Simulation and deposition of near-IR anti-reflection layers for silicon substrates

Kyeeun Kim, Gwang Yeom Song, Yong Tae Kim, Jong Ha Moon, Jaeyeong Heo*

Department of Materials Science and Engineering and Optoelectronics Convergence Research Center, Chonnam National University, Gwangju 61186, Republic of Korea

A R T I C L E   I N F O

Keywords:
Atomic layer deposition
Anti-reflection
Silicon lens
Reflectivity
Humidity test

A B S T R A C T

Anti-reflection (AR) layers for Si were investigated for potential application in optical communications in the wavelength range of 1270–1330 nm. The optical simulation module of the Essential Macleod program was used to find the optimal thickness of single-layer and double-layer structures using Al2O3 and TiO2. Al2O3 was found to be a better AR single-layer because of a lower reflectance. Less than 1% reflectance was simulated using double-layer structures for both stack sequences Si/TiO2/Al2O3 and Si/Al2O3/TiO2. For experimental work, atomic layer deposition (ALD) of Al2O3 and TiO2 was employed to fabricate two different stacks. Reflectance measurements were conducted and 1.9% and 1.7% maximum reflectance was recorded in the wavelength range 1270–1330 nm. This reflectance establishes the possibility that the two stacks can be used as effective AR layers for Si lenses designed for optical communications. Resistance against humidity was tested for the two structures and only the Si/Al2O3/TiO2 structure was impermeable. Analyses using Fourier transform infrared spectroscopy and atomic force microscopy revealed that ALD-Al2O3 is easily hydroxylated while ALD-TiO2 acts as a good humidity barrier.

1. Introduction

An expansion of the optical network is necessitated by the rapidly increasing demand for high capacity mobile services. Because of this need, transmission rates of 100 Gbps through high-speed optical networking are rapidly applied on optical modules of data centers [1–4]. Increased efforts have been concentrated on silicon photonics and on combining optical communications for condensing more information within a limited bandwidth by minimizing the number of optical devices and reducing optical loss [5–10]. The role of a coupling lens is to combine optical devices and optical fiber so that it increases the coupling efficiency [11–14].

In the recent years, the wavelength used in optical communications has been distributed on a broad range from 780 to 1770 nm. Wavelengths ranging from 1270 to 1330 nm characterized by the lowest losses are utilized to send and receive data [15]. A conventional lens consists of fused silica or glass with a low refractive index of approximately 1.4–1.5. In contrast, silicon has attracted attention as a material for optical coupling lenses because of ease of manufacturing and a high refractive index of approximately 3.5 within the near-IR range [5–8]. Optical lenses with a high refractive index are an attractive option because photonic devices can be miniaturized.

Research in anti-reflection (AR) coatings that can drive the reduction of surface reflectance of optical coupling lenses is crucial to achieving high coupling efficiency. AR coatings enhance light transmittance instead of reflection on the interface between the two materials using destructive interference. The effect of AR coatings on Si has been extensively studied for photovoltaic applications, but the wavelength of interest was mainly in the visible light range of 400–800 nm [11,12,16–22].

AR coating for optical coupling lenses usually consists of TiO2, SiO2, Al2O3, or Si3N4. There are several methods for making AR structures including a sol-gel process, electron beam evaporation, plasma enhanced chemical vapor deposition (PECVD) and atomic layer deposition (ALD). Among them, ALD offers the advantage of easy thickness control at the atomic scale [23]. It also forms thin films of high purity [24,25]. In order to reduce the reflection from the surface of the optical lens, precise control of AR coating thickness is one of the most important factors. In addition, deposited films with high purity and a smooth surface are ideal in realizing optimized AR coatings.

