Deposition of Thick SiO_2 from Tetraethylorthosilicate and H_2O by Plasma-Enhanced CVD*

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Abstract: The deposition of silicon dioxide by plasma enhanced chemical vapor deposition from tetraethylorthosilicate (TEOS) and H_2O has been studied. Silicon oxide with refractive index of 1.453 has been obtained. Tests on the 51mm wafers show that both thickness uniformity of ±1.5% and constant refractive index of 1.453 can be achieved. By raising the deposition temperature, the qualities have been improved, while the deposition rate decreased. A SiO_2 thick film deposition technique has been developed combining TEOS-PECVD technique with high temperature annealing.

Key words: silicon dioxide; plasma-enhanced CVD; planar waveguide

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1 Introduction

Commercial interest in WDM components and its system increases rapidly. WDM provides a new dimension for telecommunication network to solve the problems in capacity and flexibility. WDM system includes some key components, such as optical crossconnet (OXC), optical add/drop multiplexer (OADM), and so on. The passive waveguide devices are used as switcher and waveguide grating multiplexer. Devices reported so far can be divided into silica-based devices and InP-based ones. The former ones, often with the fiber-matched (low-contrast) waveguide structure, have the advantages of both low propagation loss and high fiber-coupling efficiency. The core is chosen as 6μm×6μm in size and the cladding layer more than 15μm in thickness, so the waveguide can match the standard single-mode fiber mode. In another word, the deposition of SiO_2 on a layer thicker than 15μm is a key technique in the fabrication of Si-based waveguide devices[1-3].

Silicon dioxide is widely used in integrated circuits. An established method of oxide deposition is to oxidize SiH_4 by chemical vapor deposition. But it is difficult to deposit thick silicon dioxide via this method, so to explore new silicious compounds is imperative, among which, tetraethylorthosilicate (TEOS) is the most promising one. With the new

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compound, esp. TEOS, a variety of SiO_2 films have been obtained at atmospheric pressure or a low pressure.

The TEOS decomposition is written as follows[4]:

$$Si(OC_2H_5)_4 \rightarrow SiO_2 + H_2O + C_2H_4$$

Small amounts of organic radicals can be observed. Due to the TEOS molecule itself containing both silicon and oxygen, no additional oxygen is required in the TEOS pyrolysis to yield the stoichiometric SiO_2 films when the reactor temperature is above 600℃. At a lower deposition temperature, the reaction can be realized by adding some oxygen or low-pressure plasma[5—8]. However, adding oxygen will impair the film uniformity; while working in the low-pressure plasma, other films that contain SiO_x, hydroxyl groups, and carbon will yield simultaneously. These problems can be avoided by improving the design of plasma reactor or using H_2O.

The deposition of SiO_2 thick film is required in the fabrication of SiO_2 waveguide device. Plasma enhanced CVD-technique has been developed to deposit SiO_2 films, with TEOS and H_2O as sources. Combining TEOS-PECVD technique with high temperature annealing, we have developed another SiO_2 thick film deposition technique, with which, a 15μm-thick SiO_2 film has been successfully deposited on the Si substrate.

2 Experiment

The deposition was performed in a low-pressure reaction chamber with diameter of 300mm. The setup is shown in Fig. 1. Two liquid sources, TEOS and H_2O, were used during the SiO_2 film deposition. The quantity of TEOS vapor introduced into the reactor was controlled by changing the bubbler temperature or flow through the TEOS. To improve the stability of this process and eliminate the TEOS decomposition products, the bubbler had to be evacuated for several minutes before each run. The pressure in the chamber was monitored by a pressure sensor and automatically adjusted with a butterfly valve. To eliminate the depletion effects, an abundance of TEOS vapor was necessary. As only a fraction of vapor was actually consumed to build the layers on the wafers, a particle trap was needed to prevent the glassy reaction products from reaching the pumps, as could be achieved by using a specially designed mechanical filter with a large inner surface. The deposition was carried out in the plasma at the temperature ranging between 150℃ and 350℃ and the pressure between 100Pa and 300Pa.

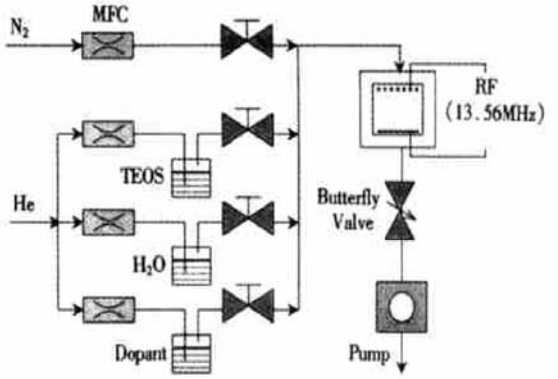

FIG. 1 Setup of TEOS-Source PECVE SiO_2 can be deposited from TEOS and H_2O in plasma. He is used as carrier gas. Reaction pressure is automatically adjusted.

