#### Conclusion

The effect of the anodization regime on the anisotropy of the porous anodization process of aluminum was investigated. The experiments revealed that the degree of anisotropy increased with the forming voltage in all the studied electrolytes. The highest degree of anisotropy,  $A_f = 0.6$ , was obtained in 0.01M oxalic acid solution in water.

SEM investigation of the porous aluminum oxide structure in contact with the mask and outside it was performed. Using the data obtained by SEM and the experimental parameters of the electrochemical process, the electric field distribution within the barrier layer of the porous oxide was calculated. The electric field concentration in the cells near the mask was shown.

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# Effects of SiH<sub>4</sub>, GeH<sub>4</sub>, and B<sub>2</sub>H<sub>6</sub> on the Nucleation and Deposition of Polycrystalline Si<sub>1-x</sub>Ge<sub>x</sub> Films

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#### ABSTRACT

In this study, the effects of different reactants, namely,  $SiH_4$ ,  $GeH_4$  and  $B_2H_6$ , on the nucleation and deposition of polycrystalline silicon-germanium (poly- $Si_{1-x}Ge_x$ ) films on oxide surface in an ultrahigh vacuum chemical vapor deposition reactor were explored. The results show that the addition of  $GeH_4$  tends to retard the nucleation process of poly films  $GeH_4$  and  $GeH_4$  tends to retard the nucleation process of poly films. while B₂H₀ is preferential for adsorbing on the oxide surface. These effects lead to different incubation duration depending on the kind of reactants used. On the deposition of  $poly-Si_{1-x}Ge_x$  films, it is observed that the Ge incorporation is only slightly related to the substrate type, but the deposition mode of  $poly-Si_{1-x}Ge_x$  films is much different from that of epitaxial growth on Si(100). The incorporation of Ge atoms also overcomes the anomalous doping effect encountered in heavily boron-doped poly-Si films and allows extremely low resistivity (below  $2 \text{ m}\Omega$ -cm) poly films to be obtained at low temperatures ( $\leq 550^{\circ}$ C).

Polycrystalline silicon (poly-Si) is one of the most important materials for integrated circuit manufacturing. 1 Usually, poly-Si films are obtained using the low pressure chemical vapor deposition (LPCVD) technique because of its high uniformity, reproducibility, and throughput. Conventional LPCVD techniques use silane (SiH4) as the deposition source, and deposition pressures of typically several hundred milliTorrs. Under these conditions, as-deposited poly-Si films with stable fine grains are obtainable at a temperature higher than  $620^{\circ}\text{C.}^{1}$  Recently, a few studies concerning the deposition<sup>2-4</sup> and device applications<sup>4-9</sup> of polycrystalline silicon-germanium (poly- $Si_{1-x}Ge_x$ ) films were reported. The main advantages associated with the poly- $Si_{1-x}Ge_x$  film deposition are the low temperature and low process thermal budget, as compared with the poly-Si ones. In addition, poly- $Si_{1-x}Ge_x$  films are compatible with mature Si technologies. All these make poly-Si<sub>1-x</sub>Ge<sub>x</sub> an attractive alternative candidate to Si for some applications demanding minimal process thermal effect, such as the fabrication of polycrystalline thin film transistors (TFTs) on glasses used for active matrix liquid crystal display (AMLCD) manufacturing, and the ultralarge scale integration (ULSI).

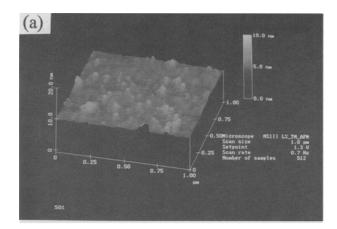
Among the published reports, poly- $Si_{1-x}Ge_x$  films were prepared mostly using the conventional LPCVD, 2,5-9 or the rapid thermal (RT) LPCVD<sup>3</sup> systems. In our earlier work, we have shown that the ultrahigh vacuum (UHV) CVD is