In this study, we first simulated the effects of Al2O3 and TiO2 AR coatings on the reflectance of Si substrates concentrating on the 1270–1330 nm wavelength range, which is common in optical communications. Single-layer and double-layer structures were compared to find the optimal thickness of each layer. Deposition of Al2O3 and TiO2 on Si substrates was performed with ALD and reflectance was...
measured to compare simulated and measured data. Reflectance values of 1.9% and 1.7% were obtained from double-layer AR coatings on Si. The respective layers deposited were 50 nm TiO2/170 nm Al2O3 and 90 nm Al2O3/50 nm TiO2. Resistance against high humidity was also investigated and it was found that the ALD-Al2O3 film exhibits poor water permeability. A 50 nm-thick TiO2 layer on the surface was found to have high resistance against moisture.

2. Experimental

The Essential Macleod software, a powerful tool for optical analysis of thin films, was used to simulate the single-layer and double-layer Al2O3 and TiO2 AR structures. The refractive indices for Si, Al2O3, and TiO2 were set to 3.88, 1.66, and 2.47, respectively, at 632.8 nm. Atomic layer deposition (Atomic classic, CN1, Korea) was used to deposit TiO2 and Al2O3 AR coatings on (100) p-type silicon substrates. Titanium(IV) isopropoxide (TTIP, EGChem, Inc.) and trimethylaluminum (TMA, Al(CH3)3, EGChem, Inc.) were used as the Ti and Al precursors, and deionized water (H2O) was used as the oxygen source. The deposition temperature was all set to be 250 °C. The pulse sequence (precursor pulse-purge-reactant pulse-purge time) was optimally arranged to be 0.05 s–0.2 s–20 s and 0.15 s–0.2 s–20 s for Al2O3 and TiO2 growth, respectively. The deposited Al2O3 and TiO2 were amorphous and crystalline (rutile) phases, respectively.

Ellipsometer (LSE-USB, Gaertner) was used to measure the thickness and refractive index of Al2O3 and TiO2 thin films. The UV-visible spectrophotometer (Cary 500, Agilent) with a wavelength range from 380 to 1100 nm was used to determine the reflectance of thin films with and without AR coatings. The optical module has to be maintained at constant humidity when used outdoors [26] so we conducted resistance tests for humidity. Si wafers with AR coatings were boiled at 80 °C for 10 h in deionized water. The reflectance before and after boiling was compared. Fourier transform infrared spectrometry (FTIR, Spectrum 400, PerkinElmer) was used to investigate the structural change after the humidity test. Surface morphologies of the fabricated AR coatings on Si wafers were scanned using the atomic force microscope (AFM, XE100, PSIA).

3. Results and discussion

3.1. Simulation with single- and double-layer structures

AR structures with TiO2 and Al2O3 single-layers on Si were simulated and Fig. 1(a) shows the maximum reflectance of each AR layer as a function of film thickness. In this graph, the maximum reflectance is defined as the highest reflectance value recorded among wavelengths ranging from 1270 to 1330 nm. The variation of thickness was from 10 to 290 nm. At 0 nm of each film, which represents a bare Si substrate without AR coating, the maximum reflectance was 31.0%. As the thickness of either TiO2 or Al2O3 increases, maximum reflectance shows a gradual drop and the lowest reflectance values are 6.38% and 1.94% for 130 nm of TiO2 and 190 nm of Al2O3, respectively. Simulated total reflectance for wavelengths in the range of 800 to 2000 nm is also shown in Fig. 1(b). The simulated reflectance of a bare Si wafer is included for comparison. Based on this simulation, we can confirm that compared to TiO2, Al2O3 is a better AR coating for Si substrates in the IR range. However, the reflectance value of 1.9% is not as low as required for higher coupling efficiency in Si lenses. Therefore, we investigated double-layer AR structures to verify if the reflectance can decrease further.

