Journal of Vacuum Science and Technology A

# SiO2/TiO2 distributed Bragg reflector near 1.5 μm fabricated by e-beam evaporation

I-Wen Feng, Sixuan Jin, Jing Li, Jingyu Lin, and Hongxing Jiang

Citation: J. Vac. Sci. Technol. A **31**, 061514 (2013); doi: 10.1116/1.4823705
View online: http://dx.doi.org/10.1116/1.4823705
View Table of Contents: http://avspublications.org/resource/1/JVTAD6/v31/i6
Published by the AVS: Science & Technology of Materials, Interfaces, and Processing

# $SiO_2/TiO_2$ distributed Bragg reflector near 1.5 $\mu$m fabricated by e-beam evaporation

I-Wen Feng, Sixuan Jin, Jing Li, Jingyu Lin, and Hongxing Jiang[a)]
*Department of Electrical and Computer Engineering, Texas Tech University, Lubbock, Texas 79409*

(Received 5 August 2013; accepted 16 September 2013; published 1 October 2013)

The authors report on the fabrication and characterization of $SiO_2/TiO_2$ distributed Bragg reflector (DBR) mirrors operating at the eye safe and optical communication wavelength window, $\lambda = 1.5\,\mu$m. Our experimental results demonstrated that $SiO_2/TiO_2$ DBR mirrors with reflectivity exceeding 95% at $\lambda = 1.5\,\mu$m can be achieved using e-beam evaporation in conjunction with postdeposition thermal annealing process in ambient air. It was found that the postdeposition annealing process transformed the crystal structure of the as-deposited $Ti_xO_y$ to $TiO_2$, leading to a significant reduction in optical absorption. Erbium doped III-nitride semiconductors incorporating DBR mirrors at 1.5 $\mu$m emission may open up many novel applications, including infrared emitters, optical amplifiers, and high power infrared lasers. 
[http://dx.doi.org/10.1116/1.4823705]

## I. INTRODUCTION

The optical loss of silica fibers, caused by Rayleigh scattering and infrared absorption, has a minimum at the spectral region near $\lambda = 1.5\,\mu$m. Coincidentally, the intra-4*f* transition of trivalent Er atoms ($Er^{3+}$) from the first excited to ground state ($^4I_{13/2} \rightarrow {}^4I_{15/2}$) offers the 1.5 $\mu$m emission. Hence, erbium doped semiconductors and dielectric materials have many applications in optical communications, ranging from infrared emitters, erbium doped fiber amplifiers (EDFA), to other photonic devices.[1,2] Many of these device applications would benefit from the development of distributed Bragg reflectors (DBRs) at $\lambda = 1.5\,\mu$m, which are expected to enhance the optical performance of the photonic devices based on Er doped materials. In particular, Er doped III-nitride semiconductors[3,4] integrated with DBRs at 1.5 $\mu$m are promising for realizing chip-scale electrically or optically pumped optoelectronic devices, including erbium doped waveguide amplifiers, infrared emitters, and high power infrared lasers with superior thermal stability.

