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Structures and electrochromic properties of $Ta_{0.1}W_{0.9}O_x$ thin films deposited by pulsed laser ablation

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Abstract:

Thin films of $Ta_{0.1}W_{0.9}O_x$ were deposited on indium tin oxide (ITO) coated glass substrates by reactive pulsed laser ablation in O_2 ambient gas at substrate temperatures of 20, 200, 400, and 600 °C. X-ray diffraction (XRD) results showed that $Ta_{0.1}W_{0.9}O_x$ films crystallized mainly to a tetragonal phase at a substrate temperature of 600 °C, while films deposited at lower substrate temperatures were amorphous. Lattice constants of polycrystalline $Ta_{0.1}W_{0.9}O_x$ film deposited in 5.32 Pa O_2 are almost the same as those of stoichiometry $Ta_{0.1}W_{0.9}O_{2.95}$ material, which indicates a truly congruent ablation has been achieved by the PLD technique. Films deposited in lower O_2 pressures have poor crystallinity and may contain defects (oxygen vacancies). For all the temperatures investigated, $Ta_{0.1}W_{0.9}O_x$ films appear almost colorless when deposited in 5.32 Pa O_2 , while films have colors of light blue, blue and black when deposited in 2.66, 1.33 and 0.13 Pa O_2 , respectively. Optical transmission measurements showed that in the wavelength range investigated (250 ~ 850 nm), the transmittance of $Ta_{0.1}W_{0.9}O_x$ films decreases with decreasing the O_2 pressure during deposition. Electrochromic properties were also measured for the $Ta_{0.1}W_{0.9}O_x$ films deposited at 600 °C in various O_2 pressures. After H^+ ions were intercalated from 0.1 M H_3PO_4 electrolyte under an applied electrical potential, the colors of the 5.32 Pa and 2.66 Pa films change from almost colorless or light blue to deep blue, while the color contrast between as-deposited and H^+ intercalated films is not so significant for the 1.33 Pa and 0.13 Pa films.

Keywords: PLD; Pulsed Laser Deposition; Thin Films; $Ta_{0.1}W_{0.9}O_x$; Electrochromic

1. Introduction

Most of the electrochromic materials that have been investigated were based on single-metal oxides, with WO_3 being by far the most extensively studied one. Mixed oxides will be of growing importance in the future since improved durability, coloration efficiency and chemical stability, as well as a desirable neutral color could be accomplished in those multicomponent films [1]. Mixed oxides that have been studied with regard to their electrochromism include binary oxides, such as Ta-Ti oxide [2], W-Mo oxide [3,4], W-V oxide [5,6], W-Nb oxide [7], W-Ti oxide [8-11], W-Cu oxide [5], W-Sn oxide [12] and Mo-V oxide [6,13], Ni-Ti oxide [14] and ternary oxide, such as W-Mo-V oxide [6] and W-Ti-Nb oxide [15,16].

Many examples have demonstrated that addition of one metal oxide into another is an effective way to alter the properties of the individual constituents. It was found that the presence of small amounts of TiO_2 within Ta_2O_5 can significantly improve the insertion capability of Li^+ ions and electrochromic efficiency [2]. Matsouka et al. have studied the influence on the durability of WO_3 films with additions of TiO_2 , ZrO_2 , Na_2O_5 , SiO_2 , Al_2O_3 , MgO , Cr_2O_3 , and NiO [11]. They found that addition of 10 mol. % TiO_2 to WO_3 films increased the number of allowed color/bleach cycles before breakdown by roughly an order of magnitude, while addition of all the other oxides diminishes the durability. Transmission values in the near IR region of electrochromic Nb_2O_5 film after mixing with TiO_2 decrease significantly at colored state indicating that the film block the electromagnetic spectra range responsible for heating conditions in building interior [14]. The ability to produce higher luminous coloration efficiency in the W-Mo mixed oxide films than for the individual constituents is another example of considerable interests. A film with an almost neutral color appearance when illuminated with daylight can be obtained on the ternary W-Mo-V oxide [6].