Two different models were designed based on Si/TiO2/Al2O3 and Si/Al2O3/TiO2 layered structures. Fig. 2(a) shows the maximum reflectance of Si/TiO2/Al2O3 AR structures with increasing TiO2 thickness. In this graph, data for three different thicknesses of Al2O3 (100, 130, and 170 nm) are illustrated. For 100 nm-thick Al2O3, the minimum reflectance is 1.68% when the thickness of TiO2 is set to 100 nm. A maximum reflectance of less than 1% is observed for a 90 nm TiO2 layer with 130 nm of Al2O3 (the actual value is 0.40%). A similar value is observed for a 50 nm TiO2 layer with 170 nm of Al2O3 (the actual value is 0.73%). The results for the structure Si/Al2O3/TiO2 with inverted layers are also illustrated in Fig. 2(b). Low maximum reflectance values of 0.25% and 0.28% were simulated when TiO2/Al2O3 thicknesses were 40 nm/105 nm and 50 nm/90 nm, respectively. The simulation demonstrates that both double-layer structures can effectively function as good AR layers. Based on the simulation, two sets of AR structures were selected for experimental validation by considering their respective maximum reflectance and TiO2 thickness. These were the Si/50 nm-TiO2/170 nm-Al2O3 and the Si/90 nm-Al2O3/50 nm-TiO2 structures. The 50 nm TiO2 thickness was fixed for both models because the growth rate of TiO2 ALD is slower than that of Al2O3 and both experiments can be performed with the reliable ALD process.

3.2. ALD fabrication of double-layer AR structure

Fig. 3(a) shows the simulated (dotted line) and measured reflectance (solid lines) of Si/50 nm-TiO2/170 nm-Al2O3 AR structures. It is noted that the overall shape of the experimentally obtained reflectance curves replicate the trend of the simulation, suggesting that the thickness of both TiO2 and Al2O3 layers are near the target values. In this regard, ALD is a powerful tool that validates the simulation results experimentally. The exception is the reflectance from 1130 to 1600 nm which is somewhat higher than the simulation result. Maximum reflectance at 1270–1330 nm was 2.9% while the simulated value is 0.73%. A wavelength of 1130 nm corresponds to the photon energy of 1.1 eV and it is consistent with the bandgap energy of Si. Longer wavelength than the bandgap energy is not well absorbed by Si.
and it is presumed that reflection from the backside of the Si slightly increases the reflectance [27]. In order to validate this hypothesis, we deliberately scratched the backside of Si to minimize the reflected light by interfering with the reflectance signal. The reflectance measurement was recorded again and the result is plotted as a solid line in red. Clearly, it is seen that the reflectance approximates the simulation result with lower reflectance values near a vertex of the curve compared to the one without scratching. The new maximum reflectance obtained from the curve was 1.9%. The Si/90 nm-Al2O3/50 nm-TiO2 structure was also prototyped using ALD (solid line, Fig. 3(b)). In this experiment, the backside surface of Si was polished to eliminate the effect of the reflectance. The reflectance spectrum follows the trend of the simulation curve (dotted line) and the maximum reflectance recorded was 1.7% for the wavelength range of 1270–1330 nm.

3.3. Humidity test for double-layer AR structures

In order to investigate the reliability of both AR coatings in the

---

**Fig. 2.** (a) Maximum reflectance from 1270 to 1330 nm for Si/TiO2/Al2O3 AR structure as a function of TiO2 thickness. Al2O3 thicknesses were selected to be 100, 130, and 170 nm. (b) Maximum reflectance in the wavelength range 1270–1330 nm for Si/Al2O3/TiO2 AR structure as a function of TiO2 thickness. Al2O3 thicknesses were selected to be 90, 95, 100, and 105 nm.

