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Disentangling nonradiative recombination processes in Ge micro-crystals on Si substrates

Fabio Pezzoli,^{1,a)} Anna Giorgioni,¹ Kevin Gallacher,² Fabio Isa,^{3,4,b)} Paolo Biagioni,⁵ Ross W. Millar,² Eleonora Gatti,¹ Emanuele Grilli,¹ Emiliano Bonera,¹ Giovanni Isella,³ Douglas J. Paul,² and Leo Miglio¹

¹LNESS and Dipartimento di Scienza dei Materiali, Università degli Studi di Milano-Bicocca, via Cozzi 55, I-20125 Milano, Italy

²School of Engineering, University of Glasgow, Rankine Building, Oakfield Avenue, Glasgow G12 8LT, United Kingdom

³LNESS, Dipartimento di Fisica del Politecnico di Milano and IFN-CNR, Polo Territoriale di Como, Via Anzani 42, I-22100 Como, Italy

⁴Laboratory for Solid State Physics, ETH Zurich, Otto-Stern-Weg 1, CH-8093 Zürich, Switzerland

⁵LNESS, Dipartimento di Fisica del Politecnico di Milano and IFN-CNR, Piazza Leonardo da Vinci 32, I-20133 Milano, Italy

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We address nonradiative recombination pathways by leveraging surface passivation and dislocation management in μm -scale arrays of Ge crystals grown on deeply patterned Si substrates. The time decay photoluminescence (PL) at cryogenic temperatures discloses carrier lifetimes approaching 45 ns in band-gap engineered Ge micro-crystals. This investigation provides compelling information about the competitive interplay between the radiative band-edge transitions and the trapping of carriers by dislocations and free surfaces. Furthermore, an in-depth analysis of the temperature dependence of the PL, combined with capacitance data and finite difference time domain modeling, demonstrates the effectiveness of GeO_2 in passivating the surface of Ge and thus in enhancing the room temperature PL emission. *Published by AIP Publishing.*

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The monolithic development of Si photonic components addresses the hurdles of charged-based computing and is expected to drastically transform information and communication technologies. The progress of Si photonics, however, has been jeopardized by fundamental limitations. The centrosymmetric crystal structure and the indirect nature of the band-gap of bulk Si imply weak electro-optical effects alongside poor absorption and emission efficiencies.¹ These obstacles have stimulated the exploration of wafer-scale fabrication methods for utilizing materials better suited for high-performance photonic circuitry.^{2,3} In this respect, direct epitaxial growth of Ge on Si substrates is a sought-after solution because Ge, being non-polar and isoelectronic with Si, turns out to be compatible with conventional microelectronic processes yielding high-volume throughput.⁴

Another crucial benefit is that Ge, an indirect band-gap semiconductor, offers an unexpectedly strong light-matter interaction.⁵ The close energy proximity of ≈ 140 meV between the fundamental conduction band minimum at the L point of the Brillouin zone and the local minimum at the zone center Γ guarantees optical access to the direct band-gap properties: a prerequisite for the fabrication of high performance photonic devices.^{6–8} Moreover, heteroepitaxial growth encompasses strain and alloying.⁹ These two degrees of freedom were leveraged for decreasing the Γ -to- L offset in the recent demonstrations of tensile-Ge^{10,11} and GeSn¹² injection lasers on Si,

eventually turning Ge even closer to a direct band gap material. All these efforts proved Ge heteroepitaxy to be viable, but at the same time heralded very poor emission efficiencies. Epitaxial growth of Ge on Si faces severe material and manufacturing issues due to the large mismatch in the lattice constants and the thermal expansion coefficients. The former leads to nucleation of dislocations, while the latter causes wafer bowing and cracking.^{9,13} As a consequence, there is an urgent need to find reliable solutions to the ubiquitous nonradiative recombination of carriers at such growth defects and free surfaces, which considerably compromise the electronic and optical properties.^{9,14,15}