Many techniques are available for preparing thin film of electrochromic oxides. The major methods are categorized into physical methods (evaporation, sputtering, laser ablation), electrochemical methods (electrodeposition and anodization), and chemical methods (vapor deposition, sol-gel, spray pyrolysis, decomposition reaction, thermal oxidation) [1]. However, for preparing thin film of multicomponent oxides, most of the methods are difficult to achieve the desired chemical stoichiometry. Pulsed laser deposition (PLD) is one of physical vapor deposition techniques capable of reproducing the target composition with relative ease under appropriate conditions; therefore it is suitable for preparing films of multicomponent oxides. Besides, this technique also produces high kinetic and internal energy of the ablated species; which enhances the adhesion of films to substrates. It has better control of the microstructure and morphology of the films, which offers better control over the film properties. The PLD technique usually can operate at low processing temperatures without deterioration of film-specific properties, which is ideal for temperature-sensitive substrates [17]. Electrochromic films that has been prepared by the PLD technique include WO_3 [18], ZnO [19], V_2O_5 [20, 21], Ce-Ti oxide [20], LiCoO_2 [20], $\text{Li}_x\text{Ni}_{1-x}\text{O}$ [22], TiO_2 [23], Ta_2O_5 [24], Sb-Sn oxide [25], and Ta-Ti oxide [26].

In this work, we have used the PLD technique to deposit thin films of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ on ITO-coated glass substrates at various substrate temperatures and oxygen pressures. To our knowledge, this paper is the first report on the deposition of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films using the PLD technique. Our emphasis is placed on the understandings of the influence of the substrate temperature and the oxygen pressure on the crystal structure and optical transmittance of the films. Primary results on the electrochromic property measurements of the films will also be given.

2. Experimental details

The $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films were deposited by ablating a 90 mm diameter rotating $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_{2.95}$ target ($\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_{2.95}$, 99.9%, SCI Engineered Materials) in an advanced deposition chamber (PVD. Inc., PLD-3000) by means of a pulsed KrF excimer laser ($\lambda = 248$ nm, Lambda Physik, LPX-210i), at a repetition rate of 50 Hz. The on-target laser beam fluence was adjusted to about $2 \sim 3 \text{ J/cm}^2$. A 25 mm x 50 mm x 1.1 mm rectangle ITO coated glass (unpolished float glass, SiO_2 passivated / indium tin oxide coated one surface, $R_s = 6 \pm 2 \Omega$, SiO_2 layer thickness: 20 ~ 30 nm, ITO layer thickness: 150 ~ 200 nm, Delta Technologies, Limited) was used as the substrate for the deposition. To achieve uniform deposition over the entire substrate surface, the laser beam was rastered over the radius of the rotating target.

Before introducing an ITO substrate into the deposition chamber, it was ultrasonicated in acetone and isopropanol to remove adsorbed organic contaminations. After loading, the process chamber was pumped down below 2.67×10^{-4} Pa using a turbo-molecular pump. A blackbody-type heater that used quartz lamps on the top of the substrate allowed non-contact, radiation-based heating.

When the temperature reached a pre-set value, oxygen gas (99.995%, Air Liquide) was introduced into the chamber and its flow was controlled through a mass-flow controller to achieve a pre-set oxygen gas pressure of 0.13 ~ 5.32 Pa. The laser was then turned on and a pre-cleaning cycle of the target was performed for two minutes. Subsequently, the shutter that hid the substrate surface from the ablation plume was opened and the deposition started. After 10 minutes processing time, the laser was stopped and the substrate was allowed to cool down. The film thickness determined by using a spectrophotometer was in the range of 250 ~ 300 nm.

The structures of the films were examined by X-ray diffraction (XRD, Philips, X-Pert MRD) in the θ_0 -2 θ thin film configuration, where θ_0 was fixed at 1° . The transmittance was measured in

the 250-850 nm ranges using a fiber-optic-based spectrophotometer (Scientific Computing International, Film Tek 3000). Electrochromic property tests were performed using a Gamry PC3 potentiostat with a three-electrode cell consisting of a Pt counter electrode and a saturated calomel reference electrode. H^+ intercalation of a $Ta_{0.1}W_{0.9}O_x$ film was accomplished by bringing the $Ta_{0.1}W_{0.9}O_x$ film in contact with 0.1 M H_3PO_4 electrolyte at a applied constant electrical potential for more than 3 minutes when no visual color change had been observed on the films. The sample was then taken out immediately, rinsed with Milli-Q water and dried with N_2 gas. Transmittance of the dried sample was measured using the spectrophotometer in the 250-850 nm ranges.

3. Results and discussion

Figure 1 shows XRD patterns of $Ta_{0.1}W_{0.9}O_x$ films deposited in 5.32 Pa O_2 at 20, 200, 400, and 600 °C, respectively. $Ta_{0.1}W_{0.9}O_x$ crystallized at 600 °C and its XRD pattern can be certainly assigned to the tetragonal crystal structure (ICDD 45-0115). The XRD data and peaks assignment of the 600 °C sample are given in Table 1. The average lattice constants of the film calculated from the data in Table 1 are: $a = 0.533$ nm and $c = 0.379$ nm. The values are very close to those of bulk $Ta_{0.1}W_{0.9}O_{2.95}$ obtained from thermal decomposition of Ta-doped peroxo-polytungstic acids at 750–900°C in air [27] (ICDD 45-0115), where $a = 0.5319$ nm, and $c = 0.3814$ nm were found. For films deposited at lower temperatures, the diffraction patterns consisted of a diffuse-scattering curve with a broad band centered at 2θ of about 25°. Such a profile indicates an amorphous-like structure. The broad band grows bigger as the substrate temperature increases from 20 °C to 400 °C, which indicates that more textured crystal structure was formed at higher substrate temperatures. Besides the broad band, a few small peaks originated from ITO layer also appear in the figure. Assignments of those peaks are shown in Table 1 as well.

