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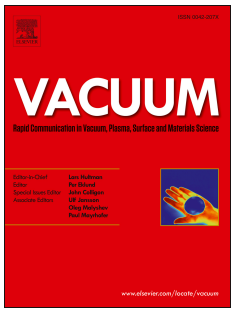

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~~Epitaxially crystalized rich layer of SixGe1-x after dry oxidation of doped silicon with germanium ions~~
Epitaxial Ge-rich silicon layers after dry oxidation of Ge implanted silicon

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Abstract

We report on formation of epi-layer of Si_xGe_{1-x} by taking standard procedure in CMOS technology. The competitive process of solid solubility of Ge dopant into Si and SiO_2 is the key to engineer atomically sharp, low defect very thin epitaxial layer at the interface of oxide-Si. Oxidation time process was used to control the distribution of the doped Ge ions at the interface of Si with oxide and in the oxide layer. Implanted samples (35 keV and 1×10^{16} Ge^+/cm^2) were oxidized at 1050 °C for 30 to 90 minutes. RBS-Channeling analysis shows two separate peaks of Ge corresponds to different depths after oxidation. Corroborate with high resolution microscopy and elemental analysis, we determined the first peak as enriched layer of Si_xGe_{1-x} at the interface of SiO_2-Si. ~~Few nm~~ Less than 10 nm epitaxially grown interfacial layer is very low in defects, and Ge ions are fully substituted into the host lattice. The second peak originated from diffusion of Ge into SiO_2 resulted in a segregated layer containing Ge in oxide film. Technological demand on forming Si_xGe_{1-x} layer for CMOS application through standard routes is what we address in this research.

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Key words: Point Defects, Solid phase epitaxy, Germanium silicon alloys, high resolution electron microscopy, ion implantation

1. Introduction

Silicon–Germanium (SiGe) nano-layer and Ge nano-crystal have attracted intensive attention during the last decade due to demand for high speed and low power electronic devices for the next generation of micro and nano-electronic and opto-electronics devices [1–5]. Therefore, SiGe is an extremely important known semiconductor alloy due to its employment in device fabrication for the wide varieties of applications to wireless communications, optical wave-guides, infrared detectors [2,4] and CMOS [6–8]. Also, another promising application of SiGe is its use in core-shell structures for photovoltaic [7,8].

Many techniques have been used to synthesize SiGe nano-layers, including sputtering, chemical vapor deposition (CVD) [6], molecular beam epitaxy (MBE) [9], pulsed laser deposition and Ge ion implantation in silicon substrate [9–11]. Among them, fabricating SiGe heterostructure and Si/SiGe heterojunctions on nano-layers of SiGe prepared by oxidation of Si implanted Ge ions is a cost effective route for devices to be used in SiGe based electronic and opto-electronics applications [12,13]. The germanium rich nano-layer between the oxide and the underlying silicon substrate was formed, because of the pile-up or snow plowing phenomena upon redistribution of Ge ions during thermal oxidation of Si [2,9,14–17]. Therefore, thermal oxidation of Ge implanted Si has been investigated in the past and last decade under a variety of oxidation conditions [9,14,16,17]. However, these researches often ended with some contradictory conclusion on the formation of mixed SiO_2+ GeO_2 [18,19], the segregation of Ge underneath the oxide [2,7,10,16], and the oxidation rate enhancement with respect to pure Si

[1,15,16,19–21]. On the other hand, the literature surveys of past reports reveal that there are many open questions on the structural properties of the interfacial Ge rich layer in particular in high temperature oxidation above 1000 °C.

This work aims to examine and investigate the interfacial Ge rich layer and redistribution of Ge impurities at the interfaces of SiO_2-Si by RBS-Channeling and high resolution electron microscopy after thermal oxidation in pure O_2 of Ge implanted Si at high temperature. Due to current technological demand on forming Si_xGe_{1-x} layer for CMOS application, we believe that our work can elucidate more the thermal processing mechanism of Silicon doped with Ge and subsequent redistribution of dopant ions at the interface.