In the quest to achieve Ge-based architectures as lossless optical components and to resolve the subtleties of the carrier dynamics, we exploit the out-of-equilibrium growth of Ge on deeply patterned Si substrates.¹⁶ Figure 1(a) demonstrates a scanning electron microscope micrograph of as-grown μm -scale crystals developed by depositing $8\ \mu\text{m}$ of Ge at $550\ ^\circ\text{C}$ by low-energy plasma enhanced chemical vapor deposition¹⁷ onto $2 \times 2\ \mu\text{m}^2$ Si pillars. Such pedestals were separated by $8\text{-}\mu\text{m}$ -deep and $3\text{-}\mu\text{m}$ -wide trenches patterned onto (001) substrates by optical lithography and reactive ion etching.¹⁶ The Ge micro-crystals are characterized by a pyramidal top surface bounded by $\{113\}$ and $\{111\}$ facets. Such facets stem from a slanted growth front that promptly steers the threading arms towards the lateral sidewalls, where they become pinned. Subsequent material deposition eventually yields a region of the crystal that is completely free from dislocations, although no annealing was performed.^{16,18,19} Such an approach has been shown to

^{a)}fabio.pezzoli@unimib.it

^{b)}Present address: Electron Microscopy Center, Empa Swiss Federal Laboratories for Materials Science and Technology, Überlandstrasse 129, CH-8600 Dübendorf, Switzerland

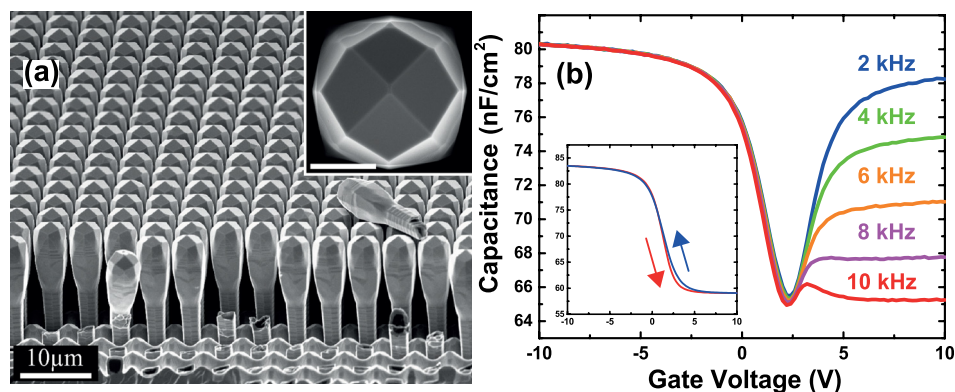


FIG. 1. (a) A cross-sectional scanning electron microscope image of the as-grown μm -scale crystals developed by depositing $8\ \mu\text{m}$ of Ge onto $2 \times 2\ \mu\text{m}^2$ Si pillars. (b) The room temperature capacitance versus voltage (C-V) characteristics as a function of low frequency (2–10 kHz) for a Pt/Si₃N₄/GeO₂/Ge capacitor. The inset shows the room temperature high frequency (1 MHz) bidirectional C-V curves.

produce space-filling arrays of Ge featuring (i) a giant enhancement in the internal quantum efficiency caused by the removal of dislocations threading through the whole epitaxial layer and (ii) an improved light extraction enabled by total internal reflection at the sidewalls of the micro-crystals.^{20,21}

Beside the as-grown and the passivated Ge micro-crystals, in this work, we selected a third sample that mimics the unpassivated micro-crystals except it encapsulates three additional Si_{0.25}Ge_{0.75} films at 2, 4, and 6 μm from the top surface, each with a thickness of 10 nm. These coherent SiGe layers are introduced to suppress the carrier diffusion towards the bottom dislocated region of the micro-crystals close to the Ge/Si interface.²⁰ In this letter, we thus utilize dislocation management provided by the Ge micro-crystals while passivating surface traps by means of conformal deposition of dielectric layers. By doing so, we can control and modulate the weight of the nonradiative recombination pathways and gather selective access to the carrier loss occurring either at extended defects or surface states in various temperature regimes. Aside from fruitful applications utilizing Ge as a photonic building block, pinpointing the parasitic sinks of charge carriers provides an in-depth knowledge of the physics of the recombination dynamics at the semiconductor interfaces, which is crucial also as a guide to design advanced semiconductor heterostructures that fulfill ever-demanding photonic and electronic functions.