DBR mirrors generally consist of many periods of two alternating materials with different refractive indices. The layer thickness of each layer is $\sim\lambda/4n$, where $\lambda$ and n refer to the light wavelength and refractive index at the targeted wavelength, respectively. In this report, we selected $SiO_2$ and $TiO_2$ dielectric materials as alternating DBR layers due to their low absorption coefficients at $\lambda = 1.5\,\mu$m and a large contrast of refractive indices ($\Delta$n), which reduce the periods of DBR mirrors needed to achieve the desired reflectivity and a relatively broad stop band.[5,6] Because of the well-known fact that heated $Ti_3O_5$ forms $TiO_2$ with a supply of oxygen, $Ti_3O_5$ has been widely used as a source material for $TiO_2$ thin films deposited by ion-assisted deposition or RF sputtering.[7–10] However, the optical performance of $TiO_2$ layers is sensitive to the substrate temperature and environmental $O_2$ pressure used during ion-assisted deposition or RF sputtering deposition.[7–10] Moreover, ion-assisted deposition and RF sputtering processes are less compatible with III-nitride optoelectronic device fabrication processes. In the present study, we prepared $SiO_2/TiO_2$ DBR mirrors via conventional e-beam evaporation using $Ti_3O_5$ as the source material from which relatively high purity materials could be achieved within the high vacuum deposition environment. To improve the performance of $SiO_2/TiO_2$ DBR mirrors deposited by e-beam evaporation, we employed a postdeposition thermal annealing process in ambient air. Our experimental results demonstrated that $SiO_2/TiO_2$ DBR mirrors with reflectivity >95% at $\lambda = 1.5\,\mu$m can be achieved using e-beam evaporation in conjunction with postdeposition thermal annealing process in ambient air, providing a new and simplified route for the realization of high quality $SiO_2/TiO_2$ DBR mirrors.

## II. EXPERIMENT

$SiO_2/TiO_2$ DBR mirrors were deposited on $Al_2O_3$ substrates using an e-beam evaporator (Edwards 306) with $SiO_2$ (CERAC, 99.99%) and $Ti_3O_5$ (CERAC, 99.9%) as the source materials with deposition rates of ~4 and ~2 nm/min, respectively. The deposition rates were monitored by a gold coated quartz crystal. Thermal annealing processes were then performed in ambient air at various annealing temperatures ($T_a$) and time ($t_a$) to improve the performance of $SiO_2/TiO_2$ DBR mirrors. The reflectivity spectra of $SiO_2/TiO_2$ DBR mirrors were measured at a near-normal incident using a reflectance probe (Stellarnet) and detected using a thermoelectrically cooled IR spectrometer (Bayspec) with a 550 nm thick aluminum thin film deposited on the $Al_2O_3$ substrate as the reference sample having a reflectivity of ~97% at $\lambda \sim 1.5\,\mu$m.

## III. RESULTS AND DISCUSSIONS

### A. $TiO_2$ thin films

To study the effects of the thermal annealing process on the properties of $Ti_xO_y$ thin films, $Ti_xO_y$ layers of 260 nm in thickness were deposited on $Al_2O_3$ substrates and then

a)Electronic mail: hx.jiang@ttu.edu

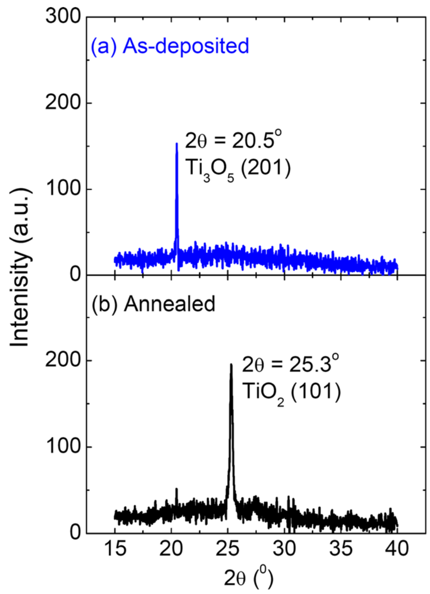


FIG. 1. (Color online) $\theta$–$2\theta$ XRD spectra measured from the (a) as-deposited and (b) annealed ($T_a = 375\,°C$ for $t_a = 30$ min in ambient air) $Ti_xO_y/Al_2O_3$ thin films prepared by e-beam evaporation.

