

Low-Temperature Annealing of Polycrystalline $\text{Si}_{1-x}\text{Ge}_x$ After Dopant Implantation

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Abstract—Hall effect measurement was employed to study the isothermal annealing of boron or phosphorus implanted polycrystalline $\text{Si}_{1-x}\text{Ge}_x$ thin films, with x varying from 0.3–0.55. X-ray diffraction and cross-sectional transmission electron microscopy were used to study the crystal structure, whereas X-ray photoelectron spectroscopy was used to determine the film composition and the chemical bonding states of the elements. In low-temperature ($\leq 600^\circ\text{C}$) annealing, the conductivity, the dopant activation, and the Hall effect mobility decreased during extended annealing. The effective activation of phosphorus was less than 20% and decreased with increasing Ge content. Boron activation could reach above 70%. It was also found that $\text{Si}_{1-x}\text{Ge}_x$ could be oxidized at 600°C in a conventional furnace even with N_2 protection, especially for phosphorus doped films with high Ge content. Consequently, a low-temperature SiO_2 capping layer is necessary during extended annealing.

I. INTRODUCTION

POLYCRYSTALLINE $\text{Si}_{1-x}\text{Ge}_x$ (poly-SiGe) is an attractive alternative to polycrystalline silicon (poly-Si) in silicon integrated circuits [1]. The advantages of poly-SiGe over poly-Si include low process temperature and high mobility. For undoped $\text{Si}_{1-x}\text{Ge}_x$, crystallization can occur at a much lower temperature or for a shorter annealing time than for silicon [2]. Some authors [3], [4] also pointed out that the boron activation in poly-SiGe was much easier than in poly-Si. The activation behavior of doped poly-SiGe as a function of the annealing conditions has been studied [4]. However, the explanations for some phenomena are still far from clear, such as why the resistivity of the phosphorus doped poly-SiGe increases rapidly with increasing x when $x > 0.5$ [4], and how and why the Hall effect mobility and the carrier concentration depend on the Ge content. Furthermore, it is still not clear what temperature-time cycles are necessary to activate dopants in poly-SiGe, particularly for low-temperature thin-film transistors.

In this paper, we present the results of a study on the low-temperature annealing of implanted poly-SiGe, with x varying from 0.3–0.55. Hall effect measurements were used

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TABLE I
SiGe DEPOSITION CONDITIONS

Sample No.	Deposition temperature ($^\circ\text{C}$)	GeH_4 flow rate (sccm)	SiH_4 flow rate (sccm)	Deposition time (min)	SiGe thickness (nm)	Si/Ge composition
49	450	22	70	45	250	0.45/0.55
50	500	10	70	22	200	0.7/0.3
51*	500	10	70	35	300	0.7/0.3
52*	500	10	70	22	200	0.7/0.3
53	450	22	70	45	250	0.45/0.55
54	450	10	70	45	210	0.55/0.45

* All the samples, except No.51 and 52, were crystallized for 7 hours at 550°C before ion implantation.

to measure the electrical characteristics of doped poly-SiGe. X-ray diffraction (XRD) and cross-sectional transmission electron microscopy (XTEM) were used to characterize the crystal structure. X-ray photoelectron spectroscopy (XPS) was used to measure the film composition and the chemical bonding states of the elements.

II. EXPERIMENTAL

Poly-SiGe films were deposited at low pressure (0.3 Torr) by chemical vapor deposition at 450 or 500°C on thermally oxidized (100), p-type, 100-mm Si wafers. The source gases were SiH_4 and GeH_4 . The deposition technique has been studied in detail by others [5]. Because of the poor nucleation of SiGe on oxide, SiH_4 was turned on 30 min earlier than GeH_4 . Other deposition conditions are shown in Table I. The deposition time listed does not include the 30-min SiH_4 pretreatment time. At 500°C , 10 nm of amorphous silicon (a-Si) was deposited during the pretreatment, but no measurable a-Si layer could be deposited at 450°C . However, we believe some silicon seeds could be formed on the oxide during the 30-min pretreatment at 450°C , which nevertheless induced SiGe deposition. The thickness of the poly-SiGe thin films was between 200–250 nm, as measured by a surface profiler. To make sure the SiGe thin films were crystallized completely, Samples 49, 50, 53, and 54 were annealed at 550°C for 7 h in N_2 immediately following the SiGe deposition. Sample 52 was not crystallized in order to find out if there was any difference

