Surface Control of Planarization Layer on Embossed Glass for Light Extraction in OLEDs

Doo-Hee Cho, Jin-Wook Shin, Jaehyun Moon, Seung Koo Park, Chul Woong Joo, Nam Sung Cho, Jin Woo Huh, Jun-Han Han, Jonghee Lee, Hye Yong Chu, and Jeong-Ik Lee

We developed a highly refractive index planarization layer showing a very smooth surface for organic light-emitting diode (OLED) light extraction, and we successfully prepared a highly efficient white OLED device with an embossed nano-structure and highly refractive index planarization layers. White OLEDs act as an internal out-coupling layer. We used a spin-coating method and two types of TiO_2 solutions for a planarization of the embossed nano-structure on a glass substrate. The first TiO_2 solution was TiO_2 sol, which consists of TiO_2 colloidal particles in an acidic aqueous solution and several organic additives. The second solution was an organic and inorganic hybrid solution of TiO_2. The surface roughness (R_a) and refractive index of the TiO_2 planarization films on a flat glass were 0.4 nm and 2.0 at 550 nm, respectively. The J–V characteristics of the OLED including the embossed nano-structure and the TiO_2 planarization film were almost the same as those of an OLED with a flat glass, and the luminous efficacy of the aforementioned OLED was enhanced by 34% compared to that of an OLED with a flat glass.

Keywords: Light extraction, OLED, out-coupling, refractive index, planarization.

I. Introduction

Since Tang and VanSlyke reported the first practical organic light-emitting diodes (OLEDs) in 1987, this field has attracted significant attention from the scientific and commercial communities [1]. Highly efficient white OLEDs are becoming more important for their potential application to flat panel displays and lighting [2]–[3]. In recent years, light extraction of white OLEDs has attracted great attention as a realization method for OLED panels with high external luminous efficacy. The external quantum efficiency (η_{ext}) of an OLED device is expressed through the internal quantum efficiency (η_{int}) and external coupling efficiency (η_{coup}) by the following relation:

$$\eta_{ext} = \eta_{coup}\,\eta_{int} = \eta_{coup}\,\gamma\,\eta_{exc}\,\varphi_p, \quad (1)$$

where γ is the electron–hole charge balance factor, η_{exc} is the fraction of excitons formed (resulting in radiative decay), and φ_p is the intrinsic quantum efficiency of the radiative decay [4]–[5]. Even when the internal quantum efficiency and cathode reflectance are near 100%, the external quantum efficiency of the bottom emission OLED depends on the external coupling efficiency. The basic device structure of a conventional OLED consists of a flat glass substrate coated with a transparent electrode layer, an OLED-stack, and a metal cathode layer. The refractive indices of the transparent electrode layer and OLED-stack are 1.8 to 1.9, and their thickness is below 300 nm. On the contrary, the refractive index of glass is about 1.5. Therefore, a large part of light emitted from the light-emitting layer in conventional OLEDs is confined to highly refractive index layers, such as OLED-stacks and transparent electrode layers. Such confinement results in low light extraction efficiency of about 20% [6]–[7]. Light extraction is

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Doo-Hee Cho (corresponding author, chodh@etri.re.kr), Jin-Wook Shin (shinjw@etri.re.kr), Jaehyun Moon (jmoon@etri.re.kr), Seung Koo Park (skpark@etri.re.kr), Chul Woong Joo (cwjoo@etri.re.kr), Nam Sung Cho (kevinchons@ etri.re.kr), Jin Woo Huh (jwhuh@etri.re.kr), Jun-Han Han (junhan@etri.re.kr), Jonghee Lee (jonghee.lee@etri.re.kr), Hye Yong Chu (hychu@etri.re.kr), and Jeong-Ik Lee (jiklee@ etri.re.kr) are with the Components & Materials Research Laboratory, ETRI, Daejeon, Rep. of Korea.

indispensable for OLEDs with high luminous efficacy, high brightness, and a long lifetime. Many research groups have proposed various light extraction methods using a surface-modified substrate [8]–[9], a low index grid [10], a high index substrate [11], photonic crystals [12], a micro-lens array [13]–[14], and so on. An embossed structure at the nanometer scale (embossed nano-structure) between a glass substrate and transparent electrode is known to be effective for increasing the out-coupling efficiency. The embossed nano-structure between glass and indium tin oxide (ITO) scatters and disturbs the waveguide mode in ITO and OLED-stack layers, resulting in an increase in out-coupling mode. However, the embossed nano-structure under a transparent electrode may deteriorate the electrical characteristics and reliability owing to its rough surface. A planarization layer on the embossed structure is, therefore, indispensable for an OLED with high efficacy and reliability. High transparency and a high refractive index are generally required for a planarization layer to avoid lowering the luminous efficacy through internal layer absorption and a strong confinement of emission light in the ITO and OLED-stack. For a stable OLED device, a very smooth surface is also needed to deposit the OLED-stack and ITO layer. The surface roughness (R_a) of the planarization layer should, therefore, be minimized. We examined the effects of several planarization layers manufactured by TiO_2 solutions on the electro-optical characteristics of OLEDs using embossed nano-structure glass.

