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# Electron-beam-induced reactivation of Si dopants in hydrogenated two-dimensional AlGaAs heterostructures: a possible new route for III–V nanostructure fabrication

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#### Abstract

Hydrogen incorporation in n-type Si-doped GaAs epilayers is a well-known process which leads to the neutralization of the active Si impurities with the formation of SiH complexes. Recently, we have shown that SiH complex dissociation and, consequently, Si-dopant reactivation could occur when the epilayers are exposed to an electron beam.

Two epilayers have been studied: the first is a 0.35  $\mu$ m thick hydrogenated Si-doped GaAs epilayer and the second is Si planar-doped AlGaAs/GaAs/InGaAs heterostructures.

Firstly, Hall effect measurements have been carried out on the epilayers exposed, after RF hydrogen plasma exposition, to increasing electron doses with different injection energies. For the 2D heterostructures, we have observed that the free carrier density  $N_s$  does not vary significantly for weak electron densities. This reactivation presents a threshold value, contrary to the 0.35  $\mu$ m epilayer in which  $N_s$  varies quite linearly. It will be shown that such phenomena might be attributed to the filling of surface states as the dopants are progressively reactivated.

Then, using a high spatial resolution electron beam lithography system, nanometric conductive patterns have been fabricated starting from hydrogenated epilayers. Electric measurements have been performed and the results obtained show that about 15 nm spatial resolution could be expected.

In conclusion, taking into account this spatial resolution, the high spatial contrast of conductivity which could be expected due to the existence of an electron dose threshold, and the high mobility of the AlGaAs/GaAs/InGaAs heterostructure, the effects described in this paper could open a new way for the fabrication of III–V 1D or 2D mesoscopic structures for electronic or optoelectronic applications.

(Some figures in this article are in colour only in the electronic version)

## 1. Introduction

Hydrogen is known to interact with a number of impurities and defects in crystalline semiconductors. For several years, the hydrogen-dopants interactions have been the most widely studied because of the problems of dopant neutralization and device reliability it raises. It is well-known that hydrogen, introduced deliberately or not, gives rise to a decrease of the free carrier concentration as well as an increase of the free carrier mobility. This passivation results from the formation of electrically neutral dopant-hydrogen complexes and, very often, one has to find out ways to dissociate these complexes in order to reactivate the dopants. Many excitations are possible to enhance the dissociation of Si-H complexes in GaAs, such as thermal annealing [1], minority carrier injection [2], hot carrier injection [3], ultraviolet illumination [4], and finally low energy electron beam irradiation [5]. In recent works, it has been shown that the reactivation of neutralized dopants in hydrogenated n-type GaAs doped with Si could also be induced by an electron beam generator. Such a process could be used for the fabrication of patterns of conductive or insulating micronic regions in hydrogenated GaAs layers, which have been observed by cathodoluminescence imaging [6]. However, in order to perform high mobility components, we study here the possibility of passivation and reactivation in an AlGaAs/GaAs/InGaAs heterostructure offering a free carrier mobility about twice as high as GaAs homoepitaxies. Another purpose of this paper is to perform fabrication at a more nanometric scale. For that, more insight about the localization of the effects giving rise to the dissociation of the Si-H complexes is necessary.

## 2. Materials and methods

These experiments were performed on two different epilayers. The first is 0.35  $\mu$ m n-type GaAs, grown on a 0.3  $\mu$ m undoped GaAs buffer layer, by molecular beam epitaxy on a semi-insulating GaAs substrate. The active layer is Si-doped GaAs with an n<sup>+</sup> doping level chosen at 4 × 10<sup>18</sup> cm<sup>-3</sup>. The second epilayer used in these experiments is an AlGaAs/GaAs/InGaAs heterostructure with a  $\delta$ -doping Si (sheet concentration =  $5 \times 10^{12}$  cm<sup>-2</sup>), presented in figure 1.