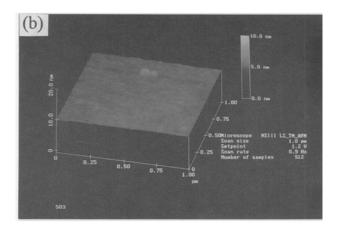
another promising approach.4 This technique features a hot-wall reactor with an extremely low base pressure  $(\sim 10^{-9} \, \text{Torr})$  as well as the reduced deposition pressures (~1 mTorr). These conditions allow extremely low temperature (below 550°C) Si/Si<sub>1-x</sub>Ge<sub>x</sub> epitaxial growth to proceed on Si substrates.  $^{10,11}$  Si<sub>1-x</sub>Ge<sub>x</sub>-based heterojunction bipolar transistors (HBTs) with a world-record cutoff frequency ( $f_T$ ) of 113 GHz were achieved with the UHV-CVD system,12 demonstrating the superior quality of the deposited films. When depositing poly films on an insulating substrate, e.g., SiO2, we have found that the fine grain structure can be retained even at a temperature as low as  $500^{\circ}\text{C.}^{4,13}$  We have also shown that the preparation of poly- $Si/poly-Si_{1-x}Ge_x$  multilayers using UHV-CVD can increase the design flexibility of poly thin film device structures.4

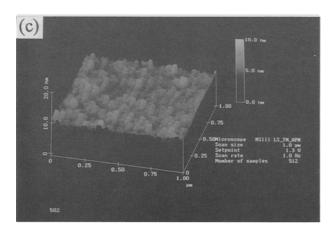
In this report, we investigated the effects of reactants on the film nucleation and deposition. The influence of Ge incorporation on the resistivity of in situ boron-doped films is also explored and discussed.

#### Experimental

A detailed description about our UHV-CVD system can be found elsewhere. 4,11 In this study, 3 in. Si wafers coated with a thermally grown oxide were used as the starting materials. Prior to deposition, the wafers were cleaned in a mixture of H<sub>2</sub>SO<sub>4</sub>/H<sub>2</sub>O<sub>2</sub>(3:1) for 15 min, followed by a 5 min deionized water rinse. Substrates were then blown dry







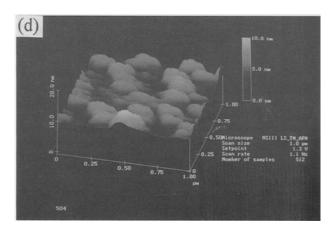


Fig. 1. The states of oxide surface investigated by AFM after exposure to input reactants for 20 min. The deposition temperature was  $550^{\circ}$ C. (a)  $SiH_4 = 20$  sccm; (b)  $SiH_4 = 20$  sccm,  $GeH_4 = 15.3$  sccm; (c)  $SiH_4 = B_2H_6 = 20$  sccm; (d)  $SiH_4 = B_2H_6 = 20$  sccm,  $GeH_4 = 15.3$  sccm.

with nitrogen gas. Before introducing the reactants, the substrates were heated in the growth chamber for 1 h at a corresponding deposition temperature. During this stage, an  $\rm H_2$  flow of 600 sccm was introduced which resulted in a pressure of 42 mTorr. Pure  $\rm SiH_4$ , 10% germane ( $\rm GeH_4$ ) in  $\rm H_2$ , and 1% diborane ( $\rm B_2H_6$ ) in  $\rm H_2$  were used as the reaction sources for deposition. The deposition pressures used in this work are in the range between 0.94 and 1.95 mTorr.

The Ge composition and the boron concentration in the as-deposited films were determined by Rutherford back-scattering spectroscopy (RBS) and secondary ion mass spectroscopy (SIMS), respectively. Electrical resistivity of the boron-doped films were measured by a four-point probe while film thicknesses were obtained by a Dektak 3030 surface profile measuring system with an accuracy within 20 nm. Transmission electron spectroscopy (TEM) and x-ray diffraction (XRD) were performed to examine the structural properties of the poly-Si<sub>1-x</sub>Ge<sub>x</sub> films. Atomic force microscopy (AFM) was used to investigate the surface state of the deposited films.

