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THE REFLECTANCE AND ADHESION OF SILVER FILMS PREPARED BY USING E-BEAM EVAPORATION ON POLYESTER SUBSTRATE

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Abstract

Thin films of silver with high reflectance of 95 % and above were fabricated successfully on polyester substrate by using e-beam evaporation processes. The optimum process condition was investigated by varying the current values applied while keeping the substrate temperature at room temperature by circulating the cooling water around it during deposition. Thin films of silver deposited with 30 mA as current revealed the highest reflectance of 96.4 %, while being illuminated with a light of 700 nm wave-length. But their adhesion showed unsatisfactory results. Though the films showed a condensation type in the cross-sectional views, they revealed crystallinity in the planes of (111) and (200) and growth orientation in <100> direction.

Key words : *silver film, thin reflective film, polyester, evaporation, room temperature deposition*

1. Introduction

The reflectors for fluorescence lamps worldwide were generally white-painted. It has been just recent that reflectors Al-anodized or vacuum-coated with a reflective thin film become popular as for their higher reflectivity values and saving electrical energy up to 40%[1] consequently. Al-anodization pre-requires an expensive facility for the industry, so high is the cost. If a higher productivity is sought to lessen this, the life time of product is sacrificed and becomes less, e.g. for 3 years only. Meanwhile, the reflective thin films are synthesized in general by using PVD methods with bright metals of Al or Ag.

We have investigated for a couple of years the possibility that e-beam evaporation[2)-5)] can be conveniently utilized to produce a quality thin film for such purposes. The reason behind is that CVD methods are improper for such polymer substrates [6] as polyester film. In this paper, we report the process conditions to fabricate silver reflective thin films on polyester film substrate and their resulting physical properties. And we also report the results of microstructural analysis for the specimens, to find some relations among them.

2. Pretreatment of Substrate

Polyester film of SH-71 grade with 50 ㎛ thickness manufactured by SKC[7] was employed as substrate. To prepare a clean specimen substrate without any contaminant particles on the substrate surface, each specimen substrate was cleaned in an ultrasonic bath with 10 % acetone, and sequentially with ethyl alcohol for 5 minutes each. Rinsing was carried out with distilled water right after the ultrasonic cleaning each time, then a blower was utilized to dry up. Finally, nitrogen gas was used to blow off any possible particles recaptured on the cleaned specimen.

3. Experimental Conditions and Depositions

As for the deposition source, a pellet type of quality silver with a purity of 99.99 % was used. The e-beam power was fixed as 7 KV and the distance between silver and specimen surfaces was maintained as 10 ㎝. For the deposition in general, vacuum chamber was evacuated down to 10^{-6} Torr level and the specimen holder was cooled with water being circulated to dissipate the thermal energy possibly accumulated during evaporation, which might be enough for distorting or damaging the substrate that is weak in heat.

During the evaporation processes kept for 2 to 10 minutes, working pressure was fixed as low as 4×10^{-6} Torr. Meanwhile the applied current was fixed to a value within a range of 10 to 40 mA to produce a sample. With various current values, corresponding samples were obtained to figure out the optimized process condition as a result.

4. Investigation Results and Discussion

The thickness of each specimen was measured with aids of an SEM and an α-step, then deposition rate of each thin film was calculated on the basis of measured thicknesses. In a case of 10 mA current, it revealed about 45 nm/min, while the current of 40 mA resulted in about 110 nm/min. The deposition rate was increased monotonically in general as the applied current increased from 10 to 40 mA as shown in Fig. 1.

Reflectance of each specimen was obtained by using a SpectroPhotometer with a light source of D65 condition in a wave-length range of 400 to 700 nm. As collected data are depicted as Fig. 2, the reflectivity values of Ag films revealed 95 to 96 % as illuminated with a light of 700 nm wave-length. The maximum reflectance showed 96.4 % with the films deposited at a current of 30 mA. This high value is well compared with a sample piece of CPF Co. revealing only 93% reflectance, as clearly seen from Fig. 3.