The different epilayers were then hydrogenated or deuterated in a 13.56 MHz capacitively coupled plasma under the following conditions: sample temperature between 190 and 210 °C, RF power 3 W, gas pressure 1 mbar and exposition duration variable according to the epilayers and to the gas used (H or D). Finally, the sheet carrier concentration  $N_s$  and the electron mobility  $\mu_n$  were deduced from Hall effect measurements performed at 300 K, with different active areas from 200 × 200 to 2000 × 2000  $\mu$ m<sup>2</sup>.

## 3. Electron-beam-induced reactivation and interpretation

The first experiments were carried out on a 0.35  $\mu$ m thick Si-doped (4–5 × 10<sup>18</sup> cm<sup>-3</sup>) GaAs epilayer grown by molecular beam epitaxy. From Hall measurements, a sheet electron concentration  $N_{\rm s} = 1.51 \times 10^{14}$  cm<sup>-2</sup> and a free carrier mobility  $\mu_{\rm n} = 1650$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> were obtained. The layers are then exposed to a hydrogen plasma under the following conditions: sample temperature 190 °C, RF power 3 W, hydrogen pressure 1 mbar, exposure duration 6 h. After plasma exposure, the samples are characterized for  $N_{\rm s} = 2.72 \times 10^{12}$  cm<sup>-2</sup> and  $\mu_{\rm n} = 3650$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>. Then, selected regions of the hydrogenated epilayers were exposed uniformly to electron beams with different electron injection energies: 10, 20, 30 and 50 keV. We can notice that these incident electron energies are much higher than the complex dissociation energy, which is about 3.5 eV. This value was obtained from experimental results [7] and confirmed by two different theoretical calculations [8, 9]. Hall measurements

GaAs:Si, type n- 20 nm	
$Al_{0.24}Ga_{0.76}As$ , type n- $30$ nm	
$\delta - Si$ N(Si)=5x10 <sup>12</sup> Si <sup>+</sup> /cm <sup>2</sup>	
$In_{0.20}Ga_{0.80}As, n.i.d.$ 13 nm	
$Al_{0.24}Ga_{0.76}As$ N s=4.5x10 <sup>11</sup> Si <sup>+</sup> /cm <sup>2</sup>	
AlGaAs/GaAs 260 nm	
GaAs n.i.d.	
Substrate GaAs S.I.	



Figure 1. The AlGaAs/InGaAs/GaAs heterostructure.

Figure 2. The evolution of the extra sheet carrier concentration  $\Delta N_s$ , measured by the Hall effect at 300 K, of hydrogenated 0.35  $\mu$ m Si-doped GaAs samples exposed to ( $\blacklozenge$ ) 10, ( $\blacksquare$ ) 20, ( $\blacktriangle$ ) 30 and ( $\textcircled{\bullet}$ ) 50 keV electron beams versus the injected electron density  $\Delta N_e$ . The electron beams on various active area surfaces (200 × 200, 300 × 300 and 2000 × 2000  $\mu$ m<sup>2</sup>). The inset shows an enlargement for injected electron densities below  $5 \times 10^{15}$  cm<sup>-2</sup>.

in the region submitted to electrons were performed for increasing injected electron dose  $\Delta N_{\rm e}$  (between  $2.7 \times 10^{14}$  and  $6.2 \times 10^{17}$  cm<sup>-2</sup>) in order to determine the variation of the free carrier density  $\Delta N_{\rm s}$ . These experiments were carried out using either a scanning electron microscope (SEM) or an electron beam pattern generator. In both cases, electron beam currents were measured using a Faraday cage. The variations of the electron density  $\Delta N_{\rm s}$  for an increasing incident electron dose are presented in figure 2.

The following features can be noted:

- (i) a strong increase of the free carrier concentration with increasing number of electrons injected in the materials,
- (ii) a higher efficiency of electron-induced dopant reactivation as the injection energy decreases.