### Results and Discussion

Nucleation.—For a poly film deposited on an  ${\rm SiO_2}$  surface at reduced pressures, a considerable amount of incubation time is observed. <sup>13-16</sup> This is due to the heterogeneous reaction which dominates at a very low deposition pressure and is very sensitive to the surface properties of the substrate used. The magnitude of incubation time is strongly related to the kind of reaction species, such as  ${\rm SiH_4}$ ,  ${\rm GeH_4}$ , and  ${\rm B_2H_6}$ , used for this study. In order to understand the effects of these reactants on the nucleation, the AFM technique was used to investigate the surface state at an early stage of deposition. The results are given in Fig. 1 in which four conditions with different input reactants are shown and compared. The deposition temperature, duration, and the flow rate of  ${\rm SiH_4}$  were  ${\rm 550^{\circ}C}$ , 20 min, and 20 sccm,

respectively, for all the samples shown in Fig. 1, while the poly-Si<sub>1-x</sub>Ge<sub>x</sub> films of Fig. 1b and d were with a GeH<sub>4</sub> (10% in H<sub>2</sub>) flow rate of 15.3 sccm, resulting in a Ge content of 21% in the deposited films. Figure 1a is the result of undoped poly-Si which exhibits scattered nuclei on the oxide surface. In the case of undoped poly- $Si_{1-x}Ge_x$ , as shown in Fig. 1b, it is found that the number of nuclei is significantly reduced, indicating that the addition of GeH<sub>4</sub> would retard the nucleation. On the other hand, the introduction of a large amount of B<sub>2</sub>H<sub>6</sub> flow would significantly change the situation. Figure 1c and d are examples of boron-doped poly-Si and poly-Si<sub>1-x</sub>Ge<sub>x</sub> films, respectively, both with a B<sub>2</sub>H<sub>6</sub> (1% in H<sub>2</sub>) flow rate of 20 sccm. It is clearly seen in Fig. 1c and d that the oxide surface has been completely covered by the films. This indicates that the boron atoms are preferentially adsorbed on the SiO2 surface and then act as a seed for film growth. Consistent results are shown in Fig. 2, in which the film thicknesses were shown as functions of the deposition time. The deposition conditions for films shown in Fig. 2 correspond to those used in Fig. 1. In this figure, the slope of the fit line and its intercept with the x-axis are defined as the deposition rate and the incubation time, respectively, for each specific condition. It is observed that the incubation time is about 36 min for undoped poly-Si, while extending to around 51 min for undoped poly- $Si_{1-x}Ge_x$ , consistent with fewer nuclei as shown in Fig. 1b. When adding the B<sub>2</sub>H<sub>6</sub>, though, the incubation times are drastically reduced to 3 and 2 min for B-doped poly-Si and poly- $Si_{1-x}Ge_x$  films, respectively, and are also well explained by the results of Fig. 1c and d.

From the above analysis it is noteworthy that the extremely different behavior of nucleation on the oxide surface between GeH<sub>4</sub> and B<sub>2</sub>H<sub>6</sub>: the former tend to retard the process while the latter is preferential to adsorb on. A more detailed investigation concerning the effect of B<sub>2</sub>H<sub>6</sub> flow on the incubation time is given elsewhere. <sup>17</sup> For some specific

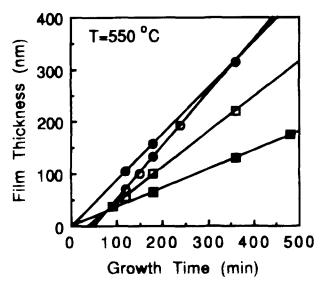


Fig. 2. Film thickness vs. growth time. ( $\square$ ) SiH<sub>4</sub> = 20 sccm; ( $\bigcirc$ ) SiH<sub>4</sub> = 20 sccm, GeH<sub>4</sub> = 15.3 sccm; ( $\blacksquare$ ) SiH<sub>4</sub> = B<sub>2</sub>H<sub>6</sub> = 20 sccm; ( $\blacksquare$ ) SiH<sub>4</sub> = B<sub>2</sub>H<sub>6</sub> = 20 sccm, GeH<sub>4</sub> = 15.3 sccm.

applications, such as the p<sup>+</sup> poly-Si<sub>1-x</sub>Ge<sub>x</sub> gate structures, <sup>5,6,9</sup> it is advantageous to use the *in situ* doping method which can avoid the long incubation time encountered at undoped poly-Si<sub>1-x</sub>Ge<sub>x</sub> deposition.

