Low Stressed In-situ Boron doped Poly SiGe Layers for High-$Q$ Resonators


Abstract—In-situ boron doped LPCVD polycrystalline silicon-germanium (poly SiGe) layers are deposited from SiH$_4$ and GeH$_4$ with 0.2% diborane (B$_2$H$_6$) in argon (Ar) as dopant precursor at 430 °C and 0.2 mbar. The characterized layers show very low tensile stress (12 MPa) for the diborane mixture flow of 50 sccm for fixed SiH$_4$ and GeH$_4$ flow. However, an increase in diborane mixture flow to 100 sccm at the same SiH$_4$ and GeH$_4$ flow results in a transition from low tensile to low compressive stress (3 MPa) in the deposited layers. The sheet resistance of these in-situ doped layers is two orders of magnitude lower compared to undoped layers with the same Ge contents. However, we have not observed any appreciable difference in the sheet resistance of these in-situ doped layers due to the saturation limit of the active dopants in the deposited layers. Hence, the deposited layers are found to be suitable for post processing high-$Q$ resonators on top of readily available CMOS circuitry not only due to low thermal budget requirements (< 450 °C) but also because of their superior electrical and mechanical properties. Additionally, the analyzed samples through XRD reveal the preferential columnar grain growth on 100 nm SiO$_2$ that can be exploited to have steep etch profiles with SF$_6$ and O$_2$ plasma at cryogenic temperatures.

Index Terms— Poly SiGe, High-$Q$, In-situ doping, Diborane, Post Processing.

I. INTRODUCTION

POLYCRYSTALLINE silicon-germanium (poly SiGe) is emerging as an exotic structural material for post processing microelectromechanical systems (MEMS) on top of foundry fabricated CMOS [1-4] apart from its potential use as metal-oxide semiconductor field effect transistor (MOSFET) gate material [5-6]. The lower deposition temperatures of doped poly SiGe alloy, dependent on Ge contents, along with its electrical and mechanical properties are the key feature for the use in post processing technologies compared to doped poly silicon at comparable deposition temperature [7]. The higher deposition rate of poly SiGe alloy with increased Ge contents while keeping the temperature to be fixed has previously been reported [8]. Therefore, the Ge contents between 60-70% is an optimal for low temperature MEMS fabrication while maintaining a higher deposition rate than low Ge content poly SiGe alloy. When the conductivity of this layer can be tailored by controlled doping, the range of application of the SiGe layer extends considerably. To that purpose, the doping techniques like diffusion and implantation can’t be employed due to the high temperature conditions for diffusion and annealing. This comes with an inherent disadvantage of non-uniform doping profile for thick layers. This leaves in-situ doping the only technique for uniform dopant profile throughout the layer thickness at temperatures compatible for post processing. Amongst the myriad of techniques being reported [9-12] from various research groups, the low pressure chemical vapor deposition (LPCVD) technique for poly SiGe films deposition is considered to be superior and most widely used to other deposition techniques due to its higher throughput, uniformity and reproducibility [13], with a set back of low deposition rate (2-30 nm) [14]. The PECVD technique is an alternate if high deposition rate, an order of magnitude higher than LPCVD [15], is required with a compromise on uniformity and throughput for a conformal deposition. Another problem associated with the layers deposited with PECVD technique is their amorphous nature which in some applications is quite critical where acoustic losses needs to be minimized for attaining higher $Q$ with deposited layers to be polycrystalline. Moreover, combination of LPCVD and PECVD techniques has also been reported for the deposition of structural layer for a gyroscope [16].

We have deposited in-situ boron doped LPCVD poly SiGe layers from silane and germane with 0.2 % diborane, as dopant gas, mixed in argon at 430 °C and 0.2 mbar. The deposited layers show their potential to use them as lowly resistive structural layer for MEMS that can be easily integrated with CMOS. These layers have a quite uniform boron profile throughout their thickness which makes these layers quite suitable to serve as structural layers for MEMS devices.

The paper is organized as follows. In Section II, we briefly describe the associated deposition parameters for poly SiGe to have 70% Ge contents and uniform boron incorporation. In Section III, the structural characteristics are presented: XRD analysis for the Ge contents and the grain orientation and SIMS analysis for the detailed depth profile of the deposited layers. Section IV would be devoted to summarize the results obtained form the characterization of the deposited layers.