II. Experiment

The nanoscale embossing structure between a glass substrate and transparent electrode is known to be an effective method for enhancing the light extraction efficiency. We developed a glass substrate with an embossed nano-structure using dry etching with an Ag dewetting mask (NS glass). This method is cost effective and scalable. On the glass substrate, a 500 nm-thick SiO_2 layer and a 60 nm-thick Ag film were deposited sequentially. SiO_2 and Ag were deposited by plasma-enhanced chemical vapor deposition and thermal evaporation methods, respectively. To form the Ag dewetting mask, the Ag-coated sample was heated to 400°C. The Ag droplets resulting from the dewetting of Ag thin films were used as a hard mask to dry etch the SiO_2 film on glass. Ag thin films were agglomerated and form nano-sized particles with a random size distribution on the SiO_2 layer/glass substrate through solid or liquid state dewetting. The exposed region between Ag droplets was dry etched using an inductively coupled plasma method to form a random nanoscale scattering layer. After the dry etching process, the Ag mask was removed using diluted HNO_3. The height and diameter of the resultant nano-structure were about 360 nm and 100 nm to 500 nm, respectively. The manufacturing process and surface morphology were well described in previous papers [15]–[16]. The scattering layer consists of irregular nano-sized pillars that can scatter incident light almost without a wavelength dependency [17]. The thermally assisted dewetting process has the advantage of being spontaneous; therefore, it may be cost effective and scalable.

The embossed nano-structure should be coated with a high refractive index planarization layer. In this study, TiO_2 films made from TiO_2 colloidal solutions and TiO_2 hybrid solutions were used as a high refractive index planarization layer. For forming the planarization layer, the TiO_2 solution was dropped onto the embossed glass, spin coated, and then baked in an electric oven at a temperature of 250°C to 400°C. The TiO_2 coating on the embossed glass was carried out after the ultrasonic cleaning of the glass with a NaOH aqueous solution, acetone, isopropyl alcohol, and deionized (DI) water (in sequence).

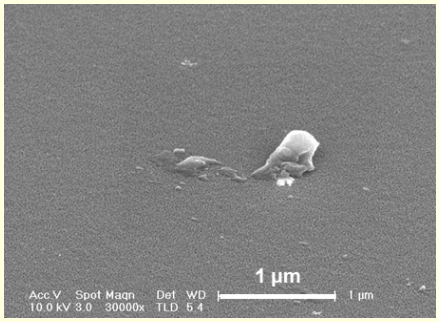
We prepared three types of TiO_2 planarization films. First, a single TiO_2 film was made from type A TiO_2 colloidal solutions prepared through the precipitation of titanium alkoxide and the peptization of its precipitates. The starting solutions were prepared by mixing titanium ethoxide (12.5 ml) and nitric acid (1.0 ml) in distilled water (17 ml) for twenty-four hours, and methanol (17 ml) was then added in the starting sol solutions to make intermediate solutions for the peptization. The pH of the intermediate solution was 1.5 to 1.6 during the peptization. After stirring of the intermediate solutions for twenty-four hours, distilled water of 20 ml was added to the intermediate solutions to make the final type A TiO_2 colloidal solutions. The coated films were baked at 400°C in an electric oven. The second type of TiO_2 film was prepared from type B TiO_2 colloidal solutions, prepared through the addition of polyvinylpyrrolidone (PVP) as a viscosity increasing agent as well as an optimization of the pH and peptization process, to make a TiO_2 colloidal solution composed of very fine TiO_2 nanoparticles. To make fine TiO_2 nanoparticles, excessive methanol was used, and a pH of three to four was maintained during the peptization process. After one hour peptization process, 1 g of PVP was added to the 15 g peptized solution, and the mixed solution was stirred for twenty-four hours at room temperature. The second type of film was also baked at 400°C in an electric oven. A third type of TiO_2 film was prepared using a two-step coating process. The type B TiO_2 solution was coated through a spin-coating method in the first step and baked at 400°C. A type C TiO_2 solution (organic–inorganic hybrid TiO_2 solution) was spin coated in the second step and baked at 250°C. The type C TiO_2 solution was made with a TiO_2 precursor (Ti-butoxide) and organic chelating agent. After triethylene glycol and titanium (IV) butoxide were

dissolved in N, N-dimethyl acetamide (DMAc) and n-butanol under nitrogen, a 37% hydrochloric acid (HCl) solution in water was drop-wise added into the DMAc solution with stirring. The reaction was carried out for one day at room temperature.

