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ITO/SiO₂/ITO Structure on a Sapphire Substrate Using the Oxidation of Ultra-Thin Si Films as an Insulating Layer for One-Glass-Solution Capacitive Touch-Screen Panels

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Abstract: The SiO₂ generated by low-temperature oxidation of ultra-thin metallic silicon (thickness = 50 nm) film was evaluated for implementation in one-glass-solution capacitive touch-screen panels (OGS-TSPs) on sapphire-based substrates. Our results show that the silicon films oxidized at 823 K exhibited the highest visible transmittance about 91% at 550 nm, compared to ~72% transmittance of the as-deposited silicon films which were deposited at room temperature. Additionally, the annealed films exhibited a more uniform, dense, and smooth surface microstructure than that of the as-deposited Si films. X-ray photoelectron spectroscopy (XPS) results revealed that the low-temperature oxidation of Si films at 823 K yielded SiO₂. Furthermore, when the insulating SiO₂ film obtained by low-temperature oxidation was sandwiched between two indium tin oxide (ITO) layers (ITO/SiO₂/ITO) on a sapphire substrate, the SiO₂ film resulted in the dielectric strength of approximately 3 MV/cm. In addition, the highest optical transmittance obtained by the ITO/SiO₂/ITO films is about 88.3%. The change in capacitance of the ITO/SiO₂/ITO structure was approximately 3.2 pF, which indicates the possibility of implementation in capacitive touch-screen panel devices.

Keywords: Thin film: sputtering; ITO; SiO₂; low-temperature oxidation; touch screen panel; sapphire; transmittance

1. Introduction

The high demand for touch-screen panels (TSPs) for implementation in various mobile communication and information devices such as cellular phones, navigation systems, and flat-panel devices continues to increase. Various techniques that employ resistive, capacitive or infrared sensors are implemented in TSP designs. Among these techniques, the capacitive touch-sensitive method is most widely used. In this method, the touch activity is identified by detecting minor changes in the electrical charge generated via human contact with a finger or object. Moreover, because capacitive TSPs enable multi-touch function and multi-tasking, they have become a replacement for conventional



resistive-type TSPs. Sapphire-based display technology is currently garnering a considerable amount of attention as a means to enhance high-end smartphone devices. The advantages of a sapphire-based display over the conventional glass-based display are good scratch resistance and sensitivity [1]. Despite these advantages, sapphire transparency remains to be a concern, although the transmittance can be optimized in the ultraviolet, infrared, and visible wavelengths by applying a suitable optical coating [2–4]. Touch-type display devices have varying types of multilayer stacks that comprise a complex series of layers of glass (or sapphire), optically clear adhesive (OCA), and indium tin oxide/polyethylene terephthalate (ITO/PET). The deposition of ITO on the PET layer has been a widely employed technique used to fabricate a transparent conducting layer in TSPs [5–8]. Although PET has been confirmed to be effective as an insulating layer, it has been reported to be a cause of the poor crystallinity of the deposited ITO, increased process complexity, and high cost. The low thermal stability of PET limits the heat-treatment temperature of ITO, which thus leads to poor transmittance. Accordingly, the direct deposition of SiO_2 on the ITO layer can be employed as an alternative to replacing the PET-based structure, as SiO₂ has a relatively high dielectric strength (maximum 10 MV/cm) as compared to PET (150~200 kV/cm). In general, SiO₂ films have been extensively employed in various technological domains [9,10], which can be formed via either physical/chemical deposition or Si wafer oxidation. The oxidation process possesses the advantage of contamination prevention, and the disadvantage of an elevated temperature process [11,12]. However, since the impregnation of nanovoids in the sputtered SiO_2 thin films is inevitable, the densification of the SiO_2 film is required to improve the dielectric strength. Silicon films can undergo a positive volume change (volume expansion) during oxidation; this phenomenon is expected to contribute to the densification of SiO₂. It is known that the thermal oxidation of bulk silicon such as silicon wafer has been well established for the formation of SiO_2 at temperatures between 1073 K and 1473 K in an oxygen ambient. However, such an oxidation temperature range cannot be suitable in this study because high-temperature annealing beyond 823 K can deteriorate the ITO properties because excessive heat affects the structure by creating voids between the grains [13]. Several researchers reported that ITO films exhibited lower resistivity and high visible transmittance (>80% at 550 nm) after annealing the films about 673 K-823 K [14,15]. Therefore, low-temperature (< 873 K) oxidation of Si can be appropriate to form the SiO₂. In this paper, we report on low-temperature oxidation of Si film and investigation of its structure and microstructural evolution, and the fabrication of ITO/SiO₂/ITO thin-film structure on a sapphire substrate and discuss its potential implementation in one-glass-solution capacitive touch-screen panels (OGS-TSPs).

