Fabrication of Ge micro-disks on free-standing SiO_2 beams for monolithic light emission

Abdelrahman Al-Attili,
Muhammad Husain, Frederic Gardes,
Hideo Arimoto, and Shinichi Saito
Faculty of Physical Sciences and Engineering
University of Southampton
Southampton, United Kingdom
Email: S.Saito@soton.ac.uk

Naoki Higashitarumizu
and Yasuhiko Ishikawa
Department of Materials Engineering
The University of Tokyo
Tokyo, Japan

Satoshi Kako, Satoshi Iwamoto,
and Yasuhiko Arakawa
Institute of Industrial Science
The University of Tokyo
Tokyo, Japan

***Abstract*—Realizing a germanium (Ge)-based monolithic light source requires high n-type doping, tensile strain, and an optical cavity. Here, we demonstrate the application of spin-on doping technique, and the use of free-standing structures to induce tensile strain on Ge micro-disks, which act as a simple micro-cavity.**

***Keywords*—Germanium, Photonics, Doping, Strain, Cavity**

I. INTRODUCTION

Innovation in short-distance communications is expected, if we realize a complementary-metal-oxide-semiconductor (CMOS) compatible laser diode on a silicon (Si) chip [1]. Compatibility with standard CMOS processes qualifies indirect-gap group IV materials for this purpose, among which Ge appears to be the most promising due to its pseudo-direct band-gap [2]. The slight difference between Γ and L valleys (136 meV) can be decreased by applying tensile strain and n-type doping [3]. Tensile strain deforms the edges of the conduction band and transforms Ge into a direct gap material at 2% bi-axial tensile strain, while n-type doping fills the L valleys with electrons and reduces the injection requirements to achieve population inversion [3]–[5]. In fact, lasing from Ge [4], [5], and more recently from GeSn [6], has been demonstrated. In order to improve the efficiency, strain and n-type doping levels have to be enhanced (Fig. 1).

In this paper, we present our work on n-type doping of Ge using spin-on dopants, and the application of tensile strain on a simple Ge micro-disk cavity, by utilizing free-standing structures using Ge-on-insulator (GOI) wafers.

II. SPIN-ON DOPING

For efficient light emission from Ge, we must achieve high active carrier concentration, while imposing minimal damage on crystalline quality upon doping. Direct-gap emission efficiency is sensitive to the crystalline quality, and it was found that the carrier lifetime seems to be limited by non-radiative recombinations [1], [7]. Consequently, ion-implantation may not be suitable for Ge light emission applications. Other diffusion-based techniques, such as $in - situ$ and delta-layer doping, can be implemented [5]. Spin-on doping (SOD) is a promising doping technique, in which a high-concentration impurity source is span-coated on top of Ge and annealed. SOD imposes minimal damage on crystalline quality because it is based on diffusion of dopants, and potentially can achieve high activation levels because the impurity concentration in the SOD solution can exceed the solid solubility in Ge [8], [9].

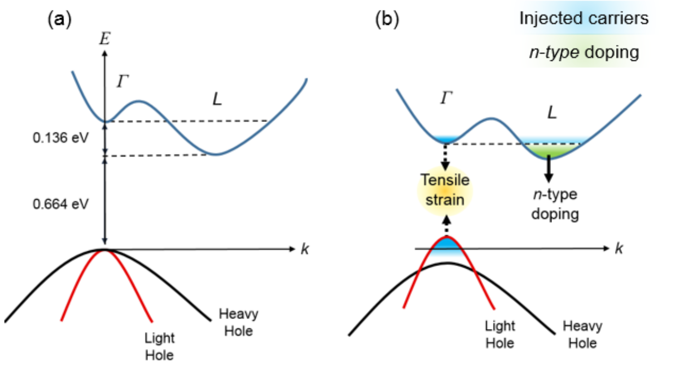

Fig. 1. Band-gap of (a) bulk Ge, and (b) tensile-strained n-doped Ge. Tensile strain deforms conduction band edges and splits the heavy-hole and light-hole bands. While n-type doping fills the L valley with electrons, and consequently increases the probability of electrons injection into the Γ valley

