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Role of heavy ion co-implantation and thermal spikes on the development of dislocation loops in nanoengineered silicon light emitting diodes

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Microstructural and electroluminescence measurements are carried out on boron implanted dislocation engineered silicon light emitting diodes (LEDs) co-implanted with the rare earth thulium to provide wavelength tuning in the infra-red. Silicon LEDs operating in the range from 1.1–1.35 μm are fabricated by co-implantation of boron and thulium into n-type Si (100) wafers and subsequently rapid thermally annealed to activate the implants and to engineer the dislocation loop array that is crucial in allowing light emission. Ohmic contacts are applied to the *p* and *n* regions to form conventional p-n junction LEDs. Electroluminescence is obtained under normal forward biasing of the devices. The influence of implantation sequence (B or Tm first), ion dose, and the post-implantation annealing on the microstructure and electroluminescence from the devices is studied. A clear role of the heavy-ion Tm co-implant in significantly modifying the boron induced dislocation loop array distribution is demonstrated. We also identify the development of dislocation loops under thermal spikes upon heavy ion (Tm) implantation into Si. The results contribute to a better understanding of the basic processes involved in fabrication and functioning of co-implanted devices, toward achieving higher light emission efficiency. © 2011 American Institute of Physics. [doi:10.1063/1.3614036]

I. INTRODUCTION

The development of integrated light emitting devices and in particular optical amplifiers in silicon are the last remaining challenge for all-silicon photonics platforms and for inter and intra-chip optical data transfer. Because of the silicon indirect gap, conventional degenerately doped laser diodes do not work. An approach suggested to achieve this is the incorporation in silicon light emitting devices of rare earth centers that can emit in the infrared and are capable of supporting lasing.^{1–4} Of these Tm has recently emerged as an exciting contender. The incorporation of Tm leads to emission of light over two important wavelength regions, 1.2–1.4 μm and 1.7–2.1 μm , which can both support lasing and indeed form the basis of commercially available, optically pumped lasers. In addition, the electrical excitation of the Tm emission has been demonstrated in silicon light emitting diodes under ordinary forward bias⁴ and, most recently,⁵ room temperature luminescence has been reported at the important, eye-safe, 2 μm wavelength. Significantly, efficient Tm emission in silicon is only achieved in samples co-implanted with B, when dislocation loops are introduced. The controlled introduction of dislocation loops into a device active region is essential to achieve efficient light emission, as their location, distribution, and size are directly correlated to the luminescence intensity. Indeed, interstitial dislocation loops induce a local strain—negative hydrostatic pressure outside the loops and positive hydrostatic pressure inside the loops; the net strain is zero. Due to the negative pressure coefficient of the indirect gap, the silicon bandgap energy

just outside the loops is increased by up to 0.75 eV. This bandgap increase outside the loops, which the injected carriers experience first, forms a potential barrier suppressing onward diffusion to non-radiative recombination centers at the surface, in favor of radiative transitions in the bulk.⁶ The boron implant and post-anneal conditions play a crucial role on the loops' microstructure. Nevertheless, the interaction of the two implants, Tm and B, is not trivial and optimizing their interaction is essential to improve the device performance. The crucial role of partial and full amorphization on the loop development at higher Tm doses is established. The important role of thermal spikes (a narrow cylindrical zone, up to ~ 10 nm, around the ion trajectory, where the local temperature can be raised up to a few thousand K for 1–100 ps in the process of energy loss of the impact projectile)⁷ due to the high mass thulium implant in modifying the device microstructure is identified. The generated thermal spikes induce ripening and growth of dislocation loops, equivalent to a prolonged thermal annealing. Thermal spikes are prominent in cases of heavy target materials and/or heavy ion projectiles ($Z \geq 20$);⁸ there is also evidence of the influence of ion induced thermal spikes in broadening of tracer profiles in silicon⁹ and numerous reports on the grain growth in polycrystalline metallic films upon ion irradiation.^{10–14} Nevertheless, nucleation and growth of extrinsic crystalline defects (dislocation loops in this case) in a host material under ion induced thermal spikes has not yet been reported.