2. Materials and Methods

The silicon thin films were deposited by radio-frequency (RF) magnetron sputtering on sapphire and ITO/sapphire substrates by using a high-purity (99.999%) silicon target of 2 in. diameter, the sputtering power was fixed at 150 W. A high-purity ITO target (2-in. diameter; 99.99% purity) containing 10 wt.% SnO₂ was used for the deposition of ITO films with a target power 150 W. Prior to sputter deposition, the vacuum chamber was evacuated to a base pressure below 2.5×10^{-6} Torr. An ultra-pure Argon (Ar) gas with a flow rate of 110 and 90 sccm was introduced through a mass-flow controller as a sputtering gas for the deposition of ITO and Si films, respectively. The distance between the target to the substrate was maintained constant at 10 cm, and both the films ITO and Si were deposited at room temperature. The sputter-deposited Si films at room temperature were subsequently oxidized at various temperatures between 723 and 823 K for four hours in ambient air via rapid thermal annealing (RTA) process and we have investigated the structural, optical, and dielectric properties of SiO₂ films prepared via low-temperature oxidation of Si. After the oxidation of Si/ITO films, a top layer ITO was deposited and further annealed for 1 h to improve the ITO structure. Thus, for the high-quality touch-screen display panel, 50-nm-thick ITO and 70-nm-thick SiO₂ films were formed on a sapphire (ITO/SiO₂/ITO) substrate by using RF magnetron sputtering. The surface microstructure of the Si and SiO₂ were observed via field-emission scanning electron microscopy (FE-SEM, TESCAN) MIRAH) (TESCAN MIRA3, Seoul, Korea) and atomic force microscopy (AFM, Park systems, XE-70)

(Park Systems, Suwon, Korea). A cross-section of the oxidized Si thin film was observed by performing high-resolution transmission electron microscopy (HR-TEM, Tecnai G2 F20) (Thermo Fisher, Hillsboro, OR, USA). The TEM analysis sample was prepared by using a focused ion beam (FIB), a very thin Pt layer was coated over the SiO₂ prior to FIB milling because the SiO₂ is an insulator. The transmittance spectra were recorded by using a ultraviolet-visible-near infrared (UV-vis-NIR) spectrophotometer (UV-3600, Shimadzu, Kyoto, Japan) Additionally, a chemical analysis of the surface of each film was enabled by X-ray photoelectron spectroscopy (XPS). Lastly, the capacitive characteristics of the TSP were evaluated via the cylindrical rod touch response by employing the conventional direction method for an LC resonance circuit (TMS 1000, FTLAB Inc., Seoul, Korea).

3. Results and Discussion

Figure 1 shows the X-ray diffraction pattern (XRD) of the as-deposited and oxidized Si/sapphire films. As can be seen, a very high-intensity peak was observed at $2\theta = 41.6^{\circ}$ corresponding to the sapphire (006) substrate. The Si-related diffraction peaks were not observed in the as-deposited films, which indicate the amorphous nature of the thin films. However, a small diffraction peak appeared around $2\theta = 20.3^{\circ}$ in the annealed films at 823 K attributed to the polycrystalline SiO₂ phase. The diffraction peak was enlarged as shown in the inset of Figure 1a in the 2θ range of 20 to 23° . It clearly suggests that the amorphous Si films were transformed into the polycrystalline SiO₂ at low-temperature (823 K). In contrast, several studies were reported that certain thermally grown SiO2 films consist of crystalline domains [16,17], so the polycrystalline SiO₂ phase can be plausibly expected along with the amorphous phase in the annealed films. Nagta et al. reported that the thermally grown crystalline SiO₂ by means of thermal oxidation possess high density which is about 4.4 g/cm³, while the amorphous phase density was about 2.2 g/cm³ [18]. The cross-sectional TEM image of the Si/sapphire film oxidized at 823 K is presented in Figure 1b. The interface between the SiO₂/sapphire was observed to be flat and devoid of any reactions or cavities at the interface, which is an indication of full oxidation and highly dense structure formation via low-temperature oxidation of Si film i.e. SiO₂ formation without any structural distortions induced by substantial volume expansion of Si during the oxidation. Such a flat SiO_2 /sapphire interface also represents that the homogeneous oxidation process. In the case of the inhomogeneous oxidation process, the interface roughness could be generally high that attributes the interface distortion energy [19,20].