Another interesting phenomenon noted in Fig. 1 is the larger grain size observed for boron-doped poly- $\mathrm{Si}_{1-x}\mathrm{Ge}_x$  than that of boron-doped poly- $\mathrm{Si}$ . This is due to the higher surface mobility of Ge atoms which facilitate the grain growth and promote the film crystallinity. The influence of this aspect on the film resistivity is discussed in the last part of this section.

Effect of  $GeH_4$  on the incubation time.—As mentioned above, the addition of  $GeH_4$  delays the nucleation process markably. In Fig. 3, the incubation time of poly- $Si_{1-x}Ge_x$  films grown at  $550^{\circ}C$  with the UHV-CVD system are shown as a function of the  $GeH_4$  flow rate. In an earlier work, <sup>13</sup> we showed that the considerable amount of incubation time (36 min) for poly-Si films deposited at  $550^{\circ}C$  is due to the rather low deposition rate ( $\sim 0.6$  nm/min) as well as the low generation rate of nuclei. In this study, we found that the addition of  $GeH_4$  increases the incubation period further.

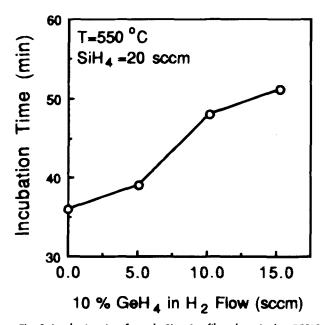


Fig. 3. Incubation time for poly-Si $_{1-x}$ Ge $_x$  films deposited at 550°C as a function of GeH $_4$  flow.

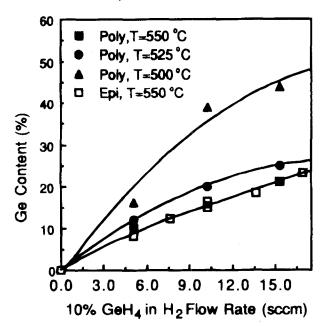


Fig. 4. Ge mole fractions incorporated in the  $Si_{1-x}Ge_x$  epitaxial layers deposited at ( $\square$ ) 550°C as well as the poly- $Si_{1-x}Ge_x$  ones deposited at three different temperatures, namely, ( $\blacksquare$ ) 550, ( $\bigoplus$ ) 525, and ( $\triangle$ ) 500°C, respectively, are shown as functions of GeH<sub>4</sub> flow.

The increase of the incubation time with increasing GeH<sub>4</sub> flow rate is in agreement with previous findings. 15,16 Accompanied by the smaller number of nuclei is the larger grain size for poly- $\mathrm{Si}_{1-x}\mathrm{Ge}_x$  films, as observed by the TEM analysis.4 According to the report of Morar et al.,18 they pointed out that the adsorbed Ge atoms on an Si substrate can break the silicon-oxygen bonds of native oxide on the surface and form germanium oxides (e.g., GeO 19) which are easy to volatilize. This finding can also well explain our observation that the introduction of GeH4 flow may effectively break the silicon-oxygen bonds between the Si nuclei and the underlying oxide layer and, thus, retard the nucleation process. We believe that this is responsible for the longer incubation time for a higher GeH<sub>4</sub> flow observed in Fig. 3. We have further examined that when only GeH<sub>4</sub> was introduced into the growth environment for 2 h at 550°C no nuclei were formed on the oxide surface. A similar result was also reported by Ozturk et al., 19 highlighting the highly selective deposition character for GeH<sub>4</sub> on SiO<sub>2</sub>.

