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Epitaxial SiC Development for High Nitrogen Incorporation

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Abstract. SiCOI is used as a lower cost substrate for power electronics and enables fabrication of MEMS and photonics platforms. Modification of the mechanical properties of SiC through doping is a potential pathway for improving resonator performance. This effort aimed to develop growth parameters for growth of 4H-SiC with nitrogen concentrations of $2x10^{20}$ cm^{-3} with a smooth surface morphology for fabrication of SiCOI wafers for MEMS fabrication. Growth conditions that were investigated include substrate polarity, growth pressure, C/Si ratio, temperature, and carrier gas flow. Highly doped grown epiwafers contained particle defects which exhibited different morphologies for C- and Si-polarities.

Introduction

Silicon Carbide for SiC-on-insulator (SiCOI) has been established as a lower cost substrate for power electronics [1]. The structure of the SiCOI substrate allows for easy fabrication of undercut structures through the removal of the intermediate oxide layer with a buffered oxide etch for micro-electromechanical systems (MEMS) [2] and photonics platforms [3]. Silicon carbide is of particular interest for improving MEMS resonator gyroscope performance over silicon devices due to small phonon Akhiezer dissipation and the in-plane isotropic hexagonal crystal lattice.

The modification of the dopant density provides a promising pathway to modify the temperature coefficient of frequency (TCF) to optimize the Q-factor for acoustic resonators. This has been demonstrated in silicon [4] and 3C-SiC [5]. The work in 3C-SiC by Sapienza et.al. noted that Young's modulus increases with increasing nitrogen incorporation in 3C-SiC grown on <111> silicon. They also observed enhancement of tensile stress for aluminum doped 3C-SiC grown on <100> silicon. Subsequent work also correlated improvement in Young's modulus with a decrease in the density of stacking faults [6].

The work presented in this paper was conducted to grow as highly doped 4H-SiC as possible so that in future work we can determine if it is possible to incorporate enough dopant to observe a similar effect in highly doped 4H-SiC. The theoretical solubility limits for nitrogen and aluminum in 4H-SiC are $>2x10^{20}$ cm^{-3} and $1x10^{21}$ cm^{-3}, respectively [7], [8]. The dopant of choice in this work was nitrogen, and not aluminum, as aluminum has a well-known memory effect. Epitaxial 4H-SiC is typically not doped above the low 10^{19} cm^{-3} dopant concentration, as nucleation of stacking faults occurs [9]. Many of the growth parameters that can be varied during hot-wall chemical vapor deposition (HWCVD) growth have been reported in the literature including substrate polarity, growth pressure, C/Si ratio, nitrogen flow and temperature over the typical (up to low 10^{19} cm^{-3}) doping range[10], while noting that Chen et.al. [11] did demonstrate doping up to $1x10^{20}$ cm^{-3} on C-face 4H-SiC.

In this work, we have varied these same parameters, and the carrier gas flow, over a larger range in an effort to incorporate nitrogen as close to the solubility limit as possible, in at least 20 μm of epitaxial material, while maintaining a smooth surface morphology for fabrication of a SiCOI layer.

Methodology and Results

This work was conducted with a well-used set of susceptor parts that were nearing the end of their lifetimes with the anticipation of developing a large n-type background. Note that this was confirmed. For a C/Si ratio of 1.3 before the growth campaign the background electron concentration was $1x10^{14}$ cm^{-3} and was in the 10^{16} – 10^{17} cm^{-3} range at the end of the growth campaign. It is known that nitrogen incorporates at a higher rate for C-face substrates [10]. However, as we were not able to obtain epi-ready C-face wafers this work started with growth on Si-face substrates. We measured the C-face of a Si-face epi-ready wafer by AFM after the initial ramp up to growth temperature and hydrogen etch of the surface and observed a bilayer stepped surface, with an rms roughness of 0.21nm, without polishing artifacts and decided this was sufficient to pursue the C-face work.

Samples were grown on N+, 100mm 4H-SiC substrates utilizing 5% silane (SiH_4) balance H_2, ethylene (C_2H_4), and 10 lpm of N_2 gas as the dopant in an LPE PE106 HWCVD system. Nitrogen concentrations ([N]) were determined on three samples, two Si-face and one C-face, grown with varying growth conditions using secondary ion mass spectroscopy (SIMS) (EAG Laboratories, Sunnyvale, CA). The samples were grown with a silane flow of 40 sccm for the two Si-face wafers and the C-face wafer was grown at 40 sccm for the C/Si ratio steps, but at 66.6 sccm for the rest of the steps to increase the growth rate for thick layer growth. Thicknesses were determined using the lengths of defects from particles, as the doping was too high for reflectance measurements. Surface profiles were acquired with a Keyence VK-X3100 3D surface profiler.

