Investigation of Combinatorial TiO_2- MoO_3 Mixed Layers to Optimize the Electrochromic Properties

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Abstract: *Electrochromic measurements have been performed to optimize the composition of reactive magnetron-sputtered mixed layers of Titanium oxide and Molybdenum oxide (TiO_2-MoO_3). Optical parameters and composition have been determined and mapped by using Spectroscopic Ellipsometry (SE). Scanning Electron Microscopy (SEM) with Energy-Dispersive X-ray Spectroscopy (EDS) has been used to check the SE results as it was used in our earlier paper [N. T. Ismaeel et al, Materials 2023, 16(12), 4204] for (TiO_2-SnO_2). Ti and Mo targets were put separately from each other, and the Indium-Tin-Oxide (ITO) covered glass and Si-probes on a glass substrate (30 cm × 30 cm) were moved under the two separated targets (Ti and Mo) in a reactive Argon-Oxygen (Ar-O_2) gas mixture. By using this combinatorial process, all the compositions (from 0 to 100%) were achieved in the same sputtering chamber after one sputtering process. The Coloration Efficiency (CE, the change of light transmission for the unit electric charge) of mixed metal oxides (TiO_2-MoO_3) that are deposited by reactive sputtering has been studied in an electrochemical cell. This paper aims to assess the results of investigations of such materials showing the enhanced electrochromic behavior compared to the pure materials.*

Keywords: *combinatorial sample; Titanium-Molybdenum oxide; reactive sputtering; electrochromic materials; Coloration efficiency.*

1 Introduction

Metal oxides are widely studied with respect to their electrochromic behavior and properties for applications such as display devices and smart windows. To decrease the absorbed heat in buildings, electrochromic films have been used as smart windows Electrochromic materials have been applied in energy-effective glazing, automobile sunroofs, smart windows, and mirrors. The structure of a smart window contains an electrochromic material layer (usually metal oxide) sandwiched between a transparent conductive layer and some solid electrolyte.

Recently, energy efficiency has been affected and focused on energy solution strategies for utilizing this important field.

The electrochromic process is based on a reversible redox process and characterized by the coloration efficiency (CE). The energy-saving windows (smart window), energy storage systems: such as electro-chromic (EC), photochromic and thermochromic have been designed on the same mechanism and both have sandwich device structures, and it was based on the electrochemical reaction of the electrode materials.

Transition metal (Titanium, Tungsten, Nickel, Vanadium, Molybdenum and others) oxide films are the most interesting and most widely studied materials for this purpose. Conventional thin film preparation methods include for instance: chemical methods (spin coating, sol–gel deposition, chemical bath deposition, Langmuir–Blodgett technique, etc.), chemical and physical vapor deposition, electrochemical methods (anodization, plating), see ref. [1] and references therein.

The EC properties of Ti oxide (TiO_2) were extensively studied prepared by the different methods: sputtering [2, 3], chemical vapor deposition [4] and spray pyrolysis [5, 6], various wet chemical techniques [7, 8], and anodization [9-11].

The most widely studied EC oxide is Tungsten oxide (WO_3), and films of this material have been prepared by several different methods. Examples for physical vapor deposition: thermal evaporation [12-16], sputtering [17, 18], pulsed laser deposition [19, 20] and spray pyrolysis [21].

Electrochromic Mo oxide (MoO_3) shows similar behaviour to W oxide, and widespread studies used films prepared by evaporation [22, 23], chemical vapor deposition [24], and wet chemical techniques [25, 26].

Regarding V-pentoxide-based materials with intermediate EC properties, we note films prepared by vacuum evaporation [27], sputter deposition [28], spray pyrolysis [29], chemical techniques [30-33], electrodeposition [34, 35] and inkjet printing [36].

Nickel oxide films (NiO) were rather considered in hydrous nickel oxide form as EC material [37, 38].

Nevertheless, relatively few publications studied the possible advantages (higher coloration efficiency) of the mixtures of different metal-oxides as electrochromic material. The electrochromic effectiveness (the change of light absorption for the same electric charge) can be higher in mixed metal-oxide layers.

