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Correlation between defect structure and luminescence spectra in monocrystalline erbium-implanted silicon

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Abstract
The transformation of structural defects and photoluminescence (PL) spectra of n- and p-type Cz-Si after implantation with erbium ions at 1 MeV energy to doses of 1×10^{13} and 1×10^{14} cm^{-2} followed by annealing at 620–1100 °C for 0.25–3.0 h in chlorine-containing atmosphere or oxygen have been studied by transmission electron microscopy, optical microscopy in combination with selective chemical etching, and PL. For the doses used, annealing at a temperature lower than 1100 °C leads to the formation of different extended defects (partial Frank or perfect prismatic dislocation loops) of submicron sizes which do not prevent the appearance of Er-related lines and do not give rise to dislocation-related lines in the PL spectrum. In contrast, high-temperature annealing at 1100 °C results in the development of similar three-dimensional networks of pure edge dislocations with a density of $\sim 10^7$ cm^{-2}. These dislocations are responsible for the appearance of quite intensive dislocation-related luminescence (DRL). For high-dose implantation, when annealing at 1100 °C is used, the same dislocation networks have been found to form in the n- and p-Si wafers with different low-temperature annealing stages (if any). However, all these parameters exert the peculiar influence upon the intensity of DRL lines.

1. Introduction

Fabrication of light-emitting structures with a wavelength of ~ 1.5 μm is of great interest for optoelectronic applications. The fabrication of structures with luminescence determined by the presence of dislocations in Si is one way of producing such structures. Traditionally,

dislocation-related luminescence (DRL) was studied in plastically deformed Si [1–3] and partly relaxed epitaxial SiGe/Si heterostructures [4, 5]. Later, DRL was observed in laser-melted Si layers [6] and liquid phase epitaxy Si films [7]. It is important to note that in all the objects the morphology of the dislocation patterns is significantly different and there are some differences in DRL spectra. This leads to a significant disagreement concerning the nature of the DRL. In particular, the pure edge dislocations, special dislocation configurations (kinks, jogs), and dislocation intersections are considered as luminescence centres responsible for DRL. Recently, we have observed DRL in silicon implanted with Er ions at doses both lower and higher than the amorphization threshold and annealed at relatively high temperature in a chlorine-containing atmosphere (CCA) [8–11]. The photoluminescence (PL) spectra under such conditions contain only two DRL lines with wavelengths of ~ 1.52 μm (D1 line) and ~ 1.42 μm (D2 line). High uniformity in the distribution of structural defects over the sample area and relatively simple morphology of dislocations open new possibilities for investigation of DRL. The purpose of this work was to study, for implantation doses lower and higher than the amorphization threshold, peculiarities of dislocation pattern formation in Si:Er during annealing as well as the influence of doping impurities and annealing regimes on the defect structure and PL spectra.

2. Experimental details

Erbium ions were implanted at 1 MeV energy and doses both lower (1×10^{13} cm^{-2}) and higher (1×10^{14} cm^{-2}) than the amorphization threshold in (100) Cz-Si at room temperature. Post-implantation annealing of the samples was carried out in a CCA which was an oxygen flow with the addition of tetrachloride carbon at a concentration of 0.5 mol%. Low-dose implantation to p-Si with a resistivity of 20 Ω cm and annealing at 1000 and 1100 °C for 0.25–3 h were carried out. For high-dose implantation, Er ions were implanted into n- and p-Si substrates and a three-step annealing at 620 °C/1 h + 900 °C/0.5 h + 1100 °C/1 h was used. The annealing procedure was carried out in such a way that, for some samples, the high-temperature stages were added by turns, and, in contrast, the early low-temperature steps were excluded by turns, for the other samples. In addition, one annealing was carried out at 1100 °C/1 h in oxygen. The PL studies were performed at the temperature of 77 K. Free carriers were excited by a mechanically chopped beam of a halogen lamp with 75 mW power. The radiation was collected by a lens, dispersed by a monochromator, registered by an InGaAs detector at room temperature, and amplified using a conventional lock-in technique. The structural defects were studied by transmission electron microscopy (TEM) on cross-sectional and plan-view samples and chemical etching/Nomarski microscopy on oblique sections.