Figure 2 shows XRD patterns of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films deposited at 600 °C in oxygen pressures of 0.13, 1.33, 2.66, and 5.32 Pa, respectively. XRD pattern of 2.66 Pa film is very similar to that of 5.32 Pa film except its peak positions shift to lower 2θ values. The shift in 2θ suggests that the lattice constants of 2.66 Pa film are larger than those of 5.32 Pa film. For the 1.33 Pa and 0.13 Pa films, not only the peak positions shift further to the lower 2θ values, but also the peak shapes become broader and their intensities become weaker. The XRD data suggest that the grain size of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films is probably getting smaller when they are deposited at lower oxygen pressures, and the crystallinity is also decreasing. The XRD peak located around $2\theta = 29^\circ$ disappears for the 1.33 Pa and 0.13 Pa films indicating that significant amount of defects (oxygen vacancies) may appear on the (101) index plane. The XRD pattern of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ film deposited in 0.13 Pa O_2 is significantly different from those of films deposited at higher O_2 pressures. XRD peaks of this film are not well-defined and their intensities are quite weak. This result suggests that the crystallinity of this film is poor and the film contains a large number of defects (oxygen vacancies). Clearly, the low O_2 pressure prevents the crystallization of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films.

The amorphous and crystalline films prepared in 5.32 Pa O_2 at different temperatures were almost colorless. Their optical transmittance spectra are similar at the whole wavelength region investigated (250 nm - 850 nm). The transmittance of 5.32 Pa films at short wavelength region (< 500 nm) is almost the same as that of the ITO glass substrate indicating very low light adsorption of the films. At long wavelength region (> 500 nm), transmittance is slightly lower than that of the ITO glass substrate, indicating some light adsorption in this wavelength region.

$\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films deposited in different oxygen pressures show very different colors. For all the temperatures investigated, the 5.32 Pa film appears almost colorless, while 2.66, 1.33, and 0.13 Pa films have colors of light blue, blue and black respectively. The optical transmittance spectra

shown in Figure 3 clearly demonstrated that transmittance of films strongly depends on the oxygen pressure during deposition. The 5.32 Pa film has excellent light transmittance, while the 0.13 Pa film is almost opaque in the whole wavelength region investigated. The fluctuations on the spectrum are relevant to the film thickness and originate from optical interference due to the multilayer (e.g., ITO, SiO₂ and Ta_{0.1}W_{0.9}O_x layers) component [18]. The origin of the coloration of the films, generally believed [18], comes from the presence of oxygen vacancies (i.e., Ta_{0.1}W_{0.9}O_{2.95-y}, where y > 0)) associated with tungsten and tantalum ions in lower oxidation state than 6⁺ and 5⁺ expected in Ta_{0.1}W_{0.9}O_{2.95} stoichiometry. Similar to the WO_{3-y} films deposited by PLD, Ta_{0.1}W_{0.9}O_x films deposited at lower oxygen pressure produce more oxygen vacancies, therefore show deeper colors. This result is consistent with the XRD results in figure 2.

Electrochromic properties were also measured for the Ta_{0.1}W_{0.9}O_x films deposited at 600 °C in various O₂ pressures. After H⁺ ions were intercalated into the Ta_{0.1}W_{0.9}O_x films from 0.1 M H₃PO₄ solution under an applied electrical potential, the color changed from almost colorless or light blue to deep blue for the 5.32 Pa and 2.66 Pa films, while not significant changes in colors were found for the films deposited in lower O₂ pressures. Figure 4a shows the results of 5.32 Pa film after H⁺ intercalation at E = - 0.5 V (vs. SCE) and E = - 0.8 V (vs. SCE). The transmittance spectrum of as-deposited film was also shown in the figure for comparison. It is clearly demonstrated that the optical transmission of Ta_{0.1}W_{0.9}O_x films decreases significantly upon H⁺ intercalation, especially in the long wavelength range (>350 nm). When the applied electrical potentials was changed to more negative values (e.g., - 0.8 V), more H⁺ ions were intercalated into the film, resulting in more light absorption of the film. Figure 4b shows the results of 1.33 Pa films after H⁺ insertion at E = - 0.5 V (vs. SCE) and E = - 0.8 V (vs. SCE). The optical transmittance of this already blue-colored film decreases much less significantly than the 5.32 Pa film upon H⁺ intercalation. Similar to the PLD WO_{3-y} film [18], the electrochromic behaviors of

$\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ could be interpreted to result from both injection of H^+ ions and electrons couple with the redox reactions $\text{W}^{6+}/\text{W}^{5+}$ and $\text{Ta}^{5+}/\text{Ta}^{4+}$. Films deposited in low oxygen pressures (i.e., 1.33 Pa and 0.13 Pa) have significant amount of oxygen vacancies, and the atomic ratio of $\text{W}^{6+}/\text{W}^{5+}$ and $\text{Ta}^{5+}/\text{Ta}^{4+}$ on those films is significantly lower than the films deposited at high oxygen pressures, therefore, under the same applied electrical potential, less amount of H^+ ions and electrons will inject into those films resulting in less significant coloration. In order to clarify those points and get a better understanding of the electrochromic behaviors of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$, more detailed studies are being pursued and the results will be reported in our future communication.

4. Conclusions

$\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films crystallized at the substrate temperature of 600 °C, while films deposited at lower substrate temperatures were amorphous. The crystallized $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films mainly contained the tetragonal phase. The lattice constants determined from XRD data for the tetragonal film deposited in 5.32 Pa O_2 are: $a = 0.533$ nm and $b = 0.379$ nm. Those values are almost the same as those of stoichiometry $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_{2.95}$ material prepared by Kudo et al. [27], which indicates a truly congruent ablation has been achieved by the PLD technique. Crystallinity of films deposited in lower O_2 pressures, especially in 1.33 and 0.13 Pa O_2 , is poor, and those low pressure films may contain defects (oxygen vacancies). For all the temperatures investigated, $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films appear almost colorless when were deposited in 5.32 Pa O_2 , while films have colors of light blue, blue and black when were deposited in 2.66, 1.33 to 0.13 Pa O_2 , respectively. Electrochromic behaviors were also observed first time for those PLD $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ films. After H^+ ions were intercalated from 0.1 M H_3PO_4 electrolyte under an applied electrical potential, the colors of the 5.32 and 2.66 Pa films change from almost colorless or light blue to deep blue, while the color contrast between as-deposited and H^+ intercalated films is not so significant for the 1.33 Pa and 0.13 Pa films

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Table 1: X-ray diffraction data for the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ film deposited at 600 °C in 5.32 Pa O_2 on ITO-coated glass substrate

	2θ	D_{hkl} (Å)	Identified planes (hkl)	Relative intensity (%)
$\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$	23.68	3.75	(110)	100
	28.91	3.08	(101)	1.33
	33.60	2.66	(111) and/or (200)	8.94
	41.42	2.18	(201)	1.31
	44.89	2.02	(211)	0.25
	47.78	1.90	(002)	1.36
	48.23	1.89	(220)	2.26
	54.36	1.69	(310)	4.14
	60.01	1.54	(311)	1.74
	62.45	1.49	(212)	0.32
ITO	31.05	2.88	(222)	Weak
	35.51	2.53	(400)	Weak
	51.40	1.78	(440)	Weak

FIGURE CAPTIONS

- Figure 1 : XRD spectra of $Ta_{0.1}W_{0.9}O_x$ films deposited at the substrate temperature of (a) 20 °C ; (b) 200°C; (c) 400 °C; and (d) 600 °C on ITO-coated glass substrate in O_2 pressure of 5.32 Pa.
- Figure 2 : XRD spectra of $Ta_{0.1}W_{0.9}O_x$ films deposited at the substrate temperature of 600 °C on ITO-coated glass substrate in O_2 pressure of (a) 0.13 Pa; (b) 1.33 Pa; (c) 2.66 Pa; and (d) 5.32 Pa.
- Figure 3 : Optical transmission spectra of $Ta_{0.1}W_{0.9}O_x$ films deposited at the substrate temperature of 600 °C on ITO-coated glass substrate in O_2 pressure of (a) 5.32 Pa; (b) 2.66 Pa; (c) 1.33 Pa; and (d) 0.13 Pa
- Figure 4 : Optical transmission spectra of $Ta_{0.1}W_{0.9}O_x$ films deposited at 600 °C in O_2 pressure of 5.32 Pa (a); 1.33 Pa (b) and on ITO-coated glass substrate at different applied voltages.

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Figure 1

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Figure 2

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Figure 3

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Figure 4