



Self assembled micro masking effect in the fabrication of SiC nanopillars by ICP-RIE dry etching

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ABSTRACT

This report presents the results of the novel fabrication of 4H-SiC pillars with nanopores using ICP-RIE dry etching. Cl₂/Ar gas plasma with various mass flow rates was used in this etching process to produce SiC nanopillars without using patterned etch mask. Cylindrical pillars of 300 nm diameter and 500 nm height with smooth side walls were etched on SiC wafer. The etching condition for the optimized fabrication of SiC nanopillars is presented in this report. Each nanopillar has been produced with a nanosize pore at the center along its length and up to the middle of the cylindrical nanopillar; it is a unique feature has not ever been reported in case of SiC. Inclusion of oxygen was found influence the formation of nanopillars by the effect of SiO₂ micro masking. The formation of self assembled SiO₂ layer and its micro masking effect in the fabrication of this unique nanostructure has been investigated using TEM, STEM and EDAX measurements.

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1. Introduction

Silicon Carbide (SiC) has recently been attractive material for electronic devices and micro-electromechanical systems (MEMS) [1] due to its properties such as wide band gap, high thermal conductivity, and large break down fields, high saturated electron drift velocity and its ability to withstand extreme environmental changes [2–7]. Nanostructures of SiC have attracted a lot of research interests due to their unique shape-induced electrical and optical properties [8,9]. It is an important class of heteronanostructures due to their properties, which is superior to carbon or Si based nanostructures. Nanostructured layers could offer potential advantages in nanoscale photonic and electronic devices. SiC is a good blue luminescence material, nanowires and nanorods of which are considered to be excellent candidates for field emission devices [10–12]. Several groups have reported the preparation of SiC nanostructures using catalyst assisted growth methods at high temperatures [13–15].

However, the high growth temperature limits the integration of the current growth method with semiconductor fabrication technology, which usually requires growth temperatures below 500 °C [16]. Etching the epitaxial thin films is a common practice for the fabrication of vertically aligned nanostructures at low temperature.

This technique can also be used to produce a high density of nanostructures with well-controlled dimensions and interesting optical properties that can be easily integrated into devices. As the bond energy between Si and C is high, it has a high chemical resistance for wet etching, hence a practical approach for patterning SiC is to use plasma based dry etching. The dry etching of patterned planar layer is a well-known catalyst free technique commonly used for the fabrication of semiconductor nanostructures. Using this top-down approach, nano structures such as nanostripes and nanopillars of various compound semiconductors can be grown either by direct etching using a focused ion beam (FIB) [17] or etching through a combination of lithography and large area etching procedures such as reactive ion beam, inductively coupled plasma (ICP) [18,19]. Inductively coupled plasma (ICP) RIE has several advantages compared to the conventional RIE, such as high etch rate, vertical profile, clean and low-damage surfaces [20].

Most of the available reports about the ICP-RIE based dry etching have used SF₆ based gas mixtures [21,22]. The damage induced by fluorine-based plasma is high; these damages could not be fully annealed out even at very high temperatures. Whereas the Cl₂/Ar plasma has been reported to cause little damages despite its poor etching rate; moreover this small amount of Cl₂/Ar plasma-induced damage can be annealed out at rather low temperatures. All the dry etching techniques used to produce nanostructures require a lithographically patterned etch mask, but in our experiment we obtained the nanopillar structure of SiC by ICP-RIE etching without using patterned etch mask. Self assembled silicon based

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oxide layers are believed to be the cause for the micromasking effect to form such etch patterns. The study of the self assembled nanometer-sized metal or semiconductor islands has recently become particularly importance for such etching and formation of nano structures [23]. These micro masking effects are becoming a recent interest in the fabrication of nanostructure.

The most interesting feature of this maskless etching is the formation of the pore structured pillars that is each pillar has been produced as a hollow structure having a pore centrally along its length. Hollow spherical and cylindrical nanostructures have sparked enormous interest in the sensor applications. These nanostructures have large surface area and it helps to have cavity-confined nanoscale reactions and transport processes, which have particular importance in bio technology for the applications of drug delivery, gene delivery, gene therapy, biosensors [24]. To our knowledge, no report is available on the fabrication of SiC nanopillars without using patterned nano mask. In this report we present the experimental results on the novel fabrication of SiC nanopillars using mask-free Cl_2 based ICP-RIE etching technique.

2. Experimental details

The 4H-SiC wafer samples used in this study were purchased from Cree Inc. Etching of the SiC wafers was performed using ICP-RIE system (KVICP – T4083) operated at 13.56 MHz. Chamber pressure was maintained at 15 mTorr under a gas mixture of Cl_2/Ar . Different samples were etched by varying ICP power (300–500 W), gas flow rates and etching time keeping the chamber pressure constant (15 mTorr). The etchant gases Cl_2 and Ar gases were introduced into the reactor chamber through independent electronic mass flow controllers (MFCs) that can control the flow rate of each gas with an accuracy of about 1 standard cubic centimeter per minute (sccm). An automatic pressure controller (APC) is placed near the exhaust end of the chamber to control the chamber pressure. The wafer surface was prepared by immersion in trichloroethylene for 3 min and rinsing in acetone, isopropyl alcohol and D.I water. In order to identify the etched region from un-etched portion a PR AZ 1512 mask was produced. Oxygen was intentionally introduced into the reaction chamber to study its effects on the etching of SiC. This intentional use of oxygen has resulted in the formation of pillar like etching pattern.