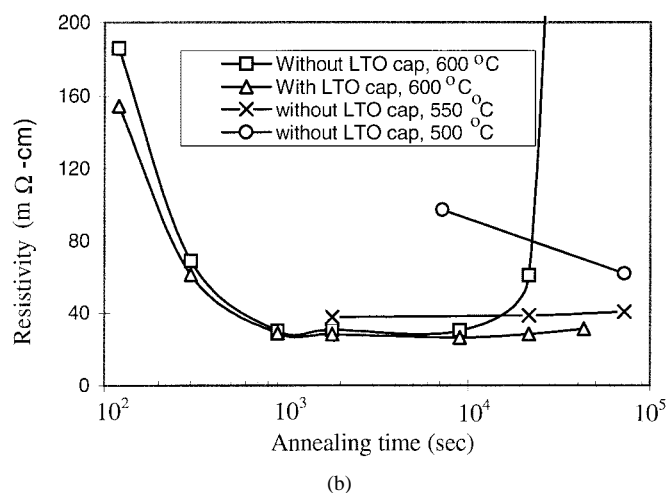
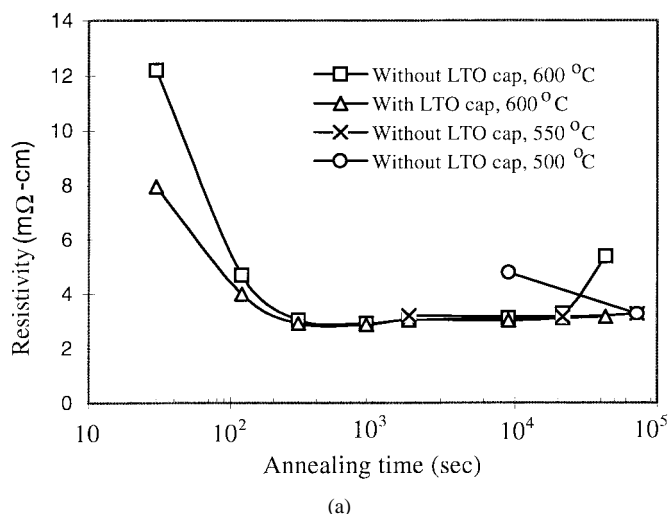


Fig. 1. Resistivity of (a) the p-type and (b) the n-type $\text{Si}_{0.45}\text{Ge}_{0.55}$ as a function of annealing time.

between the dopant activation behavior of the crystallized and the as-deposited SiGe .

Phosphorus or boron was implanted two consecutive times to obtain a smooth concentration profile. For the p-type samples, boron was implanted at 35 keV and a dose of $2 \times 10^{15} \text{ cm}^{-2}$, followed by BF_2^+ at 35 keV and a dose of $2 \times 10^{15} \text{ cm}^{-2}$. For the n-type samples, phosphorus was implanted at 80 keV and a dose of $2 \times 10^{15} \text{ cm}^{-2}$, followed by the same ion at 40 keV and a dose of $2 \times 10^{15} \text{ cm}^{-2}$. All the implanted wafers were cut into $1 \times 1 \text{ cm}^2$ square pieces. Some of them were capped with 100-nm low-temperature oxide (LTO) deposited at 425°C . The samples were annealed at different temperatures from $500\text{--}600^\circ \text{C}$. Annealing time was from 30 s to 20 h. The 30-s to 5-min anneals were done in a rapid thermal annealing (RTA) reactor, whereas the 15-min to 20-h anneals were done in a conventional furnace. All the anneals were done in an N_2 ambient evaporated from a liquid nitrogen source. The purity of the liquid nitrogen is above 99.99%. However, since the tube is not completely sealed at the loading end, oxygen, together with the laboratory air, can diffuse into the tube to change the purity of nitrogen in the tube.