Earlier, we performed experiments with mixed metal-oxides and found positive effect in electrochromic behavior. Ismaeel et al [39] determined the optimal composition of reactive magnetron-sputtered combinatorial mixed layers of Titanium oxide and Tin oxide (TiO_2-SnO_2) for electrochromic purposes. The maximum enhancement in light absorption was found at (30%) TiO_2– (70%) SnO_2 composition.

In other experiments, also combinatorial material synthesis approach has been applied for the binary MoO_3/WO_3 system. By using organic propylene carbonate

electrolyte cells in a conventional three-electrode configuration, electrochromic redox reactions have been made. Coloration efficiency data has been evaluated from the primary data plotted against the composition displayed a characteristic maximum at around 60% MoO_3. The localization of the maximum at 5% accuracy has been allowed in that combinatorial approach [40].

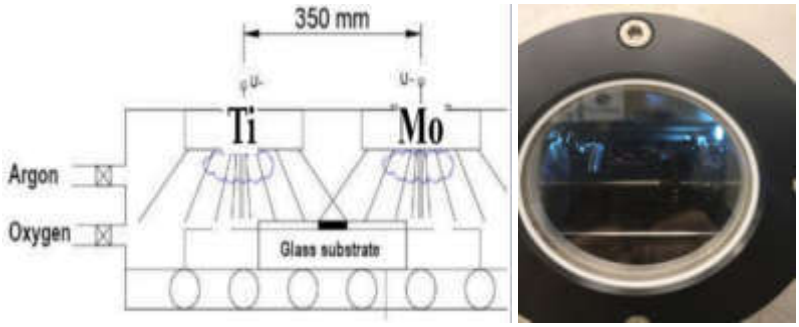
We found only a few publication about the EC behavior of Ti-Mo mixed oxide. Mahajan et al [41] reported the positive effect of doping of Ti (0, 3, 6, 9 at%) in MoO_3 thin films prepared by spray pyrolysis technique. Shrestha et al [42] successfully fabricated self-organized TiO_2–MoO_3 composite oxidenano-tubes with tunable dimensions by anodization. These nano-tube layers exhibited a significantly enhanced electrochromic color contrast compared with plain TiO_2 nano-tubes. Haiyan Yu et al [43] prepared MoO_3-TiO_2 composite core/shell nanorod films by the combination of hydrothermal and electrodeposition method. They attributed the improved electrochromic properties mainly to the porous space among the nanorods array, which makes the ion diffusion easier.

The objective of this work was to investigate the electrochromic effectiveness (the change of light absorption for the same electric charge) of TiO_2-MoO_3 mixed layers in a wide compositional range and the CE has been determined, too. Using metal atoms with different diameters in the layers would have a positive effect.

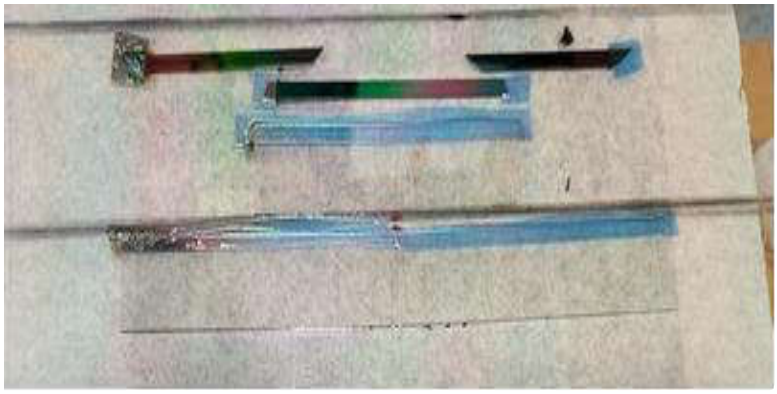
So this paper aims to assess the results of investigations of such materials showing the enhanced electrochromic behavior compared to the pure materials. One can expect that mixing metal atoms with different diameters in the layers can enhance the CE.

