

## Optical and Structural Properties of Multi-period Low-emissivity Filters by RF Magnetron Sputtering

J.-H. Lee, S.-H. Lee, K.-L. Yoo, K.-S. Lee, C. K. Hwangbo

Department of Physics, Inha University, Incheon, 402-751 Korea

(Received)

### Abstract

Multi-period low-emissivity (low- $e$ ) filters based on  $[\text{TiO}_2/\text{Ti}/\text{Ag}/\text{TiO}_2]$  layer structure were designed and fabricated by a RF magnetron sputtering method. Optical, structural, chemical, and electrical properties were investigated with various analytical tools. Interface layers consisting of Ag, Ti, and O were observed next to Ag layers by Rutherford backscattering spectrometry (RBS) analysis. The results show that Ti layers of  $\sim 1.8$  nm protect the Ag layers from oxidation better than those of  $\sim 1$  nm and the optical spectra of the filter with thicker Ti layers are in agreement with the simulated one. The average transmittance of a low- $e$  filter with thicker Ti layers is reduced and the sheet resistance is slightly increased due to the increased Ti thickness.

### 1. Introduction

For the last two decades, low-emissivity (low- $e$ ) filter has been coated on glasses of automobiles and architectural buildings to reduce the solar radiation through glasses. Recently it is known that the multiple repetition of a basic low- $e$  coating with a noble metal film can be used for display applications [1-3]. For the optimal design and the improvement of the optical, structural, and electrical properties of the filters, the quantitative investigation for the filters with various analytical tools is essential [4]. In this study, we designed and deposited a variety of conductive multi-period low- $e$  filters based on a  $[\text{TiO}_2/\text{Ti}/\text{Ag}/\text{TiO}_2]$  layer structure by an RF-magnetron sputtering method and investigated the optical, structural, chemical, and electrical properties of the filters.

### 2. Design and Experimentals

We designed three types of low- $e$  filters of Type I,

II, and III, respectively, and fabricated them by an RF magnetron sputtering:

#### Type I

[air| $\{\text{TiO}_2(30 \text{ nm})/\text{Ti}(1.5 \text{ nm})/\text{Ag}(10 \text{ nm})/\text{TiO}_2(30 \text{ nm})\}^N$ |glass(1 mm)|air]

$N = 1, 2, 3, 4$

#### Type II

[air| $\{\text{TiO}_2(24 \text{ nm})/\text{Ti}(1 \text{ nm})/\text{Ag}(17 \text{ nm})/\text{TiO}_2(24 \text{ nm})/\text{TiO}_2(24 \text{ nm})/\text{Ti}(1 \text{ nm})/\text{Ag}(13 \text{ nm})/\text{TiO}_2(24 \text{ nm})\}^2$ |glass(1 mm)|air]

#### Type III

[air| $\{\text{TiO}_2(24 \text{ nm})/\text{Ti}(1.8 \text{ nm})/\text{Ag}(17 \text{ nm})/\text{TiO}_2(24 \text{ nm})/\text{TiO}_2(24 \text{ nm})/\text{Ti}(1.8 \text{ nm})/\text{Ag}(13 \text{ nm})/\text{TiO}_2(24 \text{ nm})\}^2$ |glass(1 mm)|air]

Ar was used as a sputtering gas to deposit Ag and Ti metal films, while  $\text{O}_2$  was added as a reactive gas for  $\text{TiO}_2$  films. A working pressure was  $3.6 \times 10^{-3}$  Torr for Ag and Ti films and  $5.9 \times 10^{-3}$  Torr for  $\text{TiO}_2$

films. BK7 glasses, Si wafers, and diamond-like-carbon (DLC) coated Si wafers were employed as substrates for the optical, structural, chemical, and electrical analysis. The optical properties were investigated by the measurement of transmittance and reflectance with a spectrophotometer. The structure of the filters and the effect of the thickness of Ti blocking layer were investigated with scanning electron microscopy (SEM), Auger electron spectroscopy (AES), and Rutherford backscattering spectrometry (RBS) and the chemical composition was examined by the AES and the RBS. The electrical properties were examined by measuring the sheet resistance with a four-point probe method.

We found that 10 nm thickness of a Ag film in Type I was discontinuous in terms of microstructure and electrically insulated. Also a Ag film deposited greater than 12 nm thickness resulted in good continuity and conductivity [5]. Therefore the thickness of Ag films was increased to 13 and 17 nm in Type II and III, respectively, depending on the design for optical performance.

