IN-SITU DOPED POLY-SIGE LPCVD PROCESS USING BCL3 FOR POST-CMOS INTEGRATION OF MEMS DEVICES

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Abstract. The depositions of in-situ boron doped poly-SiGe films with BCl₃ as the dopant source in a conventional LPCVD reactor have been studied for MEMS applications. BCl₃ doped poly-SiGe films deposited at 400°C - 450 °C with 65% - 75% Ge content have similar electrical and mechanical properties as B₂H₆ doped films. To achieve the same level of doping, the required BCl₃ gas partial pressure is two orders of magnitude lower than that of B₂H₆. Poly-SiGe films with higher boron doping have larger average compressive stresses and BCl₃ doped films are more compressive than B₂H₆ doped films for the same doping level. Thicker poly-SiGe film is found to have larger grain size, lower resistivity, less compressive stress, and smaller strain gradient. The initial BCl₃ doped process shows similar or even better process uniformity and repeatability than a mature B₂H₆ doped process.

INTRODUCTION

Poly-SiGe is a promising material for the modular integration of MEMS and CMOS, due to its low process thermal budget and its good electrical and mechanical properties [1]. For MEMS applications, the poly-SiGe structural layer should have low resistivity, small tensile stress, and low strain gradient. Furthermore, a uniform and repeatable process is required for mass production of integrated systems.

Conventional LPCVD reactors can be used to deposit conformal poly-SiGe at temperature range of 400 °C – 450 °C. P-type in-situ doping is preferred as it enhances the deposition rate and enables low resistivity films. B₂H₆ has been previously used for in-situ doping of poly-SiGe MEMS structural films. However, serious problems of the B₂H₆ process make it difficult to control the electrical and mechanical properties of the deposited material. Recently, a high-throughput LPVCD process was developed using BCl₃ as the dopant source for epitaxial SiGe growth [2, 3]. Also, BCl₃ is being studied for in-situ doping of poly-Si [4].
In this work, we investigate LPCVD processes using BCl₃ as the dopant source for deposition of poly-SiGe MEMS structural films. The deposition rate, crystallinity, dopant incorporation, resistivity, residual stress, strain gradient, as well as effects of thickness on electrical and mechanical properties, and process uniformity are presented in this paper.

EXPERIMENTAL DETAILS

P-type *in-situ* doped poly-SiGe films were deposited in a Tystar hot-wall horizontal LPCVD reactor, approximately 50 inches in length and 9 inches in diameter (Fig. 1). This furnace is capable of both B₂H₆ and BCl₃ doping, and it operates in the temperature range of 350 - 450 °C, and within a pressure range of 100 - 2000 mTorr. Pure SiH₄, Si₂H₆ and GeH₄ were used as the gaseous Si and Ge precursors. 0.1% BCl₃ diluted in He was first used as the dopant gas, so that the BCl₃ partial pressure would be comparable to that in the previously reported epitaxial growth system [2, 3]. The resistivity of the films with the 0.1% BCl₃ concentration was found to be higher than desired, so the concentration of BCl₃ was changed to 1%. SiH₄ and GeH₄ were introduced at the gas ring located at the load side of the tube. The BCl₃/He mixture was introduced through the gas ring for these experiments, even though introducing BCl₃ via a multi-pore injector located at the bottom of the wafer boats is another option. Twenty-five 4”-diameter and twenty-five 6”-diameter wafers placed in open boats at the center of the reactor were used per load.

![LPCVD reactor schematic](image)

**Fig. 1** LPCVD reactor schematic
The process conditions of various depositions are summarized in Table I. Poly-SiGe films were deposited on top of Si wafers coated with ~2 µm thick low temperature (450 °C) LPCVD oxide. A ~5 nm thick undoped amorphous Si (a-Si) seeding layer was deposited first using 100 sccm of Si$_2$H$_6$ to promote adhesion of SiGe to SiO$_2$. The a-Si layer was deposited at 300 mTorr for 15 minutes at various temperatures. For the poly-SiGe deposition, the process pressure was held constant at 400 mTorr. Temperature, BCl$_3$ partial pressure, and deposition time were varied. The SiH$_4$ to GeH$_4$ gas flow ratio was held constant in order to target a 70% Ge content. The flow rates of the two gases however were reduced in some cases to increase the partial pressure of BCl$_3$. The high Ge content was chosen to ensure crystallinity for consistent resistivity measurement with boron doping. It should be noted that the selectivity of pure Ge to Si$_{30}$Ge$_{70}$ for H$_2$O$_2$ etching is degraded to 10:1 [5]. High peroxide etching selectivity is desired for integrated MEMS applications because the use of pure Ge as the conformal sacrificial layer eliminates the need to passivate the underlying CMOS.