II. EXPERIMENTAL DETAILS

The light emitting devices were fabricated by alternate implantation of 30 keV B⁺ and 400 keV Tm⁺ ions into

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n-type Si (100) wafers (2–7 Ωcm). The B implant, in addition to inducing the formation of dislocation loops, also provided *p*-type doping in Si. The Tm implanted doses ranged from 10^{12} to 10^{15} cm^{-2} , at $\sim 0.5\text{ }\mu\text{A cm}^{-2}$, while B was implanted to a constant dose of 10^{15} cm^{-2} , at $\sim 1\text{ }\mu\text{A cm}^{-2}$. Projected range of 400 keV Tm in Si is $R_p = 135\text{ nm}$ (straggle $\Delta R_p = 33\text{ nm}$), and of 30 keV B is $R_p = 110\text{ nm}$ (straggle $\Delta R_p = 39\text{ nm}$), as calculated by stopping and range of ions in matter (SRIM).¹⁵ During implantation the targets were held at room temperature, and inclined by 7° off normal to avoid channeling. In some cases the first implanted species was B and in others it was Tm. After both co-implants were done, the samples were annealed in ultra large scale integration grade (ULSI) nitrogen ambient, to prevent silicon oxidation, at 850 and 950 $^\circ\text{C}$, for 1–15 min. The diodes were fabricated by vacuum deposition of Al and AuSb eutectic on the *p*-type region and *n*-type substrate, respectively, to form ohmic contacts, and sintered at 360 $^\circ\text{C}$ for 2 min. A window was left open on the substrate side to enable the electroluminescence measurements. A mesa etch was used to isolate the *p*-*n* junction; the device area was $8 \times 10^{-3}\text{ cm}^2$. A summary of ion implantation and annealing processing of the samples is given in Table I.

Structural characterizations of the samples were done by transmission electron microscopy, using Philips EM400T, Philips CM200, and Hitachi HD2300A (STEM) microscopes. The samples were prepared for cross-sectional and plan-view analysis by ion beam milling. Electroluminescence measurements were performed at 80 K under forward bias of $\sim 1\text{ V}$ and current density of 1.25 A cm^{-2} . The devices were mounted in a continuous-flow liquid nitrogen cryostat placed in front of a conventional halfmeter spectrometer. Light was detected by a liquid nitrogen cooled Ge *p*-*i*-*n* diode and processed by a conventional lock-in amplifier.

III. RESULTS AND DISCUSSION

A. Tm dose dependence of the microstructure and electroluminescence

Interstitial dislocation loops created by ion implantation sit in $\{111\}$ Si habit planes.^{16,17} Viewed in cross section, along $[110]$ Si, they appear either as elongated oval shapes or as straight lines inclined to the surface normal. In plan view, along $[001]$ Si, they appear as oval shapes, which can be rather irregular for larger ones that consist of several loops collided in the process of their ripening. Figure 1 shows bright-field cross-sectional and corresponding plan-

FIG. 1. Bright-field cross-sectional and plan-view TEM images taken from Si samples first implanted with B to $10^{15}\text{ ions cm}^{-2}$, and subsequently with Tm at different doses: (a) 10^{12} , (b) 10^{13} , (c) 10^{14} , and (d) $10^{15}\text{ Tm cm}^{-2}$. All samples were annealed for 15 min at 850 $^\circ\text{C}$.

view transmission electron microscopy (TEM) images taken from a series of Si samples first implanted with 10^{15} B cm^{-2} and subsequently with Tm to 10^{12} – 10^{15} cm^{-2} , followed by annealing for 15 min at 850 $^\circ\text{C}$ (samples A–D from Table I). In the sample implanted to $10^{12}\text{ Tm cm}^{-2}$ (Fig. 1(a)) we observe relatively small (up to $\sim 35\text{ nm}$ diameter) dislocation loops distributed around the projected range of previously implanted boron. The loops that developed in the sample implanted to $10^{13}\text{ Tm cm}^{-2}$ (Fig. 1(b)) are still located around the boron projected range but they are considerably larger (some irregularly shaped ones are above 200 nm long) than in the previous case. The sample implanted to $10^{14}\text{ Tm cm}^{-2}$ (Fig. 1(c)) exhibits two distinct groups of loops: a deep row of collimated small loops at a depth of $\sim 200\text{ nm}$, induced by the Tm implantation, and larger shallow loops toward the surface, due to the B implantation. In the sample implanted to $10^{15}\text{ Tm cm}^{-2}$ (Fig. 1(d)) we only see a deep row of small loops, due to the Tm implantation, at a depth of $\sim 250\text{ nm}$, and some residual damage in Si toward the surface.