Surface passivation of the Ge micro-crystals was obtained by growing a high quality thermal GeO₂ layer.²² The Ge micro-crystal surfaces were first cleaned in acetone followed by rinsing in isopropanol before cyclic hydrofluoric acid/deionized water etching was performed to remove any native oxide films. The Ge micro-crystals were then immediately transferred to a furnace tube oven for thermal oxidation at 550 °C with O₂ gas for 60 min, which results in a GeO₂ thickness of about 20 nm. The GeO₂ was then subsequently capped with 50 nm of unstrained ICP-PECVD Si₃N₄.²³

To evaluate the quality of the thermally grown GeO₂, planar capacitor structures consisting of Pt/Si₃N₄/GeO₂/Ge were fabricated in parallel on the (001) surface. Figure 1(b) demonstrates the room temperature capacitance-voltage (C-V) characteristics of the GeO₂ capacitor structures at low frequencies (2–10 kHz). The well behaved threshold voltage response as a function of frequency indicates that there is a low level of interface trapped charge between the GeO₂ and Ge. The inset in Fig. 1(b) shows the high frequency (1 MHz) bidirectional C-V curves. There is no flat-band voltage shift

observed from the ideal that was calculated from the work function of Pt ($\approx 5.6\ \text{eV}$), suggesting there is negligible fixed charge present within the dielectric stack. There is a relatively small flatband voltage hysteresis observed ($\approx 300\ \text{mV}$), which is still consistent with high quality GeO₂/Ge interfaces.^{24,25} All these observations indicate an efficient electrical passivation of the dangling bonds on the surface of Ge.

In addition to the C-V investigation, we have also carried out photoluminescence (PL) decay measurements of the indirect band-gap transition to gather direct insights about the effectiveness of the surface passivation on the optical properties of the Ge micro-crystals. The exceedingly long radiative lifetime expected in an indirect gap material such as Ge is typically concealed by the presence of competitive nonradiative events occurring at the defect sites, which appreciably shorten the observable lifetime of minority carriers.²⁶ Therefore, time-resolved PL offers us a very sensitive probe of the recombination dynamics and provides a direct method to identify the carrier loss mechanisms. Notably, we expect that the diffusion length allows for an almost uniform excess carrier density profile through the entire volume of the micro-crystals²⁰ so that, to first order, the changes in the recombination rates at facets with different crystallographic orientations can be neglected.

The samples were placed inside a closed-cycle cryostat at a temperature of 14 K and were excited by the 1064 nm line of a Nd:YAG Q-switched laser. The beam diameter on the sample was of about 500 μm . The repetition frequency was 10 kHz with a temporal width of the laser pulse of about 10 ns and an estimated mean power density on the sample surface ranging from 1 to 22.5 W cm⁻². The PL was collected by a single grating monochromator with a spectral bandpass set to 1.5 meV at 0.737 eV, which corresponds to the highest energy peak of the indirect gap PL band, as clarified by low temperature continuous-wave PL data of the as-grown sample shown in the inset of Fig. 2. The emission was detected by a photomultiplier tube (PMT) operated in a single photon counting mode with a precision for the time decay of 0.3 ns. It should be noted that the weakening of the PL signal and the spectral position of the emission energy, being close to the detectivity cut-off of the PMT, prevented us from reliably implement time decay measurements above cryogenic temperatures.

Auger-mediated recombination can affect the carrier lifetime at an early stage of the recombination dynamics because of the initial high density of photogenerated carriers.

