Selective and Anisotropic Dry Etching of Ge over Si

Marcelo S. B. Castro[1], Sebastien Barnola[2] and Barbara Glück[3]

[1] Laboratório de Optrônica e Sensores, Centro Tecnológico do Exército, Rio de Janeiro, Brazil
[2,3] CEA, LETI, MINATEC, Grenoble, France
e-mail: marcelobortolini@uol.com.br

ABSTRACT

Inductively coupled plasma (ICP) etching of Ge with high selectivity over Si and anisotropic etched profiles using CF_4, HBr, SF_6, and Cl_2 reactive gases has been studied. Because pressure and biased power should be the most important parameters to drive selectivity and etch profile, they were varied from 4 to 50 mTorr and from 0 to 50 W, respectively, so as to investigate their influence on process. Total gas flow (100 sccm) and source power (350 W) were initially held constant. Selectivity greater than 100:1 of Ge over Si was achieved using 100 % Cl_2 etch gas at 50 mTorr and zero bias power but the profile of the etched features was isotropic. With the addition of N_2 to the feed gas (Cl_2) the profile became more anisotropic. A three steps ICP etch process was developed with a final Ge/Si etch selectivity of 5:1 and anisotropic profiles.

Index Terms: plasma etching, germanium, silicon, selectivity, etch profile.

I. INTRODUCTION

Germanium (Ge) and Silicon (Si) are basic materials in the fabrication of microelectronic devices such as infrared photodetectors [1, 2], sacrificial layers for structures like silicon on nothing (SON) [3], and bipolar heterojunction transistors [4]. In this work a process has been developed to selectively etch thick Ge layers (500 nm) on Si with anisotropic profile of the etched features to realize PIN photodetector structures.

For a long time dry etching processes of Si and Ge are well known and widely studied [5 - 8]. Because etching mechanisms of both materials are similar, halogen based chemistries including chlorine (Cl_2), fluorine (CF_4, SF_6) and bromine (HBr) reactive gases are generally used for both materials. Germanium is usually more rapidly etched than silicon in conventional halogen plasmas at low bombardment conditions but it is possible to reverse this result by using a gas mixture $SF_6/H_2/CF_4$ [9].

It is not easy to realize selective etching of one material to the other and control the profile of the etched features at the same time. Several authors have studied the ion etching mechanism of SiGe alloys [10-12] and high selectivity has ever been associated to isotropic etching. As it will be presented in this work, high selective etching of Ge to Si can be achieved under low ion bombardment conditions, but it is always associated to isotropic etching profiles.

The challenge of this work was to study the etching of Ge and Si using Cl_2, CF_4 , SF_6, and HBr feed gases by varying pressure and bias power so as to get a selective ICP etch process for Ge with stop on Si and with anisotropic profile of the etched structures.

II. EXPERIMENTAL PROCEDURES

The etching was performed in an ICP reactor model Centura AP1, manufactured by Applied Materials, using 13.56 MHz as power supplies for both, source and bias power. The inductively coupled source power was initially fixed at 350 W and after changed to 600 W. The bias power was varied from 0 to 50 W. CF_4, HBr, SF_6, and Cl_2 were used as main gases. The pressure was varied from 4 to 50 mTorr. All experiments were done on 200 mm silicon wafers kept at 60 ^oC.

A preliminary study of Ge/Si etch selectivity used full 2.5 μm Ge layers epitaxialy grown on Si-wafers by reduced pressure chemical vapor deposition (RP-CVD) as described by J. M. Hartmann et al. in their work [13]. Silicon samples were virgin polished 200 mm Si wafers. Ge and Si etching rates were determined by a weighing comparative process using a precision balance model AT261 Delta Range and adapted for weighing of 200 mm wafers. All wafers were measured two times before and after etching.

The thickness of Ge and Si etched layers were, respectively, calculated by equations 1 and 2, where: t_{Ge} and t_{Si} are respectively thickness of Ge and Si etched layers expressed in nanometers; ρ_{Ge} is the Ge density (5.3268 g/cm³); ρ_{Si} is the Si density (2.3280 g/cm³); r is the wafer radius in millimeters; and Δm is the wafer mass variation in grams, measured before and after etching.

$$t_{Ge} = \frac{10^7}{\rho_{Ge}.\pi r^2}.\Delta m = 5975.631.\Delta m \quad (1)$$

$$t_{Si} = \frac{10^7}{\rho_{Si}.\pi r^2}.\Delta m = 13673.105.\Delta m \quad (2)$$

To study the etch profile, new samples with 500 nm thick Ge layer were covered with resist (M78Y, 790 nm thick) and a standard photolithography process was done to define the pattern which has to be transferred during the etching experiments.