After annealing, all the samples were measured in a Hall effect measurement system to estimate the sheet resistance,

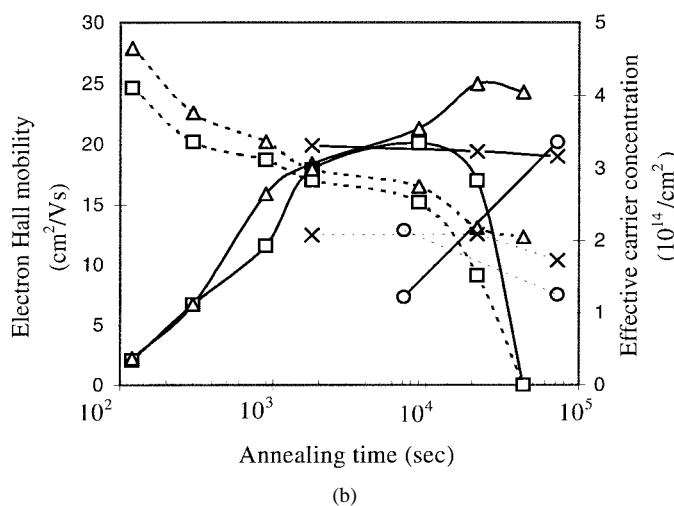
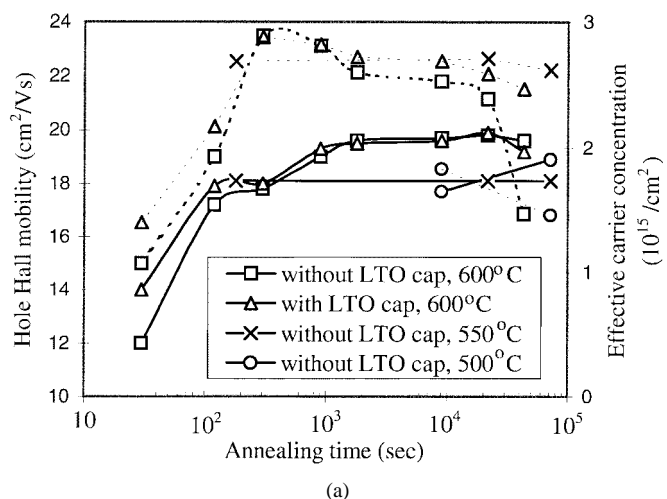


Fig. 2. Hall effect mobility and the effective dopant activation of (a) the p-type and (b) the n-type type $\text{Si}_{0.45}\text{Ge}_{0.55}$ as functions of annealing time. In both figures, the solid lines are for Hall effect mobility and the dotted lines are for effective dopant activation.

the Hall effect mobility, and the carrier concentration. Some of the samples were analyzed by XRD and XPS. XTEM analysis was done for microstructural analysis.

III. RESULTS

The resistivity of the p-type and the n-type $\text{Si}_{0.45}\text{Ge}_{0.55}$ films as a function of the annealing time at different temperatures is summarized in Fig. 1. The behavior is very similar between the n- and the p-type samples, although the resistivity of the n-type samples is always higher than that of the p-type samples. For the samples annealed at 600°C , the resistivity first decreased quickly with annealing time and then increased gradually on further annealing. For the boron-doped samples, resistivity as low as $3 \text{ m}\Omega\text{-cm}$ could be obtained after 5 min of annealing at 600°C . For the phosphorus-doped samples, the lowest resistivity of about $30 \text{ m}\Omega\text{-cm}$ was obtained after 15 min of annealing at 600°C . The behavior of the samples with and without the LTO caps was similar for annealing time less than 6 h at 600°C . Beyond 6 h, the resistivity of the samples without the LTO caps increased more quickly than that of the samples with the LTO caps, especially for the n-type samples.

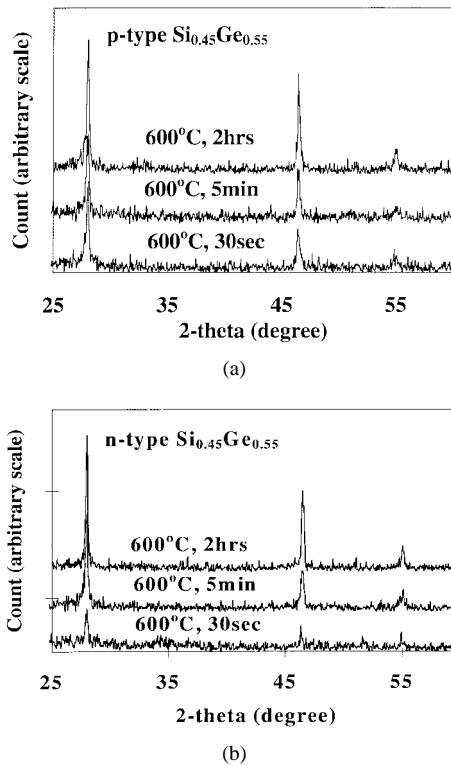


Fig. 3. XRD results of (a) the p-type and (b) the n-type type $\text{Si}_{0.45}\text{Ge}_{0.55}$ after annealed for 30 s, 5 min, and 2 h at 600°C .