annealed at $T_a = 375\,°C$ for $t_a = 30$ min in ambient air. Figure 1 shows $\theta$–$2\theta$ x-ray diffraction (XRD) spectra measured from as-deposited and annealed $Ti_xO_y/Al_2O_3$ samples. The as-deposited $Ti_xO_y/Al_2O_3$ sample exhibits a $2\theta$ peak at 20.5°, a reflection peak identified as $Ti_3O_5$. In contrast, we observed $2\theta$ peak at 25.3° from the annealed sample, which is identified as a $2\theta$ peak of $TiO_2$.[8,11–13] It appears that the thermal annealing process initiates the crystal structure transformation from $Ti_3O_5$ to $TiO_2$. The as-deposition of $Ti_xO_y$ using a conventional e-beam evaporator occurs in an environment that is deficient in $O_2$ and hence results in the formation of $Ti_3O_5$. Thermal annealing with $O_2$ transforms $Ti_3O_5$ to $TiO_2$. Figure 2 shows that the change in crystalline property of $Ti_xO_y$ thin films due to the annealing process also altered the reflectance and transmittance spectra of $Ti_xO_y/Al_2O_3$ samples.[14,15] It can be seen that the optical absorption in $Ti_xO_y$ is significantly reduced upon the thermal annealing process.

### B. $SiO_2/TiO_2$ DBR

#### *1. Annealing temperature effect*

Five periods of $SiO_2/TiO_2$ DBR mirrors were deposited on $Al_2O_3$ substrates with thicknesses of $t_{SiO_2} = 258$ nm and $t_{TiO_2} = 206$ nm, respectively, and then followed by thermal annealing in ambient air for $t_a = 60$ min at various temperature, $T_a = 325$, 375, and 425 °C. Figure 3(a) shows the optical microscopy images of the as-deposited and annealed $SiO_2/TiO_2$ DBR mirrors at different $T_a$. Notably, as $T_a$ was increased, the image of $SiO_2/TiO_2$ DBR mirrors changed gradually from opaque black to transparent chartreuse. It was found that, at $T_a \geq 425\,°C$, $SiO_2/TiO_2$ DBR mirrors started to peel off from $Al_2O_3$ substrates because of the presence of a large strain induced by the difference between the thermal expansion coefficients of $SiO_2/TiO_2$ DBR mirrors and $Al_2O_3$ substrates. Also shown in Fig. 3(b) are the reflectivity spectra measured from the as-deposited samples and samples annealed at different temperatures, $T_a = 325$ and 375 °C, of 5 periods of $SiO_2/TiO_2$ DBR mirrors. We observed that thermal annealing changed the reflectivity pattern of $SiO_2/TiO_2$ DBR mirrors, where the reflectivity at $\lambda = 1.5\,\mu m$ increased from ∼51% to ∼91% as the temperature was increased up to 375 °C.

#### *2. Annealing time dependence*

$SiO_2/TiO_2$ DBR mirrors (2, 5, and 7 periods with $t_{SiO_2} = 258$ nm and $t_{TiO_2} = 206$ nm) were annealed at $T_a = 375\,°C$ in ambient air for different annealing times ($t_a$). Figure 4(a) shows the reflectivity spectra measured from as-deposited and annealed samples. The reflectivity spectra were gradually modified with an increase of $t_a$, where the reflectivity near $\lambda = 1.5\,\mu m$ increased up to 91% for $t_a \geq 60$ min. Figure 4(b) summarizes the infrared reflectivity near $\lambda = 1.5\,\mu m$ as a function of $t_a$ measured for 2, 5, and 7 periods of $SiO_2/TiO_2$ DBR mirrors. The infrared reflectivity at $\lambda = 1.5\,\mu m$

