Deep reactive ion etching of in situ boron doped LPCVD Ge$_{0.7}$Si$_{0.3}$ using SF$_6$ and O$_2$ plasma

S.N.R. Kazmi *, C. Salm, J. Schmitz

MESA+ Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands

A R T I C L E   I N F O

Article history:
Available online xxxx

Keywords:
Deep reactive ion etching
Ge$_{0.7}$Si$_{0.3}$
Etch selectivity
Resonator

A B S T R A C T

This paper reports on deep reactive ion etching (DRIE) of in situ highly boron doped low pressure chemical vapor deposited Ge$_{0.7}$Si$_{0.3}$ alloy in SF$_6$ and O$_2$ plasma. The effect of RF power, SF$_6$ flow, O$_2$ flow and temperature on the etch rate of Ge$_{0.7}$Si$_{0.3}$ films with a boron concentration of $2.1 \times 10^{21}$ atoms/cm$^3$ is investigated. Optimized conditions for a combination of a vertical etch profile and a high selectivity towards PECVD oxide are reported. The effect of boron doping concentration on the etch rate is also investigated. The etch rate is found to decrease with an increase in the dopant concentration. The developed SF$_6$ and O$_2$ based DRIE recipes are applied to fabricate GeSi microresonators.

© 2013 Elsevier B.V. All rights reserved.

1. Introduction

Germanium–silicon (GeSi) alloys have received much attention over the past few decades due to a range of applications; for instance, in heterojunction bipolar transistors [1], MOSFET gates [2] and source/drain regions [3], solar cells [4] and thin film transistors [5]. The application of these layers in CMOS-MEMS post-processing is currently under study [6,7]. Anisotropic etching of the GeSi alloy is necessary in most of these applications.

Dry etching techniques are commonly applied in microtechnology to precisely transfer the desired pattern with a great degree of anisotropy. A myriad of halogen containing plasma chemistries has been used to demonstrate the dry etching of un-doped GeSi [8–11]. However, to our knowledge, no literature regarding deep etching of un-doped GeSi is available; although the experience with highly doped silicon provides a good starting point [12].

In this article, the DRIE of in situ highly boron doped Ge$_{0.7}$Si$_{0.3}$ layers is described. A mixture of SF$_6$ and O$_2$ is chosen for its proven ability to control the anisotropy, as observed in silicon etching [13]. The primary aim of this work is to achieve a vertical etch profile and a good selectivity, at least 50:1 towards SiO$_2$. We further studied the etch rate as a function of boron concentration in the Ge$_{0.7}$Si$_{0.3}$ layer. The optimized etch recipes are applied in the fabrication of micromechanical resonators.

2. Experimental

The starting material were 100-mm (100) oriented Si wafers covered with 100 nm SiO$_2$ deposited by PECVD (plasma-enhanced chemical vapor deposition). On top of this a 1.5 µm thick in situ boron doped Ge$_{0.7}$Si$_{0.3}$ films was deposited by means of LPCVD (low-pressure chemical vapor deposition) using SiH$_4$, GeH$_4$, Ar and B$_2$H$_6$ as gaseous precursors. The Ge$_{0.7}$Si$_{0.3}$ films were deposited at a temperature of 430 °C and total pressure of 0.2 mbar. The heavily doped films used in this work have a boron concentration of $2.1 \times 10^{21}$ atoms/cm$^3$ as determined by SIMS. For comparison also the etch rate is studied for un-doped films and in-situ-doped GeSi layers with a lower boron concentration.

In this case the boron concentration in the films was varied by changing the ratio of the partial pressures of the gases, keeping the Si to Ge ratio in the deposited films constant within 2%. More information on the fabrication of the samples can be found in Ref. [14]. Before patterning, the wafers were cleaned in 99% fuming nitric acid for 5 min followed by deionized water rinse and nitrogen drying. The patterning was done by means of optical lithography after spinning and prebaking 1.2 µm Olin 907-12 photoresist at 95 °C for 1 min (without post-bake). The GeSi native oxide was removed by dipping the samples in 1% HF immediately before loading into the etcher to avoid etch inhibition [15].

