OPTOELECTRONICS AND ADVANCED MATERIALS – RAPID COMMUNICATIONS Vol. 4, No. 4, April 2010, p. 584 - 587

Effect of OMCTS flow rate on SiO_2 films grown by flame hydrolysis deposition

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In this present study the commonly used $SiCl_4$ have been replaced by organic compound Octamethylcyclotetrasiloxane (OMCTS). We present here for the first time direct dense glassy transparent SiO_2 films deposition by indigenously developed FHD system. The grown films were characterized by ellipsometery for the optical properties. 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 461 cm^{-1} respectively. The absence of peaks corresponding to the OH bond in the As-deposited films reveals that the deposited films are most suitable for the photonic devices application. The surface and elemental analysis was carried out using SEM with EDAX attachment.

(Received September 21, 2009; accepted April 11, 2010)

Keywords: Flame hydrolysis deposition, FHD, Octamethylcyclotetrasiloxane, Photonic devices, FTIR, SEM, EDAX

1. Introduction

Recent explosion of internet data traffic and other high speed communication needs have motivated world wide efforts to develop optical fiber communication networks that continue to increase its bandwidth. This has generated a strong demand on various component technologies such as lasers, amplifiers, detectors, modulators, attenuators, multiplexers, demultiplexers and filters [1]. In most cases they are built into individual and discrete optical fibers and multi-layer dielectric thin film. The cost of making these individual components is high due to extensively manual driven fabrication and packaging processes. A marriage of optical fiber fabrication technology and LSI microfabrication technology gave birth to fiber matched silica waveguides on silicon. Thick silica content glass is deposited on silicon by flame hydrolysis, a method originally developed for fiber preform fabrication. Silica channel waveguides are then formed by photolithographic pattern definition processes followed by reactive ion etching. This "high silica (HiS) technology" [2] offers the possibility of integrating a number of passive functions on a single silicon chip, as well as the possibility of the hybrid integration of both active and passive devices on silicon. The silica waveguide can be formed on crystalline silicon substrates by a combination of glass film deposition and etching. Their simple and clear core structures, low propagation loss and almost perfect field matching to optical fibers have lead to silica waveguides on silicon attracting increasing attention in recent years. Furthermore, as the base materials are Si and SiO_2 a variety of film deposition and etching processes in the field of Si-VLSI technology can be applied to extend the functions of integrated optic components.

Various deposition techniques have been explored for the fabrication of silica waveguides [3 - 6]. The two techniques in the development of silica on silicon technology include Chemical Vapor Deposition (CVD) and Flame Hydrolysis Deposition Technology. However, the CVD technique is having slow growth rate as compared to FHD. The FHD is an effective means of producing thick films at low production cost [7 – 10]. The Aerosol process is being used whenever high purity and fast growth rate are important such as in the fabrication of optical waveguides [11]. The films deposited by FHD are porous in nature it needs annealing at higher temperature for the densification of these films. During this annealing process there is high possibility of structural damage and movement of volatile dopants. 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. 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

Fig. 1 shows the schematic of the experimental setup 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.

