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Thermal stability of germanium-tin (GeSn) fins

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We investigate the thermal stability of germanium-tin ($\text{Ge}_{1-x}\text{Sn}_x$) fins under rapid thermal annealing in N_2 ambient. The $\text{Ge}_{1-x}\text{Sn}_x$ fins were formed on a GeSn-on-insulator substrate and were found to be less thermally stable than blanket $\text{Ge}_{1-x}\text{Sn}_x$ films. The morphology change and material quality of the annealed $\text{Ge}_{1-x}\text{Sn}_x$ fin are investigated using scanning electron microscopy, Raman spectroscopy, high-resolution transmission electron microscopy, energy-dispersive X-ray spectroscopy, and electron energy loss spectroscopy. Obvious degradation of crystalline quality of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin was observed, and a thin Ge layer was formed on the SiO_2 surface near the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin region after 500 °C anneal. A model was proposed to explain the morphology change of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin. Published by AIP Publishing. <https://doi.org/10.1063/1.5006994>

Germanium-tin ($\text{Ge}_{1-x}\text{Sn}_x$) alloys have attracted great attention for their potential applications in both nano-electronic and photonic devices.^{1–7} In particular, $\text{Ge}_{1-x}\text{Sn}_x$ p-channel metal-oxide-semiconductor field-effect transistors (p-MOSFETs) have been demonstrated to have higher hole mobility than Ge channel p-MOSFETs.^{1,8–12} Recently, excellent electrostatic performance was achieved by a $\text{Ge}_{1-x}\text{Sn}_x$ p-channel fin field-effect transistor (p-FinFET) fabricated on a fully compressively strained GeSn-on-insulator (GeSnOI) substrate.¹³ In order to exploit the high mobility benefit of the $\text{Ge}_{1-x}\text{Sn}_x$ channel, the material quality and integrity should be well-maintained during the device fabrication or thermal processing steps. However, because of the low surface energy and large covalent radius of the Sn atom, it has a tendency to segregate to the surface during thermal treatment.¹⁴ This could lead to material degradation and a reduced Sn composition in the bulk $\text{Ge}_{1-x}\text{Sn}_x$ region, thus degrading the electrical properties of $\text{Ge}_{1-x}\text{Sn}_x$ MOSFETs. Therefore, investigation of the thermal stability of $\text{Ge}_{1-x}\text{Sn}_x$ is very important, as it provides a guideline on the highest thermal budget that can be used in the fabrication of $\text{Ge}_{1-x}\text{Sn}_x$ MOSFETs.

For blanket $\text{Ge}_{1-x}\text{Sn}_x$ materials grown on the Ge substrate, thermal stability studies have been reported for various Sn compositions ranging from 7.8% to 17%.^{14–17} For instance, when $\text{Ge}_{0.922}\text{Sn}_{0.078}$ is annealed at 620 °C, a Sn-rich surface layer would form.¹⁵ When $\text{Ge}_{0.915}\text{Sn}_{0.085}$ is annealed at 500 °C, reduction of bulk Sn composition and formation of three-dimensional (3D) islands were observed.¹⁴ When $\text{Ge}_{0.9}\text{Sn}_{0.1}$ was annealed at 450 °C, obvious degradation in photoluminescence (PL) intensity and surface nanodot formation can be observed.¹⁶ When the Sn

composition is very high, e.g., for $\text{Ge}_{0.83}\text{Sn}_{0.17}$, annealing at a temperature as low as 300 °C causes surface migration of Sn atoms to form Sn wires.¹⁷ The thermal stability of the $\text{Ge}_{1-x}\text{Sn}_x$ material was found to be poorer with increasing Sn mole fraction x .

For future high performance MOSFETs, fins and nanowire channel structures are needed for better control of short channel effects (SCEs). However, the thermal stability of $\text{Ge}_{1-x}\text{Sn}_x$ fins and nanowires could be different from that of blanket materials due to the existence of additional exposed surfaces which can enhance the surface diffusivity of atoms.^{18–21} Therefore, it is important to study the thermal stability of $\text{Ge}_{1-x}\text{Sn}_x$ fin structures as it is related to the realization of high performance $\text{Ge}_{1-x}\text{Sn}_x$ multi-gate transistors.

In this letter, the thermal stability and morphology change of $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins formed on a GeSnOI substrate are studied for annealing temperatures ranging from 300 °C to 500 °C. Raman spectroscopy indicates that the thermal stability of $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins is poorer than that of the blanket $\text{Ge}_{1-x}\text{Sn}_x$ film. Annealing the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins at 500 °C could result in Sn segregation and desorption, obvious Ge diffusion, and degradation of fin crystalline quality, as confirmed using high resolution transmission electron microscopy (HRTEM), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and electron energy loss spectroscopy (EELS).