### 3. Results and Discussion

#### A. Optical property

The measured transmittance of Type I was not in agreement with the simulated one because 10-nm-thickness Ag layers did not form the wet surface. Transmittance and reflectance of the optimized structures of Type II and III are shown in Fig. 1(a) and (b). The average transmittances are 61.1 % and 53.4 %, respectively. In Type II there is a discrepancy in transmittance between the measured and simulated ones due to the poor protection of Ag layers [6]. Thus we increased the Ti thickness in Type III. Although the average transmittance of the visible is decreased due to the absorption of thick Ti layers, the measured spectrum is in agreement with the simulated one and the transmittances above 800 nm is below 5.7 %. Type II and III filters block the infrared light below 10 %.

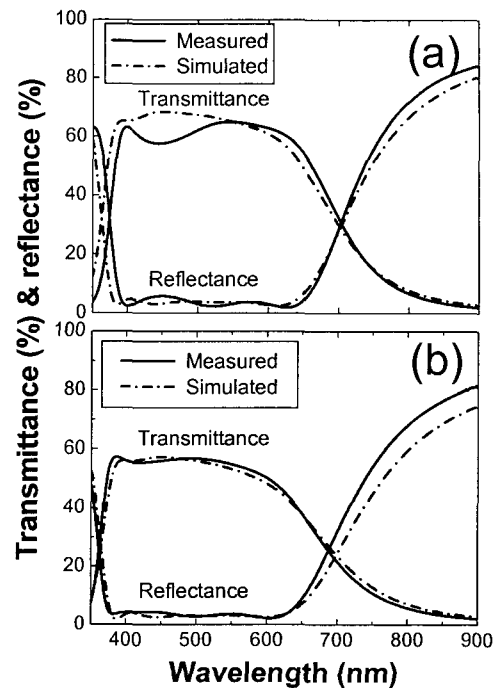


Fig. 1. Transmittance and reflectance of the filters of (a) Type II and (b) Type III.

#### B. Structural and chemical properties

SEM images of the surface profile of  $[\text{TiO}_2(24\text{nm})|\text{Ti}(\sim 1\text{nm})|\text{Ag}(13\text{nm})|\text{TiO}_2(24\text{nm})]$  layers showed many small particles on the surface and it turned out from the AES surface scan and depth profile that the composition of them was a mixture of Ti, O, and Ag due to the diffusion of Ag atoms into Ti and  $\text{TiO}_2$  layers. On the other hand, the surface of  $[\text{TiO}_2(24\text{nm})|\text{Ti}(\sim 1.8\text{nm})|\text{Ag}(13\text{nm})|\text{TiO}_2(24\text{nm})]$  showed negligible number of particles.

To investigate the discrepancy between optical spectra of Type II and III, we examined the two filters with the AES and RBS. The RBS spectra in Fig. 2 show that the Ag layers in Type III are protected better than those in Type II. In Fig. 2(a) a peak of Ag layer close to substrate is partially disrupted, whereas a higher Ag peak is shown in Fig. 2(b). Also it is shown that the third and the fourth Ti peaks around 1.25 MeV of Type II seem to merge

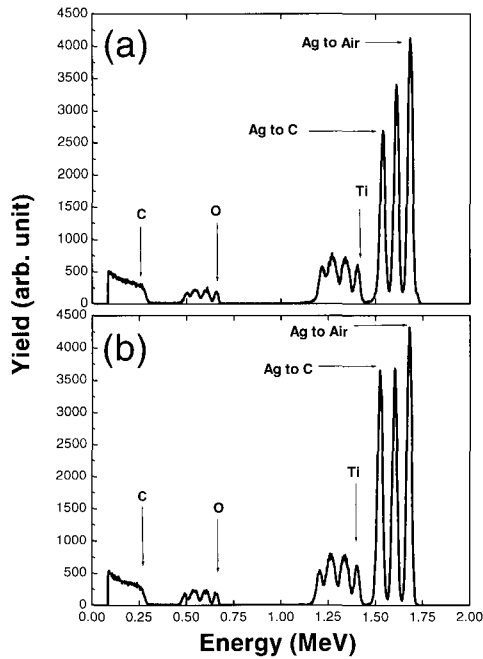


Fig. 2. RBS spectra of the filters of (a) Type II and (b) Type III.

together due to the disappearance of the Ag layer next to the substrate. Also the diffusion of Ag into TiO<sub>2</sub> layer from the magnified RBS spectra is observed. In Fig. 3, the Ag curves of Type II are higher than those of Type III in 1.55~1.58, 1.63~1.65, and 1.72~1.75 MeV. Note that the Ag of Type II is diffused farther into TiO<sub>2</sub> layers than that of Type III since those regions correspond to Ag atoms in TiO<sub>2</sub> layers. Thus, the protection of Ag layers from oxidation in Type II is poorer than Type III and the intermixed layers of Ti, O, and Ag are found above and below Ag layers.