Figure 1. (a) XRD patterns of the as-deposited and oxidized Si/sapphire films; (b) cross-sectional TEM image of the Si/sapphire film oxidized at 823 K.

Oxidation of the Si film was expected to reflect the optical transmittance change. As is shown in Figure 2, the sapphire substrate exhibits an optical transmittance of about 86% and the as-deposited Si film exhibit approximately 72% at the wavelength of 550 nm. However, the transmittance improved as the oxidation temperature was increased. The Si/sapphire film annealed at 823 K exhibited the

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highest transmittance of approximately 91%. It should be noted that, as the annealing temperature increased, the transmittance approached the transmittance of SiO_2 ; this phenomenon is believed to be due to changes in the optical constants in SiO_x films. Due to the lower refractive index ~1.45 of SiO₂ film, higher transmittance is generally expected for the SiO₂/sapphire films. In the case of the films oxidized at low-temperatures, there may be lower-valence silicon species that facilitate oxidation of the film. More specifically, oxygen atoms entering into the film increases the thickness of the film, thereby inducing volume expansion and decreasing the refractive index. Lai et al. reported that the transmittance of the films could be described by a single-layer model [21,22], suggesting that the films are homogeneous and that all structural and compositional changes uniformly occur throughout the SiO_x films under the condition of low-temperature oxidation. In addition, the uniformly structured film can effectively reduce light scattering, and thus enhance transmittance [23]. We also performed simulations to compare the transmittance of the oxidized films by implementing the optical constants of well-defined SiO₂ and physical thickness values as inputs into the Essential Macleod optical coating design program [24]. The simulated and experimentally measured transmittance results were found to be in good agreement, indicating that SiO₂ phase formation occurred in the absence of the retained Si phase, and that increased surface roughness significantly increased the amount of light that was scattered.



Figure 2. Optical transmittance spectra of the as-deposited and oxidized Si/sapphire films.

As is shown in Figure 3a,b, the as-deposited Si film had a defective surface morphology (as denoted by the arrows and the dotted circle), whereas the oxidized film at 823 K shows a smooth and uniform surface microstructure comprised of larger grains. AFM was used to observe the microstructural changes; the resulting images are presented in Figure 4a,b. The root-mean-square (R_{rms}) and peak-to-valley (R_{pv}) roughness values for the as-deposited and oxidized Si films were determined to be 7.74 and 48.67 nm, and 5.87 and 41.2 nm, respectively. As can be seen in Figure 4, the as-deposited films possess a rough surface and localized voids (i.e. nano-sized cavities) indicated with the arrow-lines, which are inevitable during the Si deposition process. In contrast, the oxidized films are shown to have a compact and dense microstructure that is generally comprised of overlapped grains and has no significant trace of voids. It has been reported that SiO₂ film growth is dependent on Si from the SiO₂/Si interface [25], and the oxidation of amorphous Si film is hindered by the reduced transport of oxygen into the amorphous Si film relative to that occurring in crystalline silicon [26]. Furthermore, assuming that the crystalline Si was oxidized, the molar volume of SiO₂ has been reported to be approximately 2.27 times higher than that of Si crystal [27]; such an increase in volume is believed to overflow into voids or cavities, leading to the formation of a dense SiO₂ film (i.e. densification of the film).



Figure 3. Field-emission scanning electron microscopy (FE-SEM) images of Si/sapphire: (**a**) as-deposited and (**b**) oxidized at 823 K.