2. Materials and methods

In the present work, p-type Si (100) wafers with resistivity in the range of 5-15 Ω-cm were used. Implantation was done using singly charged Ge^+ ions with energy of 35 keV and doses of 1×10^{16} Ge^+/cm^2 into virgin Si (100) substrate at liquid nitrogen temperature, and incident angle of 6° with respect to the normal to the substrate's surface to avoid channeling. Dry thermal oxidation was done in a standard quartz-tube furnace at 1050 °C for 30, 60 and 90 minutes in a pure O_2 ambient. Before dry oxidation of samples, the initial oxide layer was etched in a diluted HF (4%) solution. The RBS-Channeling measurements were performed with a beam of 2.0 MeV He^+ ions with a detector placed at 165° scattering angle for RBS-Channeling in IBM geometry. The resolution of the detectors estimated to vary between 12 to 14 keV and in terms of depth this range of resolution for SiO_2 thickness is about 2 nm. Measurements were made in a turbo-pump evacuated chamber at pressure $\leq 1\times10^{-6}$ Torr. SIMNRA 6.6 computer program was used to simulate the random RBS spectra according to experimental conditions [22]. The RBS-

Channeling was used to determine the oxide thickness and to investigate the redistribution of the implanted Ge ions in the samples. Samples for microscopy analysis were prepared via conventional mechanical polishing down to transparency of silicon (10 microns thickness) and subsequent ion milling using PIPS Gatan Precision ion milling. Prepared samples were characterized either using JEOL JEM 2200FS FEG TEM/STEM equipped with the Omega filter and EDS detector or Hitachi HD2700 Dedicated STEM with EDS detector both operated at 200 kV.

3. Results and discussion

Figure 1 shows RBS spectra in random (blue triangle) and channeling (black circle) directions of the Si (100) substrate implanted with 35 keV, 1×10^{16} Ge^{+}/cm^{2} without any heat treatment or thermal oxidation process. To ensure an accurate conclusion on the effects of Ge implantation in silicon, RBS-Channeling measurements along the (100) axial direction were done on both implanted and non-implanted (virgin Si) regions (red solid line) of the same sample as illustrated in Figure 1. Monte Carlo simulation of the 35 keV Ge ions implanted into a silicon target using SRIM 2013 [23] code estimates between 290 Å to 300 Å the projected range of the maximum concentration of the Ge ions into silicon. Considering the dose of 1×10^{16} Ge^{+}/cm^{2}, the maximum Ge concentration reaches 0.04×10^{22} Ge^{+}/cm^{3} assuming the atomic density of Si as 4.976×10^{22} Si/cm^{3}.

As can be seen in Figure 1, the RBS spectrum of the implanted region of silicon shows a broad peak in the energy interval of 1000 - 1150 keV in aligned spectrum. The number of backscattered probe ions along (100) axial direction of the substrate in this region reaches the same backscattering events in the random spectrum in Figure 1. This is due to the fact that

implantation of heavy ions like germanium will displace the silicon atoms from their lattice sites, leaves a heavily damaged region on top of the silicon substrate. Furthermore, the two peaks appeared around 1550-1600 keV in both random and channeled spectra of the same region correspond to the Ge ions, which almost coincide in their height. Since the area under the peak in RBS spectra gives an estimation of the amount of corresponding element, once the probe ions are meeting dopant inside crystallographic directions, the number of backscattering events from those Ge ions which are substituted in lattice sites of Si is much less than the backscattering events from those which are located in an interstitial sites of the main lattice [24-26]. Based on this criteria, our analysis in Figure 1 confirms that upon tilting the sample from random to aligned state, there is no difference on the amount of Ge ions, clearly ascertain that the dopant fully occupied the interstitial sites.