All samples were cleaved after etching and observed in a scanning electron microscope (SEM) model HITACHI S5000 so as to observe etched structure profiles.

III. RESULTS AND DISCUSSION

A. Preliminary study of Ge/Si etch selectivity

The first step of this work was to determine with which of the used gases the highest selectivity of Ge over Si could be achieved.

Based on the equipment possibilities, two different chamber total pressures (4 and 50 mTorr) and two bias powers (0 and 50 W) were used. The source power was kept constant at 350 W for all preliminary experiments. CF_4, HBr, SF_6 and Cl_2 reactive gases were separately used and the gas flow were always kept constant at 100 sccm, except for SF_6. Here it was limited by the equipment at 50 sccm.

Fig. 1 shows the Ge/Si etch selectivity for the different etch conditions (a, b, c, and d). Except the point of low pressure and low bias ("b" condition), Cl_2 was the gas, that promoted the highest Ge to Si selectivity. The peak value of 116 was found at 50 mTorr and a 0 W bias power (Fig. 1.d). With SF_6 no Ge/Si etch selectivity was found under all pressure and bias power conditions. Using CF_4 the highest Ge/Si etch selectivity was 6 at the point of low pressure and low bias power. For all other conditions the Ge/Si etch selectivity was approximately 2, independent of the etch gas. It is worth mentioning that with the Cl_2 etch gas at 50 mTorr and 0 W bias power (condition "d"), the etch rate of Si was nearly zero. This result can be explained by the fact that chlorine etching of un-doped Si follows an ion-assisted mechanism and Si etching is only made possible by disruptive effect of ions bombarding the Si surface [6].

The average error on Ge and Si etch rate were lower than 0.5 % and 2.7 % respectively. The highest values of error on Ge and Si etch rate were found under 0 (zero) W bias power and high pressure conditions, independently of the reactive gas. The greatest error on the etch rate was 6.98 %, corresponding to silicon etched by Cl_2 gas at 0 W bias power and 50 mTorr. Under these conditions, the Si etch rate was quite null and the processing time was increased 2.5 times to increase the consumption of silicon and minimize the final error.

B. Initial process parameters optimization

Based on preliminary results, Cl_2 was chosen as principal reactive gas and a more detailed analysis of the pressure and bias power parameters influence was

