GROWTH AND CHARACTERIZATION OF SIO₂ FILMS DEPOSITED BY FLAME HYDROLYSIS DEPOSITION SYSTEM FOR PHOTONIC DEVICE APPLICATION

J. P. Bange, L. S. Patil, and D. K. Gautam

Department of Electronics North Maharashtra University Post Box No. 80, Umavi Nagar, Jalgaon-425 001 (M.S.), India

Abstract—There are various techniques for the deposition of SiO_2 films on silicon. Flame Hydrolysis Deposition (FHD) techniques is the most economical technique for the deposition of SiO_2 films. In this technique the SiO_2 films are deposited by hydrolysis of $SiCl_4$ in a high temperature H_2 - O_2 flame. In the present study we present the growth of SiO₂ films by indigenously developed FHD system and organic compound Tetraethoxyorthosiliate/Tetraethoxysilane TEOS as source of silicon. The films deposited by the FHD system are porous and need annealing at higher temperatures for the densification. We present here for the first time direct dense glassy transparent SiO₂ films deposited by our FHD system. The optical properties of the deposited films were studied by ellipsometery. FTIR spectroscopy was carried out to study the various characteristic peaks of SiO_2 bonds. The peaks corresponding to Si-O-Si stretching, bending and rocking modes are observed at 1090 cm^{-1} , 812 cm^{-1} and 463 cm^{-1} respectively. The absence of peaks corresponding to the OH bond in the deposited film reveals that the deposited films are most suitable for the photonic devices application. The surface analysis was carried out using SEM. The EDAX of the deposited film confirms the composition of the Si and O in the deposited film.

1. INTRODUCTION

Next generation planar lightwave circuits (PLCs) will need circuits that have greater functionality and are larger in scale, but they must also be less expensive to fabricate. To achieve this, we must continue improving the waveguide fabrication process by carrying out computational analyses [1–11]. A PLC fabricated using a technique for fabrication of optical fiber can provide a high quality lightwave circuits on a substrate. This means that both PLC and optical fiber use the same silica based glass material, buried waveguide geometry and glass forming method. Various processes have been explored for the fabrication of silica based PLC. These are Chemical Vapor Deposition (CVD), Vacuum Deposition and Flame Hydrolysis Deposition (FHD) [12–20]. Each of these processes is used in combination with Reactive Ion Etching (RIE). Among these processes the combination of FHD and RIE has the advantage of being able to produce low-loss channel waveguides best matched to optical fiber [19].

It is known that the precise control of deposition and accurate compositional control is very difficult in this process. An additional process of densification of the porous soot into dense SiO_2 film is required in FHD process. However, despite the difficulties of the FHD process, it is extensively employed to deposit thick SiO_2 amorphous films since the deposition rate is fast and material quality is already proven in Vapor-Phase Axial Deposition (VAD) or Outside Vapor Deposition (OVD) processes for optical fibers [21].

We present here for the first time direct dense glassy transparent SiO_2 films deposition by indigenously developed FHD system. The design of the torch nozzle and other processing parameters are optimized in such a manner that the deposited films are transparent glassy films that do no need any further annealing. The effects of flow rate of TEOS (carrier gas nitrogen) on refractive index, thickness and absorbance have been studied. In the second section of the paper the experimental setup and processing parameters have been given. The results are discussed in the third section of the paper. The fourth section concludes the paper.

2. EXPERIMENTAL

Figure 1 shows the schematic of the experimental set used in the present study. The system has been developed indigenously at Department of electronics. Silicon wafers (p-100) used as substrate, were cleaned by Trichloroethylene, Acetone and Methanol for removal of contaminations. The FHD system was powered ON and the MFC were allowed to heat up for better performance. After some time the MFC of hydrogen and oxygen were set at 2.0 SLPM and 0.4 SLPM. Substrate temperature was kept constant at 700 °C throughout the process. The flame was ignited at the nozzle end and allowed to get stable. Then MFC of precursor TEOS (carrier gas nitrogen) was set to desired flow and then injected at the center of the flame. In the present study the flow of carrier gas was varied from 0.3–0.6 SLPM with a step

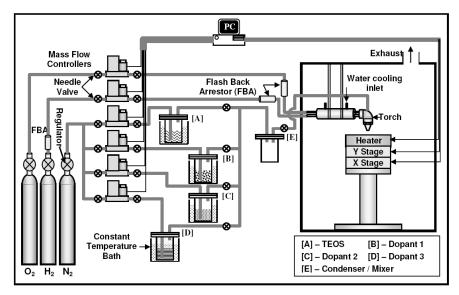


Figure 1. Schematic of the experimental set of Flame Hydrolysis Deposition system.

of 0.1 SLPM. The effects of flow rate of TEOS (carrier gas nitrogen) on refractive index, thickness and absorbance have been studied.