Ge incorporation and deposition rate.—Figure 4 shows the Ge incorporation of poly- $Si_{1-x}Ge_x$  films as a function of GeH<sub>4</sub> flow rate. In order to prevent the long incubation period, a thin Si film of 10 nm was first deposited on the SiO<sub>2</sub> surface to act as a seed and then started the poly- $Si_{1-x}Ge_x$  film deposition. The data of  $Si_{1-x}Ge_x$  layers epitaxially grown on Si(100) substrates at 550°C in the same reactor are also included in the figure. In Fig. 4, the incorporation of Ge atoms at 550°C is the same for both poly and epitaxial films. For films deposited in a polycrystalline state, grains of random orientation are distributed. Therefore, identical Ge incorporation for poly and epitaxial layers seems to indicate that the incorporation is only slightly related to the crystallite orientation. On the other hand, the Ge incorporation is promoted at lower temperatures, as observed in Fig. 4. A similar phenomenon was also observed by King  $et\ al.^6$  with an LPCVD technique. The higher Ge incorporation also promotes the film crystallinity at low temperatures. An example is given in Fig. 5 in which a plane-view TEM photograph and the diffraction pattern of an as-deposited poly- $\mathrm{Si}_{0.56}^{-}\mathrm{Ge}_{0.44}$  film deposited at 500°C are shown. The average grain size is around 180 nm and is much larger than the undoped poly-Si deposited in the same reactor<sup>13</sup> ( $\sim$ 60 nm).

It has been pointed out previously in several studies that the deposition mode of the poly-Si is identical to that of Si

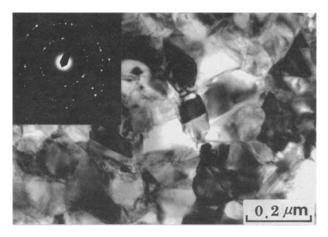


Fig. 5. The TEM micrograph and corresponding diffraction pattern of an as-deposited poly-Si<sub>1-x</sub>Ge<sub>x</sub> film grown at 500°C.

epitaxy on Si(100) in a UHV-CVD reactor. 13,20 We have also found with our system that the deposition rates are the same for both poly-Si and Si epitaxial layers. However, the situation is significantly changed as GeH4 is added. This phenomenon is shown in Fig. 6 where the variation of the deposition rate for poly-Si<sub>1-x</sub>Ge<sub>x</sub> films with GeH<sub>4</sub> flow is significantly different from that of epitaxial ones. In this figure, the deposition rate of epitaxial layers grown at 550°C is dramatically enhanced with increasing GeH<sub>4</sub> flow. This is similar to that observed by Meyerson et al., 21 and is attributed to the enhanced desorption of hydrogen atoms during deposition. However, for films deposited in a polycrystalline form, it is observed that the deposition rate is smaller than that of epitaxial growth as GeH4 is introduced, although with identical Ge incorporation. Moreover, the difference between the epitaxial and the poly films increases with increasing GeH4 flow.

The cause of this phenomenon is not very clear at this stage and is possibly due to the texture change with the

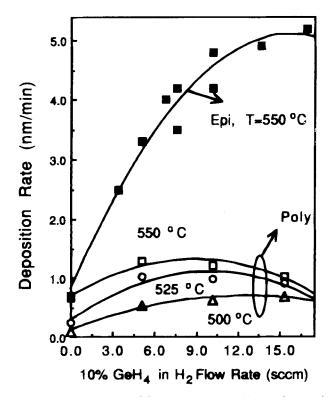


Fig. 6. Deposition rates of the  $\mathrm{Si}_{1-x}\mathrm{Ge}_x$  epitaxial layers deposited at  $|\blacksquare\rangle$  550°C as well as the poly- $\mathrm{Si}_{1-x}\mathrm{Ge}_x$  ones deposited at three different temperatures, namely,  $|\Box\rangle$  550,  $|\bigcirc\rangle$  525, and  $|\triangle\rangle$  500°C, respectively, are shown as functions of GeH<sub>4</sub> flow.

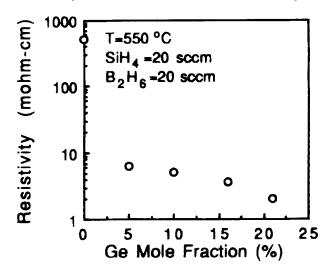


Fig. 7. The film resistivity as a function of Ge incorporation.

addition of  $\operatorname{GeH}_4$ . When an  $\operatorname{Si}_{1-x}\operatorname{Ge}_x$  layer grows epitaxially on an Si (100) substrate, the strain energy contained in the deposited films may play an important role in determining the deposition behavior. This situation, however, cannot be applied to the deposition of poly films on oxide surfaces. In a separate study<sup>22</sup> we have used the XRD method to analyze the three main textures, namely, (111), (220), and (311), of the poly– $\operatorname{Si}_{1-x}\operatorname{Ge}_x$  films. For the undoped poly– $\operatorname{Si}$  films, the main texture is (220). It was observed that, in the case of Ge incorporation, the intensity of (220) decreases while the other two textures significantly increase, especially for (311). These results indicate that the deposition mode is significantly different between the poly– $\operatorname{Si}_{1-x}\operatorname{Ge}_x$  and  $\operatorname{Si}_{1-x}\operatorname{Ge}_x$  epitaxy as  $\operatorname{GeH}_4$  is added.