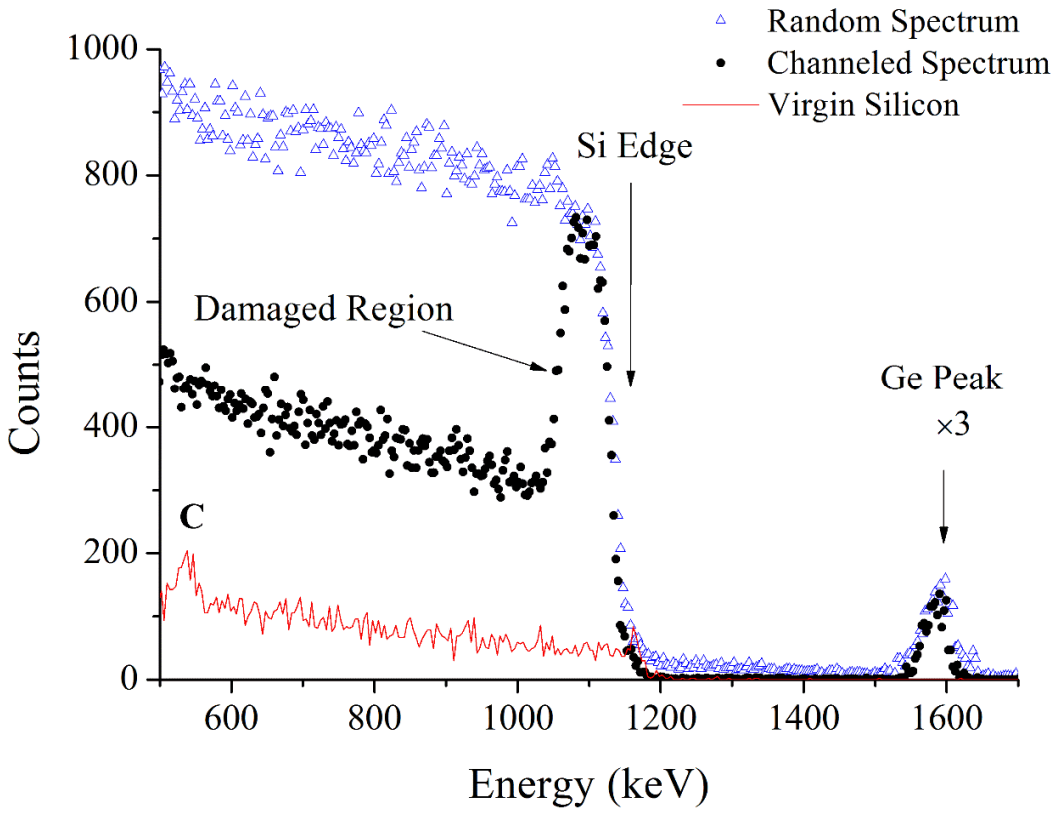

Fig. 1. RBS spectra in channeling (black circle) and random (blue triangle) states of the Si (100) substrate implanted with the dose of 1×10^{16} Ge^{+}/cm^{2} and energy of 35 keV. The channeling spectrum along the (100) direction on non-implanted region of the same sample is shown for comparison (red color). The surface contamination peak of carbon is also shown in the channeling spectrum of virgin sample as energy below 600 keV.