3. Results and discussion

3.1. Implantation with Er ions at a dose of 1×10^{13} cm^{-2}

Annealing the samples at 1000 °C for 0.5 h leads to the formation of only small Frank loops (≈ 0.2 μm) with density of about 2×10^8 cm^{-2}. Frank loops are imperfect dislocation loops with an interstitial stacking fault on the $\langle 111 \rangle$ planes and the Burgers vector $\boldsymbol{b} = (a/3)\langle 111 \rangle$. The formation of such defects is supposed to be an initial stage of their evolution during the annealing. For the short annealing at 1100 °C, small Frank loops with a density of $\leqslant 10^8$ cm^{-2} together with small perfect dislocation loops ($\sim 5 \times 10^6$ cm^{-2}) are observed. As annealing time increases, the Frank loop size increases from 0.4 to 10 μm and their density gradually decreases by about one order of magnitude. During the annealing, Frank loops transform

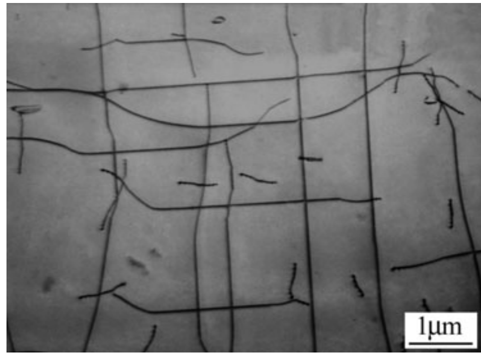

Figure 1. A plan-view TEM micrograph ($\boldsymbol{g} = \langle 400 \rangle$) of the dislocation network formed in a Si:Er sample after implantation with a 1×10^{13} cm^{-2} dose and annealing at 1100 °C for 3 h.

to perfect prismatic dislocation loops due to the nucleation of a Shockley partial dislocation at the stacking fault. Perfect dislocation loops lying mainly on $\{110\}$ planes are interstitial loops with the Burgers vector $\boldsymbol{b} = (a/2)\langle 110 \rangle$. Once nucleated, these loops gradually expand through the climb mechanism to cross each other and form large complex loops containing short segments of pure edge dislocation. Multiple intersections between perfect loops give rise to a three-dimensional dislocation network mainly composed of pure edge dislocations, which spreads from the surface to a depth of about 1 μm. A fragment of the well-developed dislocation pattern for the sample annealed at 1100 °C for 3 h is shown in figure 1.

For all the samples containing separate Frank loops or perfect loops, only Er-related luminescence lines are observed in PL spectra independently of the loop dimensions. These spectra are similar to those of Si:Er samples annealed in argon and containing no extended defects [7]. Low-intensity D1 and D2 lines appear in the samples annealed at 1100 °C when short segments of pure edge dislocation are generated at the intersecting perfect dislocation loops. During annealing for about 1 h, the dislocation network becomes well developed and tends to saturation of the dislocation density at a value of $\sim 10^7$ cm^{-2}. The intensities of the D1 and D2 lines increase in good correlation with the dislocation density and tend to saturation too. The characteristic peculiarity of the PL spectra is an absence of other DRL lines, in particular D3 and D4 lines.

3.2. Implantation with Er ions at a dose of 1 × 10^{14} cm^{-2}

The implantation with such a dose leads to the formation of a buried amorphous layer. In the transition regions between amorphous and crystalline (a–c) parts of silicon, microcrystallites slightly misoriented with respect to the bulk crystalline material are observed. Solid phase epitaxial (SPE) growth occurs during annealing at 620 °C from the two outer a–c interfaces towards the centre of the amorphous layer. A complex defect structure is formed in this case (figure 2). Near both a–c interfaces at the depths of ~ 0.25 and 0.5 μm, bands of Frank loops of high density are observed. Hairpin dislocations spread from the bands towards the middle of the regrown layer. Their formation is usually observed after SPE recrystallization of Si layers amorphized by Er-ion implantation at room temperature. There are other defects in the middle of the recrystallized layer where the two advancing interfaces meet. The nature of these defects is under investigation. Subsequent annealing at 900 °C for 0.5 h dissolves all the defects in the centre of the recrystallized layer and transforms the Frank loops into perfect dislocation loops. The loop sizes vary from 5 to 200 nm and their density is $\sim 2 \times 10^{10}$ cm^{-2}.