Many processing obstacles have to be overcome to establish a SOD recipe for heavy doping of Ge. These obstacles are due to thermal stresses during annealing, which cause the Ge layer to crack and peel off in the case of GOI wafers. Moreover, Ge oxidizes at temperatures exceeding 450 °C forming gaseous Ge mono-oxides (GeO), and this complicates the annealing process during which the diffusion of dopants occur [9], [10]. We have used two types of spin-on dopants, a water-based and an alcohol-based solutions, to dope GOI wafers with 70 nm Ge [9]. After optimizing the doping process for both solutions, mainly through dilution and patterning the Ge layer before SOD, doping of 10 μm Ge disks was possible with an activation level of $(1 - 2)\times10^{19}$ cm^{-3}. Photoluminescence (PL) measurements were conducted using a laser with 740 nm wavelength. PL spectra showed an increase in direct-gap emission intensity, which can be attributed to indirect L valleys filling by doping, and this increases the probability of electrons injection into the direct Γ valleys. Observing the power dependence of PL

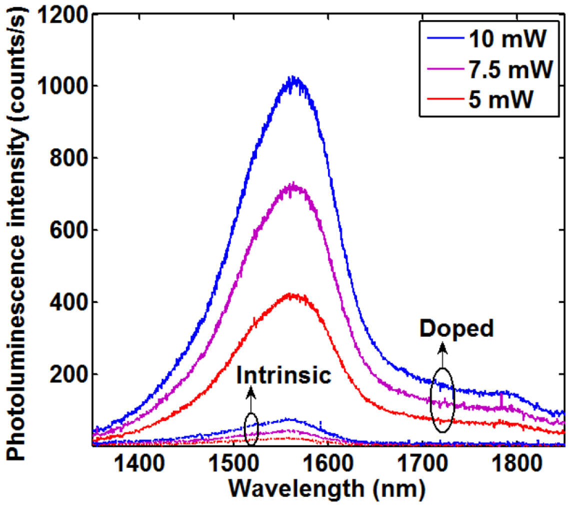

Fig. 2. Photoluminescence measurements showing the effect of doping on direct-gap emission from 10-μm-diameter Ge disk. Different colors indicated different pumping powers of 5, 7.5, and 10 mW

intensity for intrinsic and doped samples, we found a change in power-dependence behavior from quadratic to linear [9]. This indicates a faster increase in direct-gap emission intensity with increased excitation power for the doped Ge.

III. TENSILE STRAIN BY FREE-STANDING SiO_2 BEAMS

Tensile strain application by fabricating free-standing Ge beams is known for high achievable strain values [11], [12]. The dependence of this strain on beam dimensions provides an additional advantage of strain tunability. Embedding an optical cavity within free-standing structures however is not straightforward.

In order to embed the functionality of a micro-cavity within a free-standing structure, we fabricated Ge micro-disks on top of suspended SiO_2 beams, using GOI wafers (Fig. 3). Ge disks with diameters varying from 1 to 10 μm were dry-etched. Then the surface was passivated with 100 nm SiO_2 deposited using Plasma-enhanced CVD at 350 °C. Afterwards, hole openings were dry-etched in the buried-oxide (BOX) layer centered around the Ge disks to define the beams. To be suspended, the beams have to be aligned with the $\langle 001 \rangle$ direction, as shown in Fig. 4. Due to the sensitivity of alkali wet-etching that will be used afterwards to the surface roughness of Si, dry-etching was tuned such that 20 nm BOX remains un-etched, and then removed using diluted Hydrofluoric (HF) acid. Then immediately the bulk Si substrate was etched using Tetra-methyl-ammonium hydroxide (TMAH) to suspend the structures. Several beam designs were included in our chip, such as beams with curved edges (Fig. 4 (a)) to enhance beam stability upon suspension, and conventional rectangular beams. To reduce the effect of the boundaries on the beam, it is possible to utilize wet-etching anisotropy to limit the under-etching at the beam edges, as shown in Fig. 4 (b). Direction of bending was unpredictable, and beams with curved edges had better survivability compared to conventional rectangular beams.