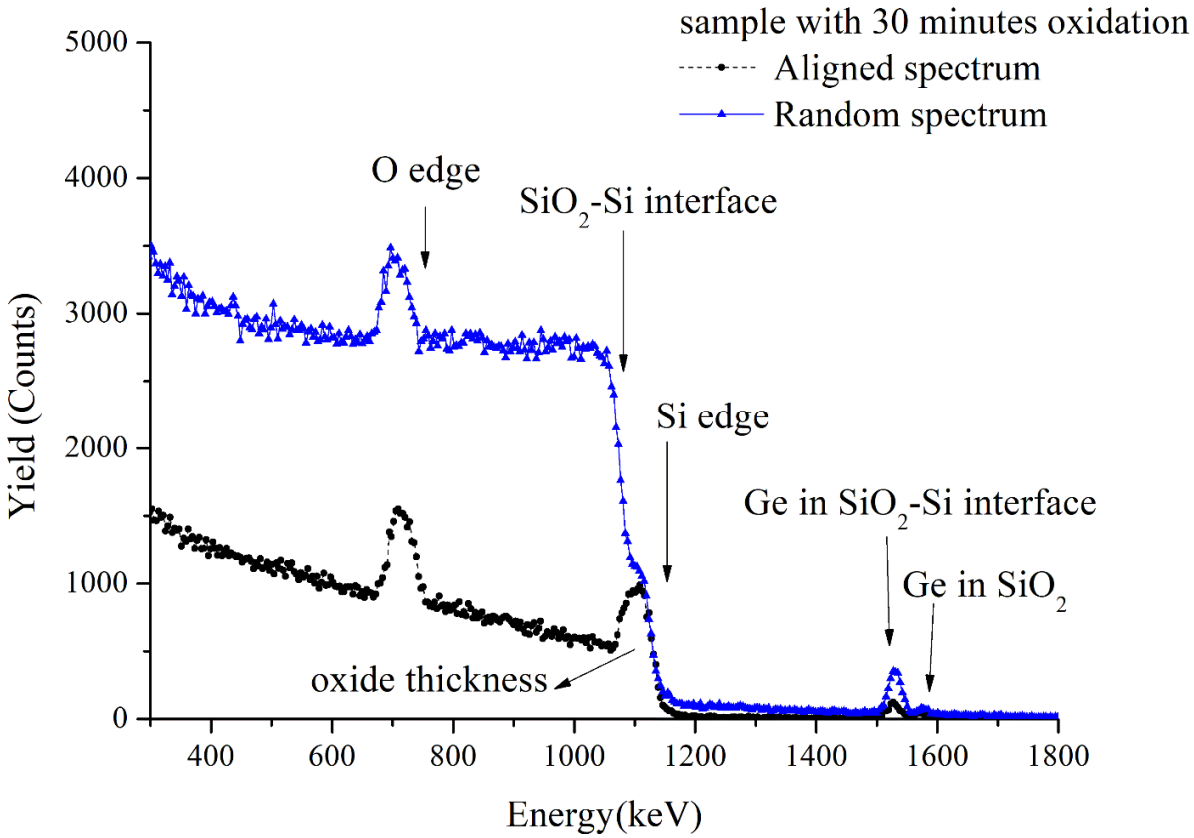

Fig. 2. Random and aligned RBS spectra of the Si (100) substrate implanted with the dose of $1x10^{16}$ Ge^{+}/cm^{2} and energy of 35 keV after dry oxidation at 1050 °C for 30 min. The energy region between 1500 keV to 1600 keV shows two Ge peaks as discussed in the text.

Figure 2 compares the random and aligned RBS spectra of the implanted region of silicon after dry oxidation at 1050 °C for 30 minutes. The position of the arrows for O, Si and Ge refers to the respective backscattering energies from these elements at the surface and the SiO_2-Si interface. In the RBS spectra, the thickness of the oxide layer can be seen as a step on the Si edge in the random spectrum or as a broad peak on the oxygen edge that represents the amorphous structure

of SiO_2. Calculations using SIMNRA 6 indicates that the thickness of SiO_2 layer for 30 minutes oxidation is about 115 nm. The distribution of Ge ions after oxidation in Figure 2 reveals interesting behavior. The Ge peak appears as two separated sharp peaks illustrating the accumulation of the Ge ions at two different depths in the sample. The peak at the energy of 1520 keV, shifted 80 keV deeper in position with respects to the surface position of Ge (1600 keV), exhibits accumulation of dopant at the SiO_2-Si interface. Simulation of RBS random spectra shows that this peak corresponds to a Ge rich layer of Si_xGe_{1-x} formed at the SiO_2-Si interface [3, 24, 25]. Such accumulation of the dopant after dry oxidation at the silicon surface results in formation of a very thin layer of Si_xGe_{1-x} of the order of few nanometers. Comparison of the scattering yield for Ge peak in this thin layer in random and aligned spectra indicates that the height (intensity) of the Ge peak drops significantly in align spectrum. Therefore, it can be concluded that this layer has high crystalline structure, as the intensity of silicon reduces in aligned spectrum, so does in the Ge peak. The other peak at energy of 1580 keV is originated from diffusion and incorporation of Ge atoms into SiO_2 layer during oxidation. The intensity of this peak in both random and aligned spectra is unchanged, as we expect, the Ge ions were randomly distributed in amorphous structure of the oxide layer. These two separate Ge peaks show the diffusivity of Ge dopant in silicon and oxide layer, faster diffusivity of Ge into silicon dioxide at this temperature pushes dopant even further away from the interface of SiO_2-Si. Also, progress of oxide layer during oxidation process will push some part of implanted Ge ions into the depth of the silicon, accumulate them as a rich layer of the Ge at the interface. The unique characteristic of this layer is that it has established an epitaxial relationship to the silicon substrate, as observed from random and aligned spectra.

Figure 3 with an emphasis on the Ge signal shows in details profiles of the Ge concentration in RBS spectra in random and aligned states of the implanted Si after dry oxidation at 1050 °C for 30, 60 and 90 minutes. As it can be seen in Figure 3.a, random spectra, in all three various oxidation cycles the height and the width of the Ge peak are the same, although the position of the Ge peak at the interface shifted slightly to the higher energies (7 keV shift) after 60 min. oxidation compared to the one after 30 minutes oxidation. This means that the longer oxidation time does not have effective role in snowplowing of Ge at high temperature dry oxidation (1050° C) and disagrees with the previously reported results above 1000 °C [20]. According to the available data on dry oxidation up to 1000 °C, the oxidation time is important to accumulate more Ge ions at the interface [1, 7, 20]. However, this scenario may change by increasing the amount of implanted Ge as was discussed in Ref. [20].