The first phenomenon can be explained by the dissociation of Si–H complexes due to the electron beam. The second observation can be explained by considering that most of the energy loss of 10 keV accelerated electrons occurs within the thickness of the first 0.35  $\mu$ m Si-doped layer, since the penetration depth of these electrons is 0.5  $\mu$ m. In contrast, if we consider 50 keV acceleration voltages, the penetration depth of electrons is 8.1  $\mu$ m and thus, most of their interactions take place in the non-doped semiconductor substrate where the Si–H complexes are not present. In order to check these considerations more accurately, Monte Carlo simulations have been performed using Sceleton code to determine electron trajectories in the n<sup>+</sup> active epilayer and the semiconductor substrate [10]. For each injection energy, the average energy lost within the thickness of the 0.35  $\mu$ m Si-doped layer by one injected electron has been determined.



**Figure 3.** The evolution, versus the electron beam injection energy, of the donor reactivation efficiency  $\Delta N_s / \Delta N_e$  resulting from experiments (full line) and of the energy lost by one electron in the 0.35  $\mu$ m hydrogenated Si-doped epilayer obtained by Monte Carlo simulations (dotted line).

The results obtained have been plotted in figure 3 versus the injection energy and compared to the experimental dissociation efficiency defined as the ratio  $\Delta N_s / \Delta N_e$  determined, using the inset of figure 2, at the beginning of the electron irradiation, when the reactivation has just begun to occur and no saturation phenomena can be observed. It can be noted that the dissociation efficiency is proportional to the energy lost by energetic electrons in the Si-doped epilayer, and such results can be quite well understood assuming an electronic excitation of the Si–H complex by energetic electrons.

## 4. The reactivation of Si planar-doped two-dimensional electron-gas heterostructures

In this last part, we study high mobility 2D heterostructures which could be used to fabricate micro or nanoelectronic structures. In this case, Si-doping is introduced in AlGaAs, instead of GaAs, and the involved physical phenomena could be slightly different. However, it has been shown in the study of UV reactivation that about the same optical cross-section is obtained and that a quite similar phenomenon probably occurs in AlGaAs [11].

The studied epilayer is presented in figure 1 and is characterized by a sheet carrier concentration, measured by the Hall effect, equal to  $3 \times 10^{12}$  cm<sup>-2</sup>. The plasma conditions are: temperature 210 °C, RF power 0.15 mW cm<sup>-2</sup>, gas pressure 1 mbar, and exposition time 10 min for hydrogen and 20 min for deuterium. The evolutions of the extra sheet carrier concentration versus electron dose, observed in hydrogenated and deuterated heterostructures, are presented in figure 4 for an electron energy of 20 keV.

The results shows the following features:

- (i) no significant increase of  $\Delta N_s$  while the electron dose does not reach a threshold value (about 10<sup>16</sup> electrons cm<sup>-2</sup>);
- (ii) a strong increase of  $\Delta N_{\rm s}$  with  $N_{\rm e}$  above this threshold value; and
- (iii) a much higher electron dose threshold for deuterated samples.

The electron dose threshold observed for the heteroepilayer, and also for bulk GaAs as the active layer thickness is reduced [12], could be explained by the existence of surface states. Due to the high speed of the incident electrons, resulting from their high acceleration energies, these electrons do not interact with the surface states. However, due to these states, a depleted region could occur in the AlGaAs layer as in the InGaAs channel. For low ionized donor concentration obtained after H or D passivation, the channel in the heterostructure epilayer is fully depleted of electrons, and very high values of resistivity are measured. Consequently, the first liberated electrons contribute to an additional filling of surface states and no significant evolution of resistivity is obtained. But, once all the surface states are filled, the complex reactivation can give rise to the increase of the free carrier concentration as already observed in  $0.35 \ \mu$ m epilayer.



Figure 4. A comparison of the evolution of the extra sheet carrier concentration versus the dose of injected electrons for a hydrogenated (squares) and deuterated (triangles) 2D heterostructure. The samples are exposed to an electronic microscope with electron energy of 20 keV.

