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Temperature-dependent photoluminescence of Ge/Si and $Ge_{1-y}Sn_y$/Si, indicating possible indirect-to-direct bandgap transition at lower Sn content

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Temperature (T)-dependent photoluminescence (PL) has been investigated for both *p*-Ge and *n*-$Ge_{1-y}Sn_y$ films grown on Si substrates. For the *p*-Ge, strong direct bandgap (E_D) along with weak indirect bandgap related (E_{ID}) PL at low temperatures (LTs) and strong E_D PL at room temperature (RT) were observed. In contrast, for the *n*-$Ge_{1-y}Sn_y$, very strong dominant E_{ID} PL at LT and strong E_D PL were observed at RT. This T-dependent PL study indicates that the indirect-to-direct bandgap transitions of $Ge_{1-y}Sn_y$ might take place at much lower Sn contents than the theory predicts, suggesting that these $Ge_{1-y}Sn_y$ could become very promising direct bandgap semiconductors. © *2013 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4803927]

The indirect nature of the electronic bandgap of group-IV semiconductors such as Si and Ge prevents their use in high efficiency light emitting devices. Phonons are usually involved in optical processes in group-IV semiconductors in order to satisfy the momentum conservation in optical transition. Therefore, they are poor light emitters, and thus there are no practical Si- or Ge-based light sources yet. In order to overcome the limitations imposed on these indirect bandgap Si and Ge semiconductors and to achieve optically active devices, an intensive research effort has been made in designing and developing artificial direct bandgap group-IV semiconductor materials.[1–9] As a result, a significant breakthrough has recently been achieved in the fabrication of Si- and Ge-based direct bandgap semiconducting materials such as $Ge_{1-y}Sn_y$ and $Ge_{1-x-y}Si_xSn_y$ alloys.[8–10] The optical properties of Ge and $Ge_{1-y}Sn_y$ alloys grown on Si substrates have recently been reported by several groups.[9–18] Grzybowski *et al.*[13] showed that for P-doped $Ge_{1-y}Sn_y$ layers grown on Si, the photoluminescence (PL) intensity was enhanced one order of magnitude compared to that of similar undoped $Ge_{1-y}Sn_y$ films. Liu *et al.*[18] demonstrated that the heavy *n*-type doping concentration, high current injection level, high laser excitation power, high temperature, and tensile strain can enhance the direct bandgap transition in Ge.

In spite of the great recent progress in the crystal growth of $Ge_{1-y}Sn_y$ alloys on Si or Ge substrate, the development of these alloys is still in the early stages, and many of the important properties of these materials are largely uncharacterized. Therefore, we have performed temperature (T)-dependent PL studies on both tensile-strained $Ge_{1-y}Sn_y$ alloys and *p*-Ge epilayers all grown on Si substrates to better understand, in particular, the indirect-to-direct bandgap transitions of $Ge_{1-y}Sn_y$ alloys, and the results are reported in this paper.

The Ge-like material of $Ge_{1-y}Sn_y$ ($y = 0.3\%$) epitaxial layer was grown using an ultra-high vacuum chemical vapor deposition method directly on *n*-type Si substrate via deuterated stannane (SnD_4) assisted reactions of pure digermane (Ge_2H_6) at 390 °C.[10,14] Below the critical concentration of $10^{19}/cm^3$ (~0.03%) Sn, the growth rate diminishes to a nearly impractical level, while above the $10^{20}/cm^3$ (~0.25%) Sn level, the material produced is more characteristic of the $Ge_{1-y}Sn_y$ alloys. The use of pure digermane under the same conditions does not produce any measurable Ge film growth, and thus, the Ge films were grown by molecular beam epitaxy (MBE) method. The *n*-type $Ge_{0.997}Sn_{0.003}$ film was doped *in situ* with P atoms, using the single source of $P(GeH_3)_3$. The donor concentration of the *n*-$Ge_{0.997}Sn_{0.003}$ film was found to be $1.5 \times 10^{19}/cm^3$ from Hall effect and infrared ellipsometry measurements.[13] The content of Sn was estimated from Rutherford backscattering measurements. After growth, the *n*-$Ge_{0.997}Sn_{0.003}$ film was annealed three times using rapid thermal annealing (RTA) at a temperature of 725 °C for 10 s, which improved crystallinity of the sample as seen from the narrowing of the XRD peak. The RTA treatment reduces the levels of threading defects and relaxes the strain in the epitaxial layers. The residual tensile strain after annealing was estimated to be 0.22% for the *n*-$Ge_{0.997}Sn_{0.003}$ film. Secondary ion mass spectrometry elemental analysis of the annealed layers revealed a highly homogeneous profile of the element throughout the crystal, and the annealed film surface remained flat (root mean square (RMS) ~1 nm). An unintentionally doped Ge film was grown directly on *p*−Si substrate for use as a reference sample by gas source MBE at 380 °C and 2×10^{-4} Torr using appropriate stock mixtures of $CH_2(GeH_3)_2$ and Ge_2H_6.[19] After growth, the Ge film was annealed at 830 °C for 30 min in a hydrogen environment. The residual tensile strain after annealing was estimated to be 0.19% for the Ge film. The thicknesses of *n*-$Ge_{0.997}Sn_{0.003}$ and *p*-Ge films are 880 and 680 nm, respectively.