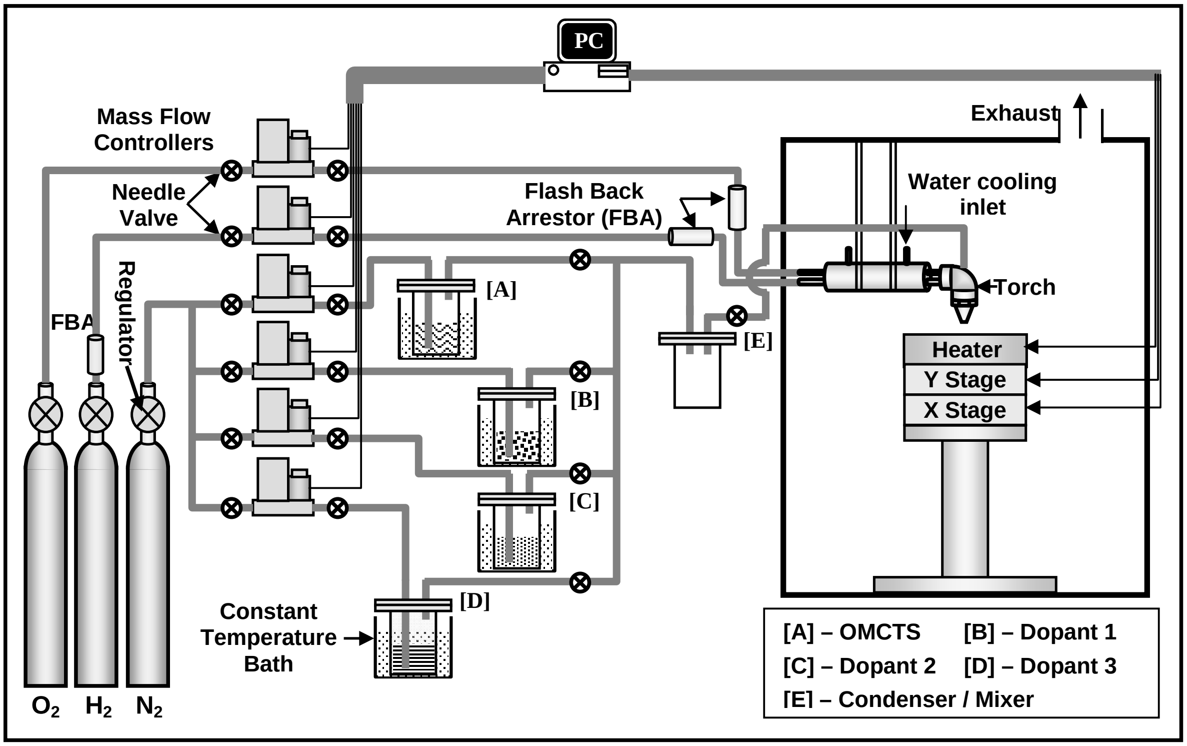

Fig. 1 Schematic of the experimental set of flame hydrolysis deposition system.

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. All the processes were carried out for 1.5 minutes duration. The flame was ignited at the nozzle end and allowed to get stable. Then MFC of precursor OMCTS (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 0.2 – 0.6 SLPM with a step of 0.1 SLPM. The effects of OMCTS (carrier gas nitrogen) flow rate on refractive index, thickness bond structure and surface morphology have been studied.

3. Results and discussion

Conventionally the $SiCl_4$ is used as a source of Silicon in FHD system for the deposition of SiO_2 films. The $SiCl_4$ is corrosive and moisture sensitive and produces HCl gas as one of its bi-products. Hence, we used the Organic compound OMCTS (Octamethylcyclotetrasiloxane) as source of SiO_2 as this chemical is safe to handle and comparably less hazardous. The SiO_2 films deposited by using indigenously developed FHD system are tested for their optical, mechanical and chemical properties.

From the Fig. 2, it is observed that there is a slight increase in refractive index from 1.4459 to 1.4468 with the increases in the OMCTS flow rate. The increase in the index of refraction in oxide films can be understood quantitatively in terms of the Lorenz-Lorentz (L-L) formalism where two factors contributes i) decrease in the mole volume as the Si contents is increased and ii) difference between the polarizations of Si-O and Si-Si bonds [12]. As the flow rate of the Silicon source material is increasing the possibility of increase in Si contents is more than the difference between the polarization of Si-O and Si-Si bonds.

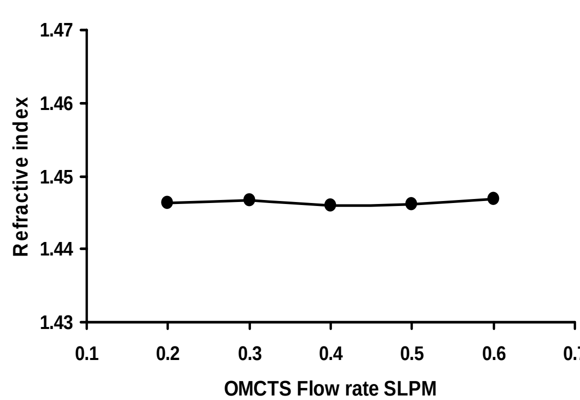

Fig. 2. Effect of OMCTS flow rate on refractive index of deposited SiO_2 films.

