

Dry Etching of Si/SiGe Multiple Stacks: Process Control by Optical Emission Spectroscopy and Surface Analysis of Etched Films

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Dry etching of Si/SiGe multiple stacks has been investigated by optical emission spectroscopy. In this way an etch stop within any of the thin films of the stacks can be achieved. Surface analysis shows a slight Ge enrichment at the etched SiGe alloy relative to its bulk concentration. The chemically shifted intensity of the Ge $2p$ core level is an indication of chlorinated Ge at the SiGe surface after reactive ion etching.

1. Introduction

Recently, Si/SiGe heterostructures have become the subject of a great deal of interest for novel electronic and optoelectronic device applications such as heterojunction bipolar transistors, quantum well devices, and infrared superlattice detectors [1-4]. As a result, there has been considerable interest in structure fabrication and transport studies in the Si/SiGe system.

In defining different devices in Si/SiGe, reactive ion etching (RIE) plays an important role in the process of pattern transfer. Such fabrication processes often require to make contact to thin buried SiGe films. Making contact to such buried regions requires etching of Si and stopping in the thin SiGe layer. Consequently, for a precise pattern transfer a method of controllable etching is demanded. Unfortunately, selective RIE techniques for SiGe have not yet been discovered. Luckily, this lack of SiGe-to-Si and Si-to-SiGe etch selectivity was overcome using optical emission spectroscopy (OES) [5]. OES is an effective and accurate technique for monitoring RIE of Si/SiGe heterostructures. It allows an exact determined etch stop in a thin buried SiGe alloy.

But the solution of this technological problem entails an additional formulation of question: How does the RIE process with etch stop on the SiGe layer influence the properties of this alloy film? If, as a result of reactive ion etching significant enrichment or depletion of Ge at the surface occurs or the RIE process leads to an evident

enhancement of microroughness, the device performance of such alloys could be degraded.

Therefore, we have investigated the dry etching mechanism of epitaxial Si/SiGe multiple stacks in $\text{Cl}_2/\text{SiCl}_4/\text{N}_2$ gas mixture. In particular, the surfaces of such etched stacks have been studied by different diagnostic techniques such as Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS).

2. Experimental

Monitoring of patterning was demonstrated using two different sample structures consisting of SiGe/Si/SiGe/Si and SiGe/SiGe/SiGe/Si multiple stacks, respectively. The SiGe layers were grown by rapid thermal chemical vapour deposition (RTCVD) using $\text{SiH}_4/\text{GeH}_4/\text{H}_2$ in a single wafer radiation heated stainless steel reactor at a deposition temperature of 500 °C and a pressure of 200 Pa.

The RIE experiments were carried out in a conventional reactive ion etching system (*SENTECH* SI 591). Prior to RIE, the background pressure is below 10^{-4} Pa. The 13.56 MHz rf-powered aluminium cathode (diameter = 250 mm) is covered with a quartz plate on which the wafer is placed. The applied power was 250 W (0.5 W/cm^2) and the chamber pressure was 20 Pa. From experiences with Si RIE a gas mixture $\text{Cl}_2/\text{SiCl}_4/\text{N}_2$ with the following flow rates was chosen: Cl_2 - 8 sccm, SiCl_4 - 35 sccm, N_2 - 50 sccm. Chlorine was used because it has been shown to produce vertical side walls due to the ion assisted etching mechanism. The addition of SiCl_4 contributes to a better control of trench formation by simultaneous redesorption. During all experiments described in this paper flow rates, power and pressure were not varied.

The experiments were done in two stages: (1) RIE of Si/SiGe multiple stacks with a precise process control by OES, and (2) investigations of composition and extent of surface reaction layers formed as a result of dry etching with etch stop in a buried SiGe film of the stack.

3. Results and discussion

During the deposition of the Si/SiGe multiple stacks by RTCVD reflection supported interferometry (with the commercial system PYRITE-RS [6]) has been applied to evaluate the thickness and optical parameters of each of the films deposited. The first line in Table 1 shows the data (thickness, Ge content) obtained by this in situ measurement procedure.

Table 1: Comparison of film thickness d and Ge content x obtained by in situ and ex situ measurements

layer sequence	SiGe		Si		SiGe		Si
	d/nm	x/%	d/nm	x/%	d/nm	x/%	d/nm
PYRITTE	30,4	20	68,4		46	20	74
OES	~30		~60		~40		~70
layer sequence	SiGe		SiGe		SiGe		Si
	d/nm	x/%	d/nm	x/%	d/nm	x/%	d/nm
PYRITTE	35	22	80	16	35	22	50
OES	~35		~65		~35		~60
AES	33	24	67	16	~33	25	~60

A main object of the RIE experiments was to prove OES is a powerful diagnostic technique suitable for use in dry etch processing of such SiGe/Si structures. Due to the high intensity the atomic emission lines of Ge at 265 nm were chosen for process monitoring.