The white OLEDs were fabricated on NS glasses covered with TiO_2 planarization layers after ultrasonic cleaning with acetone, isopropyl alcohol, and DI water (in sequence). The ITO layer was deposited onto the TiO_2 planarization layer through RF-DC sputtering and annealed at 200°C in a vacuum oven. The sheet resistance of the 100 nm-thick ITO film was 26 Ω/□. The OLED-stack and cathode were deposited by a thermal evaporation method in a high vacuum chamber below 5×10^{-7} Torr. The OLED grade materials were purchased and used without further purification. The hybrid-type white OLEDs in this study were fabricated using the following configuration: iTO (100 nm)/TCTA (170 nm)/phosphorescent green- and red-emitting layer (5 nm)/inter-layer (3 nm)/ fluorescent blue-emitting layer (10 nm)/BmPyPB (60 nm)/LiF (1 nm)/Al (120 nm). BmPyPB is 1, 3-bis (3, 5-di-pyrid-3-yl-phenyl) benzene. The OLED devices were transferred directly from a vacuum into the inert environment of a glove box, where they were encapsulated using a UV-curable epoxy and a glass cap with a moisture getter. The electroluminescence spectrum was measured using a Minolta CS-1000. The current–voltage (J–V) and luminescence–voltage (L–V) characteristics were measured with a current/voltage source/measure unit (Keithley 238) and a Minolta CS-100, respectively.

III. Results and Discussion

The embossed nano-structure can enhance the luminous efficacy of OLEDs through internal out-coupling. However, its R_a is expected to be replicated in the ITO layer, resulting in a deterioration of electrical characteristics of the OLEDs. An absolutely smooth surface is required to ensure the long-term stability of OLEDs. To obtain electrically stable OLEDs with a high out-coupling efficiency, we planarized the embossed nano-structure on the glass substrates for the OLED device. The planarization layer must have high transparency in the visible range as well as a high refractive index to achieve a high internal out-coupling efficiency. TiO_2 shows a very high transparency, and its refractive index is 2.3 (anatase); therefore, TiO_2 is the most promising material for the planarization of an embossed nano-structure. The wet-coating process accompanied with the sol-gel technique is simple and proper for the planarization. We examined the three types of TiO_2 film prepared through a spin-coating method for the planarization layer on the embossed nano-structure for the internal light extraction of OLEDs in this study.



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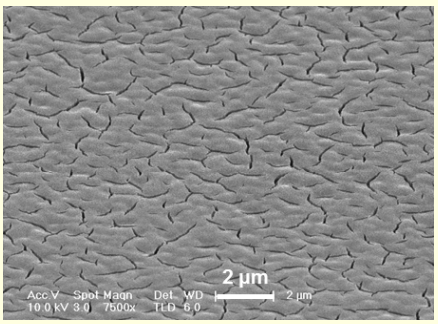
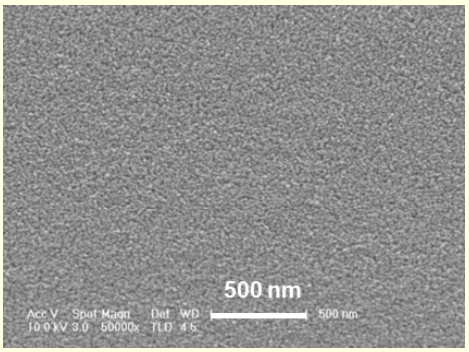


(b)

Fig. 1. SEM images of (a) Ti-A film prepared on a glass substrate and (b) Ti-A film on the NS glass.

Figure 1(a) shows the surface morphology of TiO_2 film (Ti-A film) prepared using a type A solution without filtering. The filtering of a type A solution, even with a 1 μm syringe filter, was impossible because of the large TiO_2 particles in the solution. The peptization process for the type A solution was not considered to be optimized; as a result, large agglomerates remained in the solution. Large particles were found in some places on the surface of the Ti-A film, as shown in Fig. 1(a). The large particles were considered to be originated from the inadequate peptization of the solution.

