

## ELECTROCHROMIC GLASS WITH MULTILAYER CONFIGURATION

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**Abstract-** Mirrors reflect the incident light and is an application tool of electrochromism. Self-darkening Electrochromic mirrors used at night, disallow the incident lights to dazzle by reflection from the mirror. An optically absorbing Electrochromic color is faded over the reflecting surface, reducing reflection intensity and thereby overcome driver discomfort. Opacity is to be prevented, as a muted reflection must persist in the darkened state. The back electrode is a reflective material, allowing customary mirror reflection in the bleached state. A method for retrofitting a window with a developed switchable or non-switchable window was designed to modify conventional glass or plastic windows. By combining such windows with a switchable glazing such as a Suspended Particle Device (SPD) film or with a non-switchable device such as a tinted window in a simple manner, and to the retrofitted windows produced. The method includes providing a window comprising at least one relatively transparent viewing pane with first and second opposed viewing surfaces.

**Keywords:** Electrochromic (ED), Self-darkening, Suspended Particle Device (SPD) Film, Electrochromic.

### I. INTRODUCTION

Electrochromism is a change in color as a function of an electrical field. The other behaviors of light-sensitive materials are thermochromism (color change with heat), photochromism (color change with light), and photostrictism (shape changes caused by changes in electronic configuration due to light [1]. Electrochromic (EC) a device deemed worthy of commercial exploitation, so the field is vast, [2].

The best-performance of EC mirror is described in this report, probably 90 to 95% of all self-darkening mirror follow the, process as expressed in [1]. A typical switchable glass is multilayered with an ElectroChromic Device (ECD) embedded inside. A window device may have a glass with an interior conductive oxide layer on both the top and bottom. Inside the sandwich of glass and conductive oxide is the ECD. This device consists of an EC layer, an ion storage layer, and between these two layers an ion conductor. Several different material families exhibit different behavior to a light stimulus.

An interesting light-sensitive material with both electro- and thermochromism behaviors,  $\text{Li}_x\text{VO}$ , was evaluated for a smart Windows application, [3]. Materials

have been developed to exhibit both photochromic and photographic (irreversible behavior to light) behaviors. One such system is based upon a substituted indolinospirobenzopyran embedded in a polystyrene matrix. This system performs as a photochromic system at low exposure in the UV range and as a photographic system at high exposures. The image can be devisualized by heat and can be restored with UV irradiation many times.

The most common applications are EC mirrors and windows, as below. Lampert, [4] has reviewed these and other applications on EC worldwide novel applications. Solid polymer matrix (SPMTM) mirrors for Lorries and trucks is a typical system, which relies on  $\text{WO}_3$  and NiO. A different solid-state EC mirror is based on  $\text{WO}_3$ , was reported by [5].

However, all-solid-state EC mirrors fitted to luxury cars were fabricated. The likely thermal and other stresses resulting from mounting ECDs in or on cars require particularly stringent tests, design and fabrication should be taken into the account. The durability of ECDs is discussed in Gesheva, et al. [6], which they have developed an EC mirror, that reflective to X-rays.

### II. DEVICE DESIGN AND SOME KEY CONCEPTS

One may think of many possible electrochromic device constructions. The most important and proved design is depicted in Figure 1. The materials arrangement is in the preferred sequence in almost all works aimed at the practical utilization, and the set-up is convenient also as a prototype for discussing electrochromic systems in general. The figure shows a number of layers backed by a substrate that in many cases is a glass plate.

The glass has a transparent and electrically conducting film and a film of the electrochromic oxide. This oxide has mixed conduction of ions and electrons, the ions are introduced from an adjacent electrolyte or via an adjacent ion conductor there is a corresponding charge-balancing counterflow of electrons from the transparent electron conductor. These electrons will remain in the electrochromic film as long as the ions reside there and, as indicated above, the electrons will then evoke a persistent change of the optical properties. Depending on which electrochromic oxide is used, the electron injection can increase or decrease the transparency.