3. RESULTS AND DISCUSSIONS

Conventionally the SiCl₄ is used as a source of Silicon in FHD system for the deposition of SiO₂ films. The SiCl₄ is corrosive and moisture sensitive and produces HCl gas as one of its bi-products. Hence, we used the Organic compound TEOS (Tetraethoxyorthosiliate) as source of SiO₂ as this chemical is safe to handle and comparably less hazardous. The SiO₂ films deposited by using indigenously developed FHD system are tested for their optical, mechanical and chemical properties.

The effect of TEOS (carrier gas N_2) flow rate on refractive index (n) of the deposited SiO₂ film is presented graphically in Figure 2. Refractive index was measured by Philips SD-1000 Ellipsometer at fixed wavelength 632.8 nm.

It is observed from Figure 2 that the refractive index of the deposited films initially increases and then there is decrease in the refractive index. This decrease in refractive index may be due to porousivity of the film [22]. As the flow rate of TEOS increases

Bange, Patil, and Gautam

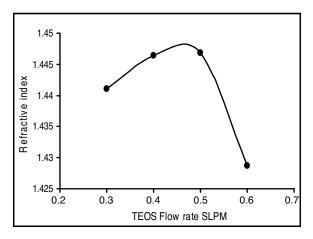


Figure 2. Effect of TEOS (carrier gas N_2) flow rate on refractive index of deposited SiO₂ films.

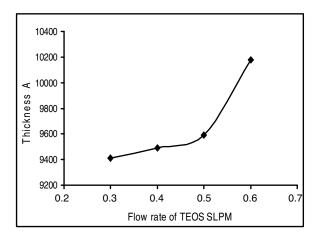


Figure 3. Effect of TEOS (carrier gas N_2) flow rate on thickness of deposited SiO₂ films.

the flame temperature decreases and hence, the sintering temperature decreases which leads to the low dense films. It is reported by various authors that the density of the film increases with increase in temperature [23–25]. The other reason of decrease in refractive index is normally due to the presence of OH bond in the deposited film but, the FTIR study (Figure 4) reveals that the peak due to OH bond is absent in the films deposited by present FHD system. The At% of Si and O

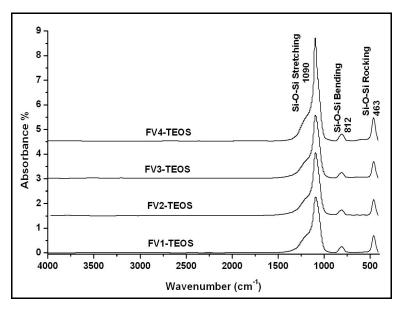


Figure 4. FTIR spectrum of SiO_2 films deposited at various TEOS flow rate.

in the deposited films also affects the properties of the deposited film. Hence, we can conclude that the decrease in the refractive index values of deposited films is mainly due to the increase in Oxygen contents as observed from the EDAX measurements (Figure 6).

The effect of TEOS (carrier gas N_2) flow rate on thickness of the deposited SiO₂ film is presented graphically in Figure 3. It is depicted from the figure that the thickness of the film increases with the corresponding increase in TEOS flow rate. It is obvious that concentration of reactant species increases with corresponding increase in flow rate of TEOS which leads to increase in growth rate of the deposited film. Hence, we can conclude that the thickness of the film increase with corresponding increase in flow rate of TEOS.

The IR spectrums of SiO_2 have three characteristic absorption bands arising from Si-O-Si groups [26–28]. The lowest frequency band is caused by the rocking mode corresponding to the out of plane motion of the oxygen atom. The weakest absorption of intermediate frequency is connected with bending vibrations in which the oxygen atom motion occurs in the Si-O-Si plane and along the Si-O-Si angle bisector. The stretching mode band is asymmetric with a broadening towards low frequencies [29], for thick SiO₂ a long high frequency tail is observed and the frequency corresponding to the maximum of this peak increases with increasing oxidation temperature [26]. The total area of the peaks also increases linearly with increasing film thickness. The band at 1056 cm^{-1} makes the main contribution to the absorption in the case of thin films, whereas the band at 1091 cm^{-1} dominates for thick films. This may cause the observed shift in the absorption maximum towards high frequencies as the oxide thickness increases [29] but no shift has been observed in the present study.