The film thickness was about 150 nm, and the R_a of the film on a flat glass was 16.0 nm. The R_a was measured using atomic force microscopy (AFM). The film thickness was insufficient for planarization of the embossed nano-structure, and the R_a was relatively poor. The R_a of the ITO layer on the Ti-A film was 18.7 nm. It was even worse than that of the Ti-A film itself. We tried to fabricate white OLED devices on Ti-A films coated on a flat soda-lime glass and NS glass. In the case of Ti-A/flat glass, the OLED device showed many dark spots at the beginning and then turned off; thus, we could not measure electro-optical characteristics. Although the R_a of the Ti-A film was not severely poor, the large particles due to the inadequate peptization caused the unstable OLEDs. In the case of the Ti-A/NS glass, the OLED device did not turn on owing to an internal short circuit of the OLED-stack. The Ti-A film did not



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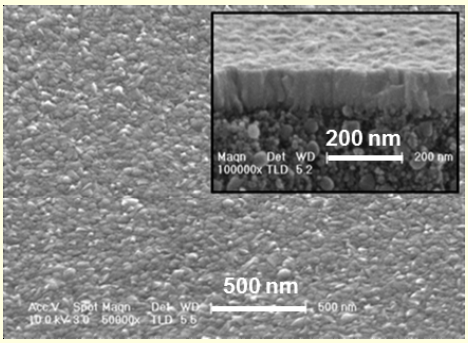


(b)

Fig. 2. SEM images of (a) Ti-B film prepared using a type B solution on a glass substrate and (b) ITO film on the Ti-B film. The SEM image in the corner box of (b) shows a cross section of the ITO film on the Ti-B film.

planarize the nano-structure of 360 nm sufficiently, as shown in Fig. 1(b); thus, small cracks, bumps, and spikes on the rough surface were considered to induce the internal short circuit.

Figure 2 shows the surface morphology of TiO_2 film (Ti-B film) prepared using a type B solution and ITO/Ti-B film on a flat glass. The Ti-B film thickness was about 400 nm, and the R_a of the film on a flat glass was 2.4 nm. The type B solution was spin coated after filtering using a 0.45 μm syringe filter. The fine R_a should be caused by fine TiO_2 particles in the type B solution owing to the optimization of the peptization process. In addition, increasing the viscosity of the coating solution by PVP is considered to increase the thickness of the final Ti-B film. The R_a of the ITO layer on the Ti-B film was 4.5 nm. The R_a worsened compared to that of the Ti-B film, as in the case of the ITO/Ti-A film. The anisotropic grain growth of ITO on the Ti-B film can be seen in Fig. 2(b). The poly-crystalline under layer can provide plenty of nucleus sites where the anisotropic grain growth is initiated. It is considered that the Ti-A and Ti-B films act as a seeding layer for the anisotropic growth of ITO; thus, this induces a poor R_a of an ITO film. The R_a of Ti-B film on the NS glass and ITO/Ti-B film on the NS glass were 7.5 nm and 8.6 nm, respectively. The sheet resistance of ITO/Ti-B films on a glass and the NS glass were

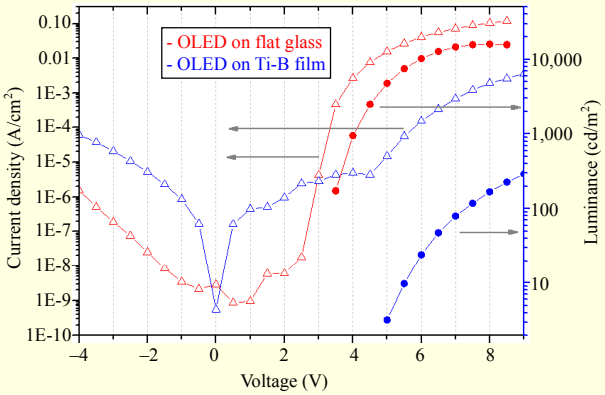
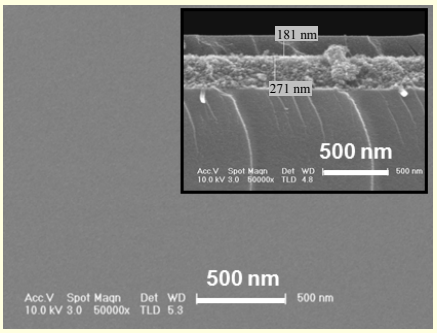


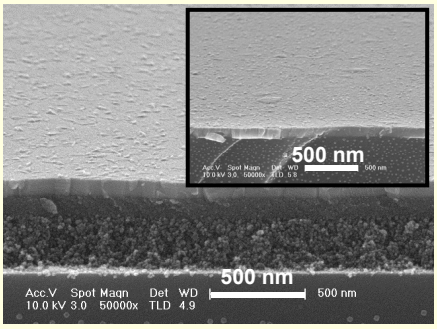
Fig. 3. J–V–L characteristics of OLED devices prepared on a flat glass and Ti-B film/NS glass.