The GeSnOI substrate used in this study is fabricated utilizing a direct wafer bonding and etch-back technique.²² Although the compressively strained $\text{Ge}_{1-x}\text{Sn}_x$ grown by chemical vapor deposition (CVD) was used in the realization of $\text{Ge}_{1-x}\text{Sn}_x$ p-FinFET, high hole mobility $\text{Ge}_{1-x}\text{Sn}_x$ p-MOSFETs have also been realized using the $\text{Ge}_{1-x}\text{Sn}_x$ material grown by molecular beam epitaxy (MBE).^{1,10} In order to avoid the strain effect on the $\text{Ge}_{1-x}\text{Sn}_x$ thermal stability, the GeSnOI substrate with a relaxed $\text{Ge}_{1-x}\text{Sn}_x$ film grown by MBE was used. The details of the GeSnOI substrate formation

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FIG. 1. (a) Top-view SEM image of one $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin test structure. The green circle shows the laser spot for the Raman measurement. (b) XTEM image of one GeSn fin ($W_{\text{Fin}} = 40 \text{ nm}$) with a HSQ capping layer on top. (c) HRTEM image of the GeSn fin region shows that the GeSn fin is single crystalline. (d) Dark-field TEM, (e) Ge, (f) Si, and (g) Sn EDX elemental mappings show the contour of the GeSn fin before annealing. The HSQ capping layer was removed before the thermal annealing process.

can be found in Ref. 23. The Sn composition is 4%, and the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ layer thickness is 100 nm. The $\text{Ge}_{0.96}\text{Sn}_{0.04}$ film has a small compressive strain of $\sim 0.1\%$ and is almost fully relaxed. After surface cleaning using diluted hydrofluoric acid (DHF, $\text{HF}:\text{H}_2\text{O} = 1:100$), dense fin patterns with a fin width (W_{Fin}) of 40 nm, a fin gap (G_{Fin}) of 100 nm, and a fin length (L_{Fin}) of $5 \mu\text{m}$ were prepared using electron beam lithography (EBL) and hydrogen silsesquioxane (HSQ) as the resist. The fin patterns were aligned along the $\langle 110 \rangle$ direction. Dense fin patterns are necessary for the purpose of obtaining strong Raman signals.²⁴ The patterns were then transferred using a Cl-based inductively coupled plasma (ICP) dry etching. After that, HSQ was removed using DHF. Figure 1(a) shows the top-view SEM image of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin test structure. 50 $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins were well-defined with two anchor pads connected at the two ends. Cross-sectional TEM (XTEM) was also used to check the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin quality before annealing. The focus-ion-beam (FIB) cut was done along the dashed line AA' as shown in Fig. 1(a). Figure 1(b) shows the XTEM image of one $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin ($W_{\text{Fin}} = 40 \text{ nm}$). The HSQ resist cap and the buried oxide layer can also be seen. The HRTEM image in Fig. 1(c) shows that the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin is single-crystalline. The dark-field TEM (DF-TEM) and EDX atomic mappings in Figs. 1(d)–1(g) clearly show the contour of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin.

After removing the HSQ resist cap, thermal annealing of these $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin structures was performed using a rapid thermal annealing (RTA) system. The samples were annealed at temperatures ranging from 300°C to 500°C in N_2 ambient with a duration of 5 min. Three samples were prepared for this annealing study: one $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin

sample, one $\text{Ge}_{0.96}\text{Sn}_{0.04}\text{OI}$ blanket sample, and one Ge fin sample formed on the Ge-on-insulator (GeOI) substrate. Raman spectroscopy was carried out to analyze the material quality of these three samples after thermal annealing. A Witec alpha 300R confocal Raman system equipped with a 532 nm green laser source was used for the measurements. The Raman laser spot size is $\sim 1 \mu\text{m}$. The laser intensity was fixed at 1.1 mW with an integration time of 2 s for each measurement. The Raman measurements were performed on the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin region as indicated by the green circle in Fig. 1(a).

