Optical Characterization of Si-Based $\text{Ge}_{1-x}\text{Sn}_x$ Alloys with Sn Compositions up to 12%

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Optical properties of germanium tin ($\text{Ge}_{1-x}\text{Sn}_x$) alloys have been comprehensively studied with Sn compositions from 0 (Ge) to 12%. Raman spectra of the GeSn samples with various Sn compositions were measured. The room temperature photoluminescence (PL) spectra show a gradual shift of emission peaks towards longer wavelength as Sn composition increases. Temperature dependent PL shows the PL intensity variation along with the temperature change, which reveals the indirectness or directness of the bandgap of the material. As temperature decreases, the PL intensity decreases with Sn composition less than 8%, indicating the indirect bandgap $\text{Ge}_{1-x}\text{Sn}_x$; while the PL intensity increases with Sn composition higher than 10%, implying the direct bandgap $\text{Ge}_{1-x}\text{Sn}_x$. Moreover, the PL study of *n*-doped samples shows bandgap narrowing compared to the unintentionally (Boron) doped thin film with similar Sn compositions due to the doping.

Key words: GeSn alloy, *n*-doped GeSn, Raman spectroscopy, photoluminescence

INTRODUCTION

Silicon-based materials and their optoelectronic devices are of great interest due to the scalable incorporation with current Si complementary metal-oxide semiconductor (CMOS) processes.¹ Among the various material systems that could be integrated on Si, the $\text{Ge}_{1-x}\text{Sn}_x$ alloy has attracted much attention recently due to the following reasons: (1) Capability of monolithic integration on Si,² (2) Availability of direct bandgap material,³ and (3) tunable bandgap covering broad shortwave- and mid-infrared (IR) wavelength range.⁴ During the

last decade, the GeSn-based optically pumped laser,⁵ light emitting diode,^{6–12} photo detector^{13–20} have been demonstrated, and the GeSn modulator has been investigated^{21,22} which make up a complete set of components for Si photonics. For these prototype devices, the material characteristics have turned out to be the decisive factor for the performance of the device. Specifically, the optical properties of the material dominate the behavior of GeSn-based optoelectronic devices. The preliminary study in analysis of optical properties of $\text{Ge}_{1-x}\text{Sn}_x$ alloy has been reported by several research groups.^{23–33} However, the overall results are still less satisfactory due to the lack of systematic investigation on a set of $\text{Ge}_{1-x}\text{Sn}_x$ alloys with

uniform material quality and various Sn compositions.

In this paper, we report systematic study of high quality $\text{Ge}_{1-x}\text{Sn}_x$ thin films. The thickness and strain of the thin film, the Sn composition, and the defect spreading were investigated by transmission electron microscopy (TEM), x-ray diffraction (XRD), and secondary ion mass spectrometry (SIMS). Optical characterization for unintentionally (Boron) doped $\text{Ge}_{1-x}\text{Sn}_x$ alloys with Sn compositions from 0% to 12% was performed. Room temperature Raman and photoluminescence spectra showing the gradually shifted peak towards a smaller wavenumber and longer wavelength as Sn composition increases were obtained, respectively, indicating the bandgap was altered by the Sn incorporation. Temperature-dependent PL spectra showing the variation of peak position and intensity were analyzed, which revealed the bandgap structure properties (indirectness or directness). Moreover, the n -doped $\text{Ge}_{1-x}\text{Sn}_x$ alloy samples were studied to compare with the unintentionally doped samples (similar Sn composition). The bandgap narrowing for n -doped samples were observed. Understanding the fundamental optical properties of $\text{Ge}_{1-x}\text{Sn}_x$ alloy could provide strong guidance for the design of future $\text{Ge}_{1-x}\text{Sn}_x$ -based optoelectronic devices.

EXPERIMENTAL METHODS

An ASM Epsilon[®] 2000 Plus reduced pressure chemical vapor deposition (RPCVD) system was used to grow $\text{Ge}_{1-x}\text{Sn}_x$ samples. A 700 nm strain-relaxed Ge buffer layer was first deposited on the Si substrate, followed by the growth of $\text{Ge}_{1-x}\text{Sn}_x$ layer. The growth temperature was kept below 450°C to be compatible with the Si CMOS process. The GeH_4 and SnCl_4 were used as Ge and Sn precursor gases,

respectively. For the n -doped samples, the PH_3 precursor gas was employed. Details of the growth information were reported in Ref. 34.

The TEM was used to verify the crystallinity and thickness of $\text{Ge}_{1-x}\text{Sn}_x$ thin films. The lattice constant was measured by XRD, and; therefore, the strain status can be calculated. The Sn composition was confirmed by SIMS. A summary of measurement results is listed in Table I.