PL measurements were made using an 830 nm of Ti:sapphire laser pumped by an Ar-ion laser. The emitted light was dispersed with a ½-m spectrometer equipped with a 600 lines/mm grating blazed at 1.6 μm and then focused onto an extended InGaAs detector. PL was measured at temperatures (Ts) ranging from 5 to 300 K.

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The T-dependent PL spectra of unintentionally doped *p*-Ge film grown on *p*-Si substrate are shown in Fig. 1(a). The PL spectra are mainly composed of two emission peaks. The very strong main peak at 0.852 eV at 20 K (0.770 eV at 300 K) is assigned to the direct bandgap (E_D) emission and the broad weak peak centered at around 0.701 eV at 20 K (almost unobservable at 300 K) is ascribed to the indirect bandgap related (E_{ID}) emission. The PL intensity of E_D emission is very strong at Ts between 20 and 50 K, it decreases gradually up to around 150 K as T increases, and then it starts to increase with T up to room temperature (RT). The photo-excitation of electrons out of the valence band directly into the Γ valley conduction-band minimum is possible with a laser wavelength of 830 nm (1.49 eV), and thus, a large number of nonthermalized electrons could be trapped in the Γ valley. This can lead to a strong E_D PL emission, showing much higher radiative E_D transitions than the E_{ID} transitions at all Ts. Apparently, the strong E_D PL intensity implies that the effect of self absorption may not be significant for this thin *p*-Ge/*p*-Si film compared with that of bulk Ge.[15]

The broad weak peak of E_{ID} emission at 20 K consists of two peaks centered at 0.737 and 0.701 eV, which are attributed to the no phonon (NP) and transverse optical (TO) phonon related peaks, respectively, with a TO phonon energy of 36 meV.[20] These assignments are consistent with previously reported PL peak positions.[20,21] The Gaussian fit of the E_{ID} PL peak shows that the intensities of these two peaks increase with T up to around 100 K with more rapid increase of NP peak than the TO peak. This fact, along with the influence of the nearby E_D PL peak, causes the broad E_{ID} PL peak to appear blue-shifted as T increases, but a more careful analysis shows that both the NP and TO peaks are in fact red-shifted. As T increases above around 100 K, the two peaks converge as one broad peak with rapidly decreasing PL intensity up to around 250 K, and then they almost disappear at RT.

The E_D PL emission energies are plotted as a function of T for this sample as shown in Fig. 1(b). Unfortunately, the T-dependent $E_{ID}(T)$ PL peaks cannot be determined reliably due to lack of clear PL peaks at higher Ts. The solid fitting curve for $E_D(T)$ is calculated using Varshni's type equation[22] with $E_D(T) = E_D(0) - \alpha T^2/(T+\beta)$, where $E_D(0) = 0.853$ eV, and the same empirical parameters of $\alpha = 5.82 \times 10^{-4}$ eV/K and $\beta = 296$ K are used as those for bulk Ge.[23] This calculated $E_D(T)$ agrees very well with the PL data as shown in Fig. 1(b) except the intermediate T region, where the influence of the E_{ID} PL obscures the accurate estimation of E_D.