Figure 2 shows the Raman spectra of annealed (a) blanket $\text{Ge}_{0.96}\text{Sn}_{0.04}$ sample, (b) $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin sample, and (c) Ge fin sample. The Raman spectra of the Ge bulk sample are also shown in each plot as a reference. For the blanket $\text{Ge}_{0.96}\text{Sn}_{0.04}$ sample, the Ge-Ge peak position does not change (298.4 cm^{-1}) for annealing temperatures up to 500°C . The negligible change in the full-width-half-maximum (FWHM) of the Ge-Ge Raman peak ($8.0 \pm 0.4 \text{ cm}^{-1}$ for blanket GeSnOI and $8.5 \pm 0.3 \text{ cm}^{-1}$ for $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins) indicates that the material quality is maintained. For the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin sample, the Ge-Ge peak position and FWHM remain the same for annealing temperatures up to 400°C . However, the Ge-Ge peak shifted back to 301.5 cm^{-1} (bulk Ge peak position) along with higher FWHM and reduced Raman intensity when the annealing temperature was increased to 500°C . The Raman peak shifting back to the bulk Ge peak position after 500°C anneal indicates that Sn segregation and desorption may have already occurred at the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin surfaces.¹⁴ The same annealing experiments were also performed on Ge fins formed on the GeOI

FIG. 2. Raman spectra of the Ge-Ge vibration mode in the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ layer from (a) blanket GeSnOI substrate, (b) $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin sample, and (c) Ge fin sample annealed at temperature ranging from 300°C to 500°C . The Raman spectra of the non-annealed bulk Ge substrate are also presented as reference.

FIG. 3. SEM images of (a) and (b) as-defined $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins and (c) and (d) 300 °C, (e) and (f) 400 °C, and (g) and (h) 500 °C annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins. (a), (c), (e), and (g) Top-view SEM images. (b), (d), (f), and (h) Tilted-view SEM images. The $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin surfaces become rough after 500 °C anneal.

substrate as shown in Fig. 2(c). No change in the Ge-Ge peak position and FWHM was observed with annealing temperature up to 500 °C. These results reveal that the thermal stability of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins is poorer than that of the blanket $\text{Ge}_{0.96}\text{Sn}_{0.04}$ film and Ge fins.

SEM was also used to examine the morphology change of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins after thermal annealing at different temperatures. Figures 3(a) and 3(b) show the top-view and tilted-view SEM images of the as-defined $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins ($W_{\text{Fin}} = 40$ nm). Figures 3(c)–3(h) show the top-view and tilted-view SEM images of the annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fins at the temperatures of 300 °C, 400 °C, and 500 °C, respectively.

No obvious fin morphology change was observed for annealing temperatures up to 400 °C as shown in Figs. 3(c)–3(f), whereas a dramatic change in the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin morphology can be seen in Figs. 3(g) and 3(h) at 500 °C. The surface morphology change could be related to the Sn segregation which was also observed on annealed $\text{Ge}_{1-x}\text{Sn}_x$ blanket samples at high temperatures.^{14,16}

HRTEM was performed on the 500 °C annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin to understand the mechanism of this morphology change. Figure 4(a) shows the HRTEM image of one $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin annealed at 500 °C. Before annealing, this $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin has the same W_{Fin} , L_{Fin} , and fin height (H_{Fin}) as the one shown in Fig. 1(b). An obvious fin morphology change, including reduction of W_{Fin} and H_{Fin} , can be observed. The $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin crystalline quality was also degraded after the 500 °C anneal. This is consistent with the Raman results for the 500 °C annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin sample shown in Fig. 2(b). Two interesting phenomena are also observed: (1) the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin shape became like a capital letter “I” with a flat top region [region (i)], vertical body region [region (ii)], and flat bottom region [region (iii)]; (2) A thin amorphous layer (3–5 nm) is observed on the SiO_2 surface near the annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin region and connected with the annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin. EELS was used to analyze the chemical composition of the annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin and the thin layer formed on the SiO_2 surface. The dark-field TEM image of one annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin, Si, Ge, and Sn EELS elemental mappings are shown in Figs. 4(b)–4(e), respectively. The Sn signal cannot be detected in the 500 °C annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin, which reveals that Sn may have segregated and desorbed from the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin surfaces.¹⁴ Ge signals were detected in both the fin region and the thin layer on the SiO_2 surface. The contour of the annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin is clearly delineated in the Ge elemental mapping. This indicates that the thin layer formed on the SiO_2 surface is a Ge layer. The thin Ge layer formed on the SiO_2 surface should be related to the Ge diffusion from the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin region to the SiO_2 surface after the 500 °C anneal. It should be noted that N_2 ambient was used in this work. Hydrogen-termination on Ge surfaces has been reported to lower the surface diffusivity of Ge atoms.^{25–27} Annealing in H_2 ambient may improve the thermal stability of $\text{Ge}_{1-x}\text{Sn}_x$ possibly from suppression of surface diffusivity of Ge and Sn atoms. Another possible method to reduce Sn desorption or segregation is to perform

FIG. 4. (a) HRTEM image, (b) dark-field TEM image, (c) Si, (d) Ge, and (e) Sn EELS elemental mappings of one $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin after 500 °C anneal. An obvious morphology change was observed after 500 °C anneal, and a thin Ge layer was observed on the SiO_2 surface as confirmed using EELS.