Experimental data was collected with five 4”-diameter wafers that were placed in slots 3, 8, 13, 18, and 23 of the wafer boats, counting from the gas inlet side. A four-point probe instrument was used to measure the sheet resistance. The films were patterned and etched for the thickness measurement using a stylus-based profiler. Wafer curvature was measured before and after SiGe deposition (backside SiGe film removed) to determine the average residual stress of the film. A cantilever beam array was patterned and released for strain gradient measurement. The strain gradient was calculated as the reciprocal of the radius of curvature of the cantilevers with various lengths. Resistivity, thickness, and strain gradient were measured at various points on each wafer, and average numbers are reported in this paper. Ge content, B and Cl concentrations were determined by secondary ion mass spectrometer with wafers at the gas inlet side of the load. The crystallinity of selected films was determined by transmission electron microscopy.

RESULTS AND DISCUSSIONS

Overall results of average deposition rate, resistivity, residual stress, Ge content, and B doping level, along with uniformity of the BCl$_3$ and B$_2$H$_6$ doped poly-SiGe processes are summarized in Table I. Both B$_2$H$_6$ and BCl$_3$ doped SiGe films have similar Cl concentration, all below 2×10$^{16}$ cm$^{-3}$, which indicates Cl incorporation is not a problem for the BCl$_3$ doping process.
**Table I Process Summary**

<table>
<thead>
<tr>
<th>Run</th>
<th>a-Si</th>
<th>SiGe layer</th>
<th></th>
<th>Results</th>
<th></th>
<th></th>
<th>Gas Inlet Side</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>Temp (°C)</td>
<td>Temp (°C)</td>
<td>SiH₄ (sccm)</td>
<td>GeH₄ (sccm)</td>
<td>BCl₃/He (sccm)</td>
<td>BCl₃ conc.</td>
<td>Time (min)</td>
</tr>
<tr>
<td>T22</td>
<td>450</td>
<td>350</td>
<td>0</td>
<td>100</td>
<td>16.5</td>
<td>0.1%</td>
<td>60</td>
</tr>
<tr>
<td>T2</td>
<td>400</td>
<td>400</td>
<td>100</td>
<td>60</td>
<td>9.2</td>
<td>0.1%</td>
<td>60</td>
</tr>
<tr>
<td>T4</td>
<td>400</td>
<td>400</td>
<td>100</td>
<td>60</td>
<td>16.5</td>
<td>0.1%</td>
<td>60</td>
</tr>
<tr>
<td>T5</td>
<td>425</td>
<td>425</td>
<td>100</td>
<td>60</td>
<td>1.8</td>
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</tr>
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<td>100</td>
<td>60</td>
<td>9.2</td>
<td>0.1%</td>
<td>60</td>
</tr>
<tr>
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<td>60</td>
<td>16.5</td>
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<td>60</td>
</tr>
<tr>
<td>T11</td>
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<td>100</td>
<td>60</td>
<td>16.5</td>
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<td>60</td>
</tr>
<tr>
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<td>100</td>
<td>60</td>
<td>16.5</td>
<td>0.1%</td>
<td>120</td>
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<tr>
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<td>0.1%</td>
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<td>48</td>
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<tr>
<td>T18</td>
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<td>50</td>
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<td>120</td>
</tr>
<tr>
<td>T9</td>
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<td>425</td>
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<td>12</td>
<td>16.5</td>
<td>0.1%</td>
<td>180</td>
</tr>
<tr>
<td>T3</td>
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<td>100</td>
<td>60</td>
<td>9.2</td>
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</tr>
<tr>
<td>T19</td>
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<td>100</td>
<td>60</td>
<td>3.6</td>
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<tr>
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<td>100</td>
<td>60</td>
<td>9.2</td>
<td>1%</td>
<td>60</td>
</tr>
<tr>
<td>T21</td>
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<td>425</td>
<td>100</td>
<td>60</td>
<td>9.2</td>
<td>1%</td>
<td>60</td>
</tr>
<tr>
<td>T23</td>
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<td>100</td>
<td>60</td>
<td>9.2</td>
<td>1%</td>
<td>60</td>
</tr>
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<td>100</td>
<td>60</td>
<td>60</td>
<td>60%</td>
<td>60</td>
</tr>
<tr>
<td>R1*</td>
<td>425</td>
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<td>60</td>
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<td>60%</td>
<td>60</td>
</tr>
<tr>
<td>R2*</td>
<td>450</td>
<td>450</td>
<td>100</td>
<td>60</td>
<td>60</td>
<td>60%</td>
<td>60</td>
</tr>
</tbody>
</table>

*B₂H₆ was introduced from the pump side through a multi-pore injector. Caged boats were used. Thirty 4"-diameter and fifteen 6"-diameter wafers were used per load.
A. Deposition Rate

Comparison of Runs T5, T1, T6, T19, T20, and T21 with BCl3 partial pressure as the only difference, we can see deposition rate is independent of BCl3 flow. Also, depositions with identical parameters but various deposition times (Runs T11, T14, and T12) show the same deposition rate, which suggests that there is no incubation period at the beginning of the deposition.