After deposition, the films were characterized by measuring their refractive index and thickness (ellipsometer).

The variation in the film thickness across a wafer was defined as follows:

$$\text{Variation}(\pm\%) = 100 \times (d_{max} - d_{min})/(d_{max} + d_{min})$$

where d_{max} is the maximum film thickness measured in the wafer center, while d_{min} is the minimum measured at 5mm from the 50mm wafer edge. To test the purity of the films, secondary ion mass spectroscopy measurement should be taken on some samples.

3 Characterization and Results

The thickness of the film must be carefully chosen so that the waveguides can support the fundamental mode at a certain wavelength, such as

1.55μm. The deposition rate is quite important, especially for the SiO_2 planar waveguide layers deposition, in which the thickness of SiO_2 film are usually required to be more than 10μm. Figure 2 shows the dependence of deposition rate on temperature. The deposition rate of SiO_2 film decreases with the temperature in the range of 150℃ to 350℃. The SiO_2 deposition from TEOS and H_2O proceeds as following two steps:

1. Hydrolization: $Si(OC_2H_5)_4 + 4H_2O \rightarrow Si(OH)_4 + 4C_2H_5OH$,
2. Condensation: $Si(OH)_4 \rightarrow SiO_2 + 2H_2O$.

In the plasma, the initial hydrolization of TEOS is carried out and yields some middle products, which are absorbed by the surface of the substrate. Then some of the middle products will further condense on the heated substrate, depositing the pure SiO_2 film on the substrate; and some of them will depart from the surface of the substrate, resulting in the decrease in the deposition rate at a higher deposition temperature.

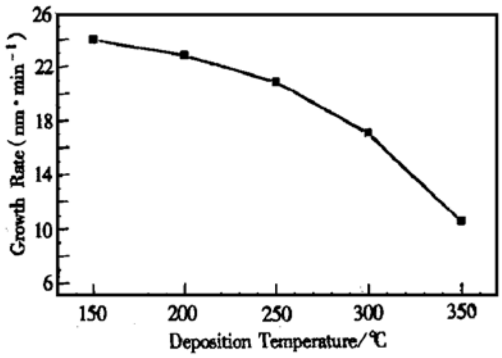

FIG. 2 Dependence of Deposition Rate on Temperature The RF power is 200W; the chamber pressure is 150Pa.

During the deposition of SiO_2 film by TEOS-sources CVD, some radicals such as SiO, SiOH, SiOC and C-H may emerge because of the incomplete reaction, which will impair the film's characteristics, including the optic properties (refractive index, optical transmission loss, etc.) and resistance to corrosion. From the temperature dependence of the refractive index shown in Figure 3, a conclusion is drawn that the temperature of 250℃ is a trade-off between high-deposition rate and good quality marked by the refractive index. When it is higher than 250℃, the constant refractive index of the films about 1.453 will not be influenced by the temperature; while when it is below 230℃, the refractive index will decrease so that some radicals will remain in the SiO_2 film owing to the incomplete reaction of TEOS.

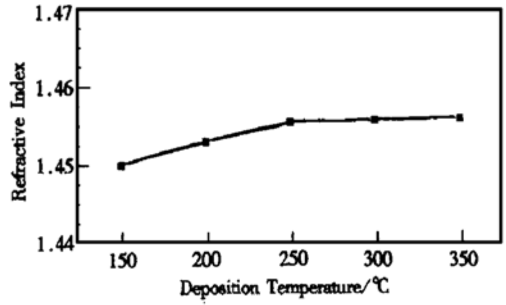

FIG. 3 Dependence of Refractive Index on Deposition Temperature The refractive index of the films is almost a constant of 1.453 when the deposition temperature is higher than 250℃.

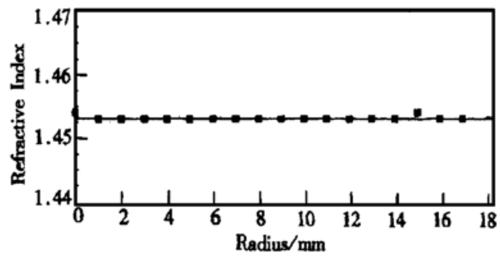

FIG. 4 Uniformity of Refractive Index Across 51mm Wafer The film has been deposited at 250℃ for 10min.