2 Materials and Methods

Optimal composition of reactive magnetron-sputtered have determined the combinatorial mixed layers of Titanium oxide and Molybdenum oxide (Mo_xTi_{1-x}) oxide thin film system, where $0 < x < 1$) for electrochromic purposes. Ti and Mo targets were put separately from each other, and the ITO-covered glass and Si-probes on a glass substrate (30 cm × 30 cm) were moved under the two separated targets (Ti and Mo) in a reactive Argon-Oxygen (Ar-O_2) gas mixture, see fig. (1a). Titanium-Molybdenum-Oxide layers were deposited onto Indium-Tin-Oxide (ITO) covered 100x25 mm size glass surfaces. Layer depositions were made by reactive sputtering in an (Ar + O_2) gas mixture at ~2×10^{-4} Pa base pressure and at ~10^{-1} Pa process pressure. The target - substrate working distance was 6 cm. 30 sccm/s Ar and 70 sccm/s O_2 volumetric flow rates were applied in the magnetron sputtering chamber. The plasma powers of the Ti and Mo metal targets were selected as (4200 and 1500) W respectively. Samples were moved back and forth at 25 cm/s of walking speed between the Mo and Ti targets and a mixed oxide film was deposited onto the ITO surface, see Fig. (1b). 5 min cooling interrupt was applied after every 50 walking cycles.



(a)



(b)

(c)

Figure 1

TiO_2-MoO_3 (a) arrangements of the two targets in a closer position (35 cm from each other) and the chamber for the DC magnetron sputtering device after being air vacuumed, blue light is from the Ar-O_2 plasma. (b) ITO-covered glass and Si-probes on a glass substrate, before-electrochromic-experiments, the Ti in the left and the Mo in the right. (c) after-electrochromic-experiments.

Spectroscopic Ellipsometry (SE) is an optical characterization technique with high-accuracy [44]. Several researchers have used SE for combinatorial or pure materials investigation [45-49]. The combinatorial approach used to investigate mixed metal oxides has several advantages. Fried et al. [49] have used SE (which is a fast, cost-effective, and non-destructive method) for the investigation and mapping of WO_3-MoO_3 mixed layers after sputtering. Different optical models, such as EMA and 2T–L, have been used to achieve the composition map and thickness map of the sample layers. We used SE similar manner to determine the composition map and thickness map of our Ti-Mo combinatorial layers.

To determine the optimal composition for the best electrochromic (EC) properties, the layers were deposited onto ITO-covered glass. The composition map and thickness map were measured on the Si-probes, see Fig. 1 (a and b). We checked the resulted compositional map on the Si-probes Fig. 1 (b and c) by using Scanning Electron Microscopy (SEM) with Energy-Dispersive X-ray Spectroscopy (EDS), see Fig. 3.

The Coloration Efficiency η is given by following equation:

$$\eta(\lambda) = \frac{\Delta OD(\lambda)}{q/A} = \frac{\ln\left(\frac{T_b}{T_C}\right)}{Qi} \tag{1}$$

where Q_i is the electronic charge inserted into the electrochromic material per unit area, ΔOD is the change of optical density, T_b is the transmittance in the bleached state, and T_c is the transmittance in the colored state. The unit of a Coloration Efficiency is $cm^2\ C^{-1}$.

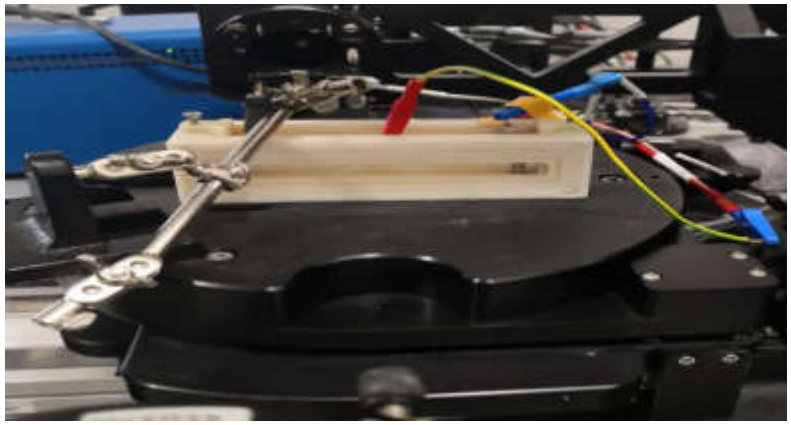


Figure 2

TiO_2-MoO_3-during-electrochromic-experiments by SE.