38 Ω/□ and 53 Ω/□, respectively. That of ITO/Ti-B film/NS glass was nearly double that of ITO film on a flat glass. The OLED devices fabricated on the Ti-B film coated on NS glass were operational. Figure 3 shows the J–V–L characteristics of white OLEDs prepared on a flat glass and Ti-B film/NS glass. The current density and luminance over 5 V were significantly low compared to those of the reference OLED. The R_a of Ti-B film was smaller than that of the Ti-A film and is not a significant problem. However, the sheet resistance of ITO/Ti-B film was higher than that of ITO/flat glass. In addition, the work function can be changed by surface morphology [18], crystallographic orientation of surface [19], and surface treatment [20] because it is a surface-related characteristic. Grain boundaries and the anisotropic grain growth of ITO film can change the hole injection properties. The work functions of ITO/flat glass was 5.55 eV and those of ITO/Ti-A and Ti-B films were 5.65 eV, while the highest occupied molecular orbital (HOMO) level of the TAPC of the hole transporting layer was known to be 5.5 eV. The work functions were measured by a photoelectron spectroscopy method. The actual work function of ITO could not be measured because the ITO was cleaned by plasma before OLED-stack deposition; however, the hole injection barrier was not considered to be changed significantly in the case of ITO/Ti-B film. Thus, the high resistance is considered to be one of the origins for the low current density of the OLED with Ti-B film/NS glass, while surface contamination and others may affect the J–V characteristics. Further studies are required to understand the reasons for lowering the current density sufficiently.

Figure 4 shows the surface morphology of a TiO_2 film (Ti-C film) prepared using the two-step coating process with type B and type C solutions as well as a 45° image of ITO film on Ti-C film. The Ti-C film thickness was about 450 nm, and the R_a of the film on a flat glass was 0.4 nm. It can be seen in Fig. 4(a) that the surface and cross section of the upper layer of



(a)



(b)

Fig. 4. SEM images of (a) surface of Ti-C film prepared using two-step coating process and (b) a 45° image of ITO film on Ti-C film. The SEM image in the corner box of (a) is a cross section of the Ti-C film, and that in (b) is a 45° image of the ITO film on a flat glass.

the Ti-C film showed a very smooth and glass-like morphology [21]. The surface morphology of the ITO/Ti-C film was almost the same as that of the ITO on flat soda-lime glass, as shown in Fig. 4(b). The work function of ITO/Ti-C film, measured by a photoelectron spectroscopy method, was also 5.55 eV, which is the same as for the ITO/flat glass. The Ti-C film is certain to provide almost the same surface quality as an amorphous glass surface. It is considered that the amorphous surface of the upper layer of the Ti-C film suppressed the anisotropic grain growth of the ITO film.

Figure 5 shows a 514 nm excited Raman spectra of the Ti-B film and the upper layer of the Ti-C film on a flat soda-lime glass. As shown in Fig. 5, the Raman spectrum of the Ti-B film showed several peaks between 100 cm^{-1} and 1000 cm^{-1}; however, that of the Ti-C film did not show any peak, but rather a smooth curve. The peaks at 146 cm^{-1}, 400 cm^{-1}, 519 cm^{-1}, and 639 cm^{-1} were assigned to the anatase phase of the TiO_2 crystal [22]. Owing to the anatase peaks in the Raman spectra [23], the Ti-B film is considered to consist of TiO_2 crystalline particles and the Ti-C film to consist of an amorphous phase. Therefore, the Ti-B film provided a relatively rough poly-crystalline surface, and the Ti-C film provided a smooth amorphous surface for the ITO deposition.

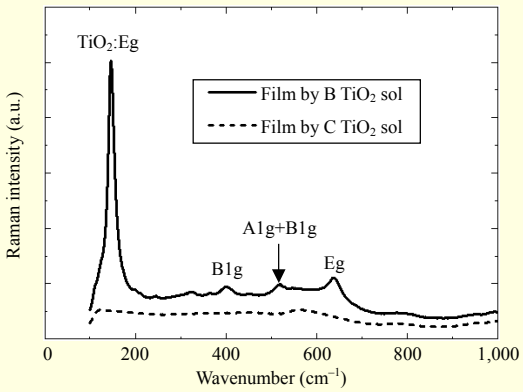


Fig. 5. Raman spectra of the thin films made using type B and type C TiO_2 solutions.

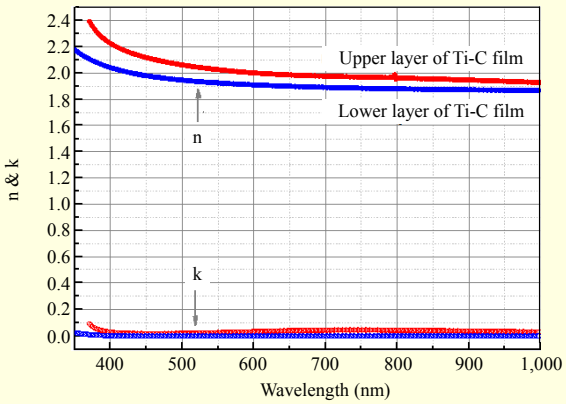


Fig. 6. Refractive indices (n) and extinction coefficients (k) of the upper and lower layers of the Ti-C film measured using an ellipsometer.