The purity of SiO_2 film deposited at 250℃ was analyzed by using a secondary ion mass spectroscopy. The result shows the film is basically composed of Si and O elements. At 250℃, SiO_2 film of good quality can be deposited by using this TEOS-source CVD system, whose refractive index is proved to be in agreement with the constant of 1.453 at the deposition temperature more than 250℃.

The uniformity of deposited SiO_2 film is important, including the uniformity of both the refractive index and the thickness. SiO_2 films used in the planar waveguide integrated devices are required totally uniform. The measurement of refractive index and thickness of SiO_2 film deposited on a 50mm wafer is taken by using an ellipsometer. The refractive indexes are in the range of 1.453 ± 0.001, which has no apparent change across a 51mm wafer (shown in Fig. 4). The refractive index measurement of SiO_2 film is restricted to the precision of the ellipsometer. The SiO_2 film in the center is thicker than that at the edge. The variation in thickness is less than $\pm 1.5\%$, as is shown in Fig. 5.

Combining TEOS-PECVD technique with

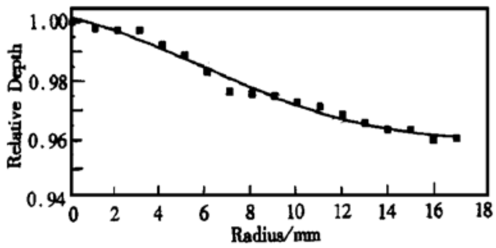

FIG. 5 Thickness Uniformity Across 51mm Wafer The film has been deposited at 250℃ for 10min.

high-temperature annealing, we have developed a SiO_2 thick film deposition technique, with which, a 15-μm-thick SiO_2 film has been successfully deposited on the Si substrate. The cross-section of thick SiO_2 is shown in the Fig. 6. The deposition of SiO_2 thick film is often used in the fabrication of SiO_2-based waveguide devices.

4 Conclusion

Silicon oxide film has been prepared by plasma enhanced CVD from TEOS and H_2O, which shows excellent refractive index uniformity (~ 1.453) and thickness uniformity. The variation in thickness across 51mm wafer is less than $\pm 1.5\%$. The deposition rate decreases with the substrate temperature. The silicon oxide films at the deposition temperature of 250℃ have good quality; while films with better quality can be obtained at a higher temperature. Combining TEOS-PECVD technique with annealing, we have developed a SiO_2 thick film deposition technique, with which, a 15μm-thickness SiO_2 film has been successfully deposited on the Si substrate.

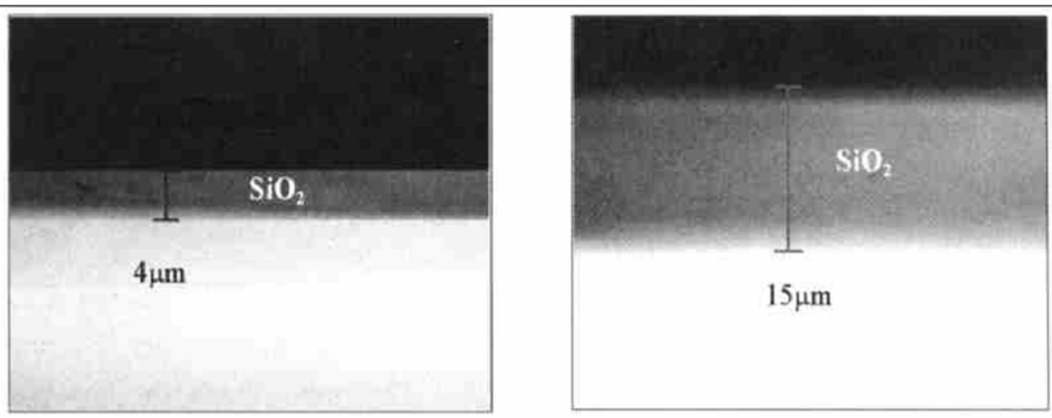

FIG. 6 Cross-Section of Thick SiO_2 Deposited from TEOS and H_2O by Plasma-Enhanced CVD

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采用 TEOS 和 H_2O 源 PECVD 方法生长氧化硅厚膜*

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摘要: 开展了使用 TEOS 和 H_2O 混合物进行 PECVD 生长 SiO_2 膜的研究工作. 氧化硅折射率分布在 1.453±0.001 的范围, 且随偏离中心距离基本不变. 薄膜厚度是中央大, 边沿薄, 其厚度相对变化不超过±1.5% (51mm 衬底). 利用 TEOS 源 PECVD, 并结合退火技术, 摸索出厚膜氧化硅生长工艺, 已成功地在硅衬底上生长出厚度超过 15μm 氧化硅厚膜, 可用于制备氧化硅平面波导器件.

关键词: 二氧化硅; PECVD; 平面波导
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