The ion conductor, that comes next on the device, can be a thin film or a bulk-like material; for practical devices one prefers a solid inorganic or organic (polymeric) material, whereas liquid electrolytes are convenient for research and exploratory work. The final components of the device are an ion storage film, with or without electrochromic properties, and a layer of the electrical conductor that must be a transparent film if the full device is for modulating light throughput.

For some devices at least, it is suitable to have a second glass plate, so that the entire system contains two substrates, each with a two-layer coating and an intervening ion conductor that can serve as a laminate material. Each of the films in the device can have a thickness less than one micrometer; such films can be produced by many different techniques.

When a voltage is applied between the (transparent) electrical conductors, as indicated in Figure 1, a distributed electrical field is set up and ions are moved uniformly into and out of the electrochromic film(s). The charge-balancing counter flow of electrons through the external circuit, then leads to a variation of the electron density in the electrochromic material(s) and thereby a modulation of their optical properties.

If the ion conductor has negligible electronic conductivity, the device will exhibit open circuit memory, so that the optical properties remain stable for long periods of time.

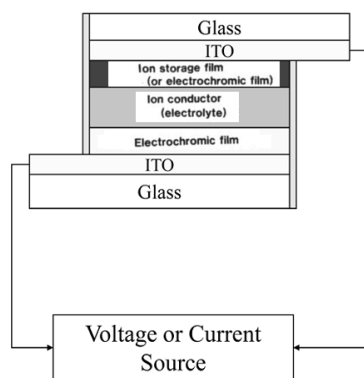


Figure 1. Basic design of an electrochromic device, indicating transport of positive ions under the action of an electric field. Five-layer electrochromic device, scheme composed of a glass-ITO substrate (GS-TC), electrochromic (EC), counter electrode (IS) film and the electrolyte (IC)

The device design in Figure 1 is appropriate for transmittance modulation, but the setup can be converted to reflectance modulation, for example by invoking a mirror or a diffuse scatterer behind the electrochromic film. In some cases the electrochromic film itself can turn reflecting, but it is more common that it exhibits absorbance modulation. The voltage applied between the outer films should be of the order of a few volts only. Higher voltages will lead to rapid deterioration of the device.

### III. EXPERIMENT

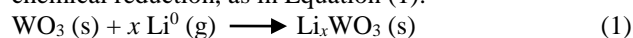
All electrochromic devices are electrochemical cells, so each contains a minimum of two electrodes separated by an ion-containing electrolyte. Since the color and optical-intensity changes occurring within the electrochromic cell define its application, the compositional changes within the ECD must be readily seen under work place illumination. In practice, high visibility is usually achieved by fabricating the cell with one or more optically transparent electrodes (OTEs), as below. Electrochromic operation of the ECD is effected via an external power supply, either by manipulation of current or potential.

Applying a constant potential in 'potentiostatic coloration', while imposing a constant current is said to be 'galvanostatic'. Galvanostatic coloration requires only two electrodes, but a true potentiostatic requires three electrodes [7], so an approximation to potentiostatic control, with two electrodes, is common. The electrolyte between the electrodes is normally of high ionic conductivity. In the early days of ECD development, the kinetics of electrochromic coloration were discussed in terms of 'types, type-I, type-II and type-III' as in the influential work of Chang, et al. [8].

In ECDs, the electrolyte viscosity can be minimized to aid a rapid response. For example, a liquid electrolyte (that actually comprises the electrochromes) is employed in the world's best-selling ECD, "Gentex rear-view mirror". The electrolyte in another type is normally solid or at least viscoelastic, e.g. a semi-solid or polymer. In fact, virtually the designed ECD remain solid during operation, as 'all-solid-state devices'. Such solid-state ECDs have multilayer structures, and a wide range of device geometries has been contemplated, [9-12] involving variations in the positions of the counter and working electrodes.

Alternatively, devices operating in a reflectance mode generally require the second electrode to be made of polished metal, the metal being chosen both for its electronic conductivity and its visual qualities, including its ability to act as a reflector. However, the color and reflectivity of the second electrode are unimportant if it is positioned behind a layer of electrolyte containing an opaque filler, [13]. Since the primary electrochrome is oxidized concurrently with reduction of the secondary (and vice versa when switching off), it is sometimes necessary to construct an all-solid-state ECD with one of the layers precharged with mobile ions, [14].

