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Optical characterization of nanostructured β – FeSi₂ layers obtained by Fe⁺ implantation

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Abstract

In this work, we present a comprehensive analysis of nanostructured β – FeSi₂ layers obtained by 40 keV Fe ion implantation in silicon, followed by rapid thermal annealing. A series of chemical, structural and optical characterizations of the samples were performed. Our results establish the formation of a 26.6 nm thick layer consisting of β – FeSi₂ nanocrystals, with an average size of 4.8 nm, embedded in the Si substrate. Optical excitation of the sample leads to a photoluminescence signal with an extremely narrow peak (1 nm full width at half maximum) at 1456 nm. This sharp emission is comparable with the radiation of semiconductor lasers and therefore, this β – FeSi₂ nanostructured layer is of interest for the fabrication of new optoelectronic devices in the near-infrared region.

Keywords: nanostructure, photoluminescence, beta-iron disilicide, ion implantation

(Some figures may appear in colour only in the online journal)

1. Introduction

 β – FeSi₂ compounds have been investigated thoroughly in the literature, due to their promising semiconducting properties, making them serious candidates for the fabrication of new near-infrared optoelectronic devices on silicon, like photonic crystals. Indeed, β – FeSi₂ is a direct band gap semiconductor, with a gap between 0.83 and 0.92 eV, which corresponds to a wavelength between 1494 and 1348 nm, very near the absorption minimum wavelength for silica optical fibers (\simeq 1550 nm) [1]. Moreover, β – FeSi₂ has a large optical absorption coefficient (over 10^5 cm^{-1}) [2–4]. Finally, the constitutive elements Fe and Si are easily found in nature and they can be safely manipulated.

Due to all these remarkable properties, $\beta - \text{FeSi}_2$ is being studied actively in the literature, especially in regard to possible optical applications. In particular, in the most recent literature, Ishiyama *et al* [5] discuss possible applications as photonic devices compatible with existing complementary metal-oxide-semiconductor (CMOS) technology, because $\beta - \text{FeSi}_2$ has infrared emission. Terai *et al* [6] and Wang *et al* [7] argue that since $\beta - \text{FeSi}_2$ shows a photoluminescence around 1.54 μ m it can be used for optoelectronic and photovoltaic applications. Rajesh *et al* [8] expound on the fact that for the direct band gap and for the large optical absorption coefficient, β – FeSi₂ has great potential for applications like telecommunication devices based on silicon, photo sensors, solar cells and thermoelectric generators (see also [9]).

Previously, different techniques for the fabrication of β – FeSi₂ have been used, such as ion beam synthesis (IBS), molecular beam epitaxy (MBE) and metal-organic chemical vapor deposition (MOCVD) [10]. With these methods one can obtain different structures, ranging from thin films [11] to nanoparticles [12] and heterojunction thin films [13]. It has also been observed that the photoluminescence signal may vary depending on the method used. Therefore it is of primary importance to obtain a type of structure with a stable optical response, allowing its use in the fabrication of new optoelectronic devices. In this regard, Rysbaev et al [14] have recently started an investigation on nanosized films of silicides obtained by ion beam implantation. The advantages of the ion beam implantation technique over other methods are the repeatability of the samples, since the distribution of the implanted ions can be controlled with high accuracy, alongside the fact that, with the use of a mask, the ion implantation can be performed in specific areas of a wafer, thus encouraging its application in thin silicon technology, which is very useful for device fabrication. This makes this technique the method of choice for the manufacture of silicon integrated circuits for example.

Previous works developed by other authors have focused on the characterization of the structural properties of β – FeSi₂ by means of Rutherford backscattering (RBS), Raman spectroscopy, and x-ray diffraction (XRD) [15, 16]. However, in order to have a complete understanding of the properties of β – FeSi₂, we need to combine the existing results with further analysis, involving other techniques.

For all these reasons, in this work a comprehensive characterization of β – FeSi₂ nanocrystals obtained by ion beam implantation is presented, focusing on the photoluminescence (PL) properties that are relevant for the fabrication of optoelectronic devices in the near-infrared region.

2. Experimental setting

 β – FeSi₂ nanocrystals have been obtained by ion beam implantation at high fluence (1 × 10¹⁷ ions cm⁻²) and low energy (40 keV). The motivation for using high-fluence implantation is because this provokes concentrations that exceed the solubility limit of the Fe ions implanted in the silicon matrix. Annealing this oversaturated system leads to the formation of β – FeSi₂ nanoparticles.

