



Research paper

Permeation barrier properties of silicon oxide films deposited on polyethylene terephthalate (PET) substrate using roll-to-roll reactive magnetron sputtering system

S.-H. Bang^a, Nong-Moon Hwang^a, H.-L. Kim^{b,*}^a Department of Materials Science and Engineering, Seoul National University, Seoul 151-742, Republic of Korea^b Power Reactor Fuel development, Korea Atomic Energy Research Institute (KAERI), Daejeon 305-353, Republic of Korea

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ABSTRACT

Silicon oxide (SiOx) barrier films were deposited on polyethylene terephthalate (PET) substrate at 20 °C using reactive dual magnetron sputtering system. The oxygen flow rate, input sputtering power and film thickness were changed to optimize the barrier properties of SiOx films. Details of sputtering power and film thickness effects on the SiOx properties in terms of residual stress, surface, roughness, density, interface of SiOx/PET, and water vapor transmission rate (WVTR) were investigated. Especially, we focused our attention on the effects of sputtering power on the final barrier properties. A high density of 2.42 g/cm³ SiOx film with high transmittance of 92% was obtained. Even at low substrate temperature of 20 °C, the 300 nm-thick SiOx barrier film exhibited superior WVTR value of 7.7×10^{-3} g/m²/day as a single barrier layer using roll to roll reactive sputtering. These experimental observations showed that the encapsulation barrier properties of SiOx films were significantly enhanced due to the change of effect of ion bombardment, which affected film density, surface roughness, and the interface of SiOx/PET.

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1. Introduction

The use of plastic substrates allows for many new applications in the field of flexible optoelectronic devices such as organic light-emitting diodes (OLEDs), organic photovoltaics (OPVs), and organic solar cell [1–6]. Especially, OLEDs are considerably attractive for display due to their inherent advantages of wide viewing angle, fast response time, high brightness, and low power consumption [7–9]. However, the long-term stability of flexible OLEDs is limited due to the instability of the organic materials and low work function metals by the permeation of moisture and oxygen into the plastic substrates which, unlike glass, have poor barrier properties [10–13]. This can lead to severe deterioration of their performance and reduces lifetime. Therefore, barrier layers are absolutely indispensable to protect OLEDs from moisture and oxygen before the great potential of OLEDs can be realized [12]. In order to ensure a long lifetime for flexible OLEDs device, water vapor transmission rate (WVTR) must be $<10^{-6}$ g/m²/day [8]. Generally, two ways are considered to get the required property. One is the optimization of barrier layers grown by atomic layer deposition (ALD) which has almost defect-free [5,8,14,15]. The other approach is a combination of inorganic and organic multi-laminated layers deposited by sputtering and chemical vapor deposition (CVD) [10–12,16–34]. Inorganic

materials such as SiOx [2,11,20], SiNx [2,5], SiOxNy [19,35], AlOx [14,20,36–38], ZnOx [14], MgO [8,9], TiOx [20,36], HfOx [38] which have high intrinsic impermeability are often used as diffusion barrier layers, while the organic layer functions as smoothing surface for the next barrier film, interrupting growth of defect, reducing mechanical stress, and lengthening the path of diffusion [20,39]. Although a perfect thin film encapsulation technique has not been developed so far, the above-mentioned of two approaches, the latter is more advantageous than the former given the economics. Reactive roll-to-roll magnetron sputtering is a low temperature deposition technique which can be used for attaining high quality oxide thin films. And also, this method has many other advantages including low cost manufacturing, easily controlled deposition process, and scalability to large area deposition with uniformity for various industrial application [40]. However, in the case of ALD process, practical application is expected to take some time due to the extremely low deposition rate about 1–3 Å/cycle and limited materials that can be deposited.

1.1. Rate

The main objective of this study is to develop the single layer of SiOx films with superior WVTR and high transmittance properties by optimizing process condition with roll-to-roll reactive magnetron sputtering system (Fig. 1), which can be applicable to the formation of multilayer barrier structures with organic materials. To achieve this

* Corresponding author.