Figure 4 shows the FTIR absorption spectrums of SiO_2 films deposited by FHD system at various flow rates. The peak intensities due to Si-O-Si stretching, bending and rocking modes are observed at 1090 cm^{-1} , 812 cm^{-1} and 463 cm^{-1} respectively. The peak values well matches with the FHD and thermally grown oxides films as reported in literatures [30–36]. As the stretching peak is observed at 1090 cm^{-1} and area of the peak increases with the increase in flow rate, we can conclude that the thickness of the film increases with corresponding increase in TEOS flow rate as observed from ellipsometer results for thickness measurements.

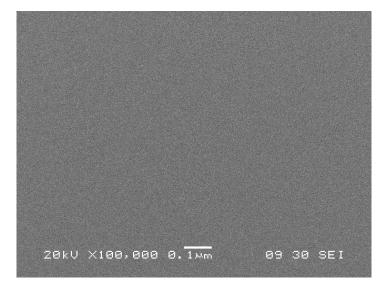


Figure 5. Scanning electron microphotograph of deposited SiO₂ films.

The peak area and peak height of the Si-O-Si stretching peaks for various flow rates of TEOS have been calculated. It can be observed that there is an increase in peak area and peak height with corresponding increase in flow rate. Hence we can conclude that the thickness of the film increase with increase in TEOS flow rate.

The surface morphology of the deposited films was studied using

170

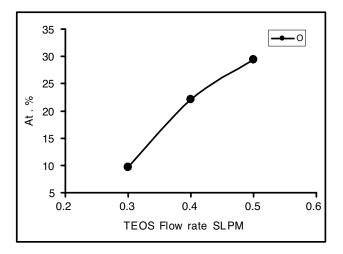


Figure 6. Effect of TEOS flow rate on oxygen percentage in deposited SiO_2 films.

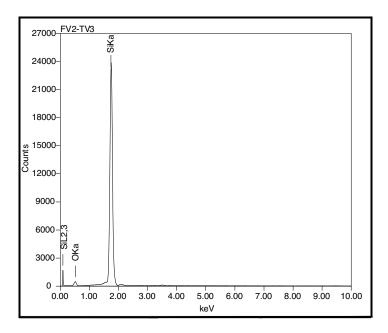


Figure 7. EDAX of SiO_2 film deposited by FHD (sample FV2-TEOS).

Scanning Electron Microscope (JEOL/EO make JSM-6360 model). The Figure 5 illustrate the scanning electron microphotograph of the sample deposited with substrate temperature 700 °C. It is clearly observed from the photographs that there is no trace of unsintered particle or pores in the deposited film. The films observed to be dense in nature.

The elemental analysis of deposited SiO_2 films for TEOS flow variations have been carried by EDAX technique. Figure 6 shows the plot of Oxygen percentage in the deposited films for TEOS flow variations.

From the plot it is clearly observed that the Oxygen count increases with corresponding increase in TEOS flow rate. Due to this increase in Oxygen count we can say that the refractive index of the deposited film decreases with increase in TEOS flow rate.

The EDAX of the sample FV2-TEOS is as show in Figure 7. From the EDAX we can confirm the deposition of SiO_2 film with no other impurities present in the deposited film.

4. CONCLUSIONS

The SiO₂ films grown by the indigenously developed Flame Hydrolysis Deposition system were found to be uniform. The refractive index of the deposited film varies with the corresponding increase in TEOS flow rate. The film thickness increases with the increase in flow rate. Absorbance spectra of the deposited films shows peak intensities due to Si-O-Si stretching, bending and rocking modes are observed at 1090 cm^{-1} , 812 cm^{-1} and 463 cm^{-1} respectively. The peaks values well match with the reported values. The absence of peaks corresponding to the OH bond in the deposited film reveals that the deposited films are most suitable for the photonic devices application. It is clearly observed from the SEM image that there is no trace of unsintered particle or porousivity in the deposited film. The film is observed to be dense in nature. The elemental analysis of deposited SiO₂ films for TEOS flow variations have been carried by EDAX technique confirms the deposition of SiO₂ film with no other impurities present in the film.

