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Plasma-treatment of MgO film in a.c. plasma display

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Abstract

We studied the influence of the plasma-treatment on MgO film in plasma display panels by XPS, SEM and AFM analysis. With the plasma-treatment, adsorbed contaminants on MgO surface were removed and the surface topography was changed smoothly. We observed that the breakdown and sustained voltages of plasma-treated panels are stabilized more rapidly than those of non-treated panels.

Keywords: Plasma-treatment; MgO; Plasma display panels

1. Introduction

A MgO film is generally used for a protecting layer in plasma display panels (PDPs) owing to its good anti-sputtering property and its large secondary electron emission coefficient. However, MgO is a very hydrophilic material so that it can be easily contaminated in atmospheric environments. The major contaminant of MgO is adsorbed water. The chemically adsorbed water reacts on the surface in order to form $Mg(OH)_2$ [1]. The physically adsorbed water de-sorbs above 150 °C in vacuum and the chemically adsorbed above 350 °C [2]. Therefore, the exhausting process of the panel heating up to approximately 350 °C is necessary right after the sealing process of front and rear panel. Generally it takes a long time (~12 h) for this process because of the low conductance of panel itself. Furthermore, the high temperature process causes a degradation of phosphor, resulted in the reduction of luminance efficiency and the changing of color coordinate [3].

The degradation of phosphor is more serious in the sealing process owing to higher process temperature (~430 °C) than exhausting process. Therefore, in order to reduce the sealing process time for cost down and to avoid the degradation of phosphor during the high temperature process, room temperature sealing processes are being investigated by the panel makers. In the case of low temperature sealing process, an alternative cleaning method is necessary instead of the conventional heating method in vacuum. Recently, plasma-cleaning process is widely used for various materials [4]. There are two kinds of mechanism in plasma-cleaning process; one is removal of oxidized organic impurities from the surface, the other is the removal of MgO itself by the sputtering process [5]. In this paper we investigated the effect of plasma-treatment on MgO film in display panels with XPS, SEM and AFM methods.

2. Experiment

The MgO thin films used for the surface analysis were deposited on a cleaned soda-lime glass substrate by the electron-beam deposition method. These films were somewhat different from those of real panels. The film in real panels was good (1 1 1)-oriented crystal, as shown in Fig. 1, but the film for this analysis was more amorphous than that in real panels. Fig. 2 shows the schematic diagram of plasma-treatment system. The base pressure of the chamber was down to several 10^{-6} Torr. Argon gas was fed for RF discharge and the process pressure was 20 mTorr. The RF power tested for the treatment ranged from 100 to 800 W, and we found that the best RF power and the time for plasma-treatment are 400 W and 10 min, respectively. The front panel for treatment was loaded on the grounded electrode for the convenience of sample-loading and the MgO coating glass was covered on the RF powered electrode to avoid the contamination of by-product from powered electrode. We prepared the plasma-treated panel in order to make a real panel. The PDPs for the measurement of aging

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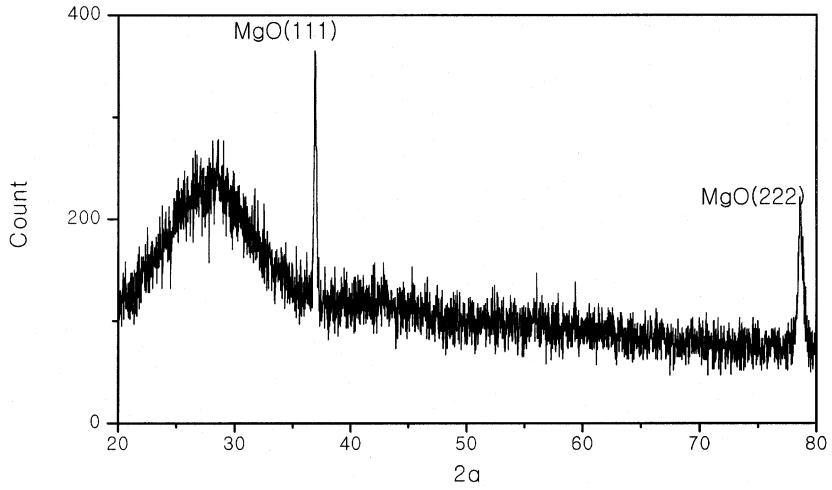

Fig. 1. XRD data of MgO thin film.

characteristics had three electrodes, two coplanar electrodes on the front panel and one electrode on the rear panel. The pixel pitch was 1080 μm, and its size was 40 in. in diagonal. A Ne–He–Xe gas mixture of 500 Torr was used and 5% Xe and Ne–He balanced with 9:1. The surface discharge having 5 kHz frequency was used for the aging process.