The implantation of 30 keV B to 10^{15} cm^{-2} is far below the amorphization threshold in Si.^{16,17} Upon post-implantation annealing at 850–950 $^\circ\text{C}$ the excess Si species nucleate

TABLE I. Sample details.

Sample	1 st Implant (ions/cm ²)	2 nd Implant (ions/cm ²)	Post-implant anneal ($^\circ\text{C}/\text{min}$)
A	10^{15} B	10^{12} Tm	850/15
B	10^{15} B	10^{13} Tm	850/15
C	10^{15} B	10^{14} Tm	850/15
D	10^{15} B	10^{15} Tm	850/15
E	10^{15} B	10^{13} Tm	950/1
F	10^{13} Tm	10^{15} B	950/1

and grow in the form of interstitial dislocation loops around the projected boron range. The dislocation loops that formed after 10^{12} and 10^{13} Tm cm $^{-2}$ irradiation are similar to those observed previously in Si substrates implanted with boron only and subsequently annealed.¹⁶ The TEM results imply that Tm ion implantation induces two effects in the Si substrate: (1) partial annealing due to thermal spikes, and (2) crystal damage with partial or full amorphization of Si for the two highest Tm doses. Indeed, by comparing the images from samples implanted at 10^{12} and 10^{13} Tm cm $^{-2}$ we observe much larger loops for the 10^{13} Tm cm $^{-2}$ sample, although both samples had the same post-implantation annealing. This suggests that ripening of loops occurred during implantation to the higher Tm dose. The collimated rows of deep loops seen in Figs. 1(c) and 1(d) are typical for those created at the original amorphous/crystalline (a/c) interface when the Si substrate was previously amorphized.^{18–20} However, the Tm dose of 10^{14} ions cm $^{-2}$ (Fig. 1(c)) caused only partial amorphization of Si, forming a buried amorphous layer around the implanted ion range. Hence, upon annealing the deep loops nucleate at the original inner a/c interface, and the shallow loops near the surface develop due to the previous B implantation in the region where Si was not amorphized. A similar formation of two rows of dislocation loops was observed after annealing of a structure containing a buried amorphous layer created in self-implanted Si.²¹ For the Tm dose of 10^{15} ions cm $^{-2}$ (Fig. 1(d)), the Si substrate was fully amorphized, deeper than in the previous case, up to around the end of range of the implanted Tm species. The deep loops formed upon annealing are so-called end of range (EOR) loops, located at the original a/c interface, and the loops due to B implantation did not develop in the process of Si recrystallization. Partial or full amorphization of the Si substrate due to high Tm doses (10^{14} and 10^{15} ions cm $^{-2}$) was confirmed by cross-sectional analysis of the corresponding samples before post-implantation annealing.

The electroluminescence (EL) responses of the corresponding diodes are shown in Fig. 2. Samples implanted to 10^{12} (A) and 10^{13} (B) Tm cm $^{-2}$ exhibit sharp EL peaks originating from Si (1.15 μ m) and six originating from Tm (1.233,

FIG. 2. (Color online) Electroluminescence response of diodes corresponding to the structures shown in Fig. 1. The implanted dose (Tm cm $^{-2}$) is also indicated.

1.251, 1.269, 1.291, 1.311, and 1.326 μ m). In the sample implanted to 10^{14} Tm cm $^{-2}$ (C) we register only a weak peak from Si, while in the one implanted to 10^{15} Tm cm $^{-2}$ (D) there is no EL response. The Si EL yield in (A) is higher than in (B), because the loops are smaller and denser.¹⁶ On the other hand, the Tm EL yield is higher in (B) compared to (A) because of the higher implanted Tm dose. The broad background (1.35–1.60 μ m) is attributed to B implantation induced damage.³ In sample (C) the deep small loops are located beneath the active region of the device, while the shallow loops are far above, and hence only lead to weak EL. Again, in (D) the small deep loops are beneath the active region of the device and have no influence on EL properties.