After 12 h of annealing at 600°C , the resistivity of the n-type samples without the LTO caps became too large to be measurable in the Hall effect measurement system.

The Hall effect mobility and the effective dopant activation of the p-type and the n-type $\text{Si}_{0.45}\text{Ge}_{0.55}$ films as functions of annealing time at different temperatures are summarized in Fig. 2. Comparing Figs. 1 and 2, we can see that for the p-type samples, the decrease of the resistivity with annealing time during the first 5 min of annealing was due to both increases in the mobility and in the effective dopant activation. However, for the n-type samples, the decrease in the resistivity was due mainly to the mobility increase during the first 15 min of annealing. Effective phosphorus activation decreased monotonically with the annealing time in all the experiments. After 5 min of annealing, both the conductivity and the effective dopant activation of the p-type samples decreased slowly with the annealing time. The reason for the decrease in effective dopant activation with annealing time was possibly grain boundary segregation [6], formation of dopant pairs [7], or both. The behavior was similar for the samples with or without the LTO caps when the annealing time was less than 6 h. However, the effective dopant activation of the samples without the LTO caps decreased rapidly with annealing time beyond 6 h. This phenomenon will be further elaborated on later in the paper.

The sheet resistance of the n-type $\text{Si}_{0.45}\text{Ge}_{0.55}$ after 30 s of annealing at 600°C was still out of the range of the Hall effect measurement system, whereas the p-type samples had sheet resistance values of lower than $500\ \Omega/\text{square}$. This was because the implant damage by phosphorus at 80 keV was more severe

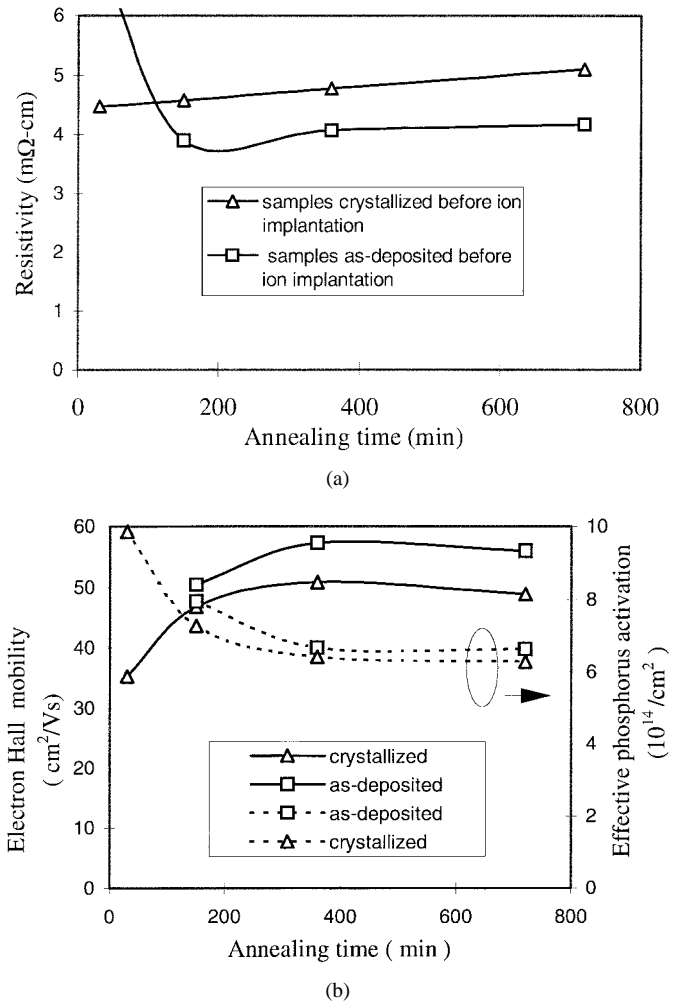


Fig. 4. Influence of crystallization before the phosphorus implantation on the resistivity, the Hall effect mobility, and the phosphorus activation as functions of annealing time after ion implantation. The Ge content is about 30%. All the samples were capped with 100 nm LTO after the ion implantation.