ACKNOWLEDGMENT

The authors wish to acknowledge the Department of Science and Technology, New Delhi for financial support to carry out the project.

REFERENCES

- 1. Abdalla, M. A. and Z. Hu, "On the study of left-handed coplanar waveguide coupler on ferrite substrate," *Progress In Electromagnetics Research Letters*, Vol. 1, 69–75, 2008.
- Yang, T., S. Song, H. Dong, and R. Ba, "Waveguide structures for generation of terahertz radiation by electro-optical process in GaAs and ZnGeP2 using 1.55 m fiber laser pulses," *Progress In Electromagnetics Research Letters*, Vol. 2, 95–102, 2008.
- 3. Wen, F. and B.-J. Wu, "Diffraction efficiency enhancement of guided optical waves by magnetostatic forward volume waves in the Yttrium-Iron-Garnet waveguide coated with perfect mental layers," *Progress In Electromagnetics Research B*, Vol. 1, 209–218, 2008.
- Sotoodeh, Z., B. Biglarbegian, F. H. Kashani and H. Ameri, "A novel bandpass waveguide filter structure on SIW technology," *Progress In Electromagnetics Research Letters*, Vol. 2, 141–148, 2008.
- Amjadi, S. M. and M. Soleimani, "Design of band-pass waveguide filter using frequency selective surfaces loaded with surface mount capacitors based on split-field update fdtd method," *Progress In Electromagnetics Research B*, Vol. 3, 271–281, 2008.
- Mondal, M. and A. Chakrabarty, "Resonant length calculation and radiation pattern synthesis of longitudinal slot antenna in rectangular waveguide," *Progress In Electromagnetics Research Letters*, Vol. 3, 187–195, 2008.
- Park, J. K., J. N. Lee, D. H. Shinan, and H. J. Eom, "A full-wave analysis of a coaxial waveguide slot bridge using the fourier transform technique," *J. of Electromagn. Waves and Appl.*, Vol. 20, No. 2, 143–158, 2006.
- Li, Y. Y., P. F. Gu, M. Y. Li, H. Yan, and X. Liu, "Research on the wide-angle and broadband 2D photonic crystal polarization splitter," *J. of Electromagn. Waves and Appl.*, Vol. 20, No. 2, 265–273, 2006.
- 9. El Sabbagh, M. A. and M. H. Bakr, "Analytical dielectric constant sensitivity of ridge waveguide filters," *J. of Electromagn. Waves* and Appl., Vol. 20, No. 3, 363–374, 2006.
- Maurya, S. N., V. Singh, B. Prasad, and S. P. Ojha, "Modal analysis and waveguide dispersion of an optical waveguide having a cross-section of the shape of a cardioid," *J. of Electromagn. Waves and Appl.*, Vol. 20, No. 8, 1021–1035, 2006.
- 11. Chang, H.-W. and W.-C. Cheng, "Analysis of dielectric waveguide

termination with tilted facets by analytic continuity method," J. of Electromagn. Waves and Appl., Vol. 21, No. 12, 1653–1662, 2007.

