Formation of antireflection structures for silicon in near-infrared region using AlO$_x$/TiO$_x$ bilayer and SiN$_x$ single-layer

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1. Introduction

Silicon photonics has experienced rapid development with the progress of silicon (Si) technologies. In particular, the development of high-speed optical transceivers has attracted significant interest [1,2]. Copper wiring for data transmission is currently replaced by Si-based optical fibers. Optical signals using light, instead of electrical signals, have been developed for communication. Devices have become smaller and more integrated, and extensive studies have been performed to improve their efficiencies [3-5]. In general, when light from a light source is incident on an optical fiber, approximately 1.2 dB of the light intensity is lost [6]. In order to reduce this loss, an optical coupling lens is used. Owing to the advancement of silicon photonics, Si is mainly used for optical coupling lenses [7]. In Si, approximately 40% of the light intensity is lost owing to reflections at wavelengths of 1310 nm and 1550 nm, which are mainly used in optical communication [8]. One approach to reduce the reflection loss is to introduce an antireflection (AR) coating on the lens surface [1,9]. The AR coating is a thin film that increases the transmittance by reducing the reflection of light at the interface of the medium [10]; it is commonly used in industries where the optical efficiency is required to be increased. It can also be used for solar cells, light-emitting diodes, polarizers, and front plates of displays to improve the device's performance [11,12].

AlO$_x$, TiO$_x$, SiO$_x$, and SiN$_x$ are commonly used as AR coatings for optical coupling lenses [9,10]. All of them have excellent passivation properties and excellent transmittance in the near-infrared (IR) range. Various techniques can be employed to coat these substances on a lens surface, such as the sol-gel process, electron-beam evaporation, plasma-enhanced chemical vapor deposition (PECVD), and atomic layer deposition (ALD). PECVD is advantageous as it can be performed at a lower temperature, compared with the other methods, and in addition, it enables a rapid deposition [13]. ALD provides an excellent step coverage and control of the thickness of the film at the atomic level, which make it suitable for the production of AR coatings whose performance depends on the thickness of the film [14].

In this study, we investigated the possibility of using an AlO$_x$/TiO$_x$ bilayer and SiN$_x$ single-layer as AR structures for Si, focusing on the wavelengths of 1270–1330 nm. The thicknesses of each layer were optimized by simulations using the Essential Macleod software. Both AR structures were experimentally fabricated using ALD and PECVD, and their performances were compared. Both AR structures lowered the reflectance, close to 0%, at the wavelength band of 1270–1330 nm, which enabled an increase of the transmittance by approximately 40%, compared with bare Si.

1.1. Experimental details

The Essential Macleod software was used to generate an efficient...
thin-film AR structure design, which is a common software package for an efficient design of thin-film optical coatings [15,16]. In this study, an $\text{AlO}_x/\text{TiO}_x$ bilayer [17] and $\text{Si}_x$ single-layer were considered. $\text{AlO}_x$ and $\text{TiO}_x$ thin films were deposited using ALD (Atomic classic, CN1, Korea) at 170 °C and 250 °C, respectively. As Al and Ti precursors, trimethylaluminum (TMA, Al(CH$_3$)$_3$, EGChem, Inc.) and tetrakis(dimethylamino)titanium (TDMA-Ti, Ti[(CH$_3$)$_2$N]$_4$, EGChem, Inc.) were used, respectively. Deionized water (H$_2$O) was used as the oxidant. The sources for TMA and H$_2$O were set to room temperature, while for TDMA-Ti to 70 °C. The ALD sequences (precursor-pulse–purge–reactant-pulse–purge) were set to 0.1 s - 10 s - 0.4 s - 25 s for $\text{AlO}_x$ and 0.5 s - 15 s - 1 s - 30 s for $\text{TiO}_x$, respectively. $\text{Si}_x$ was deposited by PECVD (VI-LA-PECVD, Unisax) at 250 °C using a silane process. Double-side-polished p-type (100) Si was used as the substrate. The structure of the sample is shown in Fig. 1. The target thicknesses of the two sides of each sample were equal.