It is depicted from the Fig. 3 that the thickness of the deposited film increases from 9679Å to 9927 Å with the corresponding increase in the OMCTS flow rate. It is obvious that concentration of reactant species increases with corresponding increase in flow rate of OMCTS 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 OMCTS. This can also be observed form the absorbance spectrums given in Fig. 4.

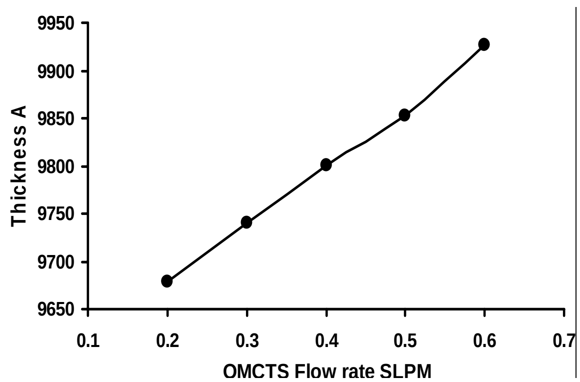

Fig. 3. Effect of OMCTS flow rate on thickness of deposited SiO_2 films.

The chemical bond analysis of the films was carried out using Thermo Nicolet 380 series FTIR. Fig. 4 shows the absorbance spectra of SiO_2 films grown by varying the flow rate of OMCTS and keeping the other process parameters constant. The peak intensities due to Si-O-Si stretching, bending and rocking modes are observed at 1090 cm^{-1}, 812 cm^{-1} and 461 cm^{-1} respectively. The peaks values well match with the reported values [13-16]. For the convenience the FTIR spectra's of individual flow of OMCTS have been stacked. Table 1 shows the peak intensities corresponding to the Si-O-Si stretching and rocking bonds.

Table 1. Peak intensity corresponding to the Si-O-Si stretching and rocking bonds.

Sample	Si-O-Si Stretching	Si-O-Si Rocking
FV1-OMCTS	1.508	0.5
FV2-OMCTS	1.834	0.6
FV3-OMCTS	1.902	0.67
FV4-OMCTS	2.061	0.72
FV5-OMCTS	2.294	0.89

The peak intensity of the Si-O-Si stretching bond increases from 1.508 to 2.294 with the corresponding increase in the OMCTS flow rate. An increase of peak intensity from 0.5 to 0.89 is observed in the Si-O-Si rocking peak. However there is a very small increase in the peak intensity of Si-O-Si bending with the corresponding increase in OMCTS flow rate. As the stretching peak is observed at 1090 cm^{-1} and area of the peak increases with the increase in flow rate, according to Tolstoy [17] we can conclude that the thickness of the film increases with corresponding increase in OMCTS flow rate as observed from ellipsometer results for thickness measurements.

From Fig. 4 we can also conclude that the peaks due to OH bonds are not present in the deposited films that are conventionally observed in the film deposited by FHD and other techniques. Therefore, the deposited film does not need further annealing for the removal of OH contents which affects the optical properties of SiO_2. However, the effect of annealing on the properties of the deposited films is further scope of study. The deposited film shows excellent chemical and optical properties suitable for their applications in optical devices.