The Coloration Efficiency (CE) has been determined in a transmission electrochemical cell, see Fig. 2. The cell was filled with 1M lithium perchlorate ($LiClO_4$) / propylene carbonate electrolyte. A 5 mm width masked (Ti-Mo oxide-free) stripe of the slides remained above the liquid level allowing direct electric contact onto the ITO layer. A Pt wire counter electrode was placed into the electrolyte alongside with a reference electrode. This arrangement was a fully functional electrochromic cell.The applied current was controlled through the cell using a Farnell U2722 Source Measurement Unit (SMU). Constant current was registered through coloration and bleaching cycles of the electrochromic layer. Simultaneous spectral transmission measurements were performed by using the Woollam M2000 spectroscopic ellipsometer into transmission mode.

The precision of the Ti/Mo ratio is 2 %, while the precision of the position is 1 mm.

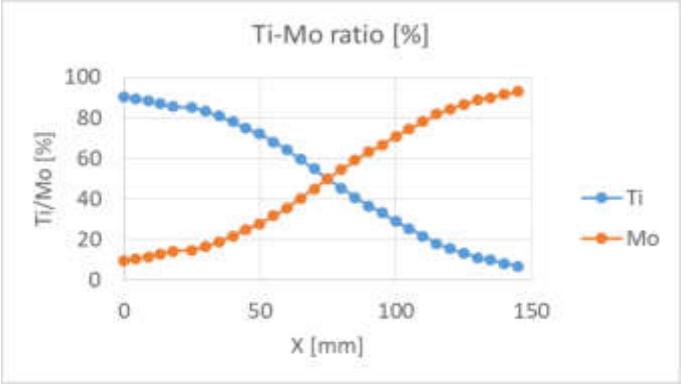


Figure 3

Ti/Mo ratio measured on the Si-probes by SEM-EDS.

3 Result and Discussion

The main criterion of the EC device performance is CE. Transmittance changes were directly measured during the coloration process, while the charge was calculated from the integral of the current vs. time data, and the electrolyte wetted area of the sample.

Figure 4 shows the calculated CE data as a function of MoO_3 fraction of the layer (individual color coded curves represent different wavelenghts), while Figure 5 is a 3D representation of the data. Individual points were calculated from the average of three independent measurements. Error is estimated as 3 %, calculated on the basis of the accuracy of sample positioning in the measuring cell and the spot size of the optical beam. The calculated data are given in Table 1 according to the equation (1).

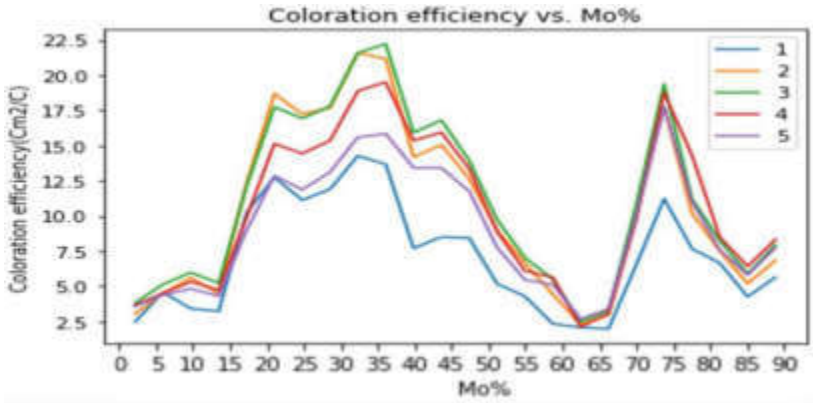


Figure 4

Coloration Efficiency of TiO_2-MoO_3. vs. Mo % for wavelengths from (400-800) nm by home-made software version 1.0 coded in Python version 3.11 language. (Individual color coded curves represent different wavelengths: 1 – 400 nm, 2 – 500 nm, 3 – 600 nm, 4 – 700 nm, 5 – 800 nm).