In addition, Figure 3.a clearly shows reduction in the height of the Ge peak along (100) direction after three oxidation processes with the maximum decrease being observed for 90 minutes oxidation. Moreover, comparison of the scattering yield of the Ge peak in random and aligned spectra and by calculating the area under the Ge peaks at different depths in random spectra, it is estimated that about 20 percent of the Ge atoms were diffused into SiO_2 layer. Simulations of the random RBS spectra of three samples in Figure 3.a gives a range of $0.065 \leq x \leq 0.085$ in rich layer of Ge formed at the interface as a Si_xGe_{1-x} mixed layer.

To investigate the crystallinity of the Ge enriched layer at SiO_2-Si interface, we can use the normalized angular yield profile along the (100) axial direction in oxidized samples and the minimum normalized yield or χ_{min} (the ratio between aligned and random counts) as one of the most important parameter to measure the crystallinity of the implanted layer [10, 26]. Figure 3.b shows the χ_{min} for Si and Ge atoms along the (100) axial direction for sample oxidized for 30

minutes at 1050 °C. In this typical profile, the vertical axis which is proportional to the number of particles backscattered from the sample in random incident is normalized to 1, and indicates the signal from conventional RBS spectra in random state. Thus, any drop in the normalized value versus angle shows that the probe ions have encountered less atoms in that specific direction before being backscattered.

From Figure 3.b the normalized angular yield profile of the Si and Ge atoms along (100) crystal direction displays similar trend, with the difference just in the value of χ_{min}; χ_{min} for silicon is estimated to be 0.4 and for Ge ions, 0.6. Therefore, these values confirm that Ge atoms have occupied lattice site positions, resulted in slightly more backscattering of the helium ions along crystallographic direction, due to induced stress/strain as expected from the difference in the ionic radii of the Si and Ge ions.

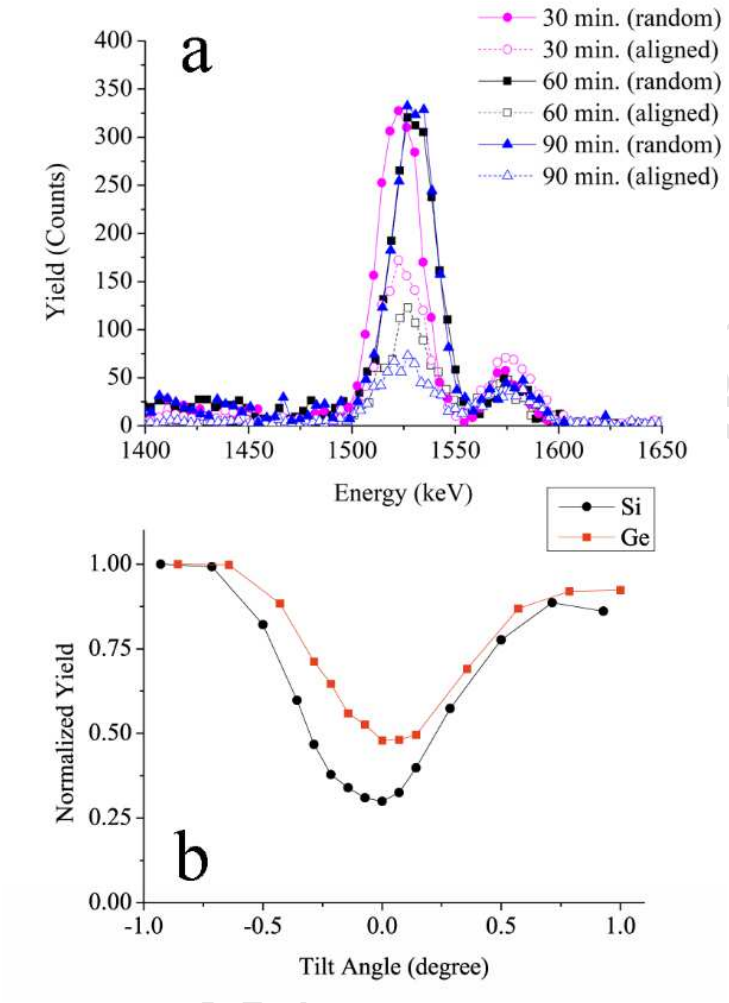

Fig. 3.a. Profiles of the Ge concentration in random and the aligned RBS spectra of the Si (100) substrate implanted with the dose of 1×10^{16} Ge/cm^2 and energy of 35 keV after dry oxidation at 1050° C for 30, 60 and 90 min. b. Normalized angular yield profile for Si and Ge atoms along the (100) axial direction in sample implanted with the dose of 1×10^{16} Ge$^+$/cm^2 and energy of 35 keV after dry oxidation at 1050° C for 30 minutes.

