light-induced coloration behavior. Copyright 2018 Elsevier.

conjugated polymer has long alkyl chains to limit ion diffusion and charge transfer near the surface of the CdSe QDs. To obtain a superb EC performance, they exchange to ligand with short chains providing a faster transport of electrolyte ion and charge, resulting in excellent electrochemical behavior on CdSe QDs-conjugated polymer films showing a wide transmittance modulation of 50.0% in visible regions and the switching speed within 1.2 s. Cong et al. group (see Figure 6) developed, the WO₃ QDs of average crystalline sizes down to 1.6 nm were using the colloid process with tungsten aryloxide as the precursor.^[31] Since the exciton Bohr radius of WO₃ is below 3 nm, this product shows a strong-quantum confinement regime to provide clear features related to size quantization effects of a blue shift in the near band edge emission and absorption. In addition, in the present study, the surface of the WO₃ QDs was stabilized by exchanging the octyalmine ligand to pyridine to improve their hydrophilicity and conductivity, which caused a superior electrochemical behavior of fast ion transport in charging/discharging for the QDs layers. These



Figure 6. Approach of the ultrafast EC performances based on downsizing to QDs region with the conductive ligand exchange, the TEM images of WO₃ QDs with average sizes of 1.6 nm, and the data of the ultrafast EC performances obtained using WO₃ QDs. Copyright 2014 Wiley-VCH Verlag GmbH & Co.

unique effects of the WO3 QDs achieved excellent EC performances with the coloration/bleaching speeds within 1.0 s and the CE value up to 154.0 cm²/C, which is superior as compared to inorganic or organic-related EC materials reported thus far. Considering that the approach of oxide-based QDs is only in its early stages of development, we believe that there is a considerable potential for ultrafast EC performances in future research on size quantization and ligand exchange using the oxide-based QDs, together with nanostructuring approaches of porous nanostructure, doped nanostructure, and composite nanostructure. More interestingly, since the attempts to form oxide-based QDs layers are based on the roomtemperature process, the possibility of success of the multifunctional EC devices, including flexibility, stretchability, and convergence with different functions with energy storage and photo-conversion, is an area to be addressed in further research.

5. Summary and Outlook

In this paper, we have reviewed the constructive approach from unique nanostructure to quantum materials for the ECDs and discussed their functions for improving the EC performances. Indeed, the nanostructuring effects of the WO₃ films,

such as porous nanostructure, doped nanostructure, and composite nanostructure, mainly include interaction of EC materials with the electrolyte, modification of optoelectronic characteristics, and complementary design with other materials. This is benefited for obtaining stable and fast EC performances by accelerating the capacity or kinetics of the EC reactions and controlling optical absorption. For the QDs, although a novel approach has only recently been reported, the potential to implement excellent EC performances is expected to be very high, as size quantization affects a blue shift in the near band edge emission and absorption. Furthermore, by exchanging the ligand on the surface of the QDs, this novel approach can accelerate their hydrophilicity and conductivity, thus resulting in the ultrafast EC performances that are much more competitive as compared to those reported so far. We believe that the technique using the QDs to provide excellent performances despite the room-temperature process will receive much attention as an attractive approach towards the success of future-oriented EC devices characterized by flexibility, stretchability, and multifunctionality.

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