E-mail address: hkim0904@kaeri.re.kr (H.-L. Kim).

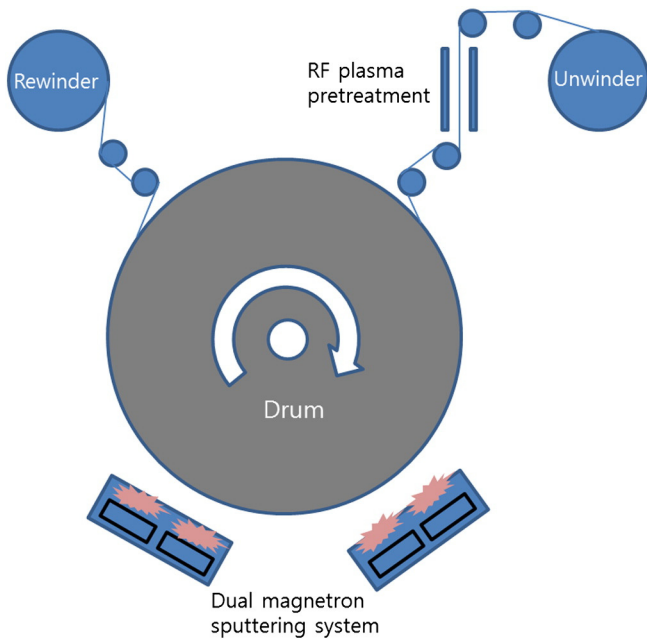


Fig. 1. Schematic diagram of the roll to roll dual magnetron sputtering system to deposit the SiOx films.

goal, chemical composition, surface roughness, density, residual stress, water vapor transmission rate were analyzed by means of X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), Rutherford backscattering spectrometry (RBS), laser profile meter. Detailed observation of the barrier films were carried out by high-resolution transmission electron microscopy (HR-TEM).

2. Experimental procedure

SiOx barrier films were deposited on polyethylene terephthalate (PET) substrate (Kimoto Tech. Inc.) with a thickness of 50 μm using roll-to-roll vacuum coater system. The machines can handle with a width of up to 300 mm and the web speed is variable between 0.1 and 10 m/min. This chamber consists of two process chambers which are pumped separately. One is for radio frequency (RF) of 13.56 MHz plasma treatment of substrate and the other is for reactive dual magnetron sputtering of SiOx. Before deposition of SiOx film, plasma treatment of the PET substrate was conducted to remove any debris existed on

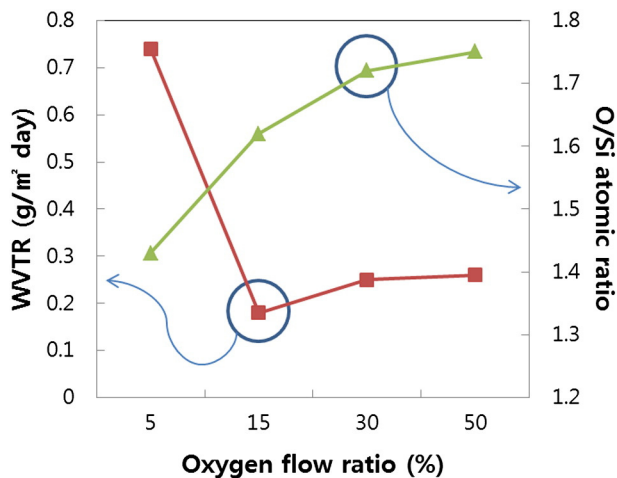


Fig. 2. Change in the WVTR and O/Si atomic ratio of the 100 nm-thick SiOx barrier films deposited on 50 μm -thick PET substrate as a function of O_2 flow ratio.

