

# Deposition of beta-SiC thin films on Si (100) substrates by MOCVD method for NSOM applications

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## ABSTRACT

Silicon carbide thin films were deposited on Si(100) substrates by metal-organic chemical vapor deposition(MOCVD) in high vacuum condition( $2.0 \times 10^{-7}$  Torr) using 1,3-disilabutane as a single source precursor which contains silicon and carbide in 1:1 ratio at various temperature in the range of 700~1000 °C. Also, silicon carbide thin films were deposited on Si (100) substrate at deposition pressure between  $5.0 \times 10^{-6}$  Torr and  $1.0 \times 10^{-6}$  Torr without carrier and bubble gas. The XPS result shows that the SiC thin film grown at 950 °C which have carbon rich for silicon and carbon at 1:1.2 ratios. XRD result shows that the SiC thin film grown at 900 °C which appeared at  $2\theta = 41.6^\circ$  for SiC (200) reflection at a large intensity and a single shape diffraction peak. SEM images result show that the SiC thin film grown at 900 °C which has influence on the small grain size and single crystallinity. AFM images result show that the SiC thin film has smooth surface at RMS = 20nm.

In this paper, we fabricated the small aperture for the better performance such as less noise, higher resonant frequencies and fast imaging. We will apply that silicon carbide thin film has smooth surface on NSOM application.

Therefore, based on experimental results from X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), atomic force microscope (AFM) the best epitaxial SiC thin films were grown at 900 °C and  $2.0 \times 10^{-6}$  Torr.

**Keywords:** SiC, MOCVD, NSOM

## 1 INTRODUCTION

In recent years, nanotechnology has become widely applied in the life sciences, electronics, and optoelectronics, where it has come to play an increasingly important role. Silicon carbide (SiC) is a material with promising potential for application in nanotechnology [1, 2]. Single crystalline SiC thin films have attracted such interest for their use in electronic and optoelectronic devices, as well as circuits designed to operate at high powers, high frequencies, high temperatures, and high radiation environments due to their exceptional electrical and mechanical characteristics, ranging from electron mobility ( $1000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ ), electron saturation velocity ( $2.0 \sim 2.7 \times 10^7 \text{ cm} \cdot \text{s}^{-1}$ ), and breakdown electric field ( $2 \sim 3 \times 10^6$ ), to high melting point and high

thermal conductivity. In particular, SiC has a wide band gap of 2.2 eV for the 3C ( $\beta$ )-SiC, at room temperature, which has been exploited for SiC-Si heterojunction bipolar transistors [3-6].

SiC-based electronic devices and sensors are currently used in automobiles and modern aircrafts to control engines and monitor emissions in harsh environmental conditions. Most of these devices are fabricated using epitaxial layers grown by chemical vapor deposition (CVD), which is the most suitable epitaxial growth technique for mass production. Thick epitaxial layers with very low defect densities and good electronic properties are especially needed for fabricating high voltage devices [7, 8].

AFM cantilevers must satisfy the following conditions in order to achieve and maintain high resolution: (1) low spring constant; (2) high resonant frequency; (3) radius of curvature is small with a sharp tip; (4) small opening angle. Considerable interest exists in fabricating a sub-wavelength aperture for a near-field optical sensor given its potential application for promising near-field optical recordings and other biological applications. There has been a dramatic increase in storage density to as high as  $\sim 100 \text{ Gbyte/in}^2$  in near-field optical recording given its ability to circumvent the diffraction limit. However, the low optical generated through the tip of the fiber probe limits the scanning speed and hinders the development of the optical storage device [9-12].

Herein, we attempt to achieve high quality SiC thin films deposited on Si (100) substrates, using a single precursor, 1,3-disilabutane (1,3-DSB), without a carrier or bubble gas. A small aperture was fabricated using silicon nitride for improved performance through less noise, higher resonant frequencies, and faster imaging. It is our intent to then manufacture small size apertures that can be applied to NSOM by a SiC thin film using the same method.

