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# Si/$\beta$-FeSi$_2$/Si heteronanostructures fabricated by ion implantation and Si MBE: growth, structural and luminescence properties

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**Abstract.** The method of ultrahigh vacuum low-temperature ($T = 850$ °C) cleaning of Si(100) and Si(111) samples implanted by iron ions (Fe$^+$) with different implanted fluencies ($\Phi = 10^{15}$–$1.8 \times 10^{17}$ cm$^{-2}$) has been applied for the first time. The possibility of the formation of atomically smooth and reconstructed Si surfaces after Fe$^+$ implantation and nanosecond pulsed ion-beam treatment has been shown. It was found that smooth Si films with thickness up to 1.7 $\mu$m and with reconstructed surface grow up to implanted fluence $\Phi = 10^{16}$ cm$^{-2}$. Preservation of $\beta$-FeSi$_2$ nanocrystallites inside silicon matrix after cleaning and formation of a cap epitaxial Si layer has been confirmed by transmission electron microscopy data, optical spectroscopy data and existing of photoluminescence peak at 0.75–0.85 eV during temperature range of 5–150 K.

## Introduction

In recent years the considerable attention has been paid to the formation of isolated precipitates and continuous layers of semiconducting iron disilicide ($\beta$-FeSi$_2$) in Si due to the possibility of application of $\beta$-FeSi$_2$ in optoelectronics as a light emitter in the 1.5–1.6 $\mu$m range. The main methods for the formation of $\beta$-FeSi$_2$ are Fe$^+$ implantation of Si [1] and reactive deposition epitaxy of Fe layers followed by molecular beam epitaxy (MBE) of Si layer [2]. Both methods include the high-temperature (up to 900 °C) and long-time (up to 20 hours) furnace annealing undesirable for device structures because their parameters essentially degrade at elevated temperatures. We have demonstrated earlier the formation of $\beta$-FeSi$_2$/Si heterostructures by low-energy Fe$^+$ implantation and pulsed treatments by ion and laser beams [3]. For subsequent fabrication of light-emitting diode the formation of Si/$\beta$-FeSi$_2$/Si heterostructures is preferable.

In this paper a new low temperature growth technology including ion implantation and Si MBE is applied for the first time. The structural and optical properties of the grown Si/$\beta$-FeSi$_2$/Si heterostructures are studied.

## 1. Experimental

The implantation of Fe$^+$ ions into monocrystalline Si wafers with (100) and (111) orientations was carried out at room temperature with ion energy $E = 40$ keV ($R_p = 37$ nm, $\Delta R_p = 13$ nm) and fluencies $\Phi = 10^{15}$–$1.8 \times 10^{17}$ cm$^{-2}$. The details of sample preparations are given in Table 1. Pulsed ion-beam treatment (PIBT) of the implanted Si layers was carried out using pulsed ion accelerator with high-energy nanosecond carbon ion beams (C$^+$, $E = 300$ keV, $\tau = 50$ ns). Pulse energy density varied between $W = 1.2$–$1.5$ J/cm$^2$ and fluence of C$^+$ ions implanted into Si during PIBT does not exceed $\approx 10^{13}$ cm$^{-2}$.

Si overgrowth was performed in two UHV chambers with base pressures $p = 10^{-10}$ Torr and $p = 10^{-9}$ Torr. Both chambers were equipped with sublimation $p$-type Si sources ($N_p = 10^{-16}$ cm$^{-3}$) for MBE growth. The first chamber was equipped with Auger electron spectrometer (AES) used for measuring of impurity concentrations on Si surface before and after cleaning procedure, the last one had the low energy electron diffraction (LEED) facility for *in-situ* studying of the structure of the grown Si layers. Si deposition rate was controlled by quartz sensor of thickness. Si samples were annealed by applying of direct current.