The results obtained for the 2D heterostructure seem to be quite interesting, since the observed effects could be used for the fabrication by electron beam writing in hydrogenated samples of patterns of conductive and insulating nanometric regions. A spatial resolution better than the one obtained with electronic lithography could be achieved, since no resist layer is used. In addition, due to the existence of an electron dose threshold, the conductivity contrast between passivated and 'written' area by the electron beam could be very important. Lastly, due to the use of 2D electron gas, very high electron mobility could be measured.

### 5. Spatial resolution evaluation on a heterostructure

This new fabrication process of nanometric components, and, in the simplest case, of conductive wires, would be useful only if the wire width experimentally observed does not exceed by too much the wire width electronic writing. To check this point, resistivity measurements of conductive lines of different widths processed on hydrogenated epilayers by electronic writing have been carried out. To achieve such experiments, it is necessary to realize, as is shown on figure 5, ohmic contacts on the heterostructure presented in figure 1. Then, the samples are exposed to a hydrogen plasma under the following conditions: temperature 190 °C, RF power 0.15 mW cm<sup>-2</sup>, gas pressure 1 mbar, and exposition time 15 min. Due to this exposure and to the hydrogen passivation of the active dopants, the square resistance strongly increases, with an experimental value above 50 M $\Omega$ . Finally, using electron beam writing, conductive wires between adjacent ohmic contacts are fabricated, according to three patterns, presented in figure 5:

- (a) a conductive line of 15  $\mu$ m width which will be the reference to determine the square resistance of the material after electronic irradiation;
- (b) a pattern of 15 lines of 1  $\mu$ m width each, spaced out 4  $\mu$ m apart; and
- (c) a pattern of 30 lines of 0.5  $\mu$ m width each, spaced out 2  $\mu$ m apart.

The electron beam writing is carried out using the following conditions: electron acceleration energy 50 keV, electron dose 0.032 Cb cm<sup>-2</sup>, beam diameter about 20 nm. Then, assuming that the wire widths of the fabricated samples are equal to the electron writing widths, the square resistance of these samples are determined. Typical results are: (a) 1800  $\Omega$ /square (b) 1790  $\Omega$ /square and (c) 1643  $\Omega$ /square. The differences observed for the various type of structures could be explained taking into account the real conductive wire widths ( $e + 2\delta$ ), greater than the electronic writing widths (e) as described in figure 6.

Using such assumptions and the different values of the square resistance, approximate values of delta are estimated and range from 8 to 20 nm. The strong dispersion of these values





**Figure 5.** Electronic writing used to determine the spatial resolution of electron beam writing on a hydrogenated heterostructure. (a) 1 line of 15  $\mu$ m width, (b) 15 lines of 1  $\mu$ m width, and (c) 30 lines of 0.5  $\mu$ m width.

**Figure 6.** A schematic representation of the conductive region (*e* width), its dispersion ( $\delta$  width) and insulating region (white) used to estimate the spatial resolution of this processing method.

can be explained by the high ratio (e/delta), especially for samples (a) and (b). Nevertheless, it appears that a spatial resolution of a few tens of a nanometre can be expected and such a value is much less than the electron diffusion length of about 1  $\mu$ m observed in a minority carrier generation effect.

## 6. Conclusion

In this paper, it has been shown that high energy electron beam irradiation is a very efficient way to dissociate dopant–hydrogen or dopant–deuterium complexes resulting from H or D plasma exposition, and that this effect occurs not only for GaAs homoepitaxy but also for AlGaAs/GaAs heteroepitaxy. This reactivation of dopants can be attributed to an electronic excitation of complexes. In addition, there appears to be an electron dose threshold for the heterostructure inducing a better contrast between insulating and conductive regions, and the spatial resolution of this new processing method has been estimated to be about 15 nm. As has been pointed out in this paper, all these results could lead to a new type of fabrication process of semiconductor nanostructures and consequently could open the route to the fabrication of new type of devices, in electronic as well as optoelectronic applications.

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