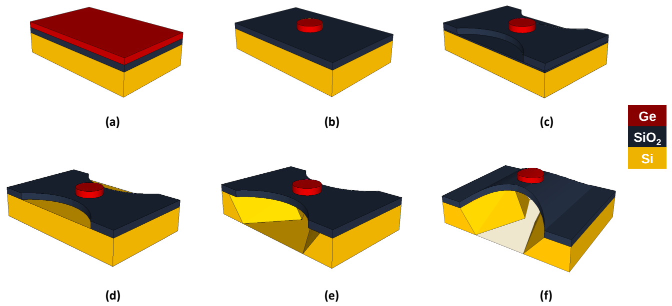

Fig. 3. Fabrication process of Ge disks on suspended SiO_2 beams: (a) GOI wafer cleaning, (b) dry-etching Ge disks, (c) dry-etching BOX layer, (d) wet-etching the remaining BOX using HF, (e) and suspension by etching the Si substrate using TMAH. (f) Shows the final structure after the beam bending

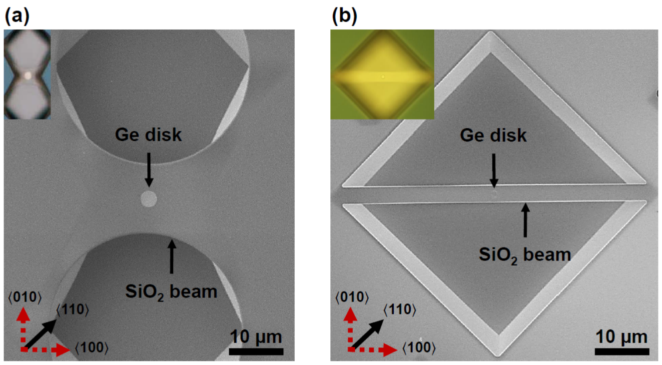

Fig. 4. Scanning electron microscopy images of a Ge micro-disk on: (a) a suspended SiO_2 beam with curved edges, and (b) a suspended SiO_2 beam with straight edges. Insets show optical microscope images of similar structures

SiO_2 beams bend upon suspension due to residual stresses within the thermally-grown SiO_2 film. BOX layer has built-in compressive stresses that originate during the high-temperature growth process, due to the difference in thermal expansion coefficient between Si and SiO_2 [13]. Releasing such a compressively-stressed beam will partially relief the stresses by beam elongation, as shown in Fig. 5 (a) and (b), while residual stresses work on deflecting the beam [13], [14]. Consequently, upward bending, for example, will cause the upper side of the beam to be tensile-strained, while the bottom side gets compressed [14], as shown in Fig. 5 (c). We expect that this tensile strain can be transferred to a Ge micro-disk on top of the beam, if there is a good adhesion between the disk and the beam. Laser microscopy height mapping (Fig. 5 (d)) shows the bending behavior of a Ge micro-disk on a free-standing SiO_2 beam with curved edges. This bending with a maximum deflection of about 0.8 μm contributed to an enhancement in tensile strain within the Ge disk, as confirmed by Raman spectroscopy.

Raman spectroscopy measurements were conducted using a laser with 532 nm wavelength and 2 μm spot size. Due to sensitivity of suspended structures to heating by laser

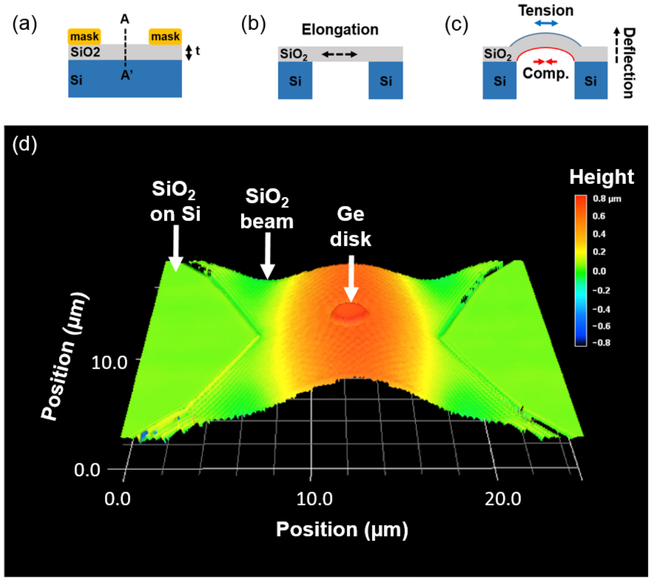

Fig. 5. Tensile strain by free-standing SiO$_2$ beams: (a) thermally-grown SiO$_2$ with built-in compressive stresses, (b) partial relief of stresses by elongation after suspension, and (c) bending due to residual stresses. (d) Laser microscopy height map of a Ge micro-disk on a free-standing SiO$_2$ beam. Upward bending of the beam is responsible for tensile strain accumulation within the disk