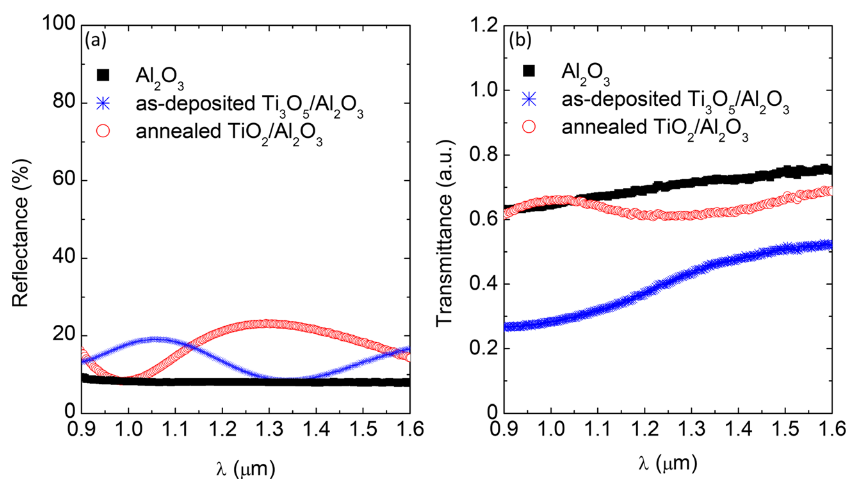


FIG. 2. (Color online) (a) Reflectance and (b) transmittance spectra measured from the as-deposited $Ti_xO_y/Al_2O_3$ and annealed $TiO_2/Al_2O_3$ samples ($T_a = 375\,°C$ for $t_a = 30$ min in ambient air).

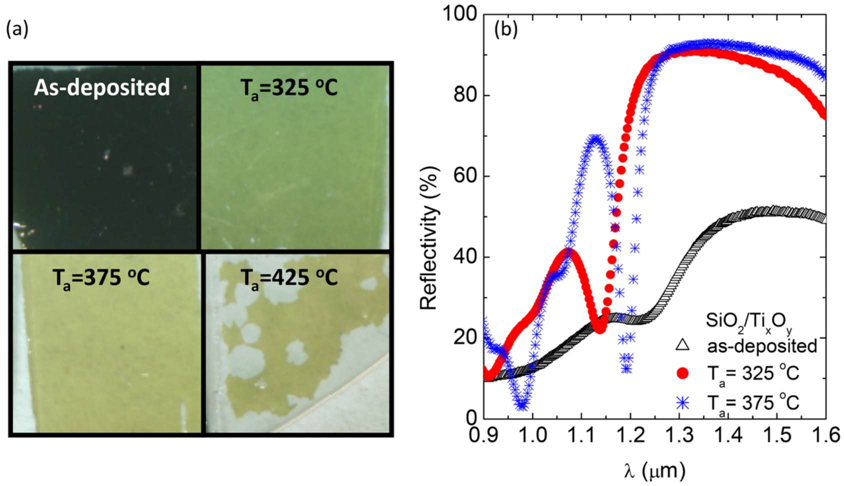


FIG. 3. (Color online) Optical microscopy images of the as-deposited $SiO_2/Ti_xO_y$ and annealed $SiO_2/TiO_2$ (258 /206 nm) DBR mirrors (5 periods) with $T_a = 325$, 375, and 425 °C and $t_a = 60$ min in ambient air. (b) Reflectivity spectra measured from the as-deposited $SiO_2/Ti_xO_y$ and annealed 5 periods of $SiO_2$/$TiO_2$ DBR mirrors with $T_a = 325$ and 375 °C and $t_a = 60$ min in ambient air.