The T-dependent PL spectra of P-doped *n*-$Ge_{0.997}Sn_{0.003}$/*n*-Si are shown in Fig. 2(a). The strong main peak at 0.711 eV at 5 K is associated with the E_{ID} transition. At present, the nature of this PL peak cannot be defined whether it is no phonon related, acoustic, or optical phonon related peak. The PL intensity of this E_{ID} emission decreases continuously as the T increases from 5 to 225 K, and then the peak almost disappears at RT because of the prevailing non-radiative recombination.

Another strong broad peak was observed at 0.720 eV at RT for this sample, which is attributed to the E_D emission. The E_D PL intensity decreases continuously as the T decreases from 300 to 100 K, and then it disappears as T decreases further as shown in the figure. For this heavily *n*-type doped sample, a higher electron population into the direct Γ valley caused by thermalization at RT is expected due to the indirect L valley states' filling effect[11] along with photo-excitation. Thus, very strong E_D emission is expected to result in from this sample. On the contrary, the PL intensity of the E_D emission at RT is not as strong as expected. Instead, the sample shows very strong E_{ID} PL at low temperature (LT). Also, the intensity of E_D PL peak of this sample decreases with T possibly at the expense of increasing E_{ID} PL intensity, whereas that of the *p*-Ge/*p*-Si increases as T decreases.

The PL peak positions of E_D at RT and E_{ID} at LT are well defined, but those peak positions at intermediate Ts are not. However, very clear two peaks are observed at T around 150 K. Therefore, each set of T-dependent PL data was fit with two Gaussian peaks of E_D (red solid squares) and E_{ID} (black solid circles) wherever possible, and the results are plotted in Fig. 2(b). The calculated $E_D(T)$ of current sample

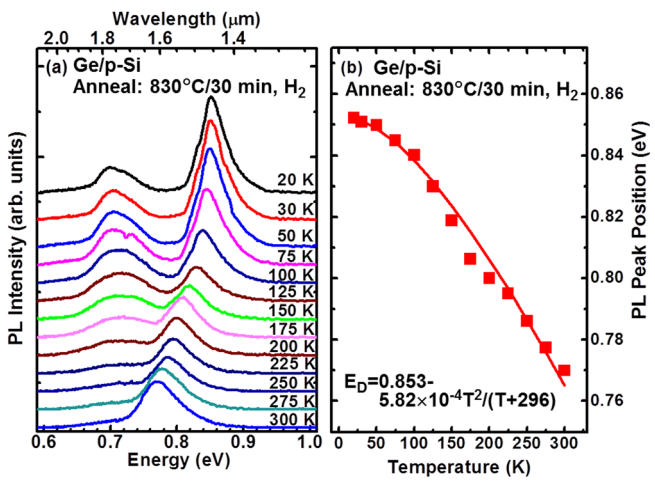

FIG. 1. (a) Temperature-dependent PL spectra of the unintentionally doped *p*-Ge film grown on *p*-Si substrate. (b) Direct bandgap related PL peak energies (E_D) plotted as a function of temperature from 20 to 300 K. The red fitting curve is the calculated direct bandgap related energies plotted as a function of temperature using Varshni's equation.

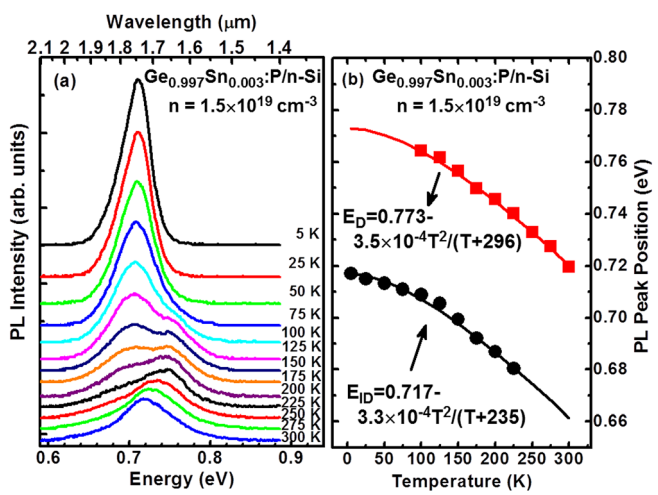

FIG. 2. (a) Temperature-dependent PL spectra of the P-doped *n*-$Ge_{0.997}Sn_{0.003}$ grown on *n*-Si substrate. (b) Direct (E_D; red squares) and indirect (E_{ID}; black circles) bandgap related PL peak energies plotted as a function of temperature from 5 to 300 K. The red and black fitting curves are the calculated direct (E_D) and indirect (E_{ID}) bandgap related energies, respectively, plotted as a function of temperature using Varshni's equation.