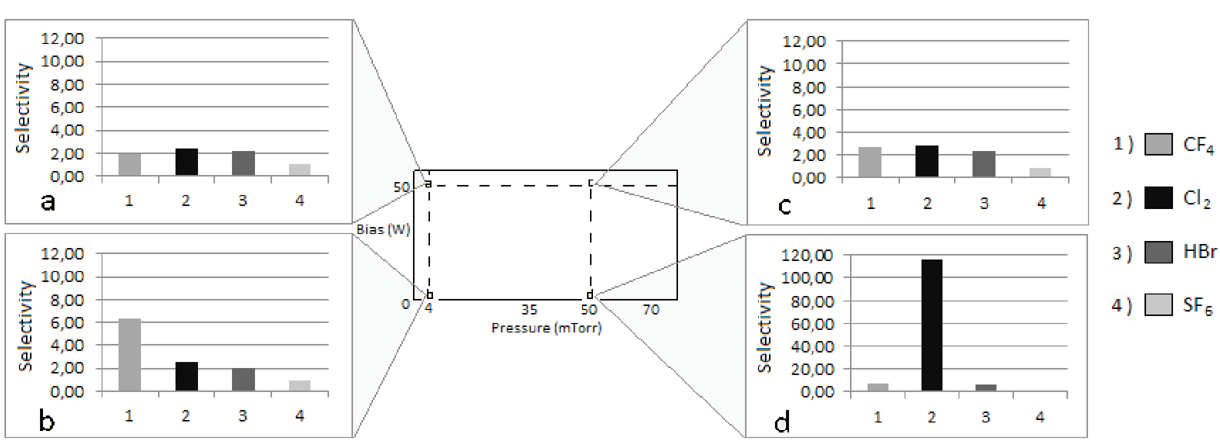

Figure 1. Comparative graph of Ge / Si plasma etch selectivity using different reactive gases (CF_4, Cl_2, SF_6 and HBr) under following different pressure and bias power conditions: a) 4 mTorr and 50 W; b) 4 mTorr and 0 (zero) W; c) 50 mTorr and 50 W; d) 50 mTorr and 0 (zero) W. It is important to observe that figure d) is intentionally presented on a scale 10 times higher than the others.

done. Source power (350 W) and the Cl_2 gas flow (100 sccm) were kept constant. Ge and Si etching rate were measured at the pressure of 4, 35, 50 and 70 mTorr and at 0, 10, 25 and 50 W bias power. The results are shown in Fig. 2 in detail.

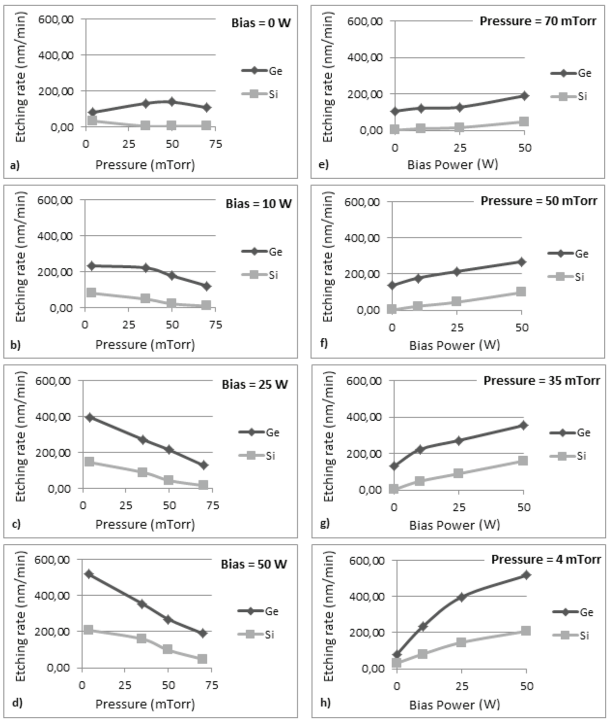

Figure 2. Results of Ge / Si plasma etching using Cl_2 at different pressures and bias powers conditions.

As expected, the higher the pressure and/or the lower the bias power the lower the etching rate of both, Ge and Si. Even though the decreasing the Ge etching rate is more pronounced than that of Si the difference between them becomes more significant. Consequently the higher the pressure and/or the lower the bias power the higher the Ge/Si etch selectivity. Those results are presented in a cartographic perspective in Fig. 3.

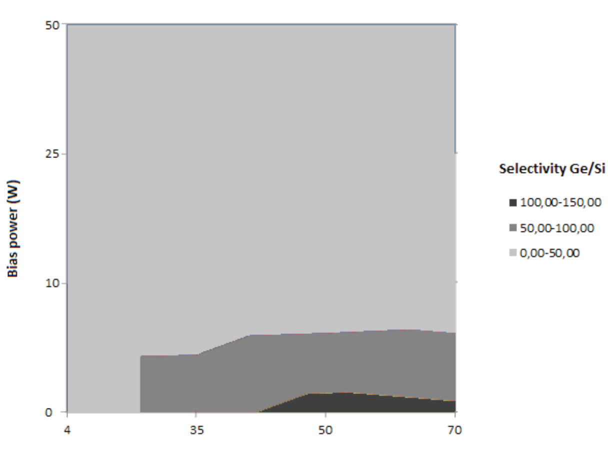

Figure 3. Results of Ge/Si plasma etching selectivity at 350 W of source power and 100sccm of Cl_2 flow.

Plasma stability is necessary to ensure feasibility and reproducibility of the process. However, at high selective conditions with high pressure (70 mTorr) and low bias power (0 – 25 W), the plasma was unstable and sometimes the reflected RF power was so high (greater than 20 %) that the reactor turned off automatically. So as to reduce this instability, the source power was increased to 600 W. At this new condition, the impedance matching was more favorable and reflected power was practically zero.