Commercially available crystalline Si (111) wafers were implanted with 40 keV Fe ions at a fluence of 1×10^{17} ions cm⁻². The Si wafers were cut into small square pieces (1×1 cm²) and subsequently they were ultrasonically cleaned in ethanol for 5 minutes. Rapid thermal annealing (RTA) was performed at 900 °C for 8 min under a chromatographic nitrogen (N₂) atmosphere. After RTA the samples



Figure 1. SIMS concentration depth profiles for the sample prepared by 40 keV Fe ion implantation in Si $(1 \times 10^{17} \text{ ions cm}^{-2})$ and annealed in N_2 at 900 °C for 8 min. The red curve corresponds to the Fe profile and the blue curve to the Si one. For comparison the Fe depth profile simulated by SRIM is included (green curve).

were etched with 3 keV Ar ions at normal incidence without sample rotation and approximately 10 nm were removed.

The chemical analysis of the samples was investigated by secondary ion mass spectroscopy (SIMS) by acquisition of a depth profile performed by a TOF-SIMS-5 spectrometer from Ion-TOF GmbH. A 2 keV cesium ion beam for sputtering and a 30 keV Bi⁺ pulsed ion beam were used for analysis of the central part of the sputter crater. The angle of incidence was 45° for both ion beams. The sputter crater size was chosen to be $300 \times 300 \ \mu\text{m}^2$ and the area of analysis was a $150 \times 150 \ \mu\text{m}^2$ raster in the center of the sputter crater.

The structural characterization, chemical analysis and PL signal were performed by Raman spectroscopy with an NTEGRA Spectra instrument from NT-MDT with a 100x objective. For structural characterization and chemical analysis a laser excitation at 532 nm was used, while for PL signal the laser excitation was at 473 nm. The exposure time was 60 seconds and the CCD was cooled to -65 °C to obtain the spectrum in the mode of single acquisition.

Morphology of the samples was investigated by atomic force microscopy (AFM) and scanning electron microscopy (SEM). The tapping mode AFM studies were carried out in air by using a Solver Next instrument from NT-MDT. We employed silicon cantilevers with a tip curvature radius of about 10 nm. The micrographs were acquired with a resolution of 512×512 pixels and a scanning frequency of 1 Hz. SEM measurements were performed using a JEOL JSM-7800 F, with secondary electrons with a voltage of 5 kV. The chemical map was obtained by energy dispersive spectroscopy (EDS) at 10 kV. The nanocrystals embedded in a silicon matrix were observed by transmission electron miscroscopy (TEM) (JEM2010 FEG).



Figure 2. AFM micrographs corresponding to the different stages of the 40 keV Fe ion implanted sample $(1 \times 10^{17} \text{ ions cm}^{-2})$: (a) as-implanted sample; (b) after the RTA in N_2 at 900 °C for 8 min; (c) after removing a 10 nm thick layer by Ar⁺ etching; (d) after removing a 60 nm thick layer.

3. Results

3.1. SIMS depth profiling

SIMS was used to determine the concentration depth profile of the 40 keV Fe ions implanted in Si substrates and annealed in N_2 at 900 °C for 8 minutes. As shown in figure 1, the Fe peak concentration is located at 32 nm. For comparison purposes, the Fe depth profile simulated by the SRIM code is included [17]. The software describes the transport properties of ions in matter, but no thermal diffusion effects are considered. In this case the simulated Fe concentration profile can be described as a Gaussian-like curve, with a maximum value at around 38 nm, similar to the shape of the experimental curve, as can be observed in figure 1. The in-depth diffusion of the Fe atoms from the surface towards the interior of the Si matrix is induced by the annealing. This explains the observed difference between the green and the red curves in figure 1 after 40 nm.

The observed surface sputtering effect due to the highfluence ion bombardment is consistent with the simulated results reported by other groups [16]. Indeed, for our 1×10^{17} ions cm⁻² implanted sample, a similar Fe depth profile as [16] was obtained, with the same maximum value. The Fe profile shows the formation of a new phase between 15 nm and 45 nm in depth scale. This gives us an indication that the thickness of the β – FeSi₂ layer is around 30 nm. To corroborate this intuition, a Gaussian approximation of the Fe profile was used and the calculated full width at half maximum (FWHM) was 26.6 nm ± 0.7 nm.