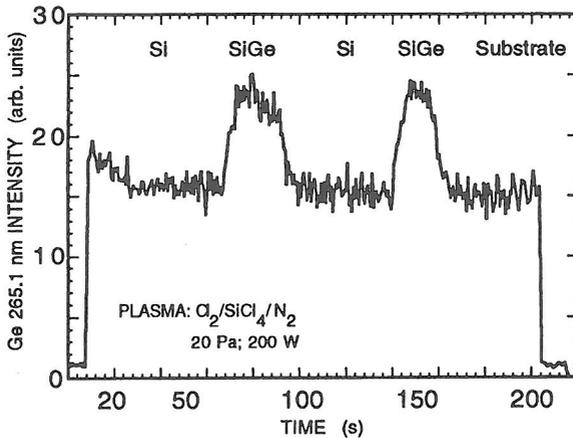


Fig. 1. Emission intensity of 265 nm Ge spectral line as function of time during RIE for a SiGe/Si/SiGe/Si stack

Fig. 1 illustrates the etching evolution of the first sample structure. About 60 seconds after plasma ignition, in which the removal of native oxide film and the silicon cap takes place the intensity of the 265 nm Ge emission lines observed rises from its base line. It is approximately constant during the first SiGe layer etching and decreases to its initial value if the RIE process continues into the silicon interlayer. Etching of the second buried SiGe film is characterized by a repeated increase of Ge emission line

intensity. The OES trace provides exact knowledge of all stages of the etching process in the different layers.

The etch rate of SiGe is a function of the Ge content and increases with increasing Ge content. This etch rate increase is consistent with the nearly 6 times greater etch rate of elemental Ge as compared to that of Si. The knowledge of the etch rates under these special plasma conditions [5] allows for given Ge contents (from the PYRITTE measurements) an estimation of the separate film thicknesses, also listed in Table 1.

Also in the second case, the OES trace at 265 nm for Ge during the etch process reflects the layer structure with different Ge contents exactly. There is a very good conformity of the OES profile with the AES depth profile measured (Fig. 2 and Table 1).

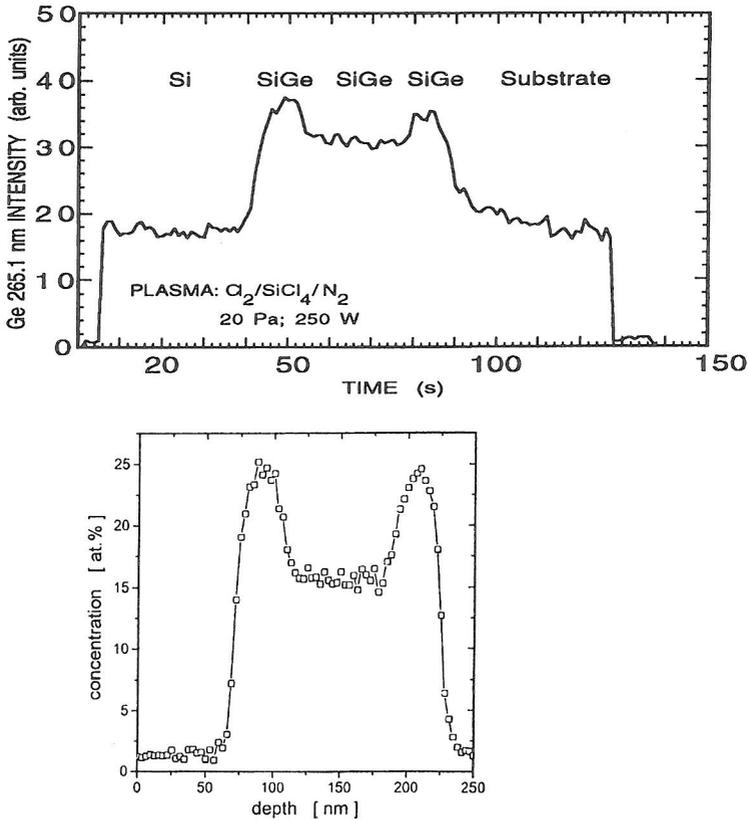


Fig. 2. OES trace at 265 nm (Ge) during RIE for a SiGe/SiGe/SiGe/Si stack (above) and the corresponding AES profile (below)

For the second objective of these RIE investigations, at some samples the etch process was stopped in the upper buried SiGe layer ($x \approx 22\%Ge$). To investigate the influence of the RIE process on prospective surface modifications AES measurements were done. The AES depth profiles illustrate a slight increased surface Ge concentration relative to its bulk value (Fig. 3).