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II. EXPERIMENTAL

Poly SiGe layers were deposited on 100 mm single side polished <100> oriented wafers (380±15 µm, n-type/P doped, 1-10 Ω-cm) with 100 nm of thermally grown oxide. The wafers were directly loaded into LPCVD system after cleaning in 99% HNO₃ for 5 min followed by DI water rinse and N₂ drying. Initially, the pressure inside the tube is maintained at 10 mbar, apart from the set pressure of 3 mbar, to heat up the wafers through convection for 30 minutes so that the temperature of each wafer inside LPCVD system reaches to 430 °C. The surface is further modified by depositing a thin (few nm) amorphous silicon layer at 0.5 mbar and 430 °C for 10 min with SiH₄ flow of 88 sccm, a necessary step for poly SiGe deposition on SiO₂ surface [8]. The in-situ boron doped poly Si₁₋ₓGeₓ deposition was then performed in the conventional LPCVD system (Fig. 1) at 430 °C and 0.2 mbar without vacuum break from the pyrolysis of pure silane (SiH₄) and pure germane (GeH₄) gasses with 0.2% diborane (B₂H₆) diluted in argon gas (Ar) as dopant precursor. All the gasses were introduced from the front side of the LPCVD tube. A total of 13 wafers were LPCVD deposited in each run including 3 dummy wafers, 1 in front and 2 at the back. The gas depletion effect was minimized with the use of roots blowers to finally achieve good cross load thickness uniformity throughout the entire boat of the wafers. The experiments were performed at varying B₂H₆ mixture flow rates 0, 50, 100 sccm with fixed SiH₄ to GeH₄ ratio (75:37) and the deposition time of 70 min to initially check for the dopant distribution and Si and Ge contents in the entire thickness of the deposited layers.

III. CHARACTERIZATION

The thickness of the deposited poly SiGe layers increases with the addition of diborane compared to undoped layers. However, the increase in the deposition rate is not linear with the addition of diborane. We observed an increase in the deposition rate of the deposited layers for a flow of 50 sccm of diborane mixture and than a decrease in the deposition rate for further increase of diborane mixture flow to 100 sccm. The deposition rate for the deposited poly SiGe is shown in fig. 2. The deposition rate is based on the thickness measured from the HRSEM of the samples. However, the thickness measured from Dektak on same samples is 70 nm (surface roughness) more than HRSEM measured thickness of 250 nm, 350 nm and 470 nm corresponding to 0 sccm, 50 sccm and 100 sccm flow of diborane mixture. The observed difference is because of the surface roughness which adds up to thickness measured from HRSEM images. The increased deposition rate with the germane contents [17] and the impact of boron incorporation to improve crystallinity while forming relatively bigger grains than in the undoped layer is well documented in the literature [18]. However, the observed increase in the deposition rate at high Ge contents (~70%) with low B₂H₆ flow rate is neither observed nor documented anywhere.

The stress in the in-situ deposited layers is calculated by Stoney’s formula while measuring the wafer curvature in two orthogonal directions. The calculated stress in the layers with 100 sccm of diborane mixture flow shows low compressive (~3 MPa) stress in the layers. Whereas, the layers deposited with 50 sccm of diborane mixture flow, keeping the silane and germane flow the same, shows up with low tensile (~12 MPa) stress. Thus, an increase in diborane mixture flow attributes to a transition from low tensile to low compressive stress. The observed shift from tensile to compressive can be explained on the basis of the incorporation of boron atoms in the
deposited layers leading to compressive stress in the deposited layers. Fig 4 shows this stress transition from tensile to compressive with increase diborane mixture flow.

Fig 3: Stress transition from tensile to compressive

The sheet resistance of in-situ doped layers is two orders of magnitude lower compared to the undoped layers. The sheet resistance of layers are 16.5 $\Omega/\square$ and 18.5 $\Omega/\square$ for 100 sccm and 50 sccm flow of diborane mixture compared to 4.5 $k\Omega/\square$ of undoped poly SiGe layers with same Ge contents of 70%. It is evident that there is no remarkable difference in the sheet resistances with the varied diborane mixture flow rates. A valid reason for this observed phenomenon could be the saturation level of the active boron atoms for the particular value of deposition temperature. Therefore further increase in the flow rate of B$_2$H$_6$ mixture to 100 sccm only appears as an increased boron concentration in the deposited SiGe layers rather than contributing towards active dopant. These excess boron atoms are supposed to occupy interstitial points or lie along the grain boundaries rather than occupying substitutional lattice point to help reduce the sheet resistance further below. This is confirmed during the SIMS analysis where boron concentrations of 1.2x10$^{21}$ cm$^{-3}$ and 6.9x10$^{20}$ cm$^{-3}$ are observed in the deposited samples with 100 sccm and 50 sccm diborane mixture flow rate. The underlying assumption for this comparison is based on the fact that the grain size for both deposited layers is the same that don’t help to lower sheet resistance with increased grain size. The cross wafer uniformity in the sheet resistance was found to be better than 2% with a cross load uniformity in sheet resistance of 5%.

The XPS analysis was done on the undoped samples and the Ge contents in the deposited layers were found to be ~70% in the deposited layers. The XRD analysis was carried out with Philips XRD model Expert system II with Cu K $\alpha$ line of wavelength 1.54 Å. The diffraction peaks (111), (220) and (311) clearly reflect the diamond crystal structure of the in-situ deposited layers. The peaks are shifted towards the Ge peaks because of Ge contents higher than Si in the samples. The Ge contents were found to be 76%, 75% and 74%, averaged obtained from the three peaks, for diborane flow rate of 0 sccm, 50 sccm and 100 sccm respectively on the deposited samples with vegard’s law [19]. The lattice constant was found to be are 5.60 Å for the all the deposited layers. Fig 4 shows the XRD pattern of the deposited poly SiGe layers. The undoped layer and the layer deposited with 100 sccm of diborane mixture flow shows a preferential V-shaped grain growth associated with (111) dominant peak. Whereas the doped layer with 50 sccm of diborane mixture shows a preferential columnar grain growth associated with (220) dominant peak. However, the (220) peak is not that much prominent over (111) peak, therefore we can assume a mix of V-shaped and columnar grains growing under the condition that corresponds to 50 sccm flow of diborane, with columnar growth to be dominant. These columnar grains thus grown can be etched very steeply and selectively to SiO$_2$ at cryogenic temperatures with SF$_6$ and O$_2$ plasma chemistry [20].