than that of boron at 35 keV. From the XRD results shown in Fig. 3, we can see that although significant crystallization has occurred in the p-type $\text{Si}_{0.45}\text{Ge}_{0.55}$ samples, the crystallization was far from complete for the n-type $\text{Si}_{0.45}\text{Ge}_{0.55}$ samples after 30 s of annealing. For the n-type samples, the time needed for significant crystallization was more than 5 min. After 15 min of annealing at 600°C , the structures became stable for both the n- and the p-type samples, as indicated by the XRD results.

In Fig. 4, the behavior of the crystallized and the as-deposited samples before the ion implantation was compared. The Ge content in the film was about 30%. After 15 min of annealing at 600°C , the sheet resistance of both kinds of samples was still larger than $1\ \text{M}\Omega/\text{square}$, which is the maximum measurable resistance of the equipment. After 30 min of annealing, the resistivity of the samples crystallized before the ion implantation was lower than $5\ \text{m}\Omega\text{-cm}$, whereas that of the as-deposited sample before the ion implantation was still out of the measurable range. From the XRD results shown in Fig. 5, we can see that the latter was still amorphous after 30 min of annealing. However, after 150 min of annealing

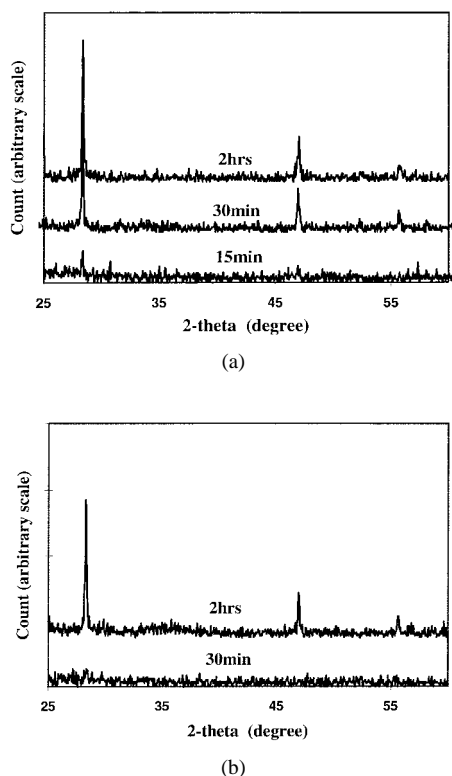


Fig. 5. XRD results of (a) the samples crystallized before the phosphorus implantation and (b) the samples as deposited before the phosphorus implantation. Each XRD trace is labeled by the corresponding implant activation time.