(red line) is given by $E_D(T) = E_D(0) - \alpha T^2/(T + \beta)$, where $E_D(0) = 0.773$ eV, $\alpha = 3.5 \times 10^{-4}$ eV/K, and $\beta = 296$ K are used. For bulk Ge, T-dependence of the direct bandgap (E_Γ) is given by $E_\Gamma(T) = 0.89 - 5.82 \times 10^{-4} \cdot T^2/(T + 296)$ (eV).[23] The black line is the calculated $E_{ID}(T)$ using $E_{ID}(T) = E_{ID}(0) - \alpha T^2/(T + \beta)$, where $E_{ID}(0) = 0.717$ eV, $\alpha = 3.3 \times 10^{-4}$ eV/K, and $\beta = 235$ K are used. For bulk Ge, T-dependence of the energy gap (E_g) is given by $E_g(T) = 0.742 - 4.8 \times 10^{-4} \cdot T^2/(T + 235)$ (eV).[23]

Schematic band diagrams (not scaled) for bulk Ge, 0.19% tensile strained p-Ge/p-Si, and 0.22% tensile strained n-$Ge_{0.997}Sn_{0.003}$/n-Si samples are plotted in Fig. 3 along with the observed PL peak energies of E_D and E_{ID} at LT and/or 300 K. The compositional dependence of the $Ge_{1-y}Sn_y$ bandgaps was calculated using a standard quadratic equation of the form: $E_i^{GeSn}(y, T) = yE_i^{Sn}(y, T) + (1 - y)E_i^{Ge}(y, T) - y(1 - y)b_i(T)$, where $i = \Gamma$,L for the direct and indirect bandgaps, respectively, with the E_i values for pure Ge and α-Sn given by Refs. 23 and 24. Bowing parameters of $b_\Gamma(0\,K) = 2.55$ eV and $b_L(0\,K) = 0.89$ eV were used for LTs, while $b_\Gamma(300\,K) = 1.95$ eV and $b_L(300\,K) = 0.68$ eV were used for RT. These bowing parameters represent a compilation of the best estimates available in the literature, including those obtained from theoretical and experimental methods.[25–27] Additionally, the effect of strain was calculated using the deformation potential theory of Van de Walle.[28]

For the p-Ge/p-Si sample, the $E_\Gamma(0)$ is expected to be reduced to 0.859 eV from the value of 0.890 eV of bulk Ge at 0 K due to 0.19% tensile strain as shown in Figs. 3(a) and 3(b). However, the estimated value of $E_D(0) = 0.853$ eV from the T-dependent PL peak energies shown in Fig. 3(b) for p-Ge sample is very slightly smaller than the predicted value of 0.859 eV, which implies that the $E_\Gamma(0)$ [$\approx E_D(0)$] reduction in Γ conduction valley minimum might be very slightly larger than the theory predicted. On the other hand, the indirect bandgap at 0 K, $E_L(0)$ [$\approx E_g(0)$], is expected to be reduced to