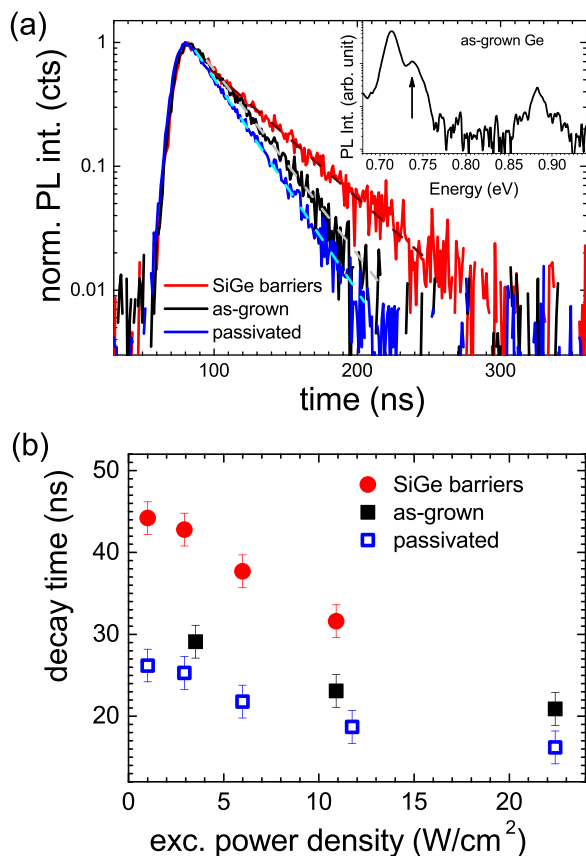


FIG. 2. (a) The PL decay curves measured at a temperature of 14 K and an average excitation power density of about 3 W/cm^2 for the as-grown (black line) Ge micro-crystals and for the micro-crystals either passivated with the oxide coating (blue line) or embedding three SiGe barriers (red line). The dashed lines are the fit of the decay curves. The inset shows a low temperature continuous-wave PL spectrum for the as-grown sample and the arrow points to the energy of the indirect band-gap peak used for the PL decay measurements. (b) The power dependence of the electron lifetime extracted from the PL decay data for the three samples: as-grown (black squares), passivated (blue open squares), and micro-crystals blending SiGe barriers (red dots).

At a low excitation power density, its weight becomes almost negligible, as demonstrated by the well-defined exponential decay observed in Fig. 2(a). Surprisingly, the decay curves for both the as-grown and passivated samples demonstrate a similar slope, that is a comparable lifetime for electrons residing in the L -valley. The slowest non-Auger-mediated decay times, summarized in Fig. 2(b), approach 30 ns and decrease by increasing the excitation power density, as a result of the local heating of the sample caused by the pulsed excitation. The negligible effect of the oxide coating on the carrier lifetime clearly demonstrates that, despite the large surface-to-volume ratio of the Ge micro-crystals, surface traps are not the most probable recombination centers at low temperature. This observation suggests the existence of competing mechanisms that are more effective than surface states in the capture of the injected carriers.

To provide insight into the origin of such parasitic recombination, we address here the Ge micro-crystals that embed the SiGe barriers. In this case, the strain and Ge molar fractions have been suitably designed in order to provide a band off-set between the alloy layers and the Ge matrix that impedes the diffusion of the photogenerated carriers towards the defective Ge/Si interface.²⁰

Remarkably, the insertion of the SiGe barriers yields the slowest decay among those reported in Fig. 2(a) lengthening the electron lifetime up to $\approx 45 \text{ ns}$ [see Fig. 2(b)]. This exceeds the literature data reported for Ge on Si heterostructures,^{14,27–29} and provides the first direct proof put forward in any recent work,²⁰ that band-gap engineered SiGe/Ge architectures are effective in mitigating the optical activity of buried dislocations.

At higher lattice temperatures, dislocations are nevertheless expected to play a less prominent role because of the augmented ionization probability of their associated localized energy levels.³⁰ To clarify this physical picture, we carried out additional PL measurements by using the 1.165 eV line of a CW Nd:YVO₄ laser. The laser spot size on the sample surface has a diameter of about $100 \mu\text{m}$, resulting in an exciting power density of about 1 kW/cm^2 . The PL was analyzed using a Fourier transform spectrometer equipped with Peltier cooled PbS detector.

Figure 3(a) reports a notable deviation from the standard temperature dependent PL quenching. As the lattice temperature increases, there exists a sharp threshold, with an activation energy of about 26 meV, above which a marked strengthening of the emission for both the as-grown and passivated sample can be observed. Such PL behavior is a hallmark of valley repopulation induced by thermal emission of carriers from the defect sites.^{20,31} Remarkably, the passivation results in a pronounced increase in the high temperature regime, yielding a room temperature PL spectrum with nearly twice the emission intensity of the as-grown Ge micro-crystals. As a comparison, we note that insertion of the SiGe barriers leads to less than 10% increase in the integrated PL intensity as compared to the as-grown Ge micro-crystals.²⁰ These results demonstrate the effectiveness of the oxide coating in mitigating the nonradiative capture by surface states and it reconciles the optical investigation with the results of the electrical characterization discussed above.