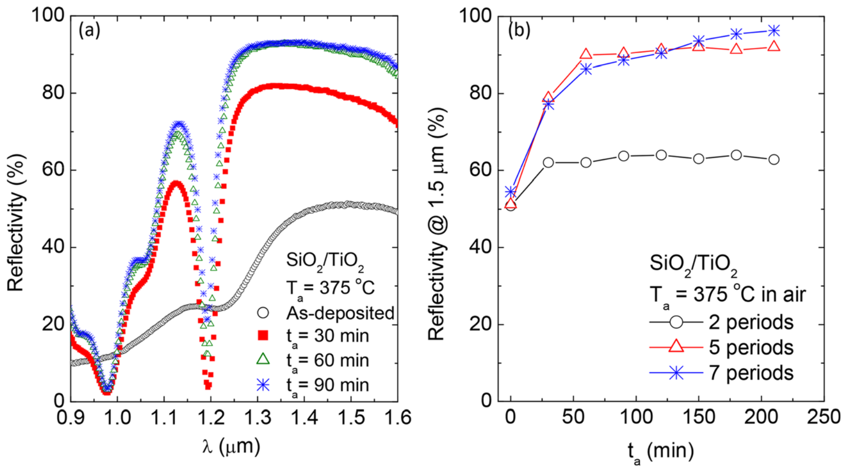


FIG. 4. (Color online) (a) Reflectivity spectra measured from the as-deposited $SiO_2/Ti_xO_y$ and annealed ($T_a = 375$ °C in ambient air) $SiO_2/TiO_2$ (258/206 nm) DBR mirrors (5 periods) with different $t_a$. (b) Reflectivity at $\lambda = 1.5$ $\mu$m as a function of $t_a$ measured from $SiO_2/TiO_2$ DBR mirrors with 2, 5, and 7 periods ($T_a = 375$ °C in ambient air).

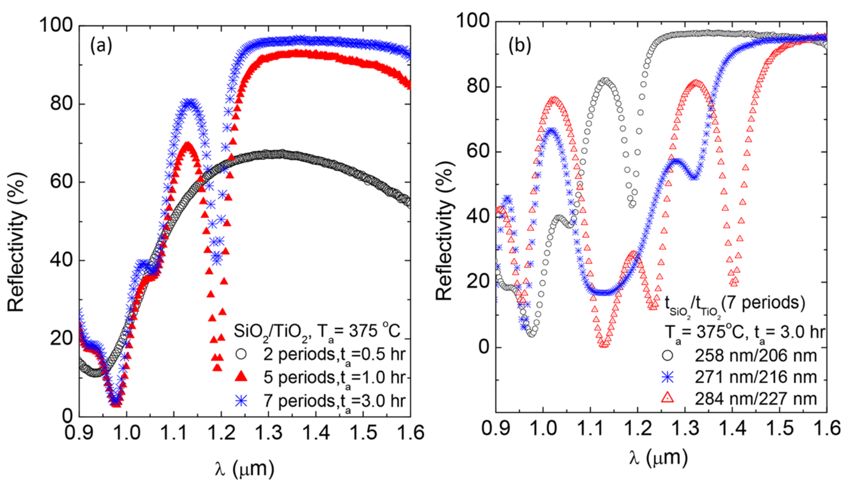


FIG. 5. (Color online) (a) Reflectivity spectra measured from annealed $SiO_2/TiO_2$ DBR mirrors with thicknesses of $t_{SiO_2} = 258$ nm and $t_{TiO_2} = 206$ nm and with 2, 5, and 7 periods annealed at each optimized $t_a$ of 0.5, 1.0, and 3.0 h, respectively. (b) Reflectivity spectra measured from the annealed $SiO_2/TiO_2$ DBR mirrors (7 periods) at $T_a = 375$ °C for $t_a = 3.0$ h with different $t_{SiO2}$/ $t_{TiO2}$ thicknesses (258/206 nm, 271/216 nm, and 284/227 nm).

increases with an increase of $t_a$ and then appears to saturate. It was also observed that $SiO_2/TiO_2$ DBR mirrors with more periods reach a higher reflectivity level but require a longer $t_a$ to reach their maximum reflectivity.

