

## Formation and properties of GeSn:C films on silicon substrates

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**Abstract.** The paper reports the formation of GeSn:C films on silicon substrates by thermal spraying of Ge/Sn superlattices followed by implantation of C<sup>+</sup> ions into them and annealing at 350 °C. It is shown that compared to Ge films, the crystallization temperature of which is ~500 °C, the crystallization temperature of the GeSn and GeSn:C films is significantly reduced, down to 300 and 350 °C, respectively. We explain this effect by the diffusion of tin atoms in the Ge lattice, which decreases the activation energy of the Ge crystallization process. Implantation of C<sup>+</sup> ions into a Ge/Sn superlattice reduces the effect of Sn atoms on the crystallization process and, accordingly, increases the crystallization temperature. We have confirmed experimentally that implantation of C<sup>+</sup> ions in Ge/Sn structures contributes to the reduction of local stresses in the Ge lattice induced by the incorporation of Sn atoms and thus possibly increases the concentration of incorporated Sn atoms in the Ge lattice. It has been established as well that laser annealing of Ge/Sn structures implanted with C<sup>+</sup> ions is more effective for embedding Sn atoms in the Ge lattice as compared to thermal annealing.

**Keywords:** GeSn films, GeSnC films, implantation, XRD, Raman spectroscopy, SIMS.

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### 1. Introduction

Today, Si technology is the base for more than 90% of all electronics in the world. However, it has a serious problem associated with the indirect gap of Si, which, in particular, does not allow the production of efficient light emitters/absorbers, which are a necessary link in the chain of transmission and registration of optical signals in electronic microcircuits made based on CMOS technology. A promising way to overcome this problem is to form Ge<sub>1-x</sub>Sn<sub>x</sub> films on Si substrates. The electronic structure of such films becomes direct-band at a certain value of  $x$  (6–9%) [1–3]. The differences in the Sn fraction in Ge<sub>1-x</sub>Sn<sub>x</sub> [1–3], at which the transition from the indirect-gap to the direct-gap electronic structure takes place, may be caused by mechanical stresses present in the films [3, 4]. A decrease in compressive stresses in GeSn films, or even better – formation of tensile stresses in them – would reduce the amount of Sn required to ensure the transition from the indirect-gap to the direct-gap structure (the ID transition, IDT). Moreover, the IDT is affected by the concentration of free electrons in Ge<sub>1-x</sub>Sn<sub>x</sub>, since at its high value, the Burstein–Moss effect can be realized, also leading to the IDT [5]. It should be noted that during the formation of Ge<sub>1-x</sub>Sn<sub>x</sub> films, not only the lattice parameters change and the electronic structure is modified as compared to pure Ge, but the film photosensitivity also expands to the long-wavelength range [6].

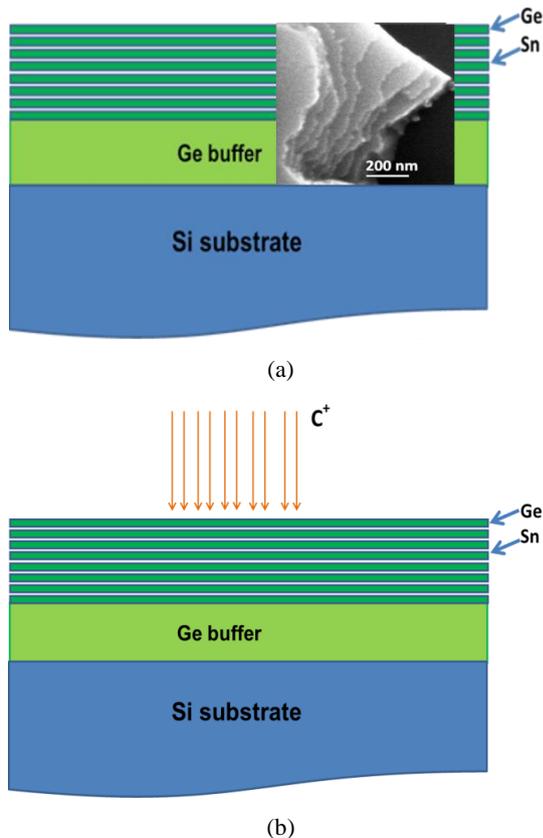
Formation of crystalline Ge<sub>1-x</sub>Sn<sub>x</sub> films with a relatively high Sn content,  $x \geq 6\%$ , is quite a difficult task since Sn is characterized by low solubility (~0.5%) in Ge. The latter is caused by a much larger covalent radius of Sn atoms compared to Ge atoms (162 pm vs 137 pm, respectively). When Sn atoms are embedded in the crystal lattice of Ge, local stresses arise, which lead to segregation of Sn at a certain concentration. As a rule, GeSn films with high tin contents are obtained by the non-equilibrium molecular beam epitaxy (MBE) method [7, 8]. However, this method is expensive and is mainly used to create laboratory samples. To embed Sn atoms in a Ge lattice by magnetron or thermal sputtering of the corresponding targets, it is necessary to compensate for the local mechanical stresses that arise. The main idea of our research is to compensate for local stresses by additional incorporation of C atoms into the Ge films because these atoms have a significantly smaller covalent radius (91 pm) compared to Sn and Ge.