In practice, this is rarely a simple procedure. To effect this with say  $\text{WO}_3$ , lithium metal can be evaporated in vacuum onto the surface of one electrochrome film before device assembly so called 'dry lithiation', [15-17]. Elemental lithium is a powerful reducing agent, so gaseous lithium diffuses into the solid layer to effect chemical reduction, as in Equation (1):



In Equation (1), the  $x$  should not exceed about 0.3, since subsequent electrochemical extraction of  $\text{Li}^+$  in attempted re-oxidation is irreversible. Figure 2 schematic of a typical all-solid-state, multi-layer electrochromic

cell. Layer (i) is an optically transparent electrode, (OTE). The second electrode (ii) could be another inward-facing OTE. Layer (iii) is the primary electrochromic and layer, (iv) is the secondary. Layer (v) is the electrolyte.

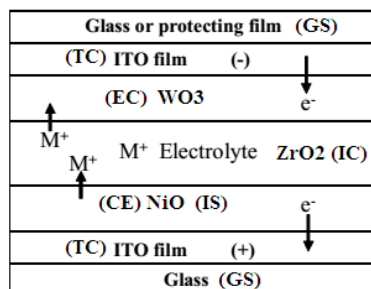


Figure 2. Schematic structure of the electrochromic process in electrochromic (EC) windows. It consists of thin EC and counter electrode (CE) coatings that are sandwiched between the two ITO films

Somewhat similarly, nickel oxide, in some commercial prototypes, is precharged using ozone; [18-19] in practice, films were irradiated with UV light in the presence of gaseous oxygen. Electrolyte layers for ECDs usage include 'Electrical and electrochemical properties of ion conducting polymers' was reported by Linford [20]. The layer of electrolyte between the two electrodes must be ionically conductive but electronically an insulator [21].

The electrochromic is dissolved in a liquid electrolyte, which can be either aqueous or a polar organic solvent such as acetonitrile or a variety of other nitriles, dimethyl-formamide, propylene carbonate or butyrolactone. The electrochromic systems working on the bases of the electrodes adaptation during electrochromic coloration. Solutions may also contain a dissolved supporting electrolyte in high concentration to suppress migration effects.

A thickener, such as acrylic polymer, poly(vinylbutyral) or colloidal silica, [22] may be added to the solution to increase its viscosity. This practice improves the appearance of an ECD because the coloration develops at different rates in different areas in a fast device, hence artificially slowing the rate of coloration helps ensure an even coloration intense. Thickening also improves the safety of a device should breakage occur, and helps minimize mass transport by convection. Gelling the electrolyte, e.g. by adding a polyether such as poly(ethylene oxide) PEO, is claimed to enhance the electrochemical stability.

In typical systems, while the electrolyte holds no soluble electrochromic, it has now enacted two roles. Firstly, during coloration and bleaching, for electroneutrality it supplies the mobile counter ions that enter and leave the facing solid-electrochromic layers. Secondly, the electrolyte still affects the accompanying conduction between the electrodes. Quite neglecting the latter, however, the electrolyte layer is called by some an 'ion-storage (IS) layer', which represents only the former action.

Thus an 'ion-storage layer' and an 'electrolyte layer' is by no means equivalent terms. Electrolyte layers for ECDs are one type ECDs operating with protons as the mobile ions can contain aqueous acids, [23].

Liquid acids are rarely used today owing to their tendency to degrade or dissolve electrochromes, and from safety considerations. A majority of particular ECDs now employs inorganic solids or viscoelastic organic polymers, the latter being flexible and resistant to mechanical shock. Solid organic acids of amorphous structure might serve similarly, although considerably higher potentials would be needed to drive any such ECD. They are apparently untested in this role, their electrical connectivity with electrochromes being critical. Ionic liquids, somewhat below their solidification temperature might also serve, but their ion-insertion capability could be questionable.