### *3. Period and thickness dependence*

Figure 5(a) shows the reflectivity spectra of the annealed $SiO_2/TiO_2$ DBR mirrors of 2, 5, and 7 periods with thicknesses of $t_{SiO_2} = 258$ nm and $t_{TiO_2} = 206$ nm at each optimized annealing condition. The reflectivity near 1.5 $\mu$m increased up to 96% with an increase of the period of the samples. Also shown in Fig. 5(b) are the reflectivity spectra of the annealed samples with varying $t_{SiO_2}/t_{TiO_2}$ thickness, 258/206 nm, 271/216 nm, and 284/227 nm, with a nearly constant $t_{SiO_2}/t_{TiO_2}$ ratio. It was observed that the stop band of the annealed samples red-shifted with an increase in the layer thickness. The annealed sample with thicknesses of $t_{SiO_2} = 271$ nm and $t_{TiO_2} = 216$ nm attains the reflectivity >95% with the stop band covering the full spectral window of the intra-4*f* transition from $^4I_{13/2}$ to $^4I_{15/2}$ of $Er^{3+}$ ions in III-nitride semiconductors.

## IV. SUMMARY AND CONCLUSIONS

In summary, we have fabricated $SiO_2/TiO_2$ DBR mirrors by e-beam evaporation in conjunction with a postdeposition thermal annealing process. The reflectivity spectra of $SiO_2/TiO_2$ DBR mirrors near 1.5 $\mu$m can be controlled by the thermal annealing conditions as well as by the period and layer thickness of the DBRs, where we are able to achieve DBR mirrors with a reflectivity >95% near $\lambda = 1.5$ $\mu$m. Erbium doped semiconductors integrated with $SiO_2/TiO_2$ DBR mirrors will open up novel applications including infrared emitters, optical amplifiers, and high power lasers operating at the technologically important and eye safe 1.5 $\mu$m spectral region.

## ACKNOWLEDGMENTS

This research was supported by JTO/ARO (W911NF-12-1-0330). Jiang and Lin acknowledge the support of Ed Whitacre and Linda Whitacre endowed chairs from AT&T Foundation.

[1]C. J. Chang-Hasnain, IEEE Commun. Mag. **41**, S30 (2003).
[2]K. Gerd, *Optical Fiber Communications* (Mc Graw Hill, Singapore, 1998).
[3]C. Ugolini, N. Nepal, J. Y. Lin, H. X. Jiang, and J. Zavada, Appl. Phys. Lett. **89**, 151903 (2006).
[4]R. Dahal, J. Y. Lin, H. X. Jiang, and J. M. Zavada, *Er-Doped GaN and InGaN for Optical Communications, Vol. 124, Topics in Applied Physics: Rare Earth Doped III-Nitrides for Optoelectronic and Spintronic Applications*, edited by K. O'Donnell and V. Dierolf (Springer, The Netherlands, 2010), pp. 116–157.
[5]H. Li and K. Iga, *Vertical-Cavity Surface-Emitting Laser Devices* (Springer, New York, 2002).
[6]G. Ghosh, Opt. Commun. **163**, 95 (1999).
[7]F. X. Wang, C. K. Hwangbo, B. Y. Jung, J. H. Lee, B. H. Park, and N. Y. Kim, Surf. Coat. Technol. **201**, 5367 (2007).
[8]K. Balasubramanian, X. F. Han, and K. H. Guenther, Appl. Opt. **32**, 5594 (1993).
[9]S. H. Jeong, J. K. Kim, B. S. Kim, S. H. Shim, and B. T. Lee, Vacuum **76**, 507 (2004).
[10]H. K. Jang *et al.*, J. Vacuum Sci. Technol. A **18**, 917 (2000).
[11]A. Y. Guo, Y. Y. Xue, X. M. Zhu, G. Y. Zhang, P. T. Guo, and X. F. Hu, J. Wuhan Univ. Technol. **21**, 101 (2006).
[12]W. Li, C. Ni, H. Lin, C. Huang, and S. I. Shah, J. Appl. Phys. **96**, 6663 (2004).
[13]S. Asbrink and A. Magneli, Acta Crystallogr. **12**, 575 (1959).
[14]K. K. Law, Opt. Commun. **108**, 8 (1994).
[15]J. P. Weber and S. Wang, Opt. Lett. **15**, 526 (1990).