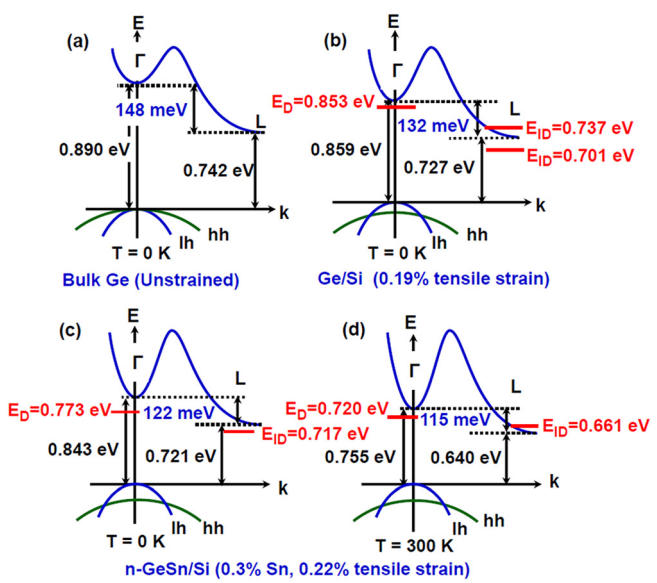

FIG. 3. Simple band diagrams (not scaled) for (a) bulk Ge at 0 K, (b) 0.19% tensile strained p-Ge/p-Si at 0 K, and (c) and (d) 0.22% tensile strained n-$Ge_{0.997}Sn_{0.003}$/Si samples at 0 and 300 K, respectively, plotted along with the observed PL peak energy positions at each temperature.

0.727 eV for this p-Ge sample, compared to the value of 0.742 eV in bulk Ge. However, the observed PL peak energy of the NP peak at 20 K ($E_{ID}(20)$) is 0.737 eV for this sample, which is slightly higher than the predicted value of 0.727 eV, indicating that the $E_L(0)$ could be as high as 0.737 eV. This implies that the reduction in the $E_L(0)$ might be smaller than the theory predicted. For this sample, the estimated energy separation between the Γ and L valleys at 0 K, $[E_\Gamma(0) - E_L(0)]$, could be about 116 meV (= 0.853 − 0.737 eV), which is smaller than the theoretically predicted value of 132 meV. This difference could be due in part to the larger tensile strain expected at lower Ts due to the thermal expansion mismatch between Ge and Si, which was not taken into account here. However, its effect cannot be as nearly close to 16 meV, because the entire 0.19% tensile strain can change only 16 meV. Therefore, it could indicate a slightly larger reduction of the $[E_\Gamma(0) - E_L(0)]$ with tensile strain than what the theory predicted.

Similarly, the direct bandgap (not shown in Fig. 3) at RT is expected to be reduced to 0.769 eV compared to the value of 0.800 eV in bulk Ge. However, the observed PL peak energy ($E_D(300)$) is 0.770 eV for this sample. If the maximum Boltzmann energy distribution of kT/2 at 300 K (13 meV) is taken into account, the $E_\Gamma(300)$ could be about 0.757 eV, which is slightly smaller than the predicted value of 0.769 eV. Unfortunately, the indirect PL peak energy ($E_{ID}(300)$) at RT could not be observed as seen in Fig. 1. Therefore, a reliable estimate of the $[E_\Gamma(300) - E_L(300)]$ could not be obtained through PL measurements.

For the n-$Ge_{0.997}Sn_{0.003}$/Si sample, the $E_\Gamma(0)$ and $E_L(0)$ at 0 K are expected to be reduced to 0.843 and 0.721 eV, respectively, due to the combination of 0.22% tensile strain and 0.3% Sn content as shown in Fig. 3(c). However, the extrapolated PL peak energy at 0 K, $E_D(0)$ [$\approx E_\Gamma(0)$], is 0.773 eV as shown in Fig. 2(b), which is about 70 meV smaller than the predicted value of 0.843 eV. Furthermore, note also here that the $E_D(0)$ of 0.773 eV for this sample is about 80 meV lower than the $E_D(0)$ of 0.853 eV for the p-Ge/p-Si. Although the heavy doping effect could influence this difference, it might be largely due to the 0.3% Sn content considering the fact that the strain is about the same for both samples. On the other hand, the estimated value of the $E_{ID}(0)$ is 0.717 eV for this sample, which is very slightly less than the predicted value of 0.721 eV. Assuming that this PL peak is due to a phonon emission, the $E_L(0)$ could be in the range of 0.717 and 0.753 eV using phonon energies of 0 meV for NP and 36 meV for TO phonon, respectively. Therefore, the estimated $[E_\Gamma(0) - E_L(0)]$ obtained through PL measurements could be less than or about 56 meV (= 0.773–0.717 eV), which is much smaller than the theoretically calculated separation of 122 meV.