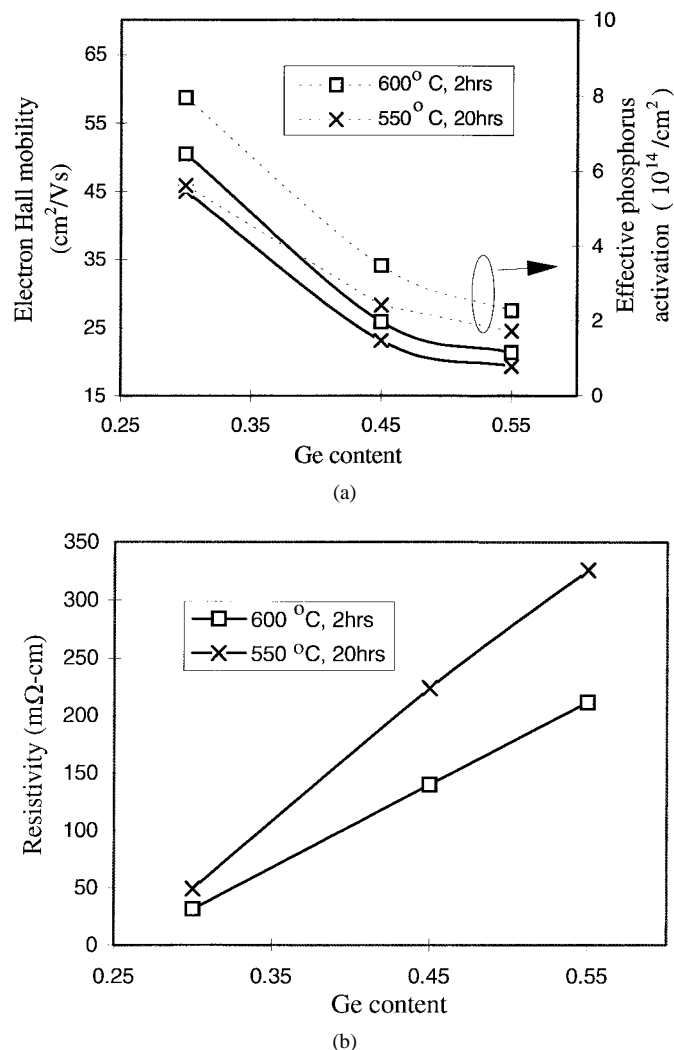


Fig. 7. Resistivity, the Hall effect mobility, and the effective dopant activation of n-type poly-SiGe as functions of the germanium content at different annealing temperatures after ion implantation. In (a), the dotted lines are for the phosphorus activation, and the solid lines are for the Hall effect mobility.



Fig. 6. XTEM micrograph of as-deposited $\text{Si}_{0.7}\text{Ge}_{0.3}$ film at 500°C on oxidized silicon. The oxide layer is about 100 nm thick. The SiGe film is about 300 nm thick. We can see the 10-nm a-Si deposited during the 30 min of SiH_4 pretreatment.

at 600°C , the conductivity, the Hall effect mobility, and the effective phosphorus activation of the as-deposited samples were higher than those of the crystallized samples.

From the XTEM micrograph (Fig. 6) of the as-deposited sample, we can see that the as-deposited $\text{Si}_{0.7}\text{Ge}_{0.3}$ was amorphous at the bottom and polycrystalline at the top. After the 80-keV phosphorus implantation, the top 100-nm layer should be amorphized completely by the phosphorus ions. Hence, during the subsequent annealing after implantation, no crystalline seeds existed. Therefore, it would take a longer time to crystallize. For the crystallized samples, no crystalline peak can be detected by XRD after the 80-keV phosphorus implantation. This means the 200-nm films were significantly damaged by the implant, although the calculated implant range should be less than 100 nm. We suspect this could be due to channeling effects in polycrystalline material. It has been noticed that the implant range in crystalline Ge was much larger than that predicted by theory [8]. However, it is still reasonable to consider that some seeds were available near the bottom of the film since the damage at the bottom should not