3. Results and discussion

The MgO is very hydrophilic material, so that in an atmospheric environment it is easily contaminated with moisture. Fig. 3 shows the XPS data of plasma-treated sample and non-treated one. An O1s peak splits to two peaks. The peak of 535.5 eV originated from MgO and that of 538 eV from $Mg(OH)_2$ [6]. Because H_2O in the air re-adsorbed on the surface during handling, the measurement was performed with ion sputtering in the analysis chamber. The relative quantities of oxygen from MgO and $Mg(OH)_2$ were calculated from the de-convolution of O1s peaks. The ratios of de-convoluted peak area were 1:0.589 in case of non-treated sample and 1:0.335 in case of treated one after 2 min-sputtering. From this result, we found that the plasma-treatment removes the adsorbed water from the surface.

The erosion rate of MgO with ion gun is ~20 Å/min and the contents of adsorbed water after 2 min-sputtering were smaller than those after 1 min. This meant that MgO surface is covered with the contaminants in tens of Angstrom when exposed to atmosphere for several hours (the thickness of $Mg(OH)_2$ layer depends on the time of exposure and humidity). Even though the further ion sputtering was performed during 6 min, the peak of 538.5 eV remains to the same degree as that sputtered for 2 min in our experimental condition. The reason of the peak of 538.5 eV not being completely

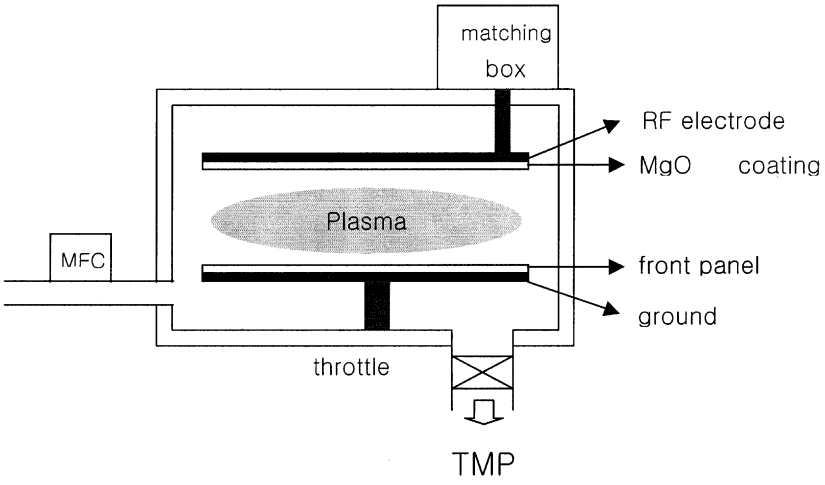

Fig. 2. Schematic diagram of the plasma-treatment system.