Simultaneous incorporation of Sn and C atoms into the Ge lattice should compensate for local stresses arising during the embedding of only Sn atoms and increase the solubility of tin in Ge. Therefore, this work is aimed at a study of the effect of carbon ion implantation into Ge/Sn layers with subsequent laser and thermal anneals of such structures on the efficiency of Sn atom incorporation into the Ge crystal lattice.

## 2. Experimental technique

To test the idea described above, a 100 nm thick Ge buffer layer was deposited on a silicon wafer using thermal sputtering in a vacuum ( $6.7 \cdot 10^{-4}$  Pa). A 10-period superlattice (SL) with  $\sim 17$  and  $\sim 1$  nm thick Ge and Sn layers, respectively, was formed on the top of the buffer layer (Fig. 1). After the sputtering, the resulting structure was cut into 2 parts, one of which was implanted with  $C^+$  ions at an energy of 50 keV and a dose of  $6.2 \cdot 10^{15}$  ion/cm<sup>2</sup>, while the other part was used for comparison in subsequent studies. The films formed in this way were annealed by two methods, namely: (1) laser anneals for 60 s at the radiation wavelength  $\lambda = 532$  nm and the power density varied from 1 to 50 kW/cm<sup>2</sup>, and (2) thermal anneals in an Ar atmosphere in the temperature range of 250 to 500 °C during the time varied from 15 min to 8 h. The formed films were studied by mass spectroscopy, X-ray diffractometry, Raman spectroscopy, and SEM microscopy.

To record the Raman spectra, we used an MDR-23 spectrometer equipped with an iDus 420 Andor cooled CCD detector. The Raman signals were excited by a solid-state laser line with a wavelength of 532 nm. The laser power density on the sample surface was less than  $10^3$  W/cm<sup>2</sup>, which excluded thermal modification of the samples. X-ray diffraction analysis was performed using a Philips X'Pert PRO-MRD setup with a Cu K $\alpha$  line

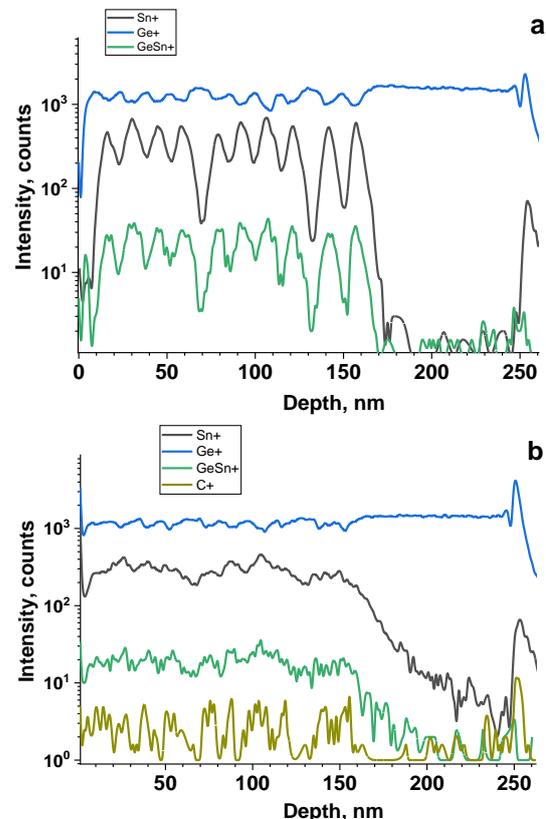


**Fig. 1.** Schematic representation of the initial structures for the formation of GeSn (a) and GeSn:C (b) films on pre-formed Ge buffer layers on Si substrates. The inset in Fig. 1a shows the SEM image of the fracture of the initial Ge/Sn structure.