On the other hand, adhesion strength was measured by using a Sebastian pull-tester. The adhesivity showed 6 to 12 kg/㎠ along with the ap-

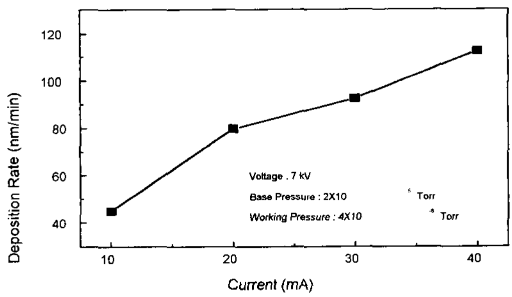

Fig. 1 The deposition rate as a function of current (Voltage : 7 KV, Current : 10~40㎃, Base pr-essure : 2×10^{-6} Torr,.Working pressure : 4×10^{-6} Torr).

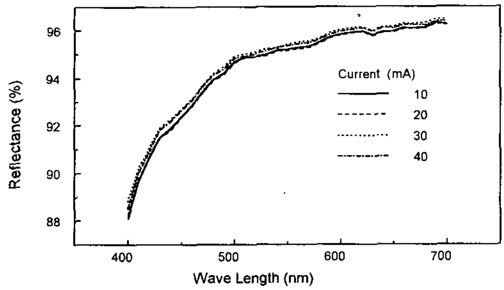

Fig. 2 The effect of current on the reflectance of Ag films, deposited by using e-beam evaporation. (Voltage : 7 KV, Base pressure : 2× 10^{-6} Torr, Working press:4× 10^{-6} Torr).

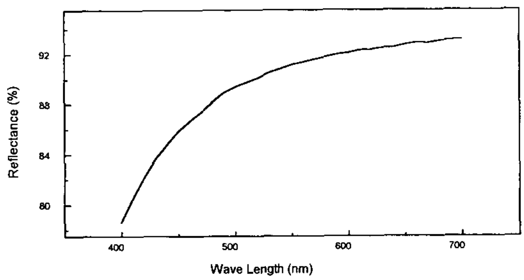

Fig. 3 The reflectance of Ag film manufactured by CPF Co.

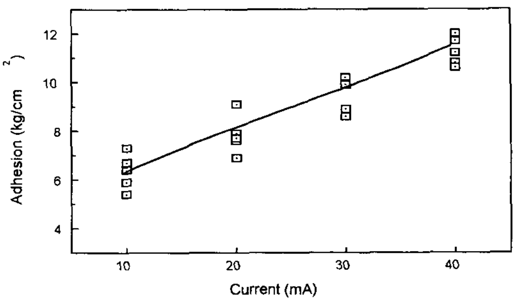

Fig. 4 The effect of current on the adhesion of Ag films (Voltage : 7 KV, Base pressure : 2× 10^{-6} Torr, Working pressure : 4× 10^{-6} Torr).

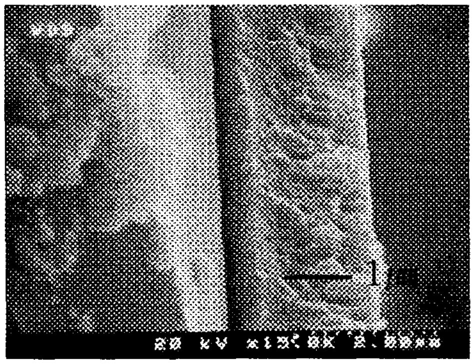

Fig. 5 A typical SEM micrograph of Ag films on the glass (D.C. power : 7 KV, Current : .30 mA,. Base pressure : 2× 10^{-6} Torr, Working pressure : 4× 10^{-6} Torr).