To further shed light on the distribution of the Ge ions in Si and oxide layer, also presence of dislocations and defects in the lattice of the Ge-rich layer at the interface of oxide/Si, high resolution TEM/STEM with elemental analysis in nano-scale have been implemented on selected samples. Figure 4.a exhibits HAADF image of the sample annealed at 1050°C for 30 minutes, where change in the contrast of the image at the interface of SiO_2-Si denotes presence of heavier

element than silicon substrate. Our interpretation in this image relies on the sensitivity of the HAADF-STEM imaging to the atomic number of the elements in the sample, heavier elements should appear with more bright contrast. Although accurate determination of the Ge-rich layer is not straightforward because of its diffuse boundaries with silicon substrate, from taken image, one can estimate 8 nm almost uniform, rich Ge layer at the interface. Further analysis of the interface using bright field detector shown in Figure 4.b reveals the atomically sharp interface between the rich Ge layer and the oxide layer within the resolution of our microscope (0.14 nm). The arrangements of the atoms in the image follows the one from the substrate, reconfirm our observation from RBS-C spectra that oxidation process has established a thin epitaxial layer of $Si_{1-x}Ge_x$ at the interface. Also, no clear dislocation is observed in the image, replicates the same layer formed during dry oxidation of the molecular beam epitaxy grown SiGe film [19]. EDS analysis in Figure 4.c shows that the thickness of the Ge rich layer may surpass 8 nm estimation from contrast change in HAADF image, it can be somewhere in between 15 nm and 20 nm thickness (tilting of the sample to increase EDS signal can increase the appeared thickness).