Fig 4: XRD foot prints of poly SiGe layers with diborane flow of (a) 0 sccm (b) 50 sccm (c) 100 sccm

The HRSEM images of the deposited poly SiGe layers are shown in following fig 5. The grain orientation can be quantified with these images, columnar for 50 sccm diborane flow and V-shaped for other deposition conditions.
Fig 5: HRSEM images of poly SiGe layers with diborane flow of (a) 0 sccm (b) 50 sccm (c) 100 sccm

To validate the results obtained from XRD the SIMS analysis of the samples is performed. SIMS analyses were performed using 3 keV O²⁺ primary ions bombardment with positive mode. The calculated values are in agreement with those by SIMS within a certain experimental accuracy. The first ~10 nm of the profiles are unreliable due to transient instrumental effects and also the profile region close to oxide, within the oxide and after oxide is unreliable due to charging and matrix effects. The instrumental conditions that minimize these effects are not chosen to avoid the enhancement of the matrix effects between Si and SiGe. The depth profile result shows that there is no variation in the Ge and Si contents throughout the thickness of the deposited SiGe layer. Also the Ge contents in the deposited layers were found to be 70 atomic percent. Fig 6 shows the depth profile of Ge and Si for the two different batches and with two different positions in the boat (first and last wafer). The observed kink in Si depth profile near the interface gives us a clear indication of a-Si seed layer that we intentionally deposited for creating nucleation sites for poly SiGe deposition.

The boron depth profile was also examined with SIMS and found to be uniform, with a systematic error of a factor of 2, throughout in-situ doped poly SiGe layers. The concentration of boron in the layer was found to be 1.2x10²¹ cm⁻³ for layers with B₂H₆ flow of 100 sccm and this value decreases to 6.9x10²⁰ cm⁻³ for the layers with B₂H₆ flow of 50 sccm. This indicates that the boron concentration in the deposited layers is almost flat and follows a linear relationship with the diborane flow. As previously discussed that this born incorporation doesn’t help reducing the sheet resistance any further because of saturation limit reached for active dopants. A saturation limit for the active boron concentration of about 5.0x10²⁰ cm⁻³ is reported for poly SiGe layers deposited at 550 °C regardless of the Ge contents in the poly SiGe alloy [21].

We expect this saturation value to be further lower than reported because of the deposition temperature 100 °C lower than reported one (550 °C). The depth profile for boron, fig 7, also reveals that the poly SiGe layers with 50 sccm flow of diborane mixture are thicker compared to the layers deposited with 100 sccm of diborane flow.

Fig 6: SIMS of poly SiGe samples across the entire thickness (a) Ge depth profile (b) Si depth profile

Fig 7: Boron depth profile in poly SiGe layers deposited at 430 °C
IV. CONCLUSIONS

Boron doped poly SiGe shows its potential to use as a structural material for MEMS components at low thermal budget IC processes compared to doped poly silicon. We have developed an optimized recipe for the in-situ boron doped poly SiGe deposition from silane and germane with 0.2% diborane in argon as dopant precursor. The process is fully compatible for post processing of the intended high-Q resonators on top of already fabricated CMOS chip with aluminum interconnects. The deposited layers exhibit the lowest tensile stress and lowest sheet resistance achieved till date at the comparable Ge contents and temperatures below 450 °C without any further treatments (annealing, laser enhanced crystallization etc). The role of low flow rate of dopant precursor on deposition rate is also carefully examined. The incorporation of diborane is generally found to increase the deposition rate at low diborane flow mixture with high germane contents ~70%. A slight increase in the deposition rate ~1.5 nm/min with the diborane mixture flow rate reduced from 100 sccm to 50 sccm of mixture is observed. However, the reason for this observed mechanism is still unknown. The boron concentration found to vary linearly with the flow rate of diborane mixture but no further reduction in the sheet resistance is measured because of a saturation limit reached for active boron incorporated in the layers at 430 °C. The depth profile measured through SIMS analysis reveals a homogeneous distribution of boron atoms throughout the deposited layers which reflects the suitability of these layers to use as MEMS structural layers. Additionally, silicon and germanium are found to be uniformly distribution throughout the entire thickness of the layer. The XRD analysis performed on the samples shows the growth of V-shaped and coloumner grains on SiO2 depending on the deposition conditions with columnar grain growth to be dominant on V-shaped grains at 50 sccm flow of B2H6 mixture flow. The reason for this observed phenomenon is still under investigation.

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