FIG. 5. (a) Schematics showing the proposed process of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin morphology change due to (b) the Sn segregation and desorption, (c) vacancy-assisted Ge diffusion, and (d) surface energy difference and Ge reaction with SiO_2 .

the annealing in a chamber with Sn overpressure, but such an annealing chamber is currently unavailable.

A model is proposed to explain the morphology change of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin after thermal treatment, as illustrated in the schematics in Fig. 5: Sn segregation and desorption [Fig. 5(b)], Ge diffusion [Fig. 5(c)], and Ge reaction with SiO_2 [Fig. 5(d)]. Due to the larger surface/volume ratio of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin structure, Sn segregation to the surface and desorption are favored at elevated temperatures as compared to those of the blanket GeSnOI material.²⁸ This process introduces vacancies in the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin body region as shown in Fig. 5(b).¹⁴ At high temperatures, vacancy-assisted Ge self-diffusion is the dominant diffusion mechanism in Ge as reported by Seeger and Chik.^{29,30} In the 500 °C annealed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin, the vacancies created by Sn atom segregation and desorption enhance the Ge atom diffusion both at the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin surfaces and in the body region, as illustrated in Fig. 5(c), leading to the degradation of crystalline quality of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin.

Since a Ge concentration gradient exists between the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin region and the SiO_2 surface, a net diffusion of Ge atoms from the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin region to the SiO_2 surface can occur.³¹ This results in the formation of a Ge thin layer on the SiO_2 surface near the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin region. The Ge atoms can also react with SiO_2 at elevated temperatures and form volatile GeO and SiO, which can desorb from the surface.^{32,33} The SiO_2 loss near the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin bottom region [region (iii) in Fig. 4(a)] can further enlarge the SiO_2 undercut area (originally caused by DHF dip during the step of HSQ cap removal) in region (iii), as shown in Fig. 5(d). This explains the formation of the thin amorphous Ge layer on the SiO_2 surface near the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin region.

The formed GeSnOI substrate has a (100) top surface, and the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin was patterned along the $\langle 110 \rangle$ direction, as shown in Fig. 3. The formed $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin sidewalls are close to (110) surfaces. However, undercuts in the fin top region [region (i) in Fig. 4(a) after anneal] after the fin etching were also observed, which are close to (111) surfaces, as shown in Fig. 1(b). It has been reported that the surface energy of the Ge (110) surface (2.127 eV) is larger than that of the Ge (100)

surface (1.691 eV) and the (111) surface (1.128 eV).³⁴ Therefore, the Ge (100) and (111) planes are more stable and the diffusion of Ge atoms is less severe at these two planes as compared to those of the Ge (110) plane during annealing. As a result, the capital letter “I”-like structure is formed as shown in Fig. 5(d). It can also be anticipated that with the further increase in the surface-to-volume ratio, the thermal stability of $\text{Ge}_{1-x}\text{Sn}_x$ fins would be poorer due to a larger percentage of atoms being surface atoms with high diffusivity and the increased area ratio of the (110) surface with large surface energy.³⁵

In conclusion, the thermal stability of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin using RTA in N_2 ambient has been studied for temperatures up to 500 °C. The $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin sample shows poorer thermal stability than the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ blanket sample. Degradation of crystalline quality of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin and obvious Ge diffusion can be observed after 500 °C anneal. The relative poorer thermal stability of the $\text{Ge}_{0.96}\text{Sn}_{0.04}$ fin could be related to the larger surface to volume ratio of the fin structure. The extra exposed surfaces could further enhance the Sn segregation and desorption and Ge diffusion. In this context, a fabrication process with a lower thermal budget should be considered for future $\text{Ge}_{1-x}\text{Sn}_x$ multi-gate MOSFETs than that of planar $\text{Ge}_{1-x}\text{Sn}_x$ MOSFETs to avoid degradation in the $\text{Ge}_{1-x}\text{Sn}_x$ channel to achieve good electrical performance. The thermal stability of $\text{Ge}_{1-x}\text{Sn}_x$ fins investigated in this work could also provide a guideline on the selection of an appropriate thermal budget for fabricating $\text{Ge}_{1-x}\text{Sn}_x$ related 3D nanostructures.

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