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Publisher's version / Version de l'éditeur:

<https://doi.org/10.1016/j.apsusc.2006.10.040>

Applied Surface Science, 253, 10, pp. 4740-4747, 2007-03-15

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Femtosecond Laser Patterning of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ /ITO Thin Film Stack

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Abstract : Selective laser patterning of thin films in a multilayered structure is an emerging technology for process development and fabrication of optoelectronics and microelectronics devices. In this work, femtosecond laser patterning of electrochromic $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ film coated on ITO glass has been studied to understand the selective removal mechanism and to determine the optimal parameters for patterning process. A 775 nm Ti:sapphire laser with a pulse duration of 150 fs operating at 1 kHz was used to irradiate the thin film stacks with variations in process parameters such as laser fluence, feedrate and numerical aperture of objective lens. The surface morphologies of the laser irradiated regions have been examined using a scanning electron microscopy and an optical surface profiler. Morphological analysis indicates that the mechanism responsible for the removal of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin films from the ITO glass is a combination of blistering and explosive fracture induced by abrupt thermal expansion. Although the pattern quality is divided into partial removal, complete removal, and ITO film damage, the ITO film surface is slightly melted even at the complete removal condition. Optimal process window, which results in complete removal of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film without ablation damage in the ITO layer, have been established. From this study, it is found that focusing lens with longer focal length is preferable for damage-free pattern generation

and shorter machining time.

PACS: 42.62.Cf; 79.20Ds; 81.20.Wk

Keywords: Femtosecond laser; Selective removal; Thin film; Electrochromic display

1. Introduction

Electrochromic metal oxides such as WO_3 and its doped derivatives have been widely used for the development of smart windows for energy efficient buildings, and large area information displays etc [1-4]. For most of these applications, electrochromic thin films need to be patterned to respective size and shape for the device of interest. When electrochromic thin films are to be used for display applications, an indium tin oxide (ITO) electrode is first patterned before depositing the electrochromic thin film. Photolithography technique is normally used to pattern the ITO layer through several complicated process steps, i.e. formation of a photoresist film on the substrate, ultraviolet light exposure, etching, etc. Although the photolithography technique is well established, it requires costly equipment and longer processing time. In addition, a patterned electrode can provide a colored pattern with diffused edges due to the diffusion of the electric field at the edges of the patterned electrode [5]. Therefore, simple and less expensive processes such as laser ablation for direct patterning of electrochromic thin film are essential.

Femtosecond lasers have been recognized as potential tools for micromachining due to their advantage in localized material removal and reduced heat-affected zone. In contrast to material processing using nanosecond or longer laser pulses where standard modes of thermal processes dominate, in the case of femtosecond laser-material interactions, only a small fraction of the laser pulse energy is transmitted as heat and transferred to the material surrounding the laser-irradiated area. Consequently, femtosecond laser pulses can induce nonthermal structural changes, driven by electronic excitation and associated nonlinear processes, before the material lattice attains equilibrium with the excited carriers. This fast mode of material modification can result

in the significant reduction of thermal stresses and minimal collateral damage for processing materials [6]. In this context, several research groups have studied the femtosecond laser machining of thin films for fabrication of various devices. Morpeceres [7] investigated the possibility of using selective ablation of ITO and amorphous silicon thin films for the development of photovoltaic position sensors with special emphasis on the morphological analysis of ablated grooves for different irradiation conditions. Park [8] investigated ultrafast laser ablation of ITO thin films as a function of laser fluence and the number of laser pulses for organic light emitting diode applications. Vankatakrishnan [9] fabricated photomask by selectively ablating the gold/chromium layer on quartz substrate using femtosecond laser pulses. Recently, Hermann [10] investigated the possibility of micromachining of CuInSe₂ (CIS) based photovoltaic devices which consist of ZnO, CIS and Mo thin films. In the case of WO₃ thin film, much work on pulsed laser deposition and surface modification using excimer lasers have been reported [11,12], however, no reports on the selective removal and patterning of electrochromic films such as WO₃ and its doped derivatives are available. In this work, selective removal of Ta_{0.1}W_{0.9}O_x layer from Ta_{0.1}W_{0.9}O_x / ITO thin film stack coated on the glass substrate has been investigated. Effect of the process parameters such as fluence, feedrate and numerical aperture of objective lens on the material removal was studied to understand the removal mechanism and to establish optimal process conditions. The surface morphologies of the grooves and patterns machined with different irradiation conditions were examined using a scanning electron microscopy and an optical surface profiler (WYKO, NT3300). The results of the morphological analysis indicate that the Ta_{0.1}W_{0.9}O_x thin film is removed by a sequential mechanism of blistering and explosive

fracture induced by abrupt thermal expansion, and that removal quality is divided into film blistering and partial removal, complete removal, and ITO film damage. Optimal process conditions for the selective removal of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ film have been finally established.