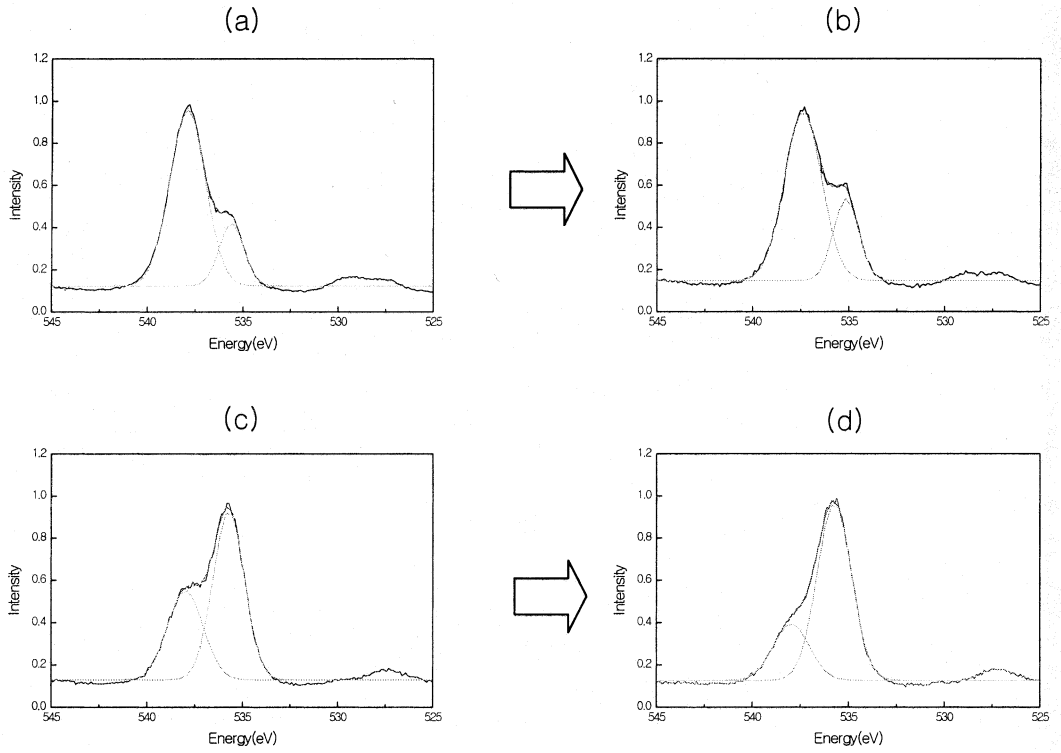

Fig. 3. XPS O1s spectra (a) before plasma-treatment (without sputtering); (b) after plasma-treatment (without sputtering); (c) before plasma-treatment (with 2 min-sputtering); (d) after plasma-treatment (with 2 min-sputtering).

disappeared after enough sputtering time was not clear. We thought that it was related to lattice water molecules generated during the deposition of MgO. We also analyzed the surface by using SEM and AFM. The roughness of MgO was bad before the plasma-treatment and became smoother after the plasma-treatment, as shown

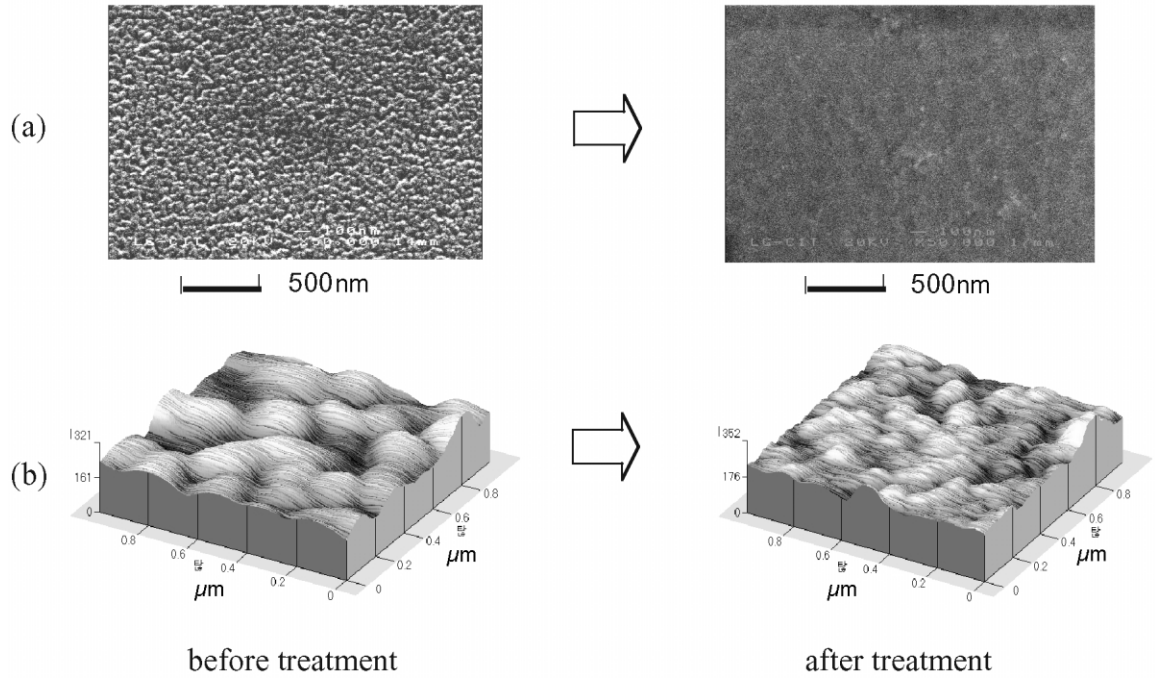

Fig. 4. (a) SEM image (×50 000) of before and after treatment; (b) AFM image (1×1 μm²) of before and after treatment.