Raman spectroscopy was performed at room temperature using a He:Ne laser operating at 632.8 nm with the output power of 10 mW. A nitrogen-cooled Si-CCD array was used as detector with wavelength coverage from 190 nm to 800 nm.

Fig. 1. Cross sectional TEM image of Si/Ge/Ge_{0.9}Sn_{0.1} film shows that the threading dislocations are trapped at the Ge/GeSn interface, resulting in a high quality GeSn layer.

Table I. Summary of measurement results of samples for this study

Sn composition	GeSn thickness (nm)	Strain	Doping	Doping concentration (cm ⁻³)
0 (Ge)	300 (Ge)	0	Unintentionally p -type (Boron) doped	1E17
1%	327	-0.0002		
2%	40	-0.0022		
3%	128	-0.0024		
4%	70	-0.0050		
5%	83	-0.0052		
6%	96	-0.0082		
7%	100	-0.0045		
8%	90	-0.0080		
9%	117	-0.0101		
10%	59	-0.0116		
12%	150	-0.0093		
5%	225	-0.0054	n -doped	6.5E19
8%	276	-0.0059		1E19
10%	338	-0.0050		2.9E18

Temperature-dependent PL measurement was performed at temperatures from 10 K to 300 K. A diode-pumped solid-state (DPSS) continuous-wave (CW) laser operating at 532 nm with output power of 500 mW was used as excitation source. Emissions from the $\text{Ge}_{1-x}\text{Sn}_x$ samples were collected by a

spectrometer, and the collected light was then sent to the detector. Two detectors were employed, an extended InGaAs photodetector (high signal-to-noise ratio) and a lead sulfide (PbS) detector (low signal-to-noise ratio) with cut-off wavelength of 2.3 μm and 3 μm , respectively. In addition, a long-pass filter was placed in front of the spectrometer entrance to eliminate the scattered laser light.

RESULTS AND DISCUSSION

Material Characterization

A typical cross sectional TEM image of a $\text{Ge}_{0.9}\text{Sn}_{0.1}$ sample is shown in Fig. 1. The Si substrate, Ge buffer, and GeSn layer are clearly identified. The film thicknesses of the Ge buffer and GeSn layers were measured at 700 nm and 150 nm, respectively. It can be seen that the threading dislocations are localized at the Ge/GeSn interface, resulting in the low defect density in the GeSn film. The high quality of the material is attributed to the optimized growth recipe.

The $2\theta-\omega$ XRD scan of the GeSn samples from the (004) plane is shown in Fig. 2a and b, for the unintentionally doped and n -doped samples, respectively. The peaks at 69° and 66° are attributed to the Si substrate and the Ge buffer layer, respectively. The peaks that were observed between 66° and 64° are from $\text{Ge}_{1-x}\text{Sn}_x$ thin films. The clearly resolved peak shifts towards lower angles as the Sn composition increases. Since the position of the peak is determined by both the Sn composition and the strain, the peak of the 7% Sn sample locates at the higher angle than that of the 6% Sn sample due to the smaller compressive strain in the 7% sample. The lattice constant of the GeSn films was calculated from the reciprocal space map (RSM, not shown here), based on which the strain can be determined, as listed in Table I. The negative value of the strain indicates the compressive strain, which

Fig. 2. $2\theta-\omega$ XRD scan from (004) plane for (a) unintentionally doped and (b) n -doped samples. The peak at 69° shows the Si substrate and the peaks between 66° and 64° belong to $\text{Ge}_{1-x}\text{Sn}_x$ films.

Fig. 3. Raman spectroscopy for (a) unintentionally doped and (b) n -doped samples. The Ge peak position locates at around 299.9 cm^{-1} , while the GeSn peak was identified ranging from 260.3 cm^{-1} to 254.3 cm^{-1} .

Table II. Raman shift of Ge-Ge and Ge-Sn bonds for unintentionally and *n*-doped samples

Sn composition (%)	Doping	Ge-Ge (cm ⁻¹)	Ge-Sn (cm ⁻¹)
0	Unintentionally (Boron) doped	299.9	
1		299.8	N.A.
2		299.8	N.A.
3		298.4	260.3
4		295.9	257.9
5		298.6	261.6
6		298.1	261.8
7		298.2	260.7
8		297.9	262.0
9		297.7	262.1
10		297.9	259.2
12		292.6	254.3
5	<i>n</i> -doped	294.9	257.9
8		295.9	258.7
10		292.8	255.7

was observed for each sample. As the Sn composition increases, the increased compressive strain was obtained due to the increased lattice constant of GeSn. On the other hand, the sample with thicker GeSn film exhibits lower strain because of the gradual relaxation as the layer thickness increases.