Spectroscopic ellipsometry (SE, V-VASE Ellipsometer, J. A. Woollam Co.) was used to determine the refractive index ($n$) and extinction coefficient ($k$) of each film. The Cauchy equation was used for the analytical model. A single-wave ellipsometer (LSE-USB, Gaertner, 632.8-nm wavelength) was also used to measure the thickness and refractive index of the $\text{AlO}_x$, $\text{TiO}_x$, and $\text{Si}_x$ thin films. An UV-Vis-near-IR spectrophotometer (Cary 5000, Agilent) with a wavelength range of 800–2000 nm was used to measure the reflectance and transmittance of the Si wafers with and without AR coatings. An atomic force microscope (AFM,XE100, PSIA) was used to scan the surface morphologies of the Si wafers with and without AR coatings. Chemical states of the grown films were analyzed using X-ray photoelectron spectroscopy (XPS, K-Alpha+, Thermo Fisher Scientific). The XPS spectra of the $\text{TiO}_x$ films were obtained after an Ar$^+$-ion beam etching for 20 s in order to remove surface contaminations. X-ray diffraction (XRD, XPert Pro MPD, PANalytical) was used to investigate the crystallinity of the $\text{TiO}_x$ film.

### 2. Results and discussion

The refractive index of each material was measured using SE. Fig. 2 shows the refractive indices of the $\text{AlO}_x$, $\text{TiO}_x$, and $\text{Si}_x$ films measured by SE in the wavelength range of 800–2000 nm.

Fig. 3. Simulated reflectance spectra of the $\text{AlO}_x/\text{TiO}_x$ (black) and $\text{Si}_x$ (red) AR structures; a magnified view of these spectra is shown in the inset. The simulated average reflectances of $\text{AlO}_x/\text{TiO}_x$ and $\text{Si}_x$ in the wavelength range of 1270–1330 nm were 0.03% and 0.30%, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 4 shows the measured reflectance and transmittance of the fabricated AR structures on both sides of Si substrates, designed according to the above simulation results. As light with a small wavelength of 1100 nm (or smaller) has a sufficiently larger energy than the band gap of Si, the light reflected from the backside is absorbed in Si; however, light with a larger wavelength is not absorbed in Si, hence it is emitted to the surface again, thus increasing the reflectance. In order to mitigate the backside reflection, we fabricated the above AR structure on both sides of the Si substrate, as depicted in Fig. 1. The measured thicknesses of the $\text{AlO}_x$, $\text{TiO}_x$, and $\text{Si}_x$ films were 98, 41, and 167 nm, respectively. The dashed curves in Fig. 4 represent the measured values for bare Si, while the solid curves represent the measured values of the $\text{AlO}_x/\text{TiO}_x$ (black) and $\text{Si}_x$ (red) AR structures. Fig. 4(a) shows that the reflectances of bare Si are approximately 33% and 47% at wavelengths of 800 nm and 1100 nm, respectively. The reflectance of the fabricated
AR structure decreased from the wavelength of 800 nm, and started to increase from a wavelength of 1300 nm, consistent with the simulations. In the wavelength band of 1270–1330 nm, the average reflectance and transmittance of bare Si were 47.0% and 52.8%, respectively. Previous studies were mainly focused on the transmittance and reflectance in the visible region [18,19]. Our results suggest that excellent AR structures for a silicon optical coupling lens in the near-IR region can be fabricated using two different combinations.

Fig. 4(b) shows that the average transmittance of bare Si was 52.8% in the wavelength range of 1270–1330 nm, while those of the AlOx/TiOx and SiNx AR structures increased to 88.1% and 99.5%, respectively. The same AR structures were used for measurement. The transmittance of the AlOx/TiOx AR structure was nearly 10% lower than that of the SiNx AR structure. In the bare Si and SiNx AR structures, most of the light was reflected and transmitted only in the medium, whereas in the AlOx/TiOx AR structure, approximately 10% of the light was absorbed or scattered inside the film, i.e., it was not transmitted to the opposite side. In order to investigate the origin of the reduced transmittance of the AlOx/TiOx AR structure, we performed further analyses, as presented below.

We performed an AFM analysis to evaluate the surface morphologies of the fabricated AR structures. Fig. 5 shows the surface morphologies of the (a) AlOx/TiOx and (b) SiNx AR structures. The surface of the SiNx AR structure was rougher than that of the AlOx/TiOx AR structure. The root-mean-square roughness ($R_q$) of the SiNx AR structure was 2.8 nm, while that of the AlOx/TiOx AR structure was 0.53 nm. Both AR structures had a relatively smooth and uniform surface with $R_q$ below 10 nm, thus minimizing the scattering of the light. The obtained result indicates that the roughness of the film surfaces does not significantly affect the transmittances of the two AR structures.

