

Growth and Characterization of “IsoPure” Epitaxial Layers for Quantum Applications

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Abstract. This paper compares ethene and methane precursors for homoepitaxial 4H-SiC growth in planetary reactors with regards to their impact on growth rate and defectivity of the epilayers. Therefore, a comprehensive experimental study has been performed in AIXTRON G10-SiC and G5WW C planetary reactors using a standard process based on ethene and trichlorosilane precursors with conventional 150 mm n-type 4H-SiC substrates from 3 different international suppliers. Methane substituted ethene as precursor in many experiments. It was found that methane precursor can compete with ethene in terms of growth rate, epilayer thickness, and defectivity of the epilayers. By using isotopically enriched methane, Si¹²C epilayers with a ¹²C concentration of 99.96 % have been grown which can be used for SiC-based quantum technology.

Introduction

Silicon Carbide (4H-SiC) has outstanding properties for applications in energy efficient power electronics and has been maturing with regards to material quality and device processing technology. Now, the SiC processing costs need to be reduced and the efficiency with regards to energy and resource consumption needs to be improved. For these reasons, we are testing alternative precursors such as methane (CH₄) and tetrachlorosilane (SiCl₄). These alternatives may enable higher growth rates, better material quality [1] and lower processing costs. Additionally, methane can act as an isotopically enriched precursor for growth of epilayers with a controlled ¹²C isotope concentration different from the natural isotope mixing ratio, which could be an enabler for SiC-based quantum technology due to the control of the nuclear spin bath concentration. SiC is a very promising candidate for quantum applications due to its silicon vacancy (V_{Si}), which acts as a color center [2] and its mature processing technology. Depending on the desired quantum application, specific isotope concentrations are required, such as a total concentration of 1.0 to 1.5 % of ¹³C and ²⁹Si for nuclear spin qubits addressable as quantum memories [3,4]. Hence, the ¹³C and ²⁹Si concentrations need to be reduced by using isotopically enriched precursors during epitaxial growth. In the carbon case, methane (CH₄) can be purchased with a nominal ¹²C concentration of 99.99 %. This paper investigates the epitaxial growth using methane as a precursor and the characterization of the grown epilayers.

Experimental

For this study, conventional n-type 4H-SiC substrates with a diameter of 150 mm from three international vendors A, B, and C were used for epitaxial growth studies in AIXTRON planetary reactors G5WW C and G10-SiC, which are operated in our joint development lab in Erlangen, Germany. The standard growth process is based on ethene and trichlorosilane precursors and uses nitrogen doping for n-type epilayers with the Multi-Ject® technology. This process was adapted to utilize methane as carbon precursor, while trichlorosilane (TCS) was further used as (standard) silicon precursor. The ethene precursor was substituted by methane. Once a suitable methane-based epitaxial

growth process was developed, isotopically purified methane with 99.99 % ^{12}C and ultra-low nitrogen concentration (< 3 ppm) was employed.

The epilayers grown in this study were characterized with regards to epilayer thickness, doping concentrations and defects by FTIR with a Semilab EIR 2300, capacitance-voltage (CV) measurements with mercury probes, and UV excited photoluminescence imaging (UVPL) in combination with differential interference contrast (DIC) microscopy with a Lasertec SICA 88. Minority carrier lifetime mappings were measured by microwave-detected photoconductivity decay (μ -PCD) method [5] with a Semilab WT-2000. The ^{12}C isotope concentration was determined by secondary ion mass spectroscopy (SIMS).

Results

Growth rate.

Many comprehensive growth experiments were conducted in both G5WW C and G10-SiC planetary reactors with ethene or methane precursor. Buffer layers with a thickness of $1\ \mu\text{m}$ to $2\ \mu\text{m}$ were grown as well as drift layers with a thickness up to $40\ \mu\text{m}$. The epilayer thickness is plotted as a function of the growth time in Fig. 1. The data points were linearly fitted and the slope of the fit yields the typical growth rate of $30\ \mu\text{m/h}$ for both ethene- and methane-based growth processes. Further experiments will be done to increase the growth rate.