This is also considered to be the origin of the difference between the ITO surfaces on the Ti-B film and Ti-C film.

Figures 6 and 7 show the refractive index and transmittance curves of the Ti-C film, respectively. The refractive indices of the lower and upper layers of the Ti-C film were about 2.0 and 1.9 at 550 nm, respectively and decreased slightly with an increase in wavelength. The refractive index of the ITO used in the OLED device (described in this study) was 1.98 at 550 nm; thus, the Ti-C film had a sufficiently high refractive index for the internal out-coupling. The transmittance of the Ti-C film was higher than 95% in the whole visible light range. Consequently, the Ti-C film showed an excellent performance for the planarization layer of the internal out-coupling.

Figure 8 shows an SEM image of the cross section of the Ti-C film used in the planarization layer on the NS glass and an AFM image of its surface. The Ti-C film covered the embossed nano-structure and planarized its surface well. When we coated the single upper layer of the Ti-C film on the NS glass, the planarization layer showed a lot of cracks and bumps

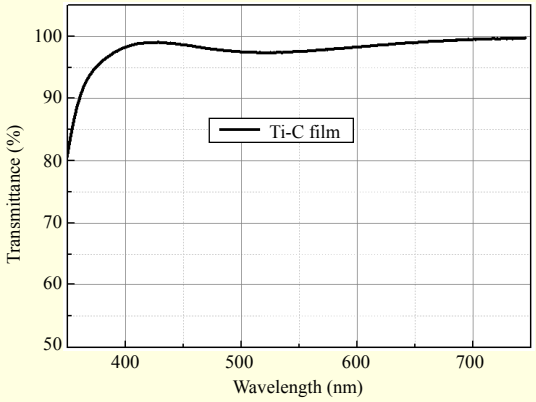
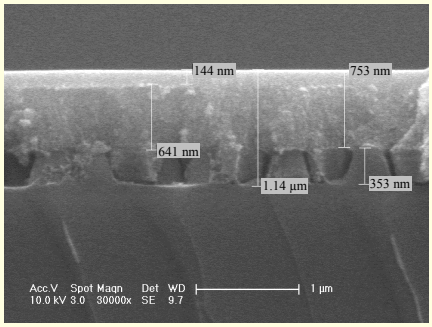


Fig. 7. Transmission spectrum of Ti-C film on a glass substrate.



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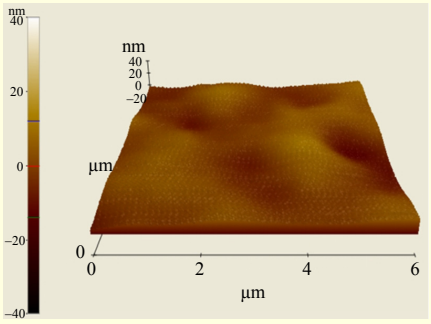

(b)

Fig. 8. (a) SEM image of the cross section and (b) AFM image of the surface of the Ti-C film on the NS glass substrate.

owing to the thinness of the upper layer. The R_a of the Ti-C film on the NS glass was 2.1 nm in spite of the 360 nm highly embossed nano-structure. The sheet resistance of ITO/Ti-C films on a glass and the NS glass was 26 Ω/□ to 27 Ω/□. It was almost the same as that of the ITO on flat glass, and the work function was also the same as that of the aforementioned ITO on flat glass. Accordingly, the J–V characteristic of a white OLED device prepared on the Ti-C film/NS glass was as stable as that on the flat soda-lime glass, as shown in Fig. 9. The leakage current level of the OLED with the Ti-C film/NS glass

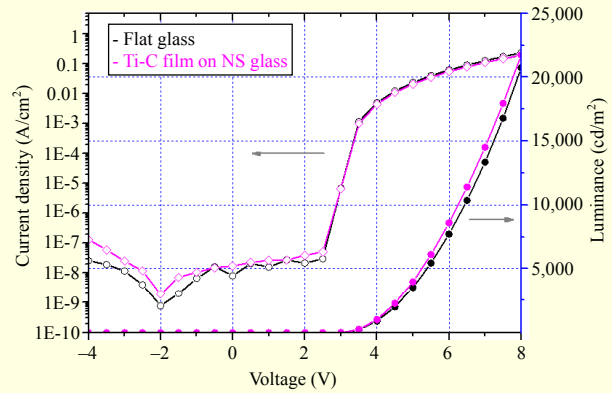

Fig. 9. J–V–L characteristics of OLED devices prepared on a flat glass and Ti-C film/NS glass.