All etch experiments were performed in a Plasmalab100 Plus system from Oxford Instruments, having a quartz dome, as shown in Fig. 1. The vacuum chamber has a process window of 6–33 $\times 10^{-3}$ mbar pressure and 10–200 sccm flow. The reactor has two power controlled plasma sources. The first is an inductively coupled plasma (ICP) source having a helicoil design to create a high radial and ion density of maximum 1200 W at 13.56 MHz. The second is a capacitively coupled CCP source of maximum 300 W also at 13.56 MHz; it can be used to direct the ions from the plasma glow region towards the wafer surface. The wafer was mechanically clamped against a liquid nitrogen cooled electrode, with helium backside cooling to allow optimum tempera-
tecture control over the entire wafer surface. The gases were injected from the top into the process chamber through a gas inlet.

Prior to performing the etching experiments the chamber is cleaned with a 20 min O₂ plasma at 20 °C, to minimize the etch variability [16,17]. The gas flows and process pressure were allowed to stabilize for 5 min before the etching was performed.

The chamber pressure, helium backside pressure and CCP power were kept fixed at 13 × 10⁻³ mbar, 20 mbar and 10 W, respectively. At the low CCP power of 10 W, corresponding to a self bias of ~20 eV, a high selectivity to the mask is guaranteed. To optimize the GeSi etch the CCP power was varied from 0 to 800 W, the SF₆ flow was varied between 0 and 200 sccm, the O₂ flow was varied between 2 and 20 sccm and the chuck temperature ranged between −110 and 20 °C. In all figures the O₂ flow is excluding the approximately 10% additional oxygen due to the quartz dome. The effects of loading and selectivity towards of photoresist were not studied in this work.

The etch depth and hence the etch rate of highly doped Ge₀.₇Si₀.₃ was determined using a Dektak 8.0 surface profilometer after removing the photoresist. The etch profile was evaluated from cross-section scanning electron microscopic (SEM) images.

Secondary Ion Mass Spectroscopy (SIMS) was carried out to determine the boron concentration in the deposited Ge₀.₇Si₀.₃ layers. The SIMS analysis was performed using 3 keV O⁺ primary ions in positive mode.

3. Results and discussion

The chamber pressure of 13 × 10⁻³ mbar yields a relatively large mean free path of the reactive species while passing through the dark sheath, nearly without collision, as reported in [18]. The ionic species bombard the wafer surface perpendicularly, a demand for anisotropic etching [19].

The CCP power was set to 10 W for all experiments, as lower power values led to a steep decrease in etch rate. Similarly, the etch rate increases with the RF power on the ICP source, as shown in Fig. 2. The etch rate seems to saturate with ICP power above 500 W under these process conditions (see the figure caption). The observed saturation is due to the sheath layer in the chamber [20], and depends on the plasma density and hence, the SF₆ flow.

The effect of the SF₆ and O₂ flows is subsequently investigated with ICP RF power fixed at 500 W. Fig. 3 shows a sublinear monotonic increase in the etch rate with an increase in SF₆ flow for chuck temperatures of −75 °C and −90 °C. The etch rate is observed to increase very slowly beyond an SF₆ flow of 100 sccm.

The dependence of etch rate on oxygen flow is shown in Fig. 4. The etch rate at −75 °C shows an initial increase with oxygen flow which is attributed to the correlated increase in fluorine atom concentration [10–12,21]. A further increase in O₂ flow results in a decreased etch rate due to the dilution of reactive species beyond

Fig. 1. Schematic layout of the Oxford Plasmalab 100 Plus system.

Fig. 2. Etch rate as a function of ICP power with T = −90 °C, 100 sccm SF₆ flow, and 5 sccm O₂ flow.

Fig. 3. Etch rate as a function of SF₆ flow with 5 sccm O₂ flow and 500 W ICP power.

Fig. 4. Etch rate versus O₂ flow with 100 sccm SF₆ flow and 500 W ICP power.
10 sccm; a similar trend is observed during the etching of Si [21]. The initial increase in etch rate is hardly visible in the 

experiments.

The etch profile changes from a slightly negative taper at low oxygen flow, to positive taper as the oxygen flow is increased to 20 sccm; see Fig. 5 (for the 90°C chuck temperature). The process

parameters as detailed in Table 1 (first row) results in a vertical etch profile of highly doped Ge0.7Si0.3 alloy.

Fig. 6 shows the etch rate between −110°C and 20°C for a 20:1 SF6:O2 plasma at 13 × 10⁻³ mbar and CCP power of 500 W. The observed increase in etch rate with the increase of temperature is due to enhanced adsorption and reactivity of fluorine atoms with the etched material during DRIE, as also reported for Si etching [19].