This increasing in source power led to an increasing in Ge and Si etch rate. This could be explained by the increasing of reactive species (radicals and ions) generated at higher source power. Despite of these variations of etching rates, the Ge/Si etch selectivity did not change significantly (Fig. 4).

C. Profile studies with patterned photoresist mask

Profile studies started using 500 nm epitaxial Ge layers on bulk silicon patterned with the photoresist mask. The etching was performed in two steps. The first one, called "breakthrough" (BT), was done to remove the native oxide layer on the wafer surface. The second one, called "main etch" (ME), was the major step and used to transfer the resist pattern in the Ge layer. The process parameters are summarized in Table 1.

Table 1. Initial parameters used to etch 500 nm epitaxial Ge layers patterned with a 790 nm thick M78Y photoresist mask.

Step	Time (s)	Source (W)	Bias (W)	Pressure (mTorr)	Flow (sccm)	
					CF_4	*Cl_2*
BT	5	350	50	4	100	0
ME	120	600	25	70	0	100

The ME's parameters were found during the initial process optimization and presented a high Ge/Si etch selectivity (7,5:1). However, it can be seen in Fig. 5a) that the resulting profile showed isotropic characteristics which were not desired.

It is known that the addition of nitrogen can help to improve the profile by a passivation process in which a protective polymer layer is formed on the flanks during the etching [8]. This is the reason why in the next set of experiments N_2 was added to Cl_2 etch gas. N_2/Cl_2 ratio flows were varied from 0 to 3.33. The N_2 addition was held constant at 50 sccm (limited by equipment) whereas the Cl_2 gas flow was lowered to 50 sccm, 25 sccm, and 15 sccm respectively. Source power, bias power and total chamber pressure were kept constant at respectively 600 W, 25 W and 70 mTorr. The profiles developed under these conditions can be compared in figures 5b, c, and d.

The best profile was obtained with a gas flow of 15 sccm Cl_2 and 50 sccm N_2 (Fig. 5.d). At this condi-

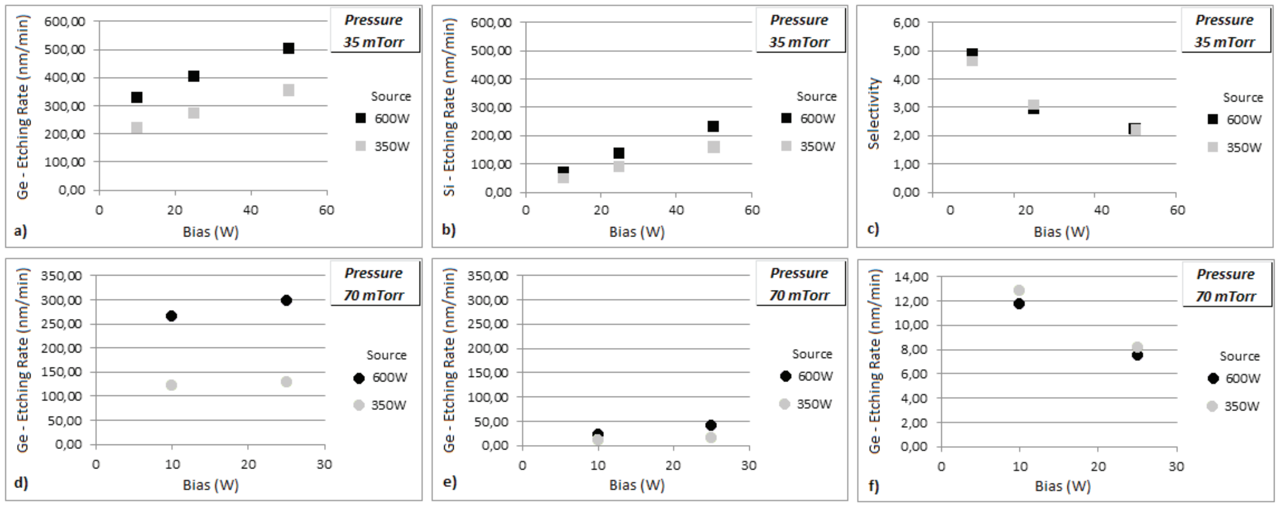

Figure 4. Etch rates and Ge/Si selectivity dependent of bias power at 35 and 70 mTorr and source power of 350 W and 600 W (Cl_2 gas flow: 100 sccm).