in Fig. 4. This surface modification was owing to the ion bombardment. The surface topography depended on the conditions of treatment. The surface topography was not changed distinctly with a low RF power, because there was a minimum kinetic energy of ions required for the erosion of MgO. As RF power was increased, the surface was smoother. However, above a certain power the surface was going to get rough again by the severe erosion of MgO. According to these results, we found that the plasma-treatment not only removed the adsorbed impurities but also etched the surface MgO itself, and there is an optimal RF power for the surface smoothness.

In the aging process of PDPs manufacturing, the panel is operated for several hours in order to stabilize the discharge characteristics [7]. During this process the surface of MgO is bombarded by the energetic ions generated from a.c.-discharge. Before the aging process, the surface is covered with residual impurity gases in spite of exhausting, and is facetted to form a sharp edge in case of a crystalline. During the aging process the adsorbed impurities are de-sorbed or dissociated on the surface and the surface is getting smoother by the ion bombardments. Figs. 5 and 6 show XPS spectra and SEM micrograph before and after the aging process. The aging process and XPS analysis were performed in situ condition using a small front panel (1.5×2 mm^2). Fig. 5 shows the O1s spectra before and after the aging process in various temperatures. As the aging process was going on, adsorbed water was removed. The contents of water were reduced more rapidly at higher temperature than those of lower temperature. At high temperature the binding energy of adsorbed water was weak, so that water can de-sorb more easily than water at low temperature. In the aging process the surface of MgO became smooth, as shown in Fig. 6. We found that the changes of the surface during the aging process are very similar to that after the plasma-treatment.

We applied the plasma-treatment to the real front panel right before the sealing process and compared its discharge characteristics during aging process to that untreated panel. Fig. 7 shows the breakdown and sustained voltage characteristics of the untreated and plasma-treated panels. The elapsed time of the aging process was monitored. The breakdown and sustained voltages were changed as the aging time went on, and stabilized after a few hours. The reason was that the ion bombardment modifies the surface of MgO during aging process. As the ions bombarded the surface, the adsorbed impurities were removed from the surface and the pure MgO was exposed to the ion bombardment. The treated surface had a larger secondary electron emission coefficient than the untreated panel [8]. It was generally known that the secondary electron emission coefficient of the surface is strongly related to the breakdown and sustained voltages [9]. In addition, the ion bombardment makes the surface smoother and more amorphous to change the surface electronic structure. This surface modification influences to the discharge characteristics.

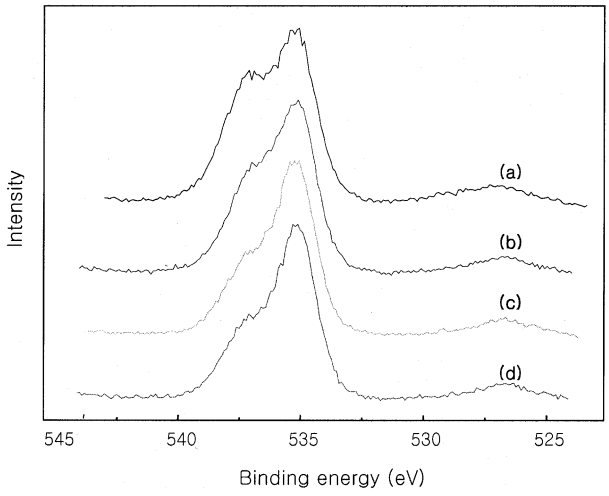

Fig. 6. XPS O1s spectra, before and after aging process in various condition (a) before aging; (b) after aging at RT; (c) after aging at 100 °C; (d) after aging at 250 °C.