Inorganic and mixed-composition electrolytes of many ECDs contain as electrolyte a thin layer of solid inorganic oxide; thin film Ta<sub>2</sub>O<sub>5</sub> is becoming widely used. Such layers are generally evaporated or sputtered. However, they are mechanically weak and cannot endure bending or mechanical shock. To overcome the problem, mixed organic/inorganic solids like tetra-alkyl-ammonium salts with small inorganic anions, or alkali metal salts containing large organic anions (provided that insertions only of the smaller ion are required) may employ; these might show greater mechanical robustness.

#### IV. ORGANIC ELECTROLYTES

Semisolid organic electrolytes fall within two general categories: polyelectrolytes and polymer electrolytes, as described below. Polyelectrolytes are polymers containing ion-labile moieties at regular intervals along the backbone. A popular example is poly(2-acrylamido-2-methyl-propane-sulfonic acid), 'poly(AMPS)', in which the proton-donor moiety is an acid. The molar ionic conductivity of polymers such as poly(AMPS) depends critically on the extent of water incorporation; wholly dehydrated poly(AMPS) is not conductive, but ionic conductivity increases rapidly as the water content increases.

Table 1 lists some polyelectrolytes used in solid state ECDs. Polymer electrolytes contain, as solvent, neutral macromolecules such as poly(ethylene oxide) - PEO, poly(propylene glycol) - PPG, or poly(vinyl alcohol) - PVA. Added inert salt acts to form an inorganic electrolyte layer. Common examples include LiClO<sub>4</sub>, triflic acid CF<sub>3</sub>SO<sub>3</sub>H, or H<sub>3</sub>PO<sub>4</sub>. The viscosity of such polymers increases with increasing molecular weight, so polymers range from liquid, at low molecular weight, through to longer polymers which behave as rigid solids.

It is quite common for polymeric electrolytes have an opaque white 'filler' powder added, such as TiO<sub>2</sub> to enhance the contrast ratio in displays. A white layer also dispenses with any need to tailor the optical properties of the secondary layer. The inclusion of particulate TiO<sub>2</sub> does not seem to affect the response times of such 'filled' ECDs, but the photocatalytic activity of TiO<sub>2</sub> may accelerate photolytic deterioration of organic materials such as the electrolyte and the stability of electrolyte layers.

Table 1. Solid ion-conducting electrolytes for use in ECDs

<i>Inorganic electrolytes</i>		<i>Organic polymers</i>	
Electrolyte	Refs.	Electrolyte	Refs.
LiAlF <sub>4</sub>	[24]	Nafion	[30]
LiNbO <sub>3</sub>	[25]	Poly(acrylic acid)	[31]
Sb <sub>2</sub> O <sub>5</sub> (inc. HSbO <sub>3</sub> )	[26]	Poly(AMPS)	[32]
HSbO <sub>3</sub> based polymer	[27]	Poly(methyl methacrylate)	[33]
Ta <sub>2</sub> O <sub>5</sub> (including 'TaOx')	[28]	PMMA ('Perspex')	[34]
TiO <sub>2</sub> (including 'TiOx')	[29]	Poly(2-hydroxyethyl methacrylate)	[35]

Multilayer EC devices, consisting of layers ITO / NiO / ZrO<sub>2</sub> / WO<sub>3</sub> / ITO using the PVD method have been deposited. The WO<sub>3</sub> is used as an EC layer, NiO as an ion-storage layer, and ZrO<sub>2</sub> as a solid electrolyte layer in the all-solid-thin film ECD. The optical transmittance varied between 3-59%. The device shows the coloration and bleaching time of 120 s and 2 s, respectively, with a good memory effect and desirable cycle-life. When a voltage is applied on ECDs or EC windows as shown in Figure 3, ions are inserted into the WO<sub>3</sub> film in the device, whose optical properties thereby are changed. It switches reversibly from transparent to dark blue upon electrochemical redox reactions.