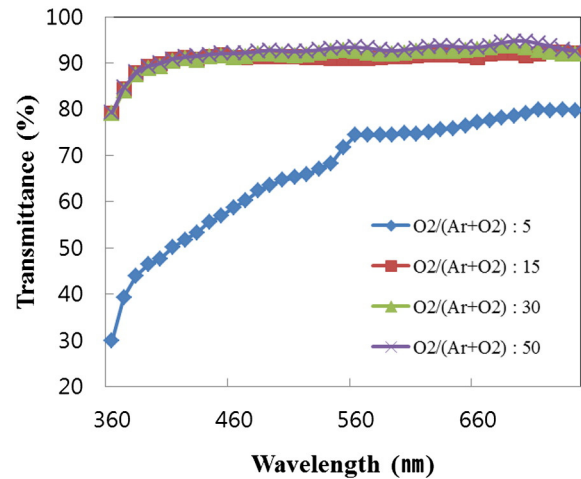


Fig. 3. Optical transmittance of 100 nm-thick SiOx films prepared by reactive sputtering according to O_2 content.

surface and to activate the surface under 0.05 kW of power generated by RF (Optimized condition in our experiment). O_2 gas with 50% Ar content was supplied at a flow rate of 100 standard cubic centimeter per minute (scm) controlled by a mass flow controller (MFC). The rolling speed and the sputtering power of sputtering systems were changed from 0.1 to 2.0 m/min and from 3 kW to 7 kW, respectively to control film thickness. The base pressure and working pressure during plasma treatment were 1×10^{-6} Torr and 3.8×10^{-2} Torr, respectively. After plasma treatment, SiOx films were deposited by the reactive dual magnetron sputtering. The target size per magnetron is $320 \times 124 \text{ mm}^2$. Working pressure and sputtering power were 3.2×10^{-3} Torr and 3.0–7.0 kW, respectively and $\text{O}_2/(\text{Ar} + \text{O}_2)$ gas flow ratio is 5–50%. SiOx films with thickness values of 100, 150, 300, and 500 nm were deposited and thickness was measured using Alpha-Step® (ASIQ3) system after averaging at least five measurements. The root-mean-square surface roughness (R_q) and surface morphologies of the SiOx films were measured by non-contact mode AFM (X-100, Park System). The surface density of the barrier films was examined by RBS with a beam of He^{+2} particles with average energy of 2.0 MeV. The chemical compositions were characterized by XPS (Thermo VG, Scientific, SIGMA PROBE) with in situ Ar sputtering cleaning right before XPS analysis and concentrations of the Si and O were calculated from the area of the Si 2p and O 1s peaks after Shirley background subtraction. The residual film stress was assessed by the beam bending method using a thin film stress measurement instrument (FLX-2320, Tencor), and

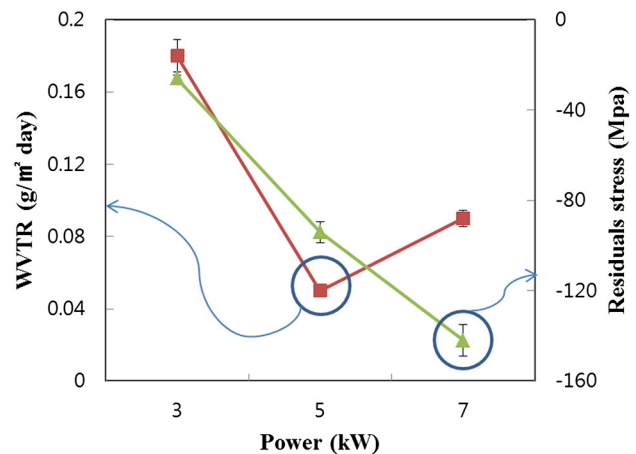


Fig. 4. Change in the WVTR and residual stress of 100 nm-thick SiOx films deposited on 50 μm -thick PET substrate as a function of sputtering power.

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