## 2 EXPERIMENT

The etching of the Si using alkaline solutions such as KOH, ethylenediamine/pyrocatechol (EDP), and tetramethylammonium hydroxide (TMAH), is anisotropic due to the different atomic density of the Si crystal surface. The etch ratio of the Si(100) surface to the Si(111) surface was on the order of a few hundred with the intersection of the Si(111) surfaces eventually forming a V-type groove or pyramidal shape at the bottom Si(100) surface [13]. The oxidation rate is dependent upon both the crystal plane and

the angle of the plane intersection and because of the difference in atomic packing density, the Si(111) surface will have an oxidation rate higher than the Si(100) surface. Due either to the stress-induced retardation of oxidation or volume expansion of the oxide at a concave surface during thermal oxidation [14, 15], inner surfaces of the V-groove or hollow pyramid will be non-uniformly oxidized during an appropriate thermal oxidation procedure, whereby the oxide layer at the bottom or apex of the hollow pyramid is thinner than the oxide at side surfaces. Isotropic oxide etching technique using aqueous HF has been employed to fabricate nano-size apertures at the apexes of the oxide pyramids [16]. Before the fabrication process, the silicon wafer was cleaned using a standard. SEM is mainly used for checking the entire fabrication processes.

In addition, the epitaxial silicon carbide thin films were grown in a homemade, thermal high vacuum metal organic chemical vapor deposition system. The p-Si (100) wafer was cut into rectangle pieces of  $28 \times 8 \text{ mm}^2$  and used as substrates for growth of the silicon carbide films in this work. Prior to the growth, the substrate surface was degreased in an ultrasonic cleaner containing acetone for 10 min, followed by a DI water wash for 10 min, then dipped in 10 wt% aqueous HF for 15 s, rinsed in DI water, and finally flushed in a nitrogen flow. The substrates were resistively heated by a DC power supply and the temperature of the silicon substrates measured with an optical pyrometer through a 4.5 in viewport in the chamber wall. The general deposition conditions were a high vacuum pressure ( $1.0 \times 10^{-7} \sim 5.0 \times 10^{-6}$  Torr) and various temperatures ( $700 \sim 1000 \text{ }^\circ\text{C}$ ).

1,3-Disilabutane was employed as a single source for the growth of the silicon carbide films. 1,3-DSB has some advantages over the conventional single sources, namely that it already possesses a Si-C bond and does not require further activation to make a Si-C bond in the film. Therefore, the deposition can be conducted without a carrier or bubbler gas for a liquid phase precursor at room temperature. 1,3-DSB was transferred into a precursor bottle, attached to deposition chamber, and further purified by freeze-pump-thaw cycles using liquid nitrogen. 3C-SiC films were deposited directly on the clean silicon (100) surface without carbonization. The deposition time for the Si(100) substrates continued throughout various hours with the growth rate dependant upon the experimental conditions. In this work, different deposition conditions were used.

### 3 RESULTS AND DISCUSSION

Figure 1 shows a schematic diagram of the basic fabrication process applied in this study for making pyramidal cantilevers and nano-aperture arrays from  $\text{Si}_3\text{N}_4$  and  $\text{SiO}_2$  thin films grown on Si(100) wafers with a thickness of  $500 \text{ }\mu\text{m}$ . To produce cantilevers with integrated tips, the required seven steps are as follows: (a) growth of  $\text{Si}_3\text{N}_4$  and  $\text{SiO}_2$  thin films on silicon wafers; (b) formation of dot array patterns by photolithography; (c) formation of

concave, pyramidal grooves by chemical etching using a TMAH (20 wt%) solution at  $80 \text{ }^\circ\text{C}$  for 8 h; (d) a re-oxidation (stress-induced retarded oxidation) process; (e) a back-side residual silicon etching process by chemical etching using a TMAH (20 wt%) solution; (f) formation of the concave, pyramidal oxide tip; and (g) formation of nano-size holes utilizing both plasma etching (MERIE dry etching) and HF chemical etching.