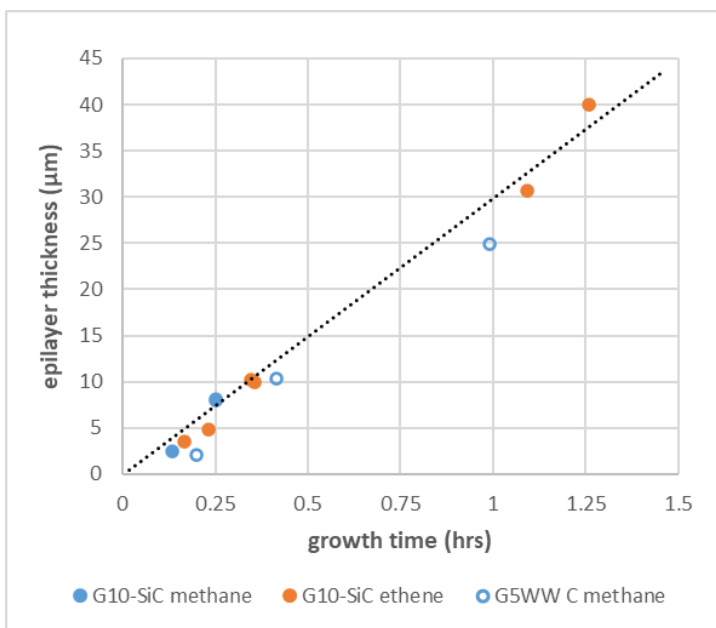


Fig. 1. Epilayer thickness as a function of growth time for different planetary reactors with different C precursors ethene (standard C precursor) and methane (new C precursor). The slope of the linear fitting, displayed as black dotted line, gives a growth rate of $30\ \mu\text{m/h}$ for these experiments.

Defectivity of epilayers.

The defectivity of all epiwafers was measured by UVPL and DIC microscopy in a Lasertec SICA 88. Those epiwafers which comply with a 1,200 V epilayer specification (i.e. thickness: $10 - 14\ \mu\text{m}$, n-type doping concentration of $(0.8 - 2) \times 10^{16}\ \text{cm}^{-3}$) were further analyzed with regards to yield prediction assuming $5 \times 5\ \text{mm}^2$ die size and $3\ \text{mm}$ edge exclusion. Stacking faults (SF), polytype inclusions, particle inclusions, PL stacking faults as well as PL propagated SF were considered for the yield prediction. Figure 2 shows the defect distribution (of mentioned defect classes) and the yield prediction for epilayers grown on substrates from vendor A, B (in G10-SiC reactor) and vendor C (in G5WW C reactor).

The killer defect density of such 1,200 V epilayers varied between $0.13\ \text{cm}^{-2}$ and $0.94\ \text{cm}^{-2}$ for different substrate vendors and reactor types. Accordingly, the device yield varied from 81.5 % to 97.3 %, which is comparable to results from conventional ethene based epigrowth processes.