---

**Fig. 3.** (a) Reflectance of Si/50 nm-TiO2/170 nm-Al2O3 double-layer structure. The dotted line indicates the result from the simulation. Solid lines in black and red color indicate the measured reflectance before and after Si backside polishing, respectively. (b) Reflectance of Si/90 nm-Al2O3/50 nm-TiO2 double-layer structure. Polishing was conducted prior to the measurement. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

---

**Fig. 4.** Reflectance spectra for (a) Si/50 nm-TiO2/170 nm-Al2O3 and (b) Si/90 nm-Al2O3/50 nm-TiO2 double-layer structures before (dotted lines) and after (solid lines) the humidity test.
presence of humidity and heat, we boiled the AR coating structures deposited on Si substrates in deionized water for 10 h at 80 °C. A color change of the deposited film was observed for the Si/50 nm-TiO2/170 nm-Al2O3 structure while no obvious change was observed for the Si/50 nm-Al2O3/50 nm-TiO2 structure. The difference is more pronounced when the reflectance spectra for both structures are compared before and after the treatment, as shown in Fig. 4. For the Si/50 nm-TiO2/170 nm-Al2O3 structure, a drastic increase in reflectance over the entire wavelength range was obtained as shown in Fig. 4(a). In contrast, no obvious change in reflectance was recorded for the Si/50 nm-Al2O3/50 nm-TiO2 structure (Fig. 4(b)). Although the two structures yielded similar maximum reflectance (1.9% and 1.7%), different resistance against the humidity and heat was observed. Since 80 °C is not a high enough temperature to deform or deteriorate the oxide films, the permeability of water through the film was suspected. To better understand resistance to water permeation, we carried out the same experiment in a high humidity environment for single-layers of TiO2 and Al2O3 deposited on Si.

Fig. 5(a) shows the reflectance spectra for 175 nm and 190 nm thick Al2O3 films grown on Si substrates before (dotted lines) and after (solid lines) the humidity test. The broad hump is noted for Al2O3 film after the test. A notable difference is more pronounced in reflectance in the range of optical communications wavelengths of 1270–1330 nm was investigated using an Essential Macleod simulation. Single-layer designs of Si/Al2O3 showed lower reflectance (1.94%) than a Si/TiO2 structure (6.38%) in terms of maximum reflectance in the

Fig. 5(b) shows the reflectance spectra for 10 nm, 20 nm, and 50 nm thick TiO2 films on Si substrate before (dotted lines) and after (solid lines) the humidity test.

FTIR was used to investigate the change in bonding states of the ALD-grown Al2O3 and TiO2 films. A notable difference was recorded for the Al2O3 film before and after the humidity test, as shown in Fig. 6(a). The broad peak in the range 3200–3500 cm−1 is only noted for the Al2O3 film after the treatment. This is the characteristic IR band due to structural –OH group [28]. In addition, we also checked the change in surface morphologies by AFM. Fig. 6(b) and (c) show the scanned surface images of Si/Al2O3 before and after the treatment, respectively. Root-mean-square roughness of the as-prepared sample was 0.27 nm, but it drastically increased to 56.4 nm after exposure to high humidity at 80 °C. Therefore, it is concluded that the ALD-Al2O3 film has poor resistance against the water permeation compared to the ALD-TiO2 film. Similar results of poor performance by ALD-Al2O3 against humidity were recently reported by other researchers [29]. This result suggests that ALD-TiO2 film is a good barrier against water permeation.

4. Conclusions

The possibility of Al2O3 and TiO2 thin films as effective AR coatings in the range of optical communications wavelengths of 1270–1330 nm was investigated using an Essential Macleod simulation. Single-layer designs of Si/Al2O3 showed lower reflectance (1.94%) than a Si/TiO2 structure (6.38%) in terms of maximum reflectance in the
is concluded that Si/90 nm-Al2O3/50 nm-TiO2 is the optimized structure for AR coating for optical communications. Development of commercialization technology for 100 Gbps small form-factor optical communication modules). The ALD-TiO2 layer was found to be an effective barrier for humidity while the ALD-Al2O3 film is easily hydroxylated. Considering the need for reliable minimum reflectance, it is concluded that Si/90 nm-Al2O3/50 nm-TiO2 is the optimized structure for AR coating for optical communications.

Acknowledgements

This work was supported by an Institute for Information & Communications Technology Promotion (IITP) grant funded by the Korea government (MSIP) (No. R6913-16-0001, Development of commercialization technology for 100 Gbps small form-factor optical communication modules).

References