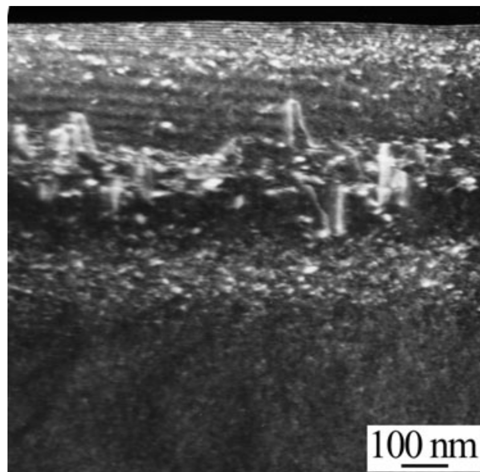

Figure 2. A cross-sectional TEM micrograph ($g = \langle 220\rangle$; dark field) of a Si:Er sample after implantation with 1×10^{14} cm^{-2} dose and SPE regrowth at 620 °C for 1 h. The sample is slightly turned around the $\langle 110\rangle$ crystallographic direction.

It is important to point out that the perfect dislocation loops do not prevent the appearance of Er-related luminescence lines and do not introduce DRL lines in the PL spectrum. After additional annealing at 1100 °C for 1 h, the dislocation network as well as perfect prismatic dislocation loops are observed. The three-dimensional dislocation network distributes from the surface to a depth of $\sim$1 μm and consists predominantly of pure edge dislocations and a small proportion of 60° dislocations. The 60° dislocations and small perfect dislocation loops are detected near the surface only. The pure edge dislocation density in the middle of the recrystallized layer was estimated, taking account of the etch pits, to be $\sim 10^7$ cm^{-2}. The transformation of structural defects and the formation of the three-dimensional network of pure edge dislocations occur identically in n- and p-Si wafers. The existence of pure edge dislocations in the appropriate samples correlates with the appearance of D1 and D2 lines in the PL spectra.

To study the influence of annealing conditions and doping impurities on the defect structure and optical properties, we additionally varied the temperature of SPE regrowth using in parallel n- and p-type substrates (see table 1). TEM investigations have shown that practically identical dislocation structure with a characteristic density of pure edge dislocations equal to $\sim 10^7$ cm^{-2} forms in all the samples if their last heat treatment is carried out at 1100 °C in an oxidizing atmosphere (CCA and pure oxygen). A typical dislocation pattern for all the samples from table 1 is shown in figure 3 for a p-Si:Er sample recrystallized at 1100 °C/1 h.

Essential changes in the DRL line intensity under the variation of experimental conditions are observed in contrast to the case for the defect structure (table 1). For all pairs of samples with identical annealing conditions, the intensity of the D1 lines for n-Si samples is about double that for p-Si samples (figure 4). To determine the dominant factor(s) responsible for the effect, further studies on the nature of optically active centres and the mechanisms of the DRL excitation should be carried out. Note that the same influence of the conductivity type of Si substrates on luminescence intensity was observed for DRL in plastically deformed Si [12]. A more than double decrease of the D1 line intensity with increasing temperature of SPE regrowth (first annealing stage) was found (table 1; samples 1, 3, and 4). This effect may be associated with the production of additional structural defects, being a channel of

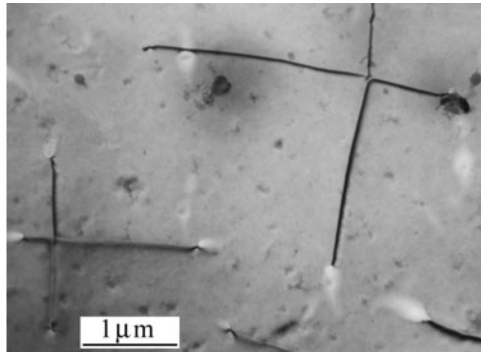

Figure 3. A plan-view TEM micrograph ($g = \langle 400 \rangle$) of the dislocation network formed in the Si:Er sample (number 4) after implantation with 1×10^{14} cm^{-2} dose and annealing at 1100 °C for 1 h.