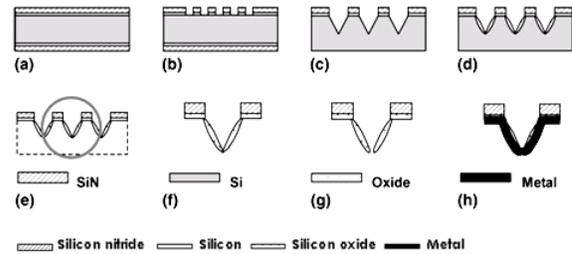


Figure 1: Fabrication procedure for pyramidal-shaped cantilevers and nano-aperture arrays.

Next, isotropic oxide etching using either 10:1 or 50:1 water:HF, was employed to open oxide apertures at pyramidal apexes, as in Fig. 2(a-b). Figure 2(a) shows an SEM image of an aperture opened by aqueous HF, as well as its dimensions ( $600 \text{ nm}$  thickness and  $120 \text{ nm}$  diameter) using the MERIE method, while Fig. 2(b) shows an SEM image of a  $267 \text{ nm}$  hole formed on the top of pyramid-tip using aqueous 50:1 HF solution. Though very careful treatment is required because of the thin oxide at the edges of the oxide tetrahedron, we have successfully brought the procedure under control for the tested arrays by adapting an aqueous 50:1 HF solution for etching rather than dry etching.

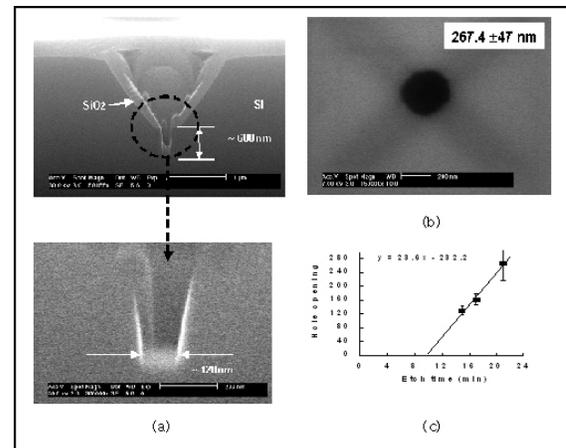


Figure 2: (a) SEM image of an aperture opened by aqueous HF and dimensions using the MERIE method. (b) SEM image of a  $267 \text{ nm}$  hole formed on top of the pyramid-tip using 50:1 aqueous HF. (c) Variations in diameters of apertures as a function of HF etch time.

From a series of repeated experiments, the progression of aperture opening within the span of the etch time has been observed, with the aperture diameter increasing linearly with etch time, Fig. 2(c). Subsequent experimentation on the oxide nano-aperture array samples with the aqueous HF etching solution was performed to study the etched shape and to control the size of hole. Briefly, after immersion of the pyramidal tip array samples into the 50:1 aqueous HF, the tip was placed into D.I water for 30 min and then dried at room temperature. Inspection of the size of the dried samples using SEM revealed 15 apertures for each 4 corners, totaling 60 apertures in the 1, inspected sample. When inspecting the hole of the aperture, width, length, and 2 diagonals in the hole were measured. Through these inspections, an increasing, linear graph with a 23.6 nm/min opening rate as a function of time was developed, Fig. 2(c). Additional experimentation revealed the hole-opening rate to be between 20.2 ~ 24.0 nm/min, on average.

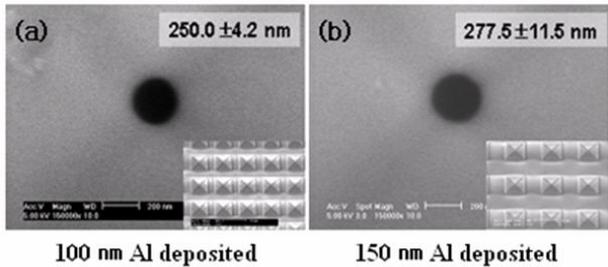


Figure 3: SEM images present the controllability of hole diameter using Al deposition thickness.