Nitrogen Incorporation Nitrogen concentrations are plotted in Fig. 1 on the same scale for various growth conditions. These plots elucidate trends for a narrow sampling of parameter space in this system which were specifically investigated to determine growth parameters which would reach [N] near the theoretical solubility limit. The legends indicate relevant growth parameters for each series as the parameter space is interdependent on the many variables explored here. All data points were taken in the carbon stable growth regime. This means that changing the silicon flow results in a change in growth rate. The inflection point to silicon stable occurs near C/Si = 1.3 for this apparatus. In Fig. 1.a) the [N] decreases with increasing C/Si, exhibiting the site competition effect [12]. Note that Arvinte et.al. have reported N incorporation in C and Si-face 4H-SIC is similar for silicon stable growth for [N] under $1x10^{18}$ cm^{-3} [13]. For the rest of the plots a C/Si ratio of 0.8 was selected, as decreasing the ratio further was detrimental to the surface morphology. Fig. 1.b) shows increasing nitrogen incorporation with increasing pressure, consistent with the trends up to $1x10^{19}$ cm^{-3} in [9]. Fig. 1.c) shows that [N] decreased with increasing temperature. Of note, at 1600°C the growth rate is measurably lower than that measured at 1580°C indicating at this temperature there is measurable etching in competition with the growth. Fig. 1.d) shows that for increasing nitrogen partial pressure (lower carrier gas flow) a higher [N] is attained.

Uniform Epilayers Uniform, nominal 13 μm epilayers were grown on a ¼ of a 100 mm 4°-off 4H-SiC wafer to determine if the growth parameters that reached the highest [N] would have a smooth surface morphology. For the Si-face growth, large triangular defects nucleated at the growth interface as shown in Fig. 2.a). The insets of Fig. 2 show the locations where a surface profile was taken for each defect, the profile is shown below the Nomarski micrograph. These defects affect large areas of the surface and extend above and below the epi-surface by a few micrometers. The growth conditions that resulted in a specular surface as observed by Nomarski micrographs, (except for the large defects) were a temperature of 1580°C, H_2 carrier gas flow of 80 slm and 400 mbar which should result in a [N] of $1.0x10^{20}$ cm^{-3}. These samples exhibited up to 20 nm steps by AFM, which could be removed with a chemo-mechanical polishing finish. Different variations of growth conditions that should result in higher [N] resulted in a rough surface with a high density of in-grown stacking faults. These in-grown stacking faults are the same as observed for high C/Si ratio growths and are found to nucleate at micropipes, mechanical damage or substrate defects [14].

Particle-nucleated defects on the C-face exhibited a different morphology as shown in Fig.2 b). These are formed by large particles that likely fell from the susceptor before the start of the growth. The particles are up to 100 μm in diameter, incorporate into the growing layer and affect morphology along the step-flow growth direction. The growth conditions that resulted in a smooth surface (except for the large defects) were a temperature of 1580°C, H_2 carrier gas flow of 100 slm and 100 mbar which should result in a [N] of 8.0x10^{19} cm^{-3}. This wafer has 40 large defects over a 100mm wafer. For growth conditions that should result in higher [N], a decrease in growth rate was observed with a large density of particles nucleating defects near the edge of the wafer. An example of this effect is shown in Fig. 3. These samples could be used to create SiCOI if the damaged outer area is removed before bonding.