B. Tm and B implant sequence dependence of the microstructure and electroluminescence

Figure 3 shows bright-field cross-sectional and plan-view TEM images taken from two samples where the B and Tm implantation sequence was swapped (samples E and F from Table I). In Fig. 3(a) the first implantation was done with 10^{15} B cm $^{-2}$, followed by implantation of 10^{13} Tm cm $^{-2}$. In Fig. 3(b) the implantation sequence was inverted: 10^{13} Tm cm $^{-2}$ followed by 10^{15} B cm $^{-2}$. After the co-implants, both samples were annealed under the same conditions, for 1 min at 950 °C. It can be seen that the implantation sequence has a striking influence on the development of dislocation loops. While in Fig. 3(b) the loops are practically in the initial stage of nucleation and growth (up to ~ 20 nm in diameter), in Fig. 3(a) they are fully developed (up to ~ 250 nm). This analysis provides further evidence about the influence of thermal spikes generated upon Tm implantation on the development of dislocation loops. Indeed, the size of the loops developed in Fig. 3(a) is equivalent to loops developed in samples implanted with boron only and annealed at the same temperature for 10 min or longer.¹⁶ The EL

FIG. 3. Bright-field cross-sectional and plan-view TEM images taken from samples with swapped implantation sequence: (a) initial B implant followed by Tm implant and (b) initial Tm implant followed by B implant. All samples were implanted at the same energy and dose of particular ions and subjected to the same post-implant anneal conditions.

FIG. 4. (Color online) Electroluminescence response of diodes corresponding to the structures shown in Fig. 3.

response of these devices is shown in Fig. 4. Both samples exhibit sharp EL peaks that originate from Si and Tm. In the sample with well-developed large loops (Fig. 3(a)), where B was the first implant, the silicon EL yield is higher. In the sample where the development of dislocation loops is in the initial stage (Fig. 3(b)), where B was the second implant, the silicon EL yield is rather lower, but the EL yield from Tm is practically at the same level.

C. The formation and location of dislocation loops

Figure 5 shows high resolution TEM (HRTEM) cross-sectional images of samples incorporating dislocation loops. The interstitial nature of dislocation loops is illustrated by the high resolution image in Fig. 5(a), where we see an inserted row in the lattice, viewed along $[110]$ Si. The image in Fig. 5(b) was taken from a sample implanted to 10^{13} Tm cm^{-2} after previous implantation of B but prior to the post-implantation annealing. It confirms the previously discussed initial formation of dislocation loops due to thermal spikes induced in Si by heavy Tm ion irradiation. In order to locate the position of Tm implanted species in the host Si lattice and their eventual clustering, apart from high-resolution TEM analysis, the samples were subjected to detailed Z-contrast imaging and electron energy loss spectroscopy (EELS).

FIG. 5. (a) HRTEM cross-sectional image along $[110]$ Si illustrating the interstitial nature of dislocation loops; (b) dislocation loops developed after Tm implantation in samples initially implanted with B but before a post-implant anneal.

However, no evidence could be observed either of the lattice occupancy or eventual clustering of Tm.

IV. CONCLUSIONS

The presented results demonstrate the importance of correct positioning of dislocation loops to enable efficient electroluminescence in Si LEDs, and the effect of Tm implanted dose. The loops that developed in the efficient devices are due to B implantation. It is well known that electrically active B species occupy substitutional sites in the Si lattice, and hence for the implanted dose of 10^{15} B cm^{-2} there is a corresponding number of Si interstitials that nucleate in the form of extrinsic dislocation loops. After initial nucleation the loops develop by the Ostwald ripening mechanism, increasing in size and decreasing in density with annealing time, but the total number of Si interstitials trapped in the loops remains constant.¹⁹ The lattice occupancy of the Tm species could not be evaluated at this stage, but the implanted doses in the efficient devices (10^{12} and 10^{13} Tm cm^{-2}) are considerably lower than that of B, so even if all Tm became substitutional the corresponding number of Si interstitials would be negligible compared to that induced by B. However, contrary to B, much heavier Tm ions induce amorphization of Si already for the doses of 10^{14} and 10^{15} Tm cm^{-2} . Creation of the amorphous zone suppresses formation of dislocation loops due to B implants, and invokes the formation of EOR loops at the end of the Tm ion distribution range. These loops are positioned below the active region of the devices and consequently the electroluminescence remains quenched. A significant phenomenon observed is the development of dislocation loops due to thermal spikes induced in Si upon implantation of the heavy Tm ions. The generated thermal spikes resulted in ripening and growth of dislocation loops, as if the samples have had a prolonged thermal annealing. This is particularly important as the time and temperature to form the optimum dislocation loop array must take this effect into account to minimize the luminescence quenching. Other rare earth dopants used for wavelength tuning of silicon LEDs have very similar masses and so these results will also be applicable to those systems.

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