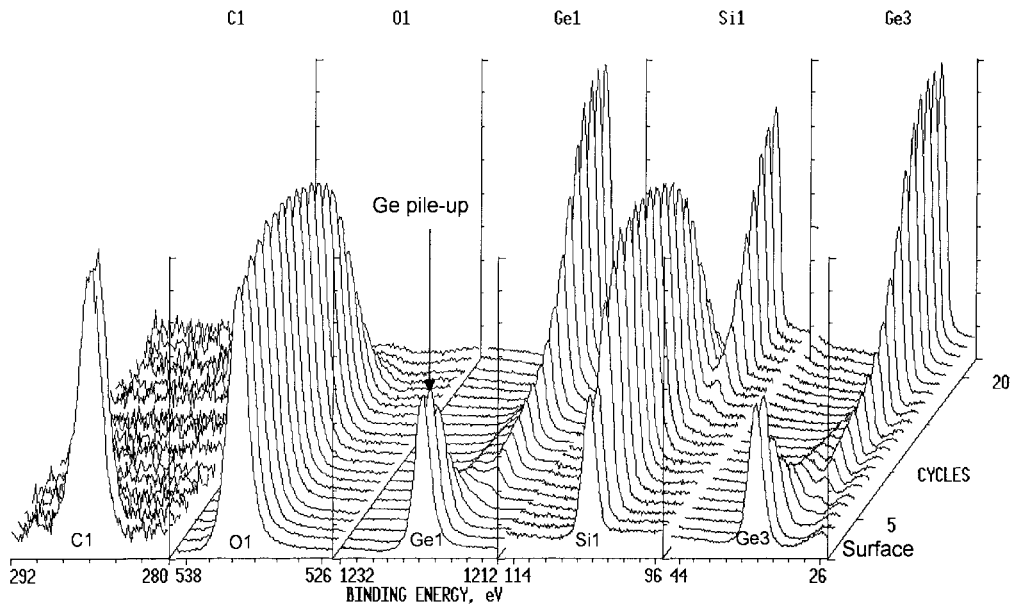


Fig. 8. XPS spectrum of the phosphorus doped $\text{Si}_{0.45}\text{Ge}_{0.55}$ without the LTO cap annealed for 6 h at 600°C in a conventional furnace with the protection of N_2 . Ar ion was employed to sputter the SiGe film at a rate of about 2 nm/min for the XPS profile. Each cycle, excluding the bottom two in the figure, represents XPS result following 1 min of sputtering. Both of the bottom two cycles represent the XPS results of the film surface.

TABLE II
ANNEALING TIME NECESSARY IN DIFFERENT TEMPERATURE TO OBTAIN SHEET RESISTANCE LOWER THAN $5\text{ k}\Omega$. THE FINAL SHEET RESISTANCE OBTAINED AFTER THE ANNEALING SCHEDULE IS ALSO SHOWN

	500°C	550°C	600°C
N-type	> 20 hours	20 hours	30 minutes
$\text{Si}_{0.7}\text{Ge}_{0.3}$	> $1\text{M}\Omega$	246Ω	179Ω
N-type	>20 hours	30 minutes	15 minutes
$\text{Si}_{0.55}\text{Ge}_{0.45}$	> $1\text{M}\Omega$	$1.8\text{K}\Omega$	559Ω
N-type	2 hours	30 minutes	5 minutes
$\text{Si}_{0.45}\text{Ge}_{0.55}$	$3.9\text{K}\Omega$	$1.5\text{K}\Omega$	$2.45\text{K}\Omega$
P-type	2 hours	30 minutes	30 seconds
$\text{Si}_{0.45}\text{Ge}_{0.55}$	128Ω	128Ω	318Ω

have been as severe as that at the surface. These seeds could shorten the necessary annealing time to recrystallize the film, but the grain size would be smaller than in the film without the seeds. We believe this is why the final conductivity and the Hall effect mobility of the as-deposited samples were higher.

In Fig. 7, the different annealing behavior of the n-type samples with different Ge contents are compared. It can be seen that the conductivity, the Hall effect mobility, and the effective dopant activation of the low Ge content samples were higher than those of the high Ge content samples when the Ge content is in the range from 0.3 to 0.55. This is similar to the results obtained by King *et al.* [4]. In their results, however, the resistivity increased much more rapidly with the Ge content than that which we observed when the Ge content was above 50%. From our experiments, we also noticed that the necessary

annealing time and temperature (Table II) were much less for the high Ge content samples.

IV. DISCUSSION

For the low Ge content SiGe films, Noguchi *et al.* [9] have shown that the resistivity was similar among samples with or without the LTO caps during the annealing. In our experiments, the resistivity of the high Ge content samples changed significantly between those with the LTO caps and those without the LTO caps, especially for the phosphorus doped samples. From Fig. 2, we can see that the carrier concentration and the conductivity decreased with long annealing time at 600°C for samples without the LTO caps. After 12 h of annealing, the resistivity of the n-type samples without the LTO caps was out of the measurement range of the equipment. It is suspected that oxidation played an important role here. In a conventional furnace, it is difficult to eliminate trace amounts of oxygen, even in a "pure" nitrogen ambient. SiGe can be oxidized at low temperature [10]. This oxidation could be enhanced by the implanted phosphorus [11]. In addition, out-diffusion of phosphorus would be serious without the LTO caps [6], especially during a lengthy annealing.

An XPS profile (Fig. 8) of $\text{Si}_{0.45}\text{Ge}_{0.55}$ without the LTO cap after 6 h of annealing in a conventional furnace at 600°C with nitrogen protection clearly showed that the top 30 nm was completely oxidized. It can also be seen that Ge piled up at the surface, similar to the thermal oxidation of SiGe [12]. Further evidence for the 30-nm oxide layer is shown in the XTEM micrograph in Fig. 9. Such thick oxide may indicate that the phosphorus doping could have significantly accelerated the oxidation rate of SiGe.