3.2. AFM and SEM analysis

The surface of the implanted samples was characterized by using AFM and SEM-EDS, both before and after the RTA. Moreover, in order to get access to the nanostructured film, a thin layer of about 10 nm was removed after annealing by etching with normal incident 3 keV Ar ions. Figures 2(a) and (b) show, respectively, the AFM images of the samples before and after the rapid thermal annealing. Structuring can be clearly observed on the silicon surface, attenuated by the RTA. The estimated root mean square roughness (R_{rms}) was 3.5 nm and 2.7 nm respectively. Figures 2(c) and (d) show the same sample after etching into two different depths. After removing a 10 nm thick layer the surface structure exhibits a variety of shapes with irregular sizes of the order of μ m (figure 2(c)). This morphology is still present even after removing a 60 nm thick layer (figure 2(d)). In this case the estimated root mean square roughness (R_{rms}) was 5.8 nm and 4.6 nm respectively.

The SEM results confirm the presence of the same patterns as described from AFM analysis (see figure 3). The structure observed in AFM and SEM can be attributed to selective sputtering. In fact, the SRIM simulation reveals that the erosion coefficients for silicon and silicide exhibit a clear difference: Y(Si) = 1 atom ion⁻¹ and $Y(FeSi_2) = 2.2$ atom ion⁻¹ respectively. Thus, this structure is produced while removing the sample surface with the Ar⁺ plasma etching at differentiated erosion coefficients.

The island morphology observed in figure 3, together with its composition is explained by figure 4, which shows the results of the SEM-EDS analysis associated with the Fe atomic distribution in the silicon substrate. In particular, figure 4(a) is a magnification of a region of the SEM micrograph presented in figure 3(b). The corresponding Si and Fe distributions are shown in figures 4(b) and (c), respectively. Accordingly, the concentration of Fe atoms is clearly higher in those regions corresponding to light grey areas in the SEM micrographs. From the SIMS profile we know that the Fe concentration diminishes at greater depth, and it is for this reason that at 60 nm we observe smaller islands than those at 10 nm. Therefore we can infer that the observed structures are not due to the presence of porosities in the Si matrix, but they are related to the formation of a new compound from the combination of Fe and Si. So far, we argue that the new compound that has been formed is iron disilicide. In the next section, this analysis is complemented with Raman spectroscopy, that proves that iron disilicide is found in its β – FeSi₂ phase.

3.3. Raman spectroscopy

The study of Raman spectroscopy before and after the annealing at 900 $^{\circ}$ C yields information about the amorphization and recrystallization of the silicon substrate. Figure 5 shows the Raman spectra obtained before (blue curve) and after





(b)

Figure 3. SEM micrographs of the Ar^+ plasma etched samples after removing a layer of: (a) 10 nm, and (b) 60 nm.

(red curve) the RTA of the Fe implanted sample. One can observe that before annealing, the shape of the Raman spectrum exhibits two regions. First, a quite broad band, approximately centered at 465 cm⁻¹, which corresponds to the superficial amorphous Si originated by the ion implantation, as discussed in the previous section. The small peak at 521 cm^{-1} is associated with the remaining crystalline Si structure in the as-implanted sample, i.e. the Si amorphization was not complete.

After RTA, the Raman signal clearly shows the 521 cm^{-1} characteristic peak associated with crystalline Si, indicating that annealing at 900 °C induces the recrystallization of the silicon lattice. Additionally, one can observe two small peaks located at 192 cm⁻¹ and 247 cm⁻¹; the characteristic



Figure 4. SEM-EDS analysis associated with the Fe atomic distribution in the Si substrate for the Ar⁺ plasma etched sample after removing a layer of 60 nm. (a) The entire analyzed region; (b) Si distribution; (c) Fe distribution.

(c)

peaks of β – FeSi₂, which are associated with the A_g phonon mode [18-20].

The difference in peak intensity at 521 cm^{-1} can be explained by considering that the crystalline structure (after annealing) has a larger scattering cross section compared to the amorphous sample due to the crystal disorder. The peaks that appear after annealing at 192 cm⁻¹ and 247 cm⁻¹ are due to the fact that the Fe ions have clustered to form the β – FeSi₂ and their intensity indicates the relative concentration of silicide in the silicon matrix. This behavior denotes the formation of β – FeSi₂.