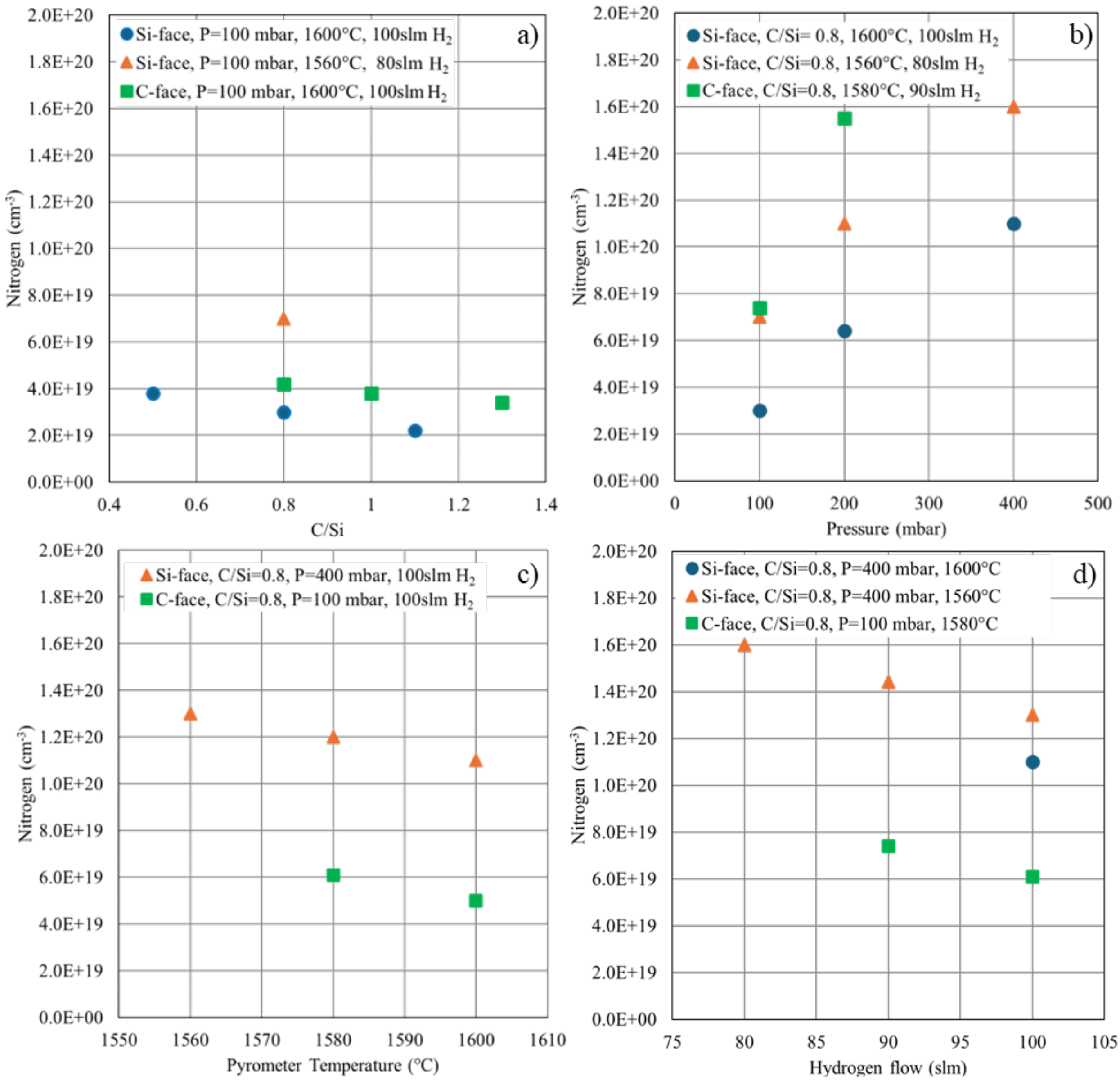

Fig. 1. Nitrogen concentration is plotted for both Si-face and C-face growth based on a) C/Si ratio, b) system pressure, c) temperature and d) the carrier gas hydrogen flow. Note that the SiH_4 flow was 40 sccm for all Si-face points and for the C-face in b). The C-face points for the rest had a SiH_4 flow of 66.6 sccm, resulting in higher growth rates.