**Table 1.** Parameters of Samples.

| Sample | Substrate | Implanted dose, cm$^{-2}$ | Conductivity type | Resistivity (Ohm cm) |
|---|---|---|---|---|
| *A* | Si(111) | $10^{16}$ | *p* | 20 |
| *B* | Si(111) | $1.8\times10^{17}$ | *p* | 20 |
| *C* | Si(100) | $10^{15}$ | *n* | 4.5 |
| *D* | Si(100) | $10^{16}$ | *n* | 4.5 |
| *E* | Si(100) | $1.8\times10^{17}$ | *n* | 4.5 |

Photoluminescence (PL) measurements (5–150 K) were carried out (ISP SB RAS) by means of He-Ne laser ($\lambda = 632.8$ nm, $J = 40$ mW/cm$^2$). PL spectra were analyzed in spectrometer on the basis of double monochromator and detected by Ge photodiode “Edinburgh Instruments”.

## 2. Results and discussion

According to TEM data [4] an implantation of Fe$^+$ ions into monocrystalline Si wafers with (100) and (111) orientations leads to nanocrystallites formation in near surface region about 100 nm in size if implanted fluence doesn’t exceed $1\times10^{16}$ cm$^{-2}$. An increase of implanted fluence results in coalescence processes up to continuous layer formation at fluence about $1 \times 10^{17}$ cm$^{-2}$.

Sample *A* surface after PIBT (Table 1) represents nearly periodical structure of alternative round or oval shaped areas, 300–500 nm in diameter. These areas are situated 3–4 nm lower than the rest of the surface and they represent the cellular structure of Si layer which was observed in [3] by TEM. Increase of implanted fluences of $Fe^+$ ions (by 18 times for the sample *B*) leads to considerable increase of surface relief; hills and holes were formed with height and depth of 20–30 nm with respect to some mean level. This attributed to higher content of iron impurity in the near-surface Si layer and significant mass transfer during PIBT due to deep melting of Si (up to 1 $\mu$m). The formation of cellular structure is related to low solubility of Fe in Si and to Fe segregation on Si cell walls due to instability of the solidifying interface.

The sample *C* implanted with the lowest fluence $\Phi = 10^{15}$ cm$^{-2}$ has the minimal root-mean square roughness ($\sigma_{\text{rms}} =$ 0.23 nm) after PIBT. Si surface implanted with fluence $\Phi = 10^{16}$ cm$^{-2}$ (sample *D*) is quite smooth ($\sigma_{\text{rms}} = 0.4$ nm). Increase of the fluence up to $1.8 \times 10^{17}$ cm$^{-2}$ (sample *E*) results in the increase of local surface non-uniformity as well as occurrence of areas with large relief ($\sigma_{\text{rms}} = 2.62$ nm) and with iron disilicide crystallites inside that have moved up and occurred on the surface in the form of granular film. The formation of $\beta$-$FeSi_2$ phase is confirmed by optical reflectance spectroscopy data, type of absorption coefficient spectra and the presence of interband transitions (2.7 eV) corresponding to $\beta$-$FeSi_2$ (2.57 eV, [5]).

A new low-temperature (LT) cleaning procedure suitable for Si overgrowth upon Si layers implanted by $Fe^+$ ions has been studied. The sample kept at $T = 850$ °C was exposed to the Si atomic flow with small rate of about 0.1 nm/min for 15–20 minutes, which provides decomposition of $SiO_2$ firstly to SiO and then to Si and $O_2$ (gas). AES data revealed that all oxide and carbon contaminations were completely removed and clean Si surface was obtained as a result of this procedure. This procedure was successfully applied for the first time for $Fe^+$ implanted samples.