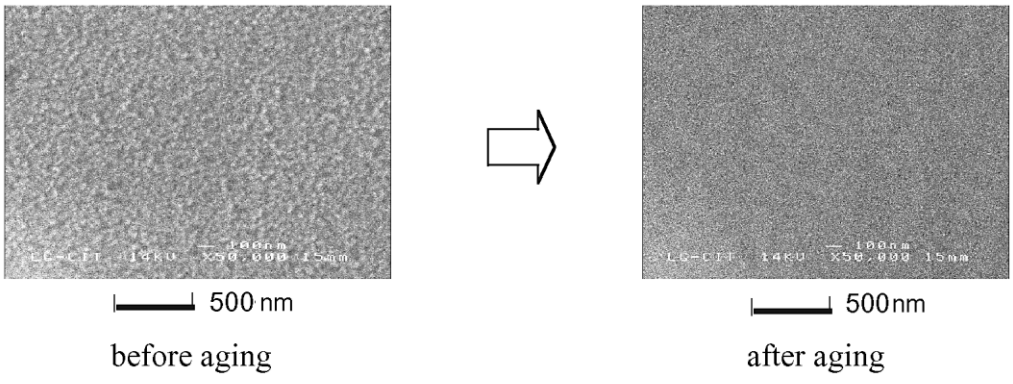

Fig. 5. SEM image ($\times$50 000) of before and after aging process.

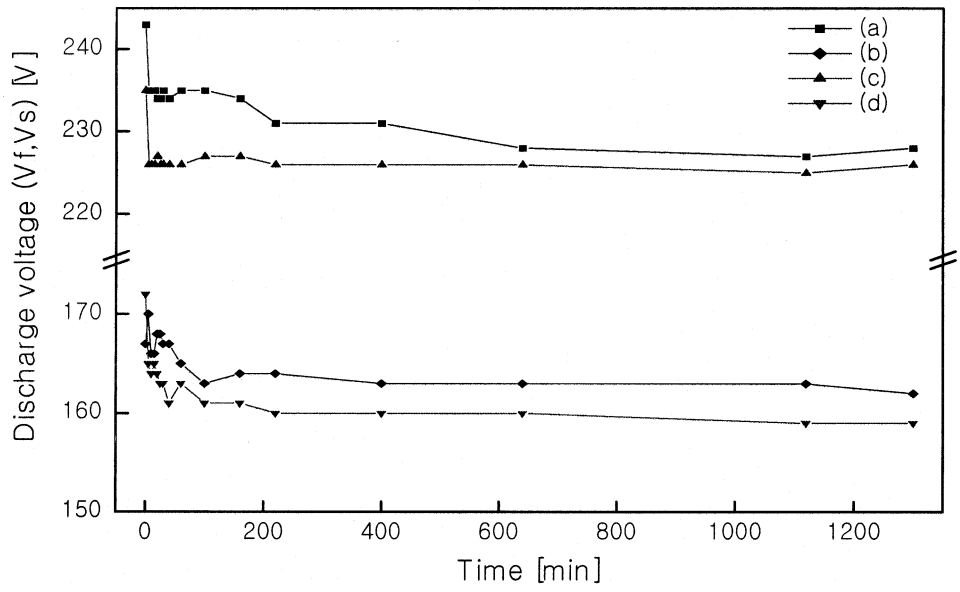

Fig. 7. Discharge voltage trend in aging process (a) breakdown voltage of reference; (b) sustained voltage of reference; (c) breakdown voltage of treated panel; (d) sustained voltage of treated panel.

Therefore, the aging process is required to make the better surface smoothness, and to stabilize the operation voltages. As shown in Fig. 7, the voltages of plasma-treated panel were stabilized more rapidly than non-treated one. It means that the plasma-treatment makes the surface-state closer to the state of the end step of aging process. Therefore, the plasma-treatment can reduce the time of the conventional aging process.

4. Conclusion

We investigated the effect of the plasma-treatment on MgO film used for protecting layer of PDPs. It was characterized by XPS, SEM and AFM analyses. We found that plasma-treatment removes the contaminants on the surface of MgO and at the optimal treatment condition the surface is smooth. The surface characteristic after plasma-treatment was compared to that after the aging process and the characteristics resembled each other. We applied the plasma-treatment to the real front panel and measured the discharge characteristics during the aging process. The breakdown and sustained voltages of plasma-treated panel were stabilized more rapidly than those of reference panel. We conclude that the plasma-treatment can play an important role to reduce the aging process time.

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