For the LTO capped n-type samples, the resistivity still increased with the Ge content. This was because of the low effective activation of phosphorus and the low electron

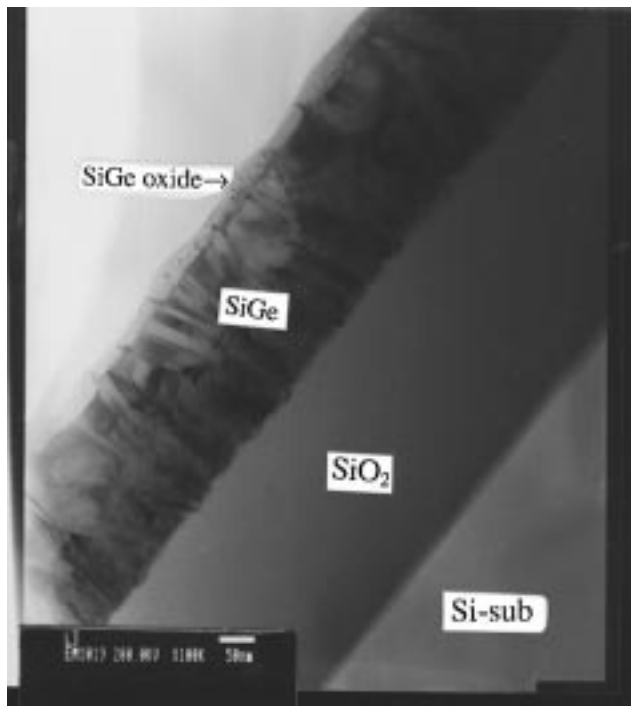


Fig. 9. XTEM micrograph of the phosphorus doped $\text{Si}_{0.45}\text{Ge}_{0.55}$ without the LTO cap annealed for 6 h at 600°C in a conventional furnace.

mobility in the samples with high Ge content, as shown in Fig. 7. Herzer and Kalbitzer [8] showed that phosphorus activation in crystalline Ge is as low as 20%, which is much lower than in crystalline Si. The mobility in polycrystalline material was also dependent on the carrier concentration [13]. Therefore, the low activation of phosphorus in high Ge content poly-SiGe was also a reason for the low mobility. The reason for the low effective activation of phosphorus may be the electronic states in the grain boundaries. It has been shown [14] that in poly-Ge grain boundaries, a partially filled band of electronic states exists near the valance band. This band would decrease the effective electron concentration and the electron mobility. The characteristics of the high Ge content poly-SiGe should be similar to those of poly-Ge. In addition, the Ge may enhance the grain boundary segregation of phosphorus. The unactivated phosphorus may exist as phosphorus clusters, especially at the grain boundaries. This would change the material structure and enhance the electron scattering. Hence, the electron mobility would decrease with increasing Ge content.

The boron activation and the hole mobility of high Ge content poly-SiGe were not bad compared with poly-Si. In our experiments, 70% effective activation was obtained. Boron activation also decreased slowly with extended annealing. This may have been due to boron pair formation [7] or grain boundary segregation. The hole mobility of high Ge content poly-SiGe can reach a value $20\text{ cm}^2/\text{Vs}$. This could be due to the high boron activation and the smaller density of hole traps in the grain boundaries, contrary to the n-type [14] samples.

It should be pointed out that the necessary annealing time for $\text{Si}_{0.7}\text{Ge}_{0.3}$ in our experiment is much longer than that used by others [3]. The film thickness here was only 200 nm

and phosphorus implantation energy was 80 keV, whereas in [3], the film thickness was 350–400 nm and the implantation energy was 60 keV. It is suspected that the films in [3] were not completely damaged by the implantation, and therefore, seed layers existed at the bottom of the SiGe layer. This would obviously shorten the recrystallization time.

V. CONCLUSION

This paper showed that the conductivity, dopant activation, and the Hall effect mobility decreased during extended annealing for the SiGe thin film when the Ge content was in the range of 0.3–0.55. The plausible reason for those decreases may be the grain boundary segregation of dopant during extend annealing. Phosphorus activation and electron mobility decreased with increasing Ge content. One of the possible reasons is the change in the grain boundary trap density with Ge content. Poly-SiGe films, especially those with high Ge content, can be oxidized at low temperature in a conventional furnace, even with “pure” nitrogen protection. This oxidation can be enhanced by heavy phosphorus doping. XPS results showed that Ge piled up at the surface when SiGe was oxidized.

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