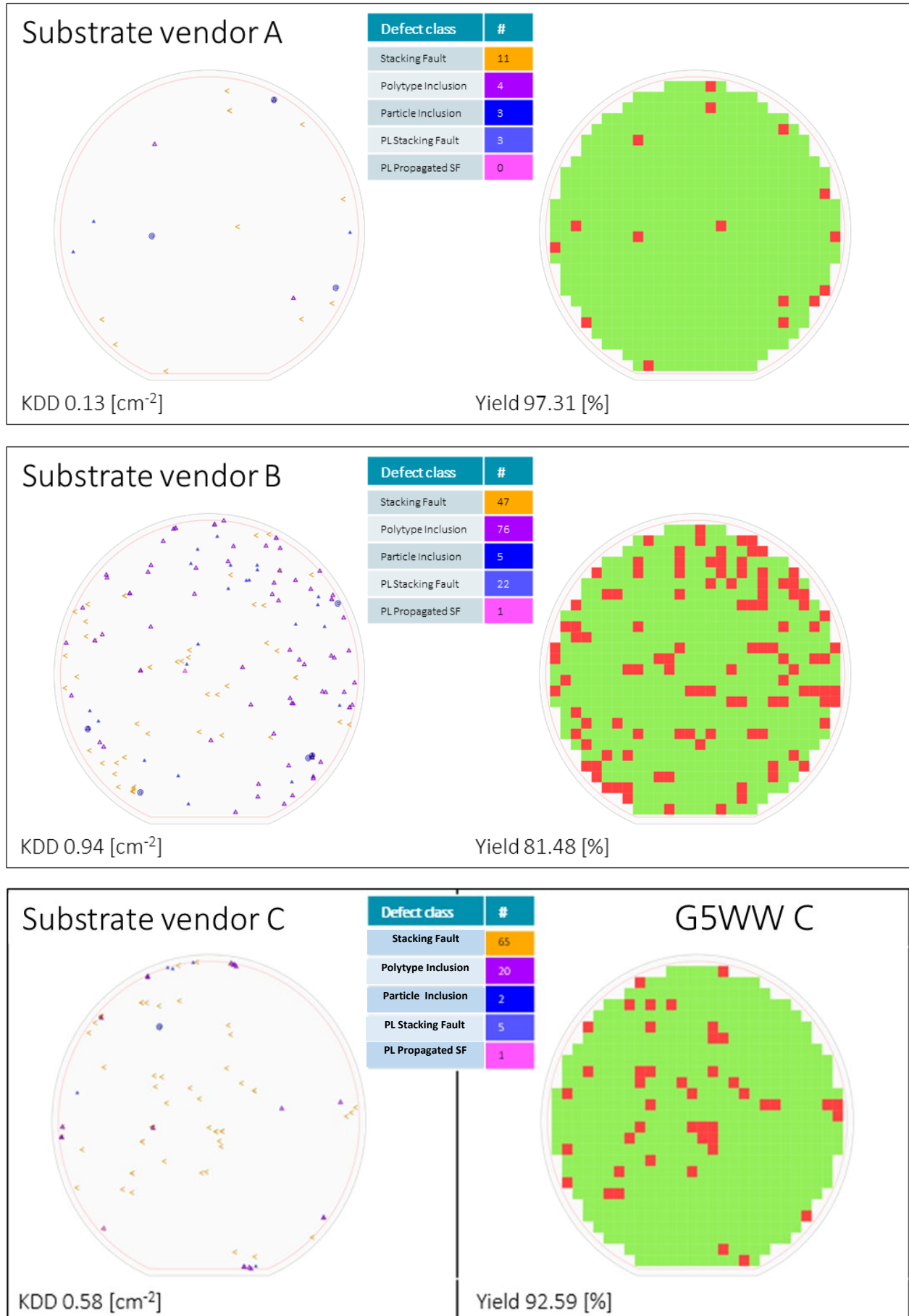


Fig. 2. Defect mappings and yield prediction for epilayers grown in different planetary reactors with methane precursor on substrates from different vendors.

Carrier lifetime in epilayers.

The *effective* minority carrier lifetime in 1,200 V epiwafers was measured by μ -PCD method without any sample preparation. *Effective* lifetimes are depending on the minority carrier lifetime in the epilayer (“bulk lifetime”), on surface recombination at the epilayer surface, and the epilayer thickness or distance to the highly doped substrate. The epilayer thickness of about 10 μm of all investigated epilayers is comparable and the as-grown surfaces were untreated, the *effective* lifetimes are an indicator for the “bulk lifetime” in the epilayer. Figure 3 shows a comparison of lifetime maps from epilayers grown in ethene- and methane-based epitaxy processes.