### C. Electrical property

It is known that the sheet resistance of a filter is related to the ability of shielding of a hazard electromagnetic wave and the lower sheet resistance leads to the better shielding of electromagnetic waves [7]. The three and four periods of Type I showed the

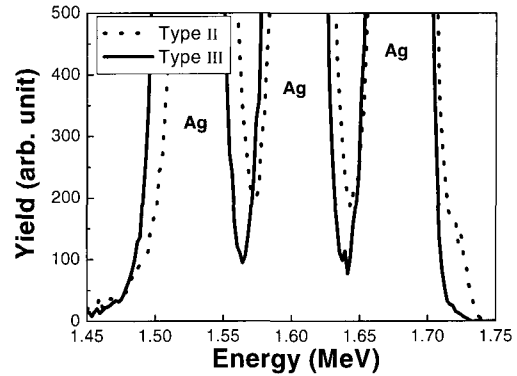


Fig. 3. The magnified view of RBS spectra of Type II and III for just Ag curves.

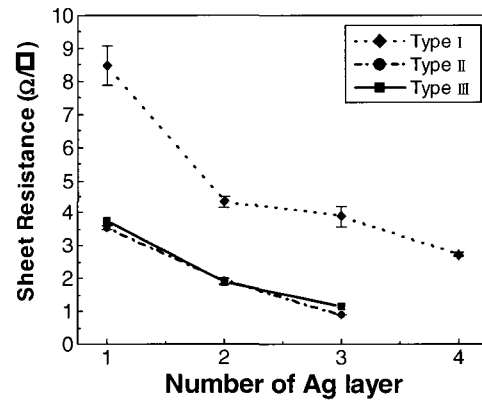


Fig. 4. Sheet resistance of Type I, II and III.

sheet resistances of 4 Ω/□ and 2.75 Ω/□, respectively. Type II and III showed 0.9 Ω/□ and 1.16 Ω/□, respectively. The sheet resistances of Type II and Type III satisfies the class B regulation of 1~2 Ω/□ for a display application, while that of three periods of Type I does not meet even the class A regulation of 2~3 Ω/□ [3].

### 4. Conclusions

We designed and fabricated three types of conductive low-*e* filters with the basic structure of [TiO<sub>2</sub>/Ti | Ag/TiO<sub>2</sub>] and the optical, chemical, structural, and electrical properties of the filters were investigated. The results show that, even if the optimized filter with

~1.0-nm-thickness Ti layers shows the highest average transmittance of 61.1 % in the visible and the lowest sheet resistance of  $0.9 \Omega/\square$  among the filters, the structure is unstable due to the disruption and diffusion of Ag layers. However, the optimized filter with the thicker Ti layers of ~1.8 nm for Type III showed better structural and chemical stability, while its average transmittance of the visible is lower and the sheet resistance is a little higher than those for Type II. The transmittances above 800 nm of Type II and III are less than 10 %. Also the RBS result shows that the intermixed layers exist around Ag layers if the protection of Ag layers by Ti blocking layers is poor.

### Acknowledgements

This work was supported by the research fund (98-0702-03-01-3) of Korea Science and Engineering Foundation, Republic of Korea.

### References

- [1] J. J. Finley, *Thin Solid Films* **351**, 264 (1999).
- [2] R. B. Farrington, 45<sup>th</sup> Annual Technical Conference of the Society of Vacuum Coaters, Orlando, O-1 (2002).
- [3] H. Ohsaki and Y. Kokubu, *Thin Solid Films* **351**, 1 (1999).
- [4] H. Weis, T. Muggenberg, P. Grosse, L. Herlitze, I. Friedrich, and M. Wutig, *Thin Solid Films* **351**, 184 (1999).
- [5] H. J. Glaser, "Large Area Glass Coatin," (Von Ardenne, Dresden, 2000) pp.220-226.
- [6] R. Hermann and G. Brauer, "DC and RF-magnetron sputtering," in *Handbook of Optical Properties*, R. E. Hummel and K. H. Guenther, eds. (CRC, Boca Raton, 1995), Chap. 6, pp. 174-175.
- [6] J. Szczyrbowski, G. Brauer, M. Ruske, H. Schilling, and A. Zmelty, *Thin Solid Films* **351**, 254 (1999).