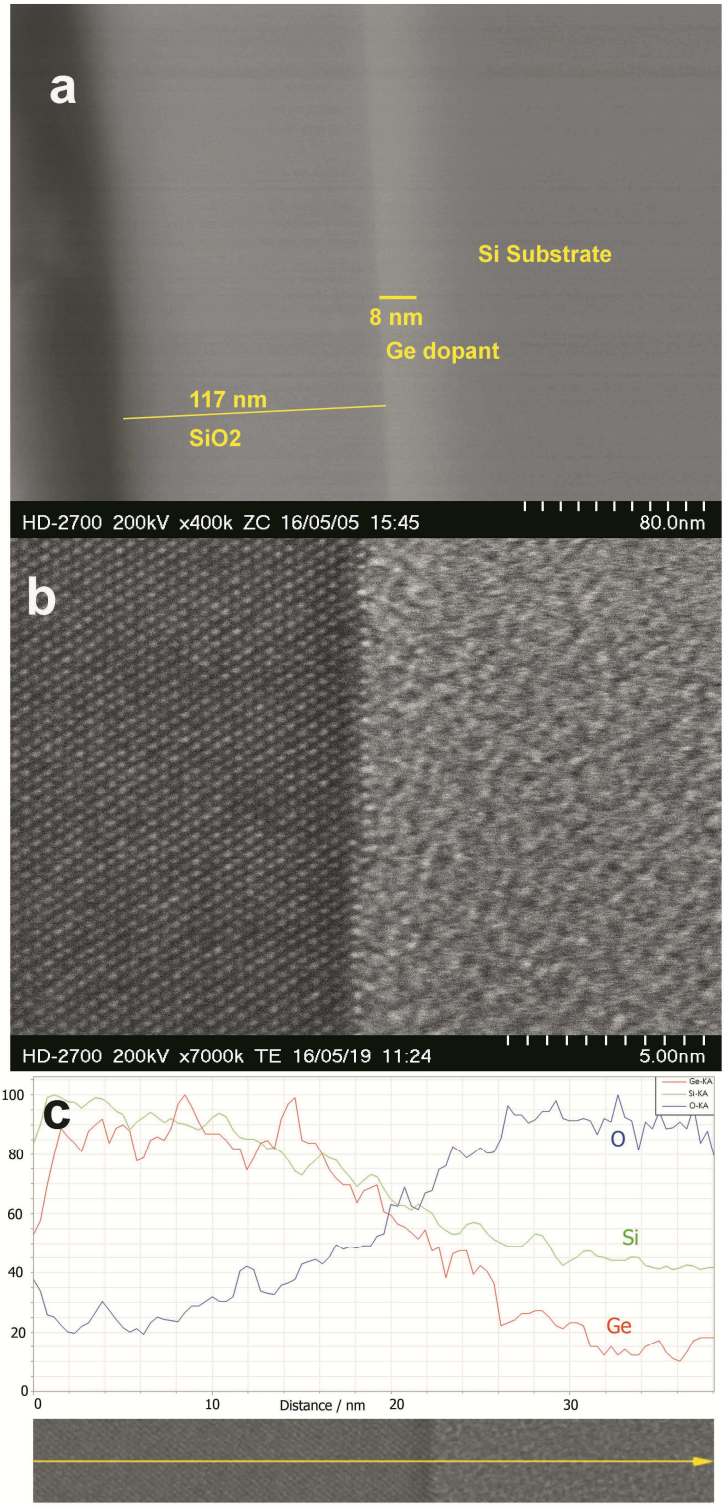

Fig. 4. For sample implanted with the dose of 1×10^{16} Ge^{+}/cm^{2} and energy of 35 keV after dry oxidation at 1050° C for 30 minutes, (a) HAADF-STEM image that is very sensitive to atomic number of elements, (b) bright field STEM image of SiO_2-Si interface showing the sharpness of the interface and (c) elemental distribution of Ge, Si and

O was obtained from EDS analysis in the SiO_2-Si interface. The profiles are not normalized and are independent from each other.