We progressed a series of Al depositions on pyramidal tips 50 nm thick using a metal sputter deposition method. After depositing Al at a thickness of 50 nm, the diameter of the hole was reduced from an initial hole diameter of 250 and 277 nm to 100 and 150 nm, Fig. 3. With these nano-aperture arrays, we investigated far-field diffraction patterns in order to ascertain possible light resonance-tunneling phenomena.

Figure 4(a) shows a schematic diagram of the basic fabrication process applied in this study for making cantilevers and nano-aperture arrays from SiC and SiO<sub>2</sub> thin films, grown on Si(100) wafers with a thickness of 500 μm.

Figure 4(b) shows the SEM image of the SiC cantilever deposited on Si using 1,3-disilabutane as a single source precursor at 900 °C and 2.0 × 10<sup>6</sup> Torr, a basic study for microelectromechanical systems (MEMS) and NSOM applications. SiC thin films deposited on Si cantilevers have smooth surfaces (RMS = 37.7 nm), a very important factor in the AFM cantilever application. SiC deposited on the Si cantilever has many advantages, ranging from a low spring constant and a high resonance frequency to small opening angles of Si<sub>3</sub>N<sub>4</sub>, and all are used in both AFM cantilevers and NSOM aperture arrays.

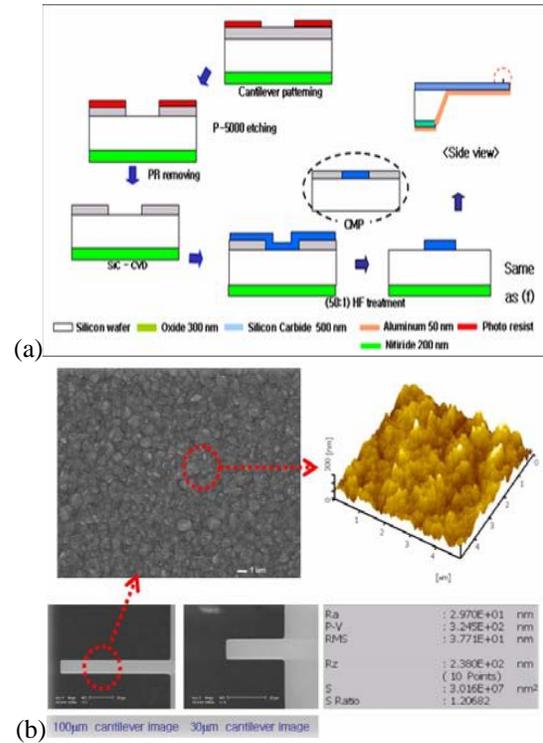


Figure 4: (a) Fabrication procedure of the SiC AFM cantilever, and (b) SEM and AFM images of the SiC cantilever.

## 4 CONCLUSION

The sub-wavelength size silicon oxide aperture array as a near-field optical probe was fabricated in order to examine the possible light resonance-tunneling phenomenon. Various semiconductor fabrication processes, including anisotropic Si etching using 20 wt% TMAH alkaline solution and isotropic HF etching technique, were used to open the nano-size pyramidal oxide apertures. HF etching time proved to be an effective control parameter for the aperture-opening rate. In this experiment, we found the oxide etch rate to be dependant upon etch time. In addition, deposition thickness of metals such as Al was also found to be a control parameter for the aperture-closing rate. The optimum deposition conditions for the epitaxial SiC films were a pressure of 2.0 × 10<sup>-6</sup> Torr, for 2 h, at 900 °C. Finally, SiC thin films deposited on Si cantilevers are predicted to outperform AFM cantilevers made using Si<sub>3</sub>N<sub>4</sub> films.

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