( $\lambda = 0.15406$  nm) as an excitation source in a symmetric ( $2\Theta - \omega$ ) mode with a scanning step of 0.025 degrees. The voltage at the tube anode was 45 kV and the current was 40 mA. The elemental composition of the Ge/Sn multilayer structures was studied by dynamic (D-) secondary mass spectrometry (SIMS). D-SIMS measurements were performed on an ATOMIKA 4000 quadrupole instrument (Perkin Elmer, Germany). The depth distribution profiles of elements were obtained using a 200 nA  $O_2^+$  primary ion beam with an energy of 5 keV. The angle of incidence of the beam with respect to the surface normal was  $0^\circ$ . The lateral square raster of the crater was  $500 \times 500 \mu m^2$ . The analyzed area was 16% in the center of the formed crater. The morphology of the surfaces of the samples and their chips was studied using a scanning electron microscope (SEM, Tescan Mira 3 MLU).

## 3. Results and discussion

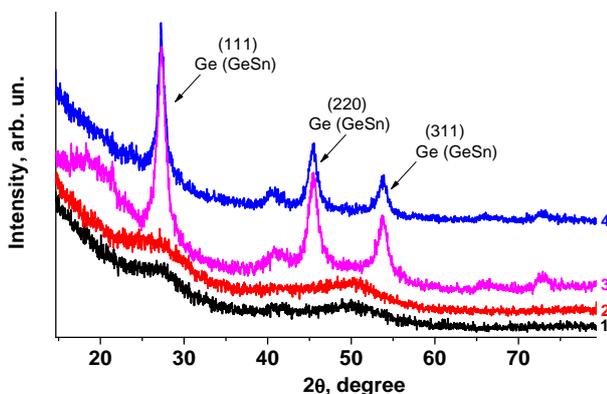
Fig. 2a shows the depth profiles of the SIMS signals from Ge, Sn, and the GeSn polyatomic complex in a 10-period Ge/Sn structure on the previously formed Ge buffer layer. It is evident from this figure that the 10-period Ge/Sn structure was indeed formed during the deposition of Ge and Sn, but the layer interfaces were not sharp, which indicates partial diffusion of the elements from different layers already during their deposition.



**Fig. 2.** Distributions of Ge and Sn atoms, and GeSn nano-clusters in the initial Ge/Sn structure (a) and Ge, Sn and C atoms, and GeSn nano-clusters in the Ge/Sn structure implanted with  $C^+$  ions followed by annealing in an Ar atmosphere at 500 °C for 15 min (b). For interpretation of the colors in the figure(s), the reader is referred to the web version of this article.

Moreover, as can be seen from Fig. 2a, GeSn polyatomic complex were already formed during the deposition process. These complexes could be precursors for formation of the GeSn layers during subsequent annealing. Fig. 2b shows the depth profiles of the SIMS signals from Ge, Sn, C, and the GeSn polyatomic complex from the studied structure after implantation of  $C^+$  ions with  $E = 50$  keV and a dose of  $6.2 \cdot 10^{15}$  ion/cm<sup>2</sup> into the above structure as well as after thermal annealing in an Ar atmosphere at 500 °C for 15 min. It is evident from this figure that annealing of such samples results in equalization of the distribution of Ge and Sn atoms over the film thickness, although minor irregularities are still observed. The latter points to the necessity to increase the annealing time of the samples or decrease the thickness of the initial layers. Moreover, the distribution of Sn atoms shown in Fig. 2b evidences that annealing at  $T = 500$  °C leads to the Sn atom diffusion to the Ge-buffer/Si-substrate interface. Therefore, the annealing temperature should be decreased in the future, since its value must only ensure crystallization of the formed films and not contribute to the diffusion of incorporated Sn and C atoms to the film surface and the film/substrate interface. We determined the onset of film crystallization using X-ray diffractometry and Raman spectroscopy.