Raman Spectroscopy

Raman spectroscopy measurements were conducted for each sample. Figure 3a and b show the Raman spectra for unintentionally and *n*-doped samples, respectively. In Fig. 3a, as Sn composition increases, the Ge-Ge peak shifts from 299.9 cm⁻¹ (0% Sn) to 292.6 cm⁻¹ (12% Sn). This can be explained as the change of the bond energy of the Ge atoms in the lattice due to the incorporation of Sn. The Ge-Sn peaks were identified ranging from 260.3 cm⁻¹ (3%) to 254.3 cm⁻¹ (12%). For the samples with Sn compositions of 1% and 2%, the intensity of the Ge-Sn peak is relatively weak and,

Fig. 4. (a) Normalized PL spectra of the unintentionally doped GeSn thin films with Sn compositions from 0% to 12% at room temperature. The PL peak position shifts towards the longer wavelength as Sn composition increases, indicating the reduced bandgap energy. (b) PL peak position attributed to the bandgap as a function of Sn composition. The solid and open symbols are extracted by Gaussian fitting, and the solid linear lines are fitted curves showing the indirect-to-direct transition.

Fig. 5. Temperature-dependent PL for GeSn samples with Sn compositions of (a) 5% and (b) 12%. Each curve is stacked for clarity. The extracted bandgap energy and integrated PL intensity as the function of temperature for the samples with Sn compositions of (c) 5% and (d) 12%.

therefore, cannot be identified. In Fig. 3b, the shift of the Ge-Ge peak was observed and Ge-Sn peaks can be identified. Increasing the Sn composition shifts the peak towards the smaller wavenumbers; while the compressive strain shifts the Raman peaks towards the higher wavenumbers. Therefore, the relaxed GeSn sample with 12% Sn would have a smaller wavenumber peak than the compressively strained one. The similar Raman peak shift tendency was also reported in Refs. 35 and 36. Also, the peak position shows the slight difference compared to the unintentionally doped sample with the similar Sn composition, which may be due to the heavy doping that alters the Ge atom's bond energy. The peak position for each sample is summarized in Table II.

Photoluminescence

Normalized PL spectra of the unintentionally doped GeSn thin films with Sn compositions from 0% to 12% at room temperature are shown in Fig. 4a. As Sn composition increases, the PL peak shifts towards longer wavelengths, indicating the reduced bandgap energy due to the incorporation of Sn. For the samples with lower Sn compositions (less than 8%), two emission peaks were clearly

observed, corresponding to the indirect and the direct bandgap transitions, respectively. While for the samples with higher Sn compositions, the indirect and direct peaks cannot be identified due to the small separation between the indirect and direct bandgap energies, resulting in a single peak PL spectrum with broad peak line-width. In addition, the indirect-to-direct transition occurs at the Sn composition of 10%,³ beyond which the GeSn alloy becomes the direct bandgap material, and therefore only a direct peak with narrowed peak line-width can be observed. Note that the noisy PL spectra of GeSn samples with Sn compositions from 8% to 12% is due to the use of PbS detector, which features a $3.0\ \mu\text{m}$ cut-off but with low signal-to-noise ratio. The GeSn sample with 12% Sn composition has lower quality compared to other samples. Therefore, the room temperature PL spectrum looks more noisy compared to the rest of the samples. A low signal to noise ratio causes a fluctuation in the room temperature PL of that sample. However, for extracting the PL peak position with Gaussian fitting function, only one peak was considered based on our theoretical study.

Using the Gaussian fitting method, the PL peak position was extracted for each sample, which

Fig. 6. Comparison of the normalized PL spectra between the unintentionally doped and n -doped GeSn samples with Sn compositions of (a) 5% (b) 8%, and (c) 10% at room temperature. The indirect and direct bandgap shrinkages are summarized in (d).

indicates the bandgap energy of $\text{Ge}_{1-x}\text{Sn}_x$, as shown in Fig. 4b. The solid and open symbols are extracted by Gaussian fitting and the linear lines are fitted curves showing the indirect-to-direct transition. As Sn composition increases, both indirect and direct bandgap energies are reduced with a more rapid decrease of direct bandgap than indirect bandgap, leading to the reduced bandgap energy separation from 89.5 meV (0% Sn) to 12.6 meV (7% Sn). Only one peak can be observed for the samples with Sn compositions from 8% to 12%, which is due to the large spectra overlap of indirect and direct bandgap transitions (8% and 9% Sn samples) or the directness of the GeSn material (10% and 12% Sn samples). The indirect-to-direct transition point locates at the Sn composition slightly below 10%, which agrees well with the discovery that was reported in Ref. 3; however, for relaxed GeSn samples, the transition point could happen for Sn compositions around 6%. The deviation between the Gaussian fitting points and the fitted lines are mainly due to the strain of the samples.