excitation, we measured the Raman shift dependence on the excitation power. Excitation power was increased in steps from 55 μW to 1 mW, and data points were fit with a Lorentzian to extract the Raman peak position. Power dependence of the Raman peak position had a linear trend, and by extrapolating this linear relation we could estimate the Raman peak position without the effect of heating. The reference un-patterned GOI wafer had a Raman shift of -0.8 cm^{-1} relative to bulk Ge, which corresponds to approximately 0.2% bi-axial tensile strain. This strain is presumably due to Ge growth on Si during the GOI manufacturing process. After dry etching the Ge disks, it was found that tensile strain relaxes and the shift in Raman peak position reduces. In fact, in small disks with diameters around 2 μm, the Raman shift after dry etching is nearly zero compared to bulk Ge. However, when the disks are suspended by releasing the SiO$_2$ beams, a slight enhancement in tensile strain is observed. For instance, Raman peak of the 3 μm Ge disk shown in Fig. 4 (a) is shifted by -1.11 cm^{-1} relative to bulk Ge. The orientation of this tensile strain, being either uni-axial or bi-axial, is not obvious. Accordingly, the tensile strain can be estimated to be 0.73% (0.28%) uni-axial (bi-axial) [15].

PL measurements of Ge micro-disks on free-standing SiO$_2$ beams were performed using a laser with 730 nm wavelength (Fig. 6). We investigated the dependence of free-standing Ge disks' spectra on the excitation power, and the broadening of the PL spectra has been observed upon increasing the excitation power. This was attributed to the sensitivity of suspended structures to heating by laser excitation. It was found that heating induced a splitting between the light-hole (LH) and heavy-hole (HH) bands, and the splitting energy increased upon heating.

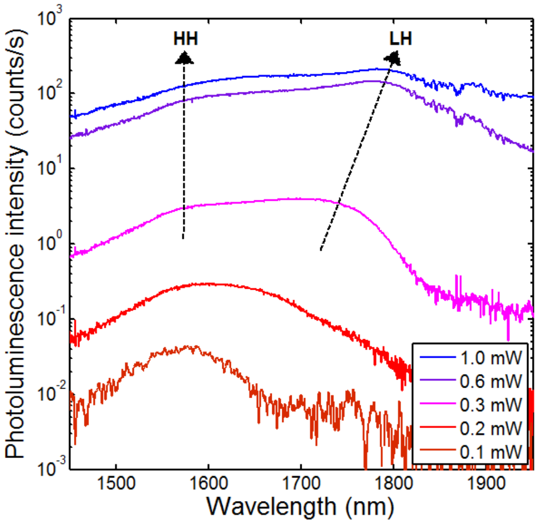

Fig. 6. Photoluminescence intensity of a free-standing Ge micro-disk for different excitation powers. Splitting of heavy-hole and light-hole bands is evident as assigned with the arrows

IV. A SIMPLE MICRO-CAVITY

Ge micro-disks are being investigated as a tempting structure for realizing an on-chip laser diode, due to their small footprint and the high quality (Q) factors of the Whispering Gallery Modes (WGM) that are confined in such structures [16], [17]. Before suspension, we could not detect any WGM resonances because of the thin Ge film and the mode leakage path through the Si substrate. On the other hand, sharp peak resonances were observed at room temperature from a 3 μm Ge disk on a free-standing SiO$_2$ beam, as shown in Fig. 7. A maximum quality factor of 192 was calculated by fitting the resonant peak with a Lorentzian function, and dividing the center frequency by the full-width half-maximum. Q-factors are degraded with increased pumping power due to heating effects and free-carrier absorption (FCA) losses [15].

Finite-domain time-difference (FDTD) simulations were done to determine the nature of these sharp resonances. An electric field was used to excite a similar structure of a 3 μm Ge disk on a free-standing SiO$_2$ beam. FDTD simulations resulted in sharp-peak WGMs centered on 1508, 1561, 1626, 1708, and 1811 nm. Assuming that only transverse-electric (TE) modes can be guided through (70 - 100) nm Ge, these TE$_{m,n}$ WGMs can be referred to as TE$_{13,1}$, TE$_{12,1}$, TE$_{11,1}$, TE$_{10,1}$, and TE$_{9,1}$, respectively [15]. Where m is the number of full wavelengths of the confined mode around the inner circumference of the disk, and n is the number of field maxima along the radius of the disk. Accordingly, we can refer to the sharp peaks in Fig. 7 as TE$_{10,1}$, and TE$_{9,1}$, with the corresponding field profiles shown in the figure.