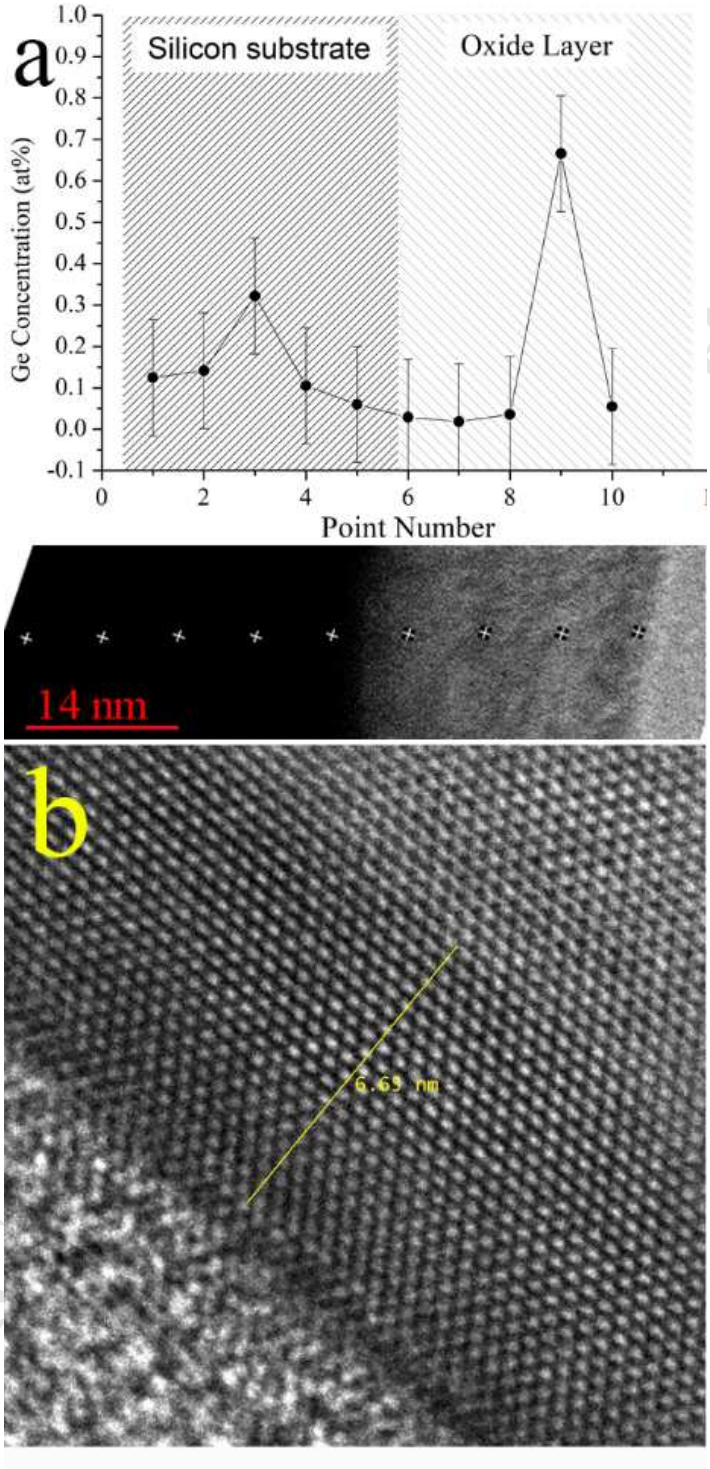

Fig. 5. STEM image and the results of the elemental analysis of the Ge along the points shown in image (a), indicating the state of the Ge distribution at the interface and inside the oxide layer for the sample implanted with the dose of 1×10^{16} Ge^{+}/cm^{2} and energy of 35 keV after dry oxidation at 1050° C for 90 minutes. (b) BF-STEM

image of the interface of oxide/Si showing epitaxially established rich layer of Ge, as the contrast of the image suggests it.

Figure 5.a indicates the result of the elemental analysis of the Ge peak along points shown in Figure 5.a from the top surface of the oxide to the silicon. The EDS analysis visibly reveals formation of two rich layers of Ge in this sample, one at the interface of SiO_2-Si and the other slightly above the interface inside the oxide layer. The concentration of Ge ions in oxide layer is higher than Si part, although a narrow tail of the Ge ions can be seen between these two peaks. This analysis corroborates with the RBS spectra in a qualitative manner where two separate peaks of the Ge ions were found, enabling us to determine the accurate location of these two Ge-rich layers in the samples. It should be mentioned that the electron beam has to be normal to the sample to avoid thickness effect for the purpose of the accurate quantitative analysis. However, it results in drop in the signal of the Ge ions substituted into host Si lattice, compared to the Ge ions in top amorphous layer. To unveil the lattice recovery of the silicon upon implantation and oxidation, and the impact of Ge ion redistribution in host lattice sites, STEM image in Figure 5.b was taken right at the interface of the oxide/Si from the same sample. The atomic arrangements of the Si ions along <111> zone axis does not indicate any presence of dislocations, which we might expect it from RBS-C analysis as a significant reduction in the signal of silicon in aligned spectrum was observed [27]. Since Ge ion has higher mass than Si, in the region doped with germanium (Si_xGe_{1-x}), one should expect change in contrast, as can be seen in Figure 5.b, a narrow region immediately at the interface shows darker contrast in compare to the remaining of the silicon. The image shows that the recovery of the damaged region and formation of the rich Ge layer does not impose visible stress/strain to the silicon lattice, mostly because of the diluted doping into the silicon lattice.

4. Conclusions

In summary, combination of ion beam analysis and electron microscopy techniques were used to reveal the redistribution of the Ge ions implanted into Si (100) substrate after dry oxidation at high temperature. Both analytical techniques confirmed accumulation of the Ge ions at the interface of the oxide/substrate and oxide layer. RBS-Channeling spectroscopy and high resolution electron microscopy were used to identify the structural quality of the rich Ge layer after oxidation processes. The interfaces of the very thin Si_xGe_{1-x} epitaxial layer with either Si or oxide are atomically sharp. It was shown that the germanium implantation as a standard route in semiconductor processing with subsequent dry oxidation would result in formation of few nanometers Ge rich layer, with epitaxial relation to the silicon substrate at the interface. The remaining of the segregated germanium in oxide layer can be chemically etched to leave the top surface of the silicon ready for the next steps on SiGe-based devices. Different solubility of Ge in Si and oxide, and in general for most common dopants is an advantage to control the concentration of Ge (dopant) in Si_xGe_{1-x} layer, which thereafter results in thickness control to form ultra-thin epi-layers at the interfaces. The depletion of the interface from extra dopant and thermal recrystallization of the interface are significant factors to create very flat interfaces after doping.

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- Ge implantation and dry oxidation were used to form very thin mixed Si_xGe_{1-x} layer.
- A rich Ge layer was formed after dry oxidation at 1050 °C.
- Very thin Si_xGe_{1-x} layer is in epitaxial relation to the silicon substrate.
- Some Ge ions are segregated in top oxide layer.