An Arrhenius plot of deposition rate is shown in Fig. 2 for 100 sccm SiH4, 60 sccm GeH4 depositions at 400 mTorr for both BCl3 and B2H6 processes. The deposition temperatures of these runs are rather low and the processes are believed to be operating in reaction-limited regime. The activation energies of the BCl3 and the B2H6 doped processes are extracted to be 0.94 eV and 0.42 eV, respectively. They are of the same order of magnitude as the undoped poly-SiGe process reported before [6]. For the 425 °C and 450 °C depositions, the deposition rates are similar for both dopant gases. B2H6 process has a much higher deposition rate at 400 °C. The resistivity of the 400 °C B2H6 recipe is very low, and boron incorporation for this film is estimated to be on the order of $1 \times 10^{21}$ cm$^{-3}$. B2H6 is known to enhance deposition for poly-Si, but the temperature effect and the doping effect cannot be separated in Fig. 2.

![Fig. 2 Deposition rate vs. Temperature](image)

B. Crystallinity

Cross-sectional TEMs for films of different thicknesses are shown in Fig. 3. These
two films have exactly the same recipe except the deposition times are 1 hour and 3 hours for the thin film and the thick film, respectively. Both films have columnar grain structure with finer grains at the bottom. The two TEMs are shown on the same scale. As the film gets thicker, the grains grow significantly larger.

![Fig. 3 Cross-sectional TEM: (a) Run T6 - 0.5 um film; (b) Run T12 - 1.5 um film](image)

C. Dopant incorporation

Fig. 4 plots boron concentration vs. partial pressure for all 425 °C poly-SiGe depositions. As a comparison, data for epi-SiGe deposited at 470 °C [2, 3] is also shown. The difference in dopant incorporation of the two systems comes from several sources: the deposition temperature of the poly-SiGe film is much lower, the oxygen contamination level of the poly-SiGe system is an order of magnitude higher, the poly-SiGe film is deposited on an oxide surface, whereas epi-SiGe film is deposited on an ultra-clean Si surface, the germanium content of the poly-SiGe film is much higher, and the deposition rate of the poly-SiGe film is more than 10× faster than that of the epi-SiGe. All of the above differences result in more than two orders of magnitude offset in boron incorporation between the films.

A data point from the B₂H₆ process is also shown in Fig. 4. A high B₂H₆ partial pressure is required to achieve similar doping levels in the film while other deposition conditions are identical. The consumption of B₂H₆ is mainly due to the decomposition inside injector rather than the disassociation on the wafer surface. Comparing the three B₂H₆ depositions (R3, R1 and R2) at various temperatures provides further evidence of
the temperature instability of $\text{B}_2\text{H}_6$. Resistivity is found to be an order of magnitude higher for an increment in temperature of 25 °C with the same $\text{B}_2\text{H}_6$ flow rate.

Fig. 4 Boron concentration vs. Dopant gas partial pressure

Resistivity vs. boron concentration for the $\text{BCl}_3$ doped process is plotted in Fig. 5 for ~70% germanium content films deposited at 425 °C with similar thicknesses. As expected, resistivity decreases linearly with boron doping. The resistivity of poly-SiGe is more than 10× higher than that of single crystalline films due to carrier trapping at the grain boundaries.

Fig. 5 Resistivity vs. Boron concentration
A comparison of BCl$_3$ runs (T1, T2, and T3) with same deposition conditions except temperatures shows higher temperature gives lower doping level and higher resistivity. This phenomenon could be explained by a lower boron surface reaction while the deposition rate goes up with temperature.

Resistivity vs. film thickness is plotted in Fig. 6(a) for runs having the same deposition conditions but different deposition times. Wafer positions are also labeled on the graph. For wafers from the same run, higher resistivity and lower deposition rate are observed at the gas outlet due to the gas depletion effect. Comparing wafers at the same position from different runs, thicker films are found to have lower resistivity. This might be a result of furnace annealing and/or crystal growth during deposition. A two-hour 425 °C anneal in N$_2$ ambient was done for several 1-hour deposition films to discriminate the two effects. There is no change in resistivity after the annealing. As shown in Fig. 3 earlier, thicker films have larger grains, which is consistent with this lower resistivity observation since carrier trapping is more significant with higher grain boundary density [8].

![Graph showing resistivity vs. film thickness](image)

**Fig. 6 (a)** Resistivity and (b) Stress vs. Film thickness

D. Stress

Fig. 6(b) shows the average residual stress vs. film thickness. Thicker film has less compressive stress. Furnace annealing at 425 °C for 2 hours does not cause significant
change in residual stress. This reduction in residual stress magnitude with thickness allows the growth of thick films without peeling off. However, the origins of the residual stress and the reasons for its decrease with film thickness are not completely understood.