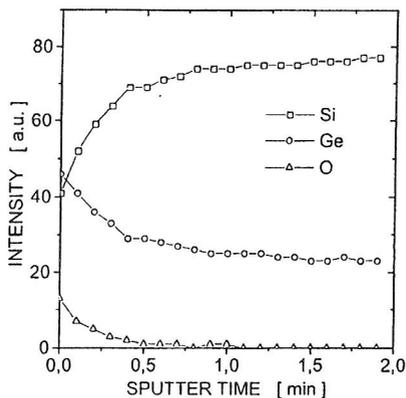


Fig. 3. AES depth profiles of a SiGe film after RIE

To understand this small Ge enrichment etched surfaces have been analysed by XPS and were compared with adequate unetched samples. The measurements were carried out using AlK_{α} radiation. The spectra for the Si $2p$ core level and the Ge $2p$ are depicted in Fig. 4. The shape of the Si peak shows only small differences related to the unetched control sample. The latter produces a single asymmetric peak centred at 99.7 eV, which is due to Si-Si and Si-Ge bonding. After RIE the Si $2p$ peak is slightly reduced in intensity, but there is no evidence of any chlorinated Si. The spectrum below illustrates the Ge $2p$ peak from unetched sample, centred at a binding energy of 1217.9 eV and is attributed to Ge-Si and possible Ge-Ge bonding. The spectra show chemically shifted Ge $2p$ core level intensity at higher binding energy. There is an overall shift in peak position of about 0.8 eV relative to the control sample. The explanation for this appears to be that the residual chlorine is bonded to Ge and the SiGe surface is riched in Ge. This result is in agreement with the AES studies.

There are some possible reasons for such an enrichment: The presence of several percent Ge in the SiGe alloy significantly increases the rate of Si atom volatilization as compared to rates measured for pure Si. In addition, the higher volatility of $SiCl_n$ (as compared to $GeCl_n$ [7]) should lead to a faster removal of this compound and results in a slight Ge enrichment at the surface. Moreover, the oxidation of the highly reactive SiGe surface after finishing the RIE procedure or during the air contact before the AES measurements can also cause a Ge enrichment beneath the thin oxide layer.

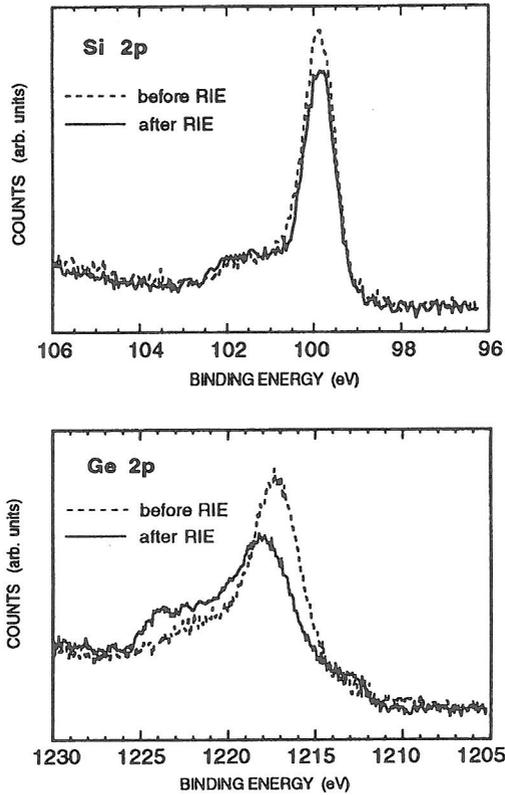


Fig. 4. Si $2p$ and Ge $2p$ photoelectron spectra of SiGe samples before and after RIE

References

- [1] K. L. Wang and R. P. G. Karunasiri, *J. Vac. Sci. Technol. B* **11**, 1159 (1993).
- [2] G. Abstreiter, *Solid State Communications* **92**, 5 (1994).
- [3] U. König, *Microelectronic Engineering* **23**, 3 (1994).
- [4] F. Schäffler, *Solid-State Electronics* **37**, 765 (1994).
- [5] H. H. Richter, A. Wolff, B. Tillack, and T. Skaloud, *Mat. Sci. Eng. B27*, 39 (1994).
- [6] G. Ritter, B. Tillack, M. Weidner, P. Zaumseil, F. G. Böbel, B. Hertel, and H. Möller, *J. Cryst. Growth* **146**, 119 (1995).
- [7] *Handbook of Chemistry and Physics*, edited by D. R. Lide, CRC Press, Boca Raton, 1992.