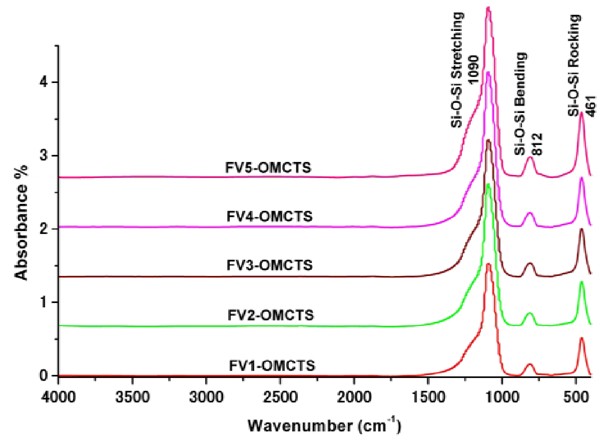

Fig. 4. FTIR spectrum of SiO_2 films deposited at various OMCTS flow rate.

The surface morphology of the deposited films was studied using Scanning Electron Microscope (JEOL/EO make JSM-6360 model). Fig. 5 illustrate the scanning electron microphotograph of the sample deposited with substrate temperature 700°C. It is clearly observed from the microphotographs that there is no trace of un-sintered particle or porousivity in the deposited film. The film is observed to be dense in nature. However, the films deposited without substrate temperature showed the traces of un-sintered particles. While the films deposited by FHD by Letian Zhang and others need annealing at higher temperature for the densification [2,10,18-21].

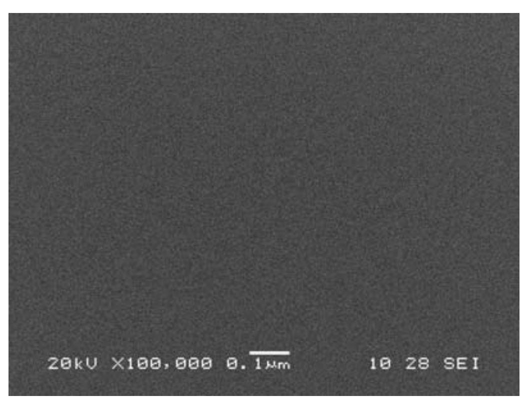

Fig. 5. Scanning electron microphotograph of the sample deposited with substrate temperature 700°C.

The elemental analysis of deposited SiO_2 films for OMCTS flow variations have been carried by EDAX technique. The EDAX of the sample FV1-OMCTS is as show in Fig. 6. From the EDAX we can confirm the deposition of SiO_2 film with no other impurities present in the film.

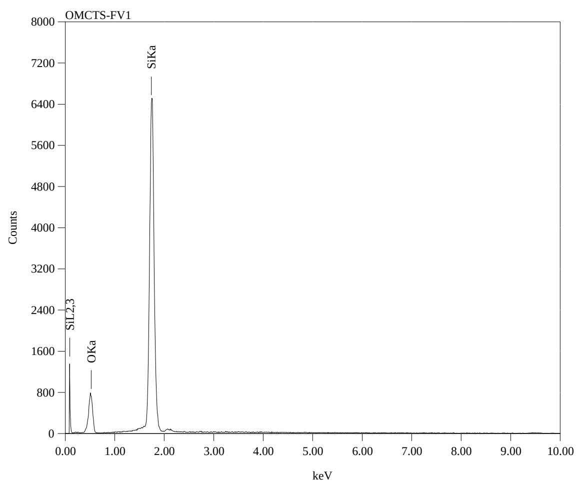

Fig. 6. EDAX of the sample FV1-OMCTS.

4. Conclusions

The SiO_2 films grown by the indigenously developed Flame Hydrolysis Deposition system were found to be uniform. The refractive index of the deposited film varies from 1.4459 to 1.4468 with the corresponding increase in OMCTS flow rate. The film thickness increases from 9679 Å to 9927Å 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 at 1090 cm^{-1}, 812 cm^{-1} and 461 cm^{-1} respectively. The peaks values well match with the reported values [13-16]. Peak intensity and area of Si-O-Si stretching peak are found to be increasing from 1.508 to 2.294. However the annealing of the samples at higher temperature will sharpen the peaks i.e. the films will be more dense as compared to the As-deposited samples. 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 SiO_2 films for OMCTS flow variations have been carried by EDAX technique, which confirms the deposition of SiO_2 film with no other impurities present in the film.

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