Figure 4. Atomic force microscopy (AFM) images (scan size: $3 \times 3 \mu m$) showing the microstructural surface differences between the (**a**) as-deposited and (**b**) oxidized Si/sapphire films at 823 K.

Figure 5 shows the XPS spectra of the surface of the as-deposited and oxidized (at 823 K) Si/ITO/sapphire thin films; the corresponding Si, O, and C peaks have been identified and accordingly labeled. The Si 2p spectra, in addition to the corresponding deconvoluted peaks, are shown in Figure 5b; the peaks in the as-deposited films at the binding energy 100.92 and 103.74 eV are associated with Si^0 of Si and Si⁴⁺ of Si-O, respectively [28]. For the oxidized films, the Si 2p peaks were slightly shifted to the higher binding energies of 104.4, 105.1, and 106.02, which are attributed to the three oxidation states Si^{2+} , Si^{3+} , and Si^{4+} of SiO_2 and SiO_x (x < 2), respectively. In Figure 5c, it is clear that the O 1s deconvoluted peaks at 532.2, 531.6, 533.3, and 532.8 eV are related to the strong Si-O bond in SiO₂; this finding is consistent with the relevant literature citing that the binding energies range from 532.2 to 533.1 eV [29]. It is known that SiO_2 is characterized by higher Si 2p binding energy; it is for this reason that the Si 2p binding energy is shown to increase with increased oxygen content [30,31]. It can also be seen in Figure 5b that the binding energy difference (i.e., chemical shift) in Si 2p between the as-deposited and oxidized films was approximately 3.5 eV; this indicates that the unstable Si-O bonds in the as-deposited films tended to form more stable stoichiometric SiO₂ during the annealing process. Consequently, in the case of the oxidized SiO_2 film, the shift of the Si 2p peak to the higher binding energy is indicative of the strong Si-O bond formation in the form of SiO₂.



Figure 5. (a) X-ray photoelectron spectroscopy (XPS) survey spectra, (b) Si 2p spectra, and (c) O 1s spectra of the as-deposited and oxidized Si/indium tin oxide (ITO)/sapphire thin films.

Optical transmittance spectra of both two-layer and three-layer structures i.e. Si/ITO/sapphire and ITO/SiO₂/ITO/sapphire were shown in Figure 6. The as-deposited Si/ITO films showed transmittance of 71.2% at 550 nm (Figure 6a). The films were oxidized at low temperatures (723–823 K) to form the SiO₂. After the oxidation, the transmittance of the films (SiO₂/ITO) was improved to 84.6%. To obtain the ITO/SiO₂/ITO structure, ITO layer was deposited over the Si/ITO films which were oxidized at 723, 773, and 823 K. Thus, ITO/SiO₂/ITO structure was obtained, and it was further annealed by RTA method in air ambient for one hour to improve the top layer ITO crystallinity (Figure 6b). Finally, the ITO/SiO₂/ITO films annealed at 823 K exhibited the highest transmittance of 88.3%, which was higher compared to the sapphire substrate (without films). Enhancement in visible light transmittance of the annealed ITO/SiO₂/ITO films can be attributed to the ITO film (top layer) structure. Hu et al. reported that the crystalline ITO films obtained by post-annealing of amorphous films exhibited much higher visible transmittance than amorphous films [32]. As a result, the post-annealing treatment of ITO/SiO₂/ITO films was effective to enhance the transmittance in the visible region.



Figure 6. Optical transmittance spectra: (**a**) as-deposited and oxidized Si/ITO/sapphire, (**b**) ITO/SiO₂/ITO/sapphire films which are annealed for 1 h after the ITO top layer deposition on SiO₂/ITO/sapphire.