Comparing films with similar thickness and boron doping as the only process variable, we can see that the average residual stress is more compressive as boron doping increases, as shown in Fig. 7. This possibly indicates the boron atoms are not residing in substitutional lattice sites, but interstitial lattice sites or grain boundaries. A data point from the B$_2$H$_6$ process shows that the average stress for B$_2$H$_6$ doped film is less compressive for the same level of doping. Further investigation on crystallinity might be able to explain this phenomenon. However, 0.6 µm films are too thin for most MEMS applications, and the 1.7 µm thick BCl$_3$ doped film with $6.0 \times 10^{18}$ cm$^{-3}$ doping yields reasonably low residual stress at -20.9 MPa.

![Average stress vs. doping](Fig. 7)

E. Strain gradient

The cantilever arrays from the thick films are released for strain gradient measurement. Other films with thickness less than 1 µm are too thin for reliable strain gradient measurements. As shown in Fig. 8, the error increases as the film gets thinner. Strain gradient is smaller for thicker film, and the reason is not well-understood. On the other hand, the trend for strain gradient vs. resistivity is not very clear, which indicates doping is a second order effect on strain gradient.
F. Process uniformity

Process uniformity summarized in Table I is being calculated as the normalized standard deviation across the wafer and across the load. A few B$_2$H$_6$ doped depositions done in the same LPCVD reactor are listed at the bottom of Table I for comparison. The dopant gas B$_2$H$_6$ is introduced from the pump side via a multi-pore injector located underneath the wafer boats to minimize the depletion effect. Caged boats were used in the B$_2$H$_6$ doped process. The pump-side injector and caged boats for the B$_2$H$_6$ doped process are not shown in Fig. 1. As a comparison, the BCl$_3$ process provides similar uniformity within a wafer and better cross-wafer uniformity in most cases with open boats and no gas injector.

The wafer flat region has lower resistivity for the BCl$_3$ process, which is due to the fact that the 4” wafers are placed below the center axis in the 9”-diamter reactor and there is more open space for gas flow on the top of the wafer flat. As a comparison, 6” wafers, which are nearly centered in the reactor, have better cross-wafer uniformity in resistivity.

Deposition rate and resistivity of selected 425 °C runs are plotted vs. wafer position in Fig. 9 to demonstrate the cross load uniformity of the BCl$_3$ process. Wafers at the gas inlet side have higher deposition rate and lower resistivity, which suggests a gas depletion effect. Increasing the gas flow rate along with using an injector to introduce BCl$_3$ could
result in better cross load uniformity. A 425 °C B₂H₆ deposition is also shown in Fig. 9 as a reference. Cross load variation in deposition rate and resistivity is similar to the BCl₃ process, despite the fact that B₂H₆ is introduced via a multi-pore injector. Cross load resistivity of the B₂H₆ shows the opposite trend compared to the BCl₃ process, because B₂H₆ was introduced from the pump side.

![Fig. 9 Deposition rate and resistivity across load](image)

Runs T6 and T11, as well as Runs T20 and T23 have exactly the same deposition parameters, but they were done separated by a few other depositions. The results show that the BCl₃ process is repeatable. Moreover, results of other similar depositions are reasonably consistent, which indicates that the process is operating in a relatively stable manner. In comparison, the decomposition of B₂H₆ causes periodic clogging of the injector, which results in large run-to-run variation unless the injector is changed approximately every 30 hours of deposition. Decomposition of BCl₃ is rather slow compared to B₂H₆, and injector clogging is not anticipated to be a problem. On the other hand, introducing B₂H₆ through the gas ring to avoid injector clogging is not an option since large across load variation in film thickness and resistivity [9] resulted from its tendency to decompose at low temperature.
CONCLUSIONS

We have investigated the depositions and properties of LPCVD poly-Si$_{30}$Ge$_{70}$ using BCl$_3$ as a dopant source. The desired doping level for poly-Si$_{30}$Ge$_{70}$ is achieved with 1% BCl$_3$ concentration. The better uniformity and higher doping efficiency for the BCl$_3$ process is clearly advantageous over the B$_2$H$_6$ process for poly-SiGe films, but degradations in deposition rate and increases in residual stress are the tradeoffs. Tradeoffs between residual stress and resistivity are also involved in optimizing boron concentration for the BCl$_3$ doped process. Both residual stress and resistivity can be improved by increasing the film thickness. Thicker films also have advantages in strain gradient and grain crystallinity, and thicker films are desired for better electro-mechanical performance in MEMS applications. Further modifications of the LPCVD reactor will be investigated to improve the BCl$_3$ process.

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REFERENCES