Table 1. Annealing conditions and the intensity of the D1 line (I) for Si implanted with Er ions at 1 MeV energy and 1×10^{14} dose.

		Annealing conditions				
		Temperature (°C)/duration (h)				
Sample	Conductivity type	620/1.0	900/0.5	1100/1.0	Ambient of 1100 °C annealing	I (a.u.)
1	n	+	+	+	CCA	21.6
1x	p					9.2
2	n	+	+	+	Oxygen	13.8
2x	p					5.8
3	n	−	+	+	CCA	8.1
3x	p					5.1
4	n	−	−	+	CCA	8.0
4x	p					3.8

nonradiative recombination. For example, microtwins were observed to be generated in (100) Si implanted with Er ions due to an increase of the SPE recrystallization temperature from 620 to 1000 °C [13]. Heat treatment in CCA at 1100 °C increases the D1 line intensity by up to 30% for both n- and p-Si in comparison with annealing in oxygen (table 1; samples 1 and 2). The effect may be related to the gettering of impurity atoms or other defects, which may be additional channels of nonradiative recombination, and decrease the efficiency of the DRL excitation.

4. Summary

Structural and optical properties of n- and p-Si implanted with Er ions and annealed in an oxidizing ambient were studied. We demonstrated that, in spite of the fact that the nucleation and evolution of extended defects during post-implantation annealing occur in different ways for the nonamorphized and amorphized Er-implanted layers, the dislocation network develops similarly during the high-temperature annealing at 1100 °C: perfect dislocation loops cross each other during their extension; and multiple interactions between the perfect loops give rise to the three-dimensional network with the density of $\sim 10^7$ cm^{-2} of pure edge dislocations. So, light-emitting structures with quite intensive DRL as well as relatively simple and sufficiently

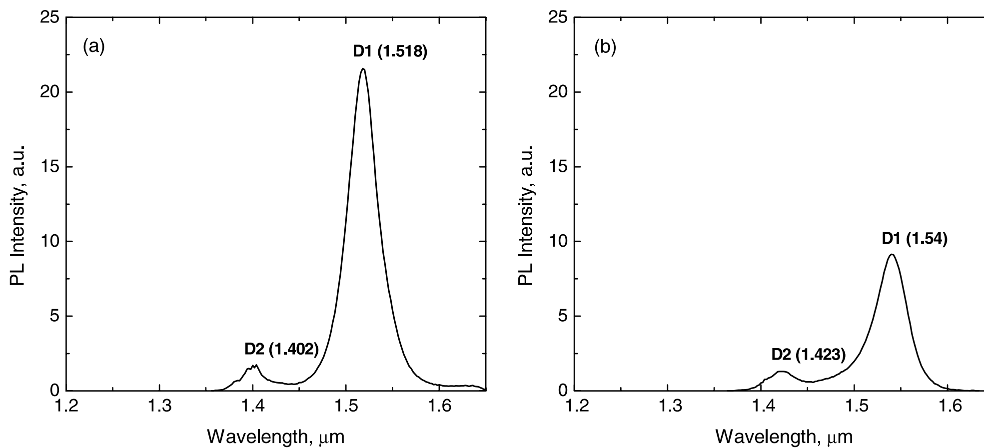

Figure 4. PL spectra measured for n-Si (a) and p-Si (b) annealed under identical conditions (samples 1 and 1x from table 1).

reproducible dislocation patterns can be fabricated by the implantation technique with the Er-ion doses lower than the amorphization threshold. For the SPE samples annealed with the use of a high-temperature stage at 1100 °C, we have found that the intensity of the D1 and D2 lines is sensitive to the type of Si substrate doping as well as to the annealing in early low-temperature stages, whereas the dislocation network is the same in the samples. The experimental results lead us to suppose that DRL is associated with the formation of pure edge dislocations, but the intensity of D1 and D2 lines can be determined by some defects distributed in the Si matrix close to the dislocations.

Acknowledgments

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