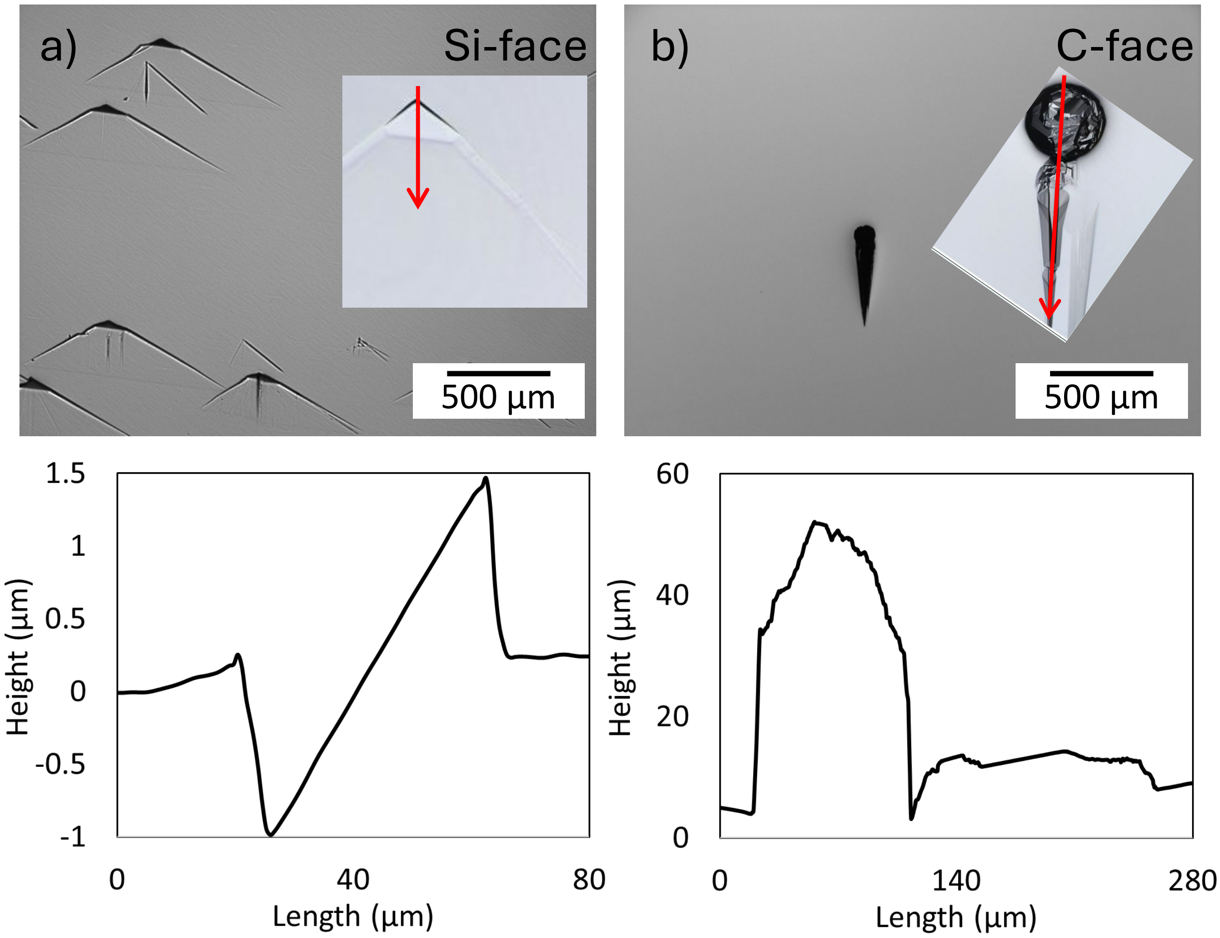

Fig. 2. a) ~13µm thick epilayer, Si- face growth with [N] ~5x10^{19} cm^{-3} and b) ~30 µm thick epilayer C-face growth with [N]~2x10^{20} cm^{-3}. The insets show the line profile, along the [1120], step-flow growth direction, (red online) taken for thickness variation as plotted below the Nomarski image.

Discussion

Growth conditions were found for both the carbon and silicon face that incorporated 1.6x10^{20} cm^{-3} nitrogen. These growth conditions had a low C/Si ratio that resulted in nucleation of silicon particles in the gas phase. The use of a well-used susceptor may have contributed to large particles which nucleate defects. These particles resulted in different polytype inclusion defects for the two faces. Growth parameters were found that minimized nucleation of particles and wafers were grown with nitrogen concentrations of 8x10^{19} cm^{-3} for carbon face and ~1x10^{20} cm^{-3} for the silicon face. The characteristic defects are shown in Figure 1 a) for the carbon face and b) for the silicon face. The defect nucleating from the particles on the carbon face growth projects above the surface, so they can be polished away for SiCOI fabrication. The polytype inclusion nucleating from the particles on the silicon face growth both protrudes and is recessed from the surface and also covers a much larger area making SiCOI fabrication challenging. The growth rate was also observed to decrease for growth conditions with higher nitrogen incorporation.

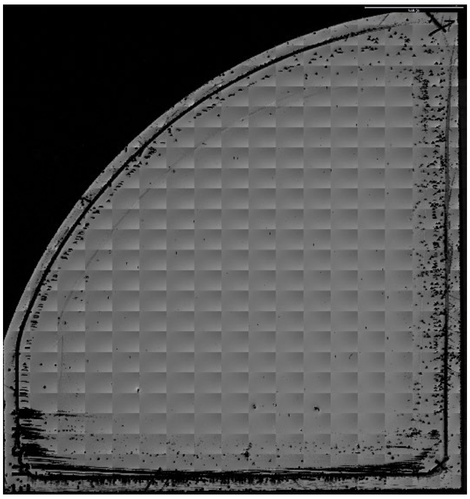

Fig. 3 Image of a quarter of a 100 mm, C-face wafer with decreased growth rate and particle issues near the edges of the sample.

Particle driven large morphological defects are the largest hindrance towards creation of commercial scale SiCOI with nitrogen concentrations above $1x10^{20}$ cm^{-3}. In future work the SiCOI layers will be fabricated into BAW resonators and characterized to determine if their inherent material properties enhance the resonator performance metrics. If the highly nitrogen doped SiC exhibits sufficient gain for MEMS resonators manufacturers can dedicate a set of susceptor parts to the high nitrogen process to isolate the memory effect and reduce the effects of using older parts.

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