Fig. 3 shows the X-ray diffraction patterns of the GeSn and GeSn:C films annealed at different temperatures. It is evident from this figure that the films annealed at 250 °C are amorphous. Annealing at  $T = 350$  °C and above leads to film crystallization. A more detailed study of the film crystallization process using Raman spectroscopy showed that GeSn films crystallize at 300 °C, and GeSn:C films at 350 °C. It is well known that pure Ge films on Si substrates crystallize at a temperature of ~500 °C [9]. Presence of a metal layer on the surface of a Ge film leads to a decrease in the crystallization temperature. Such metals as Au, Al, and Sn have a particular effect on this process [10]. Diffusion of a metal into the film leads to a decrease in the activation energy of the crystallization process. As our studies have shown, additional implantation of GeSn



**Fig. 3.** XRD diffractograms of the GeSn and GeSn:C films annealed at different temperatures: (1) GeSn at 250 °C for 8 h, (2) GeSn:C at 250 °C for 8 h, (3) GeSn:C at 350 °C for 15 min, and (4) GeSn:C at 500 °C for 15 min.

films with  $C^+$  ions leads to an increase in the crystallization temperature to 350 °C, which may be due to a decrease in the influence of Sn atoms on the activation energy of the Ge crystallization process.

Fig. 4 shows the Raman spectra of the Ge/Sn structures normalized to the intensity of Ge-Ge bands after laser annealing at different radiation powers for the case without implantation (a) and with additional implantation of  $C^+$  ions ( $E = 50$  keV,  $D = 6.2 \cdot 10^{15}$  ion/cm<sup>2</sup>) (b).

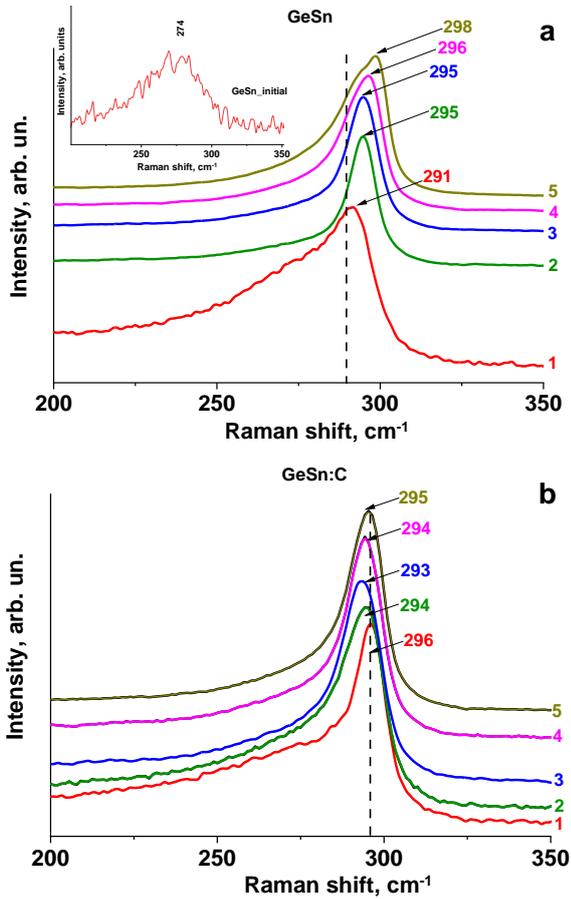
The inset in Fig. 4a shows the Raman spectrum of the initial film. The latter is characterized by a structureless band with a large half-width of 60 cm<sup>-1</sup> and a maximum at 274 cm<sup>-1</sup>, which corresponds to the spectrum of amorphous germanium. Even minor laser annealing of the initial GeSn film leads to its partial crystallization, and the experimental spectrum is characterized by the two bands with the frequencies of 274 and 291 cm<sup>-1</sup> (Fig. 4a, spectrum 1), which are characteristic of the amorphous phase of Ge and Ge nanocrystals (NCs) present in it, respectively.

It is known that, presence of Ge NCs with sizes less than ~15 nm in films leads to a shift of the Ge-Ge band to the low-frequency side, as well as to its broadening and asymmetry, compared to bulk Ge [11, 12].

At the same time, introduction of Sn atoms into the Ge lattice also leads to a low-frequency shift, broadening, and asymmetry of the Ge-Ge band [13–15]. The asymmetry in the latter case is due to the appearance of the so-called defect-activated (DA) band in the Raman spectrum of GeSn, which is a manifestation of the density of phonon states activated by the introduction of Sn atoms into the Ge lattice. The low-frequency shift of the Ge-Ge band can also be caused by tensile stresses in GeSn films. However, in our case, the Ge (or GeSn) film on the Si substrate can have only compressive stresses, which lead to a Ge-Ge band shift in the high-frequency direction.