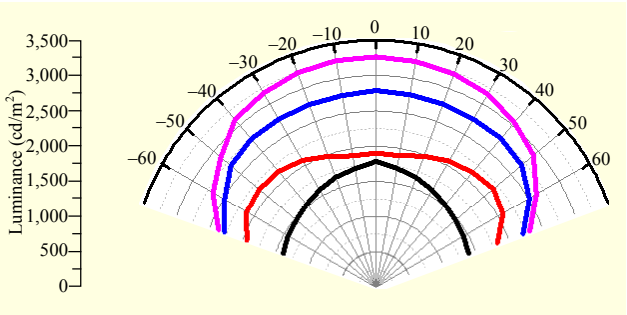

Fig. 10. Angular distributions of luminance of the white OLEDs prepared on a flat glass (black), flat glass with MLA (blue), Ti-C film/NS glass (red), and Ti-C film/NS glass with MLA (violet).

was almost the same as that of the flat glass. The luminance values, measured at the surface normal of the emitting surface, of the OLEDs fabricated on the Ti-C film/NS glass were higher than that on the flat glass.

Figure 10 shows the angular distributions of luminance of the white OLEDs prepared on a flat glass and Ti-C film/NS glass. All luminance measurements were carried out at a 20 mA input current. It can be readily seen that the luminous intensities for all directions of the OLED with Ti-C film/NS glass were remarkably enhanced compared to those of the flat glass. The luminance distribution of the OLED on Ti-C film/NS glass was different from the flat glass sample. It is considered that the extension of cavity length due to the Ti-C film, as well as the scattering effect by the nano-structure, changed the luminance distribution. External quantum efficiencies (EQE), calculated from the data in Fig. 10, of the white OLEDs prepared on a flat glass and Ti-C film/NS glass were 8.2% and 10.7%, respectively. Their luminous efficacies were 8.4 lm/W and 11.3 lm/W, respectively. The EQE and luminous efficacy were enhanced by 29% and 34%, respectively. The EQE and luminous efficacy were further

improved by the application of a microlens array (MLA) film on the light output surface of the OLEDs. Those of the Ti-C film/NS glass with MLA were 14.8% and 15.8 lm/W, respectively.

The optical and surface characteristics of the planarization layer covering the embossed nano-structure strongly influence the electrical characteristics and luminous efficacy of the OLEDs. In particular, the R_a of the planarization layer should be controlled to be extremely smooth so as to lower the leakage current and stabilize the electrical and optical characteristics of the OLEDs. In addition to the roughness, the degree of crystallinity of the planarization layer is considered to significantly affect the morphology of the ITO film and the stability of the OLEDs. The surface of the planarization layer needs to be amorphous to prevent anisotropic grain growth of the ITO, which has a negative effect on the electrical stability of the OLEDs. TiO_2 film is a promising planarization layer in OLEDs, including the embossed nano-structure for the internal out-coupling, because of its high transparency and high refractive index. The TiO_2 films prepared through the sol-gel technique result in rough and polycrystalline films, and they induce an anisotropic growth of the ITO. To achieve an extremely smooth surface and suppress the anisotropic growth of the ITO, the composition of the TiO_2 solution as well as the baking process must be modified to generate amorphous films. The TiO_2 film, using a simple sol-gel technique, was not sufficient for the planarization layer in the OLEDs owing to its rough surface and high crystallinity. The poly-crystalline TiO_2 planarization layer, using a sol-gel technique, was considered to worsen the R_a of the ITO film owing to the anisotropic growth. However, the two-step coating process with a fine TiO_2 colloidal solution and organic–inorganic hybrid TiO_2 solution can make a glass-like surface. The ITO films on the double-layer TiO_2 film showed almost the same morphology as that on the flat glass. The R_a and the amorphous surface of the highly refractive index planarization layer are very important for electrically stable OLEDs, with significant improvement of light extraction through an internal out-coupling structure.

IV. Conclusion

In this work, we studied a solution preparation method and coating process for a highly refractive index planarization layer for electrically stable and highly efficient OLED devices. We manufactured highly efficient OLED devices using an embossed nano-structure and a TiO_2 planarization film. The external quantum efficiency of a white OLED device including the embossed nano-structure and TiO_2 planarization film was increased by 29%, and its leakage current level was almost identical to that of a flat glass substrate. The embossed nano-structure and the TiO_2 planarization film were significantly effective for enhancing the out-coupling efficacy without deteriorating the electrical stability. An extremely smooth and amorphous surface, as well as a high transparency and high refractive index of the planarization layer, is very important for enhancing the out-coupling efficacy and stabilizing the electrical characteristics of OLED devices with an embossed nano-structure as an internal out-coupling structure.