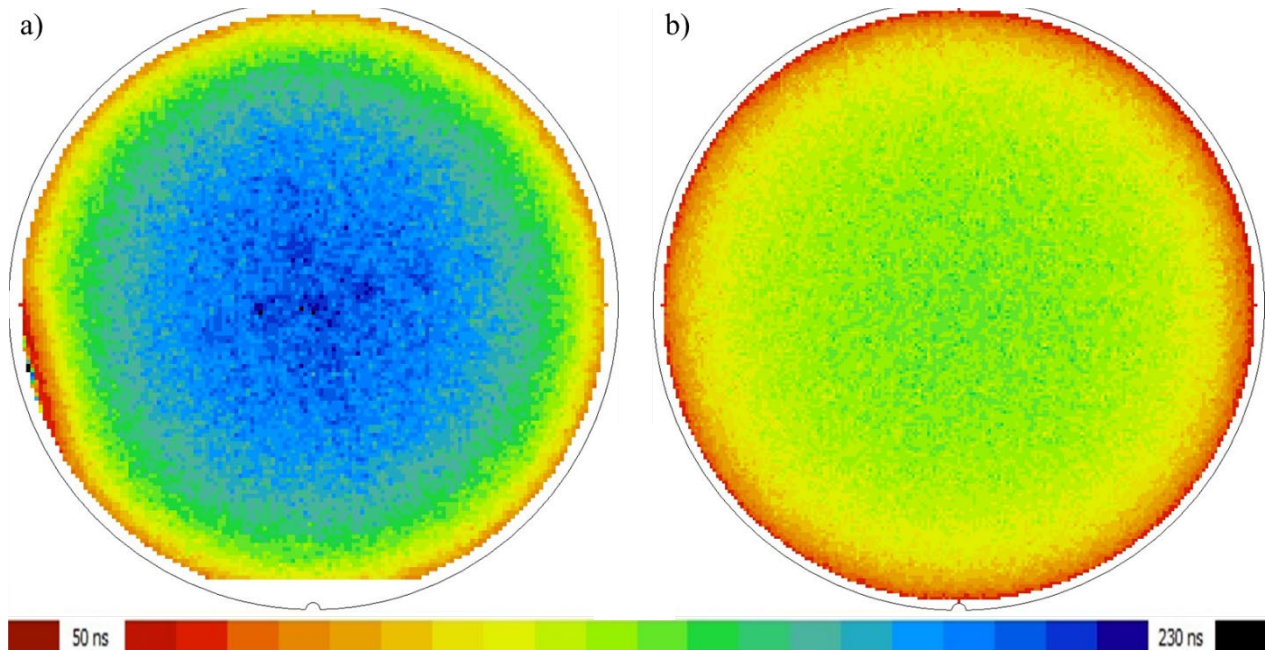


Fig. 3. *Effective* minority carrier lifetime mappings of 1,200 V epilayers grown with a methane based epigrowth process (a) and conventional ethene-based process (b) with the same color scale from 50 ns (red) to 230 ns (blue). Edge exclusion is 3 mm.

The *effective* carrier lifetime mappings show the typical radially symmetric lifetime distribution, which depends on the lateral thickness and doping concentration profiles of epilayers. No larger defects, like e.g. stacking faults, dislocation networks or defect clusters, are visible as “red spots” with shorter lifetimes. The mean *effective* minority carrier lifetime is 163 ns for the epilayer out of the methane-based process and 119 ns for the epilayer from ethene-based growth, i.e. the *effective* lifetime of the methane-based epilayer is slightly higher than that of the conventional epigrowth process. We measured more than 300 epiwafers with 1,200 V specification by μ -PCD and found that their mean *effective* lifetime is typically about 115 ns [6], which compares well to the calculated *effective* lifetime of 130 ns [6] for typical 1,200 V epilayers. We will further investigate if the methane based epigrowth can significantly increase the *effective* and bulk lifetime in the epilayer and the physical reasons. Point defect concentrations in the epilayers from different precursors and reactor types will be determined by deep level transient spectroscopy (DLTS).

IsoPure Si¹²C epilayers.

After developing a methane-based growth process with good epilayer quality at 30 $\mu\text{m}/\text{h}$ growth rate, isotopically enriched methane was used as precursor. Inserting methane with 99.99 % ¹²C into the reactor resulted in a ¹²C concentration of 99.96 % in the epilayer as shown in Fig. 4. This result indicates clearly that almost no carbon exchange takes place between the precursor species in the gas phase and the growing epilayer on one hand side and the SiC deposition in the planetary reactor on the other side.

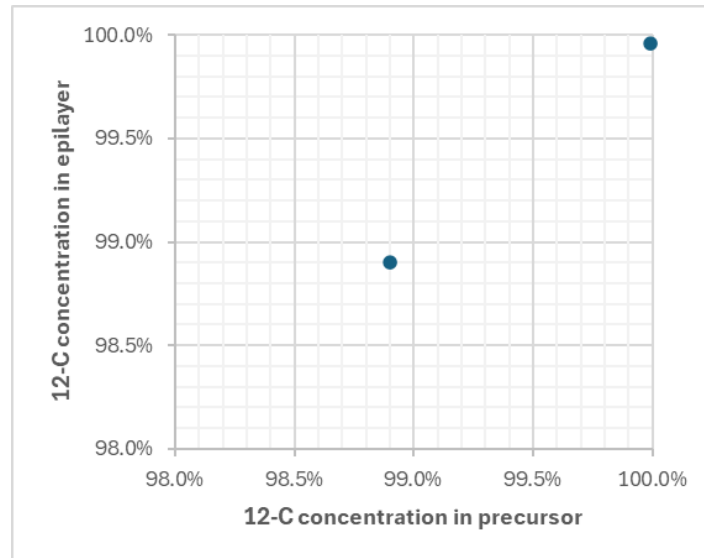


Fig. 4. Isotope concentration (determined by SIMS) for epilayers grown with different methane precursor, i.e. conventional methane and isotopically enriched methane.

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Summary

The conventional ethene precursor was substituted by methane precursor for comprehensive growth studies in AIXTRON planetary reactors G10-SiC and G5WW C on 150 mm SiC substrates. The growth rate, epilayer thickness and doping concentrations as well as the defectivity of epilayers was comparable for both methane- and ethene-based epigrowth processes. Furthermore, “IsoPure” epilayers with a ¹²C concentration of 99.96 % were grown for use in quantum-based applications. The point defect content of epilayers will be further investigated by DLTS measurements to deepen the understanding of the *effective* minority carrier lifetime. Further experiments will be done to increase the growth rate to more than 30 $\mu\text{m}/\text{h}$.

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