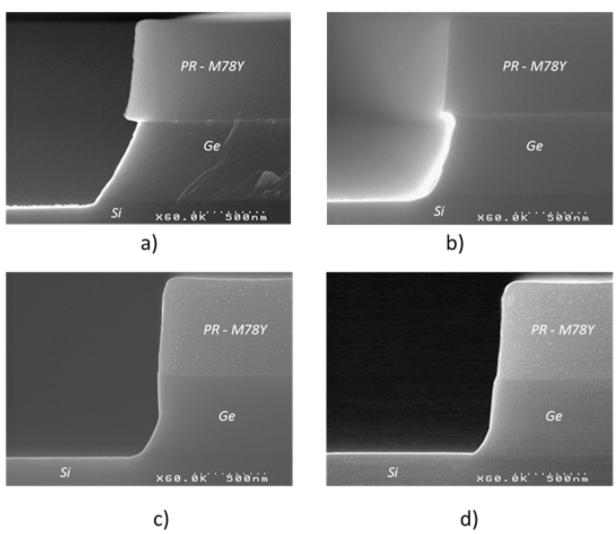

Figure 5. Profiles of Ge etched at several ME gas flow: a) 100 sccm of Cl_2 and 0 sccm of N_2; b) 50 sccm of Cl_2 and 50 sccm of N_2; c) 25 sccm of Cl_2 and 50 sccm of N_2; d) 15 sccm of Cl_2 and 50 sccm of N_2.

tion, the Ge etching rate was 300 nm/min and the Ge/Si etch selectivity was 5:1. Although this process generated an anisotropic profile with a reasonable selectivity the result was not perfect. At the bottom of the structure a kind of "foot", with a lateral tilt of about 54º remains.

D. Final etch development

In order to optimize the etched profile another anisotropic step was inserted into the process which was redefined as a three steps sequence. The first one was the same BT, so as to remove the native oxide layer. The second one, called "main etch 1" (ME1), represents a mostly anisotropic process developed during preliminary studies (350 W source power, 50 W bias power, 4 mTorr total pressure and 100 sccm Cl_2). Fig. 6 shows the straight profile obtained at these conditions.

Because this step is less selective (approximately 2:1), it was used for a partial etching of the main part of the Ge layer and has been stopped after a defined period of time, depending on the etching rate and the Ge thickness. Between 5 to 10% of Ge layer thickness should remain to be etched by a following higher selective step.

For this last step, called "main etch 2", in a first trial the process with a Ge/Si etch selectivity higher than 100 (Fig. 1.d) was chosen. However after the full 3 steps etching process, the profile is partially isotropic and presented a sort of lateral indentation in the structure of Ge (Fig. 7).

A second attempt was realized with the same process parameters which were used to achieve an anisotropic profile and a Ge/Si etch selectivity of 5:1(Fig. 5.d). The parameters of the successfully selective and anisotropic final etching process are shown in Table 2 and the profile analysis is presented in Fig. 8. None trenching or any other surface irregularities were observed.

Table 2 . Final etch process parameters.

Step	Time (s)	Source (W)	Bias (W)	Pressure (mTorr)	Flow (sccm)		
					CF_4	Cl_2	N_2
BT	5	350	50	4	100	0	0
ME1	50	350	50	4	0	100	0
ME2	40	600	25	70	0	15	50

E. Validation tests

Ending the process, the remaining photoresist was stripped by a two steps sequence. First the photoresist was burned in a downstream reactor by $O_2/N_2/H_2/CF_4$ plasma. The use of a N_2/H_2 flow promotes

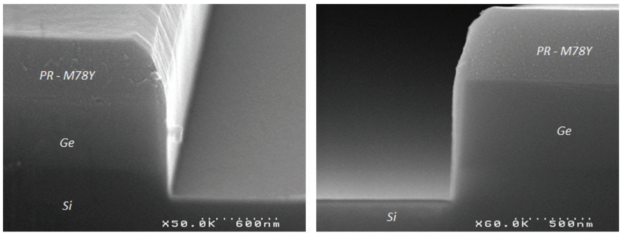

Figure 6. Profiles of Ge etched at 100 sccm of Cl_2, 4 mTorr pressure, 350 W of source power and 50 W bias power conditions.