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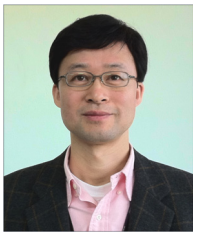

Doo-Hee Cho received his PhD degree in materials chemistry from Kyoto University, Kyoto, Japan, in 1996. He worked in the areas of float glass and low-e coating at KCC Corp., Seoul, Rep. of Korea, from 1996 to 1998. Since he joined the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 1998, he has been involved in specialty optical fiber, transparent oxide TFT, and OLED lighting research. His major research interests include optical structure and light extraction of OLEDs. He has been working on the international standardization for OLED lighting, as a member of IEC TC34, since 2009.

Jin-Wook Shin received his BS degree in electronic engineering from Myongji University, Yongin, Rep. of Korea, in 2007 and his MS degree in electronic materials engineering from Kwangwoon University, Seoul, Rep. of Korea, in 2009. He joined the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 2009. His current research interests include light extraction technologies for organic light-emitting devices.

Jaehyun Moon received his BS degree in metal engineering from Korea University, Seoul, Rep. of Korea, in 1995 and his PhD in materials science and engineering from Carnegie Mellon University, Pittsburgh, PA, USA, in 2003. From 2003 to 2004, he was a postdoctoral associate at the Max-Planck Institute, Stuttgart, Germany. He joined the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 2004. His current research interests include flexible electronics, functional nanomaterials, and organic light-emitting diodes.

Seung Koo Park received his BS degree in textile engineering and his MS and PhD degrees in textile engineering from Seoul National University, Seoul, Rep. of Korea, in 1986, 1993 and 1997, respectively. Since July 2000, he has worked for the Information & Communications Core Technology Research Laboratory of the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea. He has been interested in control of polymer film properties by designing chemical structures. He is studying new UV cross-linkable prepolymer systems for active and passive optical devices.

Chul Woong Joo received his BS and MS degrees in polymer science and engineering of organic electronics devices from Dankook University, Cheonan, Rep. of Korea, in 2008 and 2010, respectively. He joined the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 2011, and his current research interests include device architectures in organic light-emitting devices and next-generation displays.

Nam Sung Cho received his BS degree from Chung-Ang University, Seoul, Rep. of Korea, in 2000 and his MS and PhD degrees in chemistry from the Korea Advanced Institute of Science and Technology, Daejeon, Rep. of Korea, in 2002 and 2006, respectively. From 2006 to 2008, he was a postdoctoral associate at the University of California Santa Barbara, Santa Barbara, CA, USA. He worked in material development for OLEDs in LG Display R&D Center, Paju, Rep. of Korea, from 2008 to 2012. He joined the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 2012. His current research interests include OLED structures, OLED materials, and white OLEDs.

Jin Woo Huh worked for the Institute of Advanced Engineering and the Orion Electric Co., Ltd. after receiving her MS degree in physics from Ehwa Womans University, Seoul, Rep. of Korea, in 1995. Then she earned her PhD degree in electrical engineering from Korea University, Seoul, Rep. of Korea, in 2010. Thereafter, she joined the OLED lighting team at the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea. Her current research interests include transparent OLEDs and light extraction techniques.

Jun-Han Han received his BS and MS degrees in electronic engineering from Hanyang University, Seoul, Rep. of Korea, in 2007 and 2009, respectively. He joined the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 2009 and has been continuing his research on organic light-emitting diode lighting applications and next-generation displays.

Jonghee Lee received his BS, MS, and PhD degrees in chemistry from the Korea Advanced Institute of Science and Technology, Daejeon, Rep. of Korea, in 2002, 2004, and 2007, respectively. He joined the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 2007 and worked on white organic light-emitting diodes for display and lighting applications. Then he moved to the Institut für Angewandte Photophysik (IAPP, Prof. Karl Leo's group) at the Technische Universität, Dresden, Germany, as a post-doc in 2010. After two years, he joined ETRI again and has worked on new concept display modes as well as light-extraction techniques for OLEDs.

Hye Yong Chu received her BS and MS degrees in physics from Kyung Hee University, Seoul, Rep. of Korea, in 1987 and 1989, respectively. She joined the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 1989. She earned her PhD degree in information display from Kyung-Hee University, in 2008. Her current research interests include novel device architectures in organic light-emitting devices and next-generation displays.

Jeong-Ik Lee received his BS, MS, and PhD degrees in chemistry from the Korea Advanced Institute of Science and Technology, Daejeon, Rep. of Korea, in 1992, 1994, and 1997, respectively. After graduating, he joined the IBM Almaden Research Center, San Jose, CA, USA, as a post-doc, where he worked on organic light-emitting materials. He moved to the Electronics and Telecommunications Research Institute, Daejeon, Rep. of Korea, in 1999 and has been continuing his research on organic light-emitting materials and devices.