Finally, a capacitive touch-screen panel was fabricated with the films exhibited the highest transmittance (i.e., ITO/SiO₂/ITO structure annealed at 823 K) by using conducting pads with an individual pattern size of 1×1 mm to pattern the ITO layer, as is shown in schematic design Figure 7a. There are various shapes and structures of capacitive TSPs based on the method used to form the touch sensor electrodes [33]. Among various methods, a method of forming a touch screen pattern in a low surface of a window and an on-cell TSP method for forming a touch screen pattern in an upper surface of a display effectively increases the display area while reducing the thickness [34]. Schematic is shown in Figure 7b represents the cross-sectional view of the ITO/SiO₂/ITO TSP in which ITO layer of X-axis or X-channel and Y-axis or Y-channel electrodes were indicated. The capacitive characteristics of the fabricated TSP were evaluated by employing the conventional direction method for an LC resonance circuit (TMS 1000, FTLAB Inc. Seoul, Korea) to measure the touch response. In a self-capacitive display, each pad acts as a single electrode with respect to the ground voltage. Each electrode represents a separate touch-coordination pair and must be individually connected to the controller [35,36]. To evaluate touch-sensitivity, the top ITO layer was transformed into conductive pads by photolithography, and copper lines were implemented as the external contacts to the channel electrodes. Additionally, a pressing force of 0.02 MPa (~500 mN) was applied to evaluate the capacitance changes. Figure 8a,b show the measured changes in TSP capacitance $[\Delta C]$ on the sapphire substrate when the channels are switched between the on/off modes, 5 channels were evaluated. When the screen was touched by a conducting cylindrical rod with an approximate diameter of 5 mm, the capacitance of each channel was measured to change by 10 pF. Irrespective of the channel number, the change in capacitance was not observed to exceed approximately 4 pF; the average change in capacitance for the proposed ITO/SiO₂/ITO OGS-TSP was measured as approximately 3.2 pF. Because the changes in capacitance primarily reflect the dielectric properties of the SiO₂ interlayer, the dielectric breakdown strength of the proposed structure was evaluated to better understand the breakdown strength of the SiO_2 in ITO/SiO₂/ITO. In general, the change in capacitance that occurs as a finger approaches an intersection for the capacitive TSP is typically approximately 1 pF or less [37]. As compared to the conventional change in capacitance range ~1 pF, the ITO/SiO₂/ITO structure fabricated on a sapphire substrate exhibited excellent sensitivity with the change in capacitance of 3.2 pF. Moreover, its durability and scratch resistance owing to the high degree hardness of the sapphire substrate. Furthermore, the multilayer structure was found to have a dielectric breakdown strength of approximately 3.0 MV/cm, which is enough for TSP applications. This relatively high breakdown strength is related to the quality of the dense SiO_2 film. Consequently, in this study, we achieved a high sensitivity OGS-TSP by sandwiching a thin layer of SiO_2 formed by low-temperature oxidation of Si in between the ITO layers.



Figure 7. (a) Schematic design of ITO/SiO₂/ITO/sapphire capacitive touch-screen panel (TSP) (10 mm \times 10 mm), and (b) cross-section view of the TSP representing the ITO layer (X and Y-axis electrodes) and the SiO₂ dielectric layer.



Figure 8. (a) Touch sensor-induced change in capacitance $[\triangle C]$ for each channel, and (b) touch sensor-induced change in capacitance $[\triangle C]$ for various frequencies.

4. Conclusions

In this study, a well-defined smooth and dense SiO₂ layer was produced by low-temperature oxidation via RTA of the sputter-deposited ultra-thin Si films. The Si films oxidized at 823 K were found to have high visible transmittance of approximately 91% at 550 nm and the optical simulation results were in good agreement with the observed optical transmittance data. The deconvolution of XPS peaks revealed that a well-defined SiO₂ formation with strong Si-O bonding in the films oxidized at 823 K. In order to investigate the potential application of SiO₂ generated by low-temperature oxidation of Si/ITO films, ITO/SiO₂/ITO multilayer structure was fabricated on a sapphire substrate. The ITO/SiO₂/ITO films annealed at 823 K exhibited the highest transmittance ~88.3% in the visible region. Moreover, the proposed ITO/SiO₂/ITO multilayer films exhibited a relatively high dielectric breakdown strength of approximately 3 MV/cm owing to the densely formed SiO₂ films. The touch-sensitive capacitance results revealed that the proposed design, which entails sandwiching a low-temperature-oxidized

 SiO_2 film in between the ITO layers on a sapphire substrate, has the potential to be implemented in capacitive TSP devices to realize high dielectric breakdown strength.

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