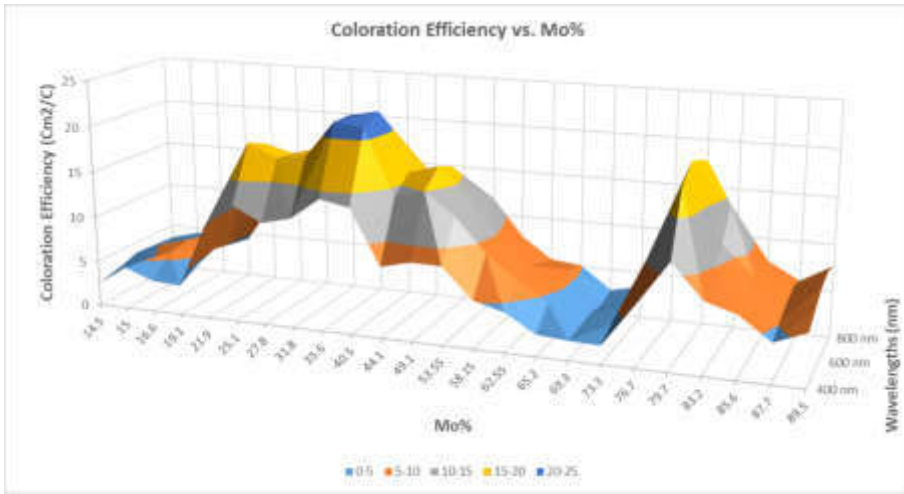


Figure 5

3D diagram of the Coloration Efficiency data of TiO_2-MoO_3. vs. Mo % for wavelengths from (400-800) nm visible spectral range.

To check the position dependent composition, Scanning Electron Microscopy (SEM) with Energy-Dispersive X-ray Spectroscopy (EDS) has been used, see Fig. 3. To explain the two maximums in CE (Fig. 4 and 5) we show here SEM-photos from the Ti-rich side and Mo-rich side, see Fig. 6 and Fig. 7. These are the proof that the material at the Ti-side is polycrystalline (several hundred nm) and the Mo-

side is amorphous or nanocrystalline. We think that the Ti-rich side was at significantly (several hundred C degree) higher temperature (plasma power 4.2 kW) during the deposition process, so the Ti-rich oxide is polycrystalline (several hundred nm grainsize) compared to the Mo-rich side where the oxide remains amorphous or nanocrystalline. This microstructure difference can be the reason for the two peaks in Fig. 4 and fig. 5, of the CE curves.

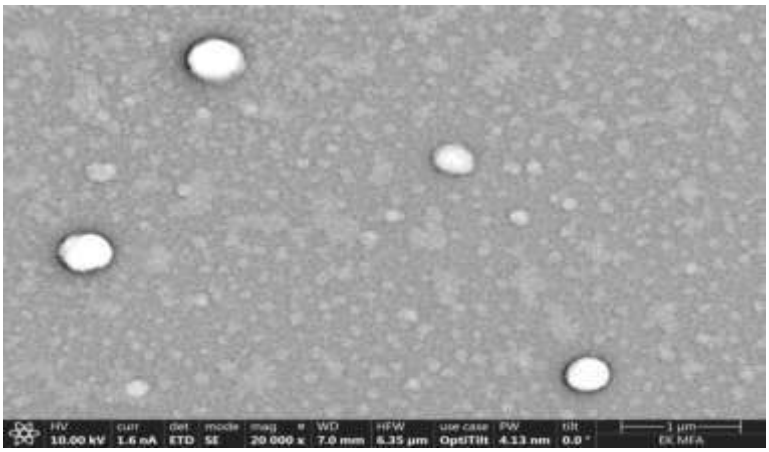


Figure 6

SEM micrograph from the TiO_2-MoO_3 surface Ti-rich side.