Figure 5a and b show the typical temperature-dependent PL spectra for unintentionally doped

GeSn samples with Sn compositions of 5% and 12%, respectively. Each curve is stacked for clarity. In Fig. 5a, at 300 K, a high energy peak at 0.648 eV and a low energy shoulder at 0.606 eV were observed, which are associated with the direct and indirect bandgap transitions, respectively. The strong direct peak indicates that the direct bandgap transition dominates the PL at room temperature. As the temperature decreases, both direct and indirect peaks decrease with the direct peak dropping more rapidly than the indirect peak, resulting in the indirect bandgap transition dominating the PL at the temperatures below 200 K. At 10 K, the direct peak almost disappears and only the indirect peak was observed. This can be explained by the fact that less electrons occupy the Γ -valley as the result of reduced thermal excitation from the lower L-valley at lower temperatures. The PL peak position and integrated PL intensity against temperature are plotted in Fig. 5c. The bandgap energy increases as temperature decreases, which follows the Varshni relation.⁴ The integrated PL intensity decrease at low temperature was observed. While GeSn with 5% Sn is an indirect bandgap material,

Fig. 7. Temperature-dependent PL spectra for n -doped GeSn samples with Sn compositions of (a) 5% (c) 8%, and (e) 10%. The corresponding bandgap shift and integrated PL intensity against temperature are plotted in (b), (d), and (f), respectively.

this PL intensity drop is expected. Although at low temperatures the defects are less active and radiative recombination should be enhanced, the number of available phonons for the indirect bandgap recombination decreases leading to a reduction of the PL intensity. A peak at 2250 nm was observed and the position and intensity were unchanged with temperature. That peak may be related to a defect,

such as dislocation in the Ge buffer layer.^{37,38} Further investigation is needed to understand the type of the defect and the source of the emission. In Fig. 5b, at 300 K, a single peak at 0.558 eV was observed, which is assigned to the direct bandgap transition. In 300 K, non-radiative recombination centers suppress the PL intensity. As the temperature decreases, the line-width keep decreasing

while the peak intensity increases. At 10 K, a single peak with narrow peak line-width was observed. The PL peak position and integrated PL intensity against temperature are plotted in Fig. 5d, which reveals the direct bandgap material of GeSn with 12% Sn.³ Since the Γ -valley minimum is lower than that of L-valley in direct bandgap GeSn, most electrons tend to populate on Γ -valley; therefore, the indirect transition was dramatically reduced, resulting in the single peak PL spectra.

The PL behavior of the *n*-doped GeSn samples with Sn compositions of 5%, 8%, and 10% were investigated. Normalized room temperature PL spectra of *n*-doped GeSn samples and their comparison with unintentionally doped samples with similar Sn composition are shown in Fig. 6a, b, and c, respectively. The direct bandgap shrinkage due to the *n*-doping is observed as 42 meV, 23 meV, and 36 meV for 5%, 8%, and 10% Sn samples, respectively. For 5% Sn sample, the indirect bandgap shrinkage of 27 meV was obtained as well. The bandgap comparison is summarized in Fig. 6d. It is worth pointing out that, since this is the first time that the doping induced bandgap shrinkage in the GeSn material was observed, more investigation is needed for further understanding the mechanism. It can be seen that the Sn composition, material strain, and doping concentration all play a part for the bandgap alteration.

Furthermore, the temperature-dependent PL spectra were obtained for *n*-doped GeSn samples, as shown in Fig. 7a, c, and e for Sn compositions of 5%, 8%, and 10%, respectively. Each curve is stacked for clarity. The temperature-dependent PL peak positions and integrated PL intensities were extracted by Gaussian fitting and are plotted in Fig. 7b, d, and f for each sample, respectively. All samples exhibit bandgap increase as the temperature decreases that follow the Varshni relation. The fluctuation of data shown in Fig. 7d and f are mainly due to the fitting error of the Gaussian distribution. For the 5% Sn sample, the integrated PL intensity decreases as temperature decreases, indicating the indirectness of the material. While for the 8% and 10% Sn samples, the integrated PL intensities increase at low temperature; this shows the direct bandgap material behavior, which suggests the directness of the material. Specifically, the integrated PL intensity of the 10% Sn sample at 10 K is more than 40 times higher than that at room temperature. This fact may be favorable for the design of a GeSn laser.

CONCLUSIONS

Material and optical characteristics were studied for both unintentionally doped and *n*-doped Ge_{1-x}Sn_x samples with Sn compositions up to 12%. The TEM and XRD show that the grown samples were consistent with the growth target in terms of

the film thickness and Sn composition. The room temperature PL spectra exhibited gradual shifts of emission peaks towards longer wavelengths as Sn composition increases. The temperature-dependent PL spectra revealed the indirectness and directness of the GeSn material. Moreover, the bandgap shrinkage was observed from a *n*-doped GeSn sample for the first time. A comprehensive optical study would benefit the GeSn technology directed towards Si-based optoelectronic devices.

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