The different layers of all-solid-thin film ECD are deposited by different physical vapor deposition techniques in a high vacuum coating unit. The Indium tin oxide (ITO) thin films, having a sheet resistance of 5-10 Ω/□, are deposited at room temperature by RF magnetron sputtering on organically cleaned glass substrate. Subsequently, NiO (250 nm) and ZrO<sub>2</sub> thin film (500 nm) are deposited by e-beam evaporation method on the ITO coated substrate held at room temperature and 200 °C, respectively. H<sup>+</sup> ions are injected into the NiO film, via ZrO<sub>2</sub> / NiO / ITO film structure by electrochemical cycling it in 0.1 M KOH electrolyte. After removing the film structure from the KOH electrolyte it is washed with distilled water to get rid of KOH and dried using dry air.

Now the WO<sub>3</sub> thin film (250 nm) is deposited by thermal evaporation method on above structure at 200 °C temperature. Finally, the ITO thin film is deposited on top of the above device structure. The substrate was continuously rotated at 40 revolutions per minute (rpm) during the deposition to improve the film's uniformity. A frame-shaped shadow mask having an area 2×2 cm<sup>2</sup> is used to avoid short-circuiting between the different layers from the edge.

We followed Figure 2 that is a typical ECD composition is formed a five-layer structure visualized in many papers. The figure described as GS / TC / EC / IC / IS / TC / GS, where GS is a Glass Substrate, TC is a Transparent Conductor, EC is an electrochromic coating, IC is an Ion Conductor and IS as an Ion Storage coating [36]. Depending on the application, the GS-TC can be a reflective (platinum or silver) or transparent coating leading to a reflectance or transmittance mode of operation, respectively. This multi-layer structure provides numerous possibilities to the manufacture devices, [37].

The ECD can be characterized by applying a constant voltage between both ITO electrodes to color and to bleach the device and alongside measuring the transmission spectra in 450-1100 nm wavelength ranges for both fully colored and bleached states. For the open-circuit memory effect measurement, a voltage pulse was applied for the coloration of the device and at the same time measured the optical transmittance spectra in an open-circuit condition. A square-wave pulse voltage was applied to the device for evaluating the switching time of the ECD and concurrently the optical measurements were performed using a laser diode (650 nm) and a Si photo-detector. For the cycle-life time measurement of the device, it was subjected to continual color-bleach cycling in the same way as for the switching time measurement.

During the EC coloration and bleaching processes of electrochemical reactions take place and the mechanisms for coloration and bleaching of the mentioned EC materials have been given, [38].

A number of different polymer materials can be used as the coating materials. These include plastisols, polyvinyl chloride, ethylene vinyl acetate and epoxies, especially useful are the ionic co-polymers of alpha-olefins and alpha-beta ethylenically unsaturated or carboxylic acids. One such ionic co-polymer material that is formed from ethylene and methacrylic acid is available commercially under the trademark "Surlyn". This material, in particular; inattention because of its clarity, elasticity and degree of adherence to glass, and perhaps the most widely used transparent coatings on glass containers.

Further description of several such coatings and methods of applying them is described in, [39-40]. Surlyn demonstrates a strong absorption band to light of about 2.4 μ (24,000 Å) wavelength. Soda-lime glass, in contrast, has a low absorption up to about 3 μ, so that the infrared light in a wavelength range around 2.4 μ will be more strongly absorbed by the coated glass than with uncoated glass. The possibility of using absorption characteristics to discriminate between coated and uncoated glasses is complex by the normal rapidity of line movement, and by the shape of the objects.

## V. CONCLUSION

Electrochromic devices have considerable commercial interest due to their controllable transmission, absorption and/or reflectance. For instance, these devices are mainly applied to glare attenuation in automobile rear view mirrors, (a mirror in an automobile that gives a view of the area behind a vehicle) and also in some smart windows that can regulate the solar gains of buildings. Most of the reports on materials for electrochromic devices and only some of them about complete systems are available. This paper describes all-solid-thin film electrochromic device, consisting of layers ITO / NiO / ZrO<sub>2</sub> / WO<sub>3</sub> / ITO using the PVD method and some of the recent ones on ECDs, whose development is possible due to the advances in nanotechnology.

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