After the etching process, the etching depth was measured by stylus profilometer. The dimension and composition of the nanopillars obtained by etching studies were estimated by Hitachi S-4800 field emission scanning electron microscope (FESEM) equipped with energy dispersive X-ray (EDX) analysis operated at 15.0 kV. XE 100 (PSIA) Atomic force microscopy (AFM) was used to investigate the surface morphology and to estimate the density of the nanopillars. The structure of the nanopillars was also analyzed by transmission electron microscope (TEM), cross section of the nanopillars and its elemental composition was analysed using focused ion beam and STM, and these results are discussed in detail.

3. Result and analysis

Etching of SiC wafer was conducted under various gas mixture pressures, ICP source powers, and chamber pressures with constant rf power (100 W). After the etching process, the dimension, density and morphology of the nanopillars were examined by the FESEM, TEM and AFM. The cross sectional view of the pillar was also obtained by FIB and STM. Etching of the samples with gas mixture of Cl_2/Ar 12/3 and 13/2, ICP source power 300–500 W and chamber pressure 15 mTorr has been found to result in the formation of nanopillars of almost uniform size. The height of the nanopillars was found increasing depending on the etching time. The etching

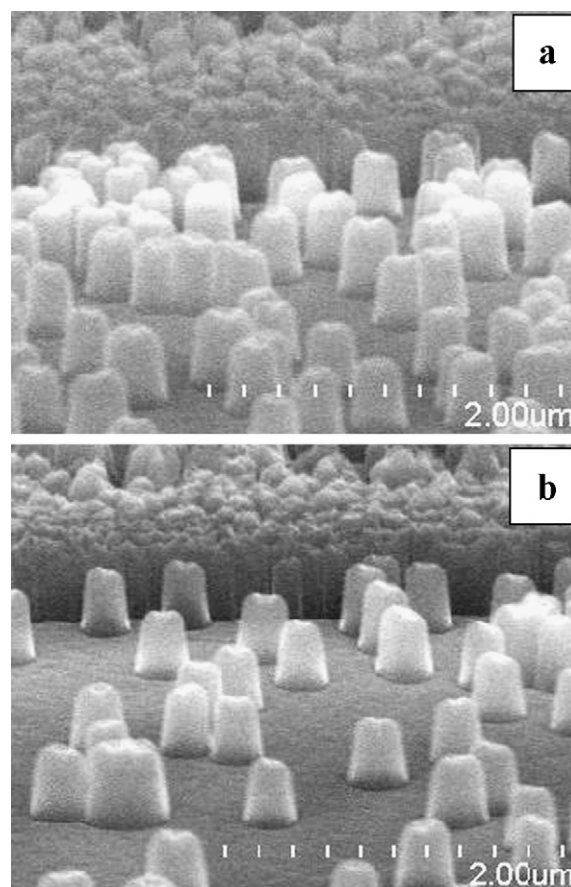


Fig. 1. SEM image of the SiC nanopillars etched with the concentration of O_2 (a) 15% and (b) 20%.

rate was also found to change with time; it was about 10 nm and 40 nm for the etching times of 5 min and 20 min respectively. Both ICP power and flow rates influenced the rate of etching, and also it has been found that the use of O_2 gas has produced pillars. The concentration of O_2 in the plasma gas has influenced the density of the pillars. Fig. 1(a) and (b) shows the samples etched with O_2 concentration 15% and 20% respectively. The increase of ICP power has resulted in lower density of SiC pillars for a particular concentration of O_2 . When the chamber pressure was reduced below 15 mTorr, no pillar formation was obtained. The Fig. 2 shows the etched sample of SiC with the chamber pressure 12 mTorr. It is not presented

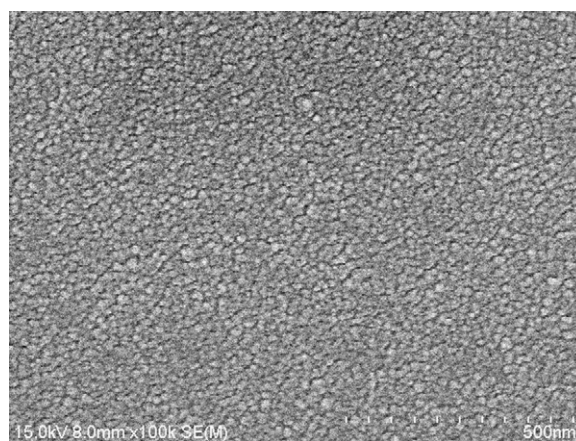


Fig. 2. SEM image of the SiC sample etched with chamber pressure 12 mTorr.

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