Broadening of the resonant peaks with increased pumping indicates that losses due to absorption, because of the shift in Ge absorption edge by heating, and FCA losses, are high and also increase with pumping [15]. These losses can be overcome by further device optimization. Higher tensile strain reduces the required pumping levels for population

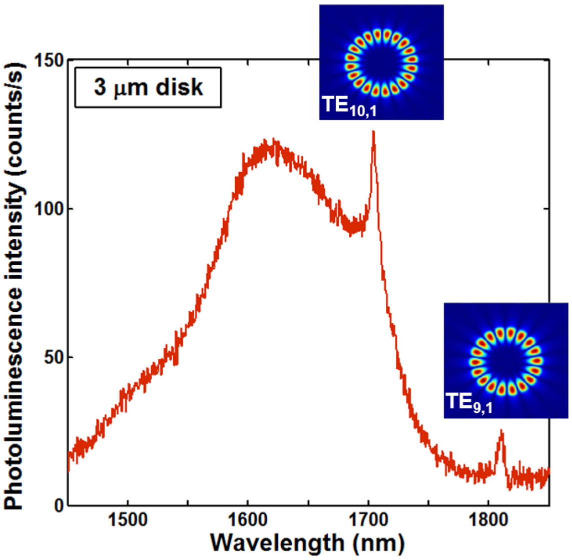

Fig. 7. Photoluminescence spectrum of a 3-μm-diameter Ge disk on a free-standing SiO_2 beam. Sharp peaks are whispering-gallery-mode resonances within the direct-gap of Ge. Mode profiles are found using finite-domain time-difference simulations, and the corresponding resonances can be assigned to be $TE_{10,1}$ and $TE_{9,1}$ at 1708, and 1811 nm, respectively

inversion, and consequently avoid excessive heating effects. We expect that enhanced adhesion between the Ge and BOX layers, in addition to optimization of the beam design, would improve the applied strain. n-type doping fills the L valleys in the conduction band with electrons, which minimizes the required injection of electron-hole pairs and consequently reduces the free hole absorption losses [2]. Spin-on doping can potentially be applied to achieve high activation levels in Ge. Additionally, further enhancement of Ge crystalline and interface quality with the substrate are important to increase the direct-gap emission efficiency [1], [7].

V. CONCLUSION

Spin-on doping is a valuable technique to achieve high activation levels in Ge, however there are many processing obstacles that originate from thermal stresses during annealing and oxidation of Ge, that have to be overcome. Suspension of Ge structures on top of SiO_2 beams, is capable of delivering tensile strain to Ge due to beams' bending, and confining optical modes in the case of Ge micro-disks. Sensitivity to heating effects is pronounced in free-standing structures, this effect was found to induce additional separation between the LH and HH bands. We believe that combining previous techniques of doping and tensile-strain is promising for Ge light emission purposes and future realization of a Si-based on-chip optical communication link.

ACKNOWLEDGMENT

Parts of the studies discussed here were supported by Japan Society for the Promotion of Science (JSPS) through its "Funding Program for World-Leading Innovation R&D on Science and Technology (FIRST)", the Project for Developing Innovation Systems, and Kakenhi 216860312, MEXT, Japan. This work is also supported by EPSRC Standard Grant (EP/M009416/1), EPSRC Manufacturing Fellowship (EP/M008975/1), EU FP7 Marie-Curie Carrier-Integration-Grant (PCIG13-GA-2013-618116), University of Southampton Zepler Institute Research Collaboration Stimulus Fund, and Hitachi. The raw data of this paper can be obtained from the University of Southampton ePrints research repository, DOI: 10.5258/SOTON/382885.

REFERENCES

[1] S. Saito, F. Y. Gardes, A. Z. Al-Attili, K. Tani, K. Oda, Y. Suwa, T. Ido, Y. Ishikawa, S. Kako, S. Iwamoto, and Y. Arakawa, "Group IV light sources to enable the convergence of photonics and electronics," *Front. Mater.*, vol. 1, pp. 1–15, 2014.

[2] J. Liu, L. C. Kimerling, and J. Michel, "Monolithic Ge-on-Si lasers for large-scale electronic-photonic integration," *Semicond. Sci. Technol.*, vol. 27, no. 9, p. 094006, 2012.

[3] M. E. Kurdi, G. Fishman, S. Sauvage, and P. Boucaud, "Band structure and optical gain of tensile-strained germanium based on a 30 band k.p formalism," *J. Appl. Phys.*, vol. 107, no. 1, p. 013710, 2010.