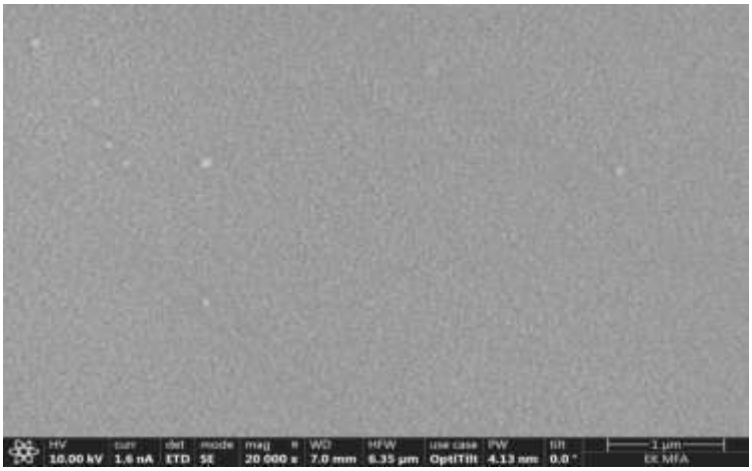


Figure 7

SEM micrograph from the TiO_2-MoO_3 surface Mo-rich side.

Table 1

calculated data for the CE according to the wavelengths (400-800)nm.

X(cm)	Mo (%)	400 nm	500 nm	600 nm	700 nm	800 nm
2	14.5	2.4	3.04	3.8	3.6	3.5
2.5	15	4.5	4.5	5.1	4.5	4.3
3	16.6	3.4	5.6	5.9	5.3	4.8
3.5	19.1	3.2	4.4	5.2	4.7	4.3
4	21.9	10.2	12.2	12.1	9.8	8.9
4.5	25.1	12.7	18.6	17.7	15.1	12.8
5	27.8	11.1	17.2	16.9	14.4	11.8
5.5	31.8	11.9	17.6	17.7	15.3	13.1
6	35.6	14.2	21.5	21.5	18.8	15.5
6.5	40.5	13.6	21.1	22.2	19.5	15.8
6.9	44.1	7.7	14.1	15.9	15.3	13.4
7.4	49.1	8.5	15.05	16.8	15.9	13.4
7.9	53.55	8.4	12.6	13.9	13.3	11.7
8.4	58.15	5.1	9	9.7	8.8	7.7
8.9	62.55	4.3	6.6	7.03	6.1	5.4
9.3	65.2	2.3	4.4	5.5	5.6	5.1
9.8	69.3	2.08	2.2	2.4	2.1	2.6
10.3	73.3	1.9	2.9	3.2	3.05	3.4
10.8	76.7	6.5	9.9	10.8	9.7	10.04
11.3	79.7	11.3	17.7	19.3	18.7	17.7
11.8	83.2	7.6	10.2	11.2	14.3	11.01
12.3	85.6	6.6	7.5	8.2	8.4	7.5
12.8	87.7	4.2	5.2	5.9	6.4	5.8
13.3	89.5	5.6	6.8	7.9	8.3	7.7

Conclusions

We could optimize the electrochromic properties of mixed Titanium oxide and Molybdenum oxide (TiO_2-MoO_3) layer deposited by reactive magnetron sputtering. We prepared combinatorial samples by moving the samples under the Ti and Mo sputtering targets in a reactive Argon-Oxygen (Ar-O_2) gas mixture.

By using this combinatorial process, all the compositions (from 0 to 100%) were achieved in the same sputtering chamber after one sputtering. The mixed metal oxides showed better EC properties than the pure oxides.

The Ti-rich side was at significantly higher temperature during the deposition process, so the Ti-rich oxide is polycrystalline compared to the Mo-rich side where the oxide remains amorphous or nanocrystalline.

Coloration Efficiency has been considered as the important parameter in this study. The maximum value of the CE is 22.2 $cm^2\ C^{-1}$ at the wavelength 600 nm at ~ 60% - 40 % Ti-Mo ratio on the Ti-rich polycrystalline material, while CE is 19.8 $cm^2\ C^{-1}$ at the wavelength 600 nm at ~ 20% - 80 % Ti-Mo ratio on the Mo-rich amorphous (or nanocrystalline) material.

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