Si overgrowth on the surface of all samples was performed after LT cleaning. After Si overgrowth ($T_{\text{Si}} = 700$ °C) on the sample *C* a smooth ($\sigma_{\text{rms}} = 0.08$ nm) epitaxial film with thickness of 1700 nm was obtained and LEED Si(100)2 × 1 pattern was observed without any additional annealing. Si overgrowth with the same growth parameters on the sample *D* resulted in the obtaining of an epitaxial film (1700 nm) demonstrating LEED Si(100)2 × 1 pattern although after LT cleaning LEED pattern was not observed. Hence, the annealing of defects on the surface takes place during Si deposition and then the growth goes on epitaxially and surface ordering takes place. The increasing of the fluence up to $1.8 \times 10^{17}$ cm$^{-2}$ (sample *E*) results in polycrystalline growth of silicon and the sample surface becomes rougher ($\sigma_{\text{rms}} = 86.2$ nm). The pinholes observed in AFM images indicate a 3D growth mechanism and agglomeration of Si crystallites. The similar tendency was observed during Si overgrowth on the samples *A* and *B* after LT cleaning; however, the surface roughness was 7.0 nm and 11.8 nm, respectively. The reflection spectroscopy data obtained from all samples after Si overgrowth reveal that in the energy range below 2 eV the behaviour of the reflection coefficient is in line with that for silicon. Hence, $\beta$-$FeSi_2$ crystallites do not occur on the surface after Si overgrowth.

The discovered optical interference in *p*-type Si layers grown onto the surface of Fe-implanted layers in the transparency wavelength range (higher 1200 nm) proves a formation of abrupt interfaces in grown heteronanostructures.

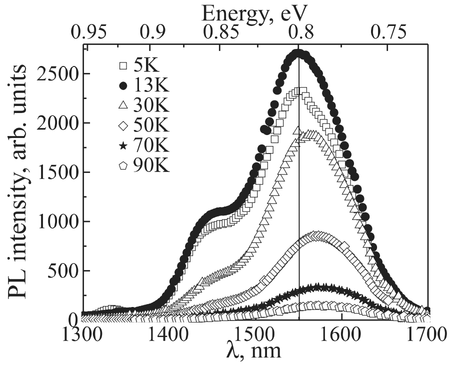

**Fig. 1.** PL spectra of sample *D* registered at different temperatures.

The study of PL properties of Si/$\beta$-$FeSi_2$/Si heteronanostructures obtained by ion implantation, PIBT and MBE has been carried out. It was shown that intensity of PL depends on a dose of implanted iron, crystalline quality of grown epitaxial silicon layer and substrate orientation. From the PL spectrums of sample *D* obtained at different temperatures (Fig. 1) we plot the temperature dependence of amplitude of the PL band near 0.8 eV and approximated it by the equation follow.

$$I(T) = \frac{I_0}{1 + C_1 \exp\left(\frac{-E_1}{kT}\right) + C_2 \exp\left(\frac{-E_2}{kT}\right)}.$$

Here, $C_1$ and $C_2$ are weighting coefficients, $E_1$ and $E_2$ are activation energies of the decay process, and $I_0$ is the PL intensity at a temperature close to absolute zero. The values of activation energies ($E_1 = 41$ meV and $E_2 = 12$ meV) were determined by fitting the theoretical curve to the experimental data. From the comparison of $E_1$ and $E_2$ values with activation energy of dislocation line $D_1$ ($\approx$10 meV) we can conclude that this line makes a contribution to PL signal and $E_1$ energy apparently relates to PL signal from $\beta$-$FeSi_2$ precipitates. Furthermore we observe a red shift of the PL band near 0.8 eV in the temperature range 5–70 K ($\approx$15 meV) whereas energy position of $D_1$ line in this range almost preserve.

Recently we have obtained planar TEM images of sample *D*. They showed us a high density of dislocations ($5.3 \times 10^9$ cm$^{-2}$), which confirms our PL data for sample *D*. An origin of these dislocations apparently is the implanted substrate because our AFM and LEED data testifies that cap silicon layer was grown epitaxially. So the dislocations just have come up from the substrate.

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## References

[1] K. Oyoshi *et al*, *Thin Solid Films* **381**, 194 (2001).
[2] T. Suemasu *et al*, *Thin Solid Films* **381**, 209 (2001).
[3] R. Bayazitov *et al*, *Nucl. Instr. Meth.* **B24**, 224 (2005).
[4] K. Oyoshi *et al*, *Thin Solid Films* **381**, 202 (2001).
[5] J. Chrost *et al*, *Surf. Sci.* **330**, 34 (1995).