2. Experimental

2.1. Sample preparation

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 laser beam was focused down to a spot size of $\sim 4 \text{ mm}^2$ on the target surface and the on-target laser beam fluence was adjusted to about $2 \sim 3 \text{ J/cm}^2$. A rectangular (25 x 50 x 1.1 mm) indium tin oxide (ITO) coated glass (unpolished float glass, SiO_2 passivated / ITO coated one surface, $R_s = 6 \pm 2 \Omega$, SiO_2 layer thickness: 20 ~ 30 nm, ITO layer thickness: 150 ~ 200 nm, Delta Technologies, Ltd) was used as the substrate for the deposition. To achieve uniform deposition over the entire substrate surface, the laser beam was raster scanned over the radius of the rotating target.

Before introducing an ITO substrate into the deposition chamber, it was ultrasonically cleaned with acetone and isopropanol to remove the adsorbed organic contaminations. After loading, the process chamber was pumped down below $2.67 \times 10^{-4} \text{ Pa}$ using a turbo-molecular pump. A blackbody-type heater using quartz lamps was used on the top of the substrate to allow 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 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 process started. After 10 minutes of processing time, the laser was stopped and the substrate was allowed to cool down. The film thickness was determined by using a spectrophotometer and measured to be ~ 200 nm.

2.2. Laser system

In our experiment, a regeneratively amplified 775 nm Ti:Sapphire laser system (CPA-2010, Clark-MXR Inc.) with a pulse duration of 150 fs operating at 1 kHz repetition rate was used. The original beam was passed through a circular aperture placed in front of the beam delivery system in order to improve the beam quality. The incoming laser beam had a Gaussian profile with a diameter of about 6 mm after magnifying 2 times using a beam expander. The laser beam was subsequently focused with a microscope objective lens (Meiji S.Plan M5X, NA = 0.1; CVI laser M8X, NA = 0.2; Nachet M20X, NA = 0.3) on the surface of the specimen which was mounted on a computer controlled x-y translation stage having a resolution of 100 nm. The pulse energy was measured using a photodiode (Ophir, PD300-3W) that was placed after the microscope objective lens and varied using a rotary half-wave plate and a linear polarizer. The specimen was moved at scan speed, ranging between 10 - 100 mm/min in the direction perpendicular to the incident beam.

3. Results and discussion

3.1. Removal characteristics

For multi-layered films, the ablation threshold of each layer is the most important controlling factor for the selective removal of a layer of interest. We first investigated the single pulse damage thresholds of the ITO film and glass substrate by measuring the laser pulse energy which resulted in visible damage to the ITO film and glass substrate respectively. Since the beam diameter can not be measured precisely, we applied the damage threshold of 1.2 J/cm^2 for the glass substrate as a reference to calculate the beam diameter because the damage threshold of glass substrate is well defined (about $1.2\text{-}1.6 \text{ J/cm}^2$ at 150 fs pulse width) [8,13]. The single shot damage threshold of ITO thin films was then found to be $\sim 0.12 \text{ J/cm}^2$ for the objective lens with $\text{NA} = 0.1$. Single lines and rectangular patterns were machined on the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ / ITO thin film stack at a wide range of fluence and feedrate conditions with three different microscope objective lenses. The morphology of the laser irradiated area was then examined using SEM. For the case of $\text{NA} = 0.1$ (Meiji, S.Plan M5X), when the laser fluence was slightly lower than the damage threshold of the ITO film, a bubble or blister feature and partial removal of the 200 nm thick $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin films was observed as shown in Fig. 1. This blister feature was straight at lower feedrate (Fig. 1a), it turned into wavy pattern as the feedrate increased higher than 60 mm/min at the higher fluence (Fig. 1b). The surface morphology of the exposed ITO layer showed irregular ripple pattern which may be resulted from the surface melting by partially overlapping successive pulses after the removal of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film, whereas the surface of undetached $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ film remained intact. Therefore it appears that the damage threshold of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ film is higher than