Finally, in order to study the possible role of the oxide as an anti-reflection coating and to clarify whether the room temperature PL enhancement is affected by photonic effects, e.g., Purcell's effect, we benchmarked our experimental data against finite-difference time-domain simulations.^{20,32,33} The steady-state PL process is mimicked by combining three simulation steps: (i) absorption of light by the Ge micro-crystals at the pump energy; (ii) local incoherent emission due to the recombination of electron-hole dipoles; and (iii) propagation of the emitted photons to the far field and their detection within the collection angle of the optics.³³ For the first step, we simulated the illumination of the Ge micro-crystals array with a Gaussian beam at 1.165 eV with an incidence angle resembling the actual experimental geometry. We then numerically evaluated the net flux of the Poynting vector entering the Ge micro-crystals and determined the total power absorption. As a second step, in order to mimic the incoherent emission process, we ran about 100 independent simulations, each one with a different randomly oriented and randomly located emitting dipole inside the Ge micro-crystals with a bandwidth covering the spectral range between 0.89 and 0.62 eV (i.e., both direct- and indirect-band emission from Ge). Such a large number of single-dipole simulations is needed in order to avoid spurious

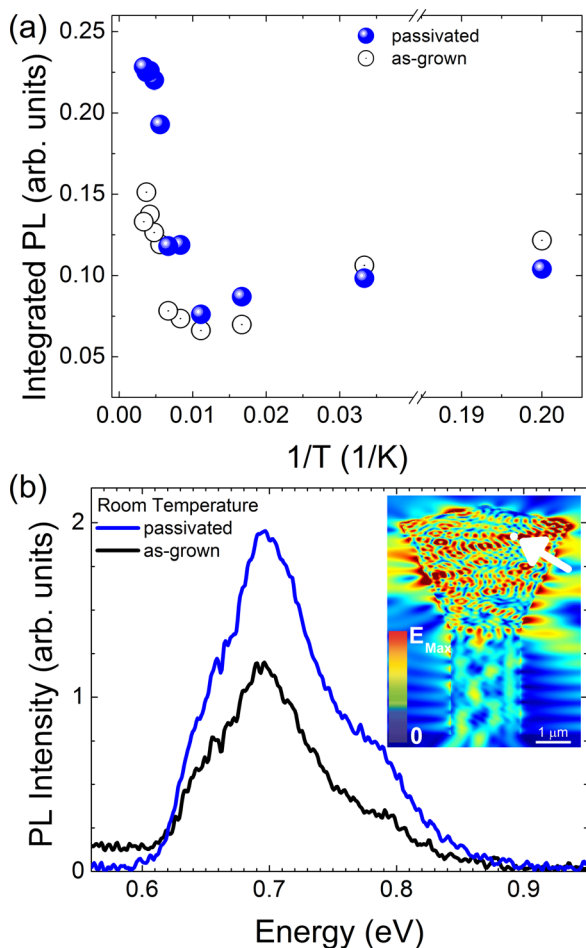


FIG. 3. (a) The temperature dependence of the integrated photoluminescence (PL) intensity of the steady-state interband emission (direct plus indirect) for the as-grown (black dots) and the passivated (blue dots) samples. (b) The room temperature PL spectra of the as-grown (black line) and passivated (blue line) Ge micro-crystals under continuous wave excitation at 1.165 eV. The inset shows a cross-sectional map of the electric field distribution obtained by finite difference time domain calculations for an emitting point dipole placed inside a Ge micro-crystal at the position of the white circle indicated by the arrow.

interference effects between different emitting dipoles, something that would have no counterpart in the actual PL process due to the lack of coherence. One representative field map resulting from such simulations is demonstrated in the inset of Fig. 3(b). Finally, we projected the local field distribution generated by each dipole to the far field and integrated the power flux within the collection angle of the optics to get an estimate of the emitted power.

By doing so, we are in the position to calculate the ratio between the expected PL intensity collected from the as-grown and passivated Ge micro-crystals under the same illumination conditions. We find that the difference in the PL intensity between the two systems is within 1%–2%, i.e., well below the experimentally observed effects. This confirms that the PL enhancement demonstrated in Fig. 3(b) must be ascribed to an improvement of the internal quantum efficiency, which stems from the reduced nonradiative recombination at the passivated sidewalls of the micro-crystals.

In conclusion, we identified a viable approach to disentangle nonradiative transitions due to dislocations and surface states and gather insights about the complex kinetics of

the recombination processes in Ge on Si heterostructures. We demonstrated the prominent role of dislocations at low temperature and the surge of room temperature PL emission under a suitable surface passivation. These findings can be used for engineering defects in key photonic building blocks such as lasers, optical resonator, and photodetectors fabricated in group IV materials. It is worth noting that our investigation can be extended to the epitaxial growth directly on silicon of other highly mismatched materials, thus enlarging even further the Si photonic toolbox.

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