3.4. Photoluminescence

The PL spectrum of the Fe⁺-implanted Si sample after the RTA is shown in figure 6. The main feature observed in the room temperature PL spectrum corresponds to an intense peak at 1456 nm (0.852 eV). This PL signal must be attributed to the formation of a β – FeSi₂ nanostructured layer, because crystalline silicon is an indirect band gap material and no PL emission is expected. This result is quite similar to those reported in previous works, where PL signals in a 0.80 eV



Figure 5. Raman spectra obtained before (blue curve) and after (red curve) the RTA of the Fe implanted sample. The inset shows the two Raman peaks corresponding to the $\beta - \text{FeSi}_2$.



Figure 6. PL spectrum after the RTA at 900 °C. The very thin and intense peak at 1456 nm is associated with the presence of monodisperse $\beta - \text{FeSi}_2$ nanocrystals. The inset is a magnification of the peak region, but in a photon energy scale.

- 0.93 eV energy range have been determined. Thus, some authors ascribe the observed fluctuations in the PL peak position to the variation in the β – FeSi₂ nanocrystal size, ranging from 0.80 eV for large nanocrystals to 0.89 eV for small ones [21, 22]. In the case of nanowires at room temperature (RT), the PL signal centered at 0.96 eV has been reported [23]. Other groups describe a shift in the PL spectrum due to different acquisition temperatures, from 0.93 eV at 3.5 K to 0.87 eV at 300 K [24, 25]. Finally, the PL signal can also depend on the β – FeSi₂ fabrication method, with PL peaks in the 0.80–0.82 eV range [26].

One striking result from the PL spectra shown in figure 6, is that the signal obtained from the nanostructured layer presents an extremely narrow peak, with a FWHM < 1 meV (the



Figure 7. Representative TEM micrographs corresponding to the Fe-implanted Si sample after the RTA. (a) Extended TEM image at high magnification; (b) HRTEM image of a β – FeSi₂ single-crystal with a size diameter of 5 nm embedded in the amorphous layer; (c) diffraction pattern obtained from the red square area indicated in figure 7(b). The β – FeSi₂ nanocrystal lattice was indexed to the [220] plane diffraction pattern.

precise value obtained being 1 nm \pm 0.02 nm), a value considerably thinner than the peak widths previously reported for β – FeSi₂. Low temperature PL measurements (at 10 K) exhibit wide PL signals, with FWHM in the 27–52 meV range for different nanostructured layers [27]. Our PL signal is even narrower than that reported for β – FeSi₂ nanocrystals (FWHM = 19 meV) [28]. By analysis of the PL spectrum and comparison with previous literature, it can be inferred that in our case, the sharp PL peak is due to fairly high monodispersity of the β – FeSi₂ nanocrystals, as discussed in the next section.

3.5. TEM analysis

In order to obtain a better morphological and structural characterization of the β – FeSi₂ nanocrystals, a detailed TEM analysis was performed. Figure 7(a) shows an extended view of the β – FeSi₂ nanocrystals embedded in the silicon matrix, randomly located in the near-surface amorphized Si layer. The depth distribution of the nanocrystals is somewhat narrow, with most nanoparticles laying 40–60 nm from the surface. Figure 7(b) shows a representative high resolution transmission electron microscopy (HRTEM) image of a β – FeSi₂ single-crystal with a size diameter of 4.8 nm ± 0.49 nm. Additionally, figure 7(c) shows the diffraction pattern obtained from the red square area indicated in figure 7(b). The β – FeSi₂ nanocrystal lattice was indexed to the [220] plane, with an interplane istance of 3.08 Å (JCPDS database, No. 01-071-0642). Our results show the formation of spherical nanocrystals with a monodisperse size distribution, with a diameter of approximately 5 nm, which is consistent with the PL signal obtained. Our method allows the fabrication of homogeneously shaped nanocrystals exhibiting an intense PL signal and therefore it can be competitive with other methods aiming for PL signal enhancements [29–33].

4. Conclusions

In this work, the formation of β – FeSi₂ nanostructured layers by low-energy and high-fluence Fe⁺ implantation into Si substrates has been demonstrated. In order to obtain a complete characterization of the samples, several different techniques such as Raman spectroscopy, AFM, SEM, EDS, TEM, SIMS and PL were used. In particular, the results obtained by SIMS suggest that the layer in which the β – FeSi₂ nanoparticles have formed is around 26.6 nm thick. The PL analysis showed that these nanoparticles emit in the near-infrared region, exhibiting a very narrow peak, with a FWHM of 1 nm, which is comparable with semiconductor laser emission. Therefore, this β – FeSi₂ nanostructured layer is potentially useful to develop near-IR lasers for fiber optics and other technological applications.

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