that of ITO film. Various patterns with interesting morphologies e.g. hillcocks [14], straight blisters [15], and wavy patterns [16], have been reported in compressed thin films. However, the details of the mechanism are still unclear. Xiao et al [17] reported the controllability of laser-induced wavy pattern formation in aluminium thin films and proposed some aspects of the mechanism. The instantaneous injection of the thermal flux separated the irradiated region of the film from the substrate immediately due to the abrupt thermal expansion of the film and the large elastic mismatch between the film and substrate. Then the film is delaminated from the substrate and deformed into wrinkles right away through the release of the internal compressive stress. Recently, McDonald et al [18] reported, for a range of laser fluence, a blister or domelike features were formed in SiO_2 films on Si (100) substrates which were irradiated with single and multiple 150 fs laser pulses. They proposed that the expansion of the delaminated film from the substrate was driven by a combination of two forces: the relaxation of intrinsic compressive stress in the film, and the interaction of the electron plasma and expanding ablated substrate material with the delaminated film, and suggested that the laser induced blister formation takes place on time scale of hundred picoseconds to nanoseconds. As the laser fluence increased, the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film was completely removed, the pattern in ITO layer became more regular and clear as shown in Fig. 2a-f. These morphologies showed two distinct features of material removal, deep grooves were formed along the centerline as a result of exposure to the peak intensity of the Gaussian beam, and the exposed ITO surface consists of a highly oriented ripple pattern. The physical mechanism to produce such fine structures is not clear. It has been observed that a ripple like periodic surface structure is formed on laser ablated material surfaces. However, the size of the ripple

spacing is on the order of the laser wavelengths used, and then the ripple structure is understood as a spatial structure produced by the interference between the incident light and the surface electromagnetic waves excited by the incident laser light [19]. When the fluence was increased further, both $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ and ITO layers were completely removed, and the same ripple pattern began to appear on the glass surface (Fig. 3a) as seen in ITO surface at low fluence range, and finally the glass substrate was severely damaged at a fluence of 1.2 J/cm^2 (Fig. 3b).

Specifically, in the case of transparent $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ / ITO bi-layer system, the mechanism for selective removal of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ is believed to be completely different from the ablation of single layer and bulk material. From the results of morphological observation, the removal of the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film is assumed to be initiated by delamination from the ITO layer due to the abrupt thermal expansion of the film and deformation of the delaminated film through the release of the internal compressive stress. This assumption is supported by observation of the wavy pattern formed at the high feedrate, because the formation of the wavy pattern is due to the existence of the isopropic biaxial compression in the film [17]. However, it is still not clear if the ablation of the underlying ITO film with lower single pulse ablation threshold occurred and resulted in the softening and delamination of the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film as McDonald et al [18] observed in their work. The $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ and ITO thin films are known to be largely transparent at the laser wavelength of 775 nm [20]. Accordingly, in the linear case, the optical penetration depth is large ($\gg 1 \mu\text{m}$) compared to the total film thickness of 400 nm. In order to figure out whether the melting or ablation of the underlying ITO layer takes place before the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film is removed, the surface morphologies were

examined after irradiating single pulse on the ITO film and $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x/\text{ITO}$ bi-layer at various fluences. Figure 4 shows the morphological changes of the two systems with the increase in laser fluence. These results reveal that some portion of the incident laser light passed through the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ layer is absorbed in the underlying ITO layer and the deposited energy was used in the heating of the ITO layer, and that the ablation threshold of the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ film is higher than this level of fluence ($F = 0.168 \text{ J/cm}^2$). It is widely assumed that the ablation starts when the free electron density reaches the critical density at which the plasma oscillation frequency is equal to the laser frequency. Once the critical density is created the originally transparent material becomes opaque and the absorbed laser energy is deposited mainly in a very thin surface layer, which leads to the ablation of the thin layer on the order of picoseconds [21]. Although the ITO film is heated and melted, there is no evidence of the delamination of the ITO layer, and the melting and ablation of the underlying ITO layer does not seem to affect the blister formation and fracture mechanism. The blister formation is expected to be initiated by the delamination of the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ layer from the ITO layer due to abrupt thermal expansion of the film via electron heating, then the blister grows until it releases the internal compressive stress. The removal mechanism of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film is attributed to the fracture which occurs at the periphery of the blister. By the sequence of these two mechanisms, the film is finally blown off in solid fragments. These results are verified from the surface morphologies presented in Fig. 2 - Fig. 4, the kerf of the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film is much wider than that of the ITO layer, and the edges are not as straight as that of the ITO layer due to the consequence of an explosive fracture.