Further, we measured the reflectance and transmittance of single layers of AlOx and TiOx grown on quartz substrates. Similar film thicknesses of 58 nm were obtained on the substrates to exclude the influence of the film thickness. Fig. 6 shows the (a) transmittance and reflectance, and (b) corresponding absorption spectra of each film. The AlOx film exhibits a high transmittance and low reflectance, while the TiOx film exhibits a relatively high reflectance and low transmittance. The absorption spectra in Fig. 6(b) reveal a large absorption of the TiOx film on the quartz substrate at a wavelength of 378 nm (3.28 eV), which corresponds to the band gap of TiOx. The increased absorption near 200 nm for the AlOx film is attributed to the absorption in the quartz substrate (band gap of 6.3 eV). A similar absorption is also observed for the bare quartz substrate, as shown in the inset. In contrast to the AlOx film, approximately 5–12% of the light is absorbed in the TiOx structure in the near-IR region. This experimental result implies that the reduced transmittance of the AlOx/TiOx AR structure can be attributed to the absorption in the TiOx layer. A similar absorption of TiOx films in the near-IR region has been reported in other studies [20,21]. According to Won et al. [20], a reduction of transmittance can occur for the TiOx film, which is correlated with the charge state of Ti$^{3+}$ in the TiOx film.
Pereira et al. attributed the absorption in the near-IR region to a high density of oxygen defects in the TiO\textsubscript{x} film [21].

An XPS analysis was performed to reveal the chemical state of the as-grown (by ALD) TiO\textsubscript{x} film. Fig. 7(a) shows the Ti 2\textit{p} XP spectra of the as-grown TiO\textsubscript{x} film. The broad peak of Ti 2\textit{p} is deconvoluted to identify the detailed chemical information. The Ti 2\textit{p}\textsubscript{3/2} peak contains Ti\textsuperscript{2+} (455.5 ± 0.2 eV), Ti\textsuperscript{3+} (457.0 ± 0.2 eV), and Ti\textsuperscript{4+} (459.0 ± 0.2 eV) peaks [22–24], while the Ti 2\textit{p}\textsubscript{1/2} peak contains Ti\textsuperscript{2+} (462.0 ± 0.2 eV), Ti\textsuperscript{3+} (462.4 ± 0.2 eV), and Ti\textsuperscript{4+} (464.4 ± 0.2 eV) peaks [22]. The as-deposited TiO\textsubscript{x} film has a comparable portion of Ti\textsuperscript{3+} and Ti\textsuperscript{4+} states. This chemical state of TiO\textsubscript{x} is partially related to the amorphous state of the as-grown TiO\textsubscript{x}. The amorphous phase of the TiO\textsubscript{x} film for the employed ALD conditions is confirmed using XRD (Fig. 8), which has also been reported in other studies [25,26]. In order to increase the oxidation state of Ti in the as-grown TiO\textsubscript{x}, an additional annealing was performed under an air environment at 300 °C for 2 h. The annealing enabled a transformation of the amorphous phase into a crystalline TiO\textsubscript{x} with an anatase phase, as shown in Fig. 8. Fig. 7(b) shows the Ti 2\textit{p} XP spectra of the 300-°C-annealed TiO\textsubscript{x} film. The annealed TiO\textsubscript{x} film mainly consists of the Ti\textsuperscript{4+} state with a reduced portion of Ti\textsuperscript{3+}, compared with that of the as-deposited TiO\textsubscript{x} film; the area percentages of the Ti\textsuperscript{4+}, Ti\textsuperscript{3+}, and Ti\textsuperscript{2+} peaks changed from 32%, 54%, and 14% to 45%, 35%, and 20%, respectively. This suggests that the air annealing enhances the oxidation of the as-deposited TiO\textsubscript{x} film. Moreover, the refractive index of the TiO\textsubscript{x} film increased from 2.42 to 2.49 at 632.8 nm, suggesting the further densification of the film.

In order to confirm the effect of the Ti oxidation states as well as crystallinity of the TiO\textsubscript{x} film, an AlO\textsubscript{x}/TiO\textsubscript{x} AR structure was fabricated, and its transmittance and reflectance before and after the annealing at 300 °C for 2 h were compared, as shown in Fig. 9. The average...
the overall portion of the Ti$^{4+}$ state in the TiO$_x$ film with an improved density. These findings reveal that both AlO$_x$/TiO$_x$ and SiNx are good candidates for AR structures for Si in the near-IR wavelength region.

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References


Fig. 9. Transmittance and reflectance spectra of the AlO$_x$/TiO$_x$ AR structure, where TiO$_x$ was annealed at 300 °C for 2 h.

Transmittance / Reflectance (%)