### Table 1

<table>
<thead>
<tr>
<th>SF6 (sccm)</th>
<th>O2 (sccm)</th>
<th>ICP (watt)</th>
<th>CCP (watt)</th>
<th>Pressure (mTorr)</th>
<th>He (mbar)</th>
<th>Temp. (°C)</th>
<th>Selectivity (GeSi:SiO2)</th>
<th>Etch angle (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>5</td>
<td>500</td>
<td>10</td>
<td>10</td>
<td>20</td>
<td>−90</td>
<td>35:1</td>
<td>90</td>
</tr>
<tr>
<td>100</td>
<td>5</td>
<td>500</td>
<td>10</td>
<td>10</td>
<td>20</td>
<td>−75</td>
<td>65:1</td>
<td>82</td>
</tr>
<tr>
<td>100</td>
<td>5</td>
<td>500</td>
<td>10</td>
<td>10</td>
<td>20</td>
<td>20</td>
<td>92:1</td>
<td>59</td>
</tr>
</tbody>
</table>

Fig. 5. Etch profiles for O2 flow of (a) 2.5 sccm, (b) 5 sccm, (c) 15 sccm and (d) 20 sccm with 100 sccm SF6 flow, 100 sccm O2 flow, and 500 W ICP power.

Fig. 6. GeSi etch rate as a function of chuck temperature with 100 sccm SF6 flow, 5 sccm O2 flow, and 500 W ICP power.

Fig. 7. Etch rate as a function of boron concentration in Ge0.7Si0.3 alloy with T = −90°C, 5 sccm O2 flow, 100 sccm SF6 flow, and 500 W ICP power.
The etch selectivity towards plasma deposited SiO₂ is measured to be 35:1 at −90 °C, and even 65:1 at −75 °C chuck temperature. We find that the etch selectivity towards oxide increases further to 92:1 at 20 °C, but at the expense of an irregular etch profile due to the deposition of eroded photoresist on the side wall, as also reported for Si at 0 °C [19].

Fig. 7 shows the etch rate of Ge₀.7Si₀.3 alloy with varied boron concentration at process conditions indicated in the caption. The boron doped layers (6.9 × 10²⁰ cm⁻³) etch four times slower than the phosphorus doped silicon wafer (≈1 × 10¹⁵ cm⁻³). The etch rate is found to decrease with an increase in the boron concentration. This decrease in the etch rate is caused due to increasing Coulomb repulsion between uncompensated boron (B⁺) and fluorine ions (F⁻), as explained by the space charge model of Lee et al. [22]. The obtained results are in line with the suppressed etch rate reported for p⁺ doped silicon [23].

4. Micromachined GeSi resonators

With the optimized etch conditions, as listed in Table 1, poly Ge₀.7Si₀.3 micro-electromechanical resonators are fabricated, as shown in Fig. 8. Both the input/output electrodes, and the resonating structure with its anchors, are realized with GeSi material. The bottom layer (forming the resonating element) is etched at −90 °C, chosen for a vertical etch profile. The upper layer, defining the electrodes, is etched at a temperature of −75 °C for the higher selectivity towards the intermediate, thin silicon dioxide layer between the two Ge₀.7Si₀.3 layers. The fabricated resonator is fully functional, with Lamé mode resonance around 48 MHz [23].

5. Conclusions

We have reported on the reactive ion etching of highly boron doped Ge₀.7Si₀.3 layers in SF₆ and O₂ plasma using a Plasmalab 100 Plus system. The effects of RF power, SF₆ flow, O₂ flow and temperature on the etch rate in SF₆ and O₂ plasma are presented. The etch rate is strongly affected by the chuck temperature, less by the flow of SF₆ and O₂. The etch profile can be controlled with the oxygen concentration in the gas mixture. Selectivity towards PECVD silicon dioxide is 35:1 up to 92:1 at chuck temperatures of −90 °C and 20 °C respectively. The developed etch recipes are successfully employed in the fabrication of GeSi microresonators.

Acknowledgements

The authors would like to thank A. A. I. Aarnink and M. J. de Boer for their technical support. We also gratefully acknowledge I. -J. Holsema and M. A. Smithers from MESA+ Institute for Nanotechnology for their help in the cleanroom and SEM imaging, respectively. This work is financially supported by the Dutch Technology Foundation STW through project Grant No. 10048.

References