[4] J. Liu, X. Sun, R. Camacho-Aguilera, L. C. Kimerling, and J. Michel, "Ge-on-Si laser operating at room temperature," *Opt. Lett.*, vol. 35, no. 5, pp. 679–681, 2010.

[5] R. E. Camacho-Aguilera, Y. Cai, N. Patel, J. T. Bessette, M. Romagnoli, L. C. Kimerling, and J. Michel, "An electrically pumped germanium laser," *Opt. Express*, vol. 20, no. 10, pp. 11 316–11 320, 2012.

[6] S. Wirths, R. Geiger, N. von den Driesch, G. Mussler, T. Stoica, S. Mantl, Z. Ikonic, M. Luysberg, S. Chiussi, J. M. Hartmann, H. Sigg, J. Faist, D. Buca, and D. Grützmacher, "Lasing in direct-bandgap GeSn alloy grown on Si," *Nat. Photonics*, vol. 9, no. 2, pp. 88–92, 2015.

[7] S. Saito, K. Oda, T. Takahama, K. Tani, and T. Mine, "Germanium fin light-emitting diode," *Appl. Phys. Lett.*, vol. 99, no. 24, p. 241105, 2011.

[8] X. Xu, K. Nishida, K. Sawano, T. Maruizumi, and Y. Shiraki, "Tensile-strained, heavily n-doped germanium-on-insulator for light emitting devices on silicon," in *Conference on Lasers and Electro-Optics (CLEO), OSA Tech. Dig.*, 2014, p. SM4H.3.

[9] A. Z. Al-Attili, S. Kako, M. K. Husain, F. Y. Gardes, H. Arimoto, N. Higashitarumizu, S. Iwamoto, Y. Arakawa, Y. Ishikawa, and S. Saito, "Spin-on doping of germanium-on-insulator wafers for monolithic light sources on silicon," *Jpn. J. Appl. Phys.*, vol. 54, no. 5, p. 052101, 2015.

[10] N. E. Posthuma, J. V. der Heide, G. Flamand, and J. Poortmans, "Emitter formation and contact realization by diffusion for germanium photovoltaic devices," *IEEE Trans. Electron Devices*, vol. 54, no. 5, pp. 1210–1215, 2007.

[11] M. J. Süess, R. Geiger, R. A. Minamisawa, G. Schiefler, J. Frigerio, D. Chrastina, G. Isella, R. Spolenak, J. Faist, and H. Sigg, "Analysis of enhanced light emission from highly strained germanium microbridges," *Nat. Photonics*, vol. 7, no. 6, pp. 466–472, 2013.

[12] D. S. Sukhdeo, D. Nam, J. H. Kang, M. L. Brongersma, and K. C. Saraswat, "Direct bandgap germanium-on-silicon inferred from 5.7% $\langle 100 \rangle$ uniaxial tensile strain," *Photon. Res.*, vol. 2, no. 3, pp. A8–A13, 2014.

[13] C. W. Wilmsen, E. G. Thompson, and G. H. Meissner, "Buckling of thermally-grown SiO_2 thin-films," *IEEE Trans. Electron Devices*, vol. 19, no. 1, p. 122, 1972.

[14] S. D. Senturia, *Microsystem Design*. Boston: Kluwer academic publishers, 2001.

[15] A. Z. Al-Attili, S. Kako, M. Husain, F. Gardes, N. Higashitarumizu, S. Iwamoto, Y. Arakawa, Y. Ishikawa, H. Arimoto, K. Oda, T. Ido, and S. Saito, "Whispering gallery mode resonances from Ge micro-disks on suspended beams," *Front. Mater.*, accepted, 2015.

[16] A. Ghrib, M. E. Kurdi, M. de Kersauson, M. Prost, S. Sauvage, X. Checoury, G. Beaudoin, I. Sagnes, and P. Boucaud, "Tensile-strained germanium microdisks," *Appl. Phys. Lett.*, vol. 102, no. 22, p. 221112, 2013.

[17] A. Ghrib, M. E. Kurdi, M. Prost, M. de Kersauson, L. Largeau, O. Mauguin, G. Beaudoin, S. Sauvage, X. Checoury, G. Ndong, M. Chaigneau, R. Ossikovski, S. David, I. Sagnes, and P. Boucaud, "Strain engineering in germanium microdisks," in *Proc. SPIE OPTO*, 2014, p. 89901C.