Effect of Ge incorporation on the film resistivity.—In one of our earlier works we investigated the electrical properties of in situ boron-doped poly–Si films.  $^{23}$  The results show that, when the incorporated boron concentration was greater than  $10^{19}~\rm cm^{-3}$ , the grain size of poly–Si films began to diminish with increasing doping level. This phenomenon was attributed to the slow surface mobility of boron atoms during deposition. As the boron doping level reaches beyond  $10^{21}~\rm cm^{-3}$ , a totally amorphous structure resulted. This kind of degradation in film crystallinity caused a drastic rise in film resistivity. Due to this anomalous doping effect, the resistivity of the as-deposited poly–Si films was prevented from being lower than 3 m $\Omega$ -cm.  $^{23}$ 

However, this restriction can be overcome with the Ge incorporation.4 Figure 7 shows the effect of Ge incorporation on the as-deposited film resistivity. The films were deposited at 550°C with both SiH4 and B2H6 flows of 20 sccm. Film thickness was around 200 nm. The high resistivity of poly-Si shown in this figure is in response to the anomalous doping effect mentioned above, although with a high boron concentration of  $2.1 \times 10^{21}$  cm<sup>-3</sup> measured by SIMS. When Ge atoms were incorporated to a level of 5% mole fraction, a rapid decrease in film resistivity is observed. Resistivity is then further decreased with increasing Ge level and reaches a value of about 2 m $\Omega$ -cm at 21% Ge incorporation. From the Hall measurements we learned that the low resistivity obtained for poly- $Si_{1-x}Ge_x$  films is due to the high concentration of activated carriers exceeding the equilibrium solid solubility of dopants. For example, the carrier concentration in the poly-Si<sub>0.79</sub>Ge<sub>0.21</sub> films is around  $4.8 \times 10^{20}$  cm<sup>-3</sup>. Such a phenomenon is known as the nonequilibrium doping effect. <sup>24</sup>

For deposition proceeding at reduced pressures, Voultsas and Hatalis have pointed out that the grain growth is closely related to the surface mobility of the reactants. <sup>25</sup> In this experiment, based on the AFM and TEM results, we postulate that, as compared with Si, B possesses a much lower surface mobility while Ge possesses a much higher

one. For B-doped poly-Si the low surface mobility of B atoms results in the grain shrinkage as well as the anomalous rising behavior in film resistivity. The addition of Ge atoms which have a much higher surface mobility can compensate in this situation and allow the nonequilibrium doping effect retained to a higher doping level. This provides a good explanation about the drastic decrease in resistivity with Ge incorporation as shown in Fig. 7.

#### Conclusions

In this work we have shown that the nucleation of poly-Si<sub>1-x</sub>Ge<sub>x</sub> films on an SiO<sub>2</sub> surface is strongly related to the kind of reactants used. As compared with the undoped poly-Si deposition, the addition of GeH4 retards the nucleation process and increases the incubation time. On the other hand, the introduction of a large amount of B<sub>2</sub>H<sub>6</sub> for either poly-Si or poly-Si<sub>1-x</sub>Ge<sub>x</sub> deposition significantly shortens the incubation time and is attributed to the preferential adsorption of boron atoms on the SiO<sub>2</sub> surface.

On the deposition of poly- $Si_{1-x}Ge_x$  films, the Ge fraction incorporated into the films is found to be almost independent of the substrate used. However, the deposition mode of poly- $Si_{1-x}Ge_x$  films is found to be different from the  $Si_{1-x}Ge_x$  epitaxial process as  $GeH_4$  is added. Ge incorporation also compensates for the anomalous doping effect encountered in heavily B-doped poly-Si films and it allows extremely low resistivity poly films to be obtained at low temperatures (e.g., below 550°C).

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