Laser annealing of Ge/Sn films with an increase in the radiation power density to 5 kW/cm<sup>2</sup> leads to a shift of the Ge-Ge band in the high-frequency direction, and decrease in its half-width ( $\Gamma$ ) from 20 to 12 cm<sup>-1</sup> (Fig. 4a, spectrum 2). Such changes are explained by increasing the average size of NCs on the one hand and precipitation of Sn atoms with the formation of clusters or/and their diffusion to the Ge/Si interface and the film surface on the other hand. Indeed, the decrease in the concentration of Sn atoms in the Ge lattice leads to an improvement of the crystal structure and, accordingly, to an increase in the lifetime of phonons ( $t_{phon} \sim 1/\Gamma$ , where  $t_{phon}$  is lifetime of phonons,  $\Gamma$  is a half-width of the corresponding phonon band) and a shift of the Ge-Ge band in the high-frequency direction. Further increase in the power density of laser radiation leads to a broadening Ge-Ge bands (spectra 3, 4, 5). Such changes are caused by unequal heating of near-surface layers and layers lying near the Ge/Sn-Ge buffer layer interface. On spectrum 5 (Fig. 4a), it is already clearly visible that the band consists of two components.

A completely different picture is observed in the Raman spectra during laser annealing of Ge/Sn layers with varying radiation power with implantation of



**Fig. 4.** Normalized Raman spectra of the Ge/Sn structures after laser annealing at different radiation power: 1(1), 5(2), 10(3), 25(4), and 20(5) kW/cm<sup>2</sup> (a) without implantation of C<sup>+</sup> ions; (b) with additional implantation of C<sup>+</sup> ions with an energy of 50 keV and a dose of  $6.2 \cdot 10^{15}$  ion/cm<sup>2</sup>.

C<sup>+</sup> ions (Fig. 4b). Laser annealing of the initial GeSn:C film at a radiation power density of 1 kW/cm<sup>2</sup> leads to its partial crystallization, and the experimental spectrum is characterized by two bands with frequencies of 274 and 296 cm<sup>-1</sup> (Fig. 4b, spectrum 1), which are characteristic of the amorphous phase of germanium and the Ge NCs present in it, respectively. An increase in the radiation power density to 5 kW/cm<sup>2</sup> leads to a shift of the Ge-Ge band in the low-frequency direction, an increase in its half-width ( $\Gamma$ ) from 10 to 16 cm<sup>-1</sup> (Fig. 4b, spectrum 2). Further increase in the radiation power density to 10 kW/cm<sup>2</sup> leads to a further slight shift of the Ge-Ge band towards low frequencies (Fig. 4b, spectrum 3). Such changes are explained by the crystallization of the deposited germanium layers and the incorporation of Sn and C atoms into them. Indeed, when incorporating both Sn and C atoms into a crystalline Ge lattice, the Ge-Ge band shifts to the low-frequency side and its half-width increases [13, 14]. The latter is due to the embedding of tin and carbon atoms, which disrupts the ordering of the germanium lattice and leads to a shorter phonon lifetime. If a film is not single-crystalline but consists of NCs, then with increasing annealing temperature the NC size increases, which may lead (at  $D_{NC} < 15$  nm) to a shift of

the Ge-Ge band toward higher frequencies and a decrease in its half-width [11]. Therefore, in our case, just the effect of the incorporation of Sn and C atoms into the Ge lattice manifests itself at increasing  $T_{ann}$  and with a shift of the Ge-Ge band in the low-frequency region. Further increase in the laser radiation power to 50 kW/cm<sup>2</sup> leads to an insignificant ( $\sim 1$  cm<sup>-1</sup>) shift of the Ge-Ge band toward higher frequencies and a decrease in its half-width (Fig. 4b, spectrum 5). Such changes in the spectral characteristics of the Ge-Ge band may be due to partial precipitation of Sn and C atoms from the lattice and formation of Sn and C clusters or/and their diffusion to the film surface and the Ge/Si interface, as well as a decrease in the effect of size limitation of Ge NCs, if it took place in the previous cases.