At RT, the $E_\Gamma(300)$ and $E_L(300)$ are expected to be reduced to 0.755 and 0.640 eV, respectively, for this n-$Ge_{0.997}Sn_{0.003}$/Si sample. Although the measured $E_D(300)$ is 0.720 eV, if the kT/2 factor is taken into account, the $E_\Gamma(300)$ could be 0.707 eV, which is much smaller than the predicted value of 0.755 eV. Also, note here that the measured $E_D(300)$ of 0.720 eV for this sample is about 50 meV lower than the $E_D(300)$ of 0.770 eV for the p-Ge, which again could be mainly due to the 0.3% Sn content. In the meantime, for the indirect bandgap, the extrapolated

$E_{ID}(300)$ is 0.661 eV for this sample, which is higher than the predicted value of 0.640 eV. However, it is a lot more difficult to discuss the relationship between the PL peak energies and the actual bandgap because the observed $E_{ID}(300)$ PL peaks at RT may be due to no-phonon, phonon absorption, or phonon emission processes. In addition, this heavy *n*-type P-doping may also fill up the conduction band above the $E_L(300)$ minimum along with the possible band tailing. Also, according to the theory for bandgap narrowing,[29] the L-valley minimum would be reduced by about 30 meV at RT. Therefore, all these could affect the observed $E_{ID}(300)$ PL peak energy as well as $E_D(300)$. Since the $[E_\Gamma(300) - E_L(300)]$ would be effectively minimally affected by the kT/2 factor, the kT/2 factor is not incorporated. If the measured PL is no-phonon related peak, then the $E_L(300)$ could be about 0.661 eV. If the measured PL peak is phonon emission related peak, then the range of $E_L(300)$ could be between 0.669 and 0.697 eV, and if it is phonon absorption related peak, which is very unlikely, then the range of $E_L(300)$ could be between 0.653 and 0.625 eV, assuming 8 meV for transverse acoustic (TA) and 36 meV for TO phonons. Therefore, the estimated value of $[E_\Gamma(300) - E_L(300)]$ could be about 59 meV (=0.720–0.661 eV) for NP related peak, between 23 and 51 meV for phonon emission related peak, and between 67 and 95 meV for phonon absorption related peak, depending on the nature of phonons involved, compared to the theoretically calculated reduced energy separation of 115 meV. It is believed that the PL data could still support the less L valley bandgap reduction than the theory predicted even after considering the kT/2 factor at RT.

In conclusion, the T-dependent PL has been investigated for 0.19% tensile-strained *p*-Ge/*p*-Si and 0.22% tensile-strained P-doped *n*-$Ge_{0.997}Sn_{0.003}$/*n*-Si samples. The *p*-Ge film shows a strong E_D transition at RT, and much stronger, dominant E_D and weak E_{ID} PL emissions at LT. On the other hand, for the *n*-$Ge_{0.997}Sn_{0.003}$, strong, dominant E_{ID} emission was observed at LT, but weak E_D transition was observed at RT. In general, the T-dependent PL measurements consistently indicate that the E_Γ [$\approx E_D$] reduction in Γ conduction valley minimum could be larger than the theory predicted, whereas the E_L reduction in L conduction valley minimum could be smaller than the theory predicted. For *p*-Ge sample, the estimated $[E_\Gamma(0) - E_L(0)]$ could be about 116 meV compared to the theoretically predicted value of 132 meV. On the other hand, for heavily doped *n*-$Ge_{0.997}Sn_{0.003}$ sample, the estimated $[E_\Gamma(0) - E_L(0)]$ could be about 56 meV compared to the predicted value of 122 meV, and $[E_\Gamma(300) - E_L(300)]$ could be about 59 meV compared to the predicted value of 115 meV. Therefore, our T-dependent PL results strongly indicate that the Sn content reduces bandgap of $Ge_{1-y}Sn_y$/Si much more effectively than the theory predicted, and the indirect-to-direct bandgap transition of *n*-$Ge_{1-y}Sn_y$/Si sample could take place at much lower Sn content than originally anticipated even after considering the heavy *n*-type doping effect. This conclusion is also consistent with the observation of indirect-to-direct transition of $Ge_{1-y}Sn_y$/Si occurring at around 6% Sn,[25,26] which is less than recently predicted values of around 10% Sn[27,30] and much less than theoretically predicted values of about 20% Sn.[31] Thus, the $Ge_{1-y}Sn_y$ alloys could be attractive materials for the fabrication of direct bandgap Si-based light emitting devices.

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