- 12. Kawachi, M., "Silica waveguides on silicon and their application to integrated optic components," *Optical and Quantum Elec.*, Vol. 22, 391, 1990.
- Valette, S., S. Renard, H. Denis, J. P. Jadot, A. Founier, P. Philippe, P. Gidon, and E. Desgranges, "Si-based integrated optics technologies," *Solid State Technol.*, Vol. 32, No. 2, 69, 1989.
- Henry, C. H., G. E. Blonder, and R. F. Kazarinov, "Glass waveguides on silicon for hybrid optical packaging," *J. Lightwave Technol.*, Vol. 7, 1530, 1989.
- Izawa, T., H. Mori, Y. Murakami, and N. Shimizu, "Deposited silica waveguide for integrated optical circuits," *Appl. Phys. Lett.*, Vol. 38, 483, 1981.
- Kawachi, M., M. Yasu, and T. Edahiro, "Fabrication of SiO₂-TiO₂ glass planar optical waveguides by Flame Hydrolysis Deposition," *Electro. Lett.*, Vol. 19, No. 5, 583, 1983.
- Kawachi, M., M. Yasu, and M. Kobayashi, "Flame Hydrolysis Deposition of SiO₂-TiO₂ glass planar optical waveguide on silicon," Jpn. J. Appl. Phys., Vol. 22, 1932, 1983.
- Nourshargh, N. A., E. M. Starr, and T. M. Ong, "Integrated optic 1 × 4 splitter in SiO/GeO₂," *Electron. Lett.*, Vol. 25, 981, 1989.
- Kashyap, R., B. J. Ainslie, and G. D. Maxwell, "Second harmonic generation in GeO₂ rigid waveguide," *Electron. Lett.*, Vol. 25, 206, 1989.
- Hickernell, F. S., "Optical waveguides on silicon," Solid State Technol., Vol. 31, No. 11, 83, 1988.
- Kim, Y. J. and D. W. Shin, "Compositional analysis of SiO₂ optical film fabricated by Flame Hydrolysis Deposition," *Journal of Ceramic Processing Research*, Vol. 3, No. 3, 186–191, 2002.
- Lucovsky, G., M. J. Manitini, J. K. Srivastava, and E. A. Irene, "Low temperature growth of silicon dioxide films: A study of chemical bonding by ellipsometry and infrared spectroscopy," J. Vac. Sci. Technol. B, Vol. 5, No. 2, 530, 1987.
- Rojas, S., et al., "Properties of silicon dioxide films prepared by low-pressure chemical vapor deposition from tetraethylorthosilicate," J. Vac. Sci. Technol. B, Vol. 8, No. 6, 1177–1184, Nov/Dec. 1990.
- 24. Becker, F. S., D. Pawlik, H. Anzinger, and A. Spitzer, "Lowpressure deposition of highquality SiO₂ films by pyrolisis of

tetraethylorthosilicate," J. Vac. Sci. Technol. B, Vol. 5, 1555, 1987.

- Sassela, A., "Tetrahedron model for the optical dielectric function of H-rich silicon oxynitride," *Phy. Rev. B*, Vol. 48, 14208, 1993.
- Gallener, F. L., "Band limits and the vibrational spectra of tetrahedral glasses," *Phy. Rev. B*, Vol. 19, 4292, 1979.
- Sen, P. N. and M. F. Thorpe, "Phonons in AX₂ glasses: From molecular to band like modes," *Phys. Rev. B*, Vol. 15, 4030, 1977.
- 28. Boyd, I. W., "Deconvolution of the infrared absorption peak of the vibrational stretching mode of silicon dioxide: Evidence for structural order?" *Appl. Phys. Lett.*, Vol. 51, 418, 1987.
- 29. Tolstoy, V. P., I. V. Chernysnova, and V. A. Skryshevsky, Handbook of Infrared Spectroscopy of Ultrathin Films, John Wiley & Sons Inc., NJ, 2003.
- 30. Kim, Y. T., S. M. Cho, Y. G. Seo, H. D. Yoon, Y. M. Im, and D. H. Yoon, "Influence of hydrogen on SiO₂ thick film deposited by PECVD and FHD for silica optical waveguide," *Cryst. Res. Technol.*, Vol. 37, No. 12, 1257–1263, 2002.
- Edahiro, T., M. Kawachi, S. Sudo, and S. Tomaru, "Deposition properties of high silica particles in the flame hydrolysis reaction for optical fiber fabrication," *Jap. J. Appl. Phys.*, Vol. 19, No. 11, 2047–2054, 1980.
- 32. Shin, H., J.-H. Yi, J.-G. Baek, and M. Choi, "Preperation and characterization of SiO₂-B₂O₃-P₂O₅ particles and films generated by flame hydrolysis deposition for planar lightwave circuits," J. Material Res., Vol. 17, No. 2, 315–322, 2002.
- Pliskin, W. A. and H. S. Lehman, "Structural evaluation of silicon oxide films," J. Electrochem Soc., Vol. 112, 1013, 1965.
- 34. Shirai, H. and R. Takeda, "Determination of thickness of thin thermal oxide layers on Czochralski grown silicon wafers from their longitudinal optical vibrational mode," *Jpn. J. Appl. Phys.*, Vol. 35, 3876, 1996.
- Devine, R. A. B., "Structural nature of the Si/SiO₂ interface through infrared spectroscopy," *Appl. Phys. Lett.*, Vol. 68, 3108, 1996.
- Bjorkman, C. H., T. Yamazaki, S. Miyazaki, and M. Hirose, "Analysis of infrared attenuated total reflection spectra from thin SiO₂ films on Si," J. Appl. Phys., Vol. 77, 313, 1995.