Therefore, comparison of the change in the Raman spectra of the GeSn films without implantation and implanted with C<sup>+</sup> ions shows that the process of incorporation of Sn ions into the Ge crystal lattice is significantly changed in the latter films. Simultaneous incorporation of Sn and C ions into the germanium lattice reduces the local stresses caused by the different covalent radii of Sn, C and Ge atoms, and allows the incorporation of Sn ions at significantly higher concentrations. The frequency position of the Ge-Ge band determines the percentage of Sn atoms embedded in the germanium lattice according to the following expression:  $\omega_{GeSn(x)}^{Ge-Ge} = \omega_{Gebulk}^{Ge-Ge} - \alpha x$ , where  $\omega_{GeSn(x)}^{Ge-Ge}$  is the frequency position of the Ge-Ge band in GeSn films;  $\omega_{Gebulk}^{Ge-Ge}$  is the frequency position of the Ge-Ge band in bulk Ge and  $\alpha = 75.4 \pm 4.5$  cm<sup>-1</sup> [13], respectively. According to this expression, the Sn content in our GeSn:C and GeSn films is 8 and 6 at. percent, respectively. However, this is true only if the frequency position of the Ge-Ge band is not affected by the size effect, which is quite difficult to estimate in this case.

The structural changes occurring in both types of the samples during thermal annealing at temperatures varying from 250 to 500 °C generally correlate with the structural changes occurring during laser annealing. However, significantly longer annealing times and absence of the effect of irradiation reduce the efficiency of embedding Sn and C atoms in the Ge lattice. This is evidenced by higher frequency positions of the Ge-Ge bands and their smaller half-widths (spectra not shown).

#### 4. Conclusions

GeSn and GeSn:C films on Ge/Si buffer layers were obtained by forming Ge/Sn superlattices with different thicknesses of the Ge and Sn layers by thermal sputtering followed by implantation of C<sup>+</sup> ions into some of them and laser and thermal annealing in an Ar atmosphere.

Using X-ray diffraction and Raman spectroscopy, the amorphous structure of the initial films was established. Changing the temperature from 200 to 500 °C during thermal annealing and the laser radiation power density from 1 to 50 kW/cm<sup>2</sup> during laser annealing made it possible to track the processes of film crystallization, incorporation of Sn atoms into the Ge lattice, and lattice relaxation during the precipitation of

Sn atoms. It was shown that, compared to pure Ge films, the crystallization temperature of which is  $\sim 500$  °C, presence of tin layers in the initial multilayer GeSn structures leads to a decrease in the crystallization temperature of GeSn and GeSn:C films to 300 and 350 °C, respectively.

Using Raman spectroscopy, it was found that laser annealing leads to more efficient incorporation of Sn and C atoms into the Ge lattice compared to thermal annealing. Laser radiation has a stronger effect on the defect subsystem and helps to reduce the activation energy of the crystallization process as well as the time during which effective diffusion of Sn and C atoms to the film surface and interface with the substrate occurs. The idea that implantation of  $C^+$  ions into GeSn helps to reduce local stresses, thereby increasing the concentration of incorporated Sn atoms in the Ge lattice, was confirmed.

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**Dubikovskiy O.V.:** SIMS measurement, data analysis.

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#### Формування та властивості GeSn:C плівок на кремнієвих підкладках

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**Анотація.** Продемонстровано формування GeSn:C плівок на кремнієвих підкладках шляхом термічного напилення Ge/Sn надграток з наступними імплантацією в них іонів C<sup>+</sup> та відпадом при температурі 350 °С. Показано, що у порівнянні з Ge плівками, температура кристалізації яких становить ~400–500 °С, температура кристалізації GeSn та GeSn:C плівок суттєво знижується до 300 та 350 °С відповідно. Такий ефект пояснено дифузєю атомів олова в Ge ґратку, що приводить до зниження енергії активації атомів Ge в процесі кристалізації. Імплантація іонів C<sup>+</sup> у Ge/Sn надгратки зменшує вплив атомів Sn на цей процес і, відповідно підвищує температуру кристалізації. Експериментально підтверджено ідею, що імплантація іонів C<sup>+</sup> у Ge/Sn структури сприяє зменшенню локальних напружень, що виникають у Ge ґратці при інкорпоруванні в ній атомів Sn, і тим самим збільшує можливість концентрацію вбудованих атомів Sn у Ge ґратці. Встановлено, що лазерний відпал Ge/Sn структур з імпантованими в них іонами C<sup>+</sup> є більш ефективним у порівнянні з термічним відпадом для вбудовування атомів Sn у Ge ґратку.

**Ключові слова:** GeSn плівки, GeSnC плівки, імплантація, XRD, раманівська спектроскопія, SIMS.