plied currents varied, as seen in Fig. 4. This seems a bit low for general use in industry. In order to find possible reasons for these low values of adhesion, cross-sectional structure was investigated by using an scanning electron microscope (SEM). Because of some difficulties in preparing SEM samples owing to the flimsiness of the polyester substrate, we prepared some more Ag films specially on glass substrate. Fig. 5 demonstrates a typical SEM photo revealing a cross-sectional view of such thin films. It was characteristic that the microstructure exhibited a type of condensation. We also examined the surface morphology by using an atomic force microscope (AFM). Fig. 6 shows a typical AFM photo for the specimens, which can be readily compared with Fig. 7 that is a typical AFM photo for the substrate sample piece of SKC Co. Meanwhile, the film crystallinity and orientation were analyzed by using an X-ray diffractometer (XRD). It resulted in the planes of (111) and (200) for the growth with a preferred orientation of <100>, in general. The typical XRD spectrum is shown as Fig. 8.

5. Concluding Remarks

The deposition rate was increased monotonically in general as the applied current increased

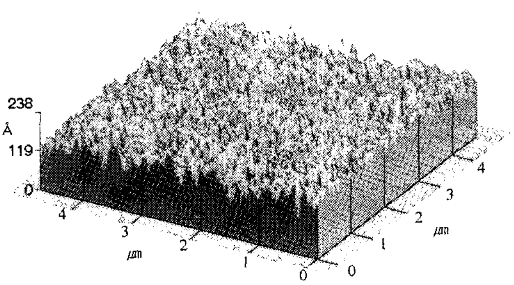

Fig. 6 A typical AFM surface topology of Ag films, deposited by using evaporation on the polyester film (D.C. power : 7 KV, Current : 30 mA, Base pressure : 2×10^{-6} Torr, Working pressure : 4×10^{-6} Torr).

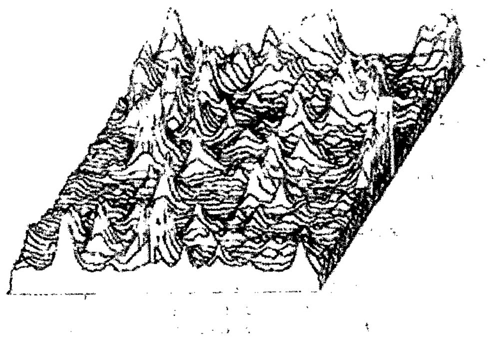

Fig. 7 A typical AFM surface topology of SH-71 polyester film manufactured by SKC.

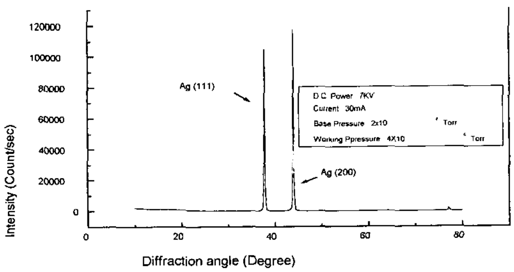

Fig. 8 A typical X-ray diffraction spectrum of Ag films, deposited by using e-beam evaporation.

from 10 to 40 mA. But optimized condition was obtained with a current of 30 mA with DC power of 7 KV. Thin films of silver deposited with 30 mA as current revealed the highest reflectance of 96.4 %, being illuminated with a light of 700 nm wave-length. But their adhesion showed unsatisfactory results. Though the films showed a condensation type in the cross-sectional views, they revealed crystallinity in the planes of (111) and (200) and growth orientation in <100> direction.

REFERENCES

1. CPF, "Sterling Silver Reflector," Engineering Manual.
2. S. Schiller, U. Heisig and S. Panzer, "Electron Beam Technology," John Wiley & Sons, Inc. (1982).
3. T. S. Sudarshan, "Surface Modification Technologies," Marcel Denker, Inc. (1989).
4. M. G. Hocking & V. Vasantasree, "Metallic and Ceramic Coatings," Longman Science & Technics (1989).
5. S. Dushman, "Scientific Foundations of Vac. Tech., (2nd ed.)," Wiley, New York (1962).
6. *RI, EuiJae*; "A Study On Domestic Status of the Metallic Thin Film Deposition Techniques Used for Various Electronic Components," KAITECH PER 91400-1 (1992).
7. SKC, Skyrol Polyester Film Technical Information.