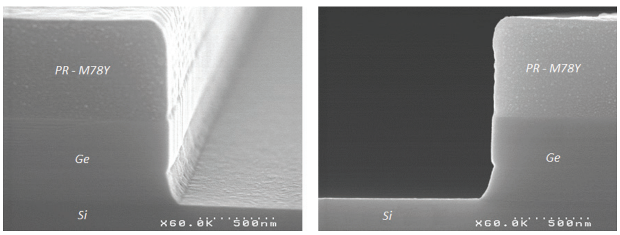

Figure 7. Sample etched by 3 steps sequence. ME2 parameters were: 50 mTorr of total pressure, 350 W source power, 0 W (zero) bias power and 100 sccm of Cl_2.

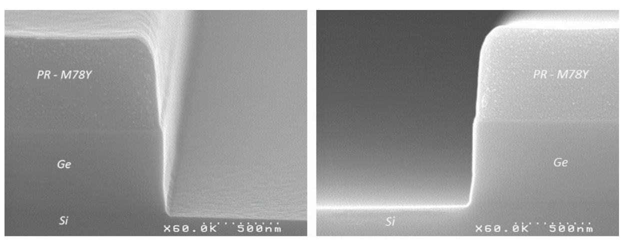

Figure 8. Sample etched by 3 steps etch process as presented at table 2.

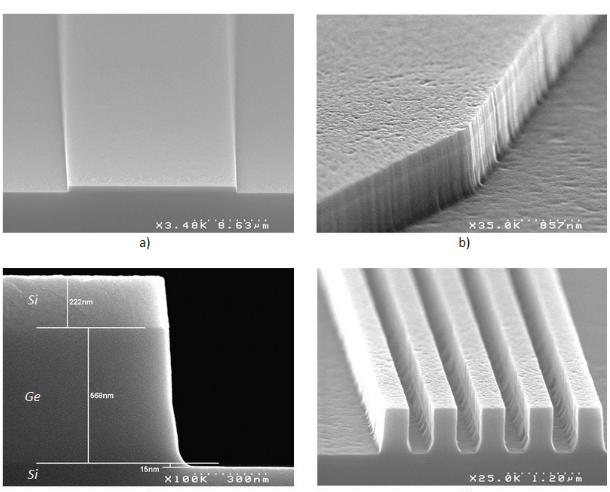

Figure 9. Final PIN structure obtained after etching and stripping.

nitridation and protection of Ge surface and avoids Ge oxidation. During the second step contaminants were removed by a wet cleaning process performed in 0.1% HF bath and rinsed by deionized water.

The full process (etching + stripping) was successfully used to realize PIN structures. For this a 220 nm poly-Si film and a 500 nm thick Ge layer have to be etched consecutively with stop on bulk silicon using a patterned 790 nm resist mask. Because of the similar etch characteristic of Ge and Poly-Si, these two materials were both etched during ME1 with the same process parameters (see table2). Only the etch time of this step was adapted to the actual thickness of the poly-Si plus Ge layers.

As to see in Fig. 9, with the developed 3 step process PIN structures with dimensions less than 1μm and anisotropic profile was realized. The Si substrate consumption was less than 15 nm. It remains a little "foot" at the bottom of the structures, more pronounced for the dense structures, that was negligible for the given application.

IV. CONCLUSIONS

Cl_2 gas, at 50 mTorr and a 0 W bias power conditions, promoted a Ge to Si selectivity of 116, the highest value among all reactive gases tested. With SF_6 no Ge/Si etch selectivity was found under all pressure and bias power conditions. The higher the total pressure and / or the lower the applied bias power the greater the Ge / Si selectivity but the lower the grade of anisotropy. A 3-step ICP anisotropic etching process was developed, using Cl_2/N_2 gas mix at 70mTorr and 25 W bias power conditions during the last step. This process was applied to realize 500 nm - PIN structures with anisotropic etch profile and minimum etch attack of the silicon stop layer on 200 mm wafers.

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