3.2. Optimal process conditions

The morphological change with respect to the laser fluence and feedrate was almost consistent regardless of the numerical aperture (NA) of the objective lens, but the available linewidth and the degree of damage to the underlying ITO layer are strongly dependent on the NA of the objective lens. Figure 5 shows the line patterns machined with three different objective lenses (NA = 0.1, 0.2, 0.3), the linewidth decreases with an increase in the NA because the laser beam diameter at the focus is linearly proportional to the focal length and is inversely proportional to the NA of the focusing lens. The measured linewidths for the patterns showing complete removal of the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film are $\sim 5.5 \pm 0.8 \mu\text{m}$, $3.2 \pm 0.5 \mu\text{m}$, and $1.8 \pm 0.3 \mu\text{m}$, respectively for the three numerical apertures. Based on these results, rectangular patterns were machined by scanning multiple lines with various spacing between scanning lines at a wide range of fluence and feedrate to seek optimal process window for the three different objective lenses. The surface morphologies of the machined patterns showed that the removal quality can be assessed into three zones of partial removal, complete removal, and ITO film damage. The results of the morphological analysis are plotted as a function of fluence and feedrate in Fig. 6. The three zones are well divided by the characteristic lines for all three NA conditions. The ITO damage zone becomes wider and the complete removal zone becomes narrower with an increase of the NA, this may be due to the difference of peak intensity at the beam center. As the NA of the focusing lens increases, the peak intensity at the beam center increases even though it has the same average intensity. Figure 7 shows the surface morphologies of the patterns whose removal quality was rated as

complete removal. With an increase in the NA, the damage of the ITO layer along the scan lines becomes severe due to higher peak intensity.

Analysis of the surface morphologies with respect to the removal quality showed that a focusing lens with longer focal length is preferable for the damage-free pattern generation, and also it has a great advantage of shorter machining time by applying wider spacing between the scanning lines.

4. Conclusion

Selective removal and patterning of electrochromic $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film from $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x/\text{ITO}$ films stack with femtosecond laser pulses are investigated. Morphological examination of the irradiated regions indicates that the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film is removed by a sequential mechanism of blistering and explosive fracture induced by abrupt thermal expansion, and that the pattern quality is divided into film blistering and partial removal, complete removal, ITO film damage, and glass substrate damage. Optimal process parametric window, which results in the selective removal of the $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ thin film with limited damage in the underlying ITO layer, become wider with increase in feedrate, fluence, and focal length of the focusing lens. These results confirm that direct patterning of $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x/\text{ITO}$ thin film stack with femtosecond laser pulses is a reliable process and can be used to replace the conventional photolithography based processes.

Acknowledgements

The authors would like to acknowledge Moe Islam, Director, Precision and Freeform Fabrication Technologies, for his support. Thanks are also due to Hugo Reshef and Matthew Shiu for their technical assistance.

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Fig. 1 SEM micrographs showing two different types of blister patterns; (a) straight, and (b) wavy

Fig. 2 SEM micrographs showing the change in surface morphology as a function of fluence (NA = 0.1, V = 30 mm/min)

Fig. 3 SEM micrographs showing the damage to the glass substrate at high fluence (NA = 0.1, V = 30 mm/min)

Fig. 4 SEM micrographs showing the surface morphologies after irradiating single pulse at various fluences near the ITO damage threshold; (a) ITO / glass, (b) $\text{Ta}_{0.1}\text{W}_{0.9}\text{O}_x$ / ITO / glass

Fig. 5 SEM micrographs showing the dependence of the linewidth on the NA of the focusing lens; (a) NA = 0.3, (b) NA = 0.2, (c) NA = 0.1

Fig. 6 Dependence of the removal quality on the process parameters

Fig. 7 Surface morphologies and profiles of the patterns with optimum removal quality; (a) NA = 0.3, (b) NA = 0.2, (c) NA = 0.1

Fig. 1

Fig. 2

Fig. 3

(a) ITO / glass

(b) Ta_{0.1}W_{0.9}O